SOUTH COAST AIR QUALITY MANAGEMENT DISTRICT

APPLIED SCIENCE & TECHNOLOGY DIVISION

LABORATORY SERVICES BRANCH

SCAQMD METHOD 300-91

ANALYSIS OF ASBESTOS IN BULK BUILDING MATERIALS

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This method applies to the determination of asbestos in samples as regulated by Rule 1403.

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1.0   Principle

1.1   This method is intended for the identification and quantitative analysis of asbestos in bulk building materials.

For the analysis of bulk building materials by polarized light microscopy, follow the procedure specified in *EPA Test Method: Method for the Determination of Asbestos in Bulk Building Materials (EPA/600/R-93/116) (Section 9.1)*.

2.0   Analysis of Asbestos in Bulk Building Materials by XRD

2.1   In XRD analysis, crystalline materials will exhibit unique diffraction patterns characteristic of their crystalline structure. The concentration of asbestos is determined by measuring the net integrated area of the diagnostic peak corrected for matrix absorption effects and comparing with calibration standards.

2.2   For specific details which are not addressed in this method, refer to *EPA Test Method: "Method for the Determination of Asbestos in Bulk Building Materials" (EPA/600/R-93/116)* (Section 9.1) and *NIOSH 9000: Chrysotile Asbestos* (Section 9.2).

3.0   Equipment

3.1   *Siemen's* D5000 X-ray Diffractometer equipped with:
   3.1.1   X-ray tube, Copper target, high intensity, fine focus
   3.1.2   Detector, X-ray solid state Peltier cooled
   3.1.3   Stage, rotating
   3.1.4   Generator, constant potential with voltage and mA stabilizers
   3.1.5   Diffractometer, automated with step-scanning mode

3.1.6   Data output accessories, digital printer, PC and software which has the capability to determine background response

3.1.7   Plate, zero-background for sample mount

3.2   Standards, *NBS* standard reference material 1866 (chrysotile, amosite, and crocidolite) and 1867 (anthophyllite, actinolite, and tremolite).

3.3   Reference material, *NIST* standard reference material 1976 (Al_2O_3).

3.4   Sonicator, *Branson* Series 7000 low level ultrasonic bath, 0.01 Watt per milliter.

3.5   Mixer/mill, *Spex* 8000 mill.

3.6   Freezer mill, *Spex* 6700 freezer/mill.

3.7   Balance, analytical, *Cahn* CA 28.

3.8   Microscope, *Olympus* BH-2 polarized light microscope (PLM).

3.9   Silver membrane filters, *Osmonics* 25 mm diameter and 0.45 micron pore size.

3.10   Test tubes, 22 mm X 105 mm round bottom centrifuge type or equivalent.

3.11   Filtering apparatus, *Schleicher & Schuell* 25 mm diameter filtering funnel.

3.12   Pipets, *Gilson* pipetman P5000.

3.13   Flasks, volumetric, 100 milliters.

3.14   Weighing paper.

3.15   Glass stirring rods.

3.16   Petri dishes, plastic.

3.17   Forceps.

3.18   Needles.

4.0   Reagents

4.1   Isopropyl Alcohol, ACS grade or better.

4.2   Surfactant, 1 percent Aerosol® (registered trademark of American Cyanamid) OT aqueous solution or equivalent (optional).

5.0   Analytical Procedure

5.1   Standard preparation

5.1.1   Reduce the size of the standard asbestos fibers by cutting, grinding, and/or teasing to diameters of approximately 10 μm and lengths of 10-20 mm. Verify the fiber size by microscopic analysis. The use of a mixer/mill or freezer mill for standard asbestos fiber reduction should be avoided because fiber size may not be effectively reduced and the physical characteristics of the fibers may be altered.

5.1.2   Prepare a stock standard by weighing 5 mg of standard asbestos fibers to the nearest 0.01 mg. Record the weight. Transfer into a 100 ml volumetric flask and add approximately 50 mls of isopropyl alcohol (IPA). Add 2-4 drops of surfactant and sonicate for approximately 20 minutes.

5.1.3   Inspect the suspension for the presence of bundles of fibers and/or fibers that may have linked together to form a strand (this is especially problematic for chrysotile asbestos).   Break down bundles by pressing against the side of the flask with a glass

stirring rod. Long strands are more difficult to break up and may require discarding of suspension, reducing fiber lengths further and repeating the preparation of the suspension.

5.1.4   Dilute to 100 mls with IPA, add a magnetic stirring bar and stir for 15 minutes before using.

5.1.5   Prepare, in triplicate, 5 working standards by pipeting 1, 2, 3, 4, and 5 ml aliquots of the stock standard (Section 4.1.2) on silver membrane filters. This corresponds to 50, 100, 150 , 200 and 250 µg of standard asbestos deposited on the silver membrane filters. For each standard, mount a silver membrane filter onto the filtering apparatus and add 2 - 5 mls of IPA into the filtering funnel **(do not turn vacuum on)**. Shake the stock standard suspension vigorously and return to the magnetic stirrer. Decrease the stirring speed until there is no vortex and pipet exactly the desired volume from the middle of the suspension. Dispense the pipet's content into the filtering funnel ensuring that the tip of the pipette is just above the liquid level. Do not immerse the tip into the liquid. Rinse the pipet with a few mls of IPA into the filtering funnel. Turn on the vacuum and filter. After filtration, keep the vacuum on and allow the filter to dry for approximately one minute. Turn the vacuum off and remove the filter. Place the filter in a uniquely labeled petri dish. Record the weight of asbestos deposited on each filter as µg asbestos.

5.2   Sample preparation

All bulk samples must be analyzed by PLM (Section 9.1) prior to XRD analysis.

Samples to be analyzed by XRD should be prepared gravimetrically (Section 9.1). Record the final weight percent of residue as G. This procedure removes matrix materials that may possibly interfere in XRD analysis and enhances detection by concentrating the asbestiform material. If particle sizes are greater than 20-30 µm after gravimetric preparation,

reduce particle size further by grinding or milling. The use of a mill may be necessary to reduce sample particle sizes for some cementitious bulk materials such as transite, stucco, etc. Opt for a standard mill over a freezer mill, when possible, and minimize milling time.

5.2.1   Using the PLM/gravimetric results, determine the amount of sample residue (Section 5.2) needed that will yield a deposition of 100-200 μg of asbestos (not to exceed 250 μg). For example, a sample residue estimated to contain 4% asbestos by PLM/gravimetry will require 2.5-5 mg of residue for a deposition of 100-200 μg asbestos. A sample residue estimated to contain 25% asbestos requires 0.8 mg of sample residue to yield 200 μg asbestos. Weigh the sample using weighing paper and transfer into a test tube. (The test tube must be of centrifuge type or better to avoid breakage). Record the weights of sample deposited on the filter as W.

5.2.2   Suspend the sample in approximately 25-50 ml IPA. Add 2-3 drops of surfactant and sonicate for at least 20 minutes.

5.2.3   Inspect the sample suspension for conglomerates of particles. Break down any conglomerates by pressing against the wall of the test tube using a glass stirring rod.

5.2.4   Mount a silver membrane filter onto the filtering apparatus. Add approximately 5 mls. IPA. **Do not turn the vacuum on**. Pour the entire sample suspension into the filtering apparatus. Rinse the test tube with several mls of IPA and add the rinsing into the filtering apparatus. Start the filtration. The sides of the filtering funnel may be rinsed with IPA during filtration. Rinsing should be stopped when the liquid level is near the filter to avoid disturbing the deposited material. After filtration, keep the vacuum on and allow the filter to dry for approximately 1 minute. Turn the vacuum off and remove the filter. Place the filter in a uniquely labeled petri dish.

6.0   X-ray Diffraction Analysis

6.1   Standard Analysis

6.1.1   The following instrument parameters may be used as guidelines. The parameters may vary depending on the conditions necessary to optimize the response for each sample. Insure that the standards and samples are analyzed under identical instrument conditions.

   Slits - 2mm divergence, 2mm anti-scatter, 1mm detector
   Scan time - 2 sec/step
   Step size - 0.02°/step
   Scan range - 5°- 42° 2θ
   Sample rotation - 60 rpm

6.1.2   Mount the standard silver membrane filters onto a zero-background plate adhered with clear tape on the outside edges of the filter for analysis.

6.1.3   Step-scan the selected diagnostic regions of the standards. See Chart 1. The standards and samples must be scanned under the same instrument conditions and 2θ range.

6.1.4   Step-scan the NIST reference material (Section 3.3) after every other standard run to correct for instrument drift.

6.1.5   Integrate the selected asbestos diffraction peak of the standard and record its integrated intensity as P_{std}. Integrate the selected reference diffraction peak (most intense) of the NIST reference material and record its integrated intensity as P_r. The procedure and units for determining the intensity must be consistent for standards, samples and reference peaks.

6.1.6   Measure the background on each side of the peak for equal time used for peak scanning. Determine the average total background by adding the integrated intensity from each side of the peak and dividing by 2. Record as B_{std} for the asbestos peak and B_r for the NIST reference material.

6.1.7   Calculate and record the net integrated intensity (the difference between the peak integrated intensity and total background integrated intensity) for the selected asbestos peak by:

$$I_{std} = P_{std} - B_{std}$$

6.1.8   Calculate and record the net integrated intensity (the difference between the peak integrated intensity and total background integrated intensity) for the NIST reference material peak by:

$$I_r = P_r - B_r$$

6.1.9   Determine a normalization scale factor, N, by averaging all I_r results and rounding to the nearest 100. Use this normalization scale factor for all standard, sample, and reference calculations.

6.1.10   Calculate and record the normalized intensity, $I^{\wedge}_{std}$, for each standard as follows:

$$I^{\wedge}_{std} = (I_{std} / I_r) \times N$$

Where: $I^{\wedge}_{std}$ = normalized intensity of the standard
        I_{std} = net integrated intensity of the standard (Section 6.1.7)
        N = normalization scale factor (Section 6.1.9)
        I_r = net intensity of the NIST reference material closest to the standard run (Section 6.1.8)

6.1.11 Calculate an average normalized intensity for each triplicate standard preparation by adding the $I^{\wedge}$std for each run and dividing by 3. Record this result as $I^{\wedge o}$std.

6.1.12 Prepare a calibration curve of averaged normalized intensity $I^{\wedge o}$std (Section 6.1.11) vs. µg asbestos (Section 5.1.5). Calculate and record the slope, m, and the intercept, b, of the calibration curve by using least squares linear regression.

6.2   Sample Analysis

6.2.1   Mount the sample filters onto a zero-background plate adhered with clear tape on the outside edges of the filter for analysis.

6.2.2   Obtain a qualitative XRD scan of the samples and determine the presence of asbestiform material (Section 9.1). The most intense diffraction peak attributed to asbestos which has been determined to be free from matrix interference should be used for quantitation. The samples and standards should be scanned under the same instrument conditions and 2θ range.

6.2.3   Step scan the NIST reference material (Section 3.3) after every other sample.

6.2.4   Integrate the selected asbestos diffraction peak of the sample and record its integrated intensity as P_x. Integrate the selected reference diffraction peak of the NIST reference material and record its integrated intensity as P_r. The procedure and units for determining the intensity must be consistent for standards, samples and reference peaks.

6.2.5   Measure the background on each side of the peak for equal time used for peak scanning. Determine the average total background by adding the integrated intensity from each side of the peak and dividing by 2. Record as B_x for the asbestos peak and B_r for the NIST reference material.

6.2.6   Calculate and record the net integrated intensity (the difference between the peak integrated intensity and total background integrated intensity) for the selected asbestos peak by:

$$I_x = P_x - B_x$$

6.2.7   Calculate and record the net integrated intensity (the difference between the peak integrated intensity and total background integrated intensity) for the NIST reference material peak by:

$$I_r = P_r - B_r$$

6.2.8   Calculate and record the normalized intensity, $I^\wedge x$, for each sample as follows:

$$I^\wedge x = (I_x / I_r) \times N$$

Where: $I^\wedge x$ = normalized intensity of the sample

I_x =   net intensity of the sample (Section   6.2.6)

N = normalization scale factor (Section 6.1.9)

I_r = net intensity of the NIST reference material closest to the sample run (Section 6.2.7)

6.2.9   Integrate the silver membrane diffraction peak at 38.2° 2θ of the sample and record its integrated intensity as P_{Ag}.

6.2.10   Measure the background on each side of the peak for equal time used for peak scanning. Determine the average total background by adding the integrated intensity from each side of the peak and dividing by 2. Record as B_{Ag} for the silver peak.

6.2.11 Calculate and record the net integrated intensity (the difference between the peak integrated intensity and total background integrated intensity) for the silver peak by:

$$I_{Ag} = P_{Ag} - B_{Ag}$$

6.2.12 Calculate and record the normalized intensity, $I^{\wedge}_{Ag}$, for each sample as follows:

$$I^{\wedge}_{Ag} = (I_{Ag}/I_r) \times N$$

Where: $I^{\wedge}_{Ag}$ =   normalized intensity of the silver membrane diffraction peak

I_{Ag} =   net intensity of the silver peak (Section 6.2.11)

N =   normalization scale factor (Section 6.1.9)

I_r =   net intensity of the NIST reference material closest to the sample run (Section 6.2.7)

6.3   Blank Silver Membrane Filter Analysis

6.3.1   For each new lot number of silver membrane filters, select six filters. Scan between 35° and 42° 2θ under the same instrument conditions as the standards and sample.

6.3.2   Step scan the NIST reference material (Section 3.3) after every other silver membrane filter scan.

6.3.3   Integrate the silver peak at 38.2° 2θ for each blank filter and record the integrated intensity as $P_{Ag(blank)}$.   Integrate the selected reference diffraction peak of the NIST reference material and record its integrated intensity as P_r. The procedure and units for determining the intensity must be consistent for standards, samples and reference peaks.

6.3.4   Measure the background on each side of the peak for equal time used for peak scanning. Determine the average total background by adding the integrated intensity from each side of the peak and dividing by 2. Record as $B_{Ag(blank)}$ for the silver peak and B_r for the NIST reference material.

6.3.5   Calculate and record the net integrated intensity (the difference between the peak integrated intensity and total background integrated intensity) for the silver peak by:

$$I_{Ag(blank)} = P_{Ag(blank)} - B_{Ag(blank)}$$

6.3.6   Calculate and record the net integrated intensity (the difference between the peak integrated intensity and total background integrated intensity) for the NIST reference material peak by:

$$I_r = P_r - B_r$$

6.3.7   Calculate and record the normalized intensity, $I^{\wedge}_{Ag(blank)}$, for the blank silver membrane filters as follows:

$$I^{\wedge}_{Ag(blank)} = (I_{Ag(blank)} / I_r) \times N$$

Where: $I^{\wedge}_{Ag(blank)}$ =   normalized blank filter silver peak intensity

$I_{Ag(blank)}$ =   blank filter silver peak intensity (Section 6.3.5)

N =   normalization scale factor (Section 6.1.9)

I_r =   net intensity of the NIST reference material (Section 6.3.6)

6.3.8   Calculate an average intensity by adding all $I^{\wedge}_{Ag(blank)}$ results and divide by 6. Record as $I^{\wedge o}_{Ag(blank)}$.

7.0   Calculations

7.1   Calculate the weight percent asbestos in bulk materials as follows:

$$C = \frac{(\hat{I}_x \times f(T)) - b}{m \times W} \times G$$

Where: C   =   weight percent asbestos

$\hat{I}_x$ =   normalized intensity for the sample peak
(Section 6.2.8)

b   =   intercept of calibration curve (Section 6.1.12)

m   =   slope of calibration curve (Section 6.1.12)

W   =   mass of sample filter deposit (Section 5.2.1)

$$f(T) = \frac{-R\,(\ln T)}{1 - T^R} = \text{absorption correction factor}$$

R   = $\sin(\theta_{Ag})/\sin(\theta_X)$

T   = $\hat{I}_{Ag} / \hat{I}^o_{Ag(blank)}$ = sample transmittance

$\hat{I}_{Ag}$ =   normalized sample silver peak intensity
(Section 6.2.12)

$\hat{I}^o_{Ag(blank)}$ =   average normalized blank filter silver
peak   intensity (Section 6.3.7)

G   =   percent residue remaining after
gravimetric procedure (Section 5.2)

8.0   Quality Control

8.1   For every 5 samples, analyze known reference materials (such as NIST-NVLAP accreditation samples) that are similar to the type of samples being analyzed for a positive control. Acceptable ranges will depend on the type of reference material used.

8.2   A blank must be analyzed with each set of 5 samples to detect contamination. Prepare according to procedures in Section 5.2, and analyze according to Section 6.0.

8.3   Prepare a new set of calibration standards every 6 months or when the positive control falls out of the acceptable range.

9.0   References

9.1   Perkins, R.L., Harvey ,B.W.; **EPA Test Method: Method for the Determination of Asbestos in Bulk Building Materials;** EPA/600/R-93/116; July 1993.

9.2   **NIOSH Method 9000: Chrysotile Asbestos;** February 1984.

10.0 Chart 1 - Reconstructed from **EPA Test Method: Method for the Determination of Asbestos in Bulk Building Materials;** *Table 2-9, page 37*

Asbestiform Mineral	Primary diagnostic peaks (approximate d spacings in A)	Interference
Serpentine Chrysotile	7.3	Nonasbestiform serpentines, (antigorite, lizardite), chlorite, vermiculite, sepiolite, kaolinite, gypsum
	3.7	Nonasbestiform serpentines (antigorite, lizardite), chlorite, vermiculite, halloysite, cellulose
Amphibole Amosite (Grunerite) Anthophyllite Crocidolite (Riebeckite)	3.1	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences, talc, carbonates
Tremolite Actinolite	8.3	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences