

DEFINITION: Total aerosol mass

NUISANCE DUST, TOTAL

METHOD: 0500

ISSUED: 2/15/84

OSHA: 15 mg/m³

NIOSH: no standard

ACGIH: 10 mg/m³, total dust less than
1% quartz

PROPERTIES: quartz less than 1% [1]

SYNONYMS: boron oxide (CAS #1303-86-2) and nuisance dusts [1] including alumina
(CAS #1344-28-1), calcium carbonate (CAS #1317-65-3), cellulose (paper fiber;
CAS #9004-34-6), glycerin mist (CAS #56-81-5), limestone (CAS #1317-65-3), etc.

SAMPLING	MEASUREMENT
SAMPLER: FILTER (tared 37-mm, 5-µm PVC filter)	! TECHNIQUE: GRAVIMETRIC (FILTER WEIGHT)
FLOW RATE: 1.5 to 2 L/min	! ANALYTE: airborne particulate material
VOL-MIN: 25 L @ 15 mg/m ³ -MAX: 133 L @ 15 mg/m ³	! BALANCE: 0.01 mg sensitivity or better; use same ! balance before and after sample ! collection
SHIPMENT: routine	! CALIBRATION: National Bureau of Standards ! Class M weights
SAMPLE STABILITY: indefinitely	! RANGE: 0.3 to 2 mg per sample
BLANKS: 2 field blanks per 10 samples	! ESTIMATED LOD: 0.2 mg per sample
BULK SAMPLE: none required	! PRECISION: 0.08 mg per sample [3]
ACCURACY	!
RANGE STUDIED: 8 to 28 mg/m ³	!
BIAS: not significant	!
OVERALL PRECISION (s _p): 0.056 [2]	!
APPLICABILITY: The working range is 3 to 20 mg/m ³ for a 100-L air sample. This method is nonspecific and determines the total dust concentration to which a worker is exposed. It may be applied, e.g., to gravimetric determination of fibrous glass [4] in addition to the other ACGIH nuisance dusts [1].	
INTERFERENCES: Organic and volatile particulate matter may be removed by dry ashing [4].	
OTHER METHODS: This method is similar to the criteria document method for fibrous glass [4] and Method 5000 for carbon black. This method replaces Method 5349 [5]. Impingers and direct-reading instruments may be used to collect total dust samples, but these have limitations for personal sampling.	

2/15/84

0500-1

DDA 115813
CONFIDENTIAL

EQUIPMENT:

1. Environmental chamber at constant temperature and humidity (e.g., $20\text{ }^{\circ}\text{C} \pm 0.3\text{ }^{\circ}\text{C}$ and $50\% \pm 5\% \text{ RH}$).
2. Sampler: 37-mm PVC, 2- to 5- μm pore size membrane or equivalent hydrophobic filter and cellulose supporting pad in 37-mm cassette filter holder.
3. Personal sampling pump, 1.5 to 2 L/min, with flexible connecting tubing.
4. Microbalance, capable of weighing to 0.01 mg.
5. Vacuum desiccator.
6. Static neutralizer: e.g., Po-210; replace nine months after the production date.

SPECIAL PRECAUTIONS: None.

PREPARATION OF FILTERS BEFORE SAMPLING:

1. Dry filters and backup pads under vacuum in the vacuum desiccator for at least 15 min.
2. Release the vacuum, remove the desiccator cover and equilibrate the filters in the environmental chamber for at least 1 hr.
3. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
4. Weigh the filters in the environmental chamber. Record the filter tare weight, W_1 (mg).
 - a. Zero the balance before each weighing.
 - b. Handle the filter with forceps (nylon forceps if further analyses will be done).
 - c. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
5. Place the weighed filters on top of the backup pads in the filter cassette bottom sections and allow to stand an additional 8 to 16 hrs in the environmental chamber.
6. Reweigh the filters. If this tare weight differs by more than 0.01 mg from the first tare weight obtained in step 4 above, discard the filter.

NOTE: Insert a rod through the outlet hole of the filter cassette bottom section to raise the backup pad and filter so that the filter can be grasped with forceps.
7. Assemble the filter in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry and mark with the same number as the backup pad.

SAMPLING:

8. Calibrate each personal sampling pump with a representative sampler in line.
9. Sample at 1.5 to 2 L/min. Do not exceed a total filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

10. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
11. Remove the top and bottom plugs from the filter cassette. Place the filter cassettes in a vacuum desiccator under vacuum for at least 15 min, followed by equilibration for at least 1 hr in the environmental chamber.
12. Remove the cassette band, pry open the cassette and remove the filter. Handle the filters very gently by the edge to avoid loss of dust.

NOTE: If the filter sticks to the underside of the cassette top, very gently lift away by using the dull side of a scalpel blade. This must be done carefully or the filter will tear.

CALIBRATION AND QUALITY CONTROL:

13. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Maintain and calibrate the balance with National Bureau of Standards Class M weights.
14. Take two to four replicate samples for every batch of field samples for quality assurance on the sampling procedures. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [6] or in the field. The quality control samples must be taken with the same equipment, procedures and personnel used in the routine field samples. The relative standard deviation calculated from these replicates should be recorded on control charts and action taken when the precision is out of control.

MEASUREMENT:

15. Weigh each filter, including field blanks. Record this post-sampling weight, W_2 (mg), beside its corresponding tare weight. Record anything remarkable about a filter (e.g., overload, leakage, wet, torn, etc.).

CALCULATIONS:

16. Calculate the concentration of total nuisance dust, C (mg/m^3), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) + B}{V} \cdot 10^3, \text{ mg}/\text{m}^3$$

where: W_1 = tare weight of filter before sampling (mg)

W_2 = post-sampling weight of sample-containing filter (mg)

B = mean change in field blank filter weights between tare and post-sampling (mg) (+ or -).

EVALUATION OF METHOD:

Lab testing with blank filters and generated atmospheres of carbon black was done at 8 to 28 mg/m^3 [2,6]. Precision and accuracy data are given on page 0500-1.

REFERENCES:

- [1] TLVs - Threshold Limit Values for 1983-84, Appendix D, ACGIH, Cincinnati, OH (1983).
- [2] This Manual, Method 5000.
- [3] Unpublished data from Non-textile Cotton Study, NIOSH/DRDS/EIB.
- [4] NIOSH Criteria for a Recommended Standard ... Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152, 119-142 (1977).
- [5] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [6] Documentation of the NIOSH Validation Tests, S262 and S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).

METHOD WRITTEN BY: Kathy Morring, Jerry Clere, and Frank Hearl, P.E., NIOSH/DRDS.

FORMULA: various

FIBERS

M.W.: various

METHOD: 7400

ISSUED: 2/15/84

REVISION #2: 8/15/87

OSHA: 0.2 asbestos fibers ($> 5 \mu\text{m}$ long)/mL [1]

PROPERTIES: solid,

NIOSH: 0.1 asbestos f/mL [1]; 3 glass fibers ($>10 \mu\text{m} \times <3.5 \mu\text{m}$)/mL [3]

fibrous

ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, f/mL

SYNONYMS: actinolite asbestos [CAS #13768-00-8], grunerite asbestos (amosite) [CAS #12172-73-5], anthophyllite asbestos [CAS #17068-78-9], chrysotile asbestos [CAS #12001-29-5], crocidolite asbestos [CAS #12001-28-4], tremolite asbestos [CAS #14567-73-8]; fibrous glass.

SAMPLING	MEASUREMENT
SAMPLER: FILTER (0.8- to 1.2- μm cellulose ester membrane, 25-mm diameter; conductive cowl on cassette)	! TECHNIQUE: LIGHT MICROSCOPY, PHASE CONTRAST ! ANALYTE: fibers (manual count) ! SAMPLE PREPARATION: acetone/triacetin "hot block" method [5]
FLOW RATE*: 0.5 to 16 L/min (see step 4)	! COUNTING RULES: Set A (required by OSHA; [1,4]) or Set B (modified CRS [6])
VOL-MIN*: 400 L @ 0.1 fiber/mL (see step 4) -MAX*: (see step 4) *Adjust for 100 to 1300 fibers/mm ² (step 4)	! EQUIPMENT: 1. positive phase-contrast microscope 2. Walton-Beckett graticule (100- μm field of view): A Rules use Type G-22; B Rules use Type G-24 3. phase-shift test slide (HSE/NPL)
SHIPMENT: routine (securely packed to reduce shock)	! CALIBRATION: HSE/NPL test slide
SAMPLE STABILITY: stable	! RANGE: 100 to 1300 fibers/mm ² filter area
FIELD BLANKS: 10% (≥ 2) of samples	! ESTIMATED LOD: 7 fibers/mm ² filter area
ACCURACY	! PRECISION: 0.10 to 0.12 (A Rules) [3] (see Evaluation of Method: B)
RANGE STUDIED: 80 to 100 fibers counted	! OVERALL PRECISION (s_p): 0.115 to 0.13 (A Rules) [3]
BIAS: see EVALUATION OF METHOD	

APPLICABILITY: The method gives an index of airborne fibers in workplace atmospheres. Phase contrast microscopy will not differentiate between asbestos and other fibers; use this method in conjunction with electron microscopy (e.g., Method 7402) for positive identification.

Fibers $< \text{ca. } 0.25 \mu\text{m}$ 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 increase the detection limit.

OTHER METHODS: This method introduces changes for improved sensitivity and reproducibility. It replaces P&CAM 239 [4,8] and Method 7400, Revision #1 (dated 5/15/85).

REAGENTS:

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

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically-conductive extension cowl and cellulose ester filter, 0.8- to 1.2- μ m pore size, and backup pad.

NOTE 1: Analyze representative filters for fiber background before use. Discard the filter lot if mean is ≥ 5 fibers per 100 graticule fields. These are defined as laboratory blanks.

NOTE 2: Use an electrically-conductive extension cowl to reduce electrostatic effects. Ground the cowl when possible during sampling.

2. Personal sampling pump, 0.5 to 16 L/min (see step 4 for flow rate), with flexible connecting tubing.
3. Microscope, positive 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, frosted-end, pre-cleaned, 25 x 75 mm.
5. Cover slips, 22 x 22 mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
6. Lacquer or nail polish.
7. Knife, #10 surgical steel, curved blade.
8. Tweezers.
9. Heated aluminum block for clearing filters on glass slides (see ref. [5] for instructions on manufacture).
10. Micropipets, 5- μ L and 100- to 500- μ L.
11. Graticule, Walton-Beckett type with 100- μ m diameter circular field (area = 0.00785 mm²) at the specimen plane (Type G-22 for A Rules; Type G-24 for B Rules). Available from PTR Optics Ltd., 145 Newton Street, Waltham, MA 02154 [phone (617) 891-6000] and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].

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 APPENDIX A).

12. HSE/NPL phase contrast test slide, Mark II. Available from PTR Optics Ltd. (address above).
13. Telescope, ocular phase-ring centering.
14. Stage micrometer (0.01-mm divisions).
15. Wire, multi-stranded, 22-gauge.

SPECIAL PRECAUTIONS: Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL 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.

U.S. A. 11544P
CONFIDENTIAL

REAGENTS:

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

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically-conductive extension cowl and cellulose ester filter, 0.8- to 1.2- μ m pore size, and backup pad.
NOTE 1: Analyze representative filters for fiber background before use. Discard the filter lot if mean is ≥ 5 fibers per 100 graticule fields. These are defined as laboratory blanks.
NOTE 2: Use an electrically-conductive extension cowl to reduce electrostatic effects. Ground the cowl when possible during sampling.
2. Personal sampling pump, 0.5 to 16 L/min (see step 4 for flow rate), with flexible connecting tubing.
3. Microscope, positive 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, frosted-end, pre-cleaned, 25 x 75 mm.
5. Cover slips, 22 x 22 mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
6. Lacquer or nail polish.
7. Knife, #10 surgical steel, curved blade.
8. Tweezers.
9. Heated aluminum block for clearing filters on glass slides (see ref. [5] for instructions on manufacture).
10. Micropipets, 5- μ L and 100- to 500- μ L.
11. Graticule, Walton-Beckett type with 100- μ m diameter circular field (area = 0.00785 mm²) at the specimen plane (Type G-22 for A Rules; Type G-24 for B Rules). Available from PTR Optics Ltd., 145 Newton Street, Waltham, MA 02154 [phone (617) 891-6000] and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].
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 APPENDIX A).
12. HSE/MPL phase contrast test slide, Mark II. Available from PTR Optics Ltd. (address above).
13. Telescope, ocular phase-ring centering.
14. Stage micrometer (0.01-mm divisions).
15. Wire, multi-stranded, 22-gauge.

SPECIAL PRECAUTIONS: Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL 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.

2. For personal sampling, fasten sampler to the worker's lapel near the worker's mouth. Remove top cover from cowl extension (open face) and orient face down. Wrap joint between cowl and monitor body with shrink tape to prevent air leaks.
NOTE: If possible, ground the cassette to remove any surface charge, using a wire held in contact (e.g., with a hose clamp) with the conductive cowl and a non-electrical metal fixture, or a cold-water pipe.
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove top covers from the field blank cassettes and store top covers and cassettes in a clean area (bag or box) with the top covers from the sampling cassettes during the sampling period. Replace the top covers in the cassettes after sampling.
4. Sample at 0.5 L/min or greater [9]. Adjust sampling flow rate, Q (L/min), and time, t (min), to produce a fiber density, E , of 100 to 1300 fibers/mm² ($3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area $A_c = 385$ mm²) for optimum accuracy. These variables are related to the action level (one-half the current standard), L (fibers/mL), of the fibrous aerosol being sampled by:

$$t = \frac{(A_c)(E)}{(Q)(L)10^3}$$

NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for 8 hrs is appropriate in non-dusty atmospheres containing ca. 0.1 fiber/mL. Dusty atmospheres require smaller sample volumes (<400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/mL, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If $\geq 50\%$ of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

NOTE 2: OSHA regulations specify a maximum sampling rate of 2.5 L/min [1].

5. At the end of sampling, replace top cover and small end caps.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.

NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

NOTE: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index ≤ 1.46 . This method collapses the filter for easier focusing and produces permanent mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" technique may be used outside the laboratory [5]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400 - revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [2,4,8,22]). A videotape of the mounting procedure is available from the NIOSH Publication Office [20].

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca. 70 °C [5].

NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.

9. Mount 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. Place wedge, dust side up, on slide.
NOTE: Static electricity will usually keep the wedge on the slide.
 - b. Insert slide with wedge into the receiving slot at base of "hot block". Place tip of a micropipet containing ca. 250 μ L acetone into the inlet port of the PTFE cap on top of the "hot block". Inject the acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding pipet firmly in place. After waiting 3 to 5 sec for the filter to clear, remove pipet and slide from their ports.
CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous, frequent use of this device in an unventilated space may produce explosive acetone vapor concentrations.
 - c. Using the 5- μ L micropipet, immediately place 3.0 to 3.5 μ L triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation.
NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.
 - d. Glue the edges of the cover slip to the slide using lacquer or nail polish [10]. Counting may proceed immediately after clearing and mounting are completed.
NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature 50 °C) for up to 15 min to hasten clearing. Heat carefully to prevent gas bubble formation.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturers instructions. At least once daily use the telescope ocular supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of microscope cleanings, adjustments, and calibrations.
 - a. Each time a sample is examined, do the following:
 - (1) Adjust the light source for even illumination across the field of view at the condenser iris. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contract optics.
NOTE: Use Köhler illumination if available.
 - (2) Focus on the particulate material to be examined.
 - (3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.
 - b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:
 - (1) Center the MSE/NPL phase-contrast test slide under the phase objective.
 - (2) Bring the blocks of grooved lines into focus in the graticule area.
NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible when observing them in the center of the graticule area. Blocks 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 resolution either too low or too high for fiber counting.
 - (3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Document the laboratory's precision for each counter for replicate fiber counts.
 - a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and PAT samples. The Quality Assurance Officer should maintain custody of the reference slides and should supply each counter with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.
 - b. From blind repeat counts on reference slides, estimate the laboratory intra- and intercounter s_p (see step 21). Obtain separate values of relative standard deviation for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, 21 to 50 fibers in 100 graticule fields, 51 to 100 fibers in 100 graticule fields, and 100 fibers in less than 100 graticule fields. Maintain control charts for each of these data files.

NOTE 1: Since fiber counting is the measurement of randomly placed fibers which may be described by a Poisson distribution, a square root transformation of the fiber count data will result in approximately normally distributed data.

NOTE 2: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [6]
12. Prepare and count field blanks along with the field samples. Report counts on each field blank.

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

NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.
13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the difference between the two counts exceeds $2.77(X)s_p$, where X = average of the two fiber counts and s_p = intracounter relative standard deviation from step 11.

NOTE: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.
14. Enroll each new counter in a training course which compares performance of counters on a variety of samples using this procedure.

NOTE: All laboratories engaged in asbestos counting should participate in a proficiency testing program such as the AIHA-NIOSH Proficiency Analytical Testing (PAT) Program and routinely exchange field samples with other laboratories to compare performance of counters.

MEASUREMENT:

15. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.
16. Adjust the microscope (Step 10) [7].

NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 μ m).
17. Select one of the following sets of counting rules:

NOTE: The two sets of rules have produced approximately equivalent mean counts on a variety of asbestos sample types [6]. OSHA regulations require the use of the A rules [1]. In either case, the rules must be strictly followed to obtain valid results. No hybridizing of the two sets of rules is permitted.

- a. A Rules (same as P&CAM 239 rules [2,4,8]; see APPENDIX B).
 1. Count only fibers longer than 5 μm . Measure 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:
 - 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, provided that the fiber meets the criteria of rules a.1. and a.2.
 - c. Do not count any fiber which crosses the graticule boundary more than once.
 - d. Reject and do not count all other fibers.
 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 graticule fields regardless of count.
- b. B Rules (see APPENDIX B)
 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. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of 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. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3 μm in diameter.
 5. Count 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 graticule fields. Stop at 100 graticule fields, regardless of count.
 7. Divide total end count by 2 to yield fiber count.
18. Start counting from the tip of the filter and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.

NOTE 1: When counting a graticule 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. A minimum counting time of 15 seconds per field is appropriate for accurate counting.

NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters > 1 μm), polarized light microscopy (e.g., NIOSH Method 7403) may be used to identify and eliminate interfering non-crystalline fibers.

CALCULATIONS AND REPORTING OF RESULTS:

19. Calculate and report fiber density on the filter, E (fibers/mm²), by dividing the total fiber count per graticule field, F/n_f , minus the mean field blank count per graticule field, B/n_b , by the graticule field area, A_f (0.00785 mm² for a properly calibrated Walton-Beckett graticule):

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

NOTE: Fiber counts above 1300 fibers/mm² and fiber counts from samples with > 50% of filter area covered with particulate should be reported as "uncountable" or "probably biased."

20. Calculate and report 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 mm² 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.

21. Report intralaboratory and interlaboratory relative standard deviations (from Step 11) with each set of results.

NOTE: Precision depends on the total number of fibers counted [4,11]. Relative standard deviation (also called coefficient of variation) is documented in references [4,11,12,13] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Fig. 1).

EVALUATION OF METHOD:

- A. This method is a revision of P&CAM 239 [2,4,8]. A summary of the revisions is as follows:

1. Sampling:

The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m³ 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 a function of flow rate in the range 0.5 to 16 L/min [9].

2. Sample Preparation Technique:

The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [2,4,5,8,14]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.

3. Measurement:

- The Walton-Beckett graticule standardizes the area observed [14,15].
- The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [7,14].

DO NOT WRITE
IN THESE SPACES

- c. An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [6]. The modified CRS (NIOSH B) Rules were found to yield equivalent counts but were more precise than the AIA (NIOSH A)* Rules. The relative standard deviations (s_r) varied with sample type and laboratory. The ranges 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.

- d. 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 variability, of 0.16 for the B Rules [16]. Another NIOSH study was conducted using field samples of asbestos [19]. This study indicated intralaboratory s_r in the range 0.17 to 0.25 and an interlaboratory s_r of 0.45. This agrees well with other recent studies [6,11,13].
- e. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/ mm^2 filter area (80 fibers total count). This level should yield intracounter s_r in the range of 0.13 to 0.17 [4,8,16,19].
- B. Interlaboratory Comparability:
- At this time, there is no independent method for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory s_r .

Theoretically, the process of counting randomly (Poisson) distributed fibers on a filter surface will give an s_r that depends on the number, N , of fibers counted:

$$s_r = 1/(N)^{1/2} \quad (1)$$

Thus s_r is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual s_r found in a number of studies is greater than these theoretical numbers [6,11,12,13].

An additional component of variability comes primarily from subjective laboratory-to-laboratory differences. In a study of ten counters in a continuing sample exchange program, Ogden [11] found this subjective component of intralaboratory s_r to be approximately 0.2 and estimated the overall s_r by the term:

$$\frac{(N + (0.2 \cdot N)^2)^{1/2}}{N} \quad (2)$$

NIOSH AIA Rules
COUNTED DATA

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were $+2 s_r$ and $-1.5 s_r$. In this program, one sample out of ten was a quality control sample. For laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association [13] also found that the variability had both a constant component and one that depended on the fiber count. These results gave a subjective interlaboratory component of s_r (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [19]. This value falls slightly above the range of s_r (0.25 to 0.42 for 1984-85) found for 80 reference laboratories in the NIOSH Proficiency Analytical Testing (PAT) program for laboratory-generated samples [12].

A number of factors influence s_r for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is chosen as 0.45. Note that, though based on at least two studies, this is a somewhat arbitrary choice. It is hoped that by the use of this number in the absence of other information, laboratories will carry out the recommended interlaboratory quality assurance programs to improve their performance and thus reduce the s_r .

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence interval on the mean count from a single sample fiber count (Figure 1). These curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results [11].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter, 0.00785 mm^2 counting field area). If this same sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

Note that the s_r of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory s_r is smaller, then it is more correct to use that smaller s_r . However, the estimated s_r of 0.45 is to be used in the absence of such information. Note also that it has been found that s_r can be higher for certain types of samples, such as asbestos cement.

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/mL standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/mL standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/mL (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/mL; i.e., $0.16 + 2.13 \times 0.16 = 0.5$.

CONFIDENTIAL

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and -49% as the upper and lower confidence values of the mean for a 100-fiber count.

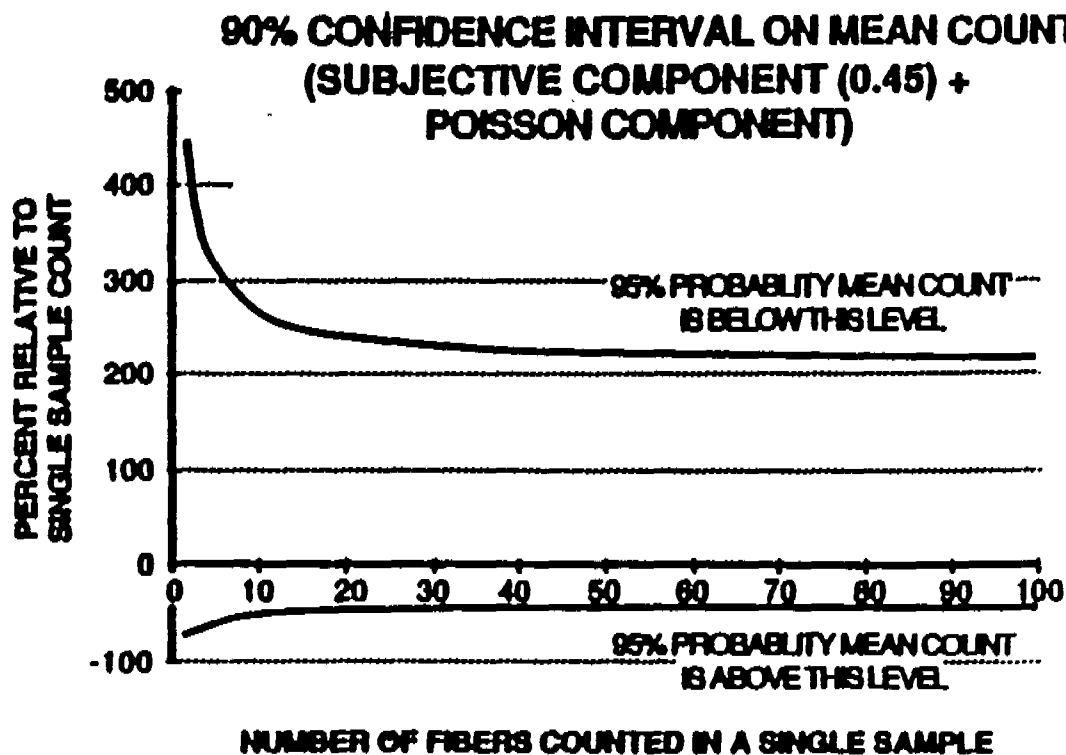


Figure 1. Interlaboratory Precision of Fiber Counts

REFERENCES:

- [1] Occupational Safety and Health Administration, U.S. Department of Labor, Occupational Exposure to Asbestos, Tremolite, Anthophyllite, and Actinolite Asbestos; Final Rules, 29 CFR Part 1910.1001 Amended June 20, 1986.
- [2] Revised Recommended Asbestos Standard, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-169 (1976).
- [3] Criteria for a Recommended Standard...Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152 (1977).
- [4] Leidel, M. A., S. G. Bayer, R. D. Zumwalde, and K. A. Busch. USPHS/NIOSH Membrane Filter Method for Evaluating Airborne Asbestos Fibers, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-127 (1979).
- [5] Baron, P. A. and G. C. Pickford. "An Asbestos Sample Filter Clearing Procedure," *Appl. Ind. Hyg.*, 1:169-171, 199 (1986).
- [6] Crawford, M. P., H. L. Thorpe, and M. Alexander. "A Comparison of the Effects of Different Counting Rules and Aspect Ratios on the Level and Reproducibility of Asbestos Fiber Counts," Part I: Effects on Level (Report No. TM/82/23), Part II: Effects on Reproducibility (Report No. TM/82/24), Institute of Occupational Medicine, Edinburgh, Scotland (December, 1982).

DDA 111651
CONFIDENTIAL

- [7] Rooker, S. J., M. P. Vaughn, and J. M. LeGuen. "On the Visibility of Fibers by Phase Contrast Microscopy," Amer. Ind. Hyg. Assoc. J., **43**, 505-515 (1982).
- [8] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 1., P&CAM 239, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [9] Johnston, A. M., A. D. Jones, and J. H. Vincent. "The Influence of External Aerodynamic Factors on the Measurement of the Airborne Concentration of Asbestos Fibers by the Membrane Filter Method," Ann. Occup. Hyg., **25**, 309-316 (1982).
- [10] Asbestos International Association, AIA Health and Safety Recommended Technical Method #1 (RTMI). "Airborne Asbestos Fiber Concentrations at Workplaces by Light Microscopy" (Membrane Filter Method), London (1979).
- [11] Ogden, T. L. "The Reproducibility of Fiber Counts," Health and Safety Executive Research Paper 18 (1982).
- [12] Schlecht, P. C. and S. A. Schulman. "Performance of Asbestos Fiber Counting Laboratories in the NIOSH Proficiency Analytical Testing (PAT) Program," Am. Ind. Hyg. Assoc. J., **47**, 259-266 (1986).
- [13] "A Study of the Empirical Precision of Airborne Asbestos Concentration Measurements in the Workplace by the Membrane Filter Method," Asbestos Information Association, Air Monitoring Committee Report, Arlington, VA (June, 1983).
- [14] Chatfield, E. J. Measurement of Asbestos Fiber Concentrations in Workplace Atmospheres, Royal Commission on Matters of Health and Safety Arising from the Use of Asbestos in Ontario, Study No. 9, 180 Dundas Street West, 22nd Floor, Toronto, Ontario, CANADA M5G 1Z8.
- [15] Walton, W. H. "The Nature, Hazards, and Assessment of Occupational Exposure to Airborne Asbestos Dust: A Review," Ann. Occup. Hyg., **25**, 115-247 (1982).
- [16] Taylor, D. G., P. A. Baron, S. A. Shulman and J. W. Carter. "Identification and Counting of Asbestos Fibers," Am. Ind. Hyg. Assoc. J. **45**(2), 84-88 (1984).
- [17] Busch, K. A. and D. G. Taylor. "Statistical Protocol for the NIOSH Validation Tests," Chemical Hazards in the Workplace, Measurement and Control, ACS Symposium Series 149, American Chemical Society, Washington, DC (1981).
- [18] Groff, Jensen. NIOSH PAT Coordinator, Private communication.
- [19] Baron, P. A. and S. Shulman. "Evaluation of the Magiscan Image Analyzer for Asbestos Fiber Counting," Am. Ind. Hyg. Assoc. J., (in press).
- [20] Sinclair, R. C. "Filter Mounting Procedure," NIOSH Publication Videotape No. 194 (1984 [updated 1986]).
- [21] Keith, L. H., W. Crummett, J. Deegan, Jr., R. A. Libby, J. K. Taylor, and G. Wentler. "Principles of Environmental Analysis," Anal. Chem., **55**:2210-2218 (1983).
- [22] Jankovic, J. T., W. Jones, and J. Clere. "Field Techniques for Clearing Cellulose Ester Filters Used in Asbestos Sampling," Appl. Ind. Hyg., **1**:145-147 (1986).

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 [2,4,8].

APPENDIX A: CALIBRATION OF THE WALTON-BECKETT GRATICULE:

Before ordering the Walton-Beckett graticule, the following calibration must be done to obtain a counting area (D) 100 μm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.

3. Install the 40 to 45X phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule, L_0 (μm), using the stage micrometer.
6. 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.
7. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule:

$$d_c = \frac{L_a}{L_0} \times D.$$

Example: If $L_0 = 112 \mu\text{m}$, $L_a = 4.5 \text{ mm}$ and $D = 100 \mu\text{m}$, then $d_c = 4.02 \text{ mm}$.

8. Check the field diameter, D (acceptable range $100 \mu\text{m} \pm 2 \mu\text{m}$) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range $0.00785 \text{ mm}^2 \pm 0.00032 \text{ mm}^2$).

APPENDIX B: COMPARISON OF COUNTING RULES:

Figure 2 shows a Walton-Beckett graticule as seen through the microscope. Although the graticule incorporates the 3:1 aspect ratio, both the "A" and "B" rules will be discussed as they apply to the labeled fibers in the figure.

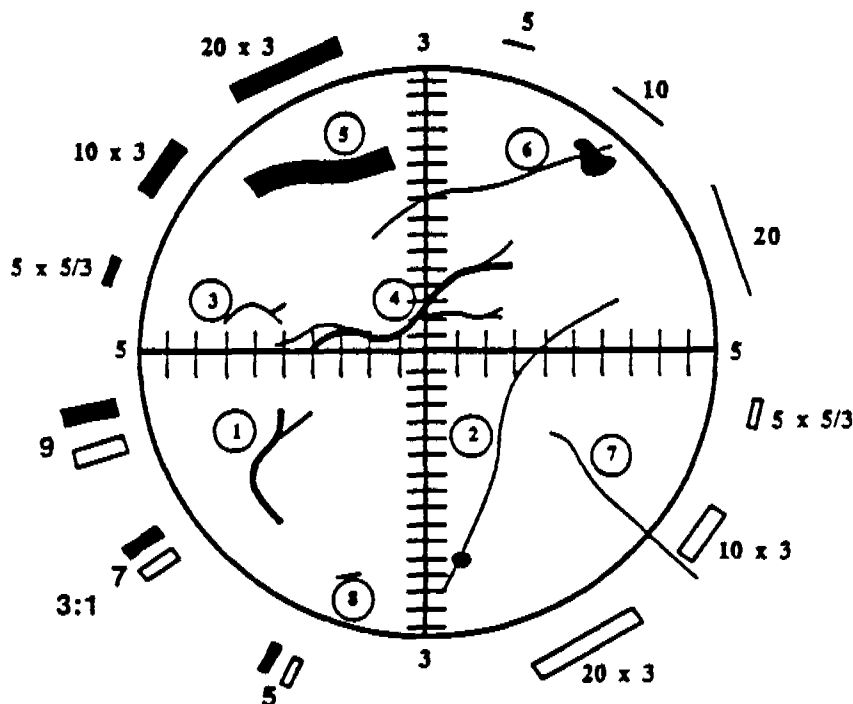


Figure 2. Walton-Beckett graticule with fibers.

CONFIDENTIAL

Fiber	FIBER COUNT		DISCUSSION
	A Rules	B Rules	
1	1 fiber	3 ends	(A) "A" rules do not allow for split ends; therefore, count one fiber. (B) Under 'B' rules, first determine whether the fiber meets dimensional criteria, (i.e., $>5\text{ }\mu\text{m}$, $>5:1$ aspect ratio, $<3\text{ }\mu\text{m}$ diameter). Next determine and count which two ends are the main trunk of the fiber. Finally, count all split ends $>5\text{ }\mu\text{m}$ as one end. Fiber #1 is counted as 3 ends.
2	1 fiber	2 ends	(A) Single fiber with small particle attached. The particle is treated as if it does not exist by the "A" rules. (B) The particle is $<3\text{ }\mu\text{m}$ diameter and therefore ignored under "B" rules.
3	1 fiber	2 ends	(A) As with Fiber 1, count one fiber under "A" rules because it meets the $>3:1$ aspect ratio, $>5\text{ }\mu\text{m}$ criteria. (B) The split end is $<5\text{ }\mu\text{m}$ long so it is not counted under "B" rules.
4	1 fiber	5 ends	(A) Fiber ends all attached to a central large fiber or bundle; therefore, count one fiber under "A" rules. (B) Count two ends as belonging to the main fiber. Three of the remaining four split ends are $>5\text{ }\mu\text{m}$, giving a total of 5 ends.
5	1 fiber	Do not count	(A) No diameter limit under "A" rules; therefore count this thick fiber because it meets the $>3:1$, $>5\text{ }\mu\text{m}$ counting criteria. (B) The fiber is $>3\text{ }\mu\text{m}$ diameter; therefore not counted under "B" rules.
6	1 fiber	1 end	(A) Ignore non-fibrous particulate matter under the "A" rules; count this as a whole fiber. (B) The short end of the fiber is $<5\text{ }\mu\text{m}$ long and obscured by a particle $>3\text{ }\mu\text{m}$ in diameter; therefore, not counted under "B" rules.
7	1/2 fiber	1 end	(A) Fibers which meet rules a.1. and a.2. and cross the graticule boundary are counted as 1/2 fiber under "A" rules unless the fiber crosses the graticule boundary more than once, in which case the fiber is not counted no matter how many ends lie within the graticule area. (B) Fiber ends lying inside the graticule boundary are counted as one end provided that the entire fiber meets rules b.1. and b.2. and each end is $>5\text{ }\mu\text{m}$. The portion of the fiber lying outside the graticule boundary must be considered in order to make this determination. Under "B" rules, it does not matter how often the fiber crosses the graticule boundary.
8	Do not count	Do not count	The fiber is $<5\text{ }\mu\text{m}$ long.

DO NOT WRITE
CONFIDENTIAL