Designation: ~~E3171 – 21ε1~~ E3171 – 21a

Standard Test Method for Determination of Total Silver in Textiles by ICP-OES or ICP-MS Analysis[1]

This standard is issued under the fixed designation E3171; 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—Editorial corrections were made throughout in May 2021.~~

1. Scope

1.1 This test method covers the use of inductively coupled plasma–optical emission spectrometry (ICP-OES) and inductively coupled plasma–mass spectrometry (ICP-MS) analyses for determination of the mass fraction of total silver in consumer textile products made of any combination of natural or manufactured fibers. Either ICP-OES or ICP-MS analysis is recommended as a first step to test for and quantify silver in a textile and results can be used to inform subsequent, more detailed analyses as part of the tiered approach described in Guide E3025 to determine if a textile contains silver nanomaterial(s).

1.2 This test method prescribes acid digestion to prepare test sample solutions from samples of textiles utilizing an appropriate internal standard followed by external calibration and analysis with either ICP-OES or ICP-MS to quantify total silver.

1.3 This test method is believed to provide quantitative results for textiles made of fibers of rayon, cotton, polyester, and lycra that contain metallic silver (see Section 17). It is the analyst's responsibility to establish the efficacy (ability to achieve the planned and desired analytical result) of this test method for other textile matrices and forms of silver.

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

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

1.6 *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

D1193 Specification for Reagent Water

[1] This test method 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.

D4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data (Withdrawn 2002)[3]
D6413 Test Method for Flame Resistance of Textiles (Vertical Test)
D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
D7439 Test Method for Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma–Mass Spectrometry
E288 Specification for Laboratory Glass Volumetric Flasks
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method
E694 Specification for Laboratory Glass Volumetric Apparatus
E1613 Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques (Withdrawn 2021)[3]
E2456 Terminology Relating to Nanotechnology
E3025 Guide for Tiered Approach to Detection and Characterization of Silver Nanomaterials in Textiles

2.2 *AATCC Standards:*[4]

AATCC 135 Dimensional Changes of Fabrics after Home Laundering

2.3 *ISO Standards:*[5]

ISO/IEC Guide 99 International vocabulary of metrology – Basic and general concepts and associated terms (VIM)
ISO 17034 General requirements for the competence of reference material producers
ISO 22036 Determination of trace elements in extracts of soil by inductively coupled plasma – atomic emission spectrometry (ICP-AES)
ISO 3585 Glass plant, pipelines and fittings – Properties of borosilicate glass
ISO 10136-1 Glass and glassware – Analysis of extract solutions – Part 1: Determination of silicon dioxide by molecular absorption spectrometry
ISO 15202-3 Workplace air – Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry – Part 3: Analysis
ISO TS 80004-1 Nanotechnologies – Vocabulary – Part 1: Core terms

2.4 *EPA Standards:*[6]

Method 200.8, Revision 5.4 Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry

2.5 *U.S. Code of Federal Regulations:*[7]

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

3. Terminology

3.1 *Definitions:*

3.1.1 For additional definitions related to textiles, see Terminology D123; for additional definitions related to nanotechnology, see ISO 80004-1 and Terminology E2456; for additional definitions related to measurements, see ISO/IEC Guide 99; and for additional definitions related to ICP-OES and ICP-MS analyses, see Test Methods D7035 and D7439, respectively. Fig. 1 shows the types of solutions used in this standard.

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

3.1.3 *background correction, n*—the process of correcting the intensity at an analytical wavelength or mass/charge (m/z) for the intensity due to the underlying spectral background of a blank. **adapted from ISO 15202-3**

3.1.4 *blank test solution, n*—solution prepared in the same way as the test sample solution but omitting the test portion. **ISO 22036**

3.1.4.1 *Discussion*—

The blank test solution enables quantification of contamination introduced during test sample solution preparation from sources such as reagents, labware, and the environment. The blank test solution must be prepared and analyzed under the same operating conditions as the test sample solutions.

[3] The last approved version of this historical standard is referenced on www.astm.org.
[4] Available from American Association of Textile Chemists and Colorists (AATCC), P.O. Box 12215, Research Triangle Park, NC 27709-2215, http://www.aatcc.org.
[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.
[6] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, https://www.epa.gov/sites/production/files/2015-08/documents/method_200-8_rev_5-4_1994.pdf
[7] Available from U.S. Government Publishing Office (GPO), 732 N. Capitol St., NW, Washington, DC 20401, http://www.gpo.gov.

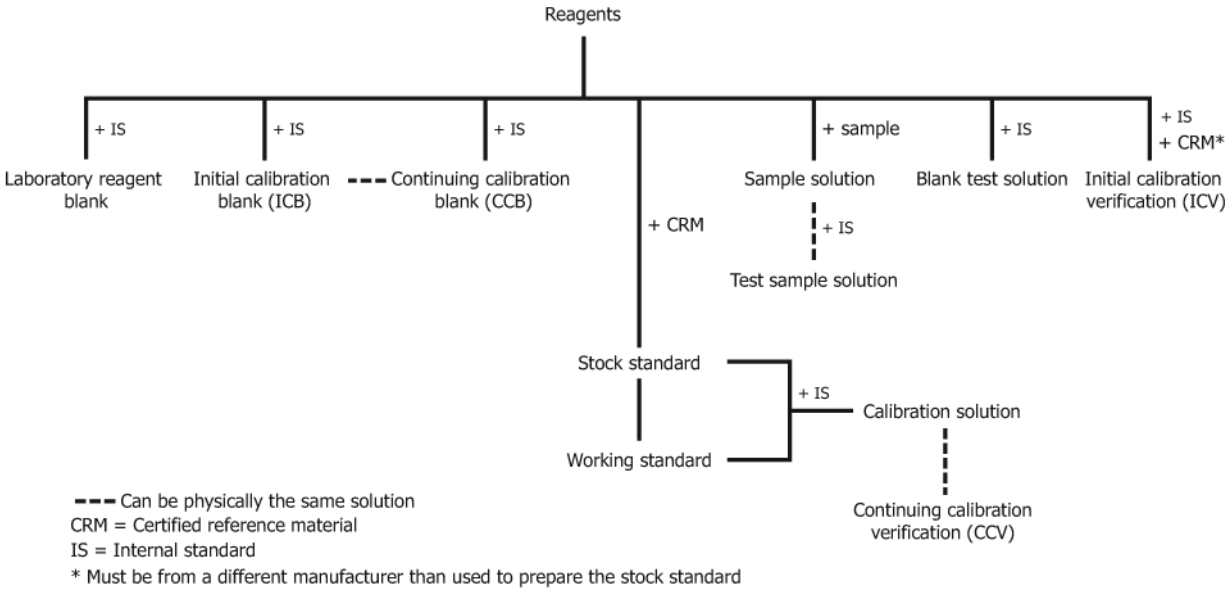

FIG. 1 Types of Solutions Used in This Test Method

3.1.5 *calibration solution, n*—solution prepared by dilution of the stock standard solution(s) or working standard solution(s), containing the analyte(s) of interest at a concentration(s) suitable for use in calibration of the analytical instrument. **ISO 15202-3**

3.1.5.1 *Discussion*—
Matrix matching is normally used when preparing calibration solutions.

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

3.1.6.1 *Discussion*—
A type of woven fabric or cloth which combine various structures and materials for a multitude of forms and purposes to satisfy human end use such as clothing, rugs, curtains.

3.1.7 *initial calibration verification standard (ICV), n*—a solution (or set of solutions) of known analyte concentration used to verify calibration standard levels; the concentration of analyte is to be near the mid-range of the linear curve that is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards.

3.1.7.1 *Discussion*—
The ICV must be matrix matched to the acid content of sample extracts or digestates. The ICV must be measured after calibration and before measuring any sample digestates or extracts. The measured value is to fall within ±10 % of the known value. **E1613**

3.1.8 *internal standard, n*—a non-analyte element, present in all calibration, blank, and sample solutions, the signal from which is used to correct for non-spectral interference or improve analytical precision. **ISO 15202-3**

3.1.9 *laboratory reagent blank (LRB), n*—a solution that must contain all of the reagents in the same volumes as used in processing the samples. This blank must be carried through the same entire preparation schemes as the samples, including digestion. **EPA 200.8**

3.1.9.1 *Discussion*—
The LRB and the blank test solution (3.1.4) are identical in substance and treatment but their functions differ. The purpose of the LRB is for computation of the method detection limit (3.1.13) and the method quantitation limit (3.1.14) prior to the preparation of the test samples and blank test solutions.

3.1.9.2 *Discussion*—
The LRB is used to assess contamination from reagents and the laboratory environment and to characterize spectral background from the reagents used in sample preparation.

3.1.10 *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 compounds, *(2)* modified or transformed natural polymers, or *(3)* glass. **D123**

3.1.11 *matrix matching, n*—a technique used to minimize the effect of the test solution matrix on the analytical results. **ISO 15202-3**

3.1.11.1 *Discussion*—
Matrix matching involves preparing calibration solutions in which the concentrations of acids and other major solvents and solutes are matched with those in the test solutions. With unknown sample matrices, exact matching is not possible. In this case, sample-specific matrix effects can be minimized by standard addition calibration method where samples are spiked with known concentration of analyte or by using an appropriate internal standard to compensate for multiplicative interference.

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

3.1.13 *method detection limit (MDL), n*—the minimum concentration of an analyte that can be reported with a 99 % confidence that the value is above zero. **D7035**

3.1.13.1 *Discussion*—
The MDL is also known as the limit of detection (LOD).

3.1.14 *method quantitation limit (MQL), n*—the minimum concentration of an analyte that can be measured with acceptable precision, ordinarily taken to be at least ten times the standard deviation of the mean blank signal. **D7035**

3.1.14.1 *Discussion*—
The MQL is also known as the limit of quantitation.

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

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

3.1.16.1 *Discussion*—
Properties that are not extrapolations from a larger size will typically, but not exclusively, be exhibited in this size range. For such properties the size limits are considered approximate.

3.1.16.2 *Discussion*—
The lower limit in this definition (approximately 1 nm) is introduced to avoid single and small groups of atoms from being designated as nano-objects or elements of nanostructures, which might be implied by the absence of a lower limit.

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

3.1.18 *sample solution, n*—solution prepared from a sample by the process of sample dissolution. **ISO 15202-3**

3.1.18.1 *Discussion*—
A sample solution might need to be subjected to further operations, for example, dilution or addition of an internal standard, or both, in order to produce a test solution that is ready for analysis.

3.1.19 *spectral interference, n*—an interference caused by the emission from a species other than the analyte of interest. **ISO 15202-3**

3.1.20 *stock standard solution, n*—solution used for preparation of working standard solutions or calibration solutions, or both, containing the analyte(s) of interest at a certified concentration(s) traceable to primary standards from internationally recognized Certified Reference Material producers (for example, National Institute of Standards and Technology or other National Metrology Institutes). **adapted from ISO 15202-3**

3.1.21 *test sample solution, n*—sample solution that has been subjected to all operations required to bring it into a state in which it is ready for analysis. **adapted from ISO 15202-3**

3.1.21.1 *Discussion*—
"Ready for analysis" includes dilution or the addition of internal standard, or both.

3.1.21.2 *Discussion*—
The test sample solution is the sample solution if these solutions are not subjected to any further operations before analysis.

3.1.22 *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**

3.1.23 *working standard solution, n*—solution, prepared by dilution of the stock standard solution(s), that contains the analyte(s) of interest at a concentration(s) better suited for preparation of calibration solutions than the concentration(s) of the analyte(s) in the stock standard solution(s). **ISO 15202-3**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *continuing calibration blank (CCB), n*—calibration solution prepared without the addition of any stock standard solution or working standard solution (adapted from ISO 15202-3) that is used to verify blank response and freedom from carryover of silver. The continuing calibration blank and the initial calibration blank may physically be the same blank solution but are identified separately to denote their position in the analytical sequence. **adapted from E1613**

3.2.1.1 *Discussion—*

The CCB must be matrix matched to the acid content of sample extracts and digestates.

3.2.1.2 *Discussion—*

The measured concentration of silver in the CCB is to be (at most) less than the method quantification limit.

3.2.2 *continuing calibration verification (CCV), n*—a solution (or set of solutions) of known analyte concentration used to verify freedom from excessive instrumental drift; the concentration of analyte is to be near the mid-range of a linear calibration curve and may be one of the actual calibration solutions. **adapted from E1613**

3.2.2.1 *Discussion—*

The CCV must be matrix matched to the acid content present in sample digestates or extracts. The CCV must be analyzed before and after all samples and at a frequency of not less than every ten samples. The measured value shall fall within ±10 % of the known value.

3.2.3 *initial calibration blank (ICB), n*—calibration solution prepared without the addition of any stock standard solution or working standard solution (adapted from ISO 15202-3) that is used to verify blank response and freedom from carryover of silver. The initial calibration blank and the continuing calibration blank may physically be the same blank solution but are identified separately to denote their position in the analytical sequence. **adapted from E1613**

3.2.3.1 *Discussion—*

The ICB must be matrix matched to the acid content of sample extracts and digestates. The ICB must be measured during and after calibration.

3.2.4 *mass fraction, n*—mass of total silver measured in a textile normalized to the mass of textile analyzed.

3.2.5 *qualitative measurement, n*—result for which the relative uncertainty is large or cannot be defined adequately for the measurand.

3.2.6 *quantitative measurement, n*—result for which there is knowledge of the sources of error that contribute to relative uncertainty for the measurand.

3.2.7 *total silver, n*—mass of element with atomic number 47 (isotopes, ions, metallic or zero-valent (Ag^0), alloys, oxide, or salt compounds, or combination thereof) in a consumer textile product.

4. Summary of Test Method

4.1 This test method utilizes acid digestion of a textile sample, addition of an appropriate internal standard, analysis with either ICP-OES or ICP-MS, and quantification by external calibration to determine total silver. The mass fraction of silver in each textile sample is calculated by normalizing the background-corrected measured mass of total silver to the dry textile sample mass. Results are reported in SI units of kg silver/kg textile though other units (for example, mg silver/kg textile) are common.

NOTE 1—If there is evidence that a precipitate (for example, silver chloride or sulfate) is present after the prescribed digestion procedure that cannot be redissolved the analyst may opt to use isotope dilution analysis (IDA) with ICP-MS to measure the mass fraction of total silver.

NOTE 2—IDA is advantageous because an enriched isotope of the analyte is used as an internal standard. After addition of the enriched isotope of the analyte to the natural sample isotopes and complete solubilization of the sample, their ratio becomes a proxy for the analyte concentration; subsequent

analyte loss (that is, precipitation) will not bias the concentration determination.

NOTE 3—IDA is considered a primary method (1)[8]; however, currently there is limited data available on the application of IDA for the determination of silver in textiles (2). As such, an IDA-ICP-MS method is provided in Appendix X1 for informational purposes only.

5. Significance and Use

5.1 Silver may be used to treat consumer textile products to provide enhanced antimicrobial (fungi, bacteria, viruses) properties (3, 4). At any point in a textile product's lifecycle, there may be a need to measure the amount of silver present. This standard prescribes a test method based on ICP-OES or ICP-MS analysis that manufacturers, producers, analysts, policymakers, regulators, and others may use for measurement of total silver in textiles. As described in Guide E3025, determination of total silver in a consumer textile product is one component of a tiered approach to determine if silver is present, possibly as nanomaterial(s) (one or more external dimensions in the nanoscale), prior to measuring the form and dimension of the Ag that is found. ICP-OES or ICP-MS analysis alone is not sufficient to determine whether a textile contains silver nanomaterial(s).

NOTE 4—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 Guide E3025.

5.2 As described in Guide E3025, the amount of silver in a textile can decrease over time as silver metal and silver compounds can react with oxygen and other oxidation-reduction (redox) active agents present in the environment to form soluble ionic species which are released by contact with moisture (for example, from ambient humidity, washing, body sweat, rain, or other sources). Hence, if silver is measured in a textile, the result may only be indicative of that moment in the article's life cycle and great care is necessary in drawing temporal inferences from the results.

5.3 If silver is measured by ICP-OES or ICP-MS analysis, additional analyses are needed to elucidate the form of silver in the textile specimen. This step is necessary because ICP-OES or ICP-MS results are for total silver independent of chemical and physical form and textiles may be treated with silver in sizes that range from the nanoscale (for example, salt nanoparticles) to the micrometer scale (for example, particulates or fibers).

5.4 If no silver is detected by ICP-OES, the more sensitive ICP-MS should be used to determine if silver is present in a test specimen. If no silver is detected in a textile sample using appropriate (fit for purpose) analytical techniques, then testing can be terminated.

NOTE 5—Typical method detection limits are 0.6 µg Ag/L by ICP-OES and 0.002 µg Ag/L by ICP-MS which are comparable to limits successfully used to detect silver in a range of products, including sports textiles and wound dressings (2).

5.5 Results of ICP-OES or ICP-MS analysis may be qualitative or quantitative, depending upon the efficacy of the digestion procedure for the textile matrix. Regardless, ICP-OES or ICP-MS analysis is recommended as a first step to screen for the presence of silver in a textile and results can be used to inform subsequent more detailed analyses as part of a tiered approach to determine if a textile contains silver nanomaterial(s).

6. Interferences

6.1 Potential exists for silver precipitates after the digestion step which would result in incomplete measurement of silver. Chloride and sulfide are known to react with dissolved silver to form poorly soluble precipitates (5, 6). These elements may be present in some reagents, textile matrices, waters (environmental, tap), and bodily fluids (for example, sweat) that could come into contact with a textile. If silver sulfide precipitates are formed, additional treatment steps shall be taken to redissolve the silver prior to ICP analysis (see Section 12). When hydrochloric acid (1% v/v) is used, the total recoverable sample digestion by this test method will effectively form a soluble form of AgCl that is suitable for the determination of silver in aqueous samples containing concentrations up to 100 µg silver/L (7). Alternatively, IDA-ICP-MS might be a useful method (see Appendix X1).

7. Apparatus

7.1 *Labware, Glassware, Beakers, and Volumetric Flasks,* that comply with the requirements of Specifications E288 and E694 and are made of borosilicate glass that complies with the requirements of ISO 3585. Glassware shall be cleaned before use by soaking in nitric acid for at least 24 hours and then rinsing thoroughly with water. Alternatively, before use, glassware shall be cleaned with

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

a suitable laboratory detergent using a laboratory washing machine. Metals-free polymer labware (for example, fluoropolymer, polypropylene, and low- or high-density polyethylene) is recommended, particularly for trace level analyses by ICP-MS.

7.2 *Analytical Balance,* calibrated to a traceable standard and capable of weighing to 1×10^{-7} kg.

7.3 *Digestion Tubes,* 0.050 L capacity with screw-cap lid, made of an inert material that is capable of withstanding temperatures of 100 °C to 120 °C (for example, polypropylene). Other types of tubes with screw-cap lid may be used provided they are inert and capable of withstanding the temperatures encountered during the digestion.

7.4 *Block Digester,* digestion apparatus that is thermostatically controlled, capable of maintaining an internal temperature of 95 °C for samples being digested, with wells appropriate for 0.050 L digestion tubes. Other size digestion tubes may be used with appropriately sized block digesters.

7.5 *Optical Emission Spectrometer,* differences exist among various makes and models of instruments and as such, detailed operating instructions are not provided. The analyst shall follow the instructions provided by the manufacturer of their particular instrument. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements of this test method and to maintain quality control data confirming instrument performance and analytical results.

7.6 *Mass Spectrometer,* differences exist among various makes and models of instruments and as such, detailed operating instructions are not provided. The analyst shall follow the instructions provided by the manufacturer of their particular instrument. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements of this test method and to maintain quality control data confirming instrument performance and analytical results.

8. Reagents and Materials

8.1 *Purity of Reagents*—Trace metal grade nitric acid (concentrated), hydrogen peroxide, ammonium hydroxide, and hydrochloric acid shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[9] 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that conforms to the resistivity requirement of 18.2 MΩ·cm (25 °C) for Type I water in Specification D1193.

8.3 *Stock Standard Solutions*—Prepare from high-purity silver standard having certified concentration(s) traceable to primary standards. Alternatively, one can use commercially available stock silver solutions manufactured by an ISO 17034 accredited supplier that are specifically prepared for ICP-OES or ICP-MS spectrometry and are traceable to primary standards.

8.4 *Internal Standard Solution*—Prepare from high-purity standards of the internal standard element of choice having certified concentration(s) traceable to primary standards. The internal standard shall be compatible with the test sample matrix and stock standard solution matrix. The internal standard should be an element that is not a component of the test ~~sample.~~ sample and should not introduce a spectral or isobaric interference for the analyte of interest. Ideally, the internal standard shall show similar chemical and analytical behavior to silver and should be chosen on the basis of a correlation study. As specified in Test Method D7439, ~~scandium has similar atomic mass to silver and is a suitable internal standard for ICP-MS analysis of silver. See Test Method~~ indium (115In) and rhodium (103Rh) have similar m/z as the isotopes of silver and might be suitable internal standards for ICP-MS. ~~D7439 for a list of other elements frequently used as an internal standard for ICP-MS analysis. Scandium, yttrium, indium,~~ Scandium, or other appropriate element chosen on the basis of a correlation study, can be used as the internal standard for ICP-OES analysis of silver.

[9] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

9. Hazards

9.1 The user shall refer to the safety data sheet (SDS) for each chemical for additional information on proper handling, compatibility, and storage.

9.2 Concentrated nitric acid is corrosive and oxidizing, and nitric acid vapor is an irritant. Avoid exposure by contact with the skin or eyes, or by inhalation of fumes. Use suitable personal protective equipment (such as impermeable gloves, safety goggles, faceshield, laboratory coat, and so forth) as established by a hazard assessment when working with concentrated nitric acid and carry out open-vessel sample digestion with nitric acid in a fume hood.

9.3 Hydrogen peroxide ~30 % (m/m) is corrosive, oxidizing, and highly reactive. Store away from strong acids. Use suitable personal protective equipment (such as impermeable gloves, safety goggles, faceshield, laboratory coat, and so forth) as established by a hazard assessment when working with hydrogen peroxide.

9.4 Ammonium hydroxide ~2.8 % (m/m) is corrosive. Avoid exposure by contact with skin or by inhalation. Use suitable personal protective equipment (such as impermeable gloves, safety goggles, faceshield, laboratory coat, and so forth) as established by a hazard assessment when working with ammonium hydroxide.

9.5 Hydrochloric acid (1 % v/v) is corrosive. Avoid exposure by contact with skin or by inhalation. Use suitable personal protective equipment (such as impermeable gloves, safety goggles, faceshield, laboratory coat, and so forth) as established by a hazard assessment when working with hydrochloric acid.

9.6 Pressure buildup during heating of sample tubes may result in eruption of sample tubes. To avoid pressure buildup, the screw-cap lids on digestion tubes must not be tightly sealed when heated in the block digester. The block digester must be used in a chemical fume hood which *(1)* at a minimum, is in compliance with authorities having jurisdiction, and *(2)* deemed appropriate by the Chemical Hygiene Officer or person(s) responsible for administering the Chemical Hygiene Plan.

10. Sampling and Test Specimens

10.1 A textile of interest is obtained and the desired number of representative samples are cut from the textile using silver-free (for example, plastic or ceramic) scissors in accordance with an appropriate sampling strategy that captures the areas that contain silver. The sampling plan (including the collection strategy and number of samples) should be fit for its intended purpose. Additionally, for textiles where silver may be distributed heterogeneously such as garments, care should be taken to collect samples using a strategy that captures spatial variability in a manner that is fit for the intended purpose of the measurement.

NOTE 6—Considerations should also be given to obtain threads, decorative trim, and other components used to assemble a textile product.

10.2 In the absence of knowledge about the distribution of silver in a textile, the analyst shall assume 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 analyst shall cut samples to capture this variability using some form of random sampling that describes the measurement distribution for their specific needs. A statistical sampling (power) calculation can be used to estimate the number of samples needed to achieve a desired level of precision.

NOTE 7—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, linens) or it may be the entire textile for smaller articles (for example, finger of a glove).

10.3 Finally, a desired number of test specimens are cut from each representative sample using silver free scissors. If the distribution of silver in a textile sample is known or could be heterogeneous, test specimens shall be cut from the samples to capture variability. The locations and dimensions of the test specimens will depend upon the specific sample.

NOTE 8—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.

NOTE 9—When preparing test specimens, consideration should be given to the contribution of the error associated with determination of mass (see 12.1) to the total analytical error. For example, the relative uncertainty on the mass of a 10 mg test specimen weighed on a 4-place balance is 1%.

10.4 Examples of textile, sample, and test specimen collection practices are described in Test Method D6413, AATCC 135, and 16 CFR Parts 1615 and 1616.

11. Calibration and Standardization

11.1 Prepare laboratory reagent blank and blank test solutions with internal standard, which are processed through the same digestion procedure as the test specimens, and contain all reagents used in sample digestion, in the same quantities used for preparation of blank and test sample solutions.

11.2 Prepare stock standard solutions of silver from a high-purity silver standard having certified concentration(s) traceable to primary standards (measurement standard established using a primary reference measurement procedure) or a commercially available stock silver solutions specifically prepared for ICP-OES or ICP-MS that is traceable to primary standards.

11.2.1 Prepare working standard solutions of silver from the stock standard solutions by serial dilution using the same acids and concentrations as test samples for ICP-OES and ICP-MS analyses (see Sections 13 and 14). Working standard solutions should be prepared before calibration measurements are started.

NOTE 10—For better accuracy, the analyst should prepare solutions on a mass fraction basis using a calibrated balance. Prepare a set of calibration solutions, the initial calibration blank (ICB) solution, and the initial calibration verification (ICV) solution.

NOTE 11—The ICV is used to assess the accuracy of the calibration standards. It must therefore be made from a different original source of stock solutions than the stock used to make the calibration standards. Use of a different serial dilution of the same original stock solution is not acceptable.

11.3 Prepare calibration solutions (preferably) from the working standard solutions using the same acids and concentrations as test sample solutions for ICP-OES and ICP-MS analyses (see Sections 13 and 14), covering the anticipated range of concentrations for the samples, but within the linear range of the instrument, that will be used to establish the analytical calibration curve. Include internal standard in the calibration solutions. Prepare calibration solutions fresh daily.

11.3.1 Prepare the ICB solution without the addition of any stock standard solution or working standard solution but including internal standard.

11.3.2 Prepare the ICV solution using a different stock solution than used to make the calibration standards and include internal standard.

11.4 Prepare the continuing calibration blank (CCB) solution without the addition of any stock standard solution or working standard solution but including internal standard (the CCB and ICB may physically be the same blank solution but are identified separately to denote their position in the analytical sequence).

11.5 Prepare the continuing calibration verification (CCV) solution by serial dilution of the same silver standard as the standard calibration source. The CCV shall consist of all the reagents in the same volumes as used in preparing the test sample solutions, including the internal standard. The CCV may be one of the actual calibration solutions.

11.6 Estimate the method detection limit (MDL) and method quantitation limit (MQL) under the working analytical conditions and repeat this exercise whenever experimental conditions are changed.

11.6.1 Subject ten laboratory reagent blank solutions to the digestion procedure used to prepare test sample solutions.

11.6.2 Make measurements (see Sections 13 and 14) on the ten laboratory reagent blank solutions.

11.6.3 Calculate the method detection limit (MDL) and method quantitation limit (MQL) as three times and ten times the standard deviation of the mean laboratory reagent blank signal, respectively. For additional details, see Test Method D7035.

NOTE 12—Calculation of the MDL in accordance with Test Method D7035 is prescribed in this standard, though alternative approaches such as that described in Practice D4210 may be used if better suited for the intended purpose of the measurement.

11.7 Determine the calibration curve under the working analytical conditions and repeat this exercise whenever experimental conditions are changed (described in 13.7 for ICP-OES and in 14.6 for ICP-MS).

11.7.1 At least five calibration standards shall be used in establishing the calibration curve. Analyze the calibration standards in order of increasing concentration. This approach permits corrective actions if results of the CCB exceed this concentration (see 13.9.1 and 14.8.1).

11.7.2 Make measurements on the ICB followed by the calibration solutions in order of increasing concentration then reanalyze the ICB followed by the ICV. Calculate the linear correlation coefficient for the calibration solutions; repeat the calibration if the correlation (R^2) is < 0.999. Additional details on the linear range of the instrument can be found in most instrument user manuals or in technical notes available on some vendor websites, or both.

12. Sample Digestion Procedure

12.1 Use a calibrated analytical balance to measure the mass of each test specimen to the nearest 1×10^{-7} kg and record the weight.

12.2 Test specimen digestion (adapted from **(8)**):

12.2.1 Place each weighed test specimen in a separate clean 0.050 L PFA (or other suitable) tube.

12.2.2 Add 0.005 L of deionized water to each tube followed by addition of 0.010 L of 70 % v/v nitric acid. If chloride is suspected in the textile, hydrochloric acid (1 % v/v) must be added to form soluble AgCl when the concentration of silver in the sample solution is up to 100 µg/L in solution **(7)**.

12.2.3 Place each tube in a block digester that is in a chemical fume hood that meets the conditions prescribed in 9.6 and heat at 95 °C for 70 minutes. Do not fully tighten the screw-cap lids on tubes to prevent pressure build up inside the tube during heating.

12.2.4 Remove each tube from the block digester, cool, and add 0.002 L water and 0.003 L of 30 % hydrogen peroxide to each tube.

12.2.5 Return tubes to the block digester at 95 °C and add 30 % hydrogen peroxide in 0.001 L increments until effervescence stops. Do not fully tighten the screw-cap lids on tubes to prevent pressure build up inside the tube during heating.

12.2.6 Heat tubes for 120 minutes at 95 °C. Do not fully tighten the screw-cap lids on tubes to prevent pressure build up inside the tube during heating.

NOTE 13—If the acid digestion is incomplete or ineffective, the analyst may dry ash the test specimen in a ceramic crucible prior to performing the wet digestion. Dry ashing procedures reported in the literature are provided in Appendix X2 for information purposes only.

NOTE 14—If a precipitate is evident in the digestate, the analyst shall redissolve the solids prior to analysis.

NOTE 15—If silver chloride precipitates are formed, one option is to evaporate off all of the acids and treat the entire sample digestate with concentrated ammonium hydroxide to redissolve the silver. Reference **(2)** provides additional options on how to redissolve silver chloride precipitates.

NOTE 16—If silver sulfide precipitates are formed, one option is to redissolve these particles using cyanide digestion procedures **(9, 10)**; however, such procedures would require special health and safety controls to prevent operator exposure when handling cyanide. An alternative that avoids the use of cyanide compounds is IDA-ICP-MS (see Appendix X1).

12.2.6.1 Reduce the acid volume by evaporation to near dryness and add internal standard to achieve the desired concentration. Quantitatively dilute to the desired final volume with deionized water or dilute nitric acid so that the final nitric acid concentration is 2 % v/v for both ICP-OES and ICP-MS analysis of the test sample solution.

NOTE 17—The digestion method prescribed herein is applicable to rayon, cotton, polyester, and lycra (see Section 17). Microwave-assisted digestion procedures may be necessary for other textile matrices **(2)** and some available procedures reported in the literature are provided for informational purposes in Appendix X3.

13. ICP-OES Analytical Procedure

13.1 Consult the manufacturer's instructions for operation of the ICP-OES and optimum analytical settings. This test method assumes that good operating procedures are followed. Design differences among instruments make it impractical to list detailed conditions.

13.2 For guidance on ICP-OES analysis, including plasma view and conditions (gas flow, radiofrequency power, viewing height, etc.), sample introduction, and quality control, the analyst is referred to Test Method D7035.

13.3 Select one or more emission lines on which to measure silver, usually the 328.068 nm line is used unless it is necessary to avoid this wavelength because of spectral interference or significant background. Take into consideration whether a wavelength is accessible on the available instrument.

NOTE 18—Agreement of results obtained using the Ag 328.068 nm and Ag 338.289 nm wavelengths provides a measure of confidence that interferences are not present.

13.4 Prior to any measurements, the analyst shall follow the instrument manufacturer's recommendations to *(1)* perform regular visual checks to ensure the instrument is in good order, and *(2)* carry out any daily performance checks to verify that the instrument is operating in accordance with specifications.

13.5 Allow the ICP-OES to warm up following manufactures recommendations or for 30-60 minutes. It is advisable to aspirate ICB or CCB solution into the plasma during warm-up.

13.6 To ensure the validity of the data obtained from an ICP-OES analysis, the quality control elements listed herein shall be considered the minimum for each analyte wavelength (see Test Method D7035):

13.6.1 Generate a spectral scan at 328.068 nm while analyzing *(1)* an ICB solution, *(2)* a calibration solution, and *(3)* a typical sample test solution into the plasma. Examine the line profiles and select points at which to make background correction measurements. Where applicable, make background correction measurements at a single point to correct for a simple background shift, that is, a shift in background intensity that is essentially constant over a given range (for example, 0.5 nm) on either side of the analyte emission line. Alternatively, for a sloping background, make background correction measurements at two points to correct for the non-constant background shift.

13.7 Calibrate the instrument at silver concentrations spanning the anticipated range in test samples (see 11.6 for additional details):

13.7.1 Beginning with the ICB, aspirate the solution into the plasma and make emission measurements at 328.068 nm. Continue with remaining calibration solutions in order of increasing concentration. The emission intensity of the ICB shall be subtracted from the emission intensities of the calibration solutions. From all background-corrected measurements generate a calibration curve for the silver response using linear regression by means of the instrument's computer.

13.7.2 Use a suitable wash-out solution, wash-out time, and wash-out rate between each measurement to ensure that there is no significant analyte carryover between measurements. The wash-out solution should have similar composition to the sample solution (2% v/v dilute nitric acid). A suitable washout protocol between sample measurements is to rinse with 2 % v/v dilute nitric acid at the same or faster pump speed than the rate used for sample analysis, but not above the maximum rated flow rate of the nebulizer.

13.8 Use the instrument software to bias correct the signal obtained from the samples and set the ICB as the baseline response. The internal standard signal is used to adjust the sample signal based on variations in sample transport in the sample introduction system. The internal standard signal response in each blank and sample test solution should be within 50 % to 125 % of the response in the ICB solution. For responses outside of this range, investigate the reasons, take corrective action, and repeat the analyses or analyze the sample diluted. Alternatively, the method of standard additions may be used.

13.9 Immediately after calibration, reanalyze the ICB solution, the ICV solution, the CCB solutions, and then the CCV solution.

13.9.1 If the measured concentration of silver in the ICB solution is above the MDL (quantitative analysis) or a minimum