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

SRC-LIBBY-02: QUANTIFICATION OF ASBESTOS IN SOIL BY SEM/EDS

Date: June 26, 2003 SOP No. SRC-LIBBY-02 (Rev. 1)

Title: QUANTIFICATION OF ASBESTOS IN SOIL BY SEM/EDS

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[a] This SOP is adapted from methods developed by the US Geological Survey (USGS 2002) and by EMSL Analytical Inc. (EMSL 2000), along with input from other Libby analytical team members.

SYNOPSIS: A method for estimating the asbestos content (mass percent) of soil using Scanning Electron Microscopy (SEM) and Energy-Dispersive X-Ray Spectroscopy (EDS) is described. The method is intended mainly for analysis of amphibole asbestos similar to that derived from the mine in Libby, and is intended mainly for soil samples that contain less than 10% asbestos by mass.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8:	[signature]	6/30/03
Syracuse Research Corp.:	W J Brattin	6/27/03

Revision Number	Revision Date	Changes
0	4/28/2003	--
1	6/26/03	Add clarifications on purpose and limitations

1.0 PURPOSE

The purpose of this document is to describe a standard operating procedure (SOP) for estimating the concentration (expressed as weight percent) of amphibole asbestos found in soils in Libby, MT, by using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS). Because confirmation of mineral type by x-ray diffraction pattern is not included, the method should be considered a screening technique. This SOP is specifically intended for use by employees of USEPA Region 8 and by contractors supporting USEPA Region 8 at the Libby, Montana, Superfund site. The reliability and utility of this method as a screening technique for the Libby site will be assessed in a performance Evaluation (PE) study being planned and performed by USEPA.

2.0 RESPONSIBILITIES

Each laboratory performing analyses of soil from the Libby site for EPA shall have a Laboratory Supervisor who is responsible for ensuring that all analyses are performed in accord with this SOP, and for ensuring the accuracy and quality of the results.

It is the responsibility of the Laboratory Supervisor to communicate with the EPA's Laboratory Coordinator regarding specific analysis objectives and anticipated situations that may require any deviation from the SOP. Any sample-specific or SDG-specific deviations from the SOP shall be recorded on deviation sheets in accord with the process described in the Contaminant Screening Study Sampling and Analysis Plan (CDM 2002). It is also the responsibility of the Laboratory Coordinator to recommend any suggestions for improvement in this SOP to the CDM Project Manager and USEPA Region 8 personnel (Remedial Project Manager or Regional Chemist).

3.0 EQUIPMENT

Equipment required to perform the analyses described in this SOP includes the following:

- Negative Air Pressure, HEPA-filtered hood (used to capture airborne dust, fibers, and other potential contaminants)
- Analytical balance - accurate to 0.0001 g, range of 0.0001 g to 100 g.
- Porcelain crucible (for sample ashing).
- Muffle furnace - used for sample ashing.
- 500 to 800 mL glass beaker
- Teflon-coated 2-inch magnetic stir bar - used in sample mounting process
- Magnetic stirring plate - used in sample mounting used in sample mounting process
- Eppendorf pipette - capable of delivering 20 uL
- Eppendorf pipette tips - modified by cutting enough material off the tip to approximately double the opening
- SEM sample stubs with conductive carbon adhesive tabs
- Sample coater - either a carbon vacuum evaporator used to apply a carbon layer to the SEM stub, or a sputter coater to apply a gold layer to the stub.
- Desiccator and/or glass petri dish used for temporary storage of sample-mounted stubs.
- SEM fitted with EDS with the following attributes:

  For SEM:
  - Resolution: 5 nm or better
  - Accelerating Voltage: 10 to 25 kV or greater
  - Minimum magnification range: x 50 to 200,000
  - SEI (secondary electron image)
  - BEI (backscattered electron image)

  For EDS:
  - Detector resolution having the ability to resolve Na, Mg, Al, Si, Ca, and Fe peaks in BIR1-g

- Minimum resolution of 150 eV at Manganese Kα
- Analyzer capability of background subtraction, peak overlap correction, correction for sum peaks and Si escape peaks.

4.0 METHOD SUMMARY

An aliquot of the soil sample is examined by SEM/EDS. In some cases, the material must be ashed to remove excessive organic matter. After ashing (if needed), a stub is prepared by suspending the sample in water, filtering an aliquot onto a polycarbonate filter, and mounting the filter on the stub. The stub is coated with carbon or gold and is examined by SEM at several different magnifications. At each magnification, a number of different fields are selected either systematically or at random, and the area coverage (all particles) and the area fraction attributable to asbestos particles in each field are estimated or measured. These data are used to estimate the asbestos content of the soil sample.

5.0 SAMPLE STUB PREPARATION

5.1 Ashing (If necessary)

Substantial organic material in the sample can result in an outgassing of material under high vacuum such that carbon coating of the SEM stub or analysis in the SEM may be impaired. Therefore, if difficulty is encountered in coating the stubs or in the analysis, if the stub is otherwise of poor quality, that sample should be ashed and then re-prepared for examination. The procedure for sample ashing is as follows:

1. Mix the bulk sample by tipping the bag upside down, and then returning it to its original position. Use a clean sample thief to remove a uniform aliquot of the material, pushing the thief all the way through the sample. Place this sample into a clean glass beaker and mix thoroughly. After mixing, transfer about 2 grams of the sample into a pre-weighed crucible, being sure the sample does not fill more than about 1/3 of the crucible. Weigh the sample to the nearest 0.05 grams and record the weight of the crucible alone and the crucible plus sample in the Lab Data Sheet for the sample (Attachment 1).

2. Place a cover on the crucible, and place the crucible into a muffle furnace.

3. Turn the furnace on and set the temperature to 480°C. [Note: if the furnace can achieve this temperature faster than 2 hours, first turn the temperature to 250°C and hold at this temperature for 1 hour before increasing the temperature to 480°C.]

4. Hold the sample at 480°C for at least 4 hours. Shut off the furnace and allow to cool overnight.

5. When the sample has cooled, weigh the residue to the nearest 0.05 g and record the final mass in the Lab Data Sheet for the sample (Attachment 1).

Note: It is understood that this ashing procedure might result in some parts of the sample reaching temperatures that may begin to decompose chrysotile asbestos (about 500°C). However, this SOP is intended for the analysis of amphibole asbestos, and amphibole asbestos is not expected to begin decomposition unless temperatures are substantially higher.

5.2 Mounting the Sample

Samples must be prepared in a clean HEPA hood. To prevent cross-contamination of samples, only one sample should be mounted onto a SEM stub at a time. The procedure to mount the soil sample onto the SEM stub is described in USGS (2002b), and is repeated below.

Set up a Millipore filter apparatus for use with 25 mm polycarbonate filters. Place a few drops of distilled water on the fritted glass surface and place a 25 mm polycarbonate filter (0.4 um or less pore size) on the water, shiny side up. Attach the top of the apparatus. Add a few milliliters of distilled water on the filter so that no part of the filter is exposed to air. Take a clean Eppendorf pipette tip and with a razor blade, cut enough material off the tip to increase the opening to approximately 1 mm. Transfer approximately 0.5 g of the sample prepared for SEM/EDS analysis to an 800 mL glass beaker containing 125 mL of distilled water and a 2-inch magnetic stirring bar, and place on a magnetic stirring plate. Slowly increase the speed of rotation until all the particles are suspended and a good vortex is achieved without excessive splashing. While the sample is mixing, and the particles are suspended, collect 20 uL of the mixture using the

Eppendorf pipette. Transfer this to the polycarbonate filter by dropping the suspension from 3 to 4 cm above the water in the Millipore apparatus. Place two more 20 uL drops of the suspension onto the polycarbonate filter for a total of 60 uL.

Allow the water to filter through the polycarbonate. If a hand-held vacuum pump is available, it may be used to decrease the time it takes for complete filtration. Do not use motorized pumps because sample will become too dry on the filter, increasing the possibility of sample loss.

Use forceps to pick up the filter just as the water has passed through but before it is dry and place it on a carbon adhesive tab on a standard SEM mount. The filter needs to be completely flat on the SEM stub. This is achieved by forming the wet filter into a gentle U-shape. Place the bottom curve of the filter onto the center of the carbon adhesive tab and slowly release the sides so they lay flat. Trim the edges of the polycarbonate filter using a razor blade. After drying, coat the stub with carbon using a carbon evaporator or with gold using a sputter coater. The stub is then ready to be taken to the SEM in a clean, covered container.

The SEM sample stub should have about 15-20% total coverage. Too little sample will require too many fields to be counted. Too much sample will cause sample charging and degrade the analyst's ability to accurately determine the asbestos content. In general, stubs that have significantly higher than 25% total coverage should be discarded and re-prepared at a lower density.

6.0 ANALYSIS BY SEM

All analyses should be run at 15 kV.

SEM analysis will be done at magnifications of 50x, 500x, and 2000x. Complete the procedure at 50x before continuing to 500x and then 2000x.

Counting Rules

At each magnification, record the occurrence of every structure that satisfies the following counting rules:

1. Particle diameter is within the range specified for the magnification:

50x	Diameter > 20 um
500x:	Diameter = 2-20 um
2000x	Diameter < 2 um

2. Aspect ratio is greater than 3:1

3. Aspect ratio is < 3:1, but particle is clearly fibrous

4. The EDS spectrum of the particle is characteristic of Libby amphibole asbestos, based on the EDS criteria presented in Attachment 3.

For analysis at 50x, the whole sample stub should be examined. This requires about 12-14 fields for a standard 1 cm-stub. For each field observed, collect the following data:

- The total area coverage (all particles). Record this in the Lab Data Sheet (Attachment 1). Whenever possible, this should be done by collecting a digital backscatter image and quantifying the particle coverage using appropriate software. When the instrument does not support this technique, the total coverage should be estimated visually using the visual reference aids provided in Attachment 2. The method used to estimate total coverage should be indicated in the Lab Data Sheet.

- The length and thickness of all structures that satisfy the counting rules above. Record these values in the Lab Data Sheet (Attachment 1).

- Record and save an EDS spectrum and prepare a photomicrograph for the first structure recorded, and for every 10th structure thereafter. Identify these structures in the Lab data Sheet.

After completion of the analysis at 50x, proceed to the analysis at 500x. Evaluate a total of 20 fields, selected using a systematic uniform random (SUR) approach to selection of fields. In this approach, the operator randomly chooses a starting field at the upper left corner of the sample stub, and then spaces the rest of the fields at grid points formed by evenly spaced horizontal and

vertical lines, with the distance between fields being sufficient to prevent field overlap. For each field at 500x, record the same information as specified at 50X, except remembering that only structures with thicknesses between 2 and 20 um should be considered. After completion of the analysis at 500x, proceed to the analysis at 2000x. View and record data for 40 fields. For each field at 2000x, record the same information as specified at 500x, except remembering that only structures with thicknesses less than 2 um should be considered.

The following table summarizes the SEM procedure:

Magnification	Fields	Counting Rules		
		Diameter	Aspect ratio	EDS
50x	12-14	> 20 um	>3:1 (all particles), or less than 3:1 if the particle is clearly fibrous	Characteristic of Libby amphiboles (see Attachment 3)
500x	20	2-20 um		
2000x	40	< 2 um		

Required Sensitivity

The sensitivity of this method depends upon the average size of asbestos particles observed at each magnification. Because data are not yet available on the expected size of these particles, no minimum sensitivity is specified at this time. A sensitivity requirement may be added to this SOP after data become available on typical asbestos particle sizes in soil samples from the site.

7.0 ESTIMATION OF MASS PERCENT

7.1 Area Fraction Approach

The mass of amphibole asbestos in the soil sample may be approximated as the area fraction of the sample that is occupied by amphibole asbestos structures. It is understood that mass fraction and area fraction are not identical, but this approximation is considered to be acceptable for the purposes of evaluating the potential utility of this method as a screening tool at the site.

The area fraction (AF) occupied by amphibole asbestos particles is computed from the raw data in the Lab Data Sheet (Attachment 1) using the following basic equations:

$$AF(total) = AF(50x) + AF(500x) + AF(2000x)$$

$$AF(m) = (1/N_m) \cdot \sum AF_{i,m}$$

$$AF_{i,m} = (1/FA_m) \cdot \sum(l_{i,j,m} \cdot w_{i,j,m}) \cdot 1E\text{-}06\ mm^2/um^2 \cdot 100$$

where:

AF(m) = Average area fraction occupied by amphibole asbestos at magnification "m"
N_m = Total number of fields examined at a magnification "m"
$AF_{i,m}$ = Area fraction occupied by amphibole asbestos in field "i" at magnification "m"
FA_m = Field area (mm^2) at magnification "m"
$l_{i,j,m}$ = length of amphibole structure "j" observed in field "i" (um) at magnification "m"
$w_{i,j,m}$ = width of amphibole structure "j" observed in field "i" (um) at magnification "m"

7.2 Structure Count Approach

The structure count approach is based on the concept that the size-distribution of asbestos particle sizes in fine-ground soil is approximately constant. If so, there will be an approximately linear relation between asbestos particle density (average particles per field) of any specified size category and the mass percent concentration in the sample.

Calibration Curve

In this approach, quantification is achieved using an empiric calibration curve. That is, each lab should measure the average number of asbestos particles (diameter = 2 to 20 um) per field at 500x in a series of calibration standards of Libby Amphibole, plus a blank. The calibration curve should be composed of at least five independent preparations (stubs) of at least 5 calibration standards (blank plus four spiked samples) that span the range of observed field samples (e.g., < 0.1% to > 5%), and each preparation should be evaluated by counting asbestos

structures in at least 20 fields at 500x. The calibration standards will be prepared and provided by USGS.

The results should be summarized in graphical format, in which the x-axis is the true concentration (mass percent) in the sample, and the y-axis is the average number of particles (2 to 20 um in diameter) per field observed at 500x.

Sample Quantification

The concentration of a sample is obtained by counting the number of amphibole asbestos structures (2 to 20 um in diameter) in each of 20 fields at one or more specified magnifications (e.g., 500x, 2000x), and using the average value (i.e., the average count across the 20 fields) to estimate the mass percent from the empiric standard curve.

8.0 SEM/EDS INSTRUMENT CALIBRATION AND MAINTENANCE

8.1 Instrument Calibration

All instrument calibrations are performed under the same operating conditions as sample analysis.

Size (Magnification) Calibration

Calibration for size (magnification) is performed using magnification gratings (obtained from E. F. Fullam or other appropriate vendor) in accord with the manufacturer's instrument-specific instructions. Instrument calibration for size (magnification) must be performed at the following minimum frequency:

- prior to initial receipt of field samples
- monthly after the first calibration
- immediately after any maintenance activities are performed

EDS Calibration

The energy dispersive x-ray will be standardized at the beginning of each day, or at any time the instrument operating conditions change significantly. The US Geological Survey has provided a BIR-1G basalt glass as a reference material for this procedure. This material is provided to assist in evaluation of performance and inter-laboratory comparisons of energy dispersive x-ray analysis data obtained on a scanning electron microscope. The EDS standardization is essential for ensuring the instrument and software capability of distinguishing mineral species that have similar compositions and may have a fibrous or similar habit (for example, distinguish amphibole from pyroxene or biotite). The EDS spectrum of the BIR1-G sample should be provided as part of the standard data package for each set of analytical results.

The basalt glass is mounted in epoxy in a 1/4 inch polished mount that has been carbon coated in a high vacuum carbon evaporator. Detailed instructions for the analysis of this sample are provided in Attachment 4. In brief, always handle this sample with gloves in a dust free environment. Do not re-polish, re-coat, or touch the surface of this reference material mount, as this could result in damage to the material or a change in its composition. The following practices and analytical conditions must be used when analyzing this sample:

1) The diameter of the primary electron beam should be less than 50 um and greater than or equal to 20 um, or an equivalent raster size should be used. (Note: some raster patterns have prolonged dwell at the comers and/or the sides)
2) Primary beam current should not exceed 10 nA.
3) Avoid repeated analysis of the same spot on the sample.
4) Do not leave the beam on the sample for prolonged periods.
5) Do not examine the sample at a high magnification that would result in a beam exposure greater than or equivalent to that specified in item 1.
6) Do not expose the epoxy to the electron beam except at very low magnification and for very brief time periods.
7) Be sure that good electrical contact is made between the sides of the brass tube and the SEM stage during analysis.
8) Always store in a dry, dust-free environment.

Evaluate the analysis by comparing the values obtained with the recommended values for BIR-1G glass. Records will be kept each day of values obtained for the standard to determine the percent variation of accuracy and precision of the instrument. Acceptance criteria will be provided in an update to this SOP after results from several laboratories are available.

In the event that EDS calibration standard results do not meet the acceptance criteria provided above, analysis can not proceed or, if in progress, should cease until the problem corrected. All EDS calibration data should be documented in the instrument logbook and should be traceable to each sample delivery group (SDG) associated with the calibration. A SDG is defined as a single set of samples submitted to the laboratory in one day or every 20 samples, whichever is more frequent.

8.2 Instrument Maintenance

Periodic instrument maintenance should be performed as necessary in accord with manufacturer's recommendations. All maintenance activities must be recorded in an instrument logbook dedicated to that particular instrument. As indicated above, if instrument maintenance is performed, the instrument should be re-calibrated and the calibration verified as described in Section 8.1.

9.0 LABORATORY QUALITY ASSURANCE/QUALITY CONTROL

Laboratory Quality Assurance/Quality Control activities are actions taken by the laboratory to obtain data that indicate if analyses are occurring within specified acceptable ranges of accuracy and precision. Any time that performance deviates from acceptable ranges, all analyses must be stopped until the source of the deviation is identified and remedied.

Accreditation and Training

All laboratories involved in the analysis must be accredited by the National Voluntary Accreditation program (NVLAP), and all analysts must be properly trained by education or experience in identification and quantification of asbestos structures by SEM/EDS. In this

regard, USEPA provides project-specific training and instruction to laboratories as described in the CSS SAP (USEPA 2002).

Laboratory Blanks

Each laboratory shall prepare at least one laboratory blank SEM stub with each set of field samples SEM stubs prepared. This blank stub will be prepared exactly the same as all field sample stubs, except that no soil will be added to the water before application to the stub. This blank is intended to evaluate the general cleanliness of the sample preparation area and the potential for stub contamination during sample handing and preparation.

10.0 DECONTAMINATION

Decontamination (decon) procedures will be performed using standard laboratory decontamination procedures.

11.0 DOCUMENTATION

Instrument Logbook

An individual instrument logbook (hard copy and/or electronic) should be kept for each piece of equipment in use at the laboratory. All initial calibration, daily calibration results, and instrument maintenance activities must be recorded in the appropriate logbook.

Photomicrographs and EDS Spectra

Each EDS spectrum and each photomicrograph collected in accord with the SOP must be identified by the unique laboratory identification number and analysis date. Any opportunistic photographs taken are also numbered, but must accompany a brief explanation of the reason for taking the picture.

Electronic Data

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

Hardcopy Data

All data sheets or logbooks, photographs, and spectrographs must be stored in a secured location with limited access (e.g., locking file cabinet) when not in use.

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

12.0 REFERENCES

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

EMSL. 2000. SOP EPA-LIBBY-01, Revision 0. Asbestos Analysis of Soil by SEM and EDS. Prepared by EMSL Analytical Inc. August 25, 2000.

USGS. 2002a. Analysis of Soil Samples for Asbestos Content by Scanning Electron Microscope and Energy Dispersive Spectroscopy. Prepared by A. Bern, G.P. Meeker, and I.M. Brownfield of the U.S. Geological Survey for the U.S. Environmental Protection Agency. October 4, 2002.

USGS. 2002b. Preparation and Analysis of Soil Samples For Amphibole Asbestos Content by Scanning Electron Microscopy And Energy Dispersive Spectrometry. U.S. Geological Survey Administrative Report prepared for the U.S. Environmental Protection Agency by A. Bern, I.M. Brownfield and G.P. Meeker, U.S. Geological Survey. December 18th, 2002.

ATTACHMENT 1

SEM LAB DATA SHEET

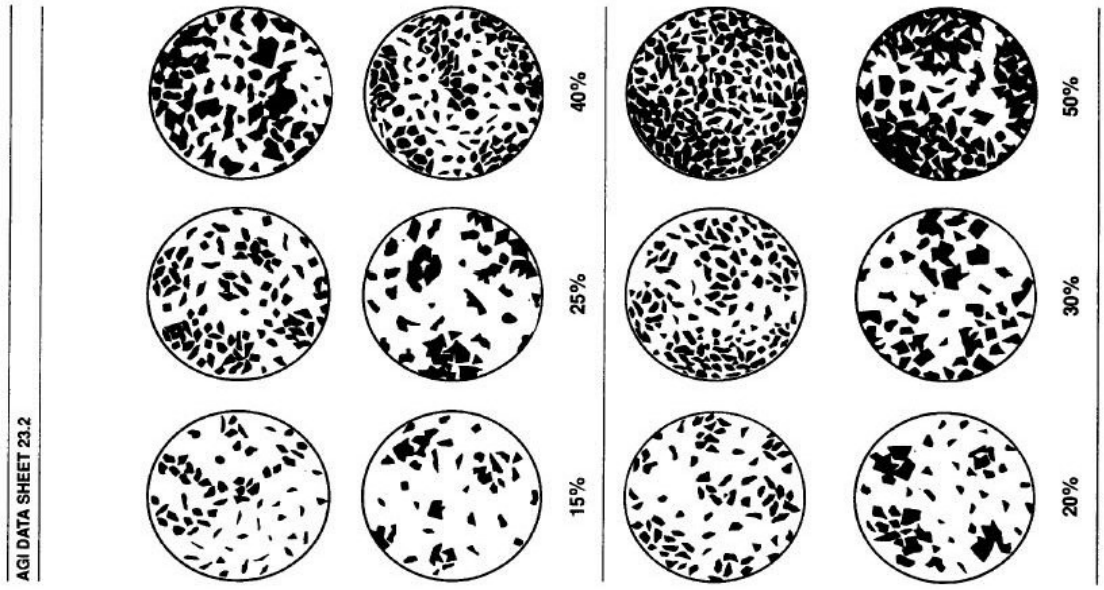
See electronic Excel file
"SEM Lab Data Sheet.xls"

(Check with Volpe or SRC to determine the most recent version number)

ATTACHMENT 2

VISUAL AID FOR ESTIMATING TOTAL COVERAGE IN A FIELD OF VIEW

Source: Dutro Jr., J. T., Dietrich, R. V., Foose, R. M. 1989. AGI Data Sheets for Geology in the Field, Laboratory, and Office, Third Edition. American Geological Institute.


AGI DATA SHEET 23.2
40%
25%
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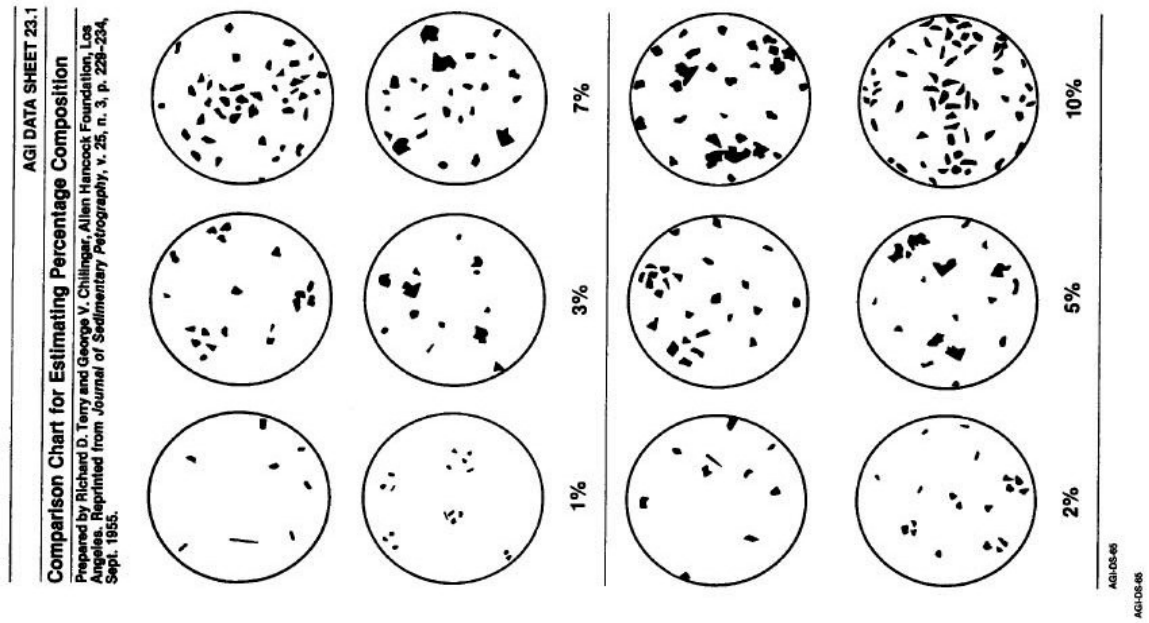
AGI DATA SHEET 23.1
Comparison Chart for Estimating Percentage Composition
Prepared by Richard D. Terry and George V. Chilingar, Allen Hancock Foundation, Los Angeles. Reprinted from Journal of Sedimentary Petrography, v. 25, n. 3, p. 229-234, Sept. 1955.
7%
3%
1%
10%
5%
2%
AGI-DS-65

ATTACHMENT 3

GUIDANCE FOR IDENTIFYING LIBBY AMPHIBOLE USING EDS

GUIDE TO ANALYSIS OF SOIL SAMPLES FROM LIBBY, MONTANA FOR ASBESTOS CONTENT BY SCANNING ELECTRON MICROSCOPE AND ENERGY DISPERSIVE SPECTROSCOPY

by

Amy Bern
Greg Meeker
Isabelle Brownfield

U.S. Geological Survey Administrative Report
For the U.S. Environmental Protection Agency

October 17, 2002

Fibrous materials can not be reliably identified as amphibole by morphology alone. Other materials, such as calcite, pyroxene, or biotite, can also have cleavage that produces long thin particles or scrolls. Libby, Montana soils contain many of these minerals. To accurately identify amphibole fibers by SEM analysis, EDS spectra must be obtained to verify that mineral composition is consistent with an amphibole.

Two EDS spectra of amphiboles are illustrated in Figure 1a. The first is an example of an amphibole from Libby Montana. The second is an International Union Against Cancer (UICC) standard tremolite. Libby amphiboles contain the elements Na, Mg, Al, Si, K, Ca, Ti, Mn, and Fe in varying amounts, but similar to those shown in Figure 1a. F and Cl also occur, but usually not in quantities great enough to show on EDS spectra. Some diagnostic spectra of other minerals are presented for comparison.

Biotites contain many of the same elements as Libby amphiboles. The Al and Mg content is used to distinguish biotite from amphibole. Biotites contain much more aluminum than Libby amphiboles. EDS spectra of Libby micas are provided in Figure 1b for comparison.

Pyroxene and Libby amphibole can be difficult to tell apart, but usually can be distinguished by the absence of K and higher Al in pyroxene. An EDS spectrum for a typical pyroxene is given in Figure 1c.

The best way to determine if the chemical composition is consistent with an amphibole is to perform a "quantitative" analysis.[1] In most cases a "standardless" analysis will provide sufficient accuracy to give confidence that the material is compatible with an amphibole. For this analysis, oxygen should be determined by stoichiometry and cation ratios for Na, Mg, Al, Si, K, Ca, Ti, Mn, and Fe should be calculated based on 23 oxygens. The cation total should equal between 15 and 16. Examples of EDS analyses for Libby Amphibole weight percentages and cations are given in Table 1.

Table 1. EDS Analysis of Libby Amphibole Fibers.

Weight Percent									
	Na2O	MgO	Al2O3	SiO2	K2O	CaO	TiO2	MnO	Fe2O3
1	4.7	21	0.43	57	1.3	7.5	0.33	0.17	7.7
2	5.5	21	0	60	2.1	4.4	0	0.03	8.0
3	1.6	23	0.14	59	0.37	12	0.12	0.01	3.1

Cation Totals (all Fe is Fe3+)										
	Na	Mg	Al	Si	K	Ca	Ti	Mn	Fe3	Total
1	1.22	4.27	0.07	7.77	0.22	1.09	0.03	0.02	0.81	15.50
2	1.44	4.11	0.00	8.07	0.35	0.63	0.00	0.00	0.84	15.43
3	0.42	4.64	0.02	7.90	0.06	1.78	0.01	0.00	0.32	15.16

	T Site Total	C Site Total	B Site Total	A Site Total	Cation Total
1	7.87	5.00	2.00	0.63	15.50
2	8.00	5.02	2.00	0.42	15.43
3	7.93	4.96	2.00	0.26	15.16

T site-Si $\cong$ 8 C site-Al + Fe + Mg + Ti + Mn $\cong$ 5 A+B sites-Ca + Na + K >= 2

1. Quantitative analysis requires matrix corrections to correct for atomic number, absorption, and fluorescence effects. Matrix correction routines (e.g. ZAF or Phi Rho Z) assume that the sample is homogeneous in the volume analyzed, and has a flat polished surface. If these conditions are not met the results can have errors that are larger than what is normally expected. The magnitude of this error will depend on the size, shape and composition of the sample or particle and also on the analytical conditions, particularly accelerating voltage (Small and Armstrong, 2000). Our experience with the analysis of single structures of Libby amphibole using the analytical conditions specified in this procedure is that the errors for all elements are typically within +/-10 % relative concentration of the expected value, and often much better.

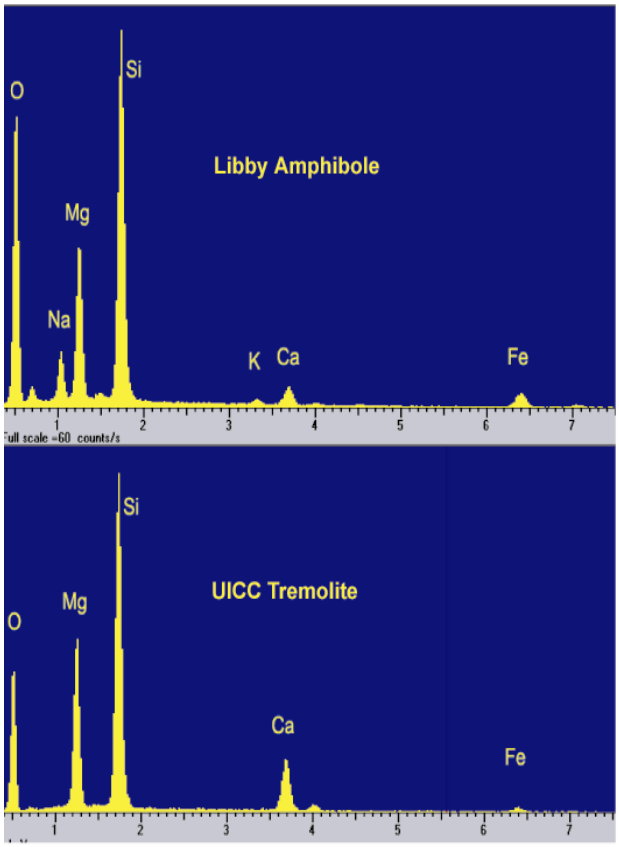

Figure 1a. EDS Spectra of Libby Amphibole and Tremolite.

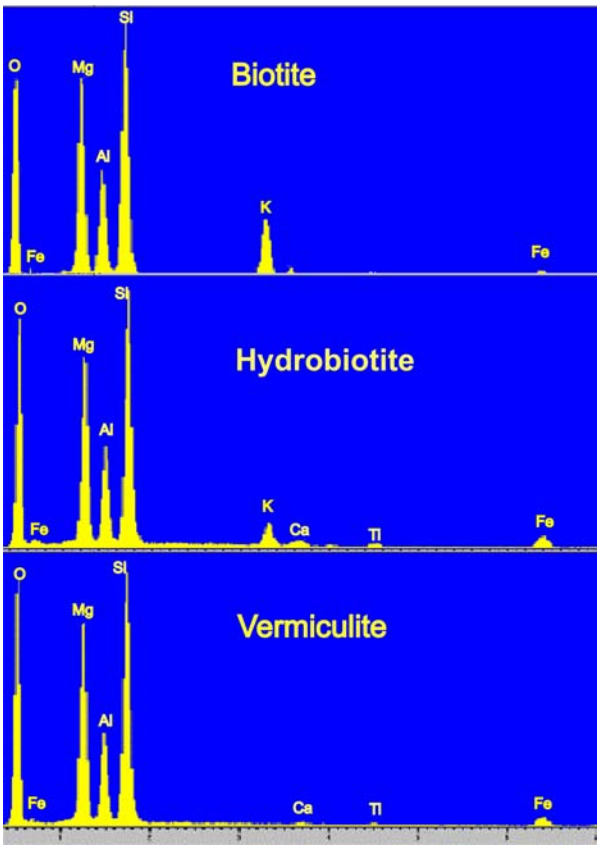

Figure 1b. EDS Spectra of micas found in Libby Samples.

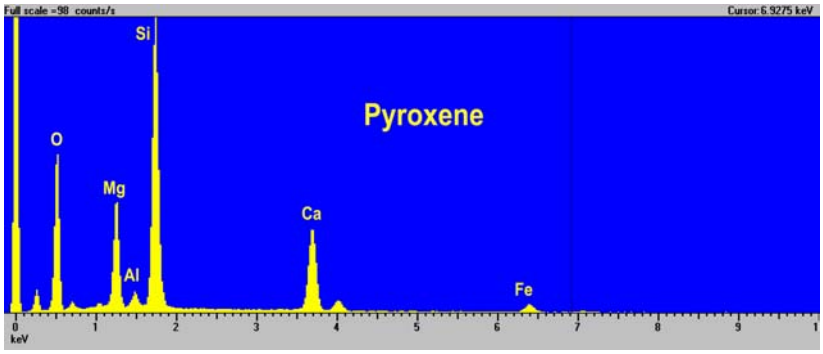

Figure 1c. EDS Spectrum of Pyroxene.

Reference

Small, J.A. and Armstrong, J.T., 2000, Improving the Analytical Accuracy in the Analysis of Particles by Employing Low Voltage Analysis. Microscopy and Microanalysis, vol. 6, supplement 2. Proceedings 2000, p.924-925.

ATTACHMENT 4

PROCEDURE FOR EVALUATION OF EDS USING GLASS STANDARD BIR1-G

Instructions for Analysis of BIR1-G Basalt Glass Reference Material

1. Follow all instructions on the handling and analysis sheet supplied with the reference material. A copy is included below.
2. Calibrate EDS system as per manufactures instructions at the proper working distance. The sample should be mounted with the surface normal to the beam unless your instrument is specifically designed otherwise. Locate basalt grains in epoxy at low magnification with the backscatter detector. Care should be taken to avoid using high current density on areas of epoxy to prevent contamination of the reference material.
3. Analyze the sample using a beam diameter (or equivalent raster) between 20 and 40 um. Do not use a beam size smaller than 20 um as this can damage the reference material.
4. Analyze the sample at 15 kV, approximately 20-40 % dead time, < 5 nA primary beam current.
5. Allow the spectrum to accumulate for at least 2 minutes (real time) or until the integrated Si peak contains approximately 50,000 counts (or about 16, 000 cts. peak height).
6. Perform a "quantitative" analysis of the spectrum using ZAF or Phi Rho Z, without standards, using the same software that you would use for Libby amphibole particles. Analyze for Na, Mg, Al, Si, K, Ca, Ti, Mn, Cr, and Fe (not all elements may be detectable). The analysis should include background subtraction, peak overlap correction, correction for sum peaks and Si escape peaks, and be normalized to 100 %.
7. Report results as weight percent oxide for Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, TiO_2, MnO, Cr_2O_3, Fe_2O_3 with oxygen calculated by stoichiometry. Report cation ratios based on 23 oxygen ions.
8. Please provide all spectra and numerical results including cation ratios.