

Part Number 1910

Standard Number 1910.1001 App A

Title OSHA Reference Method - Mandatory

This mandatory appendix specifies the procedure for analyzing air samples for asbestos and specifies quality control procedures that must be implemented by laboratories performing the analysis. The sampling and analytical methods described below represent the elements of the available monitoring methods (such as the NIOSH 7400 method) which OSHA considers to be essential to achieve adequate employee exposure monitoring while allowing employers to use methods that are already established within their organizations. All employers who are required to conduct air monitoring under paragraph (d) of the standard are required to utilize analytical laboratories that use this procedure, or an equivalent method, for collecting and analyzing samples.

Sampling and Analytical Procedure

1. The sampling medium for air samples shall be mixed cellulose ester filter membranes. These shall be designated by the manufacturer as suitable for asbestos counting. See below for rejection of blanks.

2. The preferred collection device shall be the 25-mm diameter cassette with an open-faced 50-mm electrically conductive extension cowl. The 37-mm cassette may be used if necessary but only if written justification for the need to use the 37-mm filter cassette accompanies the sample results in the employee's exposure monitoring record.

3. An air flow rate between 0.5 liter/min and 2.5 liters/min shall be selected for the 25-mm cassette. If the 37-mm cassette is used, an air flow rate between 1 liter/min and 2.5 liters/min shall be selected.

4. Where possible, a sufficient air volume for each air sample shall be collected to yield between 100 and 1,300 fibers per square millimeter on the membrane filter. If a filter darkens in appearance or if loose dust is seen on the filter, a second sample shall be started.

5. Ship the samples in a rigid container with sufficient packing material to prevent dislodging the collected fibers. Packing material that has a high electrostatic charge on its surface (e.g., expanded polystyrene) cannot be used because such material can cause loss of fibers to the sides of the cassette.

6. Calibrate each personal sampling pump before and after use with a representative filter cassette installed between the pump and the calibration devices.

7. Personal samples shall be taken in the "breathing zone" of the employee (i.e., attached to or near the collar or lapel near the worker's face).

8. Fiber counts shall be made by positive phase contrast using a microscope with an 8 to 10 X eyepiece and a 40 to 45 X objective for a total magnification of approximately 400 X and a numerical aperture of 0.65 to 0.75. The microscope shall also be fitted with a green or blue filter.

9. The microscope shall be fitted with a Walton-Beckett eyepiece graticule calibrated for a field diameter of 100 micrometers (+/-2 micrometers).

10. The phase-shift detection limit of the microscope shall be about 3 degrees measured using the HSE phase shift test slide as outlined below.

a. Place the test slide on the microscope stage and center it under the phase objective.

- b. Bring the blocks of grooved lines into focus.

NOTE: The slide consists of seven sets of grooved lines (ca. 20 grooves to each block) in descending order of visibility from sets 1 to 7, seven being the least visible. The requirements for asbestos counting are that the microscope optics must resolve the grooved lines in set 3 completely, although they may appear somewhat faint, and that the grooved lines in sets 6 and 7 must be invisible. Sets 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope that fails to meet these requirements has either too low or too high a resolution to be used for asbestos counting.

- c. If the image deteriorates, clean and adjust the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Each set of samples taken will include 10 percent blanks or a minimum of 2 blanks. The blank results shall be averaged and subtracted from the analytical results before reporting. Any samples represented by a blank having a fiber count in excess of 7 fibers/100 fields shall be rejected.

12. The samples shall be mounted by the acetone/triacetin method or a method with an equivalent index of refraction and similar clarity.

13. Observe the following counting rules.

- a. Count only fibers equal to or longer than 5 micrometers. Measure the length of curved fibers along the curve.

- b. In the absence of other information, count all particles as asbestos that have a length-to-width ratio (aspect ratio) of 3:1 or greater.

- c. Fibers lying entirely within the boundary of the Walton-Beckett graticule field shall receive a count of 1. Fibers crossing the boundary once, having one end within the circle, shall receive the count of one half (1/2). Do not count any fiber that crosses the graticule boundary more than once. Reject and do not count any other fibers even though they may be visible outside the graticule area.

- d. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of an individual fiber.

- e. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields; stop counting at 100 fields regardless of fiber count.

14. Blind recounts shall be conducted at the rate of 10 percent.

Quality Control Procedures

1. Intralaboratory program. Each laboratory and/or each company with more than one microscopist counting slides shall establish a statistically designed quality assurance program involving blind recounts and comparisons between microscopists to monitor the variability of counting by each microscopist and between microscopists. In a company with more than one laboratory, the program shall include all laboratories and shall also evaluate the laboratory-to-laboratory variability.

2. Interlaboratory program. Each laboratory analyzing asbestos samples for compliance determination shall implement an interlaboratory quality assurance program that as a minimum includes participation of at least two other independent laboratories. Each laboratory shall participate in round robin testing at least once every 6 months with at least all the other laboratories in its interlaboratory quality assurance group. Each laboratory shall submit slides typical of its own work load for use in this program. The round robin shall be designed and results analyzed using appropriate statistical methodology.

3. All individuals performing asbestos analysis must have taken the NIOSH course for sampling and evaluating airborne asbestos dust or an equivalent course.

4. When the use of different microscopes contributes to differences between counters and laboratories, the effect of the different microscope

shall be evaluated and the microscope shall be replaced, as necessary.

5. Current results of these quality assurance programs shall be posted in each laboratory to keep the microscopists informed.

[57 FR 24330, June 8, 1992]

EXAMPLE -

If $L(o) = 108 \text{ um}$, $L(a) = 2.93 \text{ mm}$ and $D = 100 \text{ um}$, then $d(c) = 2.71 \text{ mm}$.

h. Check the field diameter, D (acceptable range $100 \text{ mm} + \text{ or } - 2 \text{ mm}$) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (mm^2).

12. Microscope adjustments. Follow the manufacturer's instructions and also the following:

a. Adjust the light source for even illumination across the field of view at the condenser iris.

NOTE: Kohler illumination is preferred, where available.

b. Focus on the particulate material to be examined.

c. Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.

d. Use the telescope ocular supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric.

13. Check the phase-shift detection limit of the microscope periodically.

a. Remove the HSE/NPL phase-contrast test slide from its shipping container and center it under the phase objective.

b. Bring the blocks of grooved lines into focus.

NOTE: The slide consists of seven sets of grooves (ca. 20 grooves to each block) in descending order of visibility from sets 1 to 7. The requirements for counting are that the microscope optics must resolve the grooved lines in set 3 completely, although they may appear somewhat faint, and that the grooved lines in sets 6 to 7 must be invisible. Sets 4 and 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has either too low or too high a resolution to be used for asbestos counting.

c. If the image quality deteriorates, clean the microscope optics and, if the problem persists, consult the microscope manufacturer.

14. Quality control of fiber counts.

a. Prepare and count field blanks along with the field samples. Report the counts on each blank. Calculate the mean of the field blank counts and subtract this value from each sample count before reporting the results.

NOTE 1: The identity of the blank filters should be unknown to the counter until all counts have been completed.

NOTE 2: If a field blank yields fiber counts greater than 7 fibers/100 fields, report possible contamination of the samples.

b. Perform blind recounts by the same counter on 10 percent of filters counted (slides relabeled by a person other than the counter).

15. Use the following test to determine whether a pair of counts on the same filter should be rejected because of possible bias. This statistic estimates the counting repeatability at the 95% confidence level. Discard the sample if the difference between the two counts exceeds $2.77(F)sr$, where F =average of the two fiber counts and sr =relative standard deviation, which should be derived by each laboratory based on historical in-house data.

NOTE: If a pair of counts is rejected as a result of this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts.

16. Enroll each new counter in a training course that compares

performance of counters on a variety of samples using this procedure.

NOTE: To ensure good reproducibility, all laboratories engaged in asbestos counting are required to participate in the Proficiency Analytical Testing (PAT) Program and should routinely participate with other asbestos fiber counting laboratories in the exchange of field samples to compare performance of counters.

Measurement

17. Place the slide on the mechanical stage of the calibrated microscope with the center of the filter under the objective lens. Focus the microscope on the plane of the filter.

18. Regularly check phase-ring alignment and Kohler illumination.

19. The following are the counting rules:

a. Count only fibers longer than 5 μm . Measure the length of curved fibers along the curve.

b. Count only fibers with a length-to-width ratio equal to or greater than 3:1.

c. For fibers that cross the boundary of the graticule field, do the following:

1. Count any fiber longer than 5 μm that lies entirely within the graticule area.

2. Count as 1/2 fiber any fiber with only one end lying within the graticule area.

3. Do not count any fiber that crosses the graticule boundary more than once.

4. Reject and do not count all other fibers.

d. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.

e. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 fields regardless of fiber count.

20. Start counting from one end of the filter and progress along a radial line to the other end, shift either up or down on the filter, and continue in the reverse direction. Select fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. When an agglomerate covers ca. 1/6 or more of the field of view, reject the field and select another. Do not report rejected fields in the number of total fields counted.

NOTE: When counting a field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count.

Calculations

21. Calculate and report fiber density on the filter, E (fibers/ mm^2); by dividing the total fiber count, F ; minus the mean field blank count, B , by the number of fields, n ; and the field area, $A(f)$ (0.00785 mm^2 for a properly calibrated Walton-Beckett graticule):

$$E = \frac{(F/n(f) - (B/n(b)))}{A(f)} \text{ fibers/mm}^2$$

where:

$n(f)$ = number of fields in submission sample

$n(b)$ = number of fields in blank sample

22. Calculate the concentration, C (f/cc), of fibers in the air volume

sampled, V (L), using the effective collection area of the filter, A(c)
(385 mm(2) for a 25-mm filter):

$$C = \frac{(E) (A(c))}{V(10(3))}$$

NOTE: Periodically check and adjust the value of Ac, if necessary.

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Part Number 1910

Standard Number 1910.1001 App B

Title Detailed procedure for asbestos sampling and analysis -
Non-Mandatory

This appendix contains a detailed procedure for sampling and analysis and includes those critical elements specified in Appendix A. Employers are not required to use this procedure, but they are required to use Appendix A. The purpose of Appendix B is to provide a detailed step-by-step sampling and analysis procedure that conforms to the elements specified in Appendix A. Since this procedure may also standardize the analysis and reduce variability, OSHA encourages employers to use this appendix.

Asbestos Sampling and Analysis Method

Technique: Microscopy, Phase Contrast

Analyte: Fibers (manual count)

Sample Preparation: Acetone/triacetin method

Calibration: Phase-shift detection limit about 3 degrees

Range: 100 to 1300 fibers/mm² filter area

Estimated limit of detection: 7 fibers/mm² filter area

Sampler: Filter (0.8-1.2 μ m mixed cellulose ester membrane, 25-mm diameter)

Flow rate: 0.5 L/min to 2.5 L/min (25-mm cassette) 1.0 L/min to 2.5 L/min (37-mm cassette)

Sample volume: Adjust to obtain 100 to 1300 fibers/mm²

Shipment: Routine

Sample stability: Indefinite

Blanks: 10% of samples (minimum 2)

Standard analytical error: 0.25.

Applicability: The working range is 0.02 f/cc (1920-L air sample) to 1.25 f/cc (400-L air sample). The method gives an index of airborne asbestos fibers but may be used for other materials such as fibrous glass by inserting suitable parameters into the counting rules. The method does not differentiate between asbestos and other fibers. Asbestos fibers less than ca. 0.25 μ m diameter will not be detected by this method.

Interferences: Any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chainlike particles may appear fibrous. High levels of nonfibrous dust particles may obscure fibers in the field of view and raise the detection limit.

Reagents: 1. Acetone. 2. Triacetin (glycerol triacetate), reagent grade

Special precautions: Acetone is an extremely flammable liquid and precautions must be taken not to ignite it. Heating of acetone must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

Equipment: 1. Collection device: 25-mm cassette with 50-mm electrically conductive extension cowl with cellulose ester filter, 0.8 to 1.2 mm pore size and backup pad.

NOTE: Analyze representative filters for fiber background before use and discard the filter lot if more than 5 fibers/100 fields are found.

2. Personal sampling pump, greater than or equal to 0.5 L/min. with flexible connecting tubing.

3. Microscope, phase contrast, with green or blue filter, 8 to 10X eyepiece, and 40 to 45X phase objective (total magnification ca 400X; numerical aperture = 0.65 to 0.75.

4. Slides, glass, single-frosted, pre-cleaned, 25 x 75 mm.
 5. Cover slips, 25 x 25 mm, no. 1 1/2 unless otherwise specified by microscope manufacturer.
 6. Knife, No. 1 surgical steel, curved blade.
 7. Tweezers.
 8. Flask, Guth-type, insulated neck, 250 to 500 mL (with single-holed rubber stopper and elbow-jointed glass tubing, 16 to 22 cm long).
 9. Hotplate, spark-free, stirring type; heating mantle; or infrared lamp and magnetic stirrer.
 10. Syringe, hypodermic, with 22-gauge needle.
 11. Graticule, Walton-Beckett type with 100 μ m diameter circular field at the specimen plane (area = 0.00785 mm²). (Type G-22).
- NOTE: the graticule is custom-made for each microscope.
12. HSE/NPL phase contrast test slide, Mark II.
 13. Telescope, ocular phase-ring centering.
 14. Stage micrometer (0.01 mm divisions).

Sampling

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Fasten the sampler to the worker's lapel as close as possible to the worker's mouth. Remove the top cover from the end of the cowl extension (open face) and orient face down. Wrap the joint between the extender and the monitor's body with shrink tape to prevent air leaks.
3. Submit at least two blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove the caps from the field blank cassettes and store the caps and cassettes in a clean area (bag or box) during the sampling period. Replace the caps in the cassettes when sampling is completed.
4. Sample at 0.5 L/min or greater. Do not exceed 1 mg total dust loading on the filter. Adjust sampling flow rate, Q (L/min), and time to produce a fiber density, E (fibers/mm²), of 100 to 1300 fibers/mm² [3.85×10^4 to 5×10^5 fibers per 25-mm filter with effective collection area ($A(c) = 385$ mm²)] for optimum counting precision (see step 21 below). Calculate the minimum sampling time, $t(\text{minimum})$ (min) at the action level (one-half of the current standard), L (f/cc) of the fibrous aerosol being sampled:

$$t(\text{minimum}) = \frac{(Ac)(E)}{(Q)(L)10(3)}$$

5. Remove the field monitor at the end of sampling, replace the plastic top cover and small end caps, and store the monitor.
 6. Ship the samples in a rigid container with sufficient packing material to prevent jostling or damage.
- NOTE: Do not use polystyrene foam in the shipping container because of electrostatic forces which may cause fiber loss from the sample filter.

Sample Preparation

NOTE: The object is to produce samples with a smooth (non-grainy) background in a medium with a refractive index equal to or less than 1.46. The method below collapses the filter for easier focusing and produces permanent mounts which are useful for quality control and interlaboratory comparison. Other mounting techniques meeting the above criteria may also be used, e.g., the nonpermanent field mounting technique used in P & CAM 239.

7. Ensure that the glass slides and cover slips are free of dust and

fibers.

8. Place 40 to 60 ml of acetone into a Guth-type flask. Stopper the flask with a single-hole rubber stopper through which a glass tube extends 5 to 8 cm into the flask. The portion of the glass tube that exits the top of the stopper (8 to 10 cm) is bent downward in an elbow that makes an angle of 20 to 30 degrees with the horizontal.

9. Place the flask in a stirring hotplate or wrap in a heating mantle. Heat the acetone gradually to its boiling temperature (ca. 58 deg. C).

CAUTION. - The acetone vapor must be generated in a ventilated fume hood away from all open flames and spark sources. Alternate heating methods can be used, providing no open flame or sparks are present.

10. Mount either the whole sample filter or a wedge cut from the sample filter on a clean glass slide.

a. Cut wedges of ca. 25 percent of the filter area with a curved-blade steel surgical knife using a rocking motion to prevent tearing.

b. Place the filter or wedge, dust side up, on the slide. Static electricity will usually keep the filter on the slide until it is cleared.

c. Hold the glass slide supporting the filter approximately 1 to 2 cm from the glass tube port where the acetone vapor is escaping from the heated flask. The acetone vapor stream should cause a condensation spot on the glass slide ca. 2 to 3 cm in diameter. Move the glass slide gently in the vapor stream. The filter should clear in 2 to 5 sec. If the filter curls, distorts, or is otherwise rendered unusable, the vapor stream is probably not strong enough. Periodically wipe the outlet port with tissue to prevent liquid acetone dripping onto the filter.

d. Using the hypodermic syringe with a 22-gauge needle, place 1 to 2 drops of triacetin on the filter. Gently lower a clean 25-mm square cover slip down onto the filter at a slight angle to reduce the possibility of forming bubbles. If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours.

e. Glue the edges of the cover slip to the glass slide using a lacquer or nail polish.

NOTE: If clearing is slow, the slide preparation may be heated on a hotplate (surface temperature 50 deg. C) for 15 min to hasten clearing. Counting may proceed immediately after clearing and mounting are completed.

Calibration and Quality Control

11. Calibration of the Walton-Beckett graticule. The diameter, $d(c)$ (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

a. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.

b. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.

c. Install the 40 to 45X phase objective.

d. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.

e. Measure the magnified grid length, $L(o)$ (mm), using the stage micrometer.

f. Remove the graticule from the microscope and measure its actual grid length, $L(a)$ (mm). This can best be accomplished by using a stage fitted with verniers.

g. Calculate the circle diameter, $d(c)$ (mm), for the Walton-Beckett graticule:

$$d(c) = \frac{L(a) \times D}{L(o)}$$