FRICTION MATERIALS STANDARDS INSTITUTE, INC., EAST 210 ROUTE 4, PARAMUS, NJ 07652

B U L L E T I N

N O. 8 6 0

July 9, 1986

NIOSH METHOD 7400 FOR MEASURING ASBESTOS DUST CONCENTRATIONS

The National Institute for Occupational Safety and Health (NIOSH) has adopted their Method 7400 for making airborne asbestos fiber counts. They note 0.02 to 1.25 fibers per ml as a working range.

In the new OSHA Standards published in the FEDERAL REGISTER on June 20, 1986, NIOSH Method 7400 is listed under their Mandatory Appendix A as an essential monitoring method. While there was only limited information available at the June 1986 Meetings on the OSHA Standard (the Standard had not then been published in the FEDERAL REGISTER) it had been noted that NIOSH Method 7400 was considered an essential method for counting asbestos fiber concentrations.

At the Meeting, Ms. Grisham of Nuturn volunteered to send the Institute a copy of Method 7400 for distribution to Institute Members. A copy is enclosed.

For your information.

E. W. Drislane
Executive Director

Distribution:
Delegates and Alternates
H.E.A. Committee

FORMULA: various

M.W.: various

FIBERS

METHOD: 7400

ISSUED: 2/15/84

OSHA: 0.5 asbestos fibers (> 5 μm long)/mL
NIOSH: 0.1 asbestos f/mL [1]; 3 glass fibers (>10 μm x <3.5 μm)/mL [2]
ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, f/mL

PROPERTIES: solid, fibrous

SYNONYMS: asbestos (actinolite [CAS #77536-66-4], grunerite (amosite) [CAS #12172-73-5], anthophyllite [CAS #77536-67-5], chrysotile [CAS #12001-29-5], crocidolite [CAS #12001-28-4], tremolite [CAS #77536-68-6]); fibrous glass.

CAS #'s are incorrect

SAMPLING

SAMPLER: FILTER
(0.8-1.2 μm cellulose ester membrane, 25-mm diameter)

FLOW RATE*: ≥ 0.5 L/min

VOL-MIN*: 400 L @ 0.1 fiber/mL
-MAX*: 1920 L @ 0.1 fiber/mL
*Adjust for 100 to 1300 fibers/mm² (step 4)

SHIPMENT: routine

SAMPLE STABILITY: indefinite

BLANKS: 10% of samples (minimum 2) [3]

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: see EVALUATION OF METHOD

OVERALL PRECISION (s_r): 0.115 to 0.13 [3] (A Rules)

MEASUREMENT

TECHNIQUE: MICROSCOPY, PHASE CONTRAST

ANALYTE: fibers (manual count)

SAMPLE PREPARATION: acetone/triacetin method

COUNTING RULES: Set A (P&CAM 239 [3,4]) or Set B (modified CRS [5])

EQUIPMENT: 1. phase-contrast microscope
2. Walton-Beckett graticule (100 μm field diameter): A Rules use G-22; B Rules use Type G-24
3. phase-shift test slide (HSE/NPL)

CALIBRATION: phase-shift detection limit about 3 degrees [7]

RANGE: 100 to 1300 fibers/mm² filter area [6]

ESTIMATED LOD: 7 fibers/mm² filter area

PRECISION: 0.10 to 0.12 [3] (A Rules)

APPLICABILITY: The working range is 0.02 fiber/mL (1920-L air sample) to 1.25 fibers/mL (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 μm diameter will not be detected by this method [7].

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

OTHER METHODS: This method introduces changes for improved sensitivity and reproducibility and replaces P&CAM 239 [3,4].

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REAGENTS:

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

*See Special Precautions.

EQUIPMENT:

1. Sampler: field monitor, 25 mm, three-piece cassette with 50-mm electrically conductive extension cowl with cellulose ester filter, 0.8 to 1.2-µm 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, ≥ 0.5 L/min (see step 4 for flow rate), 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, #10 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 µm diameter circular field at the specimen plane (area = 0.00785 mm²) (Type G-22 for A Rules; Type G-24 for B Rules). Available from Graticules Ltd., Morley Road, Tonbridge TN9 1RN, Kent, England (Telephone 011-44-732-359061).
    NOTE: The graticule is custom-made for each microscope. Specify disc diameter needed to fit exactly the ocular of the microscope and the diameter (mm) of the circular counting area (see step 11).
12. HSE/NPL phase contrast test slide, Mark II. Available from PTR Optics Ltd., 145 Newton Street, Waltham, MA 02154 (Telephone (617) 891-6000).
13. Telescope, ocular phase-ring centering.
14. Stage micrometer (0.01 mm divisions).

PTR is the US distributor for the graticule and test slides ordered here. graticule #170 $150 test slides

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.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line [3].
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 monitor body with shrink tape to prevent air leaks.

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3. Submit at least two field 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 [8]. 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/mm^2), of 100 to 1300 fibers/mm^2 [3.85 • 10^4 to 5 • 10^5 fibers per 25-mm filter with effective collection area (A_c= 385 mm^2)] for optimum counting precision (see step 21). Calculate the minimum sampling time, t_{min} (min), at the action level (one-half the current standard), L (fibers/mL), of the fibrous aerosol being sampled:

$$t_{min} = \frac{(A_c)(E)}{(Q)(L)10^3}.$$

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 sampler 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 non-permanent field mounting technique used in P&CAM 239 [1,3,4]).

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 which exits the top of the stopper (8 to 10 cm) is bent downward in an elbow which makes an angle of 20 to 30° with the horizontal.
9. Place the flask on a stirring hotplate or wrap in a heating mantle. Heat the acetone gradually to its boiling temperature (ca. 58 °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% 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.

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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 [9].

NOTE: If clearing is slow, the slide preparation may be heated on a hotplate (surface temperature 50 °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 (µm), 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:

$$d_c = \frac{L_a}{L_o} \times D.$$

    Example: If L_o = 108 µm, L_a = 2.93 mm and D = 100 µm, then d_c = 2.71 mm.
    h. Check the field diameter, D(acceptable range 100 µm ± 2 µm) 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: Köhler 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 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 which fails to meet these requirements has either too low or too high a resolution to be used for asbestos counting.

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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% 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 $(F)s_r$, where F = average of the two fiber counts and s_r = 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 which compares performance of counters on a variety of samples using this procedure.
    NOTE: To ensure good reproducibility, all laboratories engaged in asbestos counting should participate in an asbestos proficiency testing program such as the NIOSH Proficiency Analytical Testing (PAT) Program and 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 Köhler illumination [7].
19. Select one of the following sets of counting rules:
    NOTE: The two sets of rules have been demonstrated to produce equivalent mean counts on a variety of asbestos sample types [5] and must be strictly followed in order to obtain valid results. No hybridizing of the two sets of rules is permitted. The calibration of the microscope with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 µm).
    a. A Rules (same as P&CAM 239 rules [1,3,4]).
       NOTE: The A Rules are required for monitoring asbestos for compliance purposes under OSHA or NIOSH standards.
       1. Count only fibers longer than 5 µm. Measure the length of curved fibers along the curve.
       2. Count only fibers with a length-to-width ratio equal to or greater than 3:1.
       3. For fibers which cross the boundary of the graticule field, do the following:
          a. Count any fiber longer than 5 µm which lies entirely within the graticule area.
          b. Count as 1/2 fiber any fiber with only one end lying within the graticule area.
          c. Do not count any fiber which crosses the graticule boundary more than once.
          d. Reject and do not count all other fibers.

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4. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
5. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 fields regardless of fiber count.

b. B Rules

NOTE: The B Rules are preferred analytically because of their demonstrated ability to improve the reproducibility of fiber counts [5].

1. Count only ends of fibers. Each fiber must be longer than 5 μm and less than 3 μm diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules b.1 and b.2.
4. Count visibly free ends which meet rules b.1 and b.2 when the fiber appears to be attached to another particle, regardless of the size of the other particle.
5. Count the free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules b.1 and b.2.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 fields. Stop at 100 fields, regardless of the fiber count.
7. Divide the total end count by 2 to yield fiber count.

NOTE: Split fibers will normally be counted as more than two ends if the free ends meet the rules b.1. and b.2.

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 [10]. 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):

$$E = \frac{(F - B)}{(n)(A_f)}, \text{ fibers/mm}^2.$$

22. Calculate the concentration, C (fibers/mL), 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):

$$C = \frac{(E)(A_c)}{V \cdot 10^3}.$$

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

EVALUATION OF METHOD:

This method is a revision of NIOSH Method P&CAM 239 [1,3,4]. A summary of the revisions is as follows:

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A. Sampling

The change from a 37-mm to a 25-mm filter size was incorporated to improve sensitivity and reduce problems associated with non-uniform fiber loading reported on the 37-mm filters [10]. The change in flow rates allows for 2 m^3 full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not affected by changes in flow rate in the range 0.5 to 16 L/min [8].

B. Sample Preparation Technique

The acetone vapor-triacetin preparation technique has been incorporated in the method as a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [1,3,4,11].

C. Measurement

1. The inclusion of the Walton-Beckett graticule in the method was made to standardize the field area observed through the eyepiece [6,11].
2. The introduction of the HSE/NPL test slide was made to standardize microscope optics for sensitivity to fiber diameter [7,11].
3. A recent international collaborative study involved 16 laboratories using prepared slides from the asbestos, cement, milling, mining, textile, and friction material industries [5]. The relative levels of count by different counting rules were:

Sample Type	Number of Samples	Aspect Ratio > 3:1 AIA	Aspect Ratio > 3:1 Mod. CRS*	Aspect Ratio > 5:1 AIA	Aspect Ratio > 5:1 Mod. CRS*
Mining	10	100	127	74	92
Milling	10	100	112	84	95
Asbestos Cement	14	100	146	90	137
Textile Chrysotile	10	100	109	89	99
Friction Material	10	100	130	87	116
Others (Insulation, Amosite)	6	100	127	92	118
TOTAL:	60 MEAN:	100	125	86	110

*Arithmetic means of counts made by different laboratories relative to the AIA counts.

The modified CRS (NIOSH B) Rules were found to be more precise than the AIA (NIOSH A)* Rules. The ranges of relative standard deviations (s_r) which varied with sample type and laboratory were:

	s_r		
	Intralaboratory	Interlaboratory	Overall
AIA (NIOSH A Rules)*	0.12 to 0.40	0.27 to 0.85	0.46
Modified CRS (NIOSH B Rules)	0.11 to 0.29	0.20 to 0.35	0.25

*Under AIA rules, only fibers having a diameter less than 3 μm are counted and fibers attached to particles larger than 3 μm are not counted. NIOSH A Rules are otherwise similar to the AIA rules.

The B Rules have also been favorably received by analysts as less ambiguous and simpler to use; these rules also showed the least bias relative to AIA rules in the collaborative study. An independent NIOSH laboratory study using amosite fibers reported a relative standard deviation, including within- and between-sample

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variability, of 0.157 for the B Rules [12]. Adding an estimated sampling pump error, s_r, of 0.05 [13] to the within-sample variability in this study results in an estimate of overall precision, s_r, of 0.102 for the B Rules.

4. Because of past inaccuracies associated with low fiber counts, the minimum loading has been increased to 100 fibers/mm^2 filter area (80 fibers total count). This level yields an overall $s_r = 0.13$, as indicated in Figure 3 (revised) of P&CAM 239 [3,4] which corresponds to a measurement $s_r = 0.12$ after removal of pump error [13]. Similarly, at the maximum count of 100 fibers, overall $s_r = 0.115$ and measurement $s_r = 0.10$ are obtained.

D. Evaluation of the method using the A and B counting rules will proceed on a continuing basis through the NIOSH Proficiency Analytical Testing (PAT) Program. The new PAT reporting form allows for reporting of results by either set of rules as of January, 1984.

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METHOD REVISED BY: James W. Carter, David G. Taylor, Ph.D., CIH, and Paul A. Baron, Ph.D., NIOSH/DPSE; based on the revised Method P&CAM 239 [1,3,4].

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