Appendix B

ARB Test Method 435 Interlaboratory Study

INTRODUCTION

Background

In 2005, concerns were raised by stakeholders regarding the repeatability of analytical results from the California Air Resources Board's (ARB) Test Method 435 (M435), "Determination of Asbestos Content of Serpentine Aggregate." Stakeholders informed the ARB that laboratories prepare and analyze soil and rock samples in different ways and obtain differing results from the same, or similar, samples.

To address these concerns, ARB staff met with M435 stakeholders, including commercial laboratory personnel, and decided to conduct an interlaboratory study (ILS) that compares laboratory sample processing and analysis procedures. The ILS was completed in July 2007. ILS results were first shared with the participating laboratories in August 2007 and subsequently shared with other stakeholders in various meetings and workshops.

Purpose

The purpose of the ILS was to investigate sources of variability among laboratories during M435 sample processing and analysis and determine whether these differences can affect M435 analytical results.

PARTICIPANTS

ARB staff coordinated the ILS. Four commercial laboratories, all offering M435 analytical services in California and located within 100 miles (160 km) of ARB offices in Sacramento, participated in the ILS. All four laboratories were accredited by the National Institute of Standards of Technology (NIST) through the National Voluntary Laboratory Accreditation Program for polarized light microscopy (PLM) analysis as a bulk test for asbestos-containing materials (ACMs) using the 1982 procedure, EPA-600-M4-82-020, "Interim Method for the Determination of Asbestos in Bulk Insulation Samples." The four participating laboratories included:

1) Asbestos TEM Laboratories, Inc.--Berkeley.
2) EMSL Analytical, Inc.--San Leandro.
3) Forensic Analytical Laboratories, Inc.--Hayward.
4) RJ Lee Group, Inc.--San Leandro.

The laboratories agreed to participate in the ILS under conditions of anonymity such that the results of the study would not be directly attributed to any laboratory.

STUDY DESIGN

The ILS was conducted in two phases. During Phase One, laboratory pulverization and analysis of a crushed field sample were observed. During Phase Two, fixed mounted slides, prepared by ARB staff, were given to laboratories for analysis in a round robin study to further assess analytical practices. The two phases of the ILS are described separately in this appendix. For each phase, the general focus was on the variability of the results rather than evaluating for accuracy. In Phase One, asbestos was highly suspected to be present in the sample. In Phase Two, asbestos was known to be present in those samples that were spiked with an asbestos standard reference material. In both cases, however, the "true" quantifiable asbestos content (by point count) was not known.

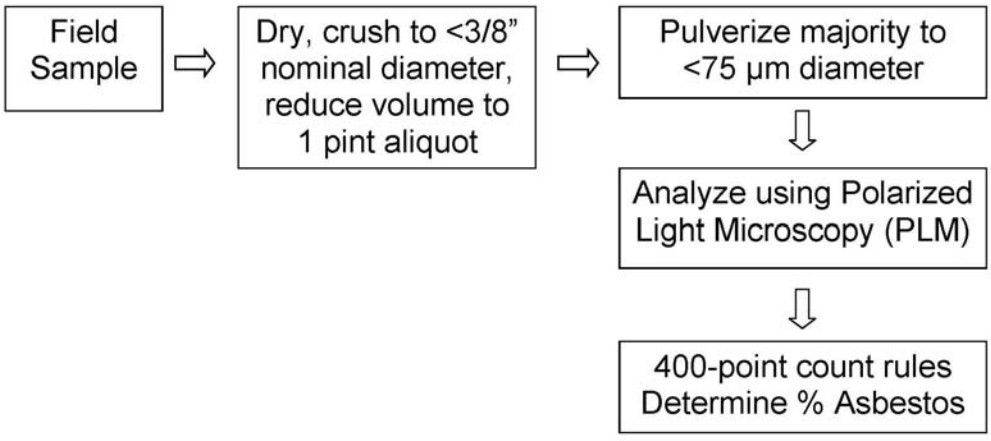
ILS PHASE ONE

Phase One: Materials and Methods

Sample Preparation

The sequence of sample preparation followed the requirements as set forth in M435 (Figure B-1). ARB obtained approximately five gallons (approximately 20 liters) of rocks and soil from an area where naturally-occurring asbestos (NOA) is known to be present. The rocks were observed through a stereoscopic microscope and a polarized light microscope and were found to contain fibers. ARB staff archived one gallon (approximately four liters) of the field sample. The remaining four gallons (approximately 16 liters) were prepared for the ILS.

Figure B-1. Test Method 435 Protocol



ARB staff supervised the field material preparation prior to distribution to the laboratories (Figure B-2). The sample was dried in a constant-temperature oven for

15 hours in shallow aluminum pans at 248 degrees Fahrenheit (ºF) (120 degrees Celsius, ºC). Many of the rocks were between one to six inches in diameter (2.5 to 15 centimeters). ARB staff supervised the crushing of rock samples to less than 3/8-inch (approximately 0.95-centimeter) diameter particles using a jaw crusher (Model Badger, Bico Braun International, Burbank, CA). The crushed material was repeatedly passed through a riffle splitter to randomly split and recombine several sample splits. About two gallons (approximately eight liters) of crushed sample were archived. ARB staff divided the remaining crushed sample into 1/2-gallon (approximately 2-liter) packages and labeled them for distribution to the laboratories.

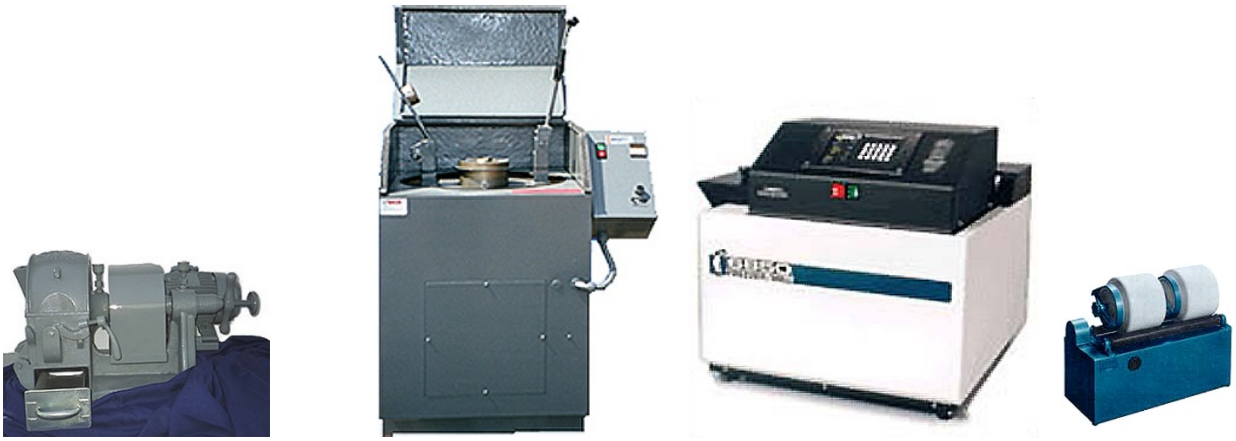
Figure B-2. Sample was (A) dried, (B) crushed, (C) riffle-split, and (D) packaged.

A. Dried **B. Crushed** **C. Riffle Split** **D. Packaged**

M435 Laboratory Pulverization

ARB staff distributed approximately one half gallon (approximately two liters) of crushed material to each laboratory and observed each laboratory's sample pulverization procedures. Each laboratory pulverized the crushed sample according to their M435 laboratory protocol, using one of the equipment shown in Figure B-3. The laboratory

Figure B-3. Laboratory Pulverization Equipment for ILS Sample



A. Plate grinder (Braun Mill) **B. Vibratory Pulverizer (Shatter Box)** **C. Freezer Mill** **D. Ball Mill**

personnel divided their powdered product into 12 aliquots and turned them over to ARB staff. ARB staff archived a portion of the powdered product and the remaining

powdered aliquots were coded and labeled by ARB staff prior to distribution to all of the four laboratories for M435 sample analysis. Three aliquots were given to each of the participating laboratories (Table B-1).

M435 Sample Analysis

In this blind study, each laboratory was asked to analyze 12 powdered aliquots according to their M435 analysis protocol. The 12 aliquots to be analyzed consisted of three replicates of the powder processed by each of the four participating laboratories, as shown in Table B-1.

Table B-1. ILS Phase One Study Design

	Analyses by Lab A			Analyses by Lab B			Analyses by Lab C			Analyses by Lab D		
Prep by Lab A	a 1	a 2	a 3	a	a	a	a	a	a	a	a	a
Prep by Lab B	b 4	b 5	b 6	b	b	b	b	b	b	b	b	b
Prep by Lab C	c 7	c 8	c 9	c	c	c	c	c	c	c	c	c
Prep by Lab D	d 10	d 11	d 12	d	d	d	d	d	d	d	d	d

Powdered Sample Characterization

ARB staff studied and characterized the powders prepared by each laboratory. The particle size distributions (PSD) of clay (less than 2-micrometer fraction), silt (2- to 50-micrometer fraction), and sand (50- to 2000-micrometer fraction) of three aliquots, taken from the powdered product of each of the four laboratories, were determined following the pipette method (Soil Survey Staff, 1996).

Following the PSD analysis by pipette, ARB staff determined the particle size distribution of the sand fractions and the greater than 2-millimeter fraction by dry sieving. The particle size cuts are shown in Figure B-4. A known mass of oven-dry, 50-micrometer or greater diameter particles were agitated through a tared nest of 3-inch (7.62-centimeter) diameter sieves having the appropriate mesh openings. After three minutes of agitation using a sample shaker, each tared sieve was weighed under a fume hood and the mass percentage of each size fraction was calculated.

Figure B-4. Methods for Particle Size Distribution Analysis

- **Particle Size Analysis by Pipette**
  Sand 50-2000 µm
  Silt 2-50 µm
  <5 µm, <10 µm, <15 µm, <20 µm
  Clay <2 µm

- **Dry Sieving**
  50 µm, 75 µm (200 mesh)
  100 µm, 250 µm, 500 µm,
  1000 µm. 2000 µm, >2000 µm

Transmission Electron Microscopy (TEM) Sample Analysis

As a follow-up, six sample powders were submitted to two laboratories for quantitative asbestos analysis by TEM using EPA/600/R-93/116, "Method for the Determination of Asbestos in Bulk Building Materials."

Phase One: Results and Discussion

M435 Analytical Results

To avoid attribution of the analytical results to any participating laboratory, Table B-2 and the following figures use letter names that have no continuity and are for discussion references only.

Table B-2. Phase One Analytical Results

	Analyses by Lab A			Analyses by Lab B			Analyses by Lab C			Analyses by Lab D		
Prep by Lab E	0.75	1.25	1.25	0.0*	0.0*	0.0*	0.75	1.00	0.75	0.00	0.00	0.00
Prep by Lab F	1.00	1.50	1.00	0.0*	0.0*	0.0*	0.25	0.50	0.50	0.00	0.00	0.00
Prep by Lab G	0.0*	0.0*	0.25	0.0*	0.0*	0.0*	0.0*	0.0*	0.0*	0.00	0.00	0.00
Prep by Lab H	0.75	1.00	1.50	0.0*	0.25	0.0*	0.75	0.50	1.00	0.00	0.00	0.00

Note: "0.00" is used for this table when no asbestos is detected ("0" or ND reported).
"0.0*" is used when "<0.25%" or "trace" is reported, meaning the asbestos fibers seen were not under a point.

The results of the M435 400-point count analyses among the four laboratories (each consisting of results from 12 aliquots) are shown in Table B-2 and depicted in box-whisker plots shown in Figures B-5 and B-6.

The analytical results ranged from 0 to 1.5 percent asbestos by point count (i.e., zero to six fibers reported from 400-point counts). The boxes in Figure B-5 indicate the 25th and 75th percentiles of the data and the line in the middle of the box is the median. Figure B-5 illustrates the data shown in Table B-2 grouped according to which laboratory did the pulverization. There is a notable "sample preparation effect" where there was statistically significantly less percentage asbestos content reported from the powder prepared by one laboratory. Out of all the 12 aliquots prepared by that particular laboratory and analyzed by all four laboratories, only one fiber in total was reported.

Figure B-5. Analytical Results: Sample Preparation Effect

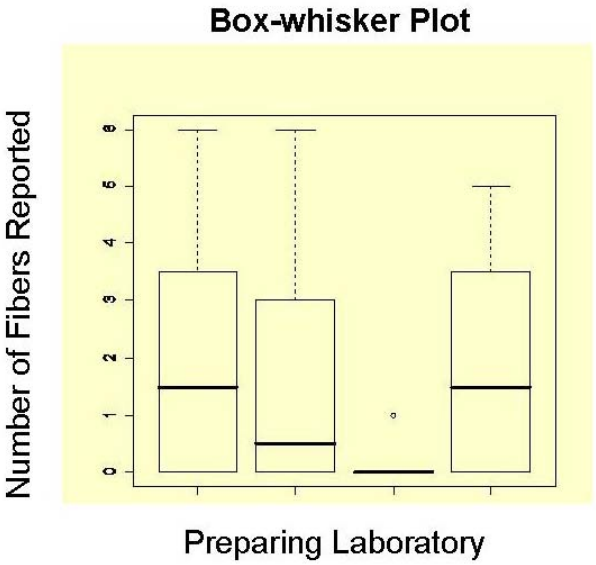
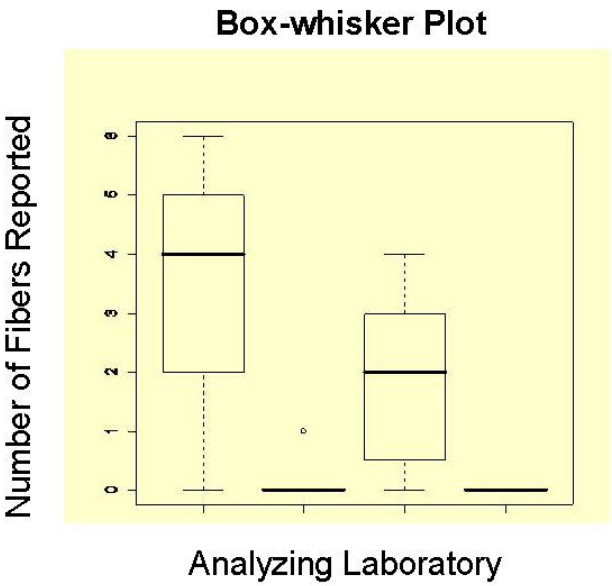


Figure B-6 depicts the same data as in Figure B-5, but this time the data are grouped according to which laboratory performed the analysis. In this case, an "analysis effect" is observed as two laboratories reported statistically significantly less percent asbestos content than the other two laboratories.

Figure B-6. Analytical Results: Laboratory Analysis Effect



Powdered Sample Characterization

ARB staff evaluated the characteristics of the pulverized powders after noticing visible differences among the powders produced by the laboratories. Specifically, a visual comparison of the powdered samples prepared by the four laboratories indicated that there were differences in particle sizes (Figure B-7). Powders 1 and 2 appeared to be fine-grained, and in comparison, Powders 3 and 4 had chunks of rocks, showing incomplete pulverization.

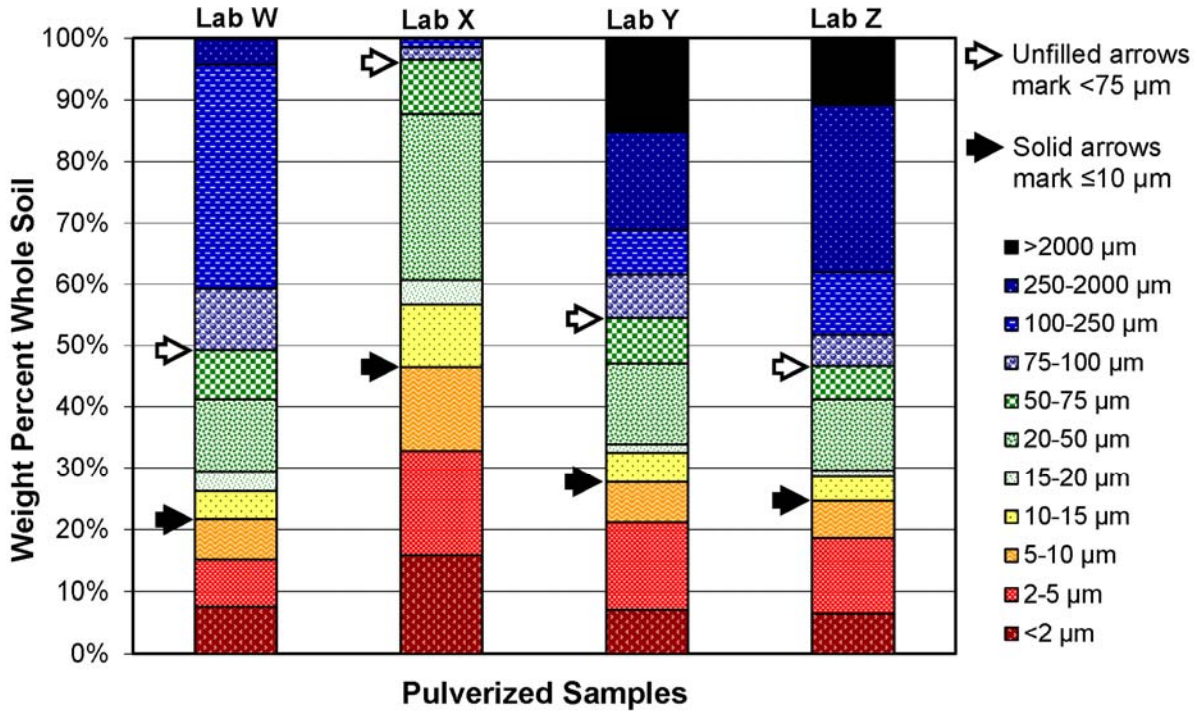
Figure B-7. Evaluation of Pulverized Samples

The analysis of the particle size distribution showed differences among the powders in the weight percentages of different particle size fractions. The particle size distribution of powders prepared by the four laboratories is shown in Figure B-8. The data presented are the averages of three replicate aliquots from each of the powders produced by the four laboratories.

Going from top to bottom in Figure B-8, the particle sizes are graphed from coarse to fine, as is similarly shown on the legend. The solid arrows indicate the weight percent of particles that are less than or equal to 10 micrometers in diameter. The powders prepared by Laboratories W, Y, and Z have between 22 to 28 percent of particles that are less than or equal to 10 micrometers in diameter. In contrast, the powder produced by Laboratory X has approximately 47 percent of particles less than or equal to 10 micrometers in diameter. These small particles are difficult to visualize at 100X magnification using PLM, as specified for point counting in M435, but could be analyzed at much higher magnifications.

The unfilled arrows indicate the upper limit of the weight percentage of particles that are less than 75 micrometers in diameter. M435 requires that the majority of the pulverized sample must pass through 75-micrometer (200 mesh) sieve. This is also the practical upper size limit of particles that can be covered by a glass slip on an oil immersion slide of powdered material for M435 PLM analysis. Almost 97 percent of the powder produced by Laboratory X is less than 75 micrometers in diameter, whereas in the other three powders this size fraction is between 47 to 55 percent. It should also be noted that the powders produced by Laboratory Y and Laboratory Z contained incompletely pulverized rock particles denoted by the solid black size fraction of particles that are greater than 2000 micrometers in diameter.

Figure B-8. Particle Size Distribution



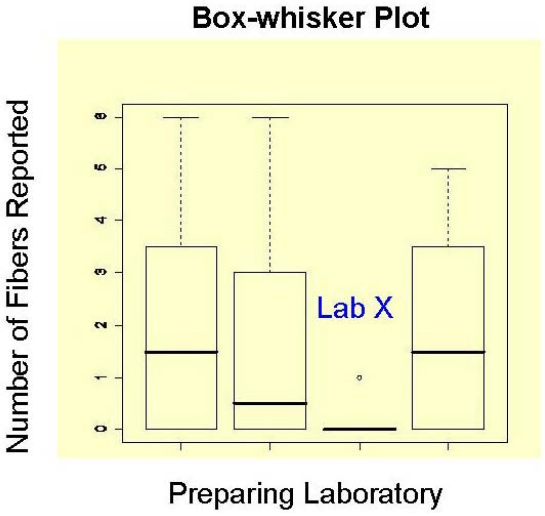
Based on the particle size analysis, it was shown that the laboratories did not produce pulverized samples with similar particle size distribution (PSD). The PSD of samples

prepared by Laboratory X were much finer. Pulverized samples from Labs Y and Z contained leftover rock chunks.

Comparing the PSD of the powders shown in Figure B-8 to the analytical results depicted in Figure B-5 revealed that the powder with very fine PSD, prepared by Laboratory X, is the same powder which reportedly had the lowest asbestos content (Figure B-9). It appears that very fine PSD significantly decreases the percent asbestos reported. It is possible that asbestos, due to its needle-like shape, may be more susceptible to the pulverization process and/or the asbestos fibers may be reduced to a size smaller than can be analyzed under conditions stipulated in M435 and may even be a size smaller than the resolution of PLM.

To further investigate the presence of asbestos in the less than or equal to 10-micrometer fraction, six of the fine-grained samples pulverized by Laboratory X were sent to two laboratories for quantitative TEM analysis of asbestos content using method EPA/600/R-93/116, "Method for the Determination of Asbestos in Bulk Building Materials." Both laboratories detected amphibole asbestos in all of the six samples, with asbestos concentrations ranging from 0.06 to 5.3 percent by weight. These results tend to show that over-pulverization of an asbestos-containing sample may reduce the size of asbestos fibers to a point where they cannot be detected by PLM.

Figure B-9. Low Asbestos Content Reported from Over-pulverized Sample



ILS PHASE TWO: Analysis of Fixed Mounted Slides

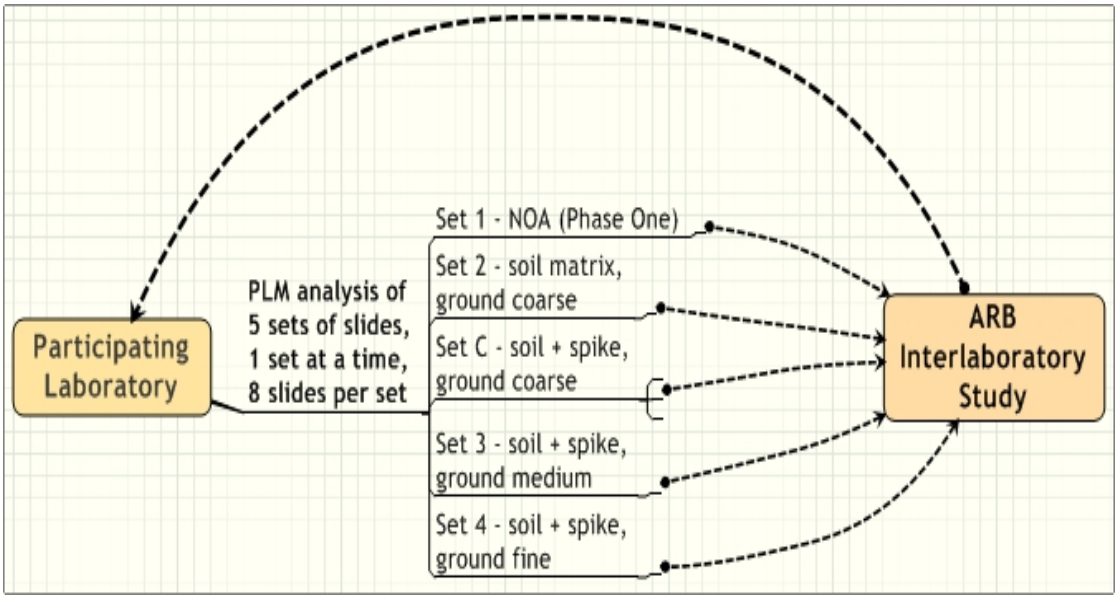
Phase Two: Materials and Method

ARB staff prepared fixed mounted slides of powdered material to remove the effect of sample preparation from the variability of the analytical results. Five sets of slides (Figure B-10), each set consisting of 8 slides, were prepared by permanently mounting powders from the following materials:

Set 1 NOA field sample, ILS Phase 1 sample aliquot.
Set 2 Soil matrix, ground coarse.
Set C Soil matrix spiked with 0.5 percent NIST asbestos tremolite, ground coarse.
Set 3 Soil matrix spiked with 0.5 percent NIST asbestos tremolite, ground medium.
Set 4 Soil matrix spiked with 0.5percent NIST asbestos tremolite, ground fine.

ARB staff gave one set of slides at a time to each participating laboratory. After analysis, the slides were returned to ARB staff, cleaned, and delivered to the next laboratory. (The descriptions of coarse, medium, and fine samples are given in the Pulverization Section on the following page.)

Figure B-10. ILS Phase Two: Round Robin Study of Fixed Slides



Soil Matrix Selection

Several soils from California were examined under the stereomicroscope. Oil immersion slides of these soils were evaluated for mineral components using PLM (BH-2, Olympus, Center Valley, PA). Selection criteria for choosing a soil matrix included the absence of asbestos fibers, minimal content of asbestos interference minerals (e.g., amphiboles and pyroxenes), and low content of minerals that may obscure asbestos fibers (e.g., iron(III) oxide-hydroxides, clay minerals).

The soil chosen was a coarse sandy loam from the Montpellier series which consists of well or moderately well drained soils formed in old alluvium from granitic rock sources, with its type location in San Joaquin County, California.

Asbestos Spike

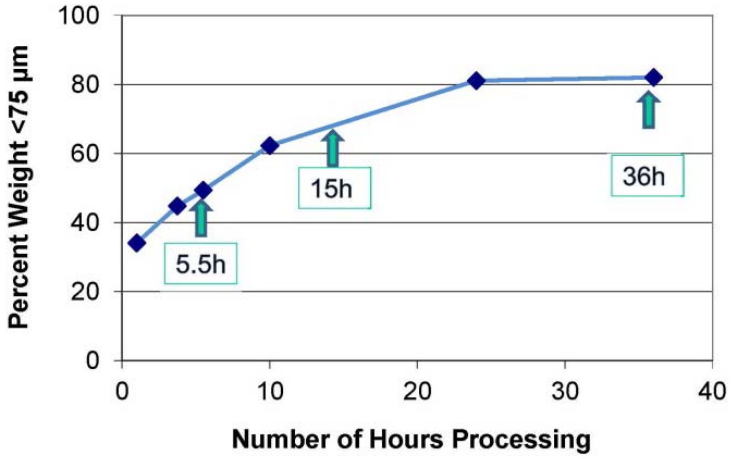
Asbestos standard reference materials (SRM) were obtained from NIST. SRM Number 1867a, Uncommon Commercial Asbestos, consisted of actinolite asbestos, anthophyllite asbestos, and tremolite asbestos. Tremolite was chosen as the spike material because it occurs in California as an asbestos mineral and is not as obvious to detect as the green, oftentimes pleiochroic, actinolite asbestos. Chrysotile, the most common asbestos in California, was also not chosen for the study because the morphology of this sheet silicate asbestos is distinctly different from the amphibole asbestos and would be very easy to identify.

Soil samples were oven-dried in a constant-temperature oven for 15 hours at 221 ºF (105 ºC), weighed, and spiked under the fume hood with the tremolite standard reference material to obtain a concentration of 0.5 percent tremolite asbestos by weight in the sample. The soil samples and tremolite were placed in cylindrical metal sample holders together with three metal grinding balls, and then labeled and double-sealed for pulverization using a ball mill, each for a specific number of hours. This was the only pulverization equipment available at the institution where the ILS fixed mounted slides were prepared.

Pulverization

Preliminary experiments were conducted on how the duration of grinding affected the percentage of the less than 75-micrometer (µm) fraction in oven-dried Montpellier soil samples that were pulverized with a ball mill. After milling, weight percentage of the less than 75-micrometer fraction was determined by dry sieving one gram of pulverized sample through a covered 75-micrometer mesh and shaking for 20 minutes. ARB staff observed an increase in the weight percentage of the less than 75-micrometer diameter particles as the grinding time increased. Based on these experiments, staff chose grinding durations of 5.5 hours, 15 hours, and 36 hours using the ball mill to obtain coarse, medium, and fine-grained samples, respectively (Figure B-11).

Figure B-11. Weight Percentage of <75-µm Particles vs. Grinding Duration



Preparation of Fixed Mounted Slides

Fixed mounted slides were prepared by an experienced laboratory technician with the guidance of ARB staff. Petropoxy 154 (Burnham Petrographics LLC, Rathdrum, ID) was the chosen mounting medium because of its well-defined refractive index (1.54) and its chemical stability. It is an epoxy-based mounting medium with low viscosity and a long shelf life.

The fixed mounted slides were prepared with particle loading of 25 to 50 percent and covered with size 1 glass cover slips (glass thickness of 0.13-0.17 millimeter). Slide identification was etched with a diamond pen on the bottom of the slide. Each set was kept in a separate box for distribution to the microscopists during the round robin study.

For the ILS Phase Two, five pulverized soil samples were used to make five sets of mounted slides, each set consisting of 10 slides. Eight slides per set were analyzed for the study and the remaining two were kept in reserve, in case replacements were needed.

Powdered Samples

For each set of fixed slides, microscopists were provided with approximately one milliliter of unmounted sample powder in a sealed glass vial. This gave the microscopist an opportunity to determine the asbestos optical characteristics of the same sample material as that mounted on the fixed slide, as indicated in M435 Table 3.

Samples and Instructions Given to Microscopists

At the beginning of each week, ARB staff gave each laboratory one set of fixed mounted slides (eight slides per set) and about one milliliter of the respective unmounted sample material. Microscopists were instructed to do a 400-point count, per M435, for each set. Analytical results were collected the same day of the following week.

Phase Two: Results and Discussion

Effect of Particle Size

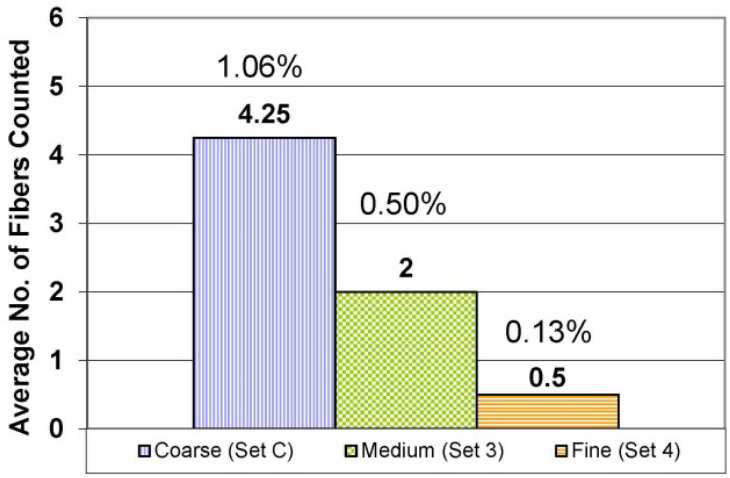
Analytical results from 400-point count of asbestos in coarse, medium, and finely-ground samples spiked with 0.5 percent tremolite showed that the average number of fibers reported decreased as the sample was ground finer (Table B-3). This was consistent with Phase One results.

Table B-3. Effect of Particle Size on Reported Asbestos from 400-Point Count

	Number of Fibers Reported				
Sample	**Lab I**	**Lab J**	**Lab K**	**Lab L**	**Observations**
Set C Coarse Spiked	**4**	**7**	**6**	**0**	Asbestos fibers reported by **three** laboratories in **coarse** sample.
Set 3 Medium Spiked	**1**	**7**	**0***	**0***	Asbestos fibers reported by **two** laboratories in **medium** sample.
Set 4 Fine Spiked	**0***	**2**	**0**	**0**	Asbestos fibers reported by **one** laboratory in **fine** sample.
	0*- indicates "trace" or "<0.25%" was reported (asbestos seen but not under a point during count).				

The average numbers of fibers reported by all four laboratories are shown in Figure B-12. Although the three samples analyzed had the same concentration of the tremolite spike, only the coarse- and medium-ground samples were reported to contain asbestos greater than 0.25 percent. If this sample was being evaluated with respect to the Asbestos Airborne Toxic Control Measure (ATCM) for Surfacing Applications that is enforceable at 0.25 percent by point count, the ATCM requirements would not be applicable for the finely-ground sample.

Figure B-12. Average Number of Fibers Reported from Coarse, Medium, and Fine Samples Spiked with 0.5% Tremolite*



*Percentage based on M435 400-point count analysis

Effect of Laboratory Asbestos Fiber Identification Criteria

ARB staff reviewed the number of asbestos fibers reported from 400-point count analyses by each laboratory during both phases of the ILS and noticed a laboratory effect first observed during the ILS Phase One (Figure B-6). The tally of asbestos fibers reported from all 400-point count analyses during both ILS phases (Table B-4) indicated that the laboratories reported a wide range in the number of asbestos fibers present in the same sample. These observations further indicated that laboratories do not have uniform asbestos fiber identification criteria.

Table B-4. Total Reported Asbestos Fibers from 400-point Counts ILS Phase One and Two

		Lab Q	Lab R	Lab S	Lab T
Phase One 400-pt count NOA	Sum of all fibers reported, 12 aliquots	1	41	24	0
Phase Two 400-pt count NIST tremolite + NOA	Sum of all fibers, Sets C, 3, 4 + Set 1, 2	6	61	5	0
	Totals	**7**	**102**	**29**	**0**

STUDY CONCLUSIONS

1. Laboratories use different M435 processing equipment and protocols. These result in varying particle size distributions of powders produced by each laboratory.

2. Although a very fine particle size distribution meets the pulverization performance requirement in M435 (i.e., majority of particles should be less than 75 micrometers in diameter), over-pulverization can lead to a lower percentage of asbestos reported.

3. Fiber identification criteria are not uniform among laboratories when analyzing for tremolite asbestos, which results in a wide range of asbestos concentration reported by different laboratories from M435 analysis of the same or similar samples. Although not tested, it is unlikely that this wide range of asbestos content would have been reported had the samples contained chrysotile, the more common form of asbestos in California, because of its distinctive fiber morphology.

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Appendix E

Crusher Cleaning Protocol and Rock Crushing Procedure

Operate and clean the jaw crusher under a negative air fume hood that uses a high-efficiency particulate air (HEPA) filter and has a minimum flow rate of 100 feet per minute (approximately 30.5 meters per minute). Be sure that this equipment is used in strict compliance with lockout/tagout and other safety procedures, as appropriate.

Crusher Cleaning Protocol

1) Purge the crusher by introducing about 1/4 liter (about 1 cup) of asbestos-free material* (e.g., white marble) into the crusher opening (Figure E-1).
2) Turn off the crusher and disconnect from electrical power. Remove the safety guard and lift out the feeder plate to clean the crusher (Figure E-2).
3) Use a vacuum cleaner equipped with a HEPA filter to clean the crusher opening and surrounding areas.
4) Use a disposable wire brush to loosen any remaining debris (Figure E-3).
5) Under a negative air fume hood enclosure with HEPA filter, use pressured air to clean the crusher.
6) Wipe down the crusher with disposable alcohol wipes (Figure E-4).
7) Clean the feeder plate using the same cleaning sequence (i.e., vacuum, brush, pressured air, alcohol wipes).
8) Change the hand gloves of the technician doing sample preparation.
9) Reinsert the feeder plate.
10) Introduce the second purge consisting of about 1/4 liter (about 1 cup) of asbestos-free material* (e.g., white marble).
11) Repeat the above cleaning procedure, taking care to use a new, clean, disposable wire brush and alcohol wipes. The cleaning procedure takes about 12-15 minutes.
12) Crusher is now ready for the next sample.

*Purging materials must have been tested by polarized light microscopy and transmission electron microscopy and shown not to contain asbestos (“blank material”). Materials used for purging during the cleaning procedures should be discarded appropriately.

Figure E-1. Purge the crusher.

Figure E-2. Lift out the feeder plate.

Figure E-3. Loosen debris with brush.

Figure E-4. Wipe down the crusher.

Rock Crushing Procedure

a) Turn on the jaw crusher and drop the M435 rock samples into the jaw crusher opening (Figure E-5).
b) After crushing, turn off the apparatus and enclose the pan in a plastic sleeve as you remove the crushed material (Figure E-6).
c) Under a fume hood enclosure with a HEPA filter, collect the crushed sample and transfer to a covered, temporary, clean pan or plastic container.
d) Store crushed sample under a negative air fume hood enclosure with a HEPA filter until pulverization.

Figure E-5. Drop sample into crusher.

Figure E-6. Enclose and remove pan.

Appendix F

Plate Grinder Cleaning Protocol

Operate and clean the plate grinder under a negative air fume hood that uses a high-efficiency particulate air (HEPA) filter. Be sure that this equipment is used in strict compliance with lockout/tagout and other safety procedures, as appropriate.

1) Turn off the plate grinder and disconnect from electrical power. Slightly increase the distance between plates (Figure F-1) by loosening the locking handle (Figure F-2) and turning the distance control knob. Reconnect to electrical power and turn on the plate grinder.
2) Introduce the first purge of the grinder using two 1/4 liter scoops (about 1 cup per scoop) of oven-dried, crushed limestone* (Figure F-3) or other asbestos-free material. The powder from the first purge should be discarded appropriately.
3) Use a negative air fume hood enclosure with HEPA filter during the entire cleaning procedure. Brush the chamber interior and surrounding parts. Slam the plate cover to loosen powdered debris.
4) For all vacuum procedures, use a vacuum cleaner equipped with a HEPA filtration system. Vacuum the interior, including the sample drawer below (Figure F-4).
5) Break open the plates and vacuum the plates' interiors.
6) Use a wire brush to clean the plates and remove caked material.
7) Vacuum the entire equipment.
8) Use alcohol wipes to clean the entire equipment.
9) Using a feeler gauge, reset the plate distance for M435 pulverization (Figure F-5). To get the fine grind, use the distance control knob to move the plates closer, so that they barely touch. Then back off the knob, about a half turn or less, to slightly increase the distance between plates. Lock the handle to set this plate distance.
10) Change the hand gloves of the technician doing sample preparation.
11) Introduce the second purge of the grinder using two 1/4 liter scoops (about 1 cup per scoop) of quartz sand. The powder from the second purge should be discarded appropriately.
12) Re-clean the interior chamber with a new brush and slam the plate cover to loosen powdered debris.
13) Vacuum the entire equipment, especially inside the plate feeder.
14) Run the plate grinder and use pressured air to clean the plate grinder.
15) Turn off the plate grinder, disconnect from power, and vacuum the entire equipment.
16) Use alcohol wipes to clean the entire equipment. The cleaning procedure takes approximately 12-15 minutes, depending on how experienced the technician is in cleaning this equipment.
17) The plate grinder is now ready for the next sample.

*Purging materials must have been tested by polarized light microscopy and transmission electron microscopy and shown not to contain asbestos ("blank material"). Materials used for purging during the cleaning procedures should be discarded appropriately.

Figure F-1. Increase distance of plates.

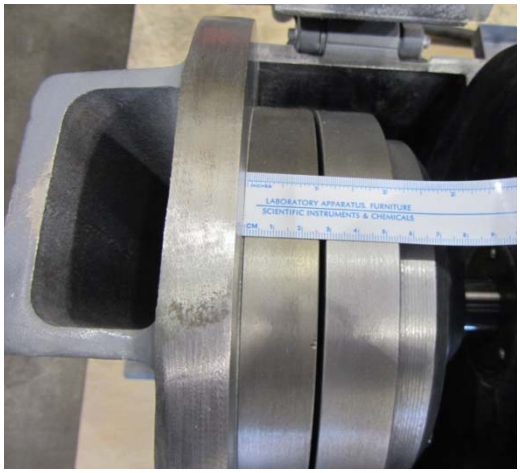


Figure F-2. Loosen the locking handle.

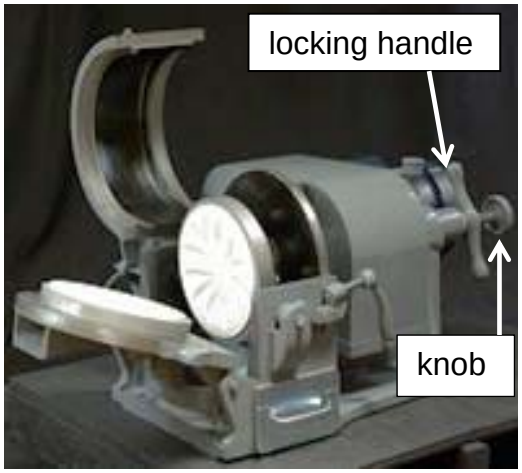


Figure F-3. First purge with limestone.

Figure F-4. Vacuum the sample drawer.

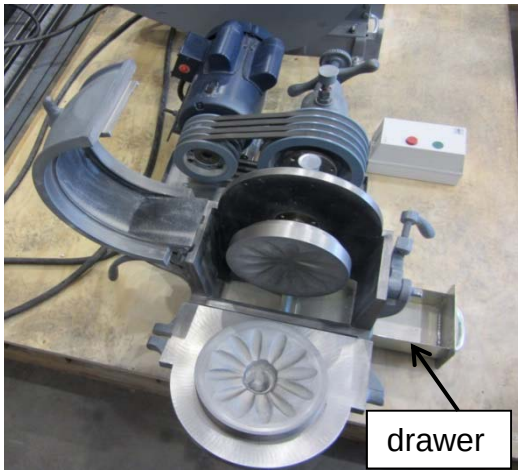
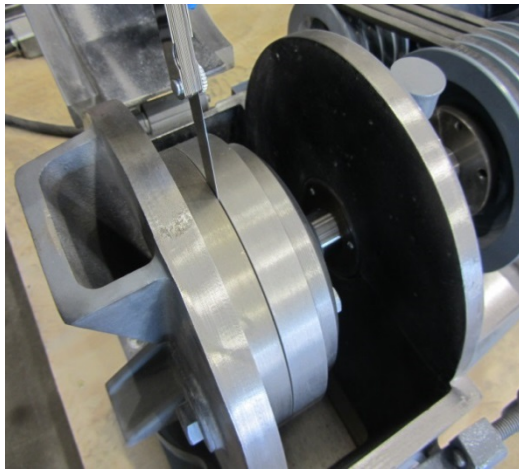


Figure F-5. Reset the plate distance.



Appendix G

Test Method 435 Sample Processing Procedures: The Addition of a Mixing Procedure and Its Effect on Sample Homogeneity

INTRODUCTION

Background

The California Air Resources Board's (ARB) Test Method 435 (M435) requires the following sample processing steps prior to analysis of possible naturally occurring asbestos-containing samples by polarized light microscopy (PLM):

1. Drying of field sample.
2. Crushing to less than 9 1/2-millimeter (3/8-inch) diameter particles.
3. Riffle splitting to reduce the size of the original field sample (to one pint).
4. Pulverizing the one pint sample to a powder sample (majority should be less than 75-micrometer diameter particles that pass through a 200 Tyler mesh).

An aliquot of the sample powder is then taken for PLM analysis. The amount of powdered sample actually viewed under the polarized light microscope for a M435 analysis is about 40 milligrams. Oftentimes, this is a sample size reduction of about a 10^{-5} (or approximately 1/100,000 of the original sample) when compared to the field sample received by the laboratory.[1] Therefore, it is important that steps be taken to ensure that this small subsample for PLM analysis is representative of the bulk field sample submitted.

Although the pulverization procedure can mix the sample to a small degree (depending on the type of equipment used), this step is not specifically intended to homogenize a large sample. Therefore, ARB staff recommends the addition of a mixing step to increase the homogeneity of the M435 sample prior to taking the aliquot for PLM analysis. This additional step would improve the accuracy and precision of M435 analyses.

After preliminary investigations, ARB staff chose to use a 3-dimensional (3-D) mixer (88 Mixer System Schatz Model 4 (1A), Inversion Mixers, Ponaka AB, Canada) to assess the benefits of adding a mixing procedure to M435 processing. (See link in References). The motion of the 88 mixer is based on the Schatz inversion kinematic, which uses a combination of three types of motions: rotation, translation (reciprocation), and inversion. The alternating motion has a thickening and thinning effect on the sample being mixed. The resulting eddies produce a changing and predictable energy gradient that mixes the sample. The Schatz kinematic 3-D mixer was selected because

[1] Assuming 1-pint (approximately 473 milliliters) field sample aliquot, particle density of 2.65 grams-per milliliter, and 30 percent porosity.

it has been established as an effective mixer of solids such as powdered medicines, ceramics, foodstuffs, and others. A sample can be mixed in its own container as long as the container size is not larger than the mixer's capacity. This helps avoid cross-contamination during sample preparation.

Purpose

The purpose of this study was to observe the effect of adding a mixing step on sample homogeneity and the resulting variation of analytical results for a target analyte.

Preliminary Study

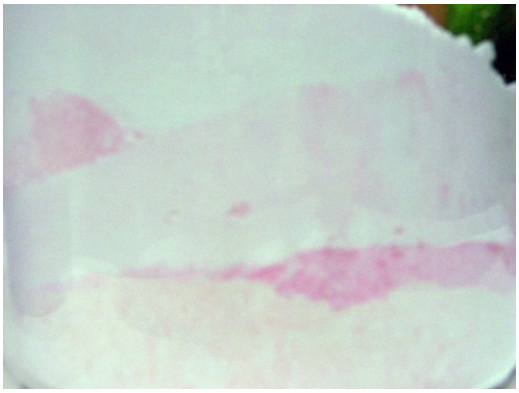
ARB staff conducted a preliminary study on mixing by observing the effect of pulverization (using a plate grinder) on the distribution of red-colored tracer material.

Approximately 3,380 grams of oven-dried salt (sodium chloride) was spiked with 2.83 grams of dyed red chalk consisting of calcium carbonate (Figure G-1A). Both materials were combined and pulverized using a plate grinder. After pulverization, there was visible uneven distribution of reddish color in the powdered sample (Figure G-1B).

Figure G-1. Preliminary Mixing Test Using Salt and Red Chalk

A. Crushed salt and red chalk (spike) before pulverization.

B. Salt and red chalk (spike) after Pulverization using a plate grinder.



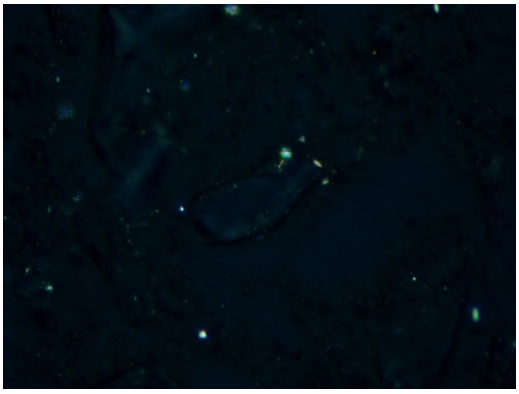
C. Stratified sample before mixing.

D. Homogenized sample after mixing.

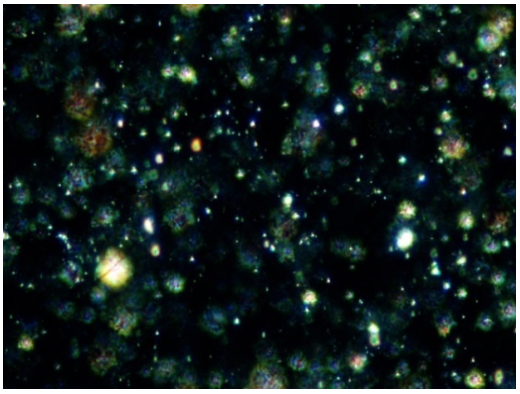
Upon transfer of the powdered sample to a one-gallon (approximately 4-liter) glass jar, distinct layering in the pulverized sample was visible (Figure G-1C). Some layers had relatively higher concentrations of spiked, red-dyed material, while other layers had virtually no spike material and were generally white. However, after homogenization using the 3-D mixer at 40 revolutions per minute (rpm) for four minutes, the entire powdered sample appeared to be uniformly *pink* and homogeneous (Figure G-1D).

Figure G-2 shows the salt sample's optical characteristics using PLM. The salt matrix before mixing appears to be mostly dark (isotropic) under crossed polars using PLM (Figure G-2A) with few impurities as indicated by the small number of bright particles. After mixing for four minutes, the homogenized powder is seen with the finely disseminated, highly birefringent, evenly-distributed chalk powder observed as bright particles against the dark salt matrix (Figure G-2B). Even at 100X magnification (mag), the chalk appears to be relatively homogeneous.

Figure G-2. PLM Analysis of Preliminary Mixing Test



A. Salt matrix (isotropic, dark) before mixing. 100X mag, PLM, crossed polars.



B. Pulverized salt matrix and chalk (bright particles) after mixing. 100X mag, PLM, crossed polars.

MATERIALS AND METHODS

Study Design

Based on the results of the preliminary study, ARB staff conducted further tests using different matrix and spike materials. Marble was chosen as the matrix because it can be easily dissolved in hydrochloric acid (HCl). Non-asbestos crystalline actinolite was chosen as the spike because it is insoluble in HCl and its prismatic crystals resemble the shape of asbestos fibers, but without the toxicity. Acid-dissolution tests of the powdered marble were done to determine the mass of insoluble crystals (impurity) in a known mass of pulverized marble.

Two actinolite spike concentrations were chosen (0.25 percent and 0.10 percent by weight, wt%) for the study. Samples were homogenized using the 3-D mixer at 40 rpm. Mixing periods of 0, 3, 5, and 7 minutes were selected.

Three replicate experiments per mixing duration were performed. Three core subsamples were obtained from each replicate sample. Core subsamples were dissolved in HCl to determine the mass of actinolite in the subsample. The study sample collection design is shown in Table G-1.

Table G-1. Sample Collection Design

Mixing Time (min)	0.25 wt%			0.10 wt%			Grand Total Samples
	No. of Replicates	No. Core Subsamples Per Replicate	Total	No. of Replicates	No. Core Subsamples Per Replicate	Total	
0	3	3	9	3	3	9	**18**
3	3	3	9	3	3	9	**18**
5	3	3	9	3	3	9	**18**
7	3	3	9	3	3	9	**18**
Total			36			36	**72**

Sample Preparation

About 130 kilograms of natural marble and one kilogram of prismatic actinolite were separately dried in a constant temperature oven at 105 degrees Celsius (ºC) for 24 hours. These materials were crushed to 9 1/2-millimeter (3/8-inch) diameter particles with a Chipmunk Jaw Crusher (Bico Braun International, Burbank, CA) (Figure G-3A). Subsequently, each material was pulverized with a plate grinder (UA Disc Pulverizer, Bico Braun International, Burbank, CA) (Figure G-3B and 3C). Crushing and pulverization were performed at different times to avoid cross-contamination between the two procedures.

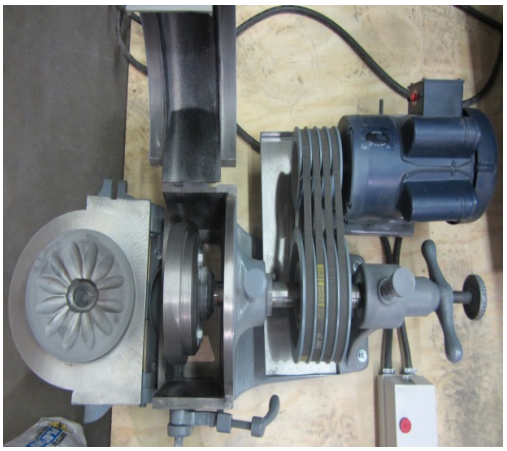
The blank marble powder was dissolved with HCl to identify and quantify the natural impurities. The pulverized actinolite spike was analyzed by PLM (Figure G-3D) to verify the composition.

Determining the Impurity of the Pulverized Marble

Prior to mixing the actinolite-marble mixture, the purity of the marble was checked. To do this, approximately 2,600 grams of pulverized marble was placed in each of pre-weighed 24 plastic containers. The plastic containers were shut and the lids were

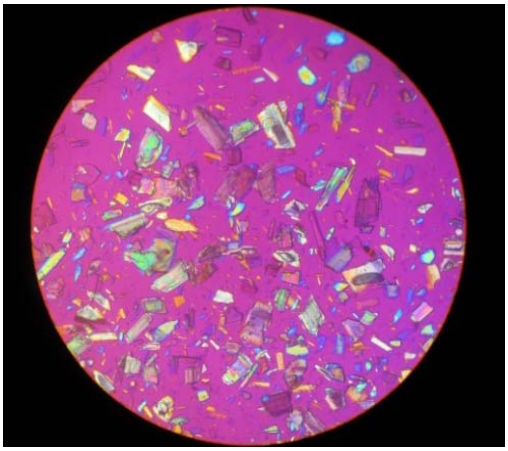
Figure G-3. Sample Preparation of Actinolite and Marble

A. Crushed sample.



B. Plate grinder for pulverization.

C. Pulverized marble.



D. Actinolite by PLM (100X mag, crossed polars, gypsum plate, field of view diameter = 2mm).

sealed with duct tape. Each marble sample was placed in the Schatz kinematic 3-D mixer, secured, and homogenized at 40 rpm for seven minutes. At the end of each mixing, the samples were allowed to stand for five minutes for dust inside to settle. Subsequently, three core subsamples of the homogenized marble powder were collected. The method for core subsample collection is provided below. The three core subsamples were mixed to provide one subsample from each container. The combined subsamples were analyzed for impurities (acid insoluble solid). Each of these marble powders in the 24 plastic containers was used for subsequent mixing tests of actinolite-marble mixture.

Preparation of Actinolite-Marble Samples

To prepare approximately a one-liter (L) actinolite-marble mixture sample with 0.25 weight percent actinolite spike, each of the approximately 2,600 grams of pulverized marble in 12 plastic containers was reduced to exactly 2,463.83-grams in the pre-weighed, plastic container (Figure G-4A). The actinolite spike (6.175 grams) for each sample was weighed on a tared glassine paper using a microbalance with sensitivity of 10^{-4} grams, labeled, and stored for use during the experiment. Twelve samples spiked with 0.25 weight percent actinolite were prepared. The remaining 12 containers of approximately 2,600 grams of pulverized marble were used to prepare a set of 12 samples spiked with 0.10 weight percent actinolite. For this sample concentration, 2.470 grams of actinolite and 2,467.53 grams of pulverized marble were mixed in each sample container.

Figure G-4. Preparation of Actinolite-Marble Sample Mixture

A. Weighing the marble matrix.

B. Adding the pre-weighed actinolite.

C. Covering actinolite with marble.

D. Container was sealed.

To have a uniform starting position of the actinolite spike in all of the marble sample replicates, an approximately 2-centimeter wide by 2-centimeter deep well was made in

the center of the upper surface of the marble powder. The actinolite powder was placed in the well (Figure G-4B) and covered with marble powder (Figure G-4C). The plastic container was shut and the lid was sealed with duct tape (Figure G-4D).

Procedure for Unmixed Samples (3-D Mixing Duration = 0 Minutes)

After spiking the marble sample with actinolite, the sealed sample container was manually inverted upside down three times in a quick succession (Figure G-5A) to represent minimal mixing that would have occurred if the target analyte were pulverized concurrently with the matrix material.[2] The sample container was placed on a flat surface for five minutes to allow inside dust to settle. Subsequently, three core subsamples of the powder were collected. Three replicate samples of "zero minute" mixing were prepared for each spike concentration resulting in nine core subsamples collected for the 0.25 weight percent actinolite spike samples and another nine core subsamples for 0.10 weight percent actinolite spike samples (Table G-1).

Procedure for Mixed Samples (3-D Mixing Duration = 3, 5, and 7 Minutes)

After inverting the sealed sample container three times, as described above, each container was placed in the Schatz kinematic 3-D mixer, secured, and mixed at 40 rpm for the appropriate experimental duration (3, 5, or 7 minutes) (Figure G-5B). The mixed sample was gently placed on a flat surface for five minutes to allow inside dust to settle. Subsequently, three core subsamples of the mixed powder were collected. Each sample was replicated three times. A total of nine samples were mixed for samples spiked at 0.25 weight percent actinolite and another nine samples mixed for samples spiked at 0.10 weight percent actinolite. Three core subsamples were collected from each replicate sample (Table G-1).

Figure G-5. Mixing Samples Using Hand and 3-D Mixer

A. Hand mixing the sample.

B. Sample was secured in 3-D mixer.

[2] Spikes were not introduced before crushing or pulverization because a small amount of material is always "lost" in these processing procedures (e.g. in equipment, through fume hood, etc.); therefore, the target concentration after processing could be slightly different.

Collection of Core Subsamples

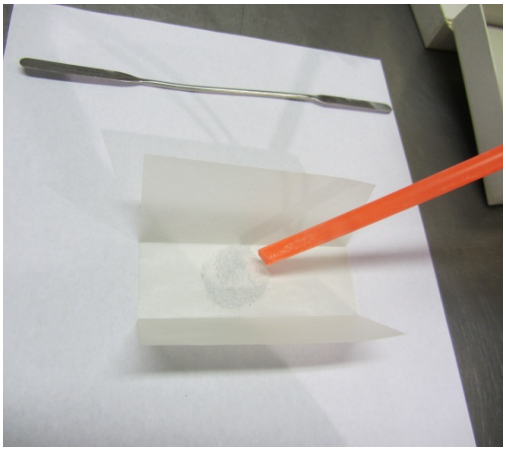
Three core subsamples were taken from each sample using a clean, single-use "sample thief" (Figure G-6A). The sample thief consisted of a large plastic straw with a diameter of approximately 8 millimeters and a length of 20 centimeters with one end diagonally cut. After homogenization, three sample thieves were inserted vertically in three locations at random on the sample surface (Figure G-6B) down to the bottom of the sample container.

Each core subsample was collected and the powder was placed on a tared glassine paper (Figure G-6C), weighed, and stored in the folded glassine paper (Figure G-6D) until the next step (dissolution). The sample thief was appropriately discarded after each use. A total of 72 core subsamples were collected for this study (Table G-1).

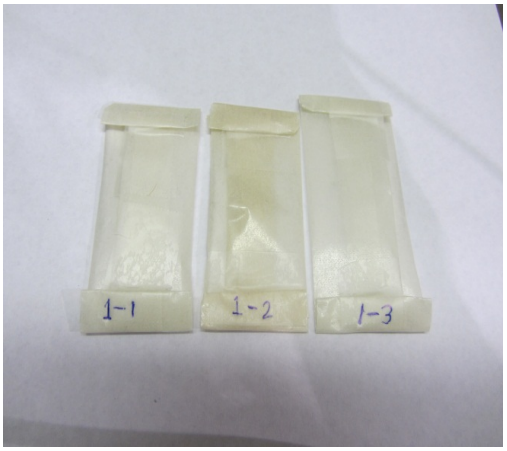
Figure G-6. Obtaining Core Subsamples

A. Sample thief.

B. Taking three core subsamples.



C. Core subsample collected.



D. Core subsamples labeled and stored.

Sample Dissolution

The objective was to determine the mass of the actinolite spike in each of the marble-actinolite core subsamples by dissolving the calcium carbonate in the marble-actinolite mixture and quantifying the acid-insoluble actinolite. The purity of the marble was determined by dissolving a known mass of pulverized marble with HCl to determine the percentage of acid-insoluble solids (impurity) in the untreated marble. Each sample container had a blank reading for initial impurity content.

Each core subsample was placed in a 250-milliliter beaker and 30 milliliters of 10 percent HCl was added to dissolve the marble (Figure G-7A). After the reaction was completed, the digest was filtered through a pre-weighed, 47-millimeter Teflon filter with 0.45-micrometer openings using a filter-suction system (Figure G-7B). The residue was rinsed with distilled deionized water. The Teflon filter with residue was placed on a clean tray and air-dried for 24 hours. It was post-weighed to determine the mass of the remaining residue from each core subsample. The difference between the mass of the dried filter with residue and the empty Teflon filter is the mass of the spike. The mass of acid-insoluble impurities from an equivalent mass of marble matrix was also deducted from the total mass of the collected residue. The same procedure was used for all 72 subsamples.

Figure G-7. Dissolution of Marble-Actinolite Mixture and Filtration

A. Dissolution of core subsamples.

B. Suction filtration of acid digest.

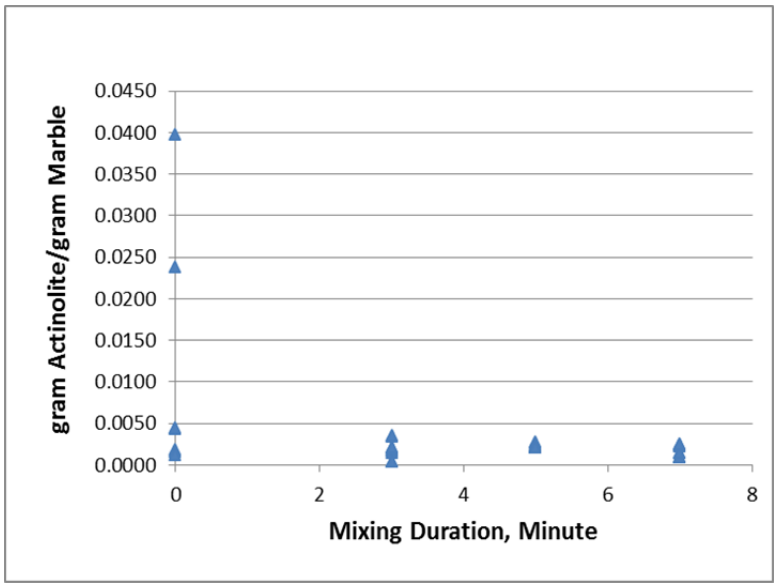
The post-dissolution concentration of actinolite in each core subsample was calculated in grams of actinolite per gram of marble. The average and standard deviation for each treatment (subsamples with the same duration of mixing) were also calculated for each concentration of actinolite (0.25 weight percent and 0.10 weight percent spike).

RESULTS AND DISCUSSION

The results for samples spiked with 0.25 weight percent actinolite are shown in Figures G-8 and G-9. At zero minute mixing (sample container was manually turned upside down three times, no use of 3-D mixer), there was great variability in actinolite concentrations measured, ranging from 0.0012 to 0.0398 g actinolite/g marble. As the mixing duration increased, the range of actinolite concentrations became narrower with a sharp decrease in the standard deviations (Figure G-9).

These results indicate that unmixed samples have much higher variability of actinolite concentrations when compared to samples that were homogenized using the 3-D mixer. When compared to the unmixed samples, the mixed samples (3, 5, and 7 minutes) consistently showed lower (and closer to the spiked concentration) average concentrations of actinolite. The standard deviations of the nine replicates of mixed samples decreased significantly indicating that the actinolite is more evenly distributed within the marble matrix. Therefore, the mixed samples resulted in more accurate and precise measurements of actinolite.

Figure G-8. Actinolite-Spiked Samples (0.25 Wt Percent) and Mixing Duration



The data from the 0.10 weight percent actinolite-marble samples follow the same trends observed in the 0.25 weight percent actinolite-marble samples (Figures G-10 and G-11). The actinolite concentrations measured from unmixed samples are significantly higher and more variable than the actinolite concentrations from the mixed samples. These samples were spiked with 0.10 weight percent actinolite but the actinolite concentrations measured from unmixed samples were approximately three times higher than 0.10 weight percent, indicating that the core subsamples from unmixed samples included pockets of unmixed actinolite.

Figure G-9. Average and Standard Deviation of Actinolite-Spiked Samples (0.25 Wt Percent) and Mixing Duration

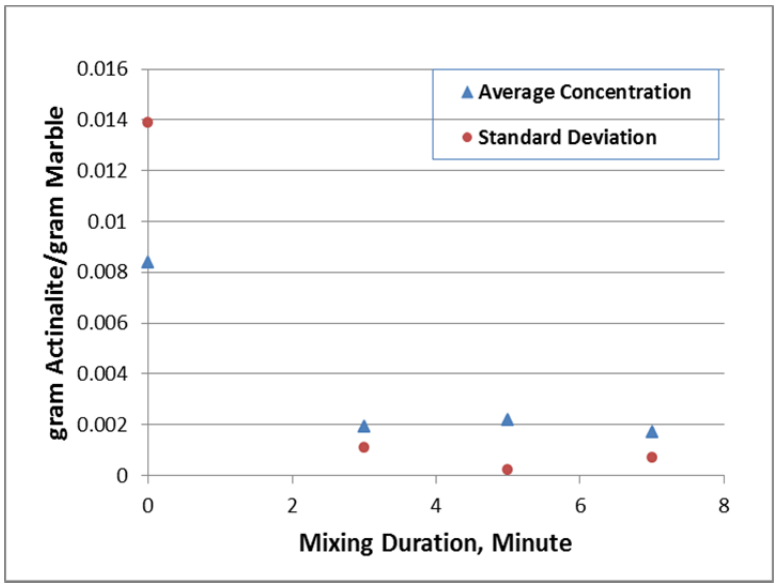
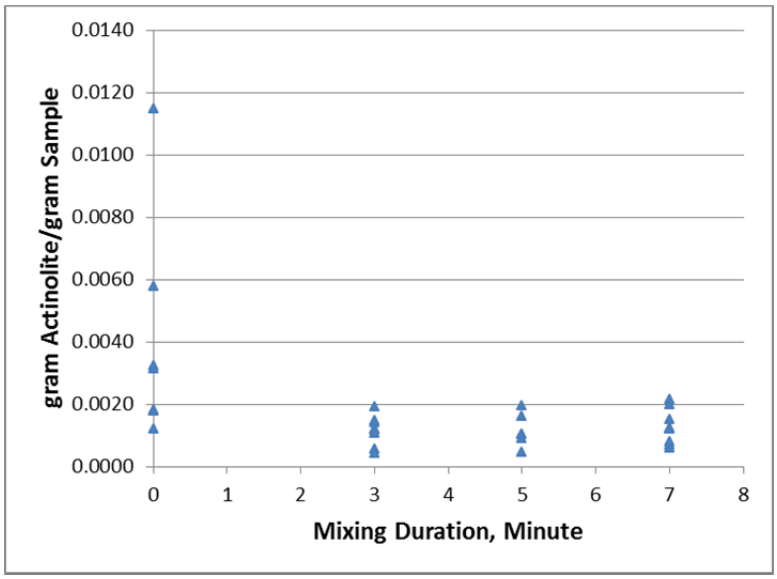
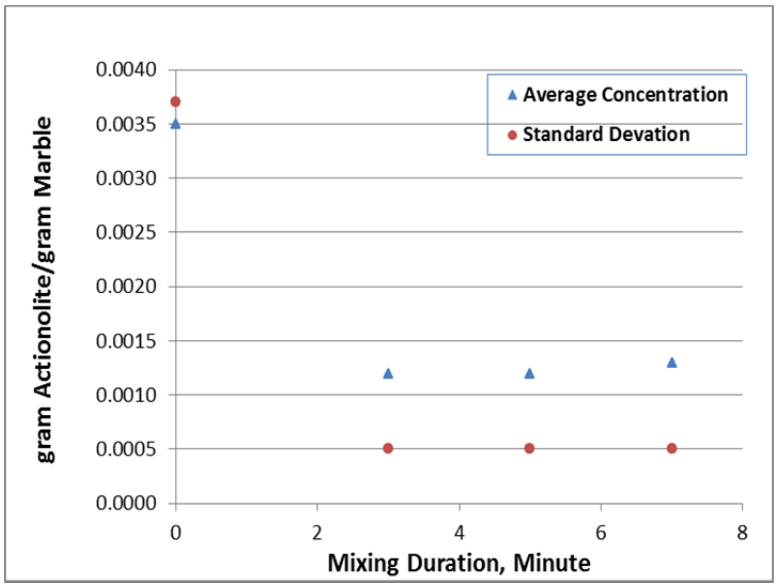


Figure G-10. Actinolite-Spiked Samples (0.10 Wt Percent) and Mixing Duration



In contrast, the mixed samples did not show this large variability. As shown in Figure G-11, the standard deviations of mixed samples were about 13 percent of that for the unmixed samples indicating that, in the mixed samples, the actinolite is more evenly distributed within the marble matrix.

Figure G-11. Average and Standard Deviations of Actinolite-Spiked Samples (0.10 Wt Percent) and Mixing Duration



The average actinolite concentrations reported in the mixed samples are about the same as the initial spike concentrations of 0.10 weight-percent or 0.25 weight-percent. The optimal mixing duration represents the minimum amount of time needed to homogenize the unmixed sample so that accurate and repeatable results can be obtained from taking the subsamples. For both concentrations, three minute mixing was sufficient to provide optimal concentrations. As shown in all four figures, there is no significant difference in mixing after three minutes, all with the lowest standard deviations. This may suggest that mixing of about a liter of powdered actinolite-marble mixture for three minutes is sufficient to get the optimal mixing.

CONCLUSIONS

In summary, the concentrations of actinolite in unmixed samples were highly variable (higher standard deviation) accompanied with a higher average. In contrast, samples that were homogenized with the 3-D mixer had much less variability (lower standard deviation) with a lower (and more accurate) mean actinolite concentration.

Both the preliminary chalk-salt mixing study and this actinolite-marble study suggest that homogenization of powdered mineral samples before analysis for asbestos content is beneficial. The results clearly show that the addition of a mixing step into M435 processing procedures can greatly improve the accuracy and precision of the analytical results.

REFERENCES

Inversion Machines Ltd. Ponoka, AB Canada (2013). One Gallon Model. Accessed April 26, 2017: https://inversionmixers.com/one-gallon-model-2/

Appendix H

Asbestos Quantification by Point-Counting: Statistical Decision-Making Errors

California Air Resources Board Test Method 435 Point-Counting Procedure

Asbestos quantification per California Air Resources Board (ARB) Test Method 435 (M435) is performed by a point-counting procedure. Point-counting is a well-established, standard technique in petrography, the description and classification of rocks, especially by microscopic examination, for determining the relative areas occupied by separate minerals in thin sections of rocks. An ocular reticle (point array) or crosshair reticle is used to visually superimpose points on the microscope field of view. Using forceps or scalpels, approximately five milligrams of powdered sample material are mounted on each of eight slides (total of 40 milligrams) using the appropriate refractive index liquid. A total of 400 points superimposed on either asbestos fibers or non-asbestos particles must be counted over at least eight different preparations of representative subsamples.

The percent (%) asbestos is calculated as follows:

% asbestos = (a/n) 100%

a = number of asbestos counts
n = number of nonempty points counted (400)

ARB staff has observed that some laboratories offer more “sensitive” M435 analyses that include counting more than 400 points. ARB staff supports counting more than 400 points but recommends that it be done in multiples of 400 (i.e., 800, 1,200, etc.). If counts greater than 400 are performed, but not in multiples of 400, the chances of reporting a sample to be less than 0.25 percent may increase, when, in fact, it is actually greater than 0.25 percent.

Introduction to Decision-Making Errors – False Positives and False Negatives

An analyst often makes conclusions about a population based on a subset of data that is available. In such instances, there is always a chance that the analyst may report a wrong conclusion when the truth is unknown. For example, in the point-count method, if the true proportion of asbestos is less than 0.25 percent, but the analyst finds a greater percentage of asbestos in a subset of data and subsequently declares the larger, original field sample to be greater than 0.25 percent, then the reported result is said to be a “false positive” (“false” indicates a wrong conclusion is being made, and “positive” indicates that the reported asbestos content is present above a certain threshold, e.g., 0.25 percent).

On the other hand, if the true proportion is greater than or equal to 0.25 percent, but the analyst finds less than 0.25 percent in the subset of data, then the reported result is said to be a “false negative” (“false” indicates a wrong conclusion is being made, and

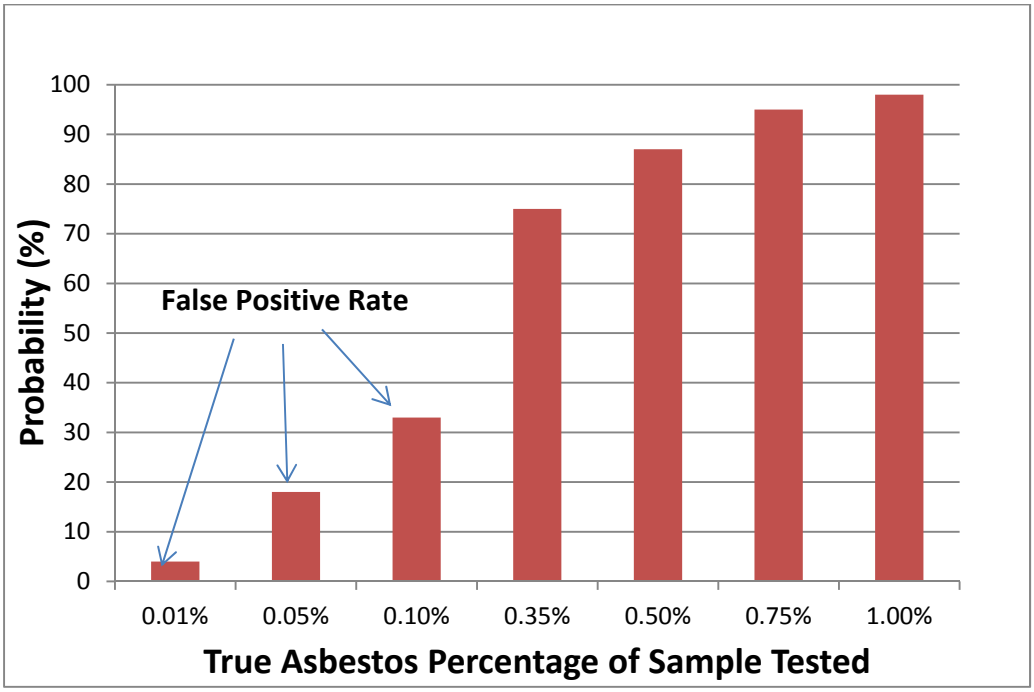
"negative" indicates that the reported amount of asbestos identified is below the threshold).

Declaring a sample to have asbestos either above (or below) a certain threshold is a "true or false" process. In statistics, this is modeled as a binomial process. The following figures and discussion take into account only the statistical probability associated with the binomial process and do not take into account the other sources of variability associated with M435.

False Positives/Negatives as a Function of Sample Concentration

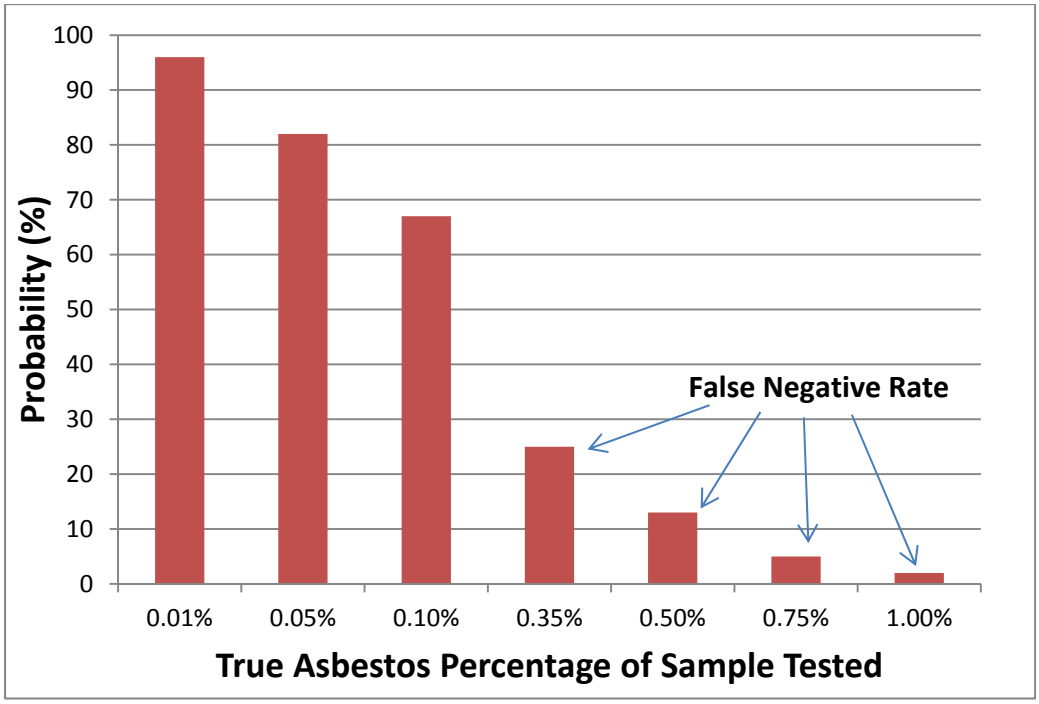
Under the scenario of counting 400 points, if the true asbestos content is 0.75 percent by point count, the point-count method will correctly identify the sample as being greater than or equal to 0.25 percent 95 percent of the time (Figure H-1). Therefore, due to chance, the point-counting under these conditions will provide false negatives five percent of the time (100 percent – 95 percent = 5 percent). That is, the "false negative" probability rate is five percent. See Figure H-2.

Figure H-1. Probability of Declaring Sample ≥ 0.25 Percent
400 Point Count



As Figure H-1 shows, if the true asbestos content is 0.05 percent by point count, the point-count method will *incorrectly* identify the sample as being greater than or equal to 0.25 percent 18 percent of the time (i.e., the false positive rate is 18 percent). Conversely, the point-count procedure will correctly identify this sample to be less than 0.25 percent 82 percent of the time (100 percent – 18 percent = 82 percent) as shown in Figure H-2.

Figure H-2. Probability of Declaring Sample < 0.25 Percent
400 Point Count



As can be seen from both figures, the probability of making a false positive or false negative declaration increases near the decision threshold (in this case, 0.25 percent) but decreases as the true asbestos percentage deviates from the threshold.

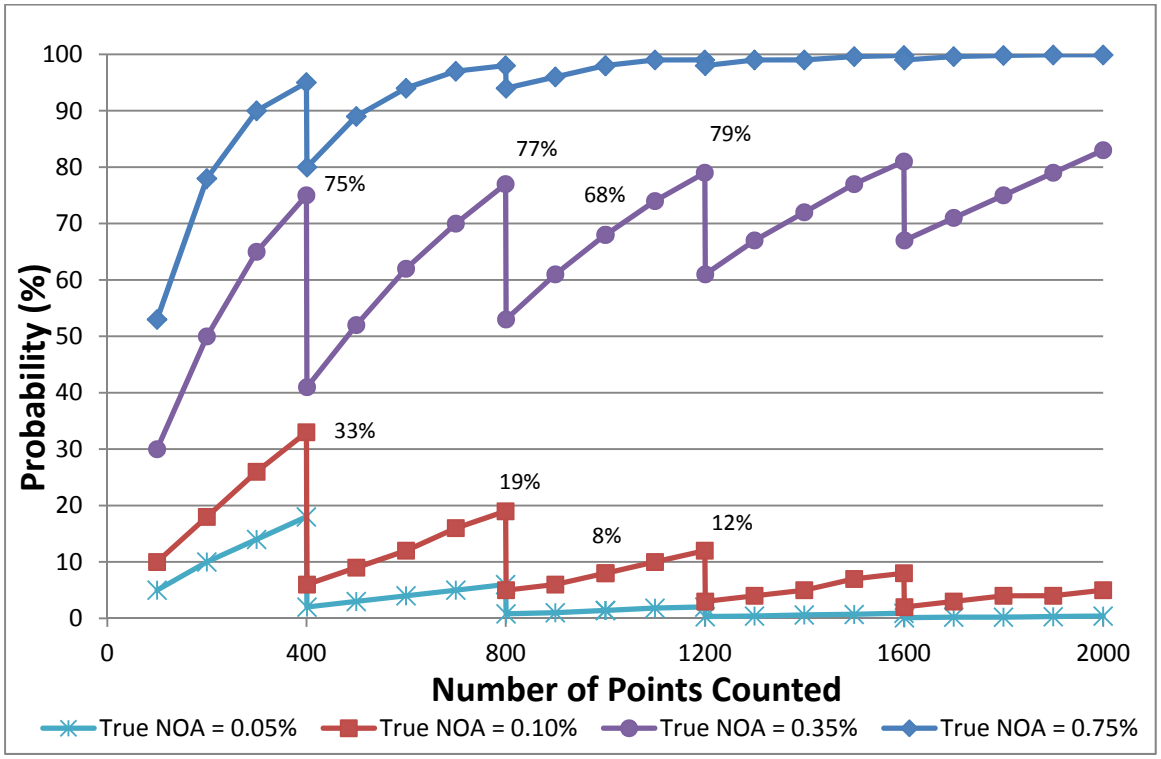
False Positives/Negatives as a Function of Number of Points Counted

The rate of correctly identifying an asbestos sample as above or below a certain threshold (e.g., 0.25 percent) also changes as the number of points counted changes; therefore, the rate of reporting false positives or false negatives will change as well. Figure H-3 illustrates this phenomenon.

The sharp peaks in probabilities occurring at increments of 400 (points counted) are due to the rejection rule of 0.25 percent. When using this threshold and counting in multiples of 400 (i.e., 400, 800, 1,200, etc.), the number of fibers it takes to "tip" the threshold is in whole numbers (e.g., for 400 points, 0.25 percent is one fiber; for 800 points, 0.25 percent is two fibers, etc.). If an analyst were to count one additional point (i.e., 401), then 0.25 percent multiplied by 401 would be 1.0025 fibers. Since an analyst cannot detect and count partial asbestos fibers, the real-world threshold in a 401 point count is two fibers. Two fibers will remain as the threshold until the point count increases above 800 points.[1] The following discussion considers cases when additional points (800, 1,000, and 1,200) are counted.

[1] The first sharp drop in probability in Figure H-3 occurs at the 401 point count. A progressive increase in probabilities is observed as the point count increases from that point on until the next multiple of 400 is reached, that is, 800 points.

Figure H-3. Probability of Declaring Sample ≥ 0.25 Percent
100-2000 Point Count



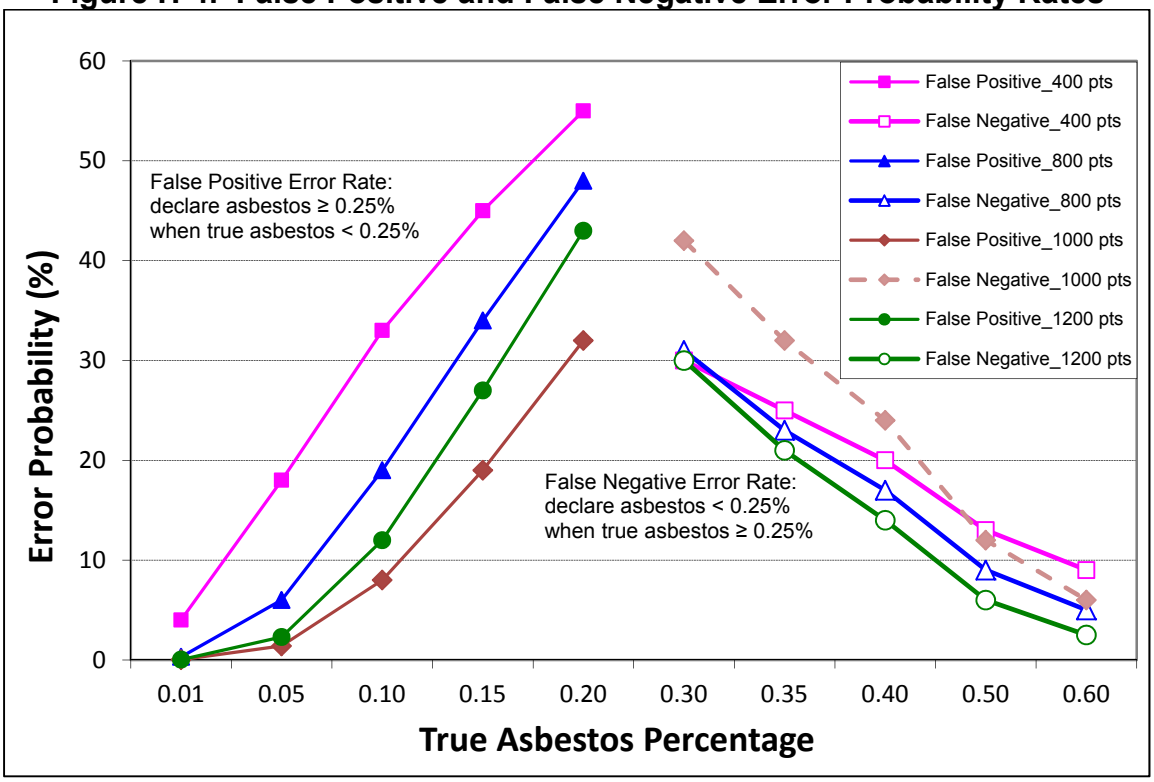
As indicated in Figure H-3, when the true asbestos content is 0.35 percent (shown by the line with circles), the point-count procedure will correctly identify the sample as being greater than or equal to 0.25 percent 75, 77, and 79 percent of the time when 400, 800, and 1,200 points are counted, respectively. However, when 1,000 points from the same sample are counted, the procedure will only correctly identify the sample as being greater than or equal to 0.25 percent 68 percent of the time. **Therefore, the false negative error rate is actually higher for the 1,000 point-count than the 400 and 800 point-count.**

The above scenario does not apply to the false positive error rate. For instance, if the true asbestos content is 0.10 percent by point count, the point-count method will *incorrectly* identify the sample as being greater than or equal to 0.25 percent 33, 19, and 12 percent of the time when 400, 800, and 1,200 points are counted, respectively. This is the false positive error rate. When 1,000 points are counted, the false positive error rate is eight percent, which is lower than the rate associated with either the 400, 800, or 1,200 point count. (The point-count procedure will correctly identify this sample to be less than 0.25 percent 67, 81, and 88 percent of the time when 400, 800, and 1,200 points are counted, respectively.)

Figure H-4 depicts yet another way of illustrating the false positive and false negative rates as a function of true asbestos content and the number of points counted. Some key points from the graph, and also shown in earlier charts, include:

1) False positive/negative error rates increase substantially near the decision threshold (e.g., 0.25 percent) but drop significantly as the true asbestos percentage deviates from the decision threshold.
2) Increasing the point-count in multiples of 400 reduces both the false positive and false negative error rate, but the statistical benefit is greater in limiting the false positive rate when compared to limiting the false negative rate.
3) The 1,000 point-count procedure will lead to lower false positives than the 400 and 800 (and 1,200) point count procedures**.** However, the false negative error rate for the 1000 point-count (shown as the dashed line with diamonds in Figure H-4) becomes substantially **higher** in comparison to the 400, 800, and 1,200 point-counts when the true asbestos percentage is above the decision threshold (i.e., 0.25 percent) and less than approximately 0.50 percent.

Figure H-4. False Positive and False Negative Error Probability Rates



Although increasing the number of points counted beyond 400 does increase the sensitivity of the quantitation of the asbestos content, doing so does have an effect on the false positive and false negative error rate. The false positive error rate will drop considerably for any point count above 400. On the other hand, **the false negative rate could increase substantially if point-counting is not done in multiples of 400.** Therefore, to maximize the benefit of any increase in the number of points counted, ARB staff recommends that such an increase be done in multiples of 400.

Appendix I

Example of Method 435 Sample Chain of Custody

Submitted by Client		Print Name:		Date:	
		Signature:		Time Submitted:	
Client Company:				Tel. No.	
Address:				Email:	
Job Site:					
Received by Laboratory		Print Name:		Date:	
		Signature:		Time Received:	
Sample No.	Sample Name	Analyst Initials	Sample Type and Volume (e.g., rocks, soil, aggregate, other)	Features for Targeted Analysis? (Y=Yes N=No)	Other Tests in Addition to M435
PLM Analysts		1. Print Name:		Other Tests Done by	Print Name:
		Signature:			
		2. Print Name:			Signature:
		Signature:			
Additional Information:					
Returned by Laboratory		Print Name:		Date:	
		Signature:		Time Returned:	
Received by Client		Print Name:		Date:	
		Signature:		Time Received:	

Appendix J

Recommended Training and Experience for M435 Asbestos PLM Analysts

All laboratory personnel who participate in the preparation and analysis of rock and soil samples using Test Method 435 (M435) should be familiar with their laboratory's safety practices for handling samples that may contain asbestos. These safety practices should be included in the laboratory's approved standard operating procedures (SOP) that are specific for this method.

A. Recommended Training

The identification of naturally occurring asbestos (NOA) in rocks and soils using M435 largely depends on the training and experience of the microscopists with polarized light microscopy (PLM).

PLM analysts should have successfully completed a course in optical mineralogy. Other helpful courses would include mineralogy and petrography, or equivalent. These courses should be taken at a college, university, or accredited learning institution for continuing education and training. Formal training or courses specifically on the identification of asbestos using PLM are highly recommended.

The following are recommended training subjects and practical experiences for microscopists who analyze rocks and soils for asbestos content using PLM, as described in M435. These training subjects should give the analyst a thorough understanding of how light is observed through a polarized light microscope and how these observations relate to the crystal structure and mineral characteristics. Furthermore, an understanding of the occurrences, mineral associations, and alteration of asbestos will prepare the analyst for the recognition and identification of asbestos in weathered rock or soil samples.

A1. Theories of light, its properties, and refraction.

A2. Optics and the petrographic microscope: assembly, illumination, mechanical and optical alignment, calibration, and routine maintenance. Sample preparation techniques for PLM and considerations of sample properties.

A3. Plane polarized light in minerals: polarization, birefringence, optical indicatrix, Michel-Levy interference colors, and extinction characteristics.

A4. Mineral crystal systems and optical crystallography: descriptions of mineral morphology and optical characteristics; principles and use of compensators.

A5. Systematic identification of asbestos minerals using PLM oil immersion technique: morphological properties and optical characteristics of asbestos minerals; M435 Table 3.

A6. Identification of asbestos using dispersion staining techniques (i.e., M435 Table 4).

A7. Asbestos occurrences, alterations, and mineral associations.

A8. Common asbestos interference minerals (look-alikes) and how to differentiate them from asbestos.

B. Recommended Experience

B1. Formal course, at least in optical mineralogy for asbestos, as described above. Other helpful courses would include mineralogy and petrography.

B2. Familiarization with naturally-occurring asbestos at different stages of mineral alteration in rocks and soils.

B3. Comparison of asbestos and their non-asbestiform, equivalent minerals of similar chemical composition.

B4. At least a two-month, full-time, practical training on asbestos identification in rock and soil samples under a supervising microscopist at an asbestos laboratory accredited by the National Voluntary Laboratory Accreditation (NVLAP), Environmental Laboratory Accreditation Program (ELAP), or equivalent.

A PLM analyst should demonstrate the ability to identify asbestos, according to asbestos characteristics described in M435 Table 3 and Table 4, and also differentiate asbestos from interference minerals that may be mistaken for asbestos. Part of the practical training should include the successful analysis of performance evaluation samples for asbestos and non-asbestos interference minerals. Following the training and experiences set forth in Sections A and B above, a supervising microscopist should oversee the analysis of laboratory samples by a newly-trained analyst for at least one week or until the supervisor is satisfied that he/she concurs with the analyses done by the new asbestos microscopist.