Designation: D 6057 – 96 (Reapproved 2001)ε1

Standard Test Method for Determining Concentration of Airborne Single-Crystal Ceramic Whiskers in the Workplace Environment by Phase Contrast Microscopy[1]

This standard is issued under the fixed designation D 6057; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Apparatus supplier name in Footnote 6 was deleted editorially in November 2001.

1. Scope

1.1 This test method covers the sampling methods and analysis techniques used to assess the airborne concentration of single-crystal ceramic whiskers (SCCW), such as silicon carbide and silicon nitride, which may occur in and around the workplace where these materials are manufactured, processed, transported, or used. This test method is based on the collection of fibers by filtration of a known quantity of air through a filter. The filter is subsequently evaluated with a phase contrast microscope (PCM) for the number of fibers meeting appropriately selected counting criteria. This test method cannot distinguish among different types of fibers. This test method may be appropriate for other man-made mineral fibers (MMMF).

1.2 This test method is applicable to the quantitation of fibers on a collection filter that are greater than 5 μm in length, less than 3 μm in width, and have an aspect ratio equal to or greater than 5:1. The data are directly convertible to a statement of concentration per unit volume of air sampled. This test method is limited by the diameter of the fibers visible by PCM (typically greater than 0.25 μm in width) and the amount and type of coincident interference particles.

1.3 A more definitive analysis may be necessary to confirm the identity and dimensions of the fibers located with the PCM, especially where other fiber types may be present. Such techniques may include scanning electron microscopy (SEM) or transmission electron microscopy (TEM). The use of these test methods for the identification and size determination of SCCW is described in Practice D 6058 and Test Methods D 6059 and D 6056.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[3]

D 4532 Test Method for Respirable Dust in Workplace Atmospheres[3]

D 6056 Test Method for Determining Concentration of Airborne Single-Crystal Ceramic Whiskers in the Workplace Environment by Transmission Electron Microscopy[3]

D 6058 Practice for Determining Concentration of Airborne Single-Crystal Ceramic Whiskers in the Workplace Environment[3]

D 6059 Test Method for Determining Concentration of Airborne Single-Crystal Ceramic Whiskers in the Workplace Environment by Scanning Electron Microscopy[3]

E 691 Practice for Conducting Interlaboratory Study to Determine the Precision of a Test Method[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *analytical sensitivity, n*—airborne fiber concentration represented by a single fiber counted in the PCM.

3.1.1.1 *Discussion*—Although the terms *fiber* and *whisker* are, for convenience, used interchangeably in this test method, whisker is correctly applied only to single-crystal fibers whereas a fiber may be single- or poly-crystalline or may be noncrystalline.

[1] This test method is under the jurisdiction of ASTM Committee D22 on Sampling and Analysis and is the direct responsibility of Subcommittee D22.04 on Analysis of Workplace Atmospheres.

Current edition approved December 10, 1996. Published February 1997.

[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

3.1.2 *aspect ratio, n*—the ratio of the length of a fiber to its width.

3.1.3 *fiber, n—for the purpose of this test method*, an elongated particle having a length greater than 5 µm, a width less than 3 µm, and an aspect ratio equal to or greater than 5:1.

3.1.4 *man-made mineral fiber, n*—any inorganic fibrous material produced by chemical or physical processes.

3.1.5 *single-crystal ceramic whisker, n*— a man-made mineral fiber that has a single-crystal structure.

3.2 For definitions of other terms used in this test method, see Terminology D 1356.

4. Summary of Test Method

4.1 The sample is collected on a mixed cellulose ester (MCE) filter by drawing air, using a sampling pump, through an open-face 25-mm electrically conductive sampling cassette assembly **(1,2)**.[5] A section of the opaque filter is converted into an optically transparent homogeneous specimen using an acetone vaporizer. The fibers are counted by PCM at a magnification of approximately 400× using the criteria discussed in Section 11. Results are expressed as a fiber concentration per unit volume of air and a fiber loading per unit area of filter. The airborne concentration is expressed as fibers per millilitre (f/mL) and the fiber loading is expressed as fibers per square millimetre (f/mm^2).

5. Significance and Use

5.1 The SCCW may be present in the workplace atmosphere where these materials are manufactured, processed, transported, or used. This test method can be used to monitor airborne concentrations of fibers in these environments. It may be employed as part of a personal or area monitoring strategy.

5.2 This test method is based on dimensional considerations only. As such, it does not provide a positive identification of the fibers counted. Analysis by SEM or TEM is required when additional fiber identification information is needed.

NOTE 1—This test method assumes that the analyst is familiar with the operation of PCM instrumentation and the interpretation of data obtained using this technique.

5.3 This test method is not appropriate for measurement of fibers with diameters less than approximately 0.25 µm due to visibility limitations associated with PCM. The SEM or TEM methods may be used to provide additional size information of SCCW if needed (refer to Practice D 6058 for additional information on the use of these methods).

5.4 Results from the use of this test method shall be reported along with 95 % confidence limits for the samples being studied. Individual laboratories shall determine their intralaboratory coefficient of variation and use it for reporting 95 % confidence limits **(1,3,4)**.

6. Interferences

6.1 All fibers meeting the dimensional criteria in Section 3 are not necessarily of the same composition. Since the PCM method does not differentiate based on chemistry or morphology, all fibers in accordance with the definitions in Section 3 shall be counted.

6.1.1 This test method has been designed to filter air for the determination of fiber concentration. However, filtration of air also involves collection of extraneous particles. Extraneous particles may obscure fibers by overlay or by discoloration of the filter. This situation can be managed by regulating the air volume sampled and thus the filter loading. Fibers should appear separated from other particles to ensure an adequate opportunity for their recognition as separate entities in the PCM and accurate counting. Some coincident particulate agglomeration does occur even with these guidelines. Analyze an alternate filter with a reduced loading if the obscuring condition appears to exceed 15 % of the filter area **(5)**. Redeposition of a portion of an overloaded filter is permitted only in circumstances where an alternate filter is not available and cannot be obtained through resampling (see 10.1.9).

7. Apparatus and Reagents

7.1 *Sampling Cassette*—Use a 25-mm, electrically conductive cassette assembly such as a three-piece cassette with an extension cowl or retainer ring, or both, containing a 0.45-µm pore size MCE filter and a support pad. Seal the cassette assembly with shrink tape. Reloading of used cassettes is not permitted.

7.2 *Personal Sampling Pump*—Use a portable battery-operated pump for personal sampling. Each pump must be capable of operating within the range from 0.5 to 4 L/min and continuously over the chosen sampling period **(1)**. The flow must be free from pulsation. All pumps shall be calibrated prior to use **(6)**.

7.3 *Area Sampling Pump*—Use a personal sampling pump or a non-portable high-volume pump for area sampling. Each pump shall be capable of operating within the range from 0.5 to 16 L/min and continuously over the chosen sampling period **(1)**. The flow shall be free from pulsation. All pumps shall be calibrated prior to use **(6)**.

7.4 *Vinyl Tubing*, or equivalent.

7.5 *Microscope*—Positive phase contrast light, with green or blue filter, 8 to 10× eyepiece, and 40 to 45× phase objective (total magnification approximately 400×); numerical aperture = 0.65 to 0.75.

7.6 *Acetone Vaporizer*—A device used to clear the MCE filter by exposure to a small amount of vaporized acetone.

7.7 *Graticule*, with standardized 100-µm diameter circular field at the specimen plane (calibrated area $\approx 7.8 \times 10^{-3}$ mm^2), with the capability to compare diameters and lengths at 3 and 5 µm, respectively, within the field of view.

NOTE 2—The graticule is custom-made for each microscope. Specify disk diameter needed to exactly fit the ocular of the microscope and the diameter (millimetres) of the circular counting area (see section 12.2.1). The Walton-Beckett Type G-24 graticule or other equivalent graticules are recommended. Graticules designed for the NIOSH 7400 A rules, such as the Walton-Beckett Type G-22, are not recommended.

NOTE 3—In some microscopes, adjustments of the interocular distance will change the tube length and hence magnification of the microscope. Each analyst shall separately measure the diameter of his or her field of view and this value shall be used in all calculations.

[5] The boldface numbers in parentheses refer to a list of references at the end of this test method.

7.8 *Phase Shift Test Slide equivalent to HSE/NPL.*[6]

7.9 *Telescope*, (ocular phase-ring centering) or Bertrand lens.

7.10 *Stage Micrometer*, (0.01-mm divisions).

7.11 *Tweezers*.

7.12 *Scalpel Blades*.

7.13 *MCE Filters*, 25 mm, 0.45 µm and 0.22 µm.

7.14 *Funnel/Filter Assembly*, 25 mm.

7.15 *Triacetin* (glycerol triacetate).

7.16 *Acetone*.

NOTE 4—**Precaution:** Acetone is a flammable liquid and requires precaution not to ignite it accidentally.

7.17 *ASTM D1193 Type II Water* (particle free).

7.18 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8. Sample Collection

8.1 Collect samples of airborne SCCW on MCE filters using sampling cassettes and pumps in accordance with Section 7.

8.2 Remove the outlet plug from the sampling cassette and connect it to a sampling pump by means of flexible, constriction-proof tubing.

8.3 Perform a leak check of the sampling system by activating the pump with the closed cassette and rotameter (or other flow measurement device) in line. Any flow indicates a leak that must be eliminated before starting the sampling operation.

8.4 Remove the inlet plug from the sampling cassette to eliminate any vacuum that may have accumulated during the leak test; then remove the entire inlet cap.

8.5 Conduct personal and area sampling as follows:

8.5.1 For personal sampling, fasten the sampling cassette to the worker's lapel in the worker's breathing zone and orient it face down. Adjust the calibrated flow rate to a value between 0.5 and 4 L/min **(1)**. Typically, a sampling rate between 0.5 and 2.5 L/min is selected **(2-5,7)**. Also see Test Method D 4532.

8.5.2 Place area samples on an extension rod facing down at a 45° angle. Adjust the calibrated flow rate to a value between 0.5 and 16 L/min **(1)**. Typically, a sampling rate between 1 and 10 L/min is selected **(8)**.

8.5.3 Set the sampling flow rate and time to produce an optimum fiber loading between 100 and 1300 f/mm^2 **(1,2)**. The time of sampling can be estimated by using the following equation:

$$t = \frac{(A_c)\,(F_L)}{(Q)\,(C_e)\,10^3} \quad (1)$$

where:

A_c = active filter collection area (~385 mm^2 for 25-mm filter),[8]
t = time, min,
F_L = fiber loading, f/mm^2,
Q = sampling flow rate, L/min,
C_e = estimated concentration of SCCW, f/mL, and
10^3 = conversion factor.

NOTE 5—While the desired minimum loading is 100 f/mm^2, the minimum loading that has statistical significance is 7 f/mm^2 after blank correction **(1)**.

NOTE 6—Experience has shown that the fiber loading should not exceed 1300 f/mm ^{2}(12 fibers/graticule area, average value for all counted fields) for the majority of sampling situations **(1)**.

8.5.4 At a minimum, check the flow rate before and after sampling. If the difference is greater than 10 % from the initial flow rate, the sample shall be rejected. Also see Test Method D 4532.

8.6 Carefully remove the cassette from the tubing at the end of the sampling period (ensure that the cassette is positioned upright before interrupting the pump flow). Replace the inlet cap and inlet and outlet plugs, and store the cassette.

NOTE 7—Deactivate the sampling pump prior to disconnecting the cassette from the tubing.

8.7 Submit at least one field blank (or a number equal to 10 % of the total samples, whichever is greater) for each set of samples. Remove the cap of the field blank briefly (approximately 30 s) at the sampling site, then replace it. The field blank is used to monitor field sampling procedures. Field blanks shall be representative of filters used in sample collection (for example, same filter lot number).

8.8 Submit at least one unused and unopened sealed blank which is used to monitor the supplies purchased as well as procedures used in the laboratory. The sealed blank shall be representative of filters used in sample collection (for example, same filter lot number).

9. Transport of Samples

9.1 Ship the samples in a rigid container with sufficient packing material to prevent jostling or damage. Care shall be taken to minimize vibrations and cassette movement.

NOTE 8—Do not use shipping material that may develop electrostatic forces or generate dust.

NOTE 9—Shipping containers for 25-mm sampling cassettes are commercially available and their use is recommended.

9.2 Include in the container a list of samples, their descriptions, and all other pertinent information.

10. Specimen Preparation

10.1 The objective of the specimen preparation technique 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

[6] The HSE/NPL Phase Shift Test Slide, Mark II.

[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[8] The active collection area (A_c) should be measured periodically, especially if different types of cassettes are used.

method noted as follows collapses the filter for easier focusing and produces permanent mounts that may be retained for quality control and interlaboratory comparison. Other mounting techniques meeting the preceding criteria may also be used (for example, the nonpermanent field mounting technique used in Physical and Chemical Analysis Method P&CAM 239 and the dimethyl formamide (DMF)/Euparal method **(1,3,4,7,9)**).

10.1.1 Wipe the exterior of the sampling cassettes with a damp cloth to minimize the possibility of contamination.

10.1.2 Perform specimen preparation in a clean area.

10.1.3 Ensure that the glass slides and cover slips are free of dust and fibers by wiping with a clean lens tissue.

10.1.4 Carefully cut a wedge of the filter area (for example, 25 %) with a curved, steel surgical blade using a rocking motion to prevent tearing.

NOTE 10—Use care not to disturb the particles on the filter surface.

10.1.5 Place the filter wedge, particle side up, on a clean glass slide.

10.1.6 Insert the slide in the acetone vaporizer centering the filter wedge under the vapor delivery spout. Inject acetone in accordance with the manufacturer's instructions to clear the filter. Remove the slide from the vaporizer.

NOTE 11—Use a minimum amount of acetone for this application. For most vaporizers, a nominal amount between 100 to 250 µL is appropriate for each slide.

10.1.7 Using a separate 5 or 10-µL syringe, place ~3 µL of triacetin on the filter. Gently lower a clean cover slip onto the filter at a slight angle to reduce the possibility of forming bubbles.

NOTE 12—If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excess triacetin remains in contact with the edge of the filter under the cover slip, fiber migration may occur at the edges.

NOTE 13—If clearing is slow, a conventional slide warmer may be used to hasten clearing. Counting may proceed immediately after clearing and mounting are completed.

10.1.8 Glue the edges of the cover slip to the glass slide using a lacquer or nail polish if retention of the slide is necessary **(10)**.

10.1.9 *Indirect Sample Preparation*—Resuspension of particulate matter collected on an overloaded filter and subsequent filtering onto another substrate may result in loss or breakup of the sample materials. Therefore, redeposition is permitted only in circumstances where an alternate filter is not available and cannot be obtained through resampling (for example, evaluation of a prototype procedure where the operational parameters cannot be duplicated). If indirect sample preparation procedures are employed, it must be clearly noted in the report. Furthermore, it must be clearly stated that results were obtained from the use of indirect sample preparation techniques and used only as an estimate of SCCW concentrations in the workplace environment. The following procedures are appropriate for this purpose.

10.1.9.1 Carefully cut a wedge (for example, one half or one fourth of the area of the original filter) as accurately as possible from the filter with a curved, steel surgical blade using a rocking motion to prevent tearing.

NOTE 14—Use care not to disturb the particles on the filter surface.

NOTE 15—The size of the wedge will depend on filter loading. If the sample is very heavily loaded, then a smaller wedge (for example, one eighth or one sixteenth of the area of the original filter) may be more appropriate.

10.1.9.2 Place the section of filter into a 100-mL beaker.

10.1.9.3 Add approximately 80 mL of filtered ASTM Type II distilled water to the beaker.

10.1.9.4 Place the beaker into the ultrasonic bath. Sonicate for approximately 1 min.

10.1.9.5 Remove the section of filter and rinse it using filtered distilled water. The rinse shall be collected in the 100-mL beaker. Add enough distilled water to result in a 100-mL suspension.

10.1.9.6 Filter the suspension using a funnel through a 25-mm, 0.22-µm MCE filter using vacuum filtration techniques. Rinse the interior of the beaker into the funnel using filtered distilled water.

NOTE 16—It is recommended that disposable funnels be used to reduce the potential for contamination.

NOTE 17—Use of a 47-mm funnel/filter assembly is permissible provided the active filter area is accounted for in the calculations provided in 13.1.2.

NOTE 18—If the resuspended filter is too heavily loaded with particles to permit analysis, then re-prepare the sample using a smaller portion of the original filter as discussed in 10.1.9.1.

NOTE 19—The MCE filters used for redeposition shall have an average blank level less than 7 f/mm^2.

10.1.9.7 Remove the funnel from the vacuum system. Place the deposited filter in a desiccator for approximately 2 h to remove moisture.

10.1.9.8 Cut a wedge of the filter (for example, 25 %) with a curved, steel surgical blade and continue to follow the procedures outlined in 10.1.5-10.1.8.

NOTE 20—Account for the area of the filter used in the resuspension process in the equations provided in 13.1.2 when calculating the estimated airborne concentration. For example, if 25 % of the original filter area was redeposited onto a 25-mm filter, then a dilution factor of 4 is used in the calculations.

11. Analysis Method

11.1 The objective of this method is to determine the concentration of fibers per cubic millilitre of air sampled based on the number of fibers observed during the PCM analysis.

11.1.1 Regularly check microscope calibration as described in Section 12.

11.1.2 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 filter using a magnification of approximately 400×.

11.1.3 *Fiber Counting Rules* **(1,7)**:

11.1.3.1 Perform fiber counting by starting at one end of the filter wedge (far enough away from the edge to ensure that no artifacts from cutting the filter are encountered) and progressing along a radial line to the other end. At the edge of the filter, the sample can be shifted either up or down to obtain an area that has not been previously examined and the counting process continues in the reverse direction. Fields are selected randomly by looking away from the eyepiece briefly while

advancing the mechanical stage. Alternatively, a microscope equipped with a motorized stage control can be used to select fields arbitrarily.

11.1.3.2 Reject the field and select another when a particle or an agglomerate of particles covers approximately 15 % or more of the field of view (graticule area) **(5)**. Do not report rejected fields in the number of fields counted. However, each rejected field shall be noted on the count sheet.

NOTE 21—Fiber loading should not exceed 12 fibers/graticule area for the average of all counted fields. Reject average fiber loadings exceeding 20 fibers/graticule area.

NOTE 22—When counting a field, continuously scan a range of focal planes by moving the fine focus knob to detect fibers which have become embedded in the filter.

11.1.3.3 Count only the ends of fibers that are greater than 5 µm in length, less than 3 µm in diameter, and have an aspect ratio equal to or greater than 5:1.

11.1.3.4 Count each fiber end that falls within the graticule area as one end, provided that the fiber is in accordance with 11.1.3.3.

11.1.3.5 Count visibly free ends that are in accordance with 11.1.3.3 and 11.1.3.4 when the fiber appears to be attached to another particle, regardless of the size of the other particle.

11.1.3.6 Count the free ends emanating from an agglomeration of fibers up to a maximum of 10 ends (5 fibers), provided that each segment is in accordance with 11.1.3.3 and 11.1.3.4.

NOTE 23—Figure 1 provides examples of fiber for possible fiber-end orientation.

11.1.3.7 Record the fiber counts on a count sheet. Record ND when no fibers are detected in a field.

11.1.3.8 Count enough graticule areas to yield 200 ends (100 fibers). Analyze a minimum of 20 fields. Stop at 100 fields, regardless of the fiber count.

11.1.3.9 Divide the total end count by 2 to yield fiber count.

12. Quality Control

12.1 Monitoring the environment for airborne fibers requires the use of sensitive sampling and analysis procedures. The sensitivity of the analysis may be influenced by a variety of factors. These include the supplies used in the sampling and analysis operation, the performance of the sampling, the preparation of the sample from the filter, and the actual examination of the sample in the microscope. Each of these unit operations must produce a product of defined quality if the analytical method is to produce a reliable and meaningful test result. Accordingly, a series of control checks and reference standards shall be performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems that might develop within the sampling and analysis operations.

12.2 *Instrument Calibration*:

12.2.1 *Graticule Calibration*—The graticule must be able to provide a counting area (*D*) of 100 µm in diameter at the image plane. Perform calibration of the graticule as described as follows:

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

NOTE 24—Specify the diameter, d_c (mm), of the circular counting area and the disk diameter when ordering the graticule.

12.2.1.2 Set the appropriate inter-pupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.

12.2.1.3 Install the 40 to 45× phase objective.

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

12.2.1.5 Measure the magnified grid length of the graticule, L_o (µm), using the stage micrometre.

12.2.1.6 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.

12.2.1.7 Calculate the circle diameter, d_c (mm), for the graticule:

$$d_c = \frac{L_a}{L_o} D \tag{2}$$

12.2.1.8 *Example*—If L_o = 108 µm, L_a = 2.93 mm, and D = 100 then d_c = 2.71 mm. Check the field diameter, D (acceptable range 100 ± 2 µm) with a stage micrometre upon receipt of the graticule from the manufacturer. Determine field area (acceptable range from 7.54×10^{-3} to 8.17×10^{-3} mm ^{2}). This area is to be used in all calculations.

NOTE 25—Calibrated graticules are not meant to be interchangeable between microscopes and this shall not be attempted under any circumstances in which the field area changes more than ±2 %.

12.2.2 *Microscope Calibration*—Conform to the manufacturer's instructions and also the following:

12.2.2.1 Focus on the particulate material to be examined.

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

NOTE 26—Kohler illumination is preferred.

12.2.2.3 Ensure the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.

12.2.2.4 Use the telescope ocular supplied by the manufacturer or Bertrand lens to ensure that the phase rings (annular diaphragm and phase shifting elements) are concentric.

12.2.3 Check the phase shift detection limit of the microscope periodically **(1,11)**.

12.2.3.1 Place the phase-shift test slide under the phase objective and bring the sets of grooved lines into focus.

NOTE 27—Calibration with the phase-shift test slide determines the minimum detectable fiber diameter (approximately 0.25 µm). The slide consists of seven sets of grooves (approximately 20 grooves to each set) in descending order of visibility from Sets 1 to 7. The requirement for fiber counting is 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 depending upon microscope quality and resolution. A microscope which fails to meet these requirements has either too low or too high a resolution to be used for counting.

12.2.3.2 Clean the microscope optics if the image quality deteriorates. Consult with instrument manufacturer if the problem persists.

12.3 *Blank Analyses*:

12.3.1 Analyze the field and sealed blanks before the field samples. If any of the field or sealed blank samples exhibit a fiber count greater than 7 fibers per 100 graticule areas, the entire sampling and analytical procedure shall be examined carefully to locate and correct any source of the contamination **(1)**.

12.3.2 Report the counts on each blank. Calculate the mean of the blank counts and subtract this value from each sample count before reporting the results.

12.3.3 Maintain as part of the laboratory quality assurance program a set of reference samples **(12)**. These samples shall consist of filter preparations including a range of loadings and background SCCW levels from a variety of sources including in-house or other laboratory field samples. The quality assurance officer shall maintain custody of the reference samples and shall supply each analyst with reference samples on a routine basis as part of the laboratory's quality assurance program. The labels on the reference samples shall be changed periodically so that an analyst does not become too familiar with the samples.

12.3.4 Estimate the laboratory intra- and inter-counter relative standard deviation from blind repeat counts expressed as fiber loading (f/mm^2) on reference slides. Obtain separate values of relative standard deviation for each sample matrix analyzed in each of the following ranges: from 5 to 20 fibers in 100 graticule fields, from >20 to 50 fibers in 100 graticule fields, from >50 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 **(1,12)**. Calculate S_R as one half of the pooled, intra-counter relative standard deviation **(12)**.

NOTE 28—The intra-counter relative standard deviation (S_R) shall be less than 0.20 for a laboratory to be considered proficient in the test method **(1,3,4)**.

12.4 *Replicate Analyses*:

12.4.1 The primary method for assessing the precision of an individual analyst is through the use of replicate analyses. A replicate analysis is a repeat analysis of the same sample, performed by the same analyst under the same analytical conditions as the original analysis.

12.4.2 Perform blind recounts by the same analyst on 10 % of filters counted using slides relabeled by a person other than the analyst.

12.4.3 Document the laboratory's precision for each analyst for replicate fiber counts.

12.4.4 The conformance expectation for the replicate analysis is that the fiber loading (f/mm^2) from the original and the replicate analyses will fall within the following control limits:

$$|\sqrt{A_1} - \sqrt{A_2}| \le 2.77 \left(\frac{\sqrt{A_1} + \sqrt{A_2}}{2} \right) S_R \quad (3)$$

where:

A_1 = original estimate of fiber loading,

A_2 = replicate estimate of fiber loading, and

S_R = one half of the pooled, intra-counter relative standard deviation **(12)**.

Control limits are established from historical data. If the original and the replicate estimate fall outside the acceptance range, the sample is reexamined to determine the cause of the count variation. If the reexamination shows the analyst may be in error due to questionable ability, the analyst may not be permitted to examine unknown samples, but must recount five reference samples. Upon acceptable performance of the analyses, the analyst may again examine unknown samples, but the frequency of replicate analyses is increased to one in every five samples for the next 100 samples, or until such replicate analyses meet the conformance expectations.

12.4.5 If the analyst fails the replicate test, all samples in the sample set shall be recounted and the new counts compared with the original count. All rejected counts shall be discarded and the samples reanalyzed.

12.5 Each new analyst shall be instructed in the operation of the instrumentation discussed in this test method.

NOTE 29—To ensure good reproducibility, all laboratories engaged in fiber counting should routinely participate with other laboratories in the exchange of field samples to compare the performance of the analysts **(13)** (also refer to Practice E 691 for guidelines).

12.6 Appropriate logs or records must be maintained by the analytical laboratory verifying that it is in compliance with the quality assurance procedures.

13. Calculations

13.1 The following information must be reported for each SCCW sample analyzed by PCM: number of fibers counted (*N*), area analyzed (mm^2), volume of air sampled in litres (*L*), fiber loading (f/mm^2), airborne fiber concentration (f/mL), and analytical sensitivity (f/mL). The calculations used to obtain fiber loading and airborne fiber concentration are as follows:

13.1.1 *Calculation for Direct Preparation*:

13.1.1.1 *Fiber Counts (N)*:

$$N = \frac{\text{number fiber ends counted}}{2} \quad (4)$$

13.1.1.2 *Fiber Loading (F_L)*:

$$F_L = \left(\frac{N}{\text{area analyzed}} \right)_{\text{SCCW Sample}} - \left(\frac{N}{\text{area analyzed}} \right)_{\text{Blank}} \quad (5)$$

13.1.1.3 *Airborne Fiber Concentration (C)*:

$$C = F_L \times \frac{\text{active filter area}}{\text{volume of air sampled } (L) \times 10^3} \quad (6)$$

13.1.1.4 *Analytical Sensitivity (A_s)*:

$$A_s = \frac{1 \text{ fiber}}{\text{area analyzed}} \times \frac{\text{active filter area}}{\text{volume of air sampled } (L) \times 10^3} \quad (7)$$

13.1.2 *Calculations for Indirect Preparation*:

13.1.2.1 *Fiber Counts (N)*:

$$N = \frac{\text{number fiber ends counted}}{2} \quad (8)$$

13.1.2.2 *Fiber Loading (F_L)*:

$$\left(\frac{N}{\text{area analyzed}} \times \text{active filter area} \times \text{dilution} \right)_{\text{redeposited area}} \quad (9)$$

$$F_L = \frac{-\left(\frac{N}{\text{area analyzed}} \times \text{active filter area}\right)_{\text{blank}}}{\text{active filter area of collection filter}}$$

13.1.2.3 *Airborne Fiber Concentration (C)*:

$$\left(\frac{N}{\text{area analyzed}} \times \text{active filter area} \times \text{dilution}\right)_{\text{redeposited filter}} \quad (10)$$

$$C = \frac{-\left(\frac{N}{\text{area analyzed}} \times \text{active filter area}\right)_{\text{blank}}}{\text{volume of air sampled } (L) \times 10^{3}}$$

13.1.2.4 *Analytical Sensitivity (A_s)*:

$$A_s = \frac{1 \text{ fiber}}{\text{area analyzed}} \quad (11)$$

$$\times \frac{\text{active redeposited filter area}}{\text{volume of air sampled } (L) \times 10^{3}} \times \text{dilution factor}$$

14. Precision and Bias

14.1 The precision of the procedure described in this test method for measuring the concentration of single-crystal ceramic whiskers is being determined.

14.2 Since there is no accepted reference material suitable for determining the bias using the procedure described in this test method for measuring the concentration of single-crystal ceramic whiskers, bias has not been determined.

15. Keywords

15.1 air monitoring; phase contrast microscopy; sampling and analysis; silicon carbide whiskers; single crystal ceramic whiskers; workplace environment

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