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Army - AT
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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Methods for CHEMICAL ANALYSIS OF ZIRCONIUM AND ZIRCONIUM ALLOYS[1]

This standard is issued under the fixed designation E 146; 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 These methods cover procedures for the chemical analysis of zirconium and zirconium alloys having chemical compositions within the following limits:

Aluminum, ppm	25 to 360
Boron, ppm	0.1 to 25
Cadmium, ppm	0.1 to 5
Carbon, ppm	100 to 600
Chromium, ppm	20 to 2000
Cobalt, ppm	0.5 to 30
Copper, ppm	5 to 300
Hydrogen, ppm	1 to 100
Iron, ppm	250 to 3000
Lead, ppm	50 to 500
Magnesium, ppm	5 to 700
Manganese, ppm	10 to 100
Molybdenum, ppm	5 to 200
Nickel, ppm	20 to 1500
Nitrogen, ppm	2 to 200
Oxygen, ppm	250 to 3500
Silicon, ppm	10 to 400
Tin, %	0.20 to 2.0
Titanium, ppm	10 to 200
Tungsten, ppm	30 to 200
Uranium (total), ppm	0.5 to 5

1.2 The techniques and procedures covered in these methods have been chosen so as to keep the consumption of sample to a minimum.

1.3 The analytical procedures appear in the following order:

	Sections
Iron by the Ortho-Phenanthroline (Photometric) Method	8 to 17
Nickel by the Dimethylglyoxime (Photometric) Method	18 to 26
Chromium by the Diphenylcarbazide (Photometric) Method	27 to 36
Aluminum by the 8-Hydroxyquinoline (Fluorometric) Method	37 to 48
Manganese by the Periodate (Photometric) Method	49 to 57
Nitrogen by the Nessler (Photometric) Method	58 to 70
Tin by the Iodate Titration (Volumetric) Method	71 to 78
Titanium by the 5-Sulfosalicylic Acid (Photometric) Method	79 to 88
Carbon by the Combustion-Conductometric Method	89 to 101
Tungsten by the Thiocyanate (Photometric) Method	102 to 111
Uranium by the Fluorometric Method	112 to 122
Oxygen by the Inert Gas Fusion Method Using the Platinum Flux Noncycling Technique	123 to 134
Copper by the Neo-Cuproine (Photometric) Method	135 to 143
Hydrogen by the Hot Extraction Method	144 to 153
Silicon by the Molybdenum Blue (Photometric) Method	154 to 164

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Sections 5, 63, 116, and Note 10.

2. Applicable Documents

2.1 *ASTM Standards:*

E 29 Recommended Practice for Indicating

[1] These methods are under the jurisdiction of ASTM Committee E-3 on Chemical Analysis of Metals and are the direct responsibility of Subcommittee E03.05 on Nonferrous Metals.

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Which Places of Figures Are to Be Considered Significant in Specified Limiting Values[2]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[2]

E 60 Practice for Photometric Methods for Chemical Analysis of Metals[2]

3. Significance and Use

3.1 These methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these methods will be trained analysts, capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

4. Apparatus, Reagents, and Photometric Practice

4.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure. The apparatus, standard solutions, and certain other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50, except that photometers shall conform to the requirements prescribed in Practice E 60.

4.2 Photometric practice prescribed in these methods shall conform to Practice E 60.

5. Safety Precautions

5.1 For precautions to be observed in the use of certain reagents in these methods, reference shall be made to Practices E 50.

6. Sampling

6.1 The sample shall be selected so as to be representative of the material to be analyzed.

6.2 For the determination of hydrogen, nitrogen, and oxygen, solid pieces are preferred to millings, turnings, or drillings, if representative, so as to minimize the effect of surface area.

7. Rounding Calculated Values

7.1 Calculated values shall be rounded to the desired number of places in accordance with the rounding method given in Section 3.4 to 3.5 of Recommended Practice E 29.

IRON BY THE ORTHO-PHENANTHROLINE (PHOTOMETRIC) METHOD[3]

8. Summary of Method

8.1 Ferrous iron, in a solution having a pH of 5.5 to 6.0, forms an orange-red complex with *o*-phenanthroline. Photometric measurement is made at 508 nm.

9. Concentration Range

9.1 The recommended concentration range is from 0.050 to 0.25 mg of iron in 100 mL of solution, using a cell depth of 1 cm.[4]

10. Stability of Color

10.1 The color is stable for at least 4 h.

11. Interferences

11.1 The elements ordinarily present in zirconium and zirconium alloys do not interfere if their concentrations are within the maximum limits shown in 1.1.

12. Apparatus

12.1 *Platinum Beakers* with matching covers, 200-mL capacity.

12.2 *pH Meter*—Hydrogen ion meter, fitted with a glass indicator electrode and a calomel reference electrode, capable of measuring 0.01 pH unit.

13. Reagents

13.1 *Hydroxylamine Hydrochloride Solution* (100 g/L)—Dissolve 50 g of hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) in water and dilute to 500 mL. Store in a refrigerator.

13.2 *Iron Standard Solution* (1 mL = 0.10 mg Fe)—Dissolve 0.1000 g of iron wire in 100 mL of HCl (1+1) and dilute with water to 1 L in a volumetric flask.

13.3 *Ortho-Phenanthroline Solution* (2 g/L)—Dissolve 1.0 g of *o*-phenanthroline monohydrate in hot water. Cool and dilute to 500 mL. Store in a refrigerator.

13.4 *Tartaric Acid Solution* (500 g/L)—Dis-

[2] *Annual Book of ASTM Standards*, Vol 03.05.

[3] Supporting data giving the results of cooperative tests have been filed at ASTM Headquarters, 1916 Race St., Philadelphia, Pa. 19103, in research report file RR: E03-197.

[4] This procedure has been written for a cell having a 1-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.