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Standard Guide for Tiered Approach to Detection and Characterization of Silver Nanomaterials in Textiles[1]

This standard is issued under the fixed designation E3025; 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 guide covers the use of a tiered approach for detection and characterization of silver nanomaterials in consumer textile products, which can include some medical devices (for example, wound dressings or face masks), made of any combination of natural or manufactured fibers.

1.2 This guide covers, but is not limited to, fabrics and parts (for example, thread, batting) used during the manufacture of textiles and production of consumer textile products that may contain silver-based nanomaterials. It does not apply to analysis of silver nanomaterials in non-consumer textile product matrices nor does it cover thin film silver coatings with only one dimension in the nanoscale.

1.3 This guide is intended to serve as a resource for manufacturers, producers, analysts, policymakers, regulators, and others with an interest in textiles.

1.4 This guide is presented in the specific context of measurement of silver nanomaterials; however, the structured approach described herein is applicable to other nanomaterials in consumer textile products, including some medical devices.

1.5 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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.*

1.7 *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]

D123 Terminology Relating to Textiles

D6413 Test Method for Flame Resistance of Textiles (Vertical Test)

2.2 *AATCC Standards:*[3]

AATCC 135 Dimensional Changes of Fabrics after Home Laundering

2.3 *ISO Standards:*[4]

ISO 10136-1 Glass and Glassware—Analysis of Extract Solutions—Part 1: Determination of Silicon Dioxide by Molecular Absorption Spectrometry

ISO 16140 Microbiology of Food and Animal Feeding Stuffs—Protocol for the Validation of Alternative Methods

ISO/IEC Guide 99 International Vocabulary of Metrology—Basic and General Concepts and Associated Terms (VIM)

ISO/TR 18196 Nanotechnologies—Measurement Technique Matrix for the Characterization of Nano-Objects

ISO/TS 80004-1 Nanotechnologies—Vocabulary—Part 1: Core Terms

2.4 *U.S. Code of Federal Regulations:*[5]

16 CFR Parts 1615 and 1616 Standards for the Flammability of Children's Sleepwear

3. Terminology

3.1 *Definitions*—For additional definitions related to textiles, see Terminology D123; for additional definitions related to nanotechnology, see ISO/TS 80004-1; and for additional definitions related to measurements, see ISO/IEC Guide 99.

[1] This guide is under the jurisdiction of ASTM Committee E56 on Nanotechnology and is the direct responsibility of Subcommittee E56.06 on Nano-Enabled Consumer Products.

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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.

[3] Available from American Association of Textile Chemists and Colorists (AATCC), P.O. Box 12215, Research Triangle Park, NC 27709-2215, http://www.aatcc.org.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[5] Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.access.gpo.gov.

3.1.1 *analyte, n*—element or constituent to be determined. **ISO 10136-1**

3.1.2 *consumer textile product, n*—textile product intended to satisfy human wants and needs. **D123**

3.1.3 *manufactured fiber, n*—class name for various genera of filament, tow, or staple produced from fiber-forming substances that may be: (*1*) polymers synthesized from chemical compound, (*2*) modified or transformed natural polymers, or (*3*) glass. **D123**

3.1.4 *measurand, n*—quantity intended to be measured or a quantity that is being determined by measurement. **ISO/IEC Guide 99**

3.1.5 *nanomaterial, n*—material with any external dimension in the nanoscale or having internal structure or surface structure in the nanoscale. **ISO/TS 80004-1**

3.1.6 *nanoscale, n*—range from approximately 1 to 100 nm. **ISO/TS 80004-1**

3.1.7 *natural fiber, n*—class name for various genera of fibers (including filaments) of (*1*) animal, (*2*) mineral, or (*3*) vegetable origin. **D123**

3.1.8 *qualitative method, n*—method of analysis whose response is either the presence or absence of the analyte detected either directly or indirectly in a certain amount of sample. **ISO 16140**

3.1.9 *quantitative method, n*—method of analysis whose response is the amount of the analyte measured either directly (enumeration in a mass or a volume), or indirectly (colour absorbance, impedance, etc.) in a certain amount of sample. **ISO 16140**

3.1.10 *textile, n*—general term for fibers, yarn intermediates, yarns, fabrics, and products that retain all the strength, flexibility, and other typical properties of the original fibers or filaments. **D123**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *characterization, n*—identification and quantification of one or more relevant physical or chemical property values of the analyte.

3.2.2 *detection, n*—qualitative recognition of the presence of the target analyte in a sample.

3.2.3 *silver, n*—element with atomic number 47 that can be in the form of ions, metallic or zero-valent (Ag^0), alloys, composites, oxide, or salt compounds, or combination thereof.

4. Significance and Use

4.1 Natural and manufactured textiles fibers can be treated with chemicals to provide enhanced antimicrobial (fungi, bacteria, viruses) properties. In some cases, silver nanomaterials may be used to treat textile fibers (**1**).[6] Silver nanomaterials are used to treat a wide array of consumer textile products, including, but not limited to, various clothing; primary garments (shirts, pants), outer wear (gloves, jackets), inner wear (socks and underwear), children's clothing (sleepwear); children's plush toys; bath towels and bedding (sheets, pillows); and medical devices (for example, wound dressings and face masks) (**2**).

4.2 There are many different chemical and physical forms of silver that are used to treat textiles and an overview of this topic is provided in Appendix X1.

4.3 Several applicable techniques for detection and characterization of silver are listed and described in Appendix X2 so that users of this guide may understand the suitability of a particular technique for their specific textile and silver measurement need.

4.4 There are many different reasons to assay for silver nanomaterials in a textile at any point in a product's life cycle. For example, a producer may want to verify that a textile meets their internal quality control specifications or a regulator may want to understand the properties of silver nanomaterials used to make a consumer textile product under their jurisdiction or what quantity of silver nanomaterial is potentially available for release from the treated textile during the washing process or during product use. Regardless of the specific reason, a structured approach to detect and characterize silver nanomaterials present in a textile will facilitate measurements and data comparison. Detection and characterterization of silver in textiles is one component of an overall risk assessment.

4.5 The approach presented in this guide (see Fig. 1) consists of three sequential tiers: obtain a textile sample (Section 7), detection of a silver nanomaterial (Section 8), and characterization of a silver nanomaterial (Section 9). If no forms of silver are detected in a textile sample using appropriate (fit for purpose) analytical techniques then testing can be terminated. If silver is detected, but present in a non-nanoscale form, the textile is a chemical or bulk silver-containing material. Silver ions may be released from silver-containing materials, and under reducing conditions these can transform into nanoscale silver-containing particles. If nanoscale silver is detected, one concludes that the textile contains a silver nanomaterial. Subsequent measurements can characterize the chemical and physical properties of the silver nanomaterial.

4.6 Numerous techniques are available to detect and characterize silver nanomaterials in textiles. The breadth of options can cause confusion for those interested in developing an analytical strategy and selecting appropriate techniques. Some techniques apply only to certain chemical forms of silver and all have limited ranges of applicability with respect to a measurand. No single technique is suitable to both detect and fully characterize silver nanomaterials in textiles. This guide describes and defines a tiered approach using commercially available measurement techniques so that manufacturers, producers, analysts, policymakers, regulators, and others may make informed and appropriate choices in assaying silver nanomaterials in textiles within a standardized framework. The user is cautioned that this guide does not purport to address all conceivable textile analysis scenarios and may not be appropriate for all situations. In all instances, professional judgment is necessary.

[6] The boldface numbers in parentheses refer to a list of references at the end of this standard.

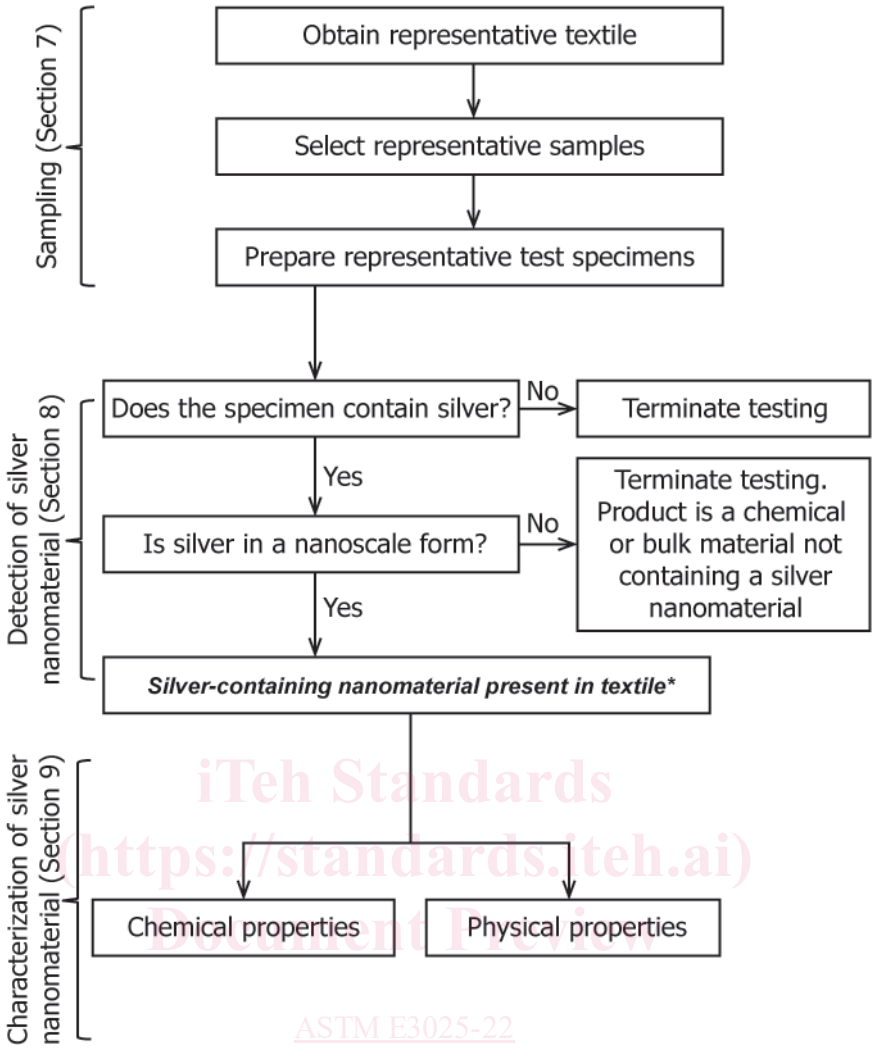

FIG. 1 Tiered Approach for Determining if a Textile Contains a Silver Nanomaterial
(*It might not be possible to know how the nanomaterial formed in the textile. It may have been engineered or intentionally applied or transformed from another silver source.)

4.7 This guide provides a tiered approach to determine an efficacious and efficient procedure for detecting and characterizing silver in textiles and determine whether any silver nanomaterial is present. This tiered approach may also be used to determine whether a reported measurand for silver nanomaterials in a textile was obtained in an appropriate and meaningful way.

4.8 Material property measurement depends on the method. Caution is required when comparing data for the same measurand from techniques that operate on different physical or chemical principles or with different measurement ranges.

4.9 The amount of silver in a textile might decrease over time. Silver metal and silver compounds can react with oxygen and other oxidation-reduction (redox) active agents present in the environment to form soluble silver species. These soluble silver species can be released by contact with moisture (for example, from ambient humidity, washing, body sweat, rain, or other sources). As described in Appendix X1, release of soluble silver species may occur at varying rates. Release rates depend on many characteristics, including chemical nature, surface area, crystallinity, and shape, where the silver is applied to the textile (on the fiber surface, in the volume of the fiber, and so forth), and in what form the silver is applied to the textile (discrete particles, with carriers, and so forth). The condition and age of textile test samples must be considered when drawing temporal inferences from the results, as only a moment in time of the textile life cycle will be captured in the results.

4.10 Textile acquisition, storage, handling, and preparation can also affect silver content.

5. Reagents

5.1 *Purity of Reagents*—Reagent-grade chemicals should be used in all tests. Unless otherwise indicated, it is intended that all reagents should 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.

6. Tiered Approach

6.1 This tiered approach is a cost-effective strategy that uses progressively more specialized instrumentation to elucidate whether a silver nanomaterial is present in a textile and to measure its chemical and physical properties (see Fig. 1) **(3)**.

6.2 Initially, a robust bulk analytical technique is used for qualitative detection of silver regardless of form (for example, ions, Ag^0, AgCl, alloy, composite) or size in a textile specimen.

6.3 If silver is detected in a textile specimen, additional measurements are made to determine if any of it is a silver nanomaterial.

6.4 If a silver nanomaterial is present in a textile, complementary and confirmatory techniques are used to characterize its chemical and physical properties.

7. Sampling

7.1 The first step of the tiered approach is to obtain a textile that represents the life-cycle stage for which measurement is needed using an appropriate sampling strategy (Fig. 1). Depending upon the user's specific measurement need, a textile may be obtained from the fabric-processing stage, finishing treatment stage, finished lot or end-product stage, or any other part of its life cycle. Consider whether to obtain threads, decorative trim, and other components used to assemble a textile product. The sampling plan should be fit for its intended purpose to deliver the required or specified variation for a heterogeneous material.

7.2 Cut the desired number of representative samples from the textile using an appropriate sampling strategy that captures the areas that may contain silver nanomaterials. In the absence of knowledge about the distribution of silver in a textile, a conservative approach assumes that any silver is distributed heterogeneously until proven otherwise. If the distribution of silver in a textile is known or assumed to be heterogeneous, the user should cut samples to capture this variability using some form of random sampling that describes the measurement distribution for their specific needs. A power calculation can be used to estimate the number of samples needed to achieve a desired level of precision for the user's specific needs (**4**).

7.2.1 The locations and dimensions of samples will also depend upon the size of the specific textile article; they may be cut from a portion of a large textile (for example, bed linens, pants) or it may be the entire textile for smaller articles (for example, finger or palm of a glove).

7.3 Cut the desired number of test specimens from each representative sample. If the distribution of silver in a textile sample is known or could be heterogeneous, test specimens should be cut from the samples to capture variability. The locations and dimensions of the test specimens will depend upon the specific sample and will be described in the report.

NOTE 1—If the distribution of silver in a textile is known to be homogeneous, representative samples (and test specimens) can be cut from any location of the article, for example, from different locations across the width of a textile.

7.4 Examples of textile, sample, and test specimen collection practices for processes that span an article's life cycle are described in Test Method D6413, AATCC 135, and 16 CFR Parts 1615 and 1616.

7.5 Textiles, samples, and test specimens should be stored in a manner that will not alter the properties of any silver present and potentially bias the intended data collection objectives. Storage considerations include, but are not limited to, temperature, relative humidity, exposure to direct sunlight, and atmosphere.

8. Detection

8.1 Qualitative determination of silver within prepared test specimens can include indirect or direct measurement methods, or both. Test specimens should be initially measured using a robust bulk analytical technique, preferably with capability for high throughput detection of silver (see Appendix X2 for examples). In this step, silver measurements do not need to be made using a quantitative method and do not need to differentiate between forms.

8.1.1 If no silver is detected, a more sensitive bulk analytical technique [lower method detection limit (MDL)] should be used to determine if silver is present in a test specimen. This confirmatory measurement does not need to be quantitative.

8.1.2 If measurement for silver is below the MDL of the more sensitive technique, the result serves as confirmation that silver is not present in a measureable quantity in the textile specimen. Therefore, all testing should be terminated at this step.

8.2 If silver is qualitatively detected above the MDL of either the initial or confirmatory bulk analytical technique, additional measurements are needed to elucidate the form of silver in the textile specimen. This step is necessary because, as noted in Appendix X1, textiles may be treated with silver materials in forms that range from silver salts and nanoparticles to micrometer scale silver fibers, and the aforementioned elemental detection step is not designed to discriminate between physical forms. As such, by itself, detection of silver is not sufficient to determine whether a textile contains a silver nanomaterial.

8.3 Available techniques to determine whether a silver nanomaterial is present in a textile include ultraviolet-visible (UV-vis) absorbance spectroscopy, electron microscopy, scanning probe microscopy, and single particle inductively coupled plasma-mass spectroscopy (SP-ICP-MS). A summary of these techniques is provided in Appendix X2. The choice of technique might be limited to the type of instrumentation to which a user has access, though any measurement made in 8.1 should be confirmed using a more sensitive technique in 8.2.

[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.

8.4 UV-vis absorbance spectroscopy measures the absorbance signal due to the surface plasmon resonance (SPR) of metallic silver nanomaterials. The absorbance wavelength will shift as particle size increases or if particles agglomerate. This technique is applicable if the metallic silver nanomaterial is on or near the surface of the textile fiber and provides only an indirect indication of the presence of metallic silver nanomaterials. The absence of an UV-Vis absorbance spectroscopy signal cannot be taken as evidence that a silver nanomaterial is not present in a textile. If the metallic silver nanomaterial is encased in the volume of the fibers, it will not exhibit SPR. Further, if silver is present as silver ions or as a silver alloy, oxide, or salt, these also do not exhibit SPR.

8.5 SP-ICP-MS is a firmware and software based modification of the traditional ICP-MS analytical technique. In SP-ICP-MS, the observed steady state signal represents the ionic, dissolved contribution form of the element (here, Ag) and discrete peaks or ion plumes represent individual nanoparticles. Software algorithms identify and separate the two signals and report the ionic, and dissolved element concentrations as well as the nanoparticle size, size distribution, and concentration of particles. The intensity of each discrete peak is proportional to the number of ions detected at the instrument detector as well as to the mass of the particle. This observed mass to charge ratio is converted to particle size and a plot of particle size distribution. SP-ICP-MS is not capable of distinguishing primary particles from aggregates or agglomerates. At present, only liquid samples may be analyzed by commercially available SP-ICP-MS instruments and, as such, textiles must first be prepared for analysis. To determine silver nanomaterials in textiles by SP-ICP-MS, one must extract the nanomaterials from the textile or dissolve the textile fibers in an appropriate solvent that will not alter the nanomaterial. Analysts must prepare dilute liquid suspensions of the nanomaterials to prevent coincidence of multiple particles at the detector simultaneously, which results in positive bias of particle size.

8.6 Among the techniques described in Appendix X2, electron microscopy and scanning probe microscopy provide direct visual confirmation of the form and dimensions of particles, which makes these techniques especially useful for evaluating whether particles are present in nanoscale form. Employing electron and scanning probe microscopy for visualization and measurement of any nanoscale particles present in a textile specimen are of particular use since they can be augmented with chemical detection techniques, such as energy dispersive X-ray analysis or selected area electron diffraction to determine if they actually contain silver. This chemical measurement does not need to be quantitative.

8.7 The type of treatment (ion exchanger, salt, metal) used on a textile is an important consideration when using electron microscopy to determine the form and dimensions of silver present in the detection step of the tiered approach.

8.7.1 With silver ion exchangers, silver is applied to a textile in the form of discrete silver ions (Ag^+) that are distributed within carrier particles such as porous zeolite or glass. Zeolites are alumina silicates that are typically in the micrometer scale (**5**). Electron microscopy augmented with chemical detection techniques is useful to verify if visible particles in a textile specimen produced using an ion exchanger treatment are carriers and not a silver nanomaterial.

8.7.2 Textile treatments with silver salts can include both neat silver salt particles (AgCl, Ag_2SO_4, and so forth) and microcomposites composed of salt particles attached to a carrier material (for example, titanium dioxide). The form of particles visible in textiles produced using silver salts could be neat silver-containing salt particles or silver-containing particles attached to the carrier material, depending upon the treatment application. Chemical detection techniques would be required to differentiate silver-containing particles from carrier material.

8.7.3 Treatment of textiles with elemental or zero-valent silver (Ag^0) can involve a variety of physical forms, which might add complexity to measuring particle dimensions by microscopy. For example, silver nanomaterials can be incorporated into the volume of the fibers themselves. Determining the size of elemental silver particles by electron microscopy may be challenging for textiles in which particles were incorporated into the volume of the fibers during manufacturing. In this situation, an appropriate solvent may be used to dissolve the fibers without affecting the silver particles and would minimize sample preparation artifacts. The exact solvent will depend on the specific type of natural or manufactured fiber. For example, spandex fibers can be dissolved using N,N-dimethylformamide, N,N-dimethylacetamide, or by heat-treating and washing with acetone or ethanol (**6**).

8.8 If silver is found in the textile but is not present in a nanoscale form (for example, ions in a carrier or particulate with all dimensions >100 nm), then it can be considered a chemical or bulk silver-containing material. There still may be potential for release of silver ions that transform into nanoscale silver-containing particles; see discussion in X1.4.

8.9 If a portion of the silver-containing particles have any external dimension in the nanoscale or have internal structure or surface structure in the nanoscale, then one concludes that silver nanomaterial is present in the textile. This conclusion only addresses the dimensional aspect of the silver and does not address any potential or actual nanoscale-specific chemical or physical properties that might exist (see Section 9).

8.10 A textile can contain silver-containing particles in the nanoscale and non-nanoscale form.

9. Characterization

9.1 If a silver nanomaterial is present in a textile, additional measurements can be made to characterize its chemical or physical properties. A summary of several applicable techniques for characterizing the chemical and physical properties of silver nanomaterials is provided in Appendix X2.

9.2 Properties of nanomaterials that may be of interest for characterization include bulk elemental composition, surface composition, speciation, and crystal structure (**7**), and will depend on the user's measurement goals. The Organization for Economic Cooperation and Development (OECD) published a framework in 2019 that the user may wish to refer to when selecting their measurement goals (**8**).

9.2.1 It is important to recognize that there currently is no chemical analytical technique that can quantitatively differentiate between amounts (in the bulk or on the surface), species, or crystal structures of nanoscale and non-nanoscale silver in a textile. Only in the case where all silver in a sample is in the form of a nanomaterial would a measurement correspond only to nanoscale silver. As such, multiple complementary and confirmatory techniques are necessary to characterize the chemical properties of silver in textiles.

9.2.1.1 Quantitative determination of the total amount of silver present in a textile specimen can be assayed using solution-based techniques that require acid-assisted digestion of the textile matrix and silver before analysis or certain direct measurement techniques such as X-ray fluorescence spectroscopy. Depending upon the treatment application (see X1.1) and the life-cycle stage, silver can be present in the textile as nanoparticles or as ions, or both. To avoid bias in determining the silver nanomaterial content, one must separate silver nanomaterials from all other forms of silver (for example, ions) during sample preparation. If necessary, the user will determine the particle size distribution of silver in a range from nanoscale to larger scales. The nano-scale fraction must be measured separately to avoid overstating the silver nanomaterial mass. Acceptable quantitative measurements are characterized by traceability (accuracy based on instrument calibration with known reference material standards or based on first principles), high precision, and knowledge of the uncertainty relating to the result. Appropriate SI units for results are mass of silver (kg)/mass of textile (kg).

9.2.1.2 Surface composition refers to the elemental composition of the surface of a textile sample. Surface analysis is generally method specific. For instance, some surface-sensitive techniques may have deeper penetration than other techniques, so the surface composition shall be given in the context of the method and with an estimate of the surface thickness contributing to the analysis. Appropriate SI units for surface composition are mass of silver (kg)/area of textile (m^2).

9.2.1.3 Speciation refers to the chemical form of silver (for example, zero-valent silver, ionic silver, oxide, sulfide, etc). Several techniques are available for determining speciation of silver. These range from commercially available microscopy based techniques of individual or ensembles of particles such as transmission electron microscopy with electron energy loss spectroscopy or with selected area electron diffraction to highly specialized techniques for determining average speciation distributions, such as X-ray absorption near edge structure spectroscopy, which requires access to a synchrotron to make measurements. Speciation is a qualitative property so no unit can be assigned.

9.2.1.4 Crystal structure may include the measurands crystallite size (size of single crystals, typically microscopic or nanoscopic in dimensions and forming polycrystalline materials when held together by then defective or amorphous layers), crystallinity (relative proportion of crystalline and amorphous material in a sample), crystal form, and physico-chemical structure (amorphous, paracrystalline, or crystalline). Crystallite size has SI units of meter, crystallinity has non-dimensional units, and crystal form and physico-chemical structure are qualitative properties, so no units can be assigned.

9.3 There are numerous physical properties of nanomaterials that may be of interest for characterization, and the specific properties will depend on the user's measurement goals. Among the most commonly cited physical properties for characterization of nanomaterials are size, size distribution, shape, and spatial distribution (**7**). In the context of textiles, spatial distribution refers to where the nanomaterial is located in the fibers (on the surface, in the fiber volume, and so forth).

9.3.1 Direct visualization by electron microscopy or scanning probe microscopy is the most suitable technique for determination of particle physical properties though some indirect techniques such as X-ray scattering and spectroscopy may also provide useful information (as described in Section 8).

9.3.1.1 In the detection phase of the tiered approach, electron microscopy (or another analytical technique) is used to identify the presence of silver nanomaterials in textile specimens. In the characterization phase, the same or other techniques, or both, are used to quantitatively determine the size, particle size distribution, and shape of silver nanomaterials in a textile specimen. As noted previously, quantitative measurements are characterized by traceability (accuracy based on instrument calibration using known reference material standards such as a grating), high precision, and knowledge of the uncertainty relating to the result. The appropriate SI units for particle size are the meter, square meter, or cubic meter, depending on the measurement method. For example, analysis of the size distribution may be limited by the number of particles counted and the ability to differentiate between particles that are touching or overlaid. As a result, size distributions and shape measures may provide only semi-quantitative or qualitative information about the breadth or general shape of the distribution of particles. The appropriate SI unit for particle size distribution will depend on the type of distribution measured (volume, mass, and so forth).

9.3.1.2 Silver nanomaterials occur in a wide variety of shapes such as spheroidal particles, wires, rods, and agglomerates or aggregates that may be in the form of neat particles or attached to microcomposite carrier material. The appropriate SI units for shape will depend on the measurement (length, area, surface area, or volume). Measurements that help to define shape such as length and width of a rod have SI units of meter, area of a plate-like particle or surface area of a 3D shape have SI units of square meter, and the volume of a sphere or pyramid have SI units of cubic meter. Additional descriptors such as aspect ratio (length/width) are non-dimensional, and certain shape descriptors such as "spheroidal" or "pyramidal" are a qualitative property, so no SI unit can be assigned.

9.3.1.3 It may be desirable to describe the spatial distribution of silver in a textile. As noted previously, silver nanomaterials may be incorporated into the volume of fibers during manufacturing. In this situation, some investigators have used thermal treatment in excess of 500 °C to combust the fibers and evaluated the particles remaining in the textile ash using electron microscopy; however, use of heat may cause sintering of neighboring particles to form larger particles. As such, the

obtained particle size information may not be representative of the silver particles *in situ* (9). Alternatively, dissolution of the fiber using an organic solvent or other means (6) may be used to remove the textile matrix prior to inspection by microscopy.

10. Report

10.1 At a minimum, report the following information:

10.1.1 Date and methods by which textile was acquired;

10.1.2 Description of textile (manufacturer, model, fiber type and composition, weight);

10.1.3 Textile storage conditions (light/dark, temperature, humidity, container composition);

10.1.4 Textile storage duration from acquisition to measurement;

10.1.5 Methods by which representative samples and test specimens were acquired;

10.1.6 Description of test specimens (dimensions and mass);

10.1.7 Analytical preparation (including manufacturer, expiration date, lot number, and purity of reagents) and handling procedures;

10.1.8 Date(s) of measurements;

10.1.9 Description of instrument(s)—make and model, software version, etc.;

10.1.10 Date of last calibration of an instrument (using a reference material standard or other means) and the result (for quantitative measurements) or calibration due date;

10.1.11 Number of replicate measurements, appropriate expression of summary statistics (where applicable), and accompanying measurement units (where applicable) for results; and

10.1.12 Description, results and discussion of analytical findings.

11. Keywords

11.1 analytical methods; characterization; detection; nanomaterials; silver; textiles; tiered approach

APPENDIXES

(Nonmandatory Information)

X1. CONTEXT FOR MEASURING SILVER IN TEXTILES

X1.1 Many Different Chemical and Physical Forms of Silver Are Used in Textile Treatments

X1.1.1 Silver can be applied or incorporated into textiles in a variety of chemical forms. When testing textiles for the presence or concentration of silver in a textile, the test method and interpretation of results shall take into account the particular chemical form of the silver material applied to the textile or fibers where known. Where the chemical form is unknown, testing and interpretation shall take into account that any silver detected may be in any of several different chemical forms, each generally falling into one of three broad classes of treatments widely used in textile manufacturing.

X1.1.1.1 *Silver Ion Exchangers Class*—These textile treatments comprise silver zirconium phosphates, silver zeolites, and silver impregnated glass. In these treatments, silver is held in the form of discrete silver ions (Ag^+) distributed within the matrix carrier (that is, zeolite, glass, and so forth). Silver ions (Ag^+) are released from the textile (or the carrier attached to the textile) when the ions come into contact with moisture from body sweat, washing, ambient humidity, and rain or water in the immediate environment.[8]

X1.1.1.2 *Silver Salts Class*—These are textile treatments composed of both neat silver chloride (AgCl) particles and microcomposites composed of AgCl particles attached to titanium dioxide as a carrier material. Silver chloride is relatively insoluble in water; however, like silver textile treatments in the ion exchangers class, silver ions (Ag^+) are released from the AgCl particles when they come into contact with water in various forms as described previously. In principle, commercial silver textile treatments also may be made with other insoluble or slightly soluble, particulate silver compounds, including silver carbonate (Ag_2CO_3), silver citrate ($C_6H_5Ag_3O_7$), silver phosphate (Ag_3PO_4), silver iodide (AgI), silver bromide (AgBr), silver oxide (Ag_2O), and silver sulfate (Ag_2SO_4).

X1.1.1.3 *Silver Metal Class*—These are textile treatments composed of silver metal, that is, not ions or compounds, but in the form of elemental or zero-valent silver (Ag^0). The silver metal may be present in a variety of physical forms, including silver metal filaments (threads), polymer fibers electrolytically coated with a silver layer, or silver metal particles (colloidal silver) applied to the surface of textiles or fibers or incorporated into the volume of the fibers themselves. The textile treatment also may be in the form of microcomposites composed of silver metal particles attached to or embedded in an inert carrier material. Like the other silver textile treatment classes, silver ions (Ag^+) may be released from silver metal particles when they come into contact with water in various forms.

X1.2 Silver Material Particle Size Characterization is Inherently Ambiguous Unless the Measurand is Specified

X1.2.1 Given the variety of chemical and physical forms of commercial silver textile treatments in measuring and reporting the size or size distribution of the silver component of the treatment to avoid ambiguity and improper comparisons, it is necessary to decide and then report whether the aspect being measured is the size of the silver metal nanomaterial, silver ion, or silver compound particle in the treatment or the size of any

[8] The presence of ligands is not required for dissolution of any form of silver (10), though silver species may be formed when released ions come into contact with ligands (for example, thiols in sweat).