ATTACHMENT C

LIBBY LABORATORY MODIFICATIONS FOR TEM ANALYSES

LB-000016
LB-000019
LB-000028
LB-000029b
LB-000030
LB-000066c
LB-000085

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Request for Modification
To
Laboratory Activities
LB-000016

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, All project labs
Individual Lab Applicable forms – copies to: EPA, Volpe, CDM-Denver, Initiating Lab

Method (circle one/those applicable): TEM-AHERA, [TEM-ISO 10312], PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other:_____

Requester: Jeanne Orr Title: President
Company: Reservoirs Environmental, Inc. Date: December 2, 2002

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

Reason for Modification:
To optimize the efficiency of air sample analysis and to provide consistency in analytical procedures and data recording in the project laboratories.

Potential Implications of this Modification:
Modifications reflect changes necessary to clarify ISO requirements in relation to project-specific issues. No negative implications to these modifications are anticipated. Positive implications are consistency in procedures between and within project laboratories and documentation of those procedures.

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

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (complete Proposed Modification Section) Effective Date: HISTORIC

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by TEM analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):
Please see the attached for the description of the TEM-ISO clarifications/modifications

Technical Review: R.K. Mahony EMSL Date: 23 April 2003
(Laboratory Manager or designate)

Project Review and Approval: [signature] Date: 4 April 2003
(Volpe: Mark Raney)

Approved By: [signature] Title: Project Chemist Date: 3 April 2003
(USEPA: Mary Goldade)

1. **Modification:**
   The ISO method requirement is if the specimen grid exhibits more than approximately 10% obscuration on the majority of the grid openings, the specimen shall be designated as overloaded. A rejection criteria of >25% obscuration and <50% intact grid openings will be used for this project. The 25 % overload criteria resulted from various communications that took place 29 December 1999 between EPA Region 8, Camp Dresser McKee, Volpe Center, and Reservoirs.
2. **Modification:**

   ISO 10312 is a direct preparation method. If samples are visibly overloaded or contain loose debris and they have not been previously analyzed (the filter is whole) they will be prepared indirectly according to procedures described in ASTM D5755-95. If the sample has been previously analyzed or rejected in the microscope (section removed from the filter), prepare the sample indirectly according to EPA/540/2-90/005a by plasma ashing a portion of the original filter and depositing an aliquot on a secondary filter. Secondary filters will be analyzed according to the ISO counting rules for this project. Calculations are modified to contain a dilution factor. This indirect preparation procedure is embraced to enable the capture of data from samples that otherwise would be rejected.

3. **Clarification:**
   Stopping rules for ISO analyses are completion of the grid opening on which the 100th asbestos structure has been recorded, or a minimum of four grid openings. For this project, a maximum of ten grid openings will be read unless specifically instructed otherwise.

   If abundant chrysotile is present, the chrysotile count may be terminated at the end of the grid opening where the 100th chrysotile structure is counted. The analysis will continue recording amphibole fibers only until the remaining grid openings to be analyzed are completed. The grid opening location designation will be followed by a "*" to indicate the grid openings where only amphibole asbestos was recorded, i.e. K6*.
   This clarification in structure counting and recording is <u>to provide consistency in analytical procedures and data recording in the project laboratories.</u>

4. **Modifications and clarifications: Structure counting and recording**
   a. **Modification:** Non-asbestos structures are not being recorded. This project-specific modification stems from our need only to quantify contaminants of concern: the asbestos levels at a given sample location.
   b. **Modification:** The overall dimensions of disperse clusters (CD) and disperse matrices (MD) will not be recorded in two perpendicular directions. The matrix type and individual structures associated with the matrix or cluster will be recorded as described in the ISO method.
   c. **Modification:** Structures that intersect a non-countable grid bar will be recorded on the count sheet but excluded from the structure density and concentration calculations.
   d. **Modification:** If a structure originates in one grid opening and extends into an adjacent grid opening, providing that it does not intersect a non-counting grid bar, the entire length of the fiber is recorded.
   e. **Clarification:** If a structure intersects both a countable and a non-countable grid bar, the observed length of the structure will be recorded.

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

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Tuesday, April 22, 2003 11:09 AM
To: 'Mahoney, Ron'
Subject: FW: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017

LB-000015_rev (MR 4-4-03 email... LB-000016_rev (MR 4-4-03 email... LB-000017_rev (MR 4-4-03 email...

FYI

> -----Original Message-----
> From: Raney, Mark
> Sent: Friday, April 04, 2003 9:31 AM
> To: 'Beckham, Richard'; 'Goldade.mary@EPAmail.epa.gov'; 'mgoldade@peakpeak.com'
> Cc: Autio, Anni
> Subject: VOLPE Approved MODS: LB-000015, LB-000016, and LB-000017
>
>
> Volpe provides approval to revised MODs LB-000015, LB-000016, & LB-000017 as attached. The attached MODs include the following changes to the previous versions (received 4/1/03).
>
> * The date indicated in the "Effective Date" field was removed and replaced with "HISTORIC"
> * Under the "Description of Modification" section the following sentence was added "The purpose of the attached is to document permanent historic modifications & clarifications."
>
> If you have any questions as to these changes or the reason behind them let me know. Please proceed with distribution of the accepted versions of the attached for final hardcopy signature.
>
> Mark.
>
> > <<LB-000015_rev (MR 4-4-03 email).doc>> > > <<LB-000016_rev (MR 4-4-03 email).doc>> > > <<LB-000017_rev (MR 4-4-03 email).doc>>
>
>
> -----Original Message-----
> From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
> Sent: Tuesday, April 01, 2003 10:47 AM
> To: 'Goldade.mary@EPAmail.epa.gov'; 'RANEY@VOLPE.DOT.GOV';
> 'mgoldade@peakpeak.com'
> Cc: Autio, Anni
> Subject: FW: LB-000015, LB-000016, and LB-000017
>
>
> For your review and approval.
>
> - Richard Beckham
>
> -----Original Message-----
> From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
> Sent: Monday, March 31, 2003 6:11 PM
> To: Beckham, Richard
> Subject: LB-000015, LB-000016, and LB-000017
>
>
> Richard,
>
> These should be final. The only recent revision is the addition of the
> Effective Date. These need to go to Mark and Mary for their final blessing.

> <<LB-000015(rev 3_31_03).doc>> <<LB-000016 rev. (3_31_03).doc>>
> <<LB-000017 rev(3_31_03).doc>>
>
> R.K. Mahoney
> Senior Analyst
> Special Projects Coordinator
> EMSL Analytical, Inc.
> Westmont, NJ
> 800.220.3675, x1218
> rmahoney@emsl.com
>
> << File: LB-000015(rev 3_31_03).doc >> << File: LB-000016 rev. (3_31_03).doc >> << File: LB-000017 rev(3_31_03).doc >>

Mahoney, Ron

From: Raney, Mark [RANEY@VOLPE.DOT.GOV]
Sent: Wednesday, April 23, 2003 9:02 AM
To: 'Mahoney, Ron'
Subject: FW: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017

LB-000015(rev 3_31_03).doc LB-000016 rev. (3_31_03).doc LB-000017 rev(3_31_03).doc

Ron,

I almost forgot to forward you this....

See Mary's earlier email below, regarding EPA's approval for MODs LB-15, 16, & 17.

Let me know if you have any questions.

Mark.

----Original Message----
From: Goldade.Mary@epamail.epa.gov [mailto:Goldade.Mary@epamail.epa.gov]
Sent: Thursday, April 03, 2003 5:49 PM
To: Beckham, Richard
Cc: Autio, Anni; 'mgoldade@peakpeak.com'; 'RANEY@VOLPE.DOT.GOV'
Subject: EPA APPROVED CONDITIONAL: LB-000015, LB-000016, and LB-000017

Richard,
Mark will modify LB-000015, 16 & 17 to indicate that the Effective Date is: Historical.
EPA approves these mods with this changed completed.

"Beckham, Richard" <BeckhamRE@cdm.com>
To: Mary Goldade/EPR/R8/USEPA/US@EPA, '"RANEY@VOLPE.DOT.GOV"' <RANEY@VOLPE.DOT.GOV>, '"mgoldade@peakpeak.com"' <mgoldade@peakpeak.com>
cc: "Autio, Anni" <AutioAH@cdm.com>
Subject: FW: LB-000015, LB-000016, and LB-000017
04/01/03 08:47 AM

For your review and approval.

- Richard Beckham

----Original Message----
From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
Sent: Monday, March 31, 2003 6:11 PM
To: Beckham, Richard

Subject: LB-000015, LB-000016, and LB-000017

Richard,

These should be final. The only recent revision is the addition of the Effective Date. These need to go to Mark and Mary for their final blessing.
<<LB-000015(rev 3_31_03).doc>> <<LB-000016 rev. (3_31_03).doc>>
<<LB-000017 rev(3_31_03).doc>>

R.K. Mahoney
Senior Analyst
Special Projects Coordinator
EMSL Analytical, Inc.
Westmont, NJ
800.220.3675, x1218
rmahoney@emsl.com

(See attached file: LB-000015(rev 3_31_03).doc)(See attached file: LB-000016 rev. (3_31_03).doc)(See attached file: LB-000017 rev(3_31_03).doc)

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
>
>
>

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Request for Modification
To
Laboratory Activities
LB-000028

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:

All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable):TEM-AHERA, TEM-ISO 10312, PCM-NIOSH 7400, PLM-NIOSH 9002, EPA/600/R-93/116, ASTM D5755-95, EPA/540/2-90/005a, Other: All TEM Methodologies

Requester: R. K. Mahoney Title: Senior Analyst / Special Projects Coordinator
Company: EMSL Analytical, Inc. Date: 17 June 2003

Description of Modification:
This is a clarification pertaining to the re-analysis of TEM samples when some of the originally read grid openings in a sample selected for re-analysis have become unreadable. In the event that more than half of the originally read grid openings have become unreadable, select the closest adjacent sample from the same sample delivery group with adequate intact grid openings for re-analysis. If half or less of the original openings on the sample selected are unreadable, make note in the Comments box in Data Entry 1 of the TEM EDD as to which grid openings are unreadable, and proceed with analysis of the original sample.

Reason for Modification:
This clarification is intended to provide more complete TEM re-analysis data.

Potential Implications of this Modification:
There are no negative implications to this clarification.

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

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 17June 2003

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

Technical Review: R. K. Mahoney EMSL Date: 18 July 2003
(Laboratory Manager or designate)

Project Review and Approval: Mark Rance Date: 7/18/03
(Volpe: Project Technical Lead or designate)

Approved By: Mary Goldade Date: 6/24/03

Title: Project Chemist
(USEPA: Project Chemist or designate)

Mary Goldade

06/24/03 01:20 PM

To: "Beckham, Richard" <BeckhamRE@cdm.com>
cc: "Autio, Anni" <AutioAH@cdm.com>, 'Bill Egeland' <begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" <Bob.Shumate@battaenv.com>, "'brattin@syrres.com'" <brattin@syrres.com>, 'Charlie LaCerra' <clacerra@emsl.com>, "'corbin77@atc-enviro.com'" <corbin77@atc-enviro.com>, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>, 'Gustavo Delgado' <gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" <gfreeman@mastest.com>, "'jeanneorr@resienv.com'" <jeanneorr@resienv.com>, "'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>, "'m_szynskie@resienv.com'" <m_szynskie@resienv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, "'Raney@volpe.dot.gov'" <Raney@volpe.dot.gov>, "'rdemalo@emsl.com'" <rdemalo@emsl.com>, "'rhatfield@mastest.com'" <rhatfield@mastest.com>, "'rmahoney@emsl.com'" <rmahoney@emsl.com>, 'Shu-Chun Su' <scsu@delanet.com>, 'William Longo' <wlongo@mastest.com>
Subject: Re: EPA Approved w/ revisions MOD LB-000028

EPA approves Mod LB-000028 with revisions as attached.

LB-000028 (MG 6-24-03).

Mary Goldade
Regional Superfund Chemist
U.S. Environmental Protection Agency, Region 8
999 19th Street, Suite 300
Mail Code: 8EPR-PS
Denver, CO 80202

Phone: (303) 312-7024
Fax: (303) 312-6065
email: goldade.mary@epa.gov

"Beckham, Richard" <BeckhamRE@cdm.com>

"Beckham, Richard" <BeckhamRE@cdm.com>

06/23/03 08:42 AM

To: 'Charlie LaCerra' <clacerra@emsl.com>, 'Charlie LaCerra' <clacerra@emsl.com>, "'jeanneorr@resienv.com'" <jeanneorr@resienv.com>, "'rdemalo@emsl.com'" <rdemalo@emsl.com>, "'rmahoney@emsl.com'" <rmahoney@emsl.com>, 'William Longo' <wlongo@mastest.com>, "'rhatfield@mastest.com'" <rhatfield@mastest.com>, 'Bill Egeland' <begeland@mastest.com>, "'Bob.Shumate@battaenv.com'" <Bob.Shumate@battaenv.com>, "'Naresh C. Batta'" <ncbatta@battaenv.com>, 'Shu-Chun Su' <scsu@delanet.com>, "'corbin77@atc-enviro.com'" <corbin77@atc-enviro.com>, 'Gustavo Delgado' <gdelgado77@atc-enviro.com>, "'Garth B. Freeman'" <gfreeman@mastest.com>, "Autio, Anni" <AutioAH@cdm.com>, "'Raney@volpe.dot.gov'" <Raney@volpe.dot.gov>, "'brattin@syrres.com'" <brattin@syrres.com>, Mary Goldade/EPR/R8/USEPA/US@EPA, "'dmazzaferro@mastest.com'" <dmazzaferro@mastest.com>, "'mgoldade@peakpeak.com'" <mgoldade@peakpeak.com>, "'m_szynskie@resienv.com'" <m_szynskie@resienv.com>
cc:

Subject: MOD LB-000028

This MOD impacts all labs. For your review and comment.

- Richard Beckham

<<LB-000028.doc>>

From: "LaCerra, Charles" <CLaCerra@EMSL.com>
To: "Carr, Kim" <KCarr@EMSL.com>; "EMSL Mobile Lab - Asbestos" <mobileasbestoslab@EMSL.com>
Sent: Friday, July 18, 2003 5:57 AM
Attach: LB-000025_rev (MG 6-04-03 email).doc; LB-000027 (MG 6-24-03).doc; LB-000028 (MG 6-24-
Subject: FW: MODs: LB-000025, 26, 27 & 28

-----Original Message-----
From: Raney, Mark [mailto:RANEY@VOLPE.DOT.GOV]
Sent: Friday, July 18, 2003 7:53 AM
To: 'Beckham, Richard'; Autio, Anni
Cc: 'Goldade, Mary'; 'Goldade, Mary (HOME)'; 'Orr, Jeaane at Reservoir Env'; 'Mahoney, Ron'; 'Demalo, Rob (EMSL)'; 'LaCerra, Charles'
Subject: MODs: LB-000025, 26, 27 & 28

Richard,

LB-000025 (EMSL): Volpe provided approval (with revisions) on 6/18/03 & EPA approved on 5/14/03 (see emails and attachment below). I have yet to see a final version for signature. EMSL should finalize, sign and distribute for signature.

LB-000026 (EMSL): Approved and signed by both Volpe and EPA.

LB-000027 (RESI): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). RESI should finalize, sign and distribute for signature.

LB-000028 (EMSL): MOD provided on 6/23/03 via Richard Beckham, Approved by EPA (with revisions) on 6/24/03. Volpe concurs with EPA and herby provides approval with EPA's revisions (see attached). EMSL should finalize, sign and distribute for signature.

Please let me know if anyone has any questions.

Mark.

-----Original Message-----
From: Beckham, Richard [mailto:BeckhamRE@cdm.com]
Sent: Wednesday, July 16, 2003 5:30 PM
To: 'RANEY@VOLPE.DOT.GOV'; Autio, Anni
Subject: MOD Status

For MODs 27 and 28, I have email approvals from EPA, but have not been able
to locate approvals from Volpe. CDM received a hardcopy of 27 with an original signature from RESI, that was subsequently forwarded to Volpe on
7/8/3. (Did I miss an approval email?) To my knowledge, a hardcopy of 28
has not been prepared.

- Richard Beckham

-----Original Message-----
From: Raney, Mark
Sent: Wednesday, June 18, 2003 10:56 AM
To: 'Mahoney, Ron'
Cc: 'Anni Autio'; 'Mary Goldade'
Subject: RE: EPA Markups: MOD LB-000025

Ron,

I concur with Mary's comments below. I provide Volpe's approval for MOD LB-000025 with Mary's changes and the addition of an estimate of the number of samples involved (i.e,. < 20).

Thanks,

Mark.

-----Original Message-----
From: Mahoney, Ron [mailto:Rmahoney@EMSL.com]
Sent: Wednesday, June 04, 2003 9:27 AM
To: 'Mark Raney'

Cc: 'Anni Autio'; 'Mary Goldade'; CDM STAFF
Subject: FW: EPA Markups: MOD LB-000025

Mark,

Do you have any other comments for this mod? Mary asked for an estimate of
the number of samples involved, and we agreed on < 20. The number is more
likely < 10, but we've deceided to err on the conservative side.

If I can get your input, we can put this one to bed.

R.K. Mahoney
Senior Analyst
Special Projects Coordinator
EMSL Analytical, Inc.
Westmont, NJ
800.220.3675, x1218
rmahoney@emsl.com

-----Original Message-----
From: Mary Goldade [mailto:mgoldade@peakpeak.com]
Sent: Wednesday, May 14, 2003 6:32 PM
To: Beckham, Richard; 'Charlie LaCerra'; jeanneorr@resienv.com; rdemalo@emsl.com; rmahoney@emsl.com; 'William Longo'; rhatfield@mastest.com; 'Bill Egeland'; Bob.Shumate@battaenv.com; 'Naresh C. Batta'; 'Shu-Chun Su'; corbin77@atc-enviro.com; 'Gustavo Delgado'; 'Garth B. Freeman'; Autio, Anni; Raney@volpe.dot.gov; brattin@syrres.com; Goldade.mary@EPAmail.epa.gov; dmazzaferro@mastest.com; m_szynskie@resienv.com
Subject: EPA Markups: MOD LB-000025

Suggested changes to the MOD are attached.
Ron-Do you already have in hand an estimate regarding the actual number of
samples this affects (i.e., are you able to quantify the term "few/limited"?)
Thanks,
Mary

----- Original Message -----
From: "Beckham, Richard" <BeckhamRE@cdm.com>
To: "'Charlie LaCerra'" <clacerra@emsl.com>; <jeanneorr@resienv.com>; <rdemalo@emsl.com>; <rmahoney@emsl.com>; "'William Longo'" <wlongo@mastest.com>; <rhatfield@mastest.com>; "'Bill Egeland'" <begeland@mastest.com>; <Bob.Shumate@battaenv.com>; "'Naresh C. Batta'" <ncbatta@battaenv.com>; "'Shu-Chun Su'" <scsu@delanet.com>;

<corbin77@atc-enviro.com>; "'Gustavo Delgado'"
<gdelgado77@atc-enviro.com>;
"'Garth B. Freeman'" <gfreeman@mastest.com>; "Autio, Anni"
<AutioAH@cdm.com>; <Raney@volpe.dot.gov>; <brattin@syrres.com>;
<Goldade.mary@EPAmail.epa.gov>; <dmazzaferro@mastest.com>;
<mgoldade@peakpeak.com>; <m_szynskie@resienv.com>
Sent: Wednesday, May 14, 2003 3:28 PM
Subject: MOD LB-000025

> This MOD impacts only EMSL. For your review and comment:
>
> <<LB-000025.doc>>
> - Richard Beckham

<<LB-000025_rev (MG 6-04-03 email).doc>> <<LB-000027 (MG 6-24-03).doc>> <<LB-000028 (MG 6-24-03).doc>>
>
>
>

Mary Goldade

07/29/03 01:57 PM

To: Anni Autio
cc: Mark Raney
cc:
Subject: LB-000027 & LB-000028 are signed and mailed

Anni & Joe,
I have mail you the original copiew of the mods LB-000027 & LB-000028.
Several of the email approval pages were not provided. I attached them.

Mary Goldade
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Request for Modification
to
Laboratory Activities
LB-000029b

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Lynn Woodbury Title: Technical consultant
Company: Syracuse Research Corporation Date: December 7, 2006

Description of Modification:
Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses of air and dust. The general concepts presented in this modification may also be used for soil and water, but specific details regarding the frequency and interpretation of laboratory QC samples will need to be adjusted for these media.

Reason for Modification:
This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures.

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

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: _____
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable): _____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 4/25/07
(Volpe Project Technical Lead or designate)

Approved By: Mary Goldade Date: 4/25/07
(USEPA Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

QC Sample Type Definitions

There are three categories of TEM laboratory QC samples: Blanks, Recounts, and Repreparations.

Blanks

Lab Blank (LB) – This is a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

Recounts

Recount Same (RS) – This is a TEM grid that is re-examined within the same laboratory and by the same microscopist who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Same TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Recount Different (RD) – This is a TEM grid that is re-examined within the same laboratory but by a different microscopist than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Different TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Interlab (IL) - This is a TEM grid that is re-examined by a microscopist from a different laboratory than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Interlab TEM analyses for air and dust will be selected in accord with the procedure presented in Attachment 2.

Verified Analysis (VA) – This is a recount of a TEM grid (same grid openings) performed in accord with the protocol for verified analysis as provided in NIST (1994) (provided as Attachment 3). Verified TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Repreparations

Repreparation (RP) – This is a TEM grid that is prepared from a new portion of the same filter that was used to prepare the original grid. Typically this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter. Repreparations will be selected in accord with the procedure presented in Attachment 1.

Frequency

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Verified analysis	1%
Repreparation	1%
Interlab	0.5%
Total	**10%**

Each laboratory should prepare and analyze lab blank, recount (same, different and verified), and repreparation samples at the minimum frequency specified in the table above. The selection procedure and laboratory SOP for the selection of samples for the purposes of recounts and repreparation are provided in Attachment 1. Samples for interlab comparisons will be selected by EPA's technical consultant (SRC) in accord with the selection procedure and laboratory SOP provided in Attachment 2.

Procedure for Evaluating QC Samples and Responses to Exceptions

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: The procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

Lab Blanks.

There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

Recounts.

All recount samples (same, different, verified, and interlab) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. All structure enumeration and measurements will adhere to the established project-specific documentation presented in LB-000016A and LB-000031A. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 3).

Measurement parameter	Concordance Rule
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent) For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent). For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more areas of discordance, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Laboratory Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

Repreparations.
Repreparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 4. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

QC Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with ≥1 asbestos structures	0% - 0.1%	0.2% - 0.5%	>0.5%
Recounts	Concordance on LA count	>95%	85-95%	<85%
	Concordance on type (chrysotile vs. amphibole)	>99%	95%-99%	<95%
	Concordance on LA length	>90%	80%-90%	<80%
	Concordance on LA width	>90%	80%-90%	<80%
Repreps	Concordance on LA concentration/loading	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

ATTACHMENT 1

Selection Procedure and Laboratory SOP for Recounts (RS, RD, VA) and Repreparations (RP)

Selection Procedure

As specified in the Frequency section above, the frequency of Recount Same (RS) should be 1%, the frequency of Recount Different (RD) should be 2.5%, the frequency of Verified Analyses (VA) should be 1%, and the frequency of Repreparations (RP) should be 1%, corresponding to a total within-laboratory QC frequency of 5.5% for these analysis types. This is approximately 1 QC sample per 20 field samples. Based on this frequency, it is possible to determine which laboratory job(s) will have one or more samples selected for recount analysis or repreparation.

For those laboratory jobs in which a recount or repreparation sample is to be selected, the analyst should record the total number of structures observed in each sample. The sample(s) selected for recount or repreparation should be those within the laboratory job with the highest number of structures per grid opening (GO) area examined (calculated as the number of GOs evaluated * the GO area). When selecting samples for repreparation, if possible, preferentially select samples in which the total number of GOs is 40 or less. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the selection of samples with 40 GOs or less will reduce analytical costs associated with repreparations. When selecting samples for recount, it is not necessary to impose a minimum or maximum number of GOs because concordance is evaluated on a GO and structure basis, rather than a concentration basis. If all samples within the laboratory job are non-detect, a non-detect sample may be selected. A non-detect sample should be preferentially selected, every 10th selection.

This selection procedure will ensure that the recount analyses and repreparations yield a dataset best suited to assess concordance[1].

Laboratory SOP for Recount Analyses

1. For recount samples, re-analyze the selected sample in accord with the appropriate procedures for each type of recount (RS, RD, or VA). If more than 10 GOs were evaluated in the original analysis, the original analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the recount analysis. The original analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures per GO area examined, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated.

2. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Recount Same", "Recount Different", or "Verified Analysis", as appropriate. Be sure that the grid and GO names match exactly with the names evaluated in the original analysis (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the original analyst or laboratory director to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. If there is one or more discordant GOs between the original analysis and the recount analysis, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

[1] It should be noted that this selection procedure will tend to result in the preferential selection of samples with the highest air concentration/dust loading values. Thus, summary statistics based on laboratory QC samples may tend to be biased high.

4. Submit the recount TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

Laboratory SOP for Repreparations

1. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.

2. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.

3. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".

4. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

ATTACHMENT 2

Selection Procedure and Laboratory SOP for Interlabs (IL)

Selection Procedure

1. On the 1st of each month, EPA's technical consultant (SRC) will compile a list of all samples for which air and dust TEM results (ISO+AHERA+ASTM) were uploaded into Libby V2 Database in the preceding month (e.g., on November 1st, specify a date range of Oct 1-31, 2005). The Libby V2 Database query will be based on the upload date rather than the analysis date to ensure that analyses with an upload in a different month as the analysis date were not excluded[2].

2. Identify the target number of air and dust interlab samples needed to meet the QC requirements for interlabs specified in the Frequency section above (0.5%). This is accomplished by multiplying the desired interlab frequency (0.5%) by the total number of air and dust analyses performed in the preceding month. For example, 178 TEM air analyses in October 2005 * 0.5% = 0.89 (which is rounded up to 1). At a minimum, at least one air and one dust sample will be selected for interlab analysis.

3. For each medium (air and dust), rank order the TEM analyses from the preceding month on the total number of LA structures per GO area examined (calculated as the number of GOs evaluated * the GO area). Selecting from analyses with a high number of LA structures per GO area examined increases the likelihood that the GOs evaluated as part of the interlab analysis will have one or more LA structures.

4. Exclude samples in which the total number of GOs is more than 40 GOs[3]. Exclude any samples that have already been selected for interlab evaluation previously.

5. Select the appropriate number of air and dust interlab samples from the available TEM analyses for which the total number of LA structures per GO area examined is higher than 0 (i.e., LA detects). If the total number of samples with LA detects is equal to the desired number of interlab samples, select all detected samples for interlab analysis. If the total number of samples with LA detects is less than to the desired number of interlab samples, select non-detect samples for interlab analysis. If the total number of samples with LA detects is higher to the desired number of samples, interlab samples will be selected to represent multiple laboratories, selecting those samples with the highest number of LA structures per GO examined first. EPA's technical consultant (SRC) will keep a running total of the number of samples selected by laboratory to ensure that the long-term frequency of interlabs for each laboratory is generally similar.

6. Submit list of selected interlab samples to the CDM Laboratory Manager.

7. Each month, the CDM Laboratory Manager will provide each laboratory with the list of samples selected for Interlab analysis.

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[2] Consider the case where the TEM analysis for sample X-12345 was performed on September 22 and the results were uploaded on October 3. The interlab selection query performed on October 1, if limited to all results analyzed from September 1-30, would not capture the results for X-12345 because they had not yet been uploaded. The interlab selection query performed on November 1, limited to all results analyzed from October 1-31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

[3] Because all interlabs will be reprepared, these interlab repreparation samples will also be evaluated for concordance with the original sample. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the focusing on samples with 40 GOs or less will reduce analytical costs associated with repreparations.

Laboratory SOP

At the Originating Laboratory:

1. Upon receipt of the interlab sample list from the CDM Laboratory Manager, locate the appropriate sample filter. If less than ¼ of the sample filter is available, contact the CDM Laboratory Manager to identify an interlab replacement sample.

2. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.

3. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.

4. Record the orientation of each grid using the instructions for grid orientation specified in NVLAP (see Attachment 5).

5. When performing the TEM analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.

6. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".

7. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

8. Identify which laboratory will perform the interlab analysis in accord with the following table:

Originating Lab	Lab for Interlab Sample #1	Lab for Interlab Sample #2	Lab for Interlab Sample #3	Lab for Interlab Sample #4	Lab for Interlab Sample #5	Lab for Interlab Sample #6...
Hygeia	Batta	MAS	RESI	EMSL-L	EMSL-W	Repeat... (beginning with the Lab identified for Sample #1)
Batta	MAS	RESI	EMSL-L	EMSL-W	Hygeia	
MAS	RESI	EMSL-L	EMSL-W	Hygeia	Batta	
RESI	EMSL-L	EMSL-W	Hygeia	Batta	MAS	
EMSL-L	EMSL-W	Hygeia	Batta	MAS	RESI	
EMSL-W	Hygeia	Batta	MAS	RESI	EMSL-L	

EMSL-L = EMSL, Mobile Lab in Libby
EMSL-W = EMSL, Westmont

9. If more than 10 GOs were evaluated in the repreparation analysis, the repreparation analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the interlab analysis. The repreparation analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures, which may be analyzed in the event that the selected GO is damaged and cannot be re-evaluated.

10. Ship the grid(s) for the interlab sample to the appropriate laboratory using standard chain of custody procedures. For each interlab sample, include a list of which GOs should be evaluated for each grid. The names of the grid and GOs provided on the chain of custody form should match exactly with those recorded in the original TEM data recording spreadsheet (including dashes, underscores, and spaces).

11. After the interlab laboratory has completed the interlab analysis, it will request copies of the hard copy laboratory benchsheet(s), the grid opening sketches, and TEM file for each interlab sample.

12. If areas of discordance are noted, the senior laboratory analyst from the interlab laboratory will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

At the Interlab Laboratory:

1. For each grid provided for interlab analysis, place the grid into the TEM grid holder ensuring that the grid orientation matches that which was specified by the originating laboratory (see Attachment 5 for details).
2. For the 10 GOs identified for interlab analysis, perform TEM analysis using the analysis method and counting rules specified on the chain of custody. Be sure that the grid and GO names match exactly with the names provided on the chain of custody (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the originating laboratory to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. When performing the TEM interlab analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.

4. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Interlab".

5. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

6. Contact the originating laboratory to request copies of the hard copy laboratory benchsheet(s), grid opening sketches, and TEM file for each interlab sample.

7. Perform a verified analysis using the procedures presented in NIST (1994) (provided as Attachment 3).

8. Assess the between-laboratory concordance, both on a GO-by-GO basis and on a structure-by-structure basis, using the Libby-specific recount concordance rules. If areas of discordance are noted, the senior laboratory analyst will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

9. Summarize the results of the verified analysis and document any changes in laboratory procedures or analyst training that were implemented to address noted discordances. Provide a copy of this report to EPA Chemist and the CDM Laboratory Manager.

10. Ship the grid(s) back to the originating lab.

ATTACHMENT 3

Airborne Asbestos Method:
Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy-Version 2.0.
NIST (1994)

NISTIR 5351

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0

Shirley Turner
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March 1994

U.S. DEPARTMENT OF COMMERCE
Ronald H. Brown, Secretary

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS
AND TECHNOLOGY
Arati Prabhakar, Director

Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

2. Terminology

2.1 *Definitions:*

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard:*

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos- containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*[1]--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*[2]--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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[1]Code Fed. Reg. **1987**, *52* (No. 210), 41826-41905.

[2]ISO 10312 **1993**, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*--the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*--structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true postive-unmatched)*--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*--structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*--reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted *vs* time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values ≥ 0.85 and FP/TNS values ≤ 0.05. The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2-- The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3-- The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4-- The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight foward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5-- The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannnot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator } 1) + TPU(\text{Operator } 2) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6-- The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

APPENDIXES

(Nonmandatory Information)

X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: _____ Date: _____

Grid slot: _____ Verifying Analyst: _____

Grid square: _____

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

*The values for these items will be the same for both analyses.

Test Report Form (continued)

1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

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

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1	/	TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

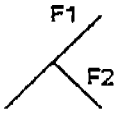
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

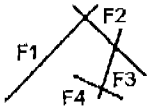
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr

a

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

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

b

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr

c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1			1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr

b

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

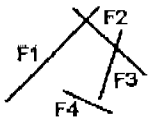
c

Fig. X2.8 (caption on previous page).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3			1	Chr

Analyst 2

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

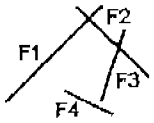
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

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

b

Fig. X2.9 (caption on previous page)

X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

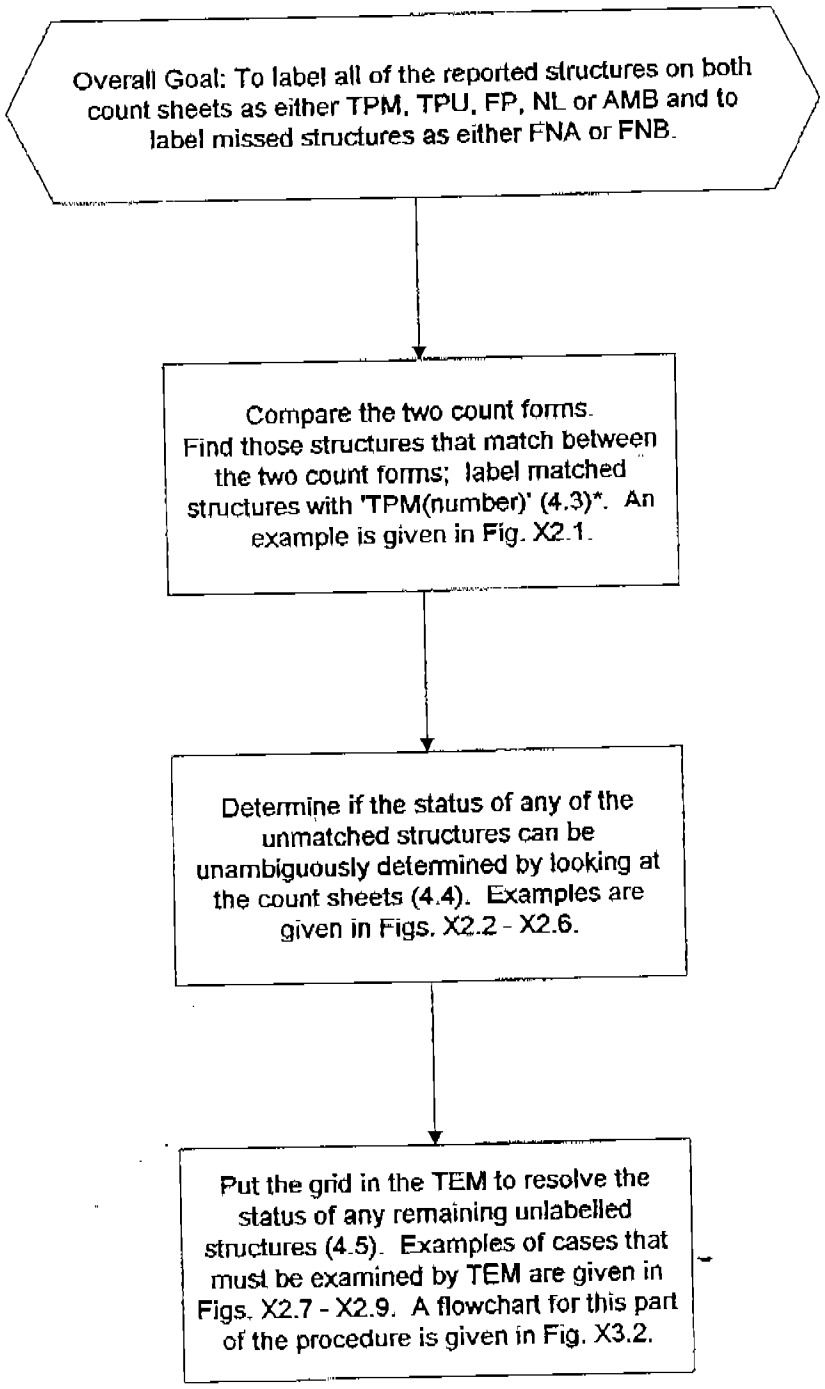


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst.
*Numbers in parentheses in each block refer to the item number in the procedure.

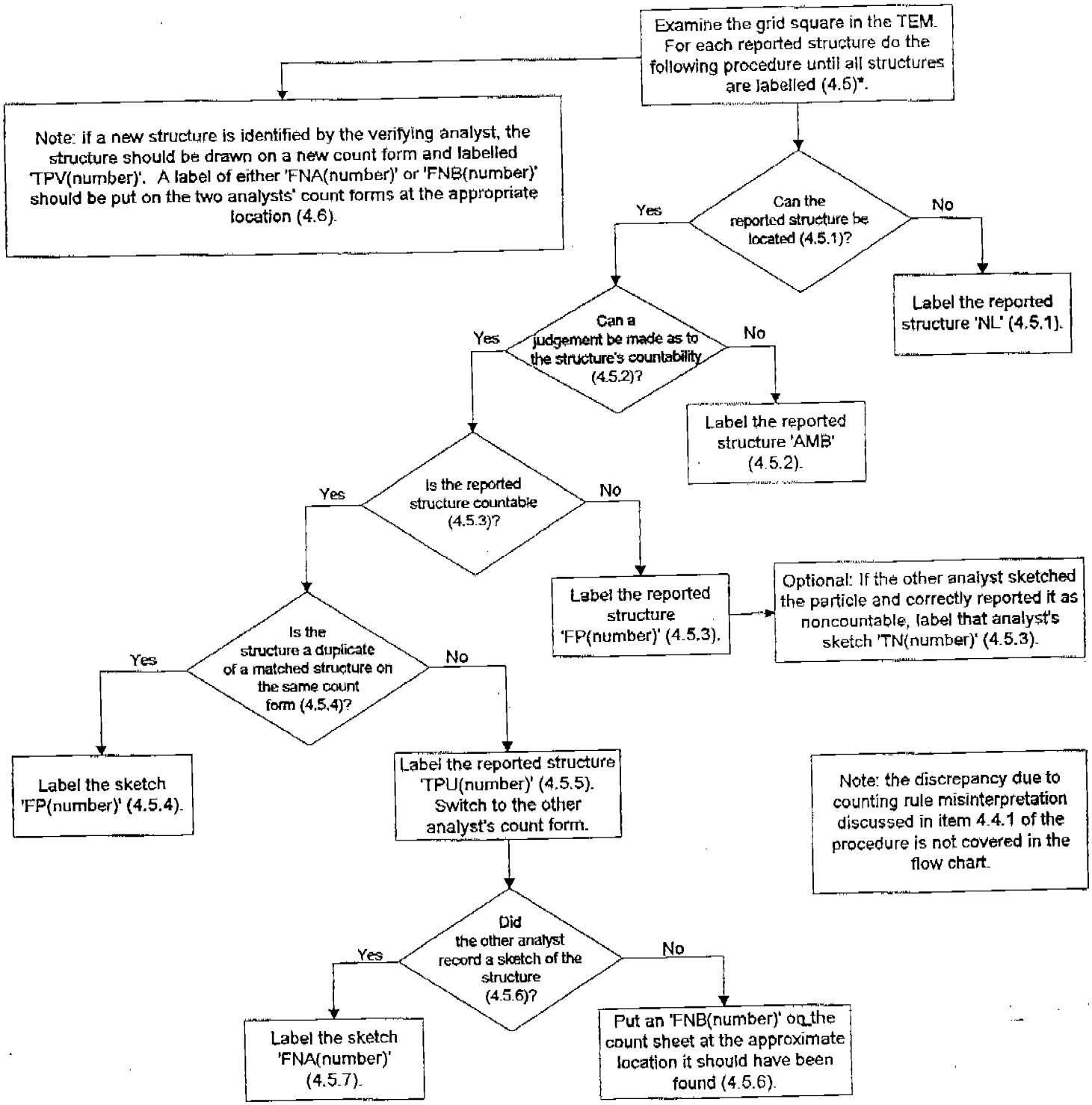


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

ATTACHMENT 4

Statistical Comparison of Two Poisson Rates

1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the repreparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from repreparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation
S1 = Sensitivity of first evaluation
N2 = Fiber count in second evaluation
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1
Rate 2 = N2 · S2
Ratio = Rate 1 / Rate 2

$$Lower\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1}{N2+1}\right) / F\left[\frac{1+\gamma}{2}; 2 \cdot N2 + 2, 2 \cdot N1\right]$$

$$Upper\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1+1}{N2}\right) \cdot F\left[\frac{1+\gamma}{2}; 2 \cdot N1 + 2, 2 \cdot N2\right]$$

where γ is the confidence interval (e.g., 0.95) and F[δ; df1, df2] is the 100δth percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is > 1, then it concluded that rate 1 is greater than rate 2 at the 100(1-γ)% significance level. If the upper bound of the ratio is < 1, then it concluded that rate 1 is less than rate 2 at the 100(1-γ)% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1-γ)% significance level.

<u>Example:</u>

N1 = 4 structures
S1 = 0.0001 $(cc)^{-1}$
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures
S2 = 0.001 $(cc)^{-1}$
Rate 2 = 6 · 0.001 = 0.006 s/cc

γ = 0.95

$$Lower\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4}{6+1}\right) / F\left[\frac{1+0.95}{2}; 2\cdot 6+2, 2\cdot 4\right] = 0.014$$

$$Upper\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4+1}{6}\right) \cdot F\left[\frac{1+0.95}{2}; 2\cdot 4+2, 2\cdot 6\right] = 0.281$$

In this example, because the upper bound of the ratio is < 1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

ATTACHMENT 5

NVLAP Airborne Asbestos Proficiency Test 98-2:
Grid Orientation

Instructions for Form 1

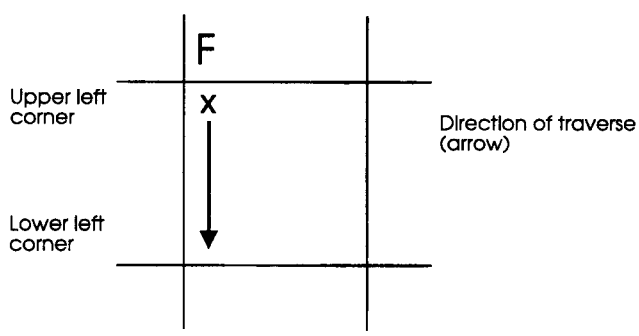
The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on *Form 1.* Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. ***Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted.*** See the example on *Form 1.*

2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.

3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1.* Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).

4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



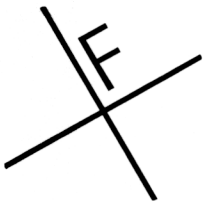
The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.)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

NVLAP Lab Code: _____

Form 1. ***Grid Orientation***

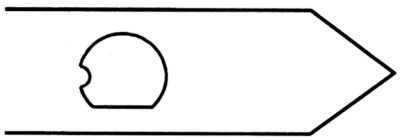
1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

EXAMPLE:



2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

EXAMPLE:



ATTACHMENT 6

Grid Opening Template for Sketching the Relative Position of Observed Structures

STRUCTURE LOCATIONS WITHIN GRID OPENING

****NOTE: Sketches only need to be completed for interlab analyses and repreps associated with interlabs*

Lab Name: _____ Lab Job Number: _____

Index ID: _____ Lab Sample ID: _____

Lab QC Type (circle one): Reprep for interlab Interlab

Grid: _____ Grid Opening: _____

upper left corner

traverse direction

Comments:

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)

Request for Modification
to
Laboratory Activities
LB-000066c

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other:_____

Requester: W. Brattin Title: Technical Consultant
Company: Syracuse Research Corporation Date: 09/11/2007

Description of Modification:
This temporary modification applies to all investigative samples (as defined by the most recent version of LB-000053) evaluated at the Libby Superfund site. Based on this temporary modification, all analytical laboratories shall: 1) begin to utilize the structure comment field to further characterize particles with regard to the levels (presence/absence) of the sodium and potassium peaks observed in the EDS spectrum; 2) record on the data sheets all NAM particles that are "close calls" (defined in attachment 1); 3) increase the frequency that EDS spectra are saved for "LA" and "close call" structures; 4) increase the frequency that photographic images of particle morphology are recorded for "LA" and "close call" structures, and 5) utilize the comment field to record mineral type of each recorded particle, including LA, OA, C and "close call" NAM particles,

Reason for Modification:
Studies of asbestos from the mine in Libby indicate that the asbestos spans several different mineralogical classes, including winchite and richterite (these are the primary forms) as well as tremolite and possibly actinolite (these are minor forms) (Meeker et al, 2003). Consequently, all analytical laboratories supporting the Libby project are currently directed to classify as "LA" any particle in an investigative sample that a) meets morphological requirements (e.g., length ≥ 0.5 um, aspect ratio ≥ 3:1), b) has an SAED diffraction pattern that is consistent with amphibole, and c) has an EDS spectrum that is consistent with the range of mineral forms observed in the mine in Libby (USEPA 2005). To date, this method for designating "LA" to a particle has worked well for samples collected at the Libby Site. However, a recent project that included collection of air samples from locations outside of Libby highlighted a potential limitation of this approach. That is, tremolite and actinolite are included in the "LA" suite and are found in Libby, but these types of fibers may also occur as the result of releases from sources that are not related to the mine in Libby (e.g., commercial products or natural sources). Also, some other minerals (e.g., pyroxenes) are sometimes difficult to distinguish from actinolite and tremolite (Bern et al. 2002). Because mineralogical data may or may not inform our understanding of the toxicity of LA, delineating amongst these mineral types is desirable at this stage of data collection. Therefore, the primary focus of this temporary modification is to collect more detailed data on the frequency of occurrence of sodium and potassium-containing particles both for samples from Libby and for samples from other locations.

Potential Implications of this Modification:
This temporary modification does not change any current procedures other than to require more detailed recording of data on particles observed under TEM. These additional requirements are not associated with a significant increase in time or cost of analysis. Hence, there are no negative implications of the modification.

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

Duration of Modification (circle one):

Temporary Date(s): 09/12/2007 until notified

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: _____

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

See Attachment 1

Note: This modification (LB-000066c) **supersedes** LB-000066b.

Technical Review: _____ Date: _____

(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 9/12/07

(Volpe: Project Technical Lead or designate)

Approved By: _____ Date: 9/11/07

(USEPA: Project Chemist or designate)

REFERENCES

Bern A, Meeker G, Brownfield I. 2002. Guide to Analysis of Soil samples from Libby, Montana for Asbestos Content by Scanning Electron Microscope and Energy Dispersive Spectroscopy. U. S. Geological Survey Administrative Report. October 17, 2002.

Meeker GP, Bern AM, Brownfield IK, Lowers HA, Sutley SJ, Hoeffen TM, and Vance JS. 2003. The Composition and Morphology of Amphiboles from the Rainy Creek Complex, Near Libby Montana. American Mineralogist 88:1955-1969.

USEPA. 2005. EDS Spectra Characteristic Study for Libby-Type Amphiboles. Report prepared by Syracuse Research Corporation, Denver CO, for USEPA, Region 8, Denver CO. March 15, 2005.

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

ATTACHMENT 1

1. Continue to classify structures as LA, OA, or C in accord with current procedures.

2. For all NAM particles that were "close calls" (i.e., they required careful assessment to determine they were not LA or OA), record the NAM particle on the bench sheet. Be sure to place a zero in the "total" column to ensure the particle is not counted as an asbestos fiber. NAM particles such as vermiculite, biotite, hydrobiotite, gypsum, titanium and other minerals that are clearly not amphibole should not be recorded.

3. For all particles that are recorded (including NAMs), use the structure comment field to record one of the following comments:

Code	Meaning
NaK	Na and K are both clearly present
NaX	Only Na is clearly present
XK	Only K is clearly present
XX	Na and K are not clearly present

4. For all particles that are recorded, whenever possible, use the structure comment field to identify a probable mineral classification. Use the designation "WRTA" (winchite/richterite/tremolite/actinolite) to indicate a particle that is consistent in morphology and chemical composition with a particle that is likely to have originated from the vermiculite mine in Libby. This will include most NaK particles and may include some NaX and some XK particles. It is unlikely that this will include any XX particles. For all other particles, use the following codes:

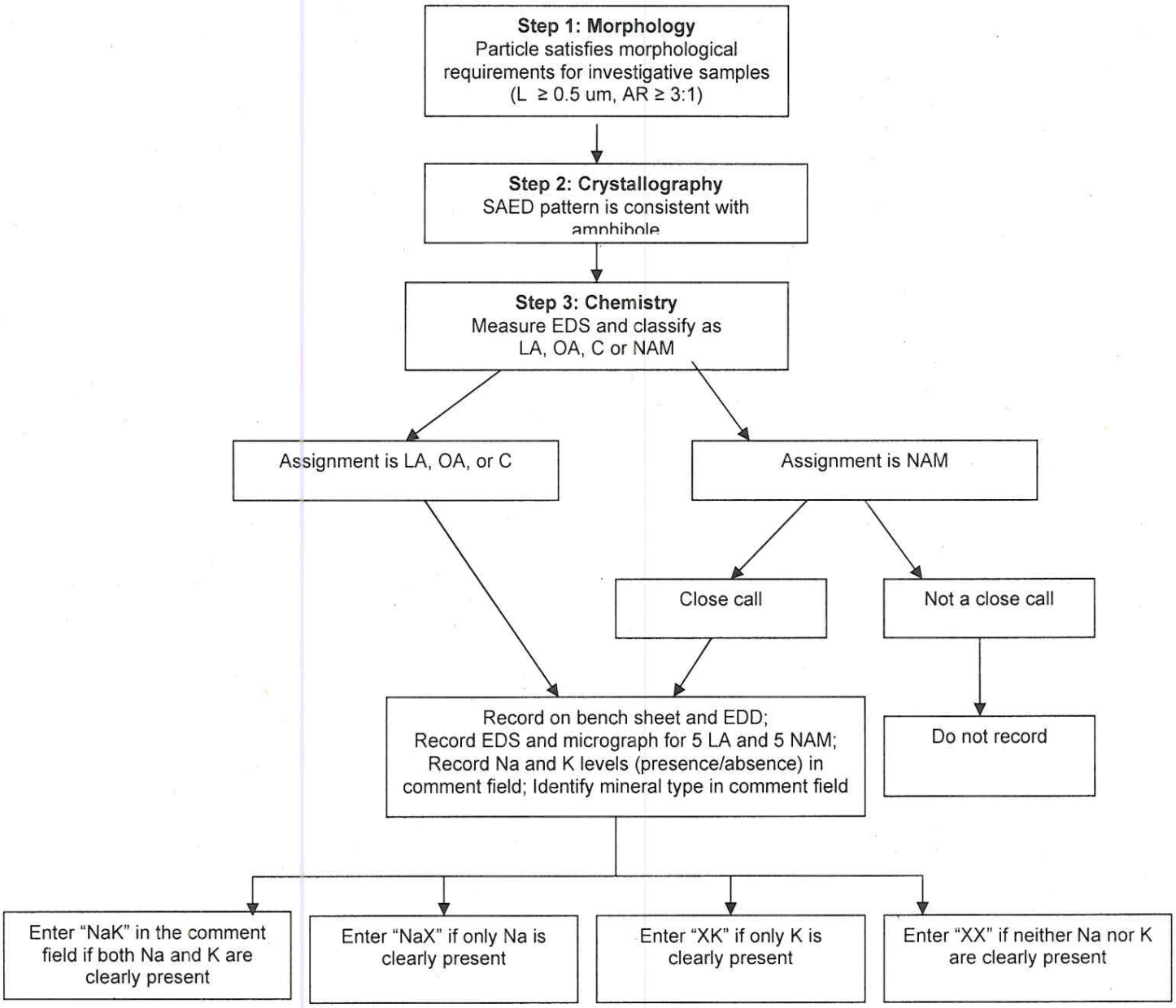
AC – actinolite
TR – tremolite
AT – actinolite/tremolite (too close to call)
AM – amosite
AN – anthophyllite
CR – crocidolite
PY – pyroxene
UN – Unknown

5. Increase the frequency that EDS spectra are recorded (saved). For each sample, record the EDS for each LA and each "close call" particle, up to a maximum of 5 LA and 5 "close call" particles per sample. To the extent practical, collect the EDS spectrum for a sufficient length of time that key peaks (e.g., sodium, potassium, aluminum), if present, can be clearly distinguished from background. Be sure that each EDS spectrum that is recorded can be linked to a specific particle in the EDD.

6. Increase the frequency that photomicrographic images of particle morphology are collected. For each particle for which an EDS spectrum is collected (up to 5 LA and 5 "close call" NAM, as discussed above), also record a photomicrograph of the same structures. Use the structure-specific comment field to record the photo identification number of each structure that is photographed. Convert all photographs to high quality electronic images (e.g., by scanning), and transmit the photos to CDM for evaluation.

7. Figure 1 provides a flow chart that summarizes the process implemented by this temporary modification.

FIGURE 1
FLOW CHART SUMMARIZING THIS TEMPORARY MODIFICATION



Request for Modification
to
Laboratory Activities
LB-000085

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): TEM-AHERA TEM-ISO 10312 PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 ASTM D5755 EPA/540/2-90/005a SRC-LIBBY-03
Other: All TEM and SEM Methods supporting Libby site investigative or Libby Action Plan (LAP) sample analysis

Requester: Mary Goldade Title: Senior Environmental Scientist/Chemist
Company: Environmental Protection Agency, Region 8 Date: April 2, 2008

Description of Modification:
Laboratories conducting transmission electron microscopy (TEM) or scanning electron microscopy (SEM) analysis in support of either the Libby Site (all operable units, including Troy) or Libby Action Plan shall perform analysis of a reference standard to calibrate the energy dispersive x-ray spectrometry (EDS) analysis. The reference standard, a glass material referred as BIR-1G, was created by the USGS. It is recommended for use for Libby Amphibole analysis because it contains sodium (Na) and potassium (K) at known levels. Na and K are important elements used in Libby Amphibole identification by EDS. The BIR-1G standard was freezer-milled by EMSL to create particles for EM analysis. While generation of thin sections of the BIR-1G using a microtome was not feasible due to the expense, analysis of the BIR-1G in particulate form is useful in standardizing the elemental measurements of the EDS and understanding the inherent variability in the EDS measurements.

The BIR-1G shall be tested daily (on days that the TEM scope is used for analysis of Libby samples) and must meet acceptance criteria prior to analysis of any field samples. Laboratories shall record the calibration information in accord with Attachment 1. As seen, not only does Attachment 1 provides the details for populating the electronic disk deliverable (EDD) used in recording the calibration information, but Attachment 1 also describes the process for generating acceptance criteria for the BIR-1G standard for each individual instrument.

Reason for Modification:
The modification provides for a standardized process for performing and recording calibration standards for EDS during Libby Amphibole analysis.

Potential Implications of this Modification: There are no negative implications to this modification. Positive impacts include a standardized process for: (1) daily calibration of a standard for the EDS used in Libby Amphibole identification; (2) reporting results of BIR-1G measurements; and (3) generating acceptance criteria for the BIR-1G standard over time.

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

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **SUPERCEDES** _____

Duration of Modification (circle one):
Temporary Date(s):_____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: April 30, 2008
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

_____

_____

Technical Review: _____N/A_____Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____Date:_____
(Volpe: Project Technical Lead or designate)

Approved By:_____Date: _____
(USEPA: Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

ATTACHMENT 1

Analyzing the BIR-1G Standard

- The BIR-1G standard shall be tested daily (on days that either the SEM or TEM microscope is used for analysis of Libby samples), prior to analyzing any field samples. Analyze for the compounds Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, TiO_2, MnO, and FeO. It is suggested that the reference publication for BIR-1G be reviewed. It is available in Volume 2 of the Analytical Guidance Documents, Tab 35, provided by CDM.
- Set up TEM instrument and orient for typical Libby field samples.
- Record the TEM instrument details in the BIR-1G Electronic Data Deliverable (EDD) spreadsheet (see most recent version of Excel file "BIR-1G EDD.xls"). Note: Use one spreadsheet per TEM instrument.
- For each daily BIR-1G evaluation, select one particle and record the measured weight % for each compound ***as oxide weight %*** in the BIR-1G EDD. Note: When recording oxide weight %, enter results as a percentage not fractions (i.e., for 30%, enter 30 not 0.3).
- When selecting particles for analysis:
    - Choose particles in the middle of the grid opening and in the center of the grid.
    - Particles should not be in close proximity to the grid bar or neighboring particles.
    - Randomly select particles within different grid openings for each analysis.
- For selected particles, focus the beam on the thin edge, not the center of the particle.
- Continue analysis until a maximum peak height count of at least 1,000 is achieved for silicon (Si). This total Si count should be sufficient to achieve optimum instrument testing conditions. It is recognized that this total Si count may not be equivalent to typical analytical conditions for field samples.
- On a monthly basis, the EDD for each TEM instrument should be provided to EPA (or designated contractors).

Acceptance Criteria

- Acceptance criteria will be TEM instrument- and element-specific and will be derived from measured results.
    - Results that are within ± 1 standard deviation of the nominal will be ranked as acceptable.
    - Results that are outside ± 1 standard deviation but within ± 2 standard deviations of the nominal will be ranked as within the warning level.
    - Results that are outside ± 2 standard deviations of the nominal will be ranked as a failure.
- The potential bias of measured results will be assessed based on a frequency evaluation of results above and below the nominal.
- As needed, EPA will re-evaluate and revise the acceptance criteria to optimize program goals.

Corrective Action

In the event that analysis results of the BIR-1G fall outside of the acceptance criteria, there should be a structured, progressive response. First, confirm that the detector/x-ray system has satisfied the acceptance criteria in the past. Next, confirm that the settings for the x-ray analysis software are correct (e.g. bias, scale). Finally, de-ice the LN2 dewar (unless it is a dry system) and carefully clean the window.

If these actions fail to rectify the problem, it will probably be necessary to send the detector/x-ray out to be serviced. The actions taken by the servicing company may include such things as baking the detector, renewing the vacuum in the dewar, checking the pre-amp or actual x-ray system for hardware defects, or replacing the crystal and/or FET (field effect transistor). In most instances the fault will not lie in the window unless the integrity of the window is compromised.

Upon the return and re-installation of the detector, re-run the BIR-1G standard to confirm that corrective action measures have resolved analysis issues.