~~Designation:E1552–93 (Reapproved2002)~~ Designation: E 1552 – 08

Standard Test Method for ~~Determining Hafnium in Zirconium and Zirconium Alloys Using the D-C Argon Plasma Spectrometer~~Determining Hafnium in Zirconium and Zirconium Alloys By Direct Current Plasma—Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation E 1552; 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 test method covers the determination of hafnium in zirconium and zirconium alloys in concentrations greater than 0.003 %.

~~1.2~~

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 8.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

B~~349~~ 349/B 349M Specification for Zirconium Sponge and Other Forms of Virgin Metal for Nuclear Application

B 350/B 350M Specification for Zirconium and Zirconium Alloy Ingots for Nuclear Application

B 351/B 351M Specification for Hot-Rolled and Cold-Finished Zirconium and Zirconium Alloy Bars, Rod, and Wire for Nuclear Application

B 352/B 352M Specification for Zirconium and Zirconium Alloy Sheet, Strip, and Plate for Nuclear Application

B 353 Specification for Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Service (Except Nuclear Fuel Cladding)

B 493 Specification for Zirconium and Zirconium Alloy Forgings

B 494/B 494M Specification for Primary Zirconium

B 495 Specification for Zirconium and Zirconium Alloy Ingots

B 523/B 523M Specification for Seamless and Welded Zirconium and Zirconium Alloy Tubes

B 550/B 550M Specification for Zirconium and Zirconium Alloy Bar and Wire

B 551/B 551M Specification for Zirconium and Zirconium Alloy Strip, Sheet, and Plate

B 614 Practice for Descaling and Cleaning Zirconium and Zirconium Alloy Surfaces

B 653/B 653M Specification for Seamless and Welded Zirconium and Zirconium Alloy Welding Fittings

B 658/B 658M Specification for Seamless and Welded Zirconium and Zirconium Alloy Pipe

B 752 Specification for Castings, Zirconium-Base, Corrosion Resistant, for General Application

B 811 Specification for Wrought Zirconium Alloy Seamless Tubes for Nuclear Reactor Fuel Cladding

E 50~~Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals~~
Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E 135~~Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials~~[4]

~~E1060Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis~~
Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E 1097 Guide for Direct Current Plasma-Atomic Emission Spectrometry Analysis

[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.06 on Ti, Zr, W, Mo, Ta, Nb, Hf, Re.

Current edition approved July 15, ~~1993.~~2008. Published ~~September~~ August 2008. Originally approved in 1993. Last previous edition approved in 2002 as E 1552 – 93 (2002).

[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* ~~, Vol 02.04.~~volume information, refer to the standard's Document Summary page on the ASTM website.

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E 135.

4. Summary of Test Method

4.1 The sample, in the form of drillings, chips, milling, turnings, or powder, is dissolved in dilute hydrofluoric acid (HF). The hafnium content is measured using a ~~d-c argon~~direct current plasma (DCP) spectrometer, which is calibrated with reference solutions of hafnium in the presence of zirconium. The microprocessor is programmed to display the results in micrograms per millilitre (µg/mL).

5. Significance and Use

5.1 When zirconium materials are used in nuclear applications, it is necessary that hafnium, a neutron absorber, be present only at very low concentrations.

~~5.2This method is useful in testing materials for compliance with the compositional requirements as given in Specifications B349, B350, B351, B352, and B353~~

5.2 This test method is useful in testing materials for compliance with the compositional requirements as given in Specifications B 349/B 349M, B 350/B 350M, B 351/B 351M, B 352/B 352M, B 353, B 493, B 494/B 494M, B 495, B 523/B 523M, B 550/B 550M, B 551/B 551M, B 653/B 653M, B 658/B 658M, B 752, and B 811.

6. Apparatus

6.1 *Plastic Labware*:

6.1.1 *Beakers*, 100-mL, disposable, polypropylene, or 125-mL polytetrafluoroethylene (PTFE) are satisfactory.

6.1.2 *Volumetric Flasks*—Linear polyethylene (LPE) or polymethylpentene (PMP) are satisfactory.

NOTE 1—Plastic volumetric flasks change dimension as they age and therefore must be recalibrated periodically.

6.2 *Spectrometer— Modified Czerny-Turner*, using an Echelle grating with 30° prism for order separation providing a reciprocal linear dispersion of about ~~1 Å mm~~0.1 nm/mm in the 80 to 85th order. The instrument is operated in the sequential mode.

6.3 ~~*Excitation Source*[6]:~~ Excitation Source:[3]

6.3.1 ~~*D-C Argon Plasma*~~D-C Plasma, formed by a tungsten cathode and two carbon anodes in an inverted "Y" configuration, having a current output of 7 A at 40 V.

6.3.2 Glass spray tube shall be replaced with one made from PTFE or pyrolytic graphite to prevent hydrofluoric acid attack on the glass.

6.4 *Argon*—Commercially available as prepurified gas or liquid is satisfactory.

7. Reagents and Materials

7.1 *Purity and Concentration of Reagents*—The purity and concentration of chemical reagents shall conform to the requirements prescribed in Practices E 50.

7.2 *Pure Metals*:

7.2.1 *Hafnium Metal or Hafnium Dioxide* , of highest purity available and having a known impurity content.

NOTE 2—Many hafnium materials contain residual zirconium in quantity sufficient to affect the hafnium value.

7.2.2 *Zirconium Metal*, of the highest purity available and having a known hafnium content.

7.3 *Reference Materials*:

7.3.1 ~~*Standard Reference Materials (SRM): Three unalloyed zirconium materials*—SRM 1234, 1235, and 1236 containing 46, 95, and 198 ppm hafnium, respectively, and three alloyed zircoloy materials—SRM 1237, 1238, and 1239 containing 31, 178, and 77 ppm hafnium, respectively.~~ Standard Reference Materials (SRM):[4] One Zircaloy-4, SRM 360b, containing 80 ppm hafnium, is available.

7.3.2 Other reference solutions can be prepared by dissolving zirconium metal in HF. A solution of hafnium metal dissolved in HF is added to the zirconium solution to produce the required concentrations.

8. Hazards

8.1 This method involves the use of concentrated hydrofluoric acid. Read and follow label precautions carefully before using.

8.2 Refer to Practices E 50, 7.4.11, for more information.

~~[3] Annual Book of ASTM Standards, Vol 3.05.~~

[3] The sole source of supply of the apparatus known to the committee at this time is Applied Research Laboratories, Inc., 5371 NW 161 St., Miami, FL 33014, http://www.arl-test.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

~~[4] Discontinued, See, *1997 Annual Book of ASTM Standards,* Vol 03.05.~~

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.