

Designation: E 1552 – 93 (Reapproved 1998)

Standard Test Method for Determining Hafnium in Zirconium and Zirconium Alloys Using the D-C Argon Plasma Spectrometer¹

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 *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:

B 349 Specification for Zirconium Sponge and Other Forms of Virgin Metal for Nuclear Application²

B 350 Specification for Zirconium and Zirconium Alloy Ingots for Nuclear Application²

B 351 Specification for Hot-Rolled and Cold-Finished Zirconium and Zirconium Alloy Bars, Rod and Wire for Nuclear Application²

B 352 Specification for Zirconium and Zirconium Alloy Sheet, Strip, and Plate for Nuclear Application²

B 353 Specification for Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Service²

B 614 Practice for Descaling and Cleaning Zirconium and Zirconium Alloy Surfaces³

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials⁴

E 1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis⁵

E 1097 Guide for Direct Current Plasma Emission Spectrometry Analysis⁵

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 plasma 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 ($\mu\text{g/mL}$).

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.

5.2 This method is useful in testing materials for compliance with the compositional requirements as given in Specifications B 349, B 350, B 351, B 352, and B 353.

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*⁶—*Modified Czerny-Turner*, using an Echelle grating with 30° prism for order separation providing a reciprocal linear dispersion of about 1 Å mm in the 80 to 85th order. The instrument is operated in the sequential mode.

*6.3 Excitation Source*⁶:

6.3.1 *D-C Argon 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 Spectrometer system manufactured by Applied Research Laboratories, Inc., Valencia, CA 91355, has been found satisfactory.

¹ This test method is under the jurisdiction of ASTM Committee E-1 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.

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² *Annual Book of ASTM Standards*, Vol 02.04.

³ *Annual Book of ASTM Standards*, Vol 3.05.

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

⁵ *Annual Book of ASTM Standards*, Vol 03.06.