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Designation: E 367 – 02

Standard Methods for Chemical Analysis of Ferroniobium[1]

This standard is issued under the fixed designation E 367; 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 the chemical analysis of ferrocolumbium having chemical compositions within the following limits:

Element	Concentration, %
Aluminum	2.00 max
Carbon	0.30 max
Chromium	2.00 max
Cobalt	0.25 max
Niobium	40.00 to 75.00
Lead	0.01 max
Manganese	3.00 max
Phosphorus	0.05 max
Silicon	4.00 max
Sulfur	0.03 max
Tantalum	7.00 max
Tin	0.15 max
Titanium	5.00 max
Tungsten	0.50 max

1.2 The methods appear in the following order:

	Sections
Niobium, Tantalum, and Titanium by the Ion-Exchange Method	9-30

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 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 Section 5.

2. Referenced Documents

2.1 *ASTM Standards:*

A 550 Specification for Ferrocolumbium[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]

E 32 Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition[4]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[4]

E 173 Practice for Conducting Interlaboratory Studies ofMethods for Chemical Analysis of Metals[5]

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 other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50. 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, refer to Practices E 50.

6. Sampling

6.1 For procedures for sampling the material, and for particle size of the sample for chemical analysis, refer to Practices E 32.

[1] These methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.01 on Iron, Steel, and Ferroalloys.

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[2] *Annual Book of ASTM Standards*, Vol 01.02.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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

[5] Withdrawn from publication, see 1997 *Annual Book of ASTM Standards*, Vol 03.05.

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7. Rounding Off Calculated Values

7.1 Calculated values shall be rounded off to the desired number of places as directed in 3.4 to 3.6 of Practice E 29.

8. Interlaboratory Studies

8.1 These methods have been evaluated in accordance with Practice E 173, unless otherwise noted in the precision and bias section.

COLUMBIUM, TANTALUM, AND TITANIUM BY THE ION-EXCHANGE METHOD

9. Scope

9.1 These methods cover the determination of columbium, tantalum, and titanium in ferrocolumbium in concentrations from 40 to 75 %, 0.25 to 7 %, and 0.05 to 5.0 %, respectively.

10. Summary of Test Method

10.1 The sample is dissolved in a hydrochloric-hydrofluoric acid mixture and transferred to an anion-exchange column. Titanium, iron, and other elements are eluted with an ammonium chloride-hydrochloric-hydrofluoric acid mixture. This eluate is treated with boric acid and cupferron, and the precipitate, containing the titanium, is ignited, fused with potassium hydrogen sulfate, and leached in dilute sulfuric acid. The titanium is oxidized to the yellow pertitanate with hydrogen peroxide. Photometric measurement is made at approximately 410 nm. Columbium is removed by eluting with an ammonium chloride-hydrofluoric acid mixture. Tantalum is removed by eluting with an ammonium chloride-ammonium fluoride solution adjusted to a pH of 5 to 6. The eluates are treated with the boric acid to complex the fluorides, and each of the elements, columbium and tantalum, is precipitated with cupferron, ignited, and weighed as the pentoxide. For tantalum in concentrations below 1 %, zirconium is added as a gatherer in the cupferron separation and the tantalum is converted to the pyrogallol complex. Photometric measurement is made at approximately 420 nm.

11. Interferences

11.1 Any bismuth present will appear in the tantalum fraction, but this element is seldom present in concentrations greater than 0.005 % in this ferroalloy. Trivalent antimony, if present, is eluted with the titanium and precipitated with cupferron, but it does not interfere in the photometric method for titanium.

12. Apparatus

12.1 *Ion-Exchange Columns*—The columns must be constructed of polystyrene tubing approximately 300-mm long and 25 mm in inside diameter. A suitable column can be prepared as follows: Insert a waxed, No. 5 rubber stopper containing a 5-mm hole into the bottom of the polystyrene tube. Insert into the hole and flush with the upper surface of the stopper a 150-mm length of polystyrene tubing, having a 5-mm outside diameter and a 2-mm bore. Attach another 150-mm length of this tubing to the smaller tube with an approximately 50-mm length of polyvinyl tubing,[6] and control the flow rate by a hosecock on the polyvinyl tubing.

12.1.1 If a number of determinations are to be made, it is convenient to arrange the columns so that they can be operated with a minimum of attention. Plastic columns equipped with fittings of polystyrene have been developed for such an assembly.[7] Inlet and outlet tubes are polyethylene; flexible connections, where necessary, are of polyvinyl tubing. The flow rate is controlled by hosecocks on these flexible connections. The system must be carefully assembled and checked to avoid possible leakage of the solutions containing hydrofluoric acid.

12.2 *Plastic Ware*—Polyethylene, polypropylene, or TFE-fluorocarbon.

12.2.1 *Bottles*, 250-mL and 1-L capacity.

12.2.2 *Graduated Cylinders*, 50 and 250-mL capacity.

12.2.3 *Griffin-Form Beakers and Covers*, 250 and 600-mL, and 1-L capacity.

13. Reagents

13.1 *Ammonium Chloride Solution* (240 g/L)—Dissolve 480 g of ammonium chloride (NH_4Cl) in 1600 mL of water by warming, cool, dilute to 2 L, and mix. Filter, if necessary. Use this stock solution to prepare the solutions described in 13.2-13.4.

13.2 *Ammonium Chloride-Ammonium Fluoride Neutral Mixture*—Transfer 600 mL of the NH_4Cl solution and 40 mL of HF to a plastic beaker. Adjust the pH to 5 to 6 with NH_4OH (approximately 80 to 85 mL will be required), dilute to 1 L with water, and mix.

NOTE 1—This solution must be prepared with care. If the pH is too low, the volume specified will not completely elute the tantalum; if the pH is too high, tantalum will precipitate in the column, thus leading to error in the determinations being run as well as the one which follows.

13.3 *Ammonium Chloride-Hydrochloric-Hydrofluoric Acid Mixture*—Transfer 240 mL of the NH_4Cl solution, 200 mL of HF and 150 mL of HCl to a plastic bottle. Dilute to 1 L with water, and mix.

13.4 *Ammonium Chloride-Hydrofluoric Acid Mixture*—Transfer 600 mL of the NH_4Cl solution and 40 mL of HF to a plastic bottle. Dilute to 1 L with water, and mix.

13.5 *Ammonium Nitrate Wash Solution* (20 g/L)—Dissolve 20 g of ammonium nitrate (NH_4NO_3) in water, and dilute to 1 L.

13.6 *Boric Acid* (H_3BO_3).

13.7 *Cupferron Solution* (60 g/L)—Reagent No. 115. This solution should be prepared fresh as needed and cooled to 5°C before use.

13.8 *Cupferron Wash Solution*—Add 25 mL of cupferron solution (13.7) to 975 mL of cold HCl (1 + 9), and mix. Prepare as needed.

13.9 *Hydrochloric-Hydrofluoric Acid Mixture*—Add 250 mL of HCl to 300 mL of water, add 200 mL of HF, dilute to 1 L with water, and mix.

[6] Tygon-R tubing has been found satisfactory for this purpose.

[7] Columns available from Ledoux and Co., Inc., Teaneck, NJ, have been found satisfactory for this purpose.

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