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

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~~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~~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~~plant, pipelines and fittings – Properties of borosilicate glass
ISO 10136-1 Glass and ~~Glassware—Analysis of Extract Solutions—Part~~ glassware – Analysis of extract solutions – Part 1: Determination of ~~Silicon Dioxide by Molecular Absorption Spectrometry~~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~~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~~ 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] 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 ~~U.S. Environmental Protection Agency,~~ 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 ~~Printing Office, Superintendent of Documents,~~ Publishing Office (GPO), 732 N. Capitol St., NW, Washington, DC ~~20401-0001, http://www.access.gpo.gov.~~20401, http://www.gpo.gov.

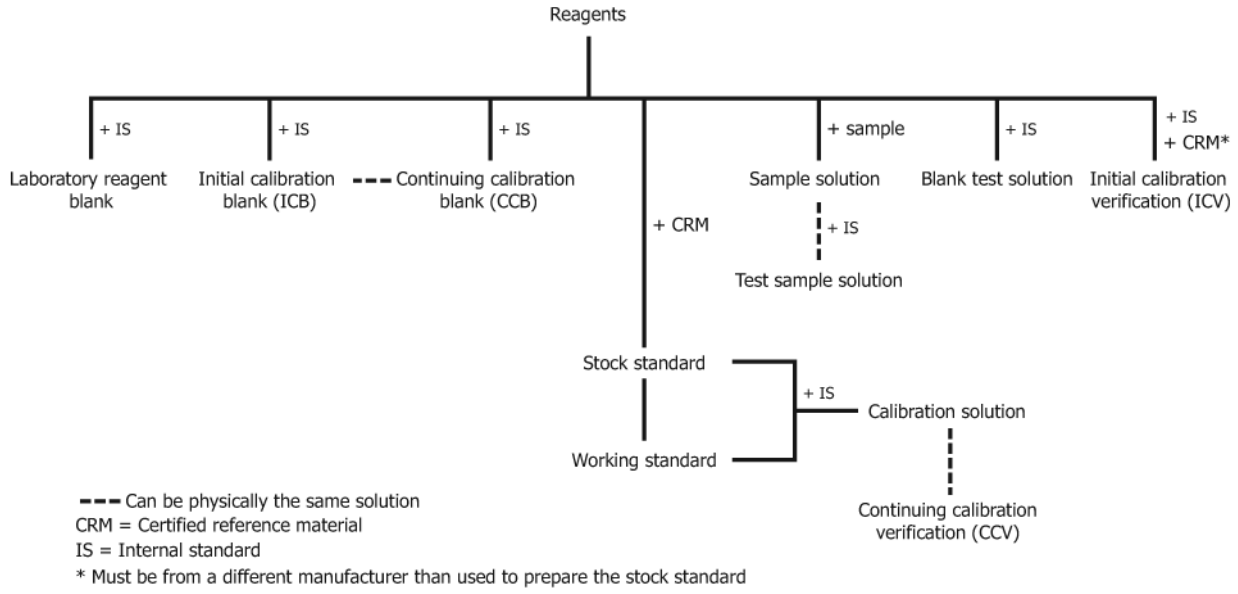

FIG. 1 Types of Solutions Used in This Test Method

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.

3.1.5 *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.