Designation: E367 – 22

Standard Test Methods for Chemical Analysis of Ferroniobium[1]

This standard is issued under the fixed designation E367; 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 test methods cover the chemical analysis of ferroniobium having chemical compositions within the following limits:

Element	Composition, %
Aluminum	2.00 max
Carbon	0.30 max
Chromium	2.00 max
Cobalt	0.25 max
Lead	0.01 max
Manganese	3.00 max
Niobium	40.00 to 75.00
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 test methods appear in the following order:

	Sections
Separation of Niobium, Tantalum, and Titanium by the Ion-Exchange Test Method	15 and 16
Titanium by the Spectrophotometric Test Method [0.05 % to 5.0 %]	17 – 21
Niobium by the Gravimetric Test Method [40 % to 75 %]	22 – 23
Tantalum by the Gravimetric Test Method [1 % to 7 %]	24 – 25
Tantalum by the Spectrophotometric Test Method [0.25 % to 1 %]	26 – 30

1.3 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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 consult and establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 6, and specific warning statements in 11.1.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A550 Specification for Ferrocolumbium (Ferroniobium)

D1193 Specification for Reagent Water

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

E32 Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1997)[3]

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 For definition of terms used in this test method, refer to Terminology E135.

4. Significance and Use

4.1 These test methods for the chemical analysis of ferroniobium alloy are primarily intended to test such materials

[1] These test 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] 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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

for compliance with compositional specifications such as Specification A550. It is assumed that all who use these test 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.

5. Apparatus, Reagents, and Spectrophotometric Practice

5.1 Apparatus, standard solutions, and other reagents required for each determination are listed in separate sections preceding the procedure. Spectrophotometers shall conform to the requirements prescribed in Practice E60. (Note 1.)

NOTE 1—In these methods, cells utilized to contain the reference material and sample solutions in spectrophotometers are referred to as "absorption cells." Please note that the radiant energy passed through the cells can be measured as absorbance or transmittance. These methods refer to absorbance measurements. Refer to Practice E60 for details.

5.2 Spectrophotometric practice prescribed in these test methods shall conform to Practice E60.

5.3 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or Type II of Specification D1193. Type III or Type IV may be used if they effect no measurable change in the blank or sample.

5.4 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6. Hazards

6.1 For precautions to be observed in the use of certain reagents in these test methods, refer to Practices E50.

6.2 Specific warning statements are given in 11.1.

7. Sampling

7.1 For procedures to sample the material, and particle size requirements of the sample, refer to Practices E32.

8. Rounding Calculated Values

8.1 Rounding of test results obtained using this test method shall be performed as directed in Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification..

9. Interlaboratory Studies

9.1 These test methods have been evaluated in accordance with Practice E173, unless otherwise noted in the precision and bias section. Practice E173 has been replaced by Practice E1601. The Reproducibility *R2* corresponds to the Reproducibility Index *R* of Practice E1601. The Repeatability *R1* of Practice E173 corresponds to the Repeatability Index *r* of Practice E1601.

10. Scope

10.1 These test methods cover the determination of niobium, tantalum, and titanium in ferroniobium from 40 % to 75 %, 0.25 % to 7 %, and 0.05 % to 5.0 %, respectively.

11. Summary of Test Method

11.1 The sample is dissolved in a HCl-HF acid mixture and transferred to an anion-exchange column. Titanium, iron, and other elements are eluted with a NH_4Cl-HCl-HF solution. This eluate is treated with boric acid (H_3BO_3) and cupferron, and the precipitate, containing the titanium, is ignited, fused with potassium hydrogen sulfate, and leached in dilute H_2SO_4. The titanium is oxidized to the yellow pertitanate with hydrogen peroxide. Spectrophotometric absorbance measurement is made at 410 nm. Niobium is removed by eluting with a NH_4Cl-HF solution. Tantalum is removed by eluting with a NH_4Cl-NH_4F solution adjusted to a pH of 5 to 6. The eluates are treated with the H_3BO_3 to complex the fluorides, and each of the elements, niobium and tantalum, is precipitated with cupferron, ignited, and weighed as the pentoxide. For tantalum below 1 %, zirconium is added as a gatherer in the cupferron separation and the tantalum is converted to the pyrogallol complex. Spectrophotometric absorbance measurement is made at 420 nm. (**Warning—**HF produces very serious burns which may or may not be painful on first contact. Such burns often damage bone and other tissue within the body. Standard procedure is to use gloves and protective clothing when handling this reagent. After the material is added, the closed container, gloves, and all surfaces that may later be touched are rinsed with large quantities of water. Even one drop of HF on the skin or fingernail must receive immediate first-aid and medical attention should be promptly sought.)

12. Interferences

12.1 Any bismuth present will appear in the tantalum fraction, but this element is seldom present 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 spectrophotometric test method for titanium.

13. Apparatus

13.1 *Ion-Exchange Columns*—The columns must be constructed of polystyrene tubing approximately 300 mm in length 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

[4] Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.