Designation: D6058 – 96 (Reapproved 2016)

Standard Practice for Determining Concentration of Airborne Single-Crystal Ceramic Whiskers in the Workplace Environment[1]

This standard is issued under the fixed designation D6058; 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.

1. Scope

1.1 This practice is intended to assist individuals in the sampling and analysis of single-crystal ceramic whiskers (SCCW), such as silicon carbide and silicon nitride, in the workplace environment. It describes sampling and analytical techniques used to assess the airborne concentration and size distribution of SCCW, which may occur in and around the workplace where these materials are manufactured, processed, transported, or used.

1.2 The protocols currently in use for asbestos and other fibrous materials have been used as a guide in developing sampling and analytical procedures for characterizing fibers produced from the manufacture and use of SCCW. The sampling and analysis protocols described here have been written specifically for SCCW, however, they may be appropriate for other man-made mineral fibers (MMMF).

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

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D6056 Test Method for Determining Concentration of Airborne Single-Crystal Ceramic Whiskers in the Workplace Environment by Transmission Electron Microscopy

D6057 Test Method for Determining Concentration of Airborne Single-Crystal Ceramic Whiskers in the Workplace Environment by Phase Contrast Microscopy

D6059 Test Method for Determining Concentration of Airborne Single-Crystal Ceramic Whiskers in the Workplace Environment by Scanning Electron Microscopy

3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology D1356.

3.2 *Definitions:*

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

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

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

4. Summary of Practice

4.1 This practice is based on a three-tier approach to the quantitative assessment of airborne SCCW levels. It includes detailed procedures to analyze standard air sampling cassettes

[1] This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.07 on Sampling and Analysis of Asbestos.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

by phase contrast microscopy (PCM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

4.2 The choice of a particular analytical method shall be based on the visibility limitation of each instrument and an understanding of the actual size distribution of the fibers being analyzed.

4.3 In general, PCM is suitable for the analysis of fibers that are greater than approximately 0.25 µm in diameter. Depending on the instrument and the sample preparation method used, the SEM may be capable of examining fibers as small as 0.10 µm in diameter. TEM has been shown to be suitable for the study of even finer fibers. The high resolution of this instrument makes it well suited for the determination of the fraction of a fiber population with diameters ≤0.10 to 0.25 µm.

4.4 In addition to an enhanced image resolution, SEM and TEM have the further advantage of providing elemental composition information on a single fiber. Furthermore, TEM may also be used to ascertain crystallographic data on the fiber. This additional information is frequently helpful in the analysis of samples which contain numerous unknown fibers and, thus, SEM or TEM, or both, are preferred in such instances.

5. Significance and Use

5.1 The SCCW may be present in the workplace atmosphere where these materials are manufactured, processed, transported, or used. The test methods discussed in this practice can be used to provide guidance when monitoring airborne concentrations of SCCW in these environments.

5.2 Because of their visibility limitations, a significant fraction of the very small thin fibers that are present in some samples may not be detected by PCM or SEM. Therefore, TEM is considered to be the reference technique for the analysis of airborne SCCW. The TEM must be used to determine both fiber count and morphology when samples are from previously uncharacterized workplaces or materials.

5.3 Although TEM is the reference technique, PCM or SEM are considered to be the primary screening methods for the analysis of airborne SCCW.

5.4 Parallel TEM measurements shall be carried out, at least initially, to provide an index or relative measure of the fraction of total fibers that are seen by PCM or SEM. Only in instances when this percentage has been shown to be at a high and reproducible level may the lower resolution techniques (that is, PCM or SEM) be relied on exclusively.

6. Evaluating Potential Methods

6.1 The following three test methods address the determination of SCCW concentrations in airborne samples. Each has its own particular scope of application and varies from the other in the type of information provided. Thus, all are relevant in different situations, and the choice of which test method is used will depend on the primary objective of the monitoring program.

6.1.1 *Test Method D6057:*

6.1.1.1 Phase contrast microscopy is the analysis method required by the Occupational Safety and Health Administration (OSHA) for the monitoring of airborne asbestos in the workplace. The asbestos permissible exposure limit and action level are based on this technique. The test method which is discussed in this practice, although closely related to the asbestos method, differs in that the counting rules recommended for SCCW are those described in NIOSH 7400 B.[3] In contrast, for asbestos the A Counting Rules are typically followed. Under the NIOSH 7400 A Counting Rules, fibers with aspect ratios ≥3:1 are counted. The B Rules count fibers with aspect ratios ≥5:1. The B Rules further place an upper limit on fiber diameter of 3 µm. The B Rules were selected to monitor SCCW based on the nature of SCCWs which are not likely to split longitudinally as are asbestos fibers. While asbestos fibers of low aspect ratio, are, in reality, bundles of finer fibrils which may split longitudinally into high aspect ratio fibrils, the SCCW do not have this structure and thus would not be expected to split into higher aspect ratio fibers.

6.1.1.2 In practice, a portion of the membrane filter containing the airborne particles is placed on a glass slide and rendered transparent by exposure to acetone vapor. The slide is transferred to a phase contrast microscope and examined at a magnification of approximately 400×. Fibers fitting the counting rules definition are counted if they lie within a measured area. The B Rules require that fiber ends be counted and that this number then be divided by two to give the fiber count. From this fiber count, and knowing the volume of air sampled, it is possible to calculate the fiber concentration in the air that was sampled. This number is generally expressed in terms of fibers per millilitre (f/mL) of air.

6.1.1.3 The PCM method only counts fibers that fit within the dimensional constraints of the counting rules. Thus, the lower limit of length to be counted will be 5 µm and the maximum diameter counted will be 3 µm. The lower limit of diameter is determined by the resolution and contrast (visibility) of the microscope which is approximately 0.25 µm.

6.1.1.4 The PCM method is also restricted to counting fibers of all types; the method does not identify or differentiate between different fiber types. In consequence, the PCM method is applicable to measurement of those populations in which SCCW is the only, or the prevalent, fiber type present. The test method is rapid, inexpensive and may be readily performed on-site. It is therefore a useful screening tool for monitoring workplace environmental levels of fibers or potential worker exposure to fibers. However, one must bear in mind that this approach is inherently limited to the examination of fibers greater than approximately 0.25 µm in diameter, depending on the difference between the refractive index of the immersion medium and the fibers.

6.1.2 *Test Method D6059:*

6.1.2.1 The SEM may be used when a more definitive estimate of airborne concentration of SCCW is required. The technique covers the size range covered by PCM and may provide information on thinner fibers down to approximately 0.1 µm in diameter. Unlike PCM, however, the technique is

[3] Baron, P., "Fibers, Method 7400 Issue 2-8-15-94," *NIOSH Manual of Analytical Methods*, 4th ed., P. M. Eller, ed., U.S. Department of Health and Human Services, DHHS (NIOSH) Publication No. 93-113, Cincinnati, OH 45226.