Designation: E367 – 16

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 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 whoever uses this standard to consult and establish appropriate safety and health 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.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A550 Specification for Ferrocolumbium (Ferroniobium)

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 1998)[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 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

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[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.

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.

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 Calculated values shall be rounded to the desired number of places as directed in Practice E29.

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 50-mm length of polyvinyl tubing,[4] and control the flow rate by a hosecock on the polyvinyl tubing.

13.1.1 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. 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 HF.

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

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

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

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

14. Reagents

14.1 *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 14.2 – 14.4.

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

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

14.2.1 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.

14.3 *Ammonium Chloride-Hydrochloric-Hydrofluoric Acid Solution*—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.

14.4 *Ammonium Chloride-Hydrofluoric Acid Solution*—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.

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

14.6 *Boric Acid* (H_3BO_3).

14.7 *Cupferron Solution* (60 g/L)—Dissolve 6 g of cupferron in 80 mL of cold water, dilute to 100 mL, and filter. This solution should be prepared fresh as needed and cooled to 5 °C before use.

14.8 *Cupferron Wash Solution*—Add 25 mL of cupferron solution (14.7) to 975 mL of cold HCl (1 + 9), and mix. Prepare as needed.

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

14.10 *Hydrogen Peroxide* (H_2O_2), 30 %.

14.11 *Ion-Exchange Resin*—Strongly basic anion-exchange resin, 200 mesh to 400 mesh, 8 % to 10 % divinyl-benzene cross linkage.[5] Since the mesh size of the resin may vary considerably from lot to lot, air-dry the resin and pass it through a No. 270 (53-µm) sieve (Note 2). Most of the fines are removed from the fraction passing the No. 270 sieve as follows: Prepare a suspension of the resin in HCl (1 + 9). Allow the coarser fraction to settle 10 min to 15 min and remove the fines by decantation. Repeat the process several times until most of the very fine material has been removed from the suspension.

NOTE 2—Material retained on the No. 270 sieve may be used for other purposes.

14.12 *Oxalate-Citrate-Sulfuric Acid Solution*—Dissolve 35 g of ammonium oxalate ($(NH_4)_2C_2O_4 \cdot H_2O$) and 35 g of diammonium hydrogen citrate ($(NH_4)_2HC_8H_5O_7$) in 1 L of H_2SO_4 (1 + 39).

14.13 *Pyrogallol* (C_6H_3-1,2,3-$(OH)_3$).

14.14 *Sodium Hydroxide Solution* (100 g/L)—Dissolve 20 g of NaOH in 150 mL of water, cool, dilute to 200 mL, and mix. Store in a plastic bottle.

14.15 *Tantalum, Standard Solution* (1 mL = 0.500 mg Ta)—Transfer 0.1221 g of tantalum pentoxide (Ta_2O_5) to a platinum crucible. Add 2.5 g of potassium hydrogen sulfate ($KHSO_4$) and heat to fuse the oxide. Dissolve the cooled melt in warm oxalate-citrate-sulfuric acid solution. Transfer to a 200-mL volumetric flask, cool, dilute to volume with oxalate-citrate-sulfuric acid solution and mix.

14.16 *Titanium, Standard Solution* (1 mL = 0.100 mg Ti)—Transfer 0.0834 g of titanium dioxide (TiO_2) to a platinum crucible. Add 1 g of $KHSO_4$, and heat to fuse the oxide. Cool, and dissolve the melt in 50 mL of warm H_2SO_4 (1 + 9). Cool, transfer to a 500-mL volumetric flask, dilute to volume with H_2SO_4 (1 + 9), and mix.

14.17 *Zirconium Solution* (1 mL = 1 mg Zr)—Dissolve 0.5 g of zirconium metal in 10 mL of HF in a plastic bottle, and dilute to 500 mL. An equivalent amount of zirconyl chloride may be substituted for the zirconium metal.

SEPARATION OF NIOBIUM, TANTALUM, AND TITANIUM BY THE ION-EXCHANGE TEST METHOD

15. Preparation of Ion-Exchange Column

15.1 Place a 6-mm to 10-mm layer of acid-resistant poly-(vinyl chloride) plastic fiber in the bottom of the column. Add the resin suspension in small portions to obtain a settled bed of the resin 150-mm to 180-mm in height. Wash the column with approximately 100 mL of HNO_3 (1 + 9), and then perform three elution cycles with alternate additions of 100 mL of HCl (1 + 9) and 100 mL of HCl (3 + 1) to remove the remainder of the fines. Finally, wash the column with 200 mL of HCl (1 + 3) to a level about 20 mm above the resin.

NOTE 3—Resin columns prepared in this way have been used for several years; the only maintenance may be to empty and refill the column with the resin charge if the flow rate becomes excessively slow due to packing.

16. Preparation of Test Solutions

16.1 Transfer a 0.5-g sample, weighed to the nearest 0.1 mg, to a 250-mL plastic beaker. Add 40 mL of the HCl-HF solution. Place a plastic cover on the beaker, and heat gently. After the reaction ceases, add HNO_3 dropwise until the solution clears (Note 4). Digest on the steam bath for 20 min to 30 min to remove nitrous oxide fumes. Rinse the plastic cover and wall of the beaker with the HCl-HF solution, and dilute to 70 mL with the HCl-HF solution.

NOTE 4—The addition of HNO_3 should be kept to a minimum because of its strong replacing power for niobium on the exchange column. Approximately 6 drops to 8 drops will be required.

16.2 Transfer 50 mL of HCl-HF solution to the column in 5-mL to 10-mL increments. Drain the acid to a level 100 mm above the resin bed, collecting the eluate in a 600-mL plastic beaker. Transfer the sample solution in 5-mL to 10-mL increments to the column. As the sample solution moves down the column, continue to add the small increments until all of the solution has been transferred. Wash the beaker four times or five times with 4-mL portions of the HCl-HF solution, transferring the washings to the column. Wash the sides of the column with 10 mL to 15 mL of the HCl-HF solution followed by several washings with the NH_4Cl-HCl-HF solution.

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[5] Dowex I anion-exchange resin has been found satisfactory. Comparable results may not be obtained with other resins.

16.3 Pass a total of 300 mL of the NH_4Cl-HCl-HF solution through the column at a flow rate of approximately 100 mL/h to 125 mL/h. Allow the solution to drain to the top of the resin. Remove the beaker containing the first fraction and reserve this solution for the determination of titanium. Replace the beaker with another 600-mL plastic beaker.

16.4 Wash the sides of the column with four or five portions (a total of about 25 mL) of the NH_4Cl-HF solution, allowing the solution to drain to the top of the resin each time. Pass a total of 300 mL of the NH_4Cl-HF solution through the column at the flow rate specified in 16.3 (Note 5). Remove the beaker containing the second fraction and reserve this solution for the determination of niobium. Replace the beaker with another 600-mL plastic beaker.

NOTE 5—This point in the preparation of the test solutions provides a convenient and satisfactory place to stop, for example overnight, if the elutions otherwise cannot be carried through as a continuous operation.

16.5 Wash the sides of the column with five or six 5-mL portions of the NH_4Cl-NH_4F neutral solution. Pass a total of 350 mL of the NH_4Cl-NH_4F neutral solution through the column, at the flow rate specified in 16.3. Remove the beaker containing the third fraction and reserve this solution for the determination of tantalum as directed in Section 24 or Section 29. Prepare the column for the next sample by adding 50 mL of HCl (1 + 3) in 10-mL increments and discarding the effluents.

TITANIUM BY THE SPECTROPHOTOMETRIC TEST METHOD

17. Concentration Range

17.1 The recommended concentration range is 0.100 mg to 2.50 mg of titanium for each 100 mL of solution, using a 2-cm cell.

NOTE 6—This test method has been written for a cell having a 2-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

18. Stability of Color

18.1 The color is stable for at least 2 h.

19. Preparation of Calibration Curve

19.1 *Calibration Solutions*—Using pipets, transfer (1, 5, 10, 15, and 25) mL of titanium solution (1 mL = 0.100 mg Ti) to 100-mL volumetric flasks, dilute to approximately 80 mL with H_2SO_4 (1 + 9), and mix. Proceed as directed in 19.3.

19.2 *Reference Solutions*—Add approximately 80 mL of H_2SO_4 (1 + 9) to a 100-mL volumetric flask. Proceed as directed in 19.3.

19.3 *Color Development*—Add 1.0 mL of H_2O_2, dilute to volume with H_2SO_4 (1 + 9), and mix.

19.4 *Spectrophotometry:*

19.4.1 *Multiple-Cell Spectrophotometer*—Measure the cell correction, using absorption cells with a 2-cm light path and a light band centered at 410 nm. Using the test cell, take the spectrophotometric absorbance readings of the calibration solutions.

19.4.2 *Single-Cell Spectrophotometer*—Transfer a suitable portion of the reference solution to an absorption cell with a 2-cm light path and adjust the spectrophotometer to the initial setting, using a light band centered at 410 nm. While maintaining this adjustment, take the spectrophotometric absorbance readings of the calibration solutions.

19.5 *Calibration Curve*—Plot the net spectrophotometric absorbance readings of the calibration solutions against milligrams of titanium per 100 mL of solution. Follow the instrument manufacturer's instructions for generating the calibration curve.

20. Procedure

20.1 Transfer the first fraction containing the titanium and iron reserved as directed in 16.3 to a 1500-mL beaker containing 50 g of H_3BO_3 dissolved in 700 mL of warm water. Add 125 mL of HCl and cool to 5 °C.

20.2 Add 50 mL of cupferron solution slowly while stirring the solution. Add filter paper pulp, stir well, and allow to stand for 10 min to 15 min. Filter, using moderate suction, on a Buchner funnel, using double thickness 9-cm, low-ash, fine filter paper precoated with a little filter paper pulp. Transfer the precipitate to the funnel, clean the beaker with a piece of moistened filter paper and add this to the funnel. Wash the paper and precipitate with 400 mL of cold (5 °C) cupferron wash solution.

20.3 Transfer the paper and precipitate to a porcelain crucible, and ignite at 550 °C to 600 °C until the carbon is destroyed.

20.4 If vanadium is present, fuse the ignited oxides with 2 g to 3 g of $KHSO_4$, cool, and dissolve the melt in 30 mL of HCl (1 + 9) in a 150-mL beaker. Add NaOH solution until alkaline to litmus and add 5 mL in excess. Boil for 3 min, and then add some filter paper pulp. Filter using a medium filter paper and wash the precipitate with the NH_4NO_3 wash solution. Transfer the paper and precipitate to a porcelain crucible and ignite at 550 °C to 600 °C until the carbon is destroyed.

20.5 Fuse the ignited oxides obtained in 20.3 and 20.4 with 2 g to 3 g of $KHSO_4$ and leach in 30 mL of H_2SO_4 (1 + 9).

20.6 Transfer the solution, selecting the size of the volumetric flask as follows:

Titanium, %	Initial Dilution, mL	Aliquot Volume, mL	Equivalent Sample Weight in Aliquot Volume, g
0.05 to 1.25	50	20	0.2
1.24 to 2.50	50	10	0.1
2.49 to 5.00	100	10	0.05

(Filter through a fine filter paper into the appropriate size volumetric flask if the solution is not clear and wash with H_2SO_4 (1 + 9).) Dilute to volume with H_2SO_4 (1 + 9) and mix.

20.7 Transfer an aliquot to a 100-mL volumetric flask, selecting the aliquot volume in 20.6. Dilute to approximately 80 mL with H_2SO_4 (1 + 9). Proceed as directed in 20.8.

20.8 *Color Development*—Proceed as directed in 19.3.

20.9 *Spectrophotometry*—Take the spectrophotometric absorbance reading of the test solution as directed in 19.4.

21. Calculation

21.1 Convert the net spectrophotometric absorbance reading of the test solution to milligrams of titanium by means of the calibration curve. Calculate the percentage of titanium as follows:

$$\text{Titanium, \%} = A/(B \times 10) \quad (1)$$

where:

A = milligrams of titanium found in 100 mL of final test solution, and
B = grams of sample represented in 100 mL of final test solution.

NIOBIUM BY THE GRAVIMETRIC TEST METHOD

22. Procedure

22.1 To the second fraction containing the niobium (see 16.4), add 15 g of H_3BO_3, 75 mL of HCl, and 95 mL of water. Heat at 30 °C to 35 °C until the H_3BO_3 is dissolved. Cool to 5 °C and proceed as directed in 20.2 using 65 mL of cupferron solution.

22.2 Transfer the precipitate and paper to a weighed platinum crucible, and ignite at a low temperature until the carbon is destroyed. Finally ignite to constant weight at 1200 °C and weigh as niobium pentoxide (Nb_2O_5).

NOTE 7—Reagent blanks usually amount to less than 0.5 mg and hence are considered to be offset by the few tenths of a milligram of earth acid lost in the precipitation.

23. Calculation

23.1 Calculate the percentage of niobium as follows:

$$\text{Niobium, \%} = (A \times 0.699/B) \times 100 \quad (2)$$

where:

A = grams of Nb_2O_5, and
B = grams of sample used.

TANTALUM BY THE GRAVIMETRIC TEST METHOD (GREATER THAN 1 %)

24. Procedure

24.1 To the third fraction containing the tantalum (see 16.5), add 9 g of H_3BO_3, 95 mL of HCl, and 85 mL of water. Heat at 30 °C to 35 °C until the H_3BO_3 is dissolved. Cool to 5 °C and proceed as directed in 20.2 using 65 mL of cupferron solution. Proceed as directed in 22.2. Weigh as tantalum pentoxide (Ta_2O_5).

25. Calculation

25.1 Calculate the percentage of tantalum as follows:

$$\text{Tantalum, \%} = (A \times 0.819/B) \times 100 \quad (3)$$

where:

A = grams of Ta_2O_5, and
B = grams of sample used.

TANTALUM BY THE SPECTROPHOTOMETRIC TEST METHOD (LESS THAN 1 %)

26. Concentration Range

26.1 The recommended concentration range is 1 mg to 5 mg of tantalum for each 100 mL of solution, using a 1-cm cell.

NOTE 8—This test method has been written for cells 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.

27. Stability of Color

27.1 The color is stable for at least 1 h.

28. Preparation of Calibration Curve

28.1 *Calibration Solutions*—Using pipets, transfer (2, 4, 7, and 10) mL of tantalum solution (1 mL = 0.50 mg Ta) to 100-mL volumetric flasks and dilute to approximately 80 mL with the oxalate-citrate-sulfuric acid solution, and mix. Proceed as directed in 28.3.

28.2 *Reference Solution*—Transfer 1 g of $KHSO_4$ to a 100-mL volumetric flask, add 80 mL of oxalate-citrate-sulfuric acid solution and proceed as directed in 28.3.

28.3 *Color Development*—Add 12 g of pyrogallol and shake to dissolve (Note 9). Dilute to volume with the oxalate-citrate-sulfuric acid solution, and mix.

NOTE 9—A mechanical shaker is desirable since dissolution time is about 10 min with continuous shaking.

28.4 *Spectrophotometry:*

28.4.1 *Multiple-Cell Spectrophotometer*—Measure the cell correction using absorption cells with a 1-cm light path and a light band centered at 420 nm. Using the test cell, take the spectrophotometric absorbance readings of the calibration solutions.

28.4.2 *Single-Cell Spectrophotometer*—Transfer a suitable portion of the reference solution to an absorption cell with a 1-cm light path and adjust the spectrophotometer to the initial setting, using a light band centered at 420 nm. While maintaining this adjustment, take the spectrophotometric absorbance readings of the calibration solutions.

28.4.3 *Calibration Curve*—Plot the net spectrophotometric absorbance readings of the calibration solutions against milligrams of tantalum per 100 mL of solution. Follow the instrument manufacturer's instructions for generating the calibration curve.

29. Procedure

29.1 *Test Solution*—To the third fraction containing the tantalum (see 16.5), add 25 mL of zirconium solution (1 mL = 1 mg Zr), 9 g of H_3BO_3, 95 mL of HCl, and 85 mL of water. Cool to 5 °C and proceed as directed in 20.2. Ignite at a temperature just sufficient to destroy carbonaceous material. Fuse the oxide with 1 g of $KHSO_4$, cool, and dissolve the melt in 80 mL of oxalate-citrate-sulfuric acid solution. Transfer the solution to a 100-mL volumetric flask.

29.1.1 If more than 5 mg of tantalum is present, transfer the dissolve melt to a 100-mL volumetric flask, dilute to volume with oxalate-citrate-sulfuric acid solution, and take a suitable aliquot.

TABLE 1 Statistical Information

	Test Specimen	Nb Found, %	Repeatability (R_1, Practice E173)[A]	Reproducibility (R_2, Practice E173)[A]
1.	Ferroniobium (NIST 340, 57.51 Nb)	57.51	0.16	0.26
2.		65.40	0.29	0.11
		Ta Found, %		
1.	Ferroniobium (NIST 340, 3.73 Ta)	3.71	0.16	0.26
2.		0.47	0.04	0.06
		Ti Found, %		
1.	Ferroniobium (NIST 340, 0.89 Ti)	0.89	0.10	0.07
2.		0.046	0.005	0.010

[A] This test method has been evaluated in accordance with Practice E173 (discontinued 1997). The Reproducibility R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. The Repeatability R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

29.2 *Reference Solution*—Proceed as directed in 28.2.

29.3 *Color Development*—Proceed as directed in 28.3.

29.4 *Spectrophotometry*—Take the spectrophotometric absorbance reading of the test solution as directed in 28.4.

30. Calculation

30.1 Convert the net spectrophotometric absorbance reading of the test solution to milligrams of tantalum by means of the calibration curve. Calculate the percentage of tantalum as follows:

$$\text{Tantalum, \%} = A/(B \times 10) \quad (4)$$

where:

A = milligrams of tantalum found in 100 mL of final test solution, and

B = grams of sample represented in 100 mL of final test solution.

31. Precision and Bias

31.1 *Precision*—Nine laboratories cooperated in testing this test method and obtained the data summarized in Table 1. Samples with compositions covering the limits of the scope were not available for testing. The user is cautioned to verify, by the use of reference materials, if available, that the precision and accuracy of this test method is adequate for the contemplated use.

31.2 *Bias*—The accuracy of this test method has been deemed satisfactory based upon the data for the certified reference material in Table 1. Users are encouraged to use this or similar reference materials to verify that the test method is performing accurately in their laboratories.

32. Keywords

32.1 chemical analysis; ferroniobium; niobium; tantalum; titanium