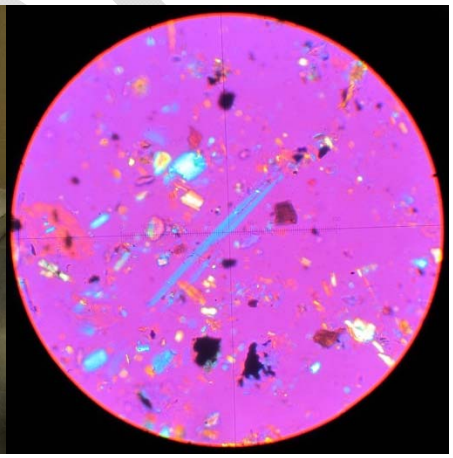
California Environmental Protection Agency

AIR RESOURCES BOARD

IMPLEMENTATION GUIDANCE DOCUMENT
Field Sampling and Laboratory Practices

Air Resources Board Test Method 435
Determination of Asbestos Content of Serpentine Aggregate



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GOOD FIELD SAMPLING AND LABORATORY PRACTICES

Air Resources Board Test Method 435
Determination of Asbestos Content of Serpentine Aggregate

EXECUTIVE SUMMARY

In 1986, asbestos was identified by the California Air Resources Board (ARB or Board) as a toxic air contaminant. In 1990, ARB adopted the first Asbestos Airborne Toxic Control Measure (ATCM) for Surfacing Applications (Surfacing ATCM) to limit the public's exposure to airborne asbestos from unpaved surfaces. At its July 2000 public hearing, ARB approved amendments to the Surfacing ATCM, further limiting the asbestos content of materials used for unpaved surfacing to less than 0.25 percent. The test method required to determine the asbestos content is ARB Test Method 435: Determination of Asbestos Content of Serpentine Aggregate (M435).

At its July 2001 Board hearing, ARB approved a second asbestos ATCM for construction, grading, quarrying, and surface mining operations. This ATCM requires operators to employ the best available dust mitigation measures during road building and maintenance activities, construction and grading operations, and quarrying and surface mining operations in areas where naturally occurring asbestos is likely to be found. This ATCM also references M435 as a laboratory test method to determine the asbestos content of bulk samples.

ARB staff research and a M435 interlaboratory study have shown that M435 sample processing and analytical procedures vary among commercial laboratories performing M435 analyses and these differences can lead to variable reported asbestos content of the same, or similar, asbestos-containing samples. In response, this guidance document was prepared to assist laboratories, consultants, local air pollution control districts, and other stakeholders in the application and performance of ARB M435. This document is intended to be used in conjunction with M435. This document provides:

a) Recommendations to help ensure that a representative field sample is obtained for a M435 analysis.
b) Recommended laboratory sample preparation procedures that will increase the representativeness of the pulverized portion of the field sample that is used for analysis by polarized light microscopy (PLM).
c) Guidance in asbestos analysis through the standardized use of PLM techniques for the optical characterization and quantification of asbestos.
d) Scientifically accepted quality control measures that can be applied to M435 to minimize field, laboratory, and analytical uncertainty.

If all parties involved in the collection and analysis of potential asbestos-containing aggregate follow the guidelines specified in this document, more accurate and

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repeatable M435 asbestos content measurements will result. This will ultimately lead to better-informed decisions regarding naturally occurring asbestos related projects.

Key recommendations are summarized below:

Sampling Practices

a) Increase the number of random (grab) samples for each test in situations of observed heterogeneity. (M435 requires a minimum of three grab samples).
b) If sampling from piles, use insertion tubes instead of round point shovels or use a front loader to obtain a smaller sample from various levels and locations of the larger pile before subsampling.
c) Choose to sample aggregates on conveyor belts closest to the final product rather than piles if at all possible.
d) Aim for a field sample volume of approximately two to three liters.

General Laboratory Processes

a) Employ chain of custody procedures and acceptance criteria for samples.
b) Prepare written laboratory standard operating procedures specific for M435.
c) Ensure equipment cleanliness during all phases of M435 activities; for some processes, specific recommended cleaning procedures are provided.

Laboratory Sample Processing

a) Use a jaw crusher and Braun Mill pulverizer to produce the rock powder.
b) Include a mixing step to increase homogeneity of the powdered sample analyzed, to increase the likelihood that the material analyzed is representative of the field sample, as well as to increase the accuracy and precision of the analytical results.
c) Perform routine particle size calibration checks to ensure that samples are not over-pulverized or incompletely pulverized.

Laboratory Sample Analysis

a) Standardize the amount of powdered sample material mounted on a slide.
b) Identify suspect fibers as asbestos using only Tables 3 and 4 of M435.
c) Use a single crosshair eyepiece for asbestos quantification and at least 200X magnification for asbestos optical characteristics verification.
d) Enhance analysis quality control to include microscopist training, routine use of asbestos proficiency evaluation and interference mineral samples, analytical replicates, instrument cross checks, method validation, and verification of some PLM non-detect M435 results using another analytical method.

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I. INTRODUCTION

I.1 Purpose

This guidance document was prepared to assist laboratories, consultants, local air pollution control districts, and other stakeholders in the application and performance of the California Air Resources Board (ARB or Board) Test Method 435--Determination of Asbestos Content of Serpentine Aggregate (M435). This document is intended to be used in conjunction with M435, which can be found in Appendix A.

ARB staff conducted an interlaboratory study (ILS) (Appendix B) that shows that sample processing and analytical procedures vary among commercial laboratories performing M435 analyses. This study indicates that these differences may lead to variable reported asbestos content of the same, or similar, asbestos-containing samples.

This document aims to clarify the stated procedures in M435 as well as provide recommendations regarding field sampling and laboratory practices. In addition, the document clarifies the different roles of using M435 in compliance with the two asbestos ATCMS for the analysis of bulk samples. The guidelines in this document, if adhered to by all parties involved in the collection and analysis of potential asbestos-containing aggregate, will yield more accurate and repeatable M435 asbestos content measurements.

I.2 Background

The California Toxic Air Contaminant Identification and Control Program, set forth in Health and Safety Code Section 39650 et seq. (H&SC §§ 39650-39675), requires ARB to identify and control toxic air contaminants (TAC). A TAC is defined as an air pollutant which may cause or contribute to an increase in mortality or in serious illness, or which may pose a present or potential hazard 1to human health (H&SC § 39655). In 1986, asbestos was identified by the Board as a TAC; the Board also determined that there is not enough scientific evidence to identify an asbestos threshold exposure level below which no significant adverse health effects are anticipated (17 CCR § 93000).

In 1990, ARB adopted the first Asbestos Airborne Toxic Control Measure (ATCM) for Surfacing Applications (Surfacing ATCM) to limit the public's exposure to asbestos from unpaved surfaces (Appendix C). At its July 2000 public hearing, ARB approved amendments to the Surfacing ATCM, further limiting the asbestos content of material used for unpaved surfacing to less than 0.25 percent. The test method required to determine the asbestos content is M435 (17 CCR § 94147).

At its July 2001 Board hearing, ARB approved a second asbestos ATCM for construction, grading, quarrying, and surface mining operations (Construction ATCM). This ATCM requires operators to employ the best available dust mitigation measures during road building and maintenance activities, construction and grading operations, and quarrying and surface mining operations in areas where NOA is likely to be found

(Appendix D). The Construction ATCM also references M435 as one among several laboratory test methods for the determination of asbestos content of bulk samples.

However, the more common circumstance, when an asbestos content determination becomes necessary under the Construction ATCM, is in relation to a geologic assessment of the presence or absence of asbestos in a certain location, a scenario which is not addressed in M435. M435 is an appropriate test for the measurement of asbestos content of individual bulk samples. It is not a substitute for a geological evaluation of the likelihood of asbestos occurrence in an area.

I.3 Safety

Asbestos is classified as a TAC and a known human carcinogen by State, federal, and international agencies. The United States Environmental Protection Agency (U.S. EPA) classifies asbestos in Group A, as a human carcinogen (CASRN 1332-21-4). Similarly, the International Agency for Research on Cancer (IARC) classifies asbestos as carcinogenic to humans (Group 1) (IARC Monographs, 1987). Asbestos dust inhalation can initiate events that could result in asbestosis, lung cancer, or other asbestos-related diseases, such as mesothelioma.

In conducting the testing described in this document, as well as any preparatory and cleanup work, parties bear the responsibility of determining and implementing all of the appropriate health and safety practices to ensure compliance with local, State, and federal health and safety regulations. All activities associated with the handling of potential NOA should proceed as if asbestos were known to be present in the rock or soil, thereby initiating the appropriate safety precautions. In performing all of the preparation and testing described in this document, all applicable safety features and procedures for the equipment involved should be employed. In addition, ARB staff recommends that field personnel and laboratories consult an outside, independent industrial hygienist and safety professional to review their respective practices and recommend additional appropriate safety procedures where needed.

II. APPLICABILITY

II.1 Asbestos ATCM for Surfacing Applications (17 CCR § 93106)

M435 was adopted in 1991 in support of the Surfacing ATCM which sought to reduce asbestos emissions from unpaved roads and other applications by limiting the sale and use of asbestos-containing serpentine rock for surfacing applications.

M435 is the referenced test method required for the determination of asbestos content of a sample of surfacing aggregate material. M435 requires representative, unbiased sampling of industrial earth products, such as bulk aggregate materials at the production plant (e.g., in piles, conveyor belts) and at existing sites where surface

covering can be assessed for asbestos content (e.g., on roads, road shoulders, driveways, parking lots, and other surfaces).

Aggregate-producing facilities operating in alluvial deposits, maintenance operations on existing roads, and construction materials of asphalt or concrete surfaces are exempted from the Surfacing ATCM. An exemption may also be sought for aggregate materials extracted from a property mapped within an ultramafic rock unit if a registered geologist has conducted a geologic evaluation and determined that serpentine or ultramafic rocks are not likely to be found on the property. A summary of this exemption appears below at Section II.3.

When the material to be tested for asbestos content consists of aggregate materials, then the use of M435 random sampling is appropriate. However, when the sampling is performed in order to assess the likelihood of geologic occurrences of asbestos, serpentine, or ultramafic rocks in an area, then a geologic evaluation of the property is necessary.

II.2 Asbestos ATCM for Construction, Grading, Quarrying, and Surface Mining Operations (17 CCR § 93105)

The Construction ATCM was adopted to reduce asbestos exposure associated with construction, grading, quarrying, and surface mining activities in areas where NOA is known or likely to be present. Specifically, these activities are subject to the Construction ATCM if they occur in mapped ultramafic rock units, or when NOA, serpentine, or ultramafic rocks are known to be present, or are discovered to be present after the start of operations in the area. M435 is referenced in the Construction ATCM as an approved test to determine the asbestos content of a bulk sample. The Construction ATCM requires work practices that will minimize dust emissions during these activities. The ATCM does not prohibit the above-mentioned activities.

A general exemption from the Construction ATCM may be sought and granted if a registered geologist conducts a geologic evaluation that determines that no serpentine or ultramafic rock is likely to be present in the area to be disturbed, a scenario which is not addressed in M435.

II.3 Exemption Via the Geologic Evaluation

This guidance document is not intended to discuss the ATCM exemption through a geologic evaluation in any great detail. It should be noted that, although M435 can be used for the analysis of bulk samples, and is referenced as a bulk analysis technique, the M435 random sampling procedure is not a substitute for a geologic evaluation of an area. The geological exemption criteria are stated in the asbestos ATCMs. Exemptions for the Surfacing ATCM and the Construction ATCM require a registered geologist to conduct a geologic evaluation of the area to be disturbed. Furthermore, the M435 random sampling methodology does not address the investigation of the presence or absence of asbestos from surface rock outcrops or subsurface rock samples which may

be needed to determine the likelihood of the presence of asbestos, serpentine, or ultramafic rocks in the area to be disturbed. Therefore, the random sample collection methodology, as written in M435, is not a substitute for targeted sampling that may be needed for the geologic evaluation of an area when seeking exemption from either asbestos ATCM. The California Geological Survey Special Publication 124 (Guidelines for Geologic Investigations of Naturally Occurring Asbestos in California) provides general procedures for geologists to use when conducting NOA site investigations. Further clarifications regarding the ATCM requirements can be given by the Emissions Evaluation Section, Transportation and Toxics Division of the ARB.

M435 is an appropriate test for the measurement of asbestos content of individual bulk samples.

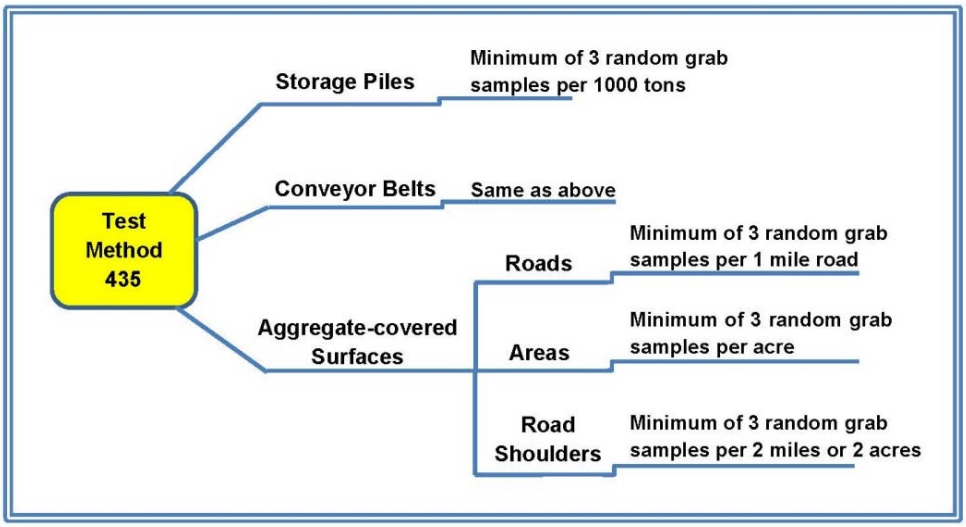
III. SAMPLING PRACTICES FOR AGGREGATE MATERIALS

III.1 Applicable Sources (M435 Section 3)

Field sampling in M435 is applicable for obtaining bulk material samples from three types of serpentine aggregate sources (Figure 1):

a) Storage piles.
b) Conveyor belts.
c) Aggregate-covered surfaces.

Figure 1. M435 Sampling Requirements for Aggregate Material



As defined in the Surfacing ATCM, the term “aggregate” means a mixture of mineral fragments, sand, gravel, cobbles, rocks, stones, or similar minerals that may or may not be crushed or screened. “Aggregate” does not include elemental metals, gemstones, petroleum products, organic materials, or mineral ore to be processed offsite of the

property from which it was extracted (17 CCR § 93106(i)(1)). All recommended sampling procedures should comply with the procedures set forth by the Mine Safety and Health Administration (MSHA), and other safety standards.

III.2 Sampling Design (M435 Section 5)

M435 sampling procedures were developed to provide a collection of unbiased samples of aggregate materials. Prior to field sampling, a sampling plan, including a description of how many samples will be collected, shall be submitted to the appropriate local air district officer for approval, if the sampling is conducted for determining compliance with a rule or regulation. This should include a conceptual site model of the area and a description of how the random collection of samples will be conducted in order to generate a composited, representative sample.

Each M435 test must consist of at least three random grab samples that are composited in a sampling container for preparation and analysis. At the discretion of the person in charge of the sampling plan, more than the required three grab samples may be collected but the method of deciding where and how to collect additional grab samples must follow the specified procedure in the approved sampling plan. One way to increase the representativeness of a grab sample is to make sure that each grab sample consists of about 20 to 30 increments, depending on the size of rock fragments in the aggregate material (Interstate Technology Regulatory Council, 2012). It is recommended that the total volume of the composited sample not exceed about three quarts (approximately three liters). This sample volume of about three quarts could present issues of sample transport and storage that may need to be discussed with the analytical laboratory prior to sample collection.

In situations of observed aggregate heterogeneity, such as notably different rock types that may indicate variable sources of aggregate material, ARB staff recommends collecting more than the minimum of three grab samples, each consisting of about 20 to 30 increments. One should take into consideration potentially variable sources of the aggregate material, as may be indicated by different lithology, rock color, etc., and total sample volume, which should not exceed about three quarts (approximately three liters). If a sample area is expected to have significant heterogeneity, the sample area should be divided into multiple units prior to sample collection and representative samples should be collected from each unit.

III.3 Sampling Equipment and Procedures (M435 Sections 4 and 5)

The different acceptable sampling equipment and procedures for the respective aggregate sources are described in this section. It is important that field sampling begins with clean sampling equipment and that the equipment be thoroughly cleaned after each sample collection, following a written protocol, to prevent cross-sample contamination.

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Storage Piles

Sampling of aggregate storage piles (Figures 2A, 2B) can be difficult because they typically have a conical shape which may be size-segregated. This is formed by the introduction of aggregate at the top of the pile, and coarser particles roll to the outside base of the pile, while leaving the finer material towards the top of the pile (sloughing). To collect a representative sample, it is important to dig into the pile to avoid the slough or size-segregated particles. One method to avoid the slough or size-segregated particles is to use a sampling tube inserted one foot (approximately 30.5 centimeters) into the pile. Another way is to use a round point shovel, and take equivolume sample increments from at least three separate locations: from the upper, middle, and lower portions of a pile. A greater number of grab samples enables one to collect material from multiple areas to better account for any variability of the aggregate material. The collected aggregate can then be transferred into a clean container of adequate size.

Figure 2A. Storage Piles of Crushed Natural Stone

The applicable diameter of the sampling tube equipment depends on the size of the aggregate particles. As described in M435 Tables 1 and 2 (Appendix A), thin-walled sampling tubes with an outside diameter between two to five inches (approximately 5.1 to 12.7 centimeters) and a length from 36 to 54 inches (approximately 91.4 to 137.2 centimeters) may be used for sampling in storage piles. The nominal diameter of the aggregate material determines the dimensions of the sampling tube. The sampling tube should have adequate strength so that it may be inserted one foot (approximately 30.5 centimeters) into the pile. Further descriptions of these tubes can be found in ASTM D 1587-83, which is incorporated in M435 by reference.

M435 also allows for the use of round point shovels. However, staff recommends the use of insertion tubes over shovels because insertion tubes do a better job of dealing with slough material. Shovels should only be used when the aggregate material is coarse or consists of mixed fine and coarse material that cannot be easily sampled with a tube.

ARB staff has also observed that aggregate sampling of stock piles in rock quarries is often done with power equipment. Using a front loader, a small sampling stockpile is made using materials taken from various levels and locations of the main stockpile (Figure 2B). After mixing the sampling stockpile with the front loader, several increments can be combined in a container as the field sample (ASTM D 75). Although different than M435 procedures, ARB staff is aware that this procedure is used in the industry to obtain representative aggregate samples for testing aggregate products. If material is taken from the upper, middle, and lower levels of the pile and then mixed, this procedure may also be effective in obtaining representative samples for M435 analysis.

Figure 2B. Front Loader Sampling Aggregate Pile

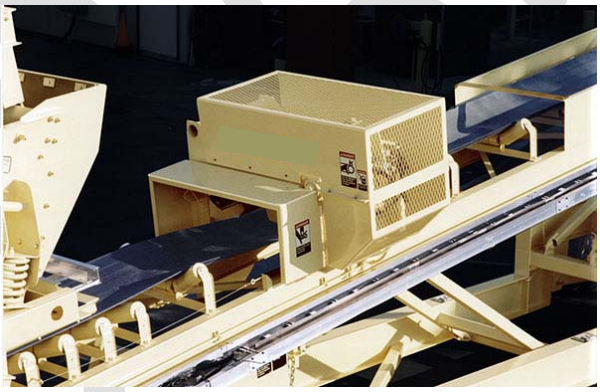
Conveyor Belts

M435 samples can also be taken from conveyor belts (Figure 3A) that are used to transport aggregate materials. To perform the sampling procedures, conveyor belts should be manually stopped, locked, and tagged out. Two steel templates, cut to the specifications given in Figure 2 of M435 (Appendix A), can be used to isolate aggregate material that will be sampled. The steel templates are placed at least six inches (approximately 15.2 centimeters) apart and, using a small shovel, brush, and dust pan, all the aggregate material between them is collected. It is important that the distance between templates be maintained for every sampling event on the conveyor belt to collect equivolume increments. Therefore, the volume of aggregate material collected would depend on the distance between the templates, the width of the conveyor belt, and the thickness of the aggregate material on the conveyor belt. An automated belt sampler, if present, may also be used (Figure 3B). Sampling is to be done at least three times in randomly chosen locations. The aggregate materials collected are composited in a container of adequate dimensions. Although potentially more disruptive to an aggregate quarry's operations than sampling from piles, conveyor belt manual sampling is less susceptible to the sloughing effects observed in aggregate piles. An automatic

belt sampler at the conveyor belt closest to the final product stream can provide representative aggregate samples most similar to the sellable product.

Figure 3A. Conveyor Belt for Aggregate Material

Figure 3B. Example of Automatic Conveyor Belt Sampler



<u>Aggregate-covered Surfaces</u>

For aggregate-covered surfaces (e.g., roads, road shoulders, parking or play areas, etc.) as shown in Figure 4, one can use manual or automatic augers, a shovel, or other suitable equipment for sampling. Sampling with an auger collects a variable volume of

Figure 4. Aggregate-covered Surfaces

aggregate materials, depending on the diameter of the sampling auger and the thickness of the compacted aggregate material bed to be sampled. The locations of sampling points are random, and the underlying soils are not included during sampling. Auger sampling is done at least three times and all the material collected is composited in one container.

Examples of the different auger types are given in M435 Section 4.3 (Appendix A) and detailed descriptions of these augers are found in ASTM D1452-80, which is incorporated in M435 by reference. The type of auger used depends on characteristics of the aggregate to be sampled, such as the nominal diameter, aggregate hardness, water content, sampling depth, etc. For example, a helical auger (Figure 5A) is good for boring holes quickly, but is difficult to use for removal of material. An orchard barrel auger (Figure 5B) works well in most soil conditions, but may bore more slowly than a helical auger through hard material. A clam shell type auger (Figure 5C) works for alternate digging and retrieving of sample materials, while the Iwan-type auger (Figure 5D) works well in stony soils. All sample increments collected are composited in a sample container.

<u>Field Sample Volume</u>

The volume of the field sample is not specifically stated in M435. However, a one-pint aliquot of the crushed field sample is required for pulverization. Therefore, by inference, one pint of aggregate material is the minimum size of a field sample.

Although laboratory personnel should not dictate sample volume, field personnel should be mindful of the dimensions of rocks they collect so that samples may be readily processed as determined by typical laboratory equipment size specifications or sample

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Figure 5. Augers: A. Helical, B. Orchard Barrel, C. Clam Shell, D. Iwan-type

A B C D

handling capacities. For instance, some laboratories use rock crushers (e.g., jaw crushers, etc.) to reduce the nominal size of aggregate to less than 3/8-inch (less than 0.95-centimeter) diameter before pulverization. These rock crushers can process, within minutes, a two-quart (approximately two-liter) sample of aggregate rock fragments, ranging from approximately 1/2 to three inches (approximately 1.3 to 7.6 centimeters) in diameter. In addition, a laboratory may be using a mixer for homogenization (as recommended in this document). These mixers are also limited in terms of sample size mixing capacity. The three-dimensional (3-D) mixer tested by ARB staff has a mixing sample capacity of four quarts (approximately four liters).

In view of the equipment size limitations discussed above, ARB staff has determined that a rock aggregate sample volume of about two to three quarts (approximately two to three liters) is appropriate. Pulverization of approximately two quarts (approximately two liters) of fine rock aggregate with less than 3/8-inch (0.95 centimeter) diameter can result in a rock powder volume of about three quarts (approximately three liters). The volume increases due to an increase in interparticle spaces. Homogenization of this rock powder, when placed in a four-quart (approximately four-liter) mixing container, will require that some volume of head space be available within the container for the powder to be thoroughly mixed.

III.4 Sample Documentation (M435 Section 6)

As written in M435, a sample log must be kept showing:

a) Unique sample number.
b) Facility name and MSHA Mine ID number if applicable.
c) Facility address or location where sample was taken.
d) Rough sketch, video, or photograph of the specific sampling location.
e) Date and time of sampling.
f) Name of person performing sampling.

ARB staff believes that, absent unusual circumstances, these sample log requirements are sufficient.

IV. SAMPLE PROCESSING PRACTICES IN THE LABORATORY

Although sample preparation processes, such as drying, crushing, and sample size reduction, are discussed in the field sampling portion of M435 (M435 Section 5, Appendix A), these activities are best performed in a laboratory setting.

IV.1 Chain of Custody Procedures

Chain of custody (CoC) documentation maintains the integrity of samples by providing records regarding their source, control, transfer, processing, and analysis. In general, the purpose of CoC procedures is to provide accountability for, and documentation of, sample integrity from the time samples are collected to sample disposal. Sample custody documentation is just one of the many important components of data defensibility. M435 does not explicitly prescribe the use of CoC procedures, but such procedures are widely recognized as producing vital documentation when using data for regulatory and/or enforcement decisions. For M435 samples, ARB staff recommends a detailed CoC record that is initiated by field sampling personnel and documents at least the following:

a) Name and signature of client submitting the samples.
b) Company name, address, telephone numbers, and email address.
c) Date and time of submission.
d) Job site where samples were collected (may be coded).
e) Sample identification (may be coded).
f) Sample type description (e.g., rock, soil, aggregate, etc.) and sample volume.
g) Name and signature of laboratory personnel accepting custody.
h) Date and time of acceptance of samples.

ARB staff recommends the use of a laboratory information management system to track the location of samples, analytical results, identification of microscopists who performed the analyses, and the location of sample archives.

In order to maintain an adequate CoC, the laboratory may also choose to render some samples inadmissible for M435 analysis for several reasons, some of which may include but are not limited to the following:

a) Sample container is breached.
b) Several samples appear to have become commingled, contaminating each other (e.g., broken bags).

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c) Insufficient volume of sample (i.e., less than the implicitly defined M435 minimum volume of one pint) or sample volume is different than what is indicated on the CoC.
d) Samples are not clearly identified and labeled.

Appendix I shows an example of what could be used as a Method 435 sample CoC. Further guidance on general CoC procedures can be found from many sources, one of which is provided below:

ASTM: http://www.astm.org/Standards/D4840.htm

IV.2 Drying

M435 requires that the sample be adequately dried before it is crushed, but does not provide details on how to accomplish this. The object of drying samples is to remove moisture that would hinder complete pulverization of the sample. Because complete pulverization is an important component in producing accurate and repeatable asbestos analytical results, appropriate and standardized laboratory drying procedures should be utilized.

ARB staff suggests the following drying steps to aid the pulverization process while reducing the potential for cross-contamination:

a) Use disposable metal pans for oven drying.
b) Label drying pans or place labeled tags in the pan for sample identification.
c) Transfer the sample into drying pan(s) under a negative air fume hood equipped with a high-efficiency particulate air (HEPA) filter, taking care that the depth of materials does not exceed 1.5 inches (approximately 3.8 centimeters) for uniform drying of samples. If a shorter drying time is needed, spread the sample to a thickness of about 0.5 inch (approximately 1.2 centimeters) and use several drying pans.
d) Remove and discard organic materials such as leaves, plant stems, roots, twigs, etc.
e) Completely cover the drying pans with clean paper towels fastened to the pan with clips.
f) Dry the samples at 230 degrees Fahrenheit (ºF) (110 degrees Celsius, ºC) in a constant-temperature oven with plus-or-minus 5 ºC accuracy for about 15 hours (overnight). Staff recommends keeping the oven-drying temperatures below 392 ºF (200 ºC) to avoid possible mineral alterations when a quicker oven-drying time is needed. The dried samples should have a gravimetric water content of about two to four percent, depending on the sample particle sizes (i.e., coarser samples retain less water and clayey samples retain more water).
g) Record the drying temperature and drying time on the sample analytical bench sheets.
h) Cool samples under a negative air fume hood that uses a HEPA filter.

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i) Place disposable items used for drying in plastic bags that can be sealed and marked for proper waste disposal.
j) Ultrasonically clean non-disposable items used (e.g., clips, forceps, etc.).

IV.3 Crushing

Per M435, the composited sample must be crushed to produce a material with a nominal size of less than 3/8 inch (approximately 0.95 centimeter). Although not explicitly stated, this procedure is required so that the crushed material can be introduced into the sample intake of the Braun Mill pulverizer (plate grinder).

ARB staff is aware of four methods that commercial laboratories use to ensure that the sample product is crushed to a size that is compatible with their pulverizing equipment:

a) Using a mechanical jaw crusher to reduce sample to a nominal size of less than 3/8 inch (approximately 0.95 centimeter).
b) Using a hammer to manually crush sample (usually contained in one or more plastic bags).
c) Requiring the submitted field sample to have specified size restrictions (e.g., small rock fragments) suitable only for the respective laboratory's pulverizing equipment.
d) Removing and discarding portions of the field sample submitted that are not compatible with or too large for the laboratory's pulverizing equipment.

ARB staff recommends the use of jaw crushers (a) because they are reliable at producing less than 3/8-inch (approximately 0.95-centimeter) crushed rock material with relative ease. Rock samples with cross sections up to about 2.5 to three inches (approximately five to seven centimeters) are easily and uniformly crushed within minutes.

The jaw crusher should be operated in strict compliance with lockout/tagout and other safety procedures, as appropriate. (See reference section for OSHA Lockout/Tagout Fact Sheet [2002]). The crusher should be adequately cleaned prior to use and operated under a HEPA filter enclosure with a minimum flow rate of 100 feet per minute (approximately 30.5 meters per minute). Recommended procedures on how to operate and clean the jaw crusher can be found in Appendix E.

ARB staff discourages the use of hammers (b) to crush rocks because of the increased likelihood of losing sample volume and the risk of spreading potentially asbestos-containing fragments in the processing room, even with the use of bags to enclose the sample. ARB staff believes that items (c) and (d) go against the original intent of M435 and are not consistent with good field and laboratory practices, especially if these practices are not documented in the appropriate standard operating procedure (SOP).

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IV.4 Sequence of Post-crushing Sample Processing Procedures

There are three general sample processing procedures that may be performed after sample crushing. These include:

a) Sample size reduction—procedure to obtain a smaller volume of test material while attempting to keep the degree of representativeness of the original sample intact.
b) Homogenization—blending of diverse rock and soil particles into a uniform mixture so that a representative sample may be obtained. This procedure is not included in M435, but is a recommended processing activity that will increase the accuracy and repeatability of the analytical results.
c) Pulverization—sample particle diminution to ensure that the resulting powder can be examined under the microscope, using PLM.

The sequence in which these post-crushing procedures are done to prepare the sample for analysis greatly affects the representativeness of the material that will be analyzed by the microscopist. As written in M435, the sequence of post-crushing sample preparation procedures is as follows:

a) Reduce volume of crushed sample to a one pint aliquot (ASTM Method C-702 80).
b) Further crush (pulverize) the one pint aliquot using a Braun Mill or equivalent to produce a material of which the majority is less than 200 Tyler mesh (less than 75 micrometers).

ASTM C-702-80, "Standard Practice for Reducing Field Samples of Aggregate to Testing Size," is included in M435 as a reference method to reduce a large field sample to a convenient size for conducting the test. This method is performed in a manner so that the smaller portion, which will be further pulverized and then analyzed, is likely to be representative of the field sample. For dry aggregates, the ASTM-preferred method for size reduction is the mechanical splitter (riffle splitter), which divides the sample into two halves.

ASTM C-702-80 further states that when the test is for certain contaminants that occur as a few discrete fragments in only small percentages, as is usually the case with NOA in aggregate samples, the entire field sample should be tested. ASTM C-702-80 states that caution should be used in interpreting the results from analysis of a reduced size test sample. Because of this, ARB staff encourages laboratories, if at all possible, to avoid sample size reduction immediately after the crushing procedure. This guidance document will discuss sample size reduction in greater detail in Section IV.7.

Because laboratory processing equipment (and the associated specifications) vary, there is no "one size fits all" post-crushing procedure that staff can recommend. Although one sequence may be deemed more advantageous than another, the presence of certain laboratory processing equipment largely determines the order in

which processing steps may be done.

That said, there may be value in considering the addition of a homogenization step that will greatly increase sample representativeness and will lead to more accurate and repeatable analytical results. Homogenization is discussed in more detail in Section IV.6.

The following table (Table 1) shows some recommended post-crushing processing sequences. As a baseline, the M435 post-crushing sequence of sample preparation is shown in the bottom row of Table 1. The potential changes to this post-crushing sequence are shown in the rows above. ARB staff's discussions on homogenization enhancements are based on available laboratory equipment.

Going from top to bottom, the most-recommended order sequence of post-crushing activities (i.e., pulverization, homogenization, sample size reduction) depends on whether a laboratory has a Braun Mill and a large-capacity mixer. The Braun Mill (also known as plate grinder) can pulverize the entire crushed sample in a reasonable amount of time. Using this equipment, pulverization of a two-quart (approximately two-liter) crushed field sample should take less than 15 minutes. This processing sequence also depends on whether the laboratory has a large-capacity (four-quart or approximately four-liter) sample homogenizer. The potential post-crushing sequences are shown in Table 1 and discussed below.

Available equipment: Braun Mill and mixer with four-quart (approximately four-liter) capacity
Sequence 1: crush--pulverize--homogenize obtain one pint (approximately 0.5 liter) for test

This is the recommended sequence if the laboratory can quickly pulverize the crushed sample and has a large-capacity mixer. The entire crushed sample is pulverized, and then homogenized. After homogenization, a one-pint (approximately 0.5-liter) test sample can be obtained for M435 analysis even without passing the powder through a riffle splitter. The powdered sample is homogenized at this point and the use of a riffle splitter to obtain a smaller-volume test sample is no longer needed.

Available equipment: Braun Mill (no large-capacity mixer)
Sequence 2: crush--pulverize--manually homogenize--obtain 1 pint for test

In this sequence, the presence of a Braun Mill that can pulverize the entire crushed sample allows the inclusion of all materials in the pulverization and manual mixing (homogenization) may be done by agitating the closed container or churning the powder with a disposable spatula. After this, a one-pint (approximately 0.5-liter) test sample can be obtained for M435 analysis.

Available equipment: Shatterbox (SB), Ball Mill (BM), or Freezer Mill (FM) and large-capacity mixer

Sequence 3: crush--homogenize--manually reduce sample size--pulverize

The crushed sample is first homogenized. One pint is manually obtained from the mixed crushed sample and then pulverized for M435 analysis. The representativeness of the sample portion that is pulverized depends on how well the crushed material was homogenized prior to sample size reduction.

Table 1. Recommended Post-crushing Sample Processing Sequences

Available Post-crushing Equipment	Step 1	Step 2	Step 3
Braun Mill + Mixer (Sequence 1)	Pulverize entire crushed sample.	Use mixer to homogenize entire powdered sample.	Manually take 1 pint for analysis.
Braun Mill (no Mixer) (Sequence 2)	Pulverize entire crushed sample.	Manually homogenize powdered sample.	Manually take 1 pint for analysis.
Shatterbox (SB), Ball Mill (BM), or Freezer Mill (FM) + Mixer (Sequence 3)	Use mixer to homogenize entire crushed sample.	Manually take 1 pint crushed sample for pulverization.	Pulverize 1 pint crushed sample for analysis.
SB, BM, or FM (no Mixer) (Sequence 4)	Riffle split entire crushed sample and take 1 pint aliquot.	Pulverize 1 pint crushed sample.	Manually homogenize powdered sample for analysis.
M435: Braun Mill or Equivalent (no Mixer)	Riffle split entire crushed sample and take 1 pint aliquot.	Pulverize 1 pint crushed sample for analysis.	

Available equipment: SB, BM, or FM (no large-capacity mixer)
Sequence 4: crush--reduce sample size--pulverize--homogenize or mix

If the laboratory has a small-capacity pulverizer and has no large-capacity mixer, then the entire crushed sample should be repeatedly poured through a mechanical splitter (i.e., riffle splitter), and the sample size is reduced to a one-pint (approximately 0.5-liter)

aliquot. This pint of crushed material is pulverized. The powdered sample is then mixed using a smaller-capacity homogenizer, if available, or manually mixed, and then analyzed.

Of the four sequences, all provide some enhancements to what is stated in M435. Under normal conditions, Sequence 1 reflects staff's most recommended post-crushing sequence in obtaining a representative subsample for analysis.

IV.5 Pulverization

M435 requires that the majority of the particles in the pulverized sample be finer than 200 Tyler mesh (less than 75 micrometers in diameter). The powder size stipulation is important because particles that are greater than 75 micrometers in diameter can be difficult to analyze by PLM due to particle thickness. In addition to meeting this M435-specific particle size distribution (PSD) criterion staff also recommends that all laboratories strive to limit the amount of material less than 10 micrometers in diameter (an indicator of over-pulverization). They should also limit the amount of material greater than two millimeters in diameter (an indicator of incomplete pulverization).

The recommended PSD would be one where:

a) At least 98 percent of the pulverized material passes through the 250-micrometer mesh sieve.
b) The 75- to 250-micrometer fraction is between 40 to 50 percent of the total mass of the sample processed.
c) The less than 75-micrometer fraction is between 50 to 60 percent of the initial sample mass.

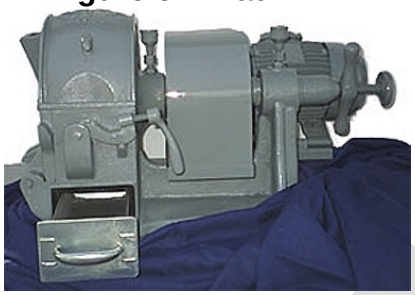
The particle size distribution recommended above is intended to reduce the risk of overgrinding the M435 sample so that asbestos, if present, can be identified using PLM.

<u>Equipment</u>

M435 states that the crushed sample shall be pulverized using the Braun Mill (Figure 6), or an equivalent pulverizer. The method leaves it up to laboratory personnel to determine what is equivalent to the Braun Mill. Pulverization equipment should be operated in strict compliance with lockout/tagout and other safety procedures, as appropriate.

The only explicitly stated performance criterion specified in M435 pertaining to pulverizing equipment is that the majority of the powdered material produced must pass through a 200 Tyler mesh (i.e., less than 75 micrometers in diameter). The ARB-led ILS (Appendix B) showed that even though all the equipment reviewed satisfied this one performance objective, the laboratory equipment and procedures employed affected the particle sizes of the sample powder produced, and in one case, the amount of asbestos content detected and reported. Therefore, staff recommends additional performance objectives as described in this section.

Figure 6. Braun Mill*



*Also known as a disc pulverizer or plate grinder

The M435 ILS was performed to assess the variable sample processing and analytical procedures used by laboratories and whether these differences affect the reported asbestos content. During the ILS, staff observed that in addition to the Braun Mill, other pulverization equipment used by laboratories include the vibrating pulverizer (shatter box), freezer mill, and ball mill. See Figure 7.

Figure 7. A. Vibrating Pulverizer (Shatter Box); B. Freezer Mill; C. Ball Mill

The ILS showed that there were visible differences in the pulverized rock powder processed using the above four pulverizing equipment, as shown in Figure 8. A subsequent quantitative PSD analysis was performed and illustrated in detail in Appendix B. This guidance document will not identify which powder was made by which pulverizer, except that produced by the plate grinder. This is because the laboratories had participated in the ILS under conditions of anonymity and it was well known at that time which laboratory used certain pulverizing equipment. However, the following results can be provided:

a) All four pulverizers met the M435 criterion of producing a powder where the majority of particles are less than 75 micrometers in diameter.

b) Powders 3 and 4 (Figure 8) showed remaining chunks of rock material (incomplete pulverization). Rock chunks cannot be mounted on a slide for subsequent analysis by PLM.
c) Nearly 50 percent of the particles from powder 2 were less than 10 micrometers (defined here as over-pulverization); these particles were very difficult to analyze at 100X magnification, as stipulated in M435. They are near the resolving limit of the light microscope as used in M435 and are difficult to visualize under PLM.
d) M435 analysis of powder 2 samples during the study resulted in statistically significant less asbestos reported than powders produced from other equipment.
e) The pulverized powder 1 was prepared using the Braun Mill, met the M435 criterion, did not leave leftover chunks, and was not over-pulverized.

More generally, the Braun Mill also has the ability to pulverize two to three quarts (approximately two to three liters) of dry, crushed rock material in 15 minutes or less. Furthermore, the Braun Mill can be calibrated to consistently avoid incomplete and over-pulverization. These results and capabilities support the use of the Braun Mill for pulverization.

Figure 8. M435 Interlaboratory Study Sample Powders

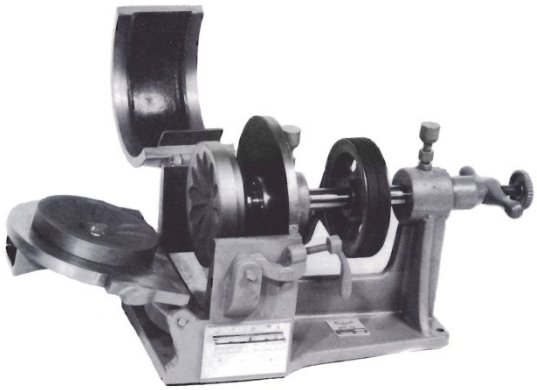
Powder 1 Powder 2 Powder 3 Powder 4

M435 allows equivalent pulverizers to be used which could include the equipment shown in Figure 7 and potentially other types of pulverizers. ARB staff recommends that laboratories show equivalency of these other pulverizing equipment to the Braun Mill, in terms of particle size distribution and length of time and efficiency of pulverization, and be able to provide appropriate documentation upon request. Pulverization protocols should be developed that result in acceptable PSD (per M435) of the powder produced and equivalent size characteristics to a powder processed using the Braun Mill. This may be done by calibrating the duration of pulverization when using these other equipment. Such duration would vary when pulverizing soft rocks or hard rocks.

Pulverization Using the Braun Mill

The reduction of rocks and soils to a fine powder using the Braun Mill is done by adjusting the distance between the grinding plates. Figure 9 shows the grinding plates separated on the left, over which a metal hood is mounted and locked when the equipment is in use.

Figure 9. Inside the Braun Mill



The procedure for pulverization is not explicitly discussed in M435, but the recommended procedure is as follows:

a) Set the plates to barely touching each other, and slowly back off to increase their distance of separation to 0.10 millimeter. Using a metric calibrated metal feeler gauge, measure this distance between the two plates at several locations to make sure that the plates are parallel and that the distance of separation is uniform throughout. The suggested plate separation of 0.10 millimeter may be adjusted, depending on how parallel the plates are mounted and the results of the PSD determination. It is important to calibrate the plate distance used for each plate grinder as this plate distance will affect the PSD of the powder produced.
b) Gradually feed the 3/8-inch (about 0.95-centimeter) crushed material through the sample intake.
c) After pulverization, collect the powdered material in the pan. Make sure to gradually cover the pan with a plastic sleeve as the pan is extracted from the plate grinder to prevent airborne dust.
d) After pulverization, clean the plate grinder by purging (grinding) non-asbestos material, brushing, vacuuming, and wiping down the plates and the entire equipment. Appendix F provides a recommended cleaning protocol for the plate grinder. This cleaning protocol was developed after testing by ARB staff using high-concentration asbestos samples.

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IV.6 Homogenization

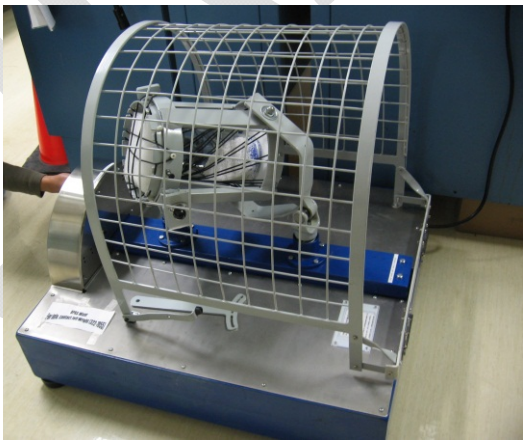
A procedure for homogenization is not addressed in M435. However, ARB staff believes that this is one of the most significant sample processing enhancements a laboratory can make.

ARB staff has observed that pulverization with a Braun Mill results in a heterogeneous powder, where the first materials that enter the sample intake are the first to go out through the plates and exit into the collecting pan. While pulverizers that use impaction for particle size diminution (i.e., shatter box, freezer mill, ball mill, etc.) may do a better job of mixing the powder during pulverization, these equipment are not purposely used to homogenize the powdered sample.

The addition of a mixing procedure in M435 sample processing would greatly increase homogeneity of the pulverized material before an aliquot of powder is taken for analysis. This procedure would increase the likelihood that the aliquot is representative of the field sample as well as increase the accuracy and repeatability of the analytical results.

ARB staff recommends the use of a three-dimensional (3-D) mixer (e.g., 88 Mixer System Schatz Model 4 (1A)) which mixes the sample in three dimensions using Schatz inversion-kinematic movement. (See Figure 10.) Staff's literature search and laboratory testing of the Schatz 3-D mixer showed that it produces a highly homogenized powder in a very short amount of time (approximately 5 to10 minutes). In addition, multiple designs are available to handle increasing sample sizes, easily large enough to handle M435 samples. The removable sample container makes cleaning relatively easy.

Figure 10. Three-dimensional Mixer - 88 Mixer System Schatz Model 4 (1A)



For purposes of M435 processing, it is recommended that the pulverized sample be homogenized with the 3-D mixer at 40 revolutions per minute for 10 minutes to ensure thorough mixing. Other mixing equipment (e.g., V-blender, rotary mixer, etc.) may be suitable for homogenization, but the optimal mixing procedures for each of these

equipment would need to be determined by the laboratory. Results of an ARB study showing the advantages of homogenizing the sample are shown in Appendix G.

IV.7 Sample Size Reduction

As specified in M435, sample size reduction is done to reduce the amount of the crushed sample prior to pulverization. However, as indicated in Section IV.4, there may be instances when this step may not be needed, based on available laboratory processing equipment. When a laboratory can easily pulverize a two- to three-quart volume (approximately two to three liters) of crushed rock sample then there is no need to reduce the sample volume immediately after crushing. Following the homogenization of the powder product, this allows for a more representative powder to be analyzed at the microscope. For those laboratories that need to follow the sample size reduction step, further clarification and guidance is provided below.

For guidance on reducing sample size, ASTM Method C-702-80 is referenced in M435 and the applicable procedures (Methods A and B) for reducing sample size are discussed at length. ARB staff recommends Method A—Mechanical Splitter (Figure 11) because it is deemed more accurate (Schumacher et al., 1990).

A sample splitter is required to have an even number of equal width chutes, but not less than eight chutes for coarse aggregate, or 12 chutes for fine aggregate (ASTM Method C-702-80). The minimum width of the individual chutes should be approximately 50 percent larger than the largest particles in the sample. Two receptacles on either side of the splitter will hold the two halves of the sample after splitting. The hopper, through which the sample is introduced at a controlled rate to the splitter, should have a width equal to, or slightly less than, the total width of all the chutes. The rate at which the sample is introduced should be controlled to ensure a free flow of material through the chutes into the receptacles. Material from one receptacle is taken and the splitting procedure is repeated as necessary until a one-pint (approximately 0.5-liter) aliquot is obtained for M435 analysis.

Figure 11. Mechanical Splitter

The procedure of cone and quartering (Method B) is not preferred because it results in greater loss of fine particles than Method A (Schumacher et al., 1990), but still may be used for coarse aggregates or mixtures of coarse and fine aggregates. The material is placed on a hard surface or canvas and shoveled into a conical pile. The pile is flattened to a uniform thickness and diameter and divided into four quarters using a shovel. Two opposing quarters are taken with a scoop or shovel, mixed, and the procedure is repeated until the desired volume is obtained. ARB staff refers to the ASTM Method C-702-80 for details of this method.

V. LABORATORY SAMPLE ANALYSIS PROCEDURES

V.1 Principles (M435 Section 7)

Asbestos identification by M435 depends on the morphology and optical characteristics of the minerals analyzed. A low-magnification stereoscopic examination at 10X to 40X magnification gives the microscopist an overall view of the homogeneity of the sample and the morphology of particles. However, positive identification of asbestos can only be done with PLM, even when fibers may be observed under the stereoscopic microscope. In addition to the morphology requirements, the optical characteristics of each asbestos mineral (M435 Table 3) have to be determined and verified using PLM to complete asbestos identification.

Details of the equipment required for a stereoscopic microscope and a petrographic microscope are given in M435 Section 7.5 (Appendix A). One suggested addition to the list of equipment would be a 20X PLM microscope objective so that, together with a 10X eyepiece, the minerals may be observed at 200X magnification when assessing the optical characteristics and particle morphology. Use of a 40X objective together with the 10X eyepiece increases magnification to 400X, which would be even better for mineral identification.

The reference asbestos minerals listed in M435 may no longer be available from the listed sources. ARB staff suggests that the laboratories obtain standard reference materials from the National Institute of Standards and Technology, if available, to help microscopists review the characteristics of the six regulated asbestos.

V.2 Polarized Light Microscopy Limits of Resolution

The limit of resolution for PLM is the smallest distance between two points on a specimen that can be distinguished as separate entities. PLM resolution is determined by the microscope optics (i.e., magnification, numerical aperture) and the wavelength of light used to image the specimen (Nesse, 2004).

M435 requires a two-step approach that includes both the visualization of fibers and the verification of optical characteristics.

M435 point-counting begins with the visualization of the sample, which should be done at a magnification of 100X (M435 Appendix A) so that a larger area of the PLM slide can be considered for the M435 analysis. Therefore, although experienced microscopists have informed ARB that at 400X magnification PLM can resolve very fine particles and fibers that are at least 2 micrometers in length and at least 0.15 micrometers in thickness, the sizes of particles and fibers investigated during a M435 point-count analysis need to be considerably larger because the point-counting is done at 100X magnification.

Following the visualization of fibers, M435 asbestos identification requires the verification of asbestos morphology and optical characteristics, as described in M435 Tables 3 and 4. This can be done using PLM analysis at higher magnifications, often at 400X magnification. If asbestos fibers below the limits of PLM resolution are present in the sample, they cannot be detected and identified with the use of PLM.

Staff has observed other analytical techniques used by laboratories to identify/quantify these smaller asbestos fibers. These include X-ray diffractometry (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

It should be noted that other analyses, such as TEM, are not within the scope of M435 and should not be considered as part of a M435 PLM analysis. Care should be taken by the laboratory to make this clear to the client and explain why the additional analysis is not provided, unless requested by the client as an additional analytical test. However, an alternative analytical method may be used as long as it produces results substantially equivalent to the results produced by the point counting method and is approved by the Executive Officer of the Air Resources Board.

XRD is an analytical technique that can identify minerals through the constructive interference of monochromatic X-rays that are diffracted by a crystalline sample. However, XRD is a bulk analytical method that in its standard form is typically used when the mineral of interest has a concentration in excess of 5 percent by weight of the total sample mass. In contrast, M435 has a sensitivity of 0.25 percent by point-count. Therefore, XRD, as it is commonly used for bulk analysis, would not be a sensitive technique to verify non-detect M435 PLM results.

While SEM may be used to characterize the morphology and elemental composition of particles, it does not provide information on crystallographic characteristics that should be used to identify the asbestos mineral, as described in Table 3 of M435 (Appendix A). Therefore, SEM alone is not sufficient for asbestos verification.

ARB staff is aware that some regulatory agencies complement PLM analysis with TEM. Samples are first analyzed using PLM because a much larger (and likely more representative) mass of sample powder is analyzed using PLM than when using TEM. The mass of a M435 sample analyzed by PLM is approximately one million times greater than the mass of a TEM sample, but TEM has a resolving power of 500 to 20,000X magnification (compared to 50 to 1000X magnification by PLM). This higher

resolving power enables TEM microscopists to distinguish and identify finer particles and fibers not seen with the use of PLM. For example, the California Department of Toxic Substances Control has used PLM, followed by TEM, as part of a tiered analytical approach to verify the absence of asbestos fibers determined by M435 PLM analyses in its Schools Program.

V.3 Procedures for Quantification of Asbestos Content (M435 Section 8)

Testing Volume

Per M435, the material to be analyzed shall be the one pint aliquot of pulverized material for the assessment of asbestos content (M435 Section 8.1, Appendix A). M435 does not state a given volume or mass of powdered material that should be mounted separately on each glass slide. After repeatedly observing microscopists perform this procedure, ARB staff recommends that approximately five milligrams of powdered sample material be used for each PLM slide (total of 40 milligrams for eight slides). This would standardize the amount of powder analyzed per sample, taking care to use enough material so that the particle loading is approximately 30 percent in the field of view (FOV), thereby minimizing particle overlap. Forceps or scalpels should be used to take the powder from different locations throughout the pint aliquot. Also, a coring device (i.e., disposable hollow tube) may be used to obtain material from the interior of a powdered sample. A detailed discussion on representative laboratory subsampling of particulate laboratory samples can be found in the document EPA/600/R-03/02 (2003), Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples.

Fiber Identification

Fiber identification by M435 requires that the asbestos fibers have a length-to-width aspect ratio of at least three-to-one (3:1) and positive identification of morphological and optical characteristics listed in M435 Table 3 (i.e., color, pleochroism, refractive indices, birefringence, extinction characteristics, and sign of elongation).

The asbestos criterion of a minimum length-to-width aspect ratio of 3:1 is only the beginning in a series of tests on the morphological and optical characteristics that need to be met before a fiber is identified as “asbestos.” Through a series of verifications on the optical characteristics enumerated above, one is able to identify asbestos. However, despite these asbestos identification tables, the wide range of asbestos fibers counted from asbestos-spiked samples from the ARB ILS suggests that laboratories use different identification criteria for asbestos. It should be stressed that only the asbestos characteristics, as described in M435 Table 3 and Table 4, should be used for determination of what is asbestos.

Attempts to disqualify suspect asbestos fibers using criteria outside of what is described in M435 Table 3 for morphology or optical characteristics, as may be expected from asbestos-containing materials (ACMs), are discouraged. NOA may not always exhibit

the unweathered characteristics of asbestos more often found in ACMs due to the added matrix materials. For purposes of asbestos identification using M435 analysis, ARB staff recommends that fibers which meet the morphological and optical characteristics of one of the six asbestos types, as defined in M435 Table 3, be reported as asbestos; otherwise, the reported analysis by the laboratory is not considered a M435 analysis.

Asbestos Quantification

The laboratory is given the option to use a 100- or 25-point Chalkley array in the microscope eyepiece or a standard crosshair eyepiece. Among several asbestos laboratories visited by ARB staff, none were observed to use a 100-point Chalkley array.

As described in M435 Section 8.3, a total of 400 particles are counted over at least eight slide preparations containing representative sample powder mounted in the appropriate refractive liquid. Fifty particles are counted in each of the eight slides and analyzed at 100X magnification. For example, when using a standard single crosshair reticle (Figure 12, left), the reticle is randomly moved across the slide. When the crosshair lands on a particle, its morphological and optical characteristics are analyzed to determine whether that particle is a fiber that is asbestos. By moving the crosshair to 50 different locations, ideally using a mechanical stage, a large portion of the slide is examined and individual particles are tested during the quantification procedure.

In contrast to this, the 25-point Chalkley array (Figure 12, right) is superimposed on a portion of the slide. All particles that land under a point are part of the point-count. However, to do a proper M435 analysis, each fiber suspected to be asbestos needs to be examined under the crosshair and the optical properties verified using M435 Table 3. In the process of moving the 25-point reticle and examining a suspect fiber under the crosshair, a microscopist can lose track of other particles, originally under a point, in the FOV. Some microscopists work around this problem by assuming that other suspect fibers they may have observed in the FOV under a point are the same mineral as the suspect fiber that they examined and verified. Other microscopists perform an initial scan of the PLM slide to determine if suspect fibers are asbestos. Then they proceed to point-counting and assume that other suspect fibers they may notice during the point-count are the same minerals as the ones they had initially verified. In both cases, each suspect fiber may not go through the formal optical characteristics verification in the course of point-counting with the 25-point reticle.

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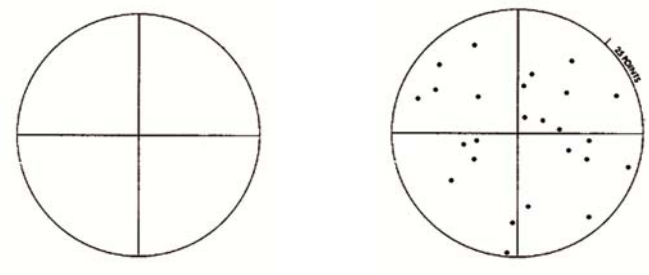
Figure 12. Single Crosshair (left) and 25-point Chalkley Array Reticles (right)



Another detriment of using a 25-point reticle is that a smaller portion of the slide is analyzed under the microscope. For instance, if all of the 25 points land on a particle, it is possible that the 25-point reticle may be used to count particles only in two FOVs of the slide. In this case, the majority of the sample powder mounted on the PLM slide will not be examined.

Staff recommends the use of a standard crosshair reticle for point-counting. This technique has two distinct advantages over the 25-point reticle:

a) A larger area of the slide is viewed during the 50 particle point-count (per slide).
b) It allows the microscopist to verify the optical characteristics of each suspect fiber that falls under the crosshair without losing track of the other particles already counted.

Staff also recommends that although the quantitation is done at 100X magnification, the microscopist should have a 20X PLM objective, allowing for 200X magnification when verifying the optical characteristics of each fiber.

Per M435, it is required that even if one asbestos mineral is confirmed, the microscopist should continue the analysis and verify the presence of other asbestos minerals (M435 Section 8.1, Appendix A) using the other appropriate refractive index liquids.

The calculations for percent asbestos using point-counting are based on an assessment of 50 particles in each of eight PLM slides for a total of 400 particles per M435 sample. Some laboratories attempt to increase the sensitivity of the test by counting more particles (e.g., 1,000 particles). ARB staff supports assessing more than 400 points only if the increase in points is done in multiples of 400 (e.g., 800, 1,200, 1,600, etc.). Otherwise, the additional point-counting may have an unintended negative effect by increasing the number of Type II (false negative) errors. An explanation of this is provided in Appendix H.

V.4 Procedures for Exceptions (M435 Section 8.3)

M435 describes two situations where an exception for the required point-count analysis may be granted.

Exception I is possible when a sample is suspected to contain no asbestos. Three PLM slides are prepared and 10 FOVs per slide are observed under PLM. Optical characteristics listed in M435 Section 8.2 must be determined to positively identify asbestos. When no asbestos fibers are observed from 30 FOVs, it can be reported that no asbestos was found using the visual technique of analysis.

Exception II is possible when a sample is suspected to contain asbestos in excess of 10 percent. Three PLM slides are prepared and if the asbestos content is estimated to exceed 10 percent by visual technique, then the particle point-count will not be necessary. It can be reported that the asbestos content exceeds 10 percent using the visual technique of analysis.

Using the visual technique, microscopists familiarize themselves with charts that show known areal percentages of asbestos fibers observed within a matrix of other particles. By repeatedly comparing what is observed under the microscope with charts of known percentages, a microscopist may be able to visually estimate the percentage of asbestos in a sample.

However, if one or more asbestos fiber(s) are identified from 30 FOVs in the three PLM slides prepared or the asbestos content is estimated by visual technique to be less than 10 percent, then the analyst is required to continue with the point-counting procedure using an additional five slides or more, for a total of at least eight PLM slides.

VI. QUALITY CONTROL (QC)

Through a method review and consultations with geologists and asbestos laboratories, staff has identified additional field sampling and laboratory analysis QC practices and principles applicable to M435. Beginning with field sampling, laboratory processing, and analysis, the QC measures summarized in this section build upon related recommendations that may be discussed elsewhere in this document. While many of these quality control practices are not addressed in M435, their use would increase the likelihood of obtaining more accurate and repeatable M435 results.

VI.1 Sampling QC

Maintenance of equipment cleanliness during sampling is of primary importance, particularly when using field equipment such as sampling tubes, augers, and shovels. A protocol for equipment cleaning and storage procedures in between collection of samples should be developed and followed.

Every effort should be taken to maintain the integrity of field samples. It is recommended that field samples be double-bagged using reclosable sample bags that are at least 4/1,000 inch (4 mil) thick (101.6 micrometers) to avoid sample spillage. In case the first sample container is breached, the sample will still be held intact by the second sample container.

Sample identity can be protected by placing a sample identification tag inside the sample container and, at the same time, using a permanent marker to write the sample name outside the sample container.

It is good practice for sampling personnel to maintain an ongoing list of M435 samples while in the field. Details on the field sample list should include at least the information required for the sample log, as enumerated in Section III.4 of this document.

Records of activities for planning and conducting field collection of M435 samples should be complete and safely stored. These include, but are not limited to, the approved sampling plan, sampling methodology, sample log, and the field sampling report. A CoC, beginning with sample collection, should be made and maintained when samples are submitted to the laboratory for analysis.

VI.2 Processing QC

Although sample processing QC is not addressed in M435, the following are recommended QC practices for the laboratory processing of field samples.

Chain of Custody

As detailed in Section IV.1, a sample chain of custody should be initiated by the field sampling personnel and continued upon receipt of the samples by the laboratory. Criteria should be identified to determine if there are any inadmissible M435 samples. A laboratory information management system can efficiently track not only the physical location of a sample in the laboratory, but also information on who performed which test on each of the samples.

Detailed M435 Standard Operating Procedure (SOP)

Each laboratory that performs M435 analysis should have a written SOP specific for M435. This includes all equipment and procedures that the laboratory uses for M435 sample processing and analysis. This SOP should also be used for laboratory training of technicians for M435 sample processing and microscopists for M435 sample analysis. This M435 SOP should be readily available to each technician or microscopist during M435 sample preparation and analysis. The SOP should also be made available to local air districts or ARB staff upon request.

Equipment Cleaning Protocols

Rigorous equipment cleaning protocols should be written for the equipment used in processing the M435 samples. Examples of cleaning protocols that were tested by ARB staff for the crusher and the plate grinder are given in Appendix E and Appendix F, respectively. Included in the protocols is the use of equipment cleaning purges (blanks)

to make sure that no asbestos is detected from asbestos-free samples that are processed through the clean equipment.

Blanks

Processing blanks, consisting of materials tested and found to have no asbestos, should be processed alongside regular field samples. These processing blanks, when analyzed blind, should yield negative results for asbestos. There should be a minimum of one processing blank for each job site to verify that no sample cross-contamination has occurred during processing. If a processing blank yields a positive result for asbestos, then sample processing should be halted. Processing procedures should be reviewed, amended, and re-tested to ensure that there is no cross-contamination of samples. One suggestion would be to ensure that at least one processing blank is produced and verified to not contain asbestos before processing samples from a new job site.

Calibrations

Calibration of processing equipment should be done to make sure that the sample powder produced meets the requirements for M435 analysis by PLM. When using a plate grinder, the gap between the two plates can be adjusted to approximately 0.1-millimeter distance. When using other pulverization equipment (e.g., freezer mill, shatter box, ball mill), the duration of the impaction can be adjusted to result in a finer or coarser grind.

Sample Processing Calibration Check

A particle size calibration check can be done to make sure that the powder produced after grinding is acceptable for M435 analysis by PLM (i.e., majority of particles are less than 75 micrometers in diameter). To do this sieve test, approximately 30 milliliters of powdered sample can be weighed and sieved through a covered stack of a 250-micrometer mesh sieve, over a 75-micrometer mesh sieve, over a bottom pan (see Figure 13).

Figure 13. Stack of Sieves for Particle Size Check

This PSD would indicate that the powder can be analyzed by PLM (10-75 micrometer particle diameter) and that the sample is not over-pulverized beyond the resolving power of a light microscope. Equipment or duration time should be adjusted accordingly if the sieve test is not met.

VI.3 Analysis QC

Section 8.3 of M435 (Appendix A) states the need for analyst cross-checks, where a second microscopist analyzes one in 10 samples of those analyzed by the first microscopist. This is done to verify and confirm the quantitation result. This is an important, common QC check. However, staff recommends the implementation of other analytical QC measures. These additions are briefly discussed below.

Microscope Alignment / Calibration of Refractive Index Liquids

Before any analysis is done, microscope alignment should be done every day and calibration of refractive index liquids should be done every three months (New York State ELAP Certification Manual, Method 198.6 (2016)).

Training

Microscopists should be trained to recognize asbestos morphology and determine optical characteristics of asbestos. The asbestos proficiency training should include analysis of not only the six forms of asbestos identified as TAC, but also the non-asbestos fibrous minerals that may be mistaken for asbestos (asbestos interferences). Microscopists who have analytical experience would still benefit from routine analysis of asbestos performance evaluation samples to refresh their familiarization with the six regulated asbestos. Staff recommends performing routine analyses of performance evaluation samples as part of the microscopists' weekly review of fixed slides of asbestos standard reference materials and asbestos interference minerals. See Appendix J, Recommended Training and Experience for Asbestos PLM Analysts.

Replicates

Blind analytical replicates of the same sample are recommended at a frequency of at least one in every 20 samples. Results of the primary and replicate analyses should match (i.e., either both analyses detect asbestos greater than or equal to 0.25 percent concentration, or both result in non-detect for asbestos). If replicate analyses do not match, the QA supervisor or laboratory manager should determine the reason for the difference and ensure that an appropriate control action is taken. All results and any corrective actions should be documented.

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Instrument Cross Checks

Instrument cross checks, include analyzing the same sample on different microscopes by the same microscopist, should be done periodically (e.g., every 20 analyses). The results of these cross checks should match. If they do not, the reason for the difference should be identified and the appropriate control action implemented and documented.

Method Validation

Method validation tests the ability of the laboratory to correctly process M435 samples and accurately detect asbestos when present. If not already performed and documented, the laboratory should perform a method validation study for M435. Using a set of performance evaluation (PE) samples (i.e., asbestos-containing and asbestos-free M435 PE samples), each laboratory can test their M435 SOP to make sure that asbestos is consistently detected when present in PE samples as well as to avoid false asbestos identifications.

Documentations

Documentation of analytical results is important and preferably done with a laboratory information management system (LIMS). The data recorded should include all bench analysis information such as the sample description using a stereoscopic microscope and crystallographic characteristics used to identify or rule out suspect fibers. Proof of the identification criteria (e.g., micrographs showing particle morphology, particles' refractive indices, birefringence, optical sign, etc.) should be recorded and saved in the LIMS. Clients should be informed of the option for photomicrographs of fibers identified as asbestos in their samples, for an additional cost. All type(s) of asbestos observed to be present should be identified and reported.

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VIII. APPENDICES

Appendixes A through J are on the following pages.

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