



## Standard Specification for Primary Zirconium<sup>1</sup>

This standard is issued under the fixed designation B 494/B 494M; 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 two grades of zirconium metal commonly designated as sponge or chunklets, but may also take other forms. This specification does not include crystal bar zirconium.

1.2 Unless a single unit is used, for example corrosion mass gain in  $\text{mg}/\text{dm}^2$ , 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 *The following precautionary caveat pertains only to the test method portions of this specification. 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:*

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>2</sup>

### 3. Terminology

3.1 *Lot Definitions:*

3.1.1 *castings*—a lot shall consist of all castings produced from the same pour.

3.1.2 *ingot*—no definition required.

3.1.3 *rounds, flats, tubes, and wrought powder metallurgical products (single definition, common to nuclear and non-nuclear standards)*—a lot shall consist of a material of the same size, shape, condition, and finish produced from the same ingot or powder blend by the same reduction schedule and the same heat treatment parameters. Unless otherwise agreed between manufacturer and purchaser, a lot shall be limited to the product of an 8 h period for final continuous anneal, or to a single furnace load for final batch anneal.

3.1.4 *sponge*—a lot shall consist of a single blend produced at one time.

3.1.5 *weld fittings*—definition is to be mutually agreed upon between manufacturer and the purchaser.

### 4. Classification

4.1 Primary zirconium is furnished in two grades:

4.1.1 *Grade R60702*—Unalloyed zirconium.

4.1.2 *Grade R60703*—Unalloyed zirconium for metallurgical alloying.

### 5. Ordering Information

5.1 Orders for material under this specification shall include the following information:

5.1.1 Quantity (weight),

5.1.2 Name of material (zirconium sponge or chunklet),

5.1.3 Grade number (see 4.1),

5.1.4 ASTM designation and year of issue,

5.1.5 Check analysis (see 7.2),

5.1.6 Sampling of small blends (see 8.1.1),

5.1.7 Inspection (see 11.1),

5.1.8 Certification report (Section 13),

5.1.9 Product marking (Section 15),

5.1.10 Packaging (Section 16),

5.1.11 Oxygen limits, when needed (Table 1), and

5.1.12 Additions to the specification and supplementary requirements, as required.

NOTE 1—A typical ordering description is as follows: 3000 lb (2000kg) zirconium sponge, ASTM B 494 - 01, Grade R60703.

### 6. Materials and Manufacture

6.1 Zirconium metal is usually prepared by reduction of zirconium tetrachloride, and it 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 metal 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.

### 7. Chemical Composition

7.1 The material shall conform to the requirements as to chemical composition prescribed in Table 1.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.02.

**TABLE 1 Chemical Requirements**

Element	Composition, %	
	Grade R60702	Grade R60703
Zirconium + hafnium	99.2 min	98.0 min
Hafnium	4.5 max	4.5 max
Iron + chromium	0.2 max	...
Hydrogen	0.005 max	...
Nitrogen	0.025 max	...
Carbon	0.05 max	...
Oxygen	<sup>A</sup>	<sup>A</sup>

<sup>A</sup>Limits as specified by the purchaser.

7.2 The purchaser may perform a check analysis for any elements listed in Table 1.

7.2.1 The manufacturer's analysis shall be considered as verified if the check analysis confirms the manufacturer's reported values within the tolerances prescribed in Table 2. Practice E 29 shall be used to establish significant digits.

## 8. Sampling

### 8.1 Sampling for Chemical Analysis:

8.1.1 The following method shall be used for blends of 3000 lb (1362 kg) or more. Smaller blends shall be sampled as agreed upon between the manufacturer and the purchaser.

8.1.2 The method shall produce a representative sample amounting to a minimum of 1 % of the quantity sampled. The sample shall be prepared by running the full quantity through a proportioner or splitter so arranged as to give the required amount of sample material. A minimum of 30 lb (14 kg) of this sample shall be taken to prepare the evaluation ingot described in 10.1.

8.1.3 If analytical samples are required for the determination of magnesium or sodium and chloride, they must be taken from the primary zirconium metal since these constituents are volatilized in melting.

8.1.4 Analytical samples for the determination of impurities other than magnesium or sodium and chloride shall be taken from the evaluation ingot (Sections 8 and 10).

## 9. Number of Tests and Retests

### 9.1 Number of Tests:

9.1.1 A minimum of three chemical tests shall be performed on the evaluation ingot.

### 9.2 Retests:

9.2.1 If any sample or specimen exhibits obvious surface contamination or improper preparation disqualifying it as a truly representative sample, it shall be discarded and a new sample or specimen substituted.

**TABLE 2 Permissible Variation in Check Analysis Between Different Laboratories**

Element	Permissible Variation in Check Analysis, %
Hydrogen	0.005
Nitrogen	0.01
Carbon	0.02
Hafnium	0.1
Iron + chromium	0.03
Oxygen	0.02

9.2.2 If the results of any tests of any lot do not conform to the requirements specified, retests shall be made on additional samples of double the original number from the same lot. Each of the retest values shall conform to the requirements specified.

## 10. Specimen Preparation

10.1 The sample for chemical analysis shall be obtained in accordance with 8.1.2 and shall be compacted into consumable electrodes, then melted 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:

10.1.1 A longitudinal section shall be taken from the center of the ingot. The longitudinal section shall be analyzed on samples taken from the longitudinal section at approximately equal intervals, as prescribed in 9.1.1.

10.1.2 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 equally spaced locations along the axial length of the ingot, as prescribed in 9.1.1.

## 11. Inspection

11.1 The manufacturer shall inspect the material covered by this specification prior to shipment. If so specified in the purchase order, the purchaser or his representative may witness the testing and inspection of the material at the place of manufacture. In such cases the purchaser shall state in his purchase order which tests he desires to witness. The manufacturer shall give ample notice to the purchaser as to the time and place of the designated tests. If the purchaser's representative does not present himself at the time agreed upon for the testing, the manufacturer shall consider the requirement for purchaser's inspection at place of manufacture to be waived.

11.2 The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with this specification. This inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

## 12. Rejection and Rehearing

12.1 Material that fails to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of the test, the producer or supplier may make claim for a rehearing.

## 13. Certification

13.1 When specified in the purchase order or contract, 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. When specified in the purchase order or contract, a report of the test results shall be furnished.

## 14. Referee

14.1 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.

### **15. Product Marking**

15.1 The containers shall be marked legibly and permanently per the agreement between the manufacturer and the purchaser.

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### **16. Packaging**

16.1 The method of packaging shall be as agreed upon between the manufacturer and the purchaser and must be acceptable by any common carrier. Packaging methods must take due recognition of the pyrophoric nature of finely divided zirconium.

### **17. Keywords**

17.1 primary zirconium metal; zirconium sponge