

# Standard Specification for Zirconium Sponge and Other Forms of Virgin Metal for Nuclear Application<sup>1</sup>

This standard is issued under the fixed designation B349/B349M; 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 (\$\epsilon\$) indicates an editorial change since the last revision or reapproval.

#### 1. Scope

- 1.1 This specification covers one grade of virgin zirconium metal commonly designated as sponge because of its porous, sponge-like texture, but it may also take other forms such as chunklets, suitable for use in nuclear applications.
- 1.2 Unless a single unit is used, for example corrosion mass gain in mg/dm², the values stated in either inch-pound or SI units are to be regarded separately as standard. The values stated in each system are not exact equivalents; therefore each system must be used independently of the other. SI values cannot be mixed with inch-pound values.
- 1.3 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 and health practices and determine the applicability of regulatory limitations prior to use.

#### 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

#### 3. Terminology

- 3.1 Forms:
- 3.1.1 *chunklets*, *n*—zirconium metal produced from the reduction of the chloride, usually by sodium.
- 3.1.1.1 *Discussion*—In this process, the reduced metal is melted and dripped onto a rotating disk to form chunklets.
- 3.1.2 *sponge*, *n*—zirconium metal produced from the reduction of the chloride, usually by magnesium.
- 3.1.2.1 *Discussion*—The process is one where the metal condenses to the solid state and does not melt.

3.2 Lot Definition—a lot shall consist of a single blend produced at one time.

#### 4. Classification

4.1 Primary zirconium is furnished in one grade designated as Reactor Grade R60001, suitable for nuclear applications. The main characteristic of the reactor grade is its low neutron capture cross section as achieved by removal of hafnium. The manufacturer must use procedures to prevent contamination with other high cross-section materials.

#### 5. Ordering Information

- 5.1 Purchase orders for material under this specification shall include the following information, as required, to describe adequately the desired material:
  - 5.1.1 Quantity (weight),
  - 5.1.2 Name of material (zirconium sponge or chunklets),
  - 5.1.3 Grade designation (see 4.1),
  - 5.1.4 ASTM designation and year of issue.
- 5.2 In addition to the data specified in 5.1, the following options and points of agreement between the manufacturer and the purchaser shall be specified in the purchase order, as required.
  - 5.2.1 Sampling and duplicate samples (see 8.2 and 8.3).
  - 5.2.2 Certification reports (Section 14), and
  - 5.2.3 Packaging (Section 16).

Note 1—A typical ordering description is as follows: 5000 lb (2000 kg) reactor grade zirconium, Grade R60001, ASTM Specification B349/B349M - 09.

#### 6. Materials and Manufacture

- 6.1 Zirconium metal is usually prepared by reduction of zirconium tetrachloride, and gets its physical characteristics from the processes involved in production. These characteristics may be expected to vary greatly with manufacturing methods. This specification, however, is not limited to material prepared by reduction of tetrachloride or to material of any specific physical form.
- 6.2 Only virgin zirconium metal, in identified, uniform, well-mixed blends, shall be supplied under this specification.

<sup>&</sup>lt;sup>1</sup> This specification is under the jurisdiction of ASTM Committee B10 on Reactive and Refractory Metals and Alloys and is the direct responsibility of Subcommittee B10.02 on Zirconium and Hafnium.

Current edition approved May 1, 2016. Published May 2016. Originally approved in 1960. Last previous edition approved in 2009 as B349/B349M-09. DOI:  $10.1520/B0349\_B0349M-16$ .

<sup>&</sup>lt;sup>2</sup> 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.

# 7. Chemical Composition

7.1 The zirconium metal supplied under this specification shall conform to the requirements for chemical composition prescribed in Table 1.

#### 8. Sampling

- 8.1 This sampling method detailed in this section shall be considered the industry standard, but alternate methods are acceptable when agreed to by the manufacturer and the purchaser. All sampling methods shall be such that the analysis is representative of the sponge blend.
- 8.2 A blend of sponge shall be sampled by running a full quantity through a proportioner or splitter to obtain a representative sample of at least 1% of the blend weight. The samples for chemical analyses shall be made on a compacted briquette and an evaluation ingot. The method for splitting this sample and preparing an evaluation ingot and compacted briquette shall be agreed upon between the manufacturer and the purchaser.
- 8.2.1 The evaluation ingot shall be greater than 30 lb (14 kg).
  - 8.2.2 The compacted briquette shall be at least 1 lb (0.5 kg).
- 8.3 When specified in the purchase order, a duplicate sample or portions of the briquette and evaluation ingot shall be supplied to the purchaser.
- 8.4 For metal forms other than sponge, such as chunklets, the sampling procedures shall be as agreed upon between purchaser and manufacturer.

## 9. Methods of Chemical Analysis

9.1 The preparation and analytical methods detailed in this section shall apply for the industry standard sampling methods detailed in Section 8. Alternate methods are acceptable when agreed to by the manufacturer and the purchaser. At a minimum, analytical samples must consist of a melted solid metal sample and an unmelted sponge sample. The solid

TABLE 1 Chemical Requirements of Zirconium Sponge, Reactor Grade R60001

Element	Permissible Impurities, max, ppm
Aluminum	75
Boron	0.5
Cadmium	0.5
Carbon	250
Chlorine	1300
Chromium	200
Cobalt	20
Copper	30
Hafnium	100
Iron	1500
Manganese	50
Molybdenum	50
Nickel	70
Nitrogen	50
Oxygen	1400
Silicon	120
Titanium	50
Tungsten	50
Uranium (total)	3.0

Note 2 — Magnesium or Sodium need to be tested only when they are used in the reduction step.

sample shall be analyzed for all elements listed in Table 1 except for chlorine, magnesium or sodium which must be analyzed from the unmelted sponge sample. All preparation and analytical methods shall be such that the analysis is representative of the sponge blend.

## 9.2 Preparation of Sample:

- 9.2.1 Compact the sponge sample taken in accordance with Section 8 into a consumable electrode and melt to ingot form in an arc furnace of a type conventionally used for reactive metals. The ingot shall be prepared for analysis by either of the following two methods:
- 9.2.1.1 Take a longitudinal section through the center of the ingot. Sample this section and analyze by appropriate means at a minimum of three places at approximately equal intervals diagonally from the top to the bottom of the section.
- 9.2.1.2 Samples for chemical tests shall be taken from solid metal below the surface porosity of the as-cast ingot. The samples shall be taken from a minimum of three places equally spaced along the axial length of the ingot.
- 9.3 Analytical samples for the determination of chlorine, magnesium or sodium must be taken from the sponge, since these elements are volatilized in melting. Obtain this sample by drilling a compacted sample of the sponge or chunklets. Sample the briquette by drilling, without water or other lubricant, a minimum of three holes, each 3/8 in. (9.5 mm) or larger in diameter, at equal intervals on a circle, concentric with the rounded surface of the briquette. Reject chips until the flutes of the drill are 1/4 in. (6 mm) below the surface of the briquette. Take the sample chips from this point until the point of the drill is within 1/4 in. (6 mm) of the opposite surface of the briquettes. Crush chips taken in this manner to pass a No. 4 (4760-µm) sieve and thoroughly mix.
- 9.4 Analyze one of the samples taken in accordance with 9.2.1 for its uranium content. Analyze all samples taken in accordance with 9.2.1 for all elements listed in Table 1 except for chlorine, magnesium, sodium, and uranium.
- 9.5 Analysis—Analysis shall be made using the manufacturer's standard methods. In the event of disagreement as to the chemical composition of the metal, chemical analysis for referee purposes shall be determined by a mutually acceptable laboratory. The average of the analyses for each impurity shall conform to the requirements of this specification, with no individual value greater than 30 % above the maximum specified limit for that impurity. Practice E29 shall be used to establish significant digits.

# 10. Particle Size

10.1 Zirconium sponge supplied under this specification shall pass a 1 in. (25 mm) screen and shall contain less than 2 % minus 20 mesh particles.

### 11. Retest

11.1 If any sample or specimen exhibits obvious contamination, or improper preparation, or flaws which disqualify it as a representative sample, the sample shall be discarded and a new sample or specimen substituted.

#### 12. Rejection

12.1 Rejection for failure of the material to meet the requirements of this specification shall be reported to the manufacturer within 60 calendar days from the receipt of the material by the purchaser. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense, unless the purchaser receives, within three weeks of the notice of rejection, other instructions for disposition.

#### 13. Referee

13.1 In the event of disagreement between the manufacturer and the purchaser on the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee shall perform the tests in question. The results of the referee's testing shall be used in determining conformance of the material to this specification.

#### 14. Certification

14.1 A producer's or supplier's certification shall be furnished to the purchaser that the material was manufactured, sampled, tested, and inspected in accordance with this specification and has been found to meet the requirements. A report of the test results shall be furnished.

# 15. Product Marking

- 15.1 The containers shall be legibly and permanently marked with the following information:
  - 15.1.1 Purchase order number.
  - 15.1.2 Name of material,
  - 15.1.3 Particle size range,
  - 15.1.4 Net weight,
  - 15.1.5 ASTM designation, and
  - 15.1.6 Date packed.

# 16. Packaging and Package Marking

16.1 The method of packaging shall be as agreed upon by the manufacturer and the purchaser. Packaging shall be such as to assure safe delivery when shipped by any common carrier. The size and nature of the containers used are generally determined by the time and length of storage and the amount of handling involved. Where fire hazards or sponge deterioration during prolonged storage are primary considerations, zirconium sponge should be packed in airtight, moisture-proof, sealed metal cans or drums which have been backfilled with argon.

#### 17. Keywords

17.1 nuclear; virgin zirconium metal; zirconium chunklets; zirconium sponge

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/