Designation: ~~E351 – 93 (Reapproved 2006)~~ E351 – 13

Standard Test Methods for Chemical Analysis of Cast Iron—All Types[1]

This standard is issued under the fixed designation E351; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the chemical analysis of pig iron, gray cast iron (including alloy and austenitic), white cast iron, malleable cast iron, and ductile ~~(nodular~~(nodular)~~)~~ iron having chemical compositions within the following limits:

~~Element~~ Element	~~Concentration Range, %~~ Composition Range, %
~~Aluminum[4]~~	~~0.003 to 0.50~~
Aluminum	0.003 to 0.50
Antimony	0.005 to 0.03
~~Arsenic[4]~~	~~0.02 to 0.10~~
Arsenic	0.02 to 0.10
~~Bismuth[4]~~	~~0.001 to 0.03~~
Bismuth	0.001 to 0.03
~~Boron[4]~~	~~0.001 to 0.10~~
Boron	0.001 to 0.10
~~Cadmium[4]~~	~~0.001 to 0.005~~
Cadmium	0.001 to 0.005
Carbon	1.25 to 4.50
~~Cerium[4]~~	~~0.005 to 0.05~~
Cerium	0.005 to 0.05
Chromium	0.01 to 30.00
~~Cobalt[4]~~	~~0.01 to 4.50~~
Cobalt	0.01 to 4.50
Copper	0.03 to 7.50
~~Lead[4]~~	~~0.001 to 0.15~~
Lead	0.001 to 0.15
Magnesium	0.002 to 0.10
Manganese	0.06 to 2.50
Molybdenum	0.01 to 5.00
Nickel	0.01 to 36.00
Phosphorus	0.01 to 0.90
~~Selenium[4]~~	~~0.001 to 0.06~~
Selenium	0.001 to 0.06
~~Silicon~~	~~0.10 to 6.0~~
Silicon	0.10 to 6.0
Sulfur	0.005 to 0.25
~~Tellurium[4]~~	~~0.001 to 0.35~~
Tellurium	0.001 to 0.35
~~Tin[4]~~	~~0.001 to 0.35~~
Tin	0.001 to 0.35
~~Titanium[4]~~	~~0.001 to 0.20~~
Titanium	0.001 to 0.20
~~Tungsten[4]~~	~~0.001 to 0.20~~
Tungsten	0.001 to 0.20
~~Vanadium[4]~~	~~0.005 to 0.50~~
Vanadium	0.005 to 0.50
~~Zinc[4]~~	~~0.005 to 0.20~~
Zinc	0.005 to 0.20

1.2 The test methods in this standard are contained in the sections indicated below:

[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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1.3 Procedures for the determination of ~~several elements~~ carbon and sulfur not included in these test methods can be found in Test Methods ~~E30 and Test Methods~~ E1019.

1.4 Some of the ~~concentration~~composition ranges given in 1.1 are too broad to be covered by a single method and therefore this standard contains multiple methods for some elements. The user must select the proper method by matching the information given in the Scope and Interference sections of each method with the composition of the alloy to be analyzed.

1.5 The values stated in SI units are to be regarded as standard. ~~In some cases, exceptions allowed in Practice E380 are also used.~~

1.6 *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.* Specific hazards statements are given in Section ~~5~~6 and in special "Warning" paragraphs throughout these Methods.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

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

~~E30 Test Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron.~~[5]

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]

E350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron

E352 Test Methods for Chemical Analysis of Tool Steels and Other Similar Medium- and High-Alloy Steels

E353 Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys

E380 Practice for Use of the International System of Units (SI) ~~(Modernized~~ (the Modernized Metric System) (Withdrawn 1997)[3]

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques

E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry (Withdrawn 2004)[3]

E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition

2.2 *Other Document:*[4]

ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility for Inter-Laboratory Tests

3. Terminology

3.1 For definitions of terms used in these test methods, refer to Terminology E135.

4. Significance and Use

4.1 These test methods for the chemical analysis of metals and alloys are primarily intended as referee methods to test such materials for compliance with compositional specifications, particularly those under the jurisdiction of ASTM Committee A04 on Iron Castings. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory

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

~~The last approved version of this historical standard is referenced on www.astm.org.~~

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory under appropriate quality control practices such as those described in Guide E882.

5. Apparatus, Reagents, and Instrumental Practices

5.1 *Apparatus*—Specialized apparatus requirements are listed in the Apparatus section in each method. ~~In some cases reference may be made to Practices E50.~~

5.2 *Reagents:*

5.2.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, ~~all reagents used in these test methods shall~~ it is intended that all reagents conform to the ~~Reagent Grade Specifications of the American Chemical Society.~~specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[5] Other ~~chemicals~~grades may be used, provided it is first ascertained that ~~they are~~ the reagent is of sufficiently high purity to permit ~~their~~its use without ~~adversely affecting the expected performance of the determination, as indicated in the section on "Precision and Bias."~~lessening the accuracy of the determination.

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

5.3 *~~Photometric~~Spectrophotometric Practice*—~~Photometric~~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 and equipment in these methods, refer to Practices E50.

7. Sampling

7.1 For procedures for sampling the material, reference shall be made to Practice E1806.

8. Interlaboratory Studies and Rounding Calculated Values

8.1 These test methods have been evaluated ~~using~~ in accordance with Practice E173 (withdrawn 1997) or ISO 5725. The Reproducibility R2 of E173 corresponds to the Reproducibility Index R of E1601. The Repeatability R1 of E173 corresponds to the Repeatability Index r of E1601.

8.2 Calculated values shall be rounded to the desired number of places ~~as directed in 3.4 to 3.6~~in accordance with the Rounding Method of Practice E29.

MANGANESE BY THE METAPERIODATE ~~PHOTOMETRIC~~SPECTROPHOTOMETRIC METHOD

9. Scope

9.1 This test method covers the determination of manganese in ~~concentrations~~compositions from 0.10 % to 2.00 %.

10. Summary of Method

10.1 Manganous ions are oxidized to permanganate ions by reaction with metaperiodate ions. Solutions of the samples are fumed with ~~perchloric~~$HClO_4$ ~~acid~~ so that the effect of metaperiodate ion is limited to the oxidation of manganese. ~~Photometric~~Spectrophotometric measurement is made at approximately 545 nm.

11. Concentration Range

11.1 The recommended concentration range is 0.15 mg to 0.8 mg of manganese per 50 mL of solution, using a 1-cm cell (Note 1) and a spectrophotometer with a band width of 10 nm or less.

NOTE 1—This method has been written for cells having a 1-cm light path and a narrow-band instrument. The concentration range depends upon band width and spectral region used as well as cell optical path length. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

12. Stability of Color

12.1 The color is stable for at least 24 h.

[5] *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.

13. Interferences

13.1 The elements ordinarily present do not interfere. ~~Perchloric~~$HClO_4$ ~~acid~~ treatment, which is used in the procedure, yields solutions which can be highly colored due to the presence of Cr (VI) ions. Although these ions and other colored ions in the sample solution undergo no further change in color quality upon treatment with metaperiodate ion, the following precautions must be observed when filter ~~photometers~~spectrophotometers are used: Select a filter with maximum transmittance between 545 nm and 565 nm. The filter must transmit not more than 5 % of its maximum at a wavelength shorter than 530 nm. The band width of the filter should be less than 30 nm when measured at 50 % of its maximum transmittance. Similar restrictions apply with respect to the wavelength region employed when other wide-band instruments are used.

13.2 The spectral transmittance curve of permanganate ions exhibits two useful minima, one at approximately 526 nm, and the other at 545 nm. The latter is recommended when a narrow-band spectrophotometer is used.

14. Reagents

14.1 *Manganese, Standard Solution* (1 mL = 0.032 mg Mn)—Transfer the equivalent of 0.4000 g of assayed, high-purity manganese (purity: 99.99 % minimum), to a 500-mL volumetric flask and dissolve in 20 mL of HNO_3 by heating. Cool, dilute to volume, and mix. Using a pipet, transfer 20 mL to a 500-mL volumetric flask, dilute to volume, and mix.

14.2 *Nitric-Phosphoric Acid Mixture*—Cautiously, while stirring, add 100 mL of HNO_3 and 400 mL of H_3PO_4 to 400 mL of water. Cool, dilute to 1 L, and mix. Prepare fresh as needed.

14.3 *Potassium Metaperiodate Solution* (7.5 g/L)—Dissolve 7.5 g of potassium metaperiodate (KIO_4) in 200 mL of hot HNO_3 (1 + 1), add 400 mL of H_3PO_4, cool, dilute to 1 L, and mix.

14.4 *Water, Pretreated with Metaperiodate*—Add 20 mL of KIO_4 solution to 1 L of water, mix, heat at not less than ~~90°C~~ 90 °C for 20 min to 30 min, and cool. Use this water to dilute solutions to volume that have been treated with KIO_4 solution to oxidize manganese, and thus avoid reduction of permanganate ions by any reducing agents in the untreated water. **Warning**—Avoid the use of this water for other purposes.

15. Preparation of Calibration Curve

15.1 *Calibration Solutions*—Using pipets, transfer ~~5, 10, 15, 20,~~ 5 mL, 10 mL, 15 mL, 20 mL, and 25 mL of manganese standard solution (1 mL = 0.032 mg Mn) to 50-mL borosilicate glass volumetric flasks, and, if necessary, dilute to approximately 25 mL. Proceed as directed in ~~14.3~~15.3.

15.2 *Reference Solution*—Transfer approximately 25 mL of water to a 50-mL borosilicate glass volumetric flask. Proceed as directed in ~~14.3~~15.3.

15.3 *Color Development*—Add 10 mL of KIO_4 solution, and heat the solutions at not less than ~~90°C~~ 90 °C for 20 min to 30 min (Note 2). Cool, dilute to volume with pretreated water, and mix.

NOTE 2—Immersing the flasks in a boiling water bath is a preferred means of heating them for the specified period to ensure complete color development.

15.4 ~~*Photometry:*~~*Spectrophotometry:*

15.4.1 *Multiple-Cell* ~~*Photometer—*~~*Spectrophotometer—*Measure the cell correction using the Reference Solution (~~14.2~~15.2) in absorption cells with a 1-cm light path and using a light band centered at approximately 545 nm. Using the test cell, take the ~~photometric~~spectrophotometric readings of the calibration solutions versus the Reference Solution (~~14.2~~15.2).

15.4.2 *Single-Cell* ~~*Photometer—*~~*Spectrophotometer—*Transfer a suitable portion of the Reference Solution (~~14.2~~15.2) to an absorption cell with a 1-cm light path and adjust the ~~photometer~~spectrophotometer to the initial setting, using a light band centered at approximately 545 nm. While maintaining this adjustment, take the ~~photometric~~spectrophotometric readings of the calibration solutions.

15.5 *Calibration Curve*—~~Plot the net photometric readings of the calibration solutions against milligrams of manganese per 50 mL of solution.~~Follow the instrument manufacturer's instructions for generating the calibration curve.

16. Procedure

16.1 *Test Solution:*

16.1.1 Select and weigh a sample in accordance with the following:

Maganese, %	Sample Weight, g	Tolerance in Sample Weight, mg	Dilution, mL
0.01 to 0.5	0.80	0.5	100
0.45 to 1.0	0.35	0.3	100
0.85 to 2.0	0.80	0.5	500

Transfer it to a 300-mL Erlenmeyer flask.

16.1.2 To dissolve samples that do not require HF, add 8 mL to 10 mL of HCl (1 + 1), and heat. Add HNO_3 as needed to hasten dissolution, and then add 3 mL to 4 mL in excess. When dissolution is complete, cool, then add 10 mL of $HClO_4$; evaporate to fumes to oxidize chromium, if present, and to expel HCl. Continue fuming until salts begin to separate. Cool, add 50 mL of water, and digest if necessary to dissolve the salts. Cool and transfer the solution to either a ~~100-~~100-mL or 500-mL volumetric flask as indicated in ~~15.1.1~~16.1.1. Proceed to ~~15.1.4~~16.1.4.

16.1.3 For samples whose dissolution is hastened by HF, treat them by adding 8 mL to 10 mL of HCl (1 + 1), and heating. Add HNO_3 and a few drops of HF as needed to hasten dissolution, and then add 3 mL to 4 mL of HNO_3. When dissolution is complete, cool, then add 10 mL of $HClO_4$, evaporate to fumes to oxidize chromium, if present, and to expel HCl. Continue fuming until salts begin to separate. Cool, add 50 mL of water, digest if necessary to dissolve the salts, cool, and transfer the solution to either a ~~100-~~100-mL or 500-mL volumetric flask as indicated in ~~15.1.1~~16.1.1.

16.1.4 Cool the solution to room temperature, dilute to volume, and mix. Allow insoluble matter to settle, or dry-filter through a coarse paper and discard the first 15 mL to 20 mL of the filtrate, before taking aliquots.

16.1.5 Using a pipet, transfer 20 mL aliquots, to two 50-mL borosilicate glass volumetric flasks. Treat one portion as directed in ~~15.3~~16.3. Treat the other portion as directed in ~~15.4.1~~16.4.1.

16.2 *Reagent Blank Solution*—Carry a reagent blank through the entire procedure using the same amounts of all reagents with the sample omitted.

16.3 *Color Development*—Proceed as directed in ~~14.3~~15.3.

16.4 *Reference Solutions:*

16.4.1 *Background Color Solution*—To one of the sample aliquots in a 50-mL volumetric flask, add 100 mL of ~~nitric-phosphoric~~HNO_3-H_3PO_4 ~~acid~~ mixture, and heat the solution at not less than ~~90°C~~ 90 °C for 20 min to 30 min (Note 2). Cool, dilute to volume (with untreated water), and mix.

16.4.2 *Reagent Blank Reference Solution*—Transfer the reagent blank solution (~~15.2~~16.2) to the same size volumetric flask as used for the test solutions and transfer the same size aliquots as used for the test solutions to two 50-mL volumetric flasks. Treat one portion as directed in ~~15.3~~16.3 and use as reference solution for test samples. Treat the other as directed in ~~15.4.1~~16.4.1 and use as reference solution for Background Color Solutions.

16.5 ~~*Photometry—*~~*Spectrophotometry—*Establish the cell corrections with the Reagent Blank Reference solution to be used as a reference solution for Background Color solutions. Take the ~~photometric~~spectrophotometric readings of the Background Color Solutions and the test solutions versus the respective Reagent Blank Reference Solutions as directed in ~~14.4~~15.4.

17. Calculation

17.1 Convert the net ~~photometric~~spectrophotometric reading of the test solution and of the background color solution to milligrams of manganese by means of the calibration curve. Calculate the percentage of manganese as follows:

~~$$Manganese, \% = (A - B)/(C \times 10) \quad (1)$$~~

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

where:

A = manganese, mg, found in 50 mL of the final test solution,
B = apparent manganese, mg, found in 50 mL of the final background color solution, and
C = sample weight, g, represented in 50 mL of the final test solution.

18. Precision and Bias

18.1 *Precision*—Nine laboratories cooperated in testing this method and obtained the data summarized in Table 1. Although a

TABLE 1 Statistical Information—Manganese by the Metaperiodate ~~Photometric~~Spectrophotometric Method

~~colwidth="0.4in"/COLSPEC~~

Test Specimen	Manganese Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
1. White cast iron (NIST 3a, 0.317 Mn)	0.318	0.006	0.017
2. Cast iron (NIST 4i, 0.793 Mn)	0.793	0.018	0.028
3. Cast iron (B.C.S. 236/2, 1.14 Mn)	1.15	0.03	0.06
4. White cast iron (NIST 1175, 1.64 Mn)	1.64	0.02	0.08
5. Low-alloy steel (NIST 100b, 1.89 Mn)	1.91	0.02	0.04

sample covered by this method with manganese ~~concentration~~composition of approximately 2.0 % was not available, the precision data for this ~~concentration~~composition should be similar to those obtained for material 5.

18.2 *Bias*—No information on the accuracy of this method is known. The accuracy of this method may be judged by comparing accepted reference values with the corresponding arithmetic average obtained by interlaboratory testing.

PHOSPHORUS BY THE MOLYBDENUM BLUE ~~PHOTOMETRIC~~SPECTROPHOTOMETRIC METHOD

19. Scope

19.1 This method covers the determination of phosphorus in ~~concentrations~~compositions from 0.02 % to 0.90 %.

20. Summary of Method

20.1 ~~See Section~~The sample ~~19 of Test Methods~~is dissolved in mixed ~~E350~~acids and the solution is fumed with $HClO_4$. Ammonium molybdate is added to react with the phosphorus to form the heteropoly phosphomolybdate. This species is then reduced with hydrazine sulfate to form the molybdenum blue complex. Spectrophotometric measurement is made at 650 nm or 825 nm, depending upon the concentration.

21. Concentration Range

21.1 ~~See Section~~The recommended ~~20 of Test Methods~~concentration range is from 0.005 mg to 0.05 mg of phosphorus per ~~E350.~~100 mL of solution when measured at 825 nm and from 0.05 mg to 0.3 mg of phosphorus per 100 mL of solution when measured at 650 nm, using a 1-cm cell.

NOTE 3—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.

22. Stability of Color

22.1 ~~See Section~~The molybdenum ~~21 of Test Methods~~blue complex is stable ~~E350.~~for at least 2 h.

23. Interferences

23.1 ~~See Section~~ None ~~22~~of ~~Test Methods~~the elements ~~E350.~~usually present interfere except arsenic, which is removed by volatilization as the bromide.

24. Apparatus

24.1 ~~See Section~~Glassware must ~~23 of Test Methods~~be phosphorus- and arsenic-free. ~~E350.~~Boil the glassware with HCl and rinse with water before use. It is recommended that the glassware used for this determination be reserved for this use only. Many detergents contain phosphorus and must not be used for cleaning purposes.

25. Reagents

25.1 *Ammonium Molybdate Solution* (20 g/L)—Cautiously, while stirring and cooling, add 300 mL of H_2SO_4 to 500 mL of water and cool. Add 20 g of ammonium heptamolybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$), cautiously dilute to 1 L, and mix.

25.2 *Ammonium Molybdate-Hydrazine Sulfate Solution*—Dilute 250 mL of the ammonium molybdate solution to 600 mL, add 100 mL of the hydrazine sulfate solution, dilute to 1 L, and mix. Do not use a solution that has stood for more than 1 h.

25.3 *Hydrazine Sulfate Solution* (1.5 g/L)—Dissolve 1.5 g of hydrazine sulfate ($(NH_2)_2\cdot H_2SO_4$) in water, dilute to 1 L, and mix. Discard any unused solution after 24 h.

25.4 *Phosphorus Standard Solution A* (1 mL = 1.0 mg P)—~~Proceed as directed~~ Transfer 2.292 g of anhydrous disodium hydrogen phosphate (Na_2HPO_4~~in 24.1 through 24.7 of Test Methods~~), previously dried to constant weight at 105 °C, to a 500-mL volumetric flask; dissolve in about 100 mL of water, dilute ~~E350.~~to volume, and mix.

25.5 *Phosphorus Standard Solution B* (1 mL = 0.01 mg P)—Using a pipet, transfer 10 mL of Solution A (1 mL = 1.0 mg P) to a 1-L volumetric flask, add 50 mL of $HClO_4$ (1 + 5), dilute to volume, and mix.

25.6 *Phosphorus Standard Solution C* (1 mL = 0.10 mg P)—Using a pipet, transfer 50 mL of Solution A (1 mL = 1.0 mg P) to a 500-mL volumetric flask, add 50 mL of $HClO_4$ (1 + 5), dilute to volume, and mix.

25.7 *Sodium Sulfite Solution* (100 g/L)—Dissolve 100 g of sodium sulfite (Na_2SO_3) in water, dilute to 1 L, and mix.

26. Preparation of Calibration Curve for Concentrations from 0.005 mg/100 mL to 0.05 mg/100 mL

26.1 *Calibration Solutions*—~~Proceed as directed~~ Using pipets, transfer 5 mL, 10 mL, 15 mL, 25 mL, and 50 mL of Phosphorus Standard Solution B (1 mL = 0.01 mg P) to 100-mL volumetric flasks. Add 20 mL of $HClO_4$~~in 25.1 through 25.6~~, dilute to volume, and mix. Using a pipet, transfer 10 mL of each solution to a 100-mL borosilicate glass volumetric flask. Proceed in accordance with 26.3 ~~of Test Methods E350~~.

26.2 *Reagent Blank*—Transfer 12 mL of $HClO_4$ (1 + 5) to a 100-mL borosilicate glass volumetric flask.

26.3 *Color Development:*

26.3.1 Add 15 mL of Na_2SO_3 solution, boil gently for 30 s, and add 50 mL of ammonium molybdate-hydrazine sulfate solution that has been prepared within the hour.

26.3.2 Heat the solutions at not less than 90 °C for 20 min, quickly cool, dilute to volume, and mix.

NOTE 4—Immersing the flasks in a boiling water bath is the preferred means of heating them for complete color development.

26.4 *Reference Solution*—Water.

26.5 *Spectrophotometry:*

26.5.1 *Multiple-Cell Spectrophotometer*—Measure the reagent blank (which includes the cell correction) versus the reference solution (26.4) using absorption cells with a 1-cm light path and using a light band centered at approximately 825 nm. Using the test cell, take the spectrophotometric readings of the calibration solutions versus the reference solution.

26.5.2 *Single-Cell Spectrophotometer*—Transfer a suitable portion of the reference solution (26.4) to an absorption cell with a 1-cm light path and adjust the spectrophotometer to the initial setting using a light band centered at approximately 825 nm. While maintaining this adjustment, take the spectrophotometric readings of the reagent blank solution and of the calibration solutions.

26.6 *Calibration Curve*—Follow the instrument manufacturer's instructions for generating the calibration curve.

27. Preparation of Calibration Curve for Concentrations from 0.05 mg/100 mL to 0.30 mg/100 mL

27.1 *Calibration Solutions*—Using pipets, transfer 5 mL, 10 mL, 15 mL, 20 mL, 25 mL, and 30 mL of Phosphorus Standard Solution C (1 mL = 0.10 mg P) to 100-mL volumetric flasks. Add 20 mL of $HClO_4$, dilute to volume, and mix. Using a pipet, transfer 10 mL of each solution to a 100-mL borosilicate glass volumetric flask.

27.2 *Reagent Blank*—Proceed ~~as directed~~ in ~~26.1 through 26.6~~accordance with 26.2~~ of Test Methods E350~~.

27.3 *Color Development*—Proceed in accordance with 26.3.

27.4 *Reference Solution*—Water.

27.5 *Spectrophotometry:*

27.5.1 *Multiple-Cell Spectrophotometer*—Measure the reagent blank (which includes the cell correction) versus the reference solution (27.4) using absorption cells with a 1-cm light path and a light band centered at approximately 650 nm. Using the test cell, take the spectrophotometric readings of the calibration solutions versus the reference solution.

27.5.2 *Single-Cell Spectrophotometer*—Transfer a suitable portion of the reference solution (27.4) to an absorption cell with a 1-cm light path and adjust the spectrophotometer to the initial setting using a light band (no change) centered at approximately 650 nm. While maintaining this adjustment, take the spectrophotometric readings of the reagent blank solution and of the calibration solutions.

27.6 *Calibration Curve*—Follow the instrument manufacturer's instructions for generating the calibration curve.

28. Procedure

28.1 *Test Solution:*

28.1.1 Select and weigh a sample in accordance with the following:

Phosphorus, %	Sample Weight, g	Tolerance in Sample Weight, mg
0.020 to 0.30	1.0	0.5
0.30 to 0.60	0.5	0.3
0.60 to 0.90	0.25	0.1

Transfer it to a 250-mL Erlenmeyer flask.

28.1.2 If the sample is other than white iron, proceed as directed in 28.1.2.1 and 28.1.2.2.

28.1.2.1 Add 15 mL of a freshly prepared mixture of 1 volume of HNO_3 and 3 volumes of HCl, slowly and in small portions. When the reaction has ceased, add 10 mL of $HClO_4$ and evaporate to fumes. Remove the flask immediately to avoid undue loss of $HClO_4$, cool, and add 20 mL of HBr (1 + 4). Evaporate the solution to copious white fumes and then, without delay, fume strongly enough to cause the white fumes to clear the neck of the flask, and continue at this rate for 1 min.

28.1.2.2 Cool the solution, add 60 mL of $HClO_4$ (1 + 5), and swirl to dissolve the salts. Transfer to a 100-mL volumetric flask, cool, dilute to volume, and mix. Allow insoluble matter to settle or dry filter the solution. Using a pipet, transfer 10-mL portions to two 100-mL borosilicate glass volumetric flasks; treat one in accordance with 28.3 and the other in accordance with 28.4.2.

28.1.3 ~~If the sample is other than white iron, proceed as directed in~~Treat ~~27.1.2 and 27.1.3 of Test Methods E350; treat~~ samples of white iron as directed in ~~27.1.2.1~~28.1.3.1 and ~~27.1.2.2~~28.1.3.2.

28.1.3.1 Crush the material in an iron mortar and weigh only particles passing through a No. 50 (300-μm) sieve. Transfer the weighed sample to a 250-mL Erlenmeyer flask. Add 15 mL of HNO_3 and 5 mL of HBr. Heat until dissolution is complete. Add

10 mL of $HClO_4$, evaporate to copious white fumes; then, without delay, fume strongly enough to cause the white fumes to clear the neck of the flask, and continue at this rate for 1 min.

28.1.3.2 ~~Proceed as directed~~ Cool the solution, add 60 mL of $HClO_4$~~in 27.1.3 of Test~~ (1 + 5), and swirl to dissolve the salts. Transfer to a 100-mL volumetric flask, cool, dilute to volume, and mix. Allow insoluble matter to settle or dry filter the solution. Using a pipet, transfer 10-mL portions to two 100-mL borosilicate glass volumetric flasks; treat one in accordance with 28.3 ~~Methods~~and ~~E350~~the other in accordance with 28.4.2.

28.2 *Reagent Blank Solution*—Carry a reagent blank through the entire procedure using the same amount of all reagents with the sample omitted.

28.3 *Color Development*—Proceed with one of the 10-mL portions obtained in 28.1.2.2 or 28.1.3.2, in accordance with 26.3.

28.4 ~~Proceed as directed in 27.2 through 27.5 Molybdenum Blue Photometric Method of Test Methods E350.~~*Reference Solutions:*

28.4.1 *Water*—Use this as the reference solution for the reagent blank solution.

28.4.2 *Background Color Reference Solution*—Add 15 mL of Na_2SO_3 solution to the second 10-mL portion obtained in 28.1.2.2 or 28.1.3.2. Boil gently for 30 s, add 50 mL of H_2SO_4 (3 + 37), cool, dilute to volume, and mix. Use this as the reference solution for the test solution.

28.5 *Spectrophotometry*—Take the spectrophotometric readings of the reagent blank solution and of the test solution (using the respective reference solutions) in accordance with 26.5 or 27.5 depending upon the estimated composition of phosphorus in the sample.

29. Calculation

29.1 ~~Proceed as directed in Section~~Convert the net spectrophotometric reading ~~28~~of ~~Test Methods~~the test ~~E350.~~solution and of the reagent blank solution to milligrams of phosphorus by means of the appropriate calibration curve. Calculate the percent of phosphorus as follows:

$$\text{Phosphorus, \%} = (A - B)/(C \times 10) \tag{2}$$

where:

A = phosphorus found in 100 mL of the final test solution, mg,
B = phosphorus found in 100 mL of the final reagent blank solution, mg, and
C = sample represented in 100 mL of the final test solution, g.

30. Precision and Bias

30.1 Nine laboratories cooperated in testing this method and obtained the data summarized in Table 2.

TABLE 2 Statistical Information—Phosphorus

Test Specimen	Phosphorus Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
~~1. Cast iron 15Ni-2Cr-5Cu (NBS 115, 0.114 P)~~	~~0.107~~	~~0.013~~	~~0.014~~
~~2. Cast iron (NBS 5k, 0.263 P)~~	~~0.257~~	~~0.016~~	~~0.012~~
~~3. Cast iron (NBS 7g, 0.794 P)~~	~~0.779~~	~~0.020~~	~~0.053~~

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SULFUR BY THE GRAVIMETRIC METHOD

This test method, which consisted of Sections 30 through 36, was discontinued in 1988.

SULFUR BY THE COMBUSTION-IODATE TITRATION METHOD

This test method, which consisted of Sections 37 through 45, was discontinued in 2012.

~~SULFUR BY THE COMBUSTION-IODATE TITRATION METHOD~~

~~37. Scope~~

~~37.1 This method covers the determination of sulfur in concentrations from 0.005 to 0.25 %.~~

~~38. Summary of Test Method~~

~~38.1 See Section 38 of Test Methods E350.~~

~~39. Interferences~~

~~39.1 The elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.~~

~~40. Apparatus~~

~~40.1 See Section 40 of Test Methods E350.~~

~~41. Reagants~~

~~41.1 Proceed as directed in 41.1 through 41.6 of Test Methods E350.~~

~~42. Calibration~~

~~42.1 Proceed as directed in 42.1 through 42.6 of Test Methods E350.~~

~~43. Procedure~~

~~43.1 Proceed as directed in 43.1 through 43.2 of Test Methods E350.~~

~~44. Calculation~~

~~44.1 Proceed as directed in Section 44 of Test Methods E350.~~

~~45. Precision~~

~~45.1 Twenty-two laboratories cooperated in testing this method; six used resistance furnaces and reported eight sets of values (Note 3) and sixteen used induction furnaces (Note 4). They obtained the data summarized in Table 3 for specimen 5. Although samples covered by this method with sulfur concentrations near the lower limit of the scope were not available for testing, the precision data obtained using the methods indicated in Table 4 should apply. None was available to permit a test near the upper limit of the scope.~~

~~NOTE 3—The recovery of sulfur as SO_2 ranged from 72 to 97 %, an average value of 83 % based on calibration standards designated *b*,*c*, and *d* in Table 3.~~

~~NOTE 4—The recovery of sulfur as SO_2 ranged from 80 to 96 %, an average value of 88 % based on calibration standards designated *b*,*c*, and *d* in Table 3.~~

SILICON BY THE GRAVIMETRIC METHOD

46. Scope

46.1 This method covers the determination of silicon in ~~concentrations~~compositions from 0.1 % to 6.1 ~~%~~%.

47. Summary of Test Method

47.1 ~~See Section 47 of Test Methods E350.~~After dissolution of the sample, silicic acid is dehydrated by fuming with H_2SO_4 or $HClO_4$. The solution is filtered, and the impure silica is ignited and weighed. The silica is then volatilized with HF. The residue is ignited and weighed; the loss in weight represents silica.

TABLE 3 Statistical Information—Sulfur

Test Specimen	Sulfur Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E 173)
Induction Furnace			
1. No. 1, E352	0.006[A]	0.002	0.003
2. No. 2, E352	0.008[A]	0.001	0.004
3. No. 3, E350	0.014[A]	0.003	0.003
4. No. 4, E350	0.016[A]	0.002	0.002
5. Cast iron (NBS 6f, 0.106S)	0.106[B]	0.009	0.008
6. No. 7, E350	0.141[C]	0.007	0.013
7. No. 7, E353	0.286[D]	0.014	0.020
Resistance Furnace			
1. No. 1, E352	0.006[A]	0.001	0.002
2. No. 2, E352	0.009[A]	0.001	0.002
3. No. 3, E350	0.014[A]	0.001	0.003
4. No. 4, E350	0.015[A]	0.002	0.003
5. Cast iron (NBS 6f, 0.106 S)	0.105[B]	0.007	0.008
6. No. 7, E350	0.140[C]	0.007	0.011
7. No. 7, E353	0.288[D]	0.012	0.021

[A] Calibration standards: See Footnote[A], Table 6, Methods E350.
[B] Calibration standards: See Footnote[B], Table 6, Methods E350.
[C] Calibration standards: See Footnote[C], Table 6, Methods E350.
[D] Calibration standards: See Footnote[D], Table 6, Methods E350.

TABLE 43 Statistical Information—Silicon

Test Specimen	Silicon Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
$HClO_4$ Dehydration			
1. Cast iron 1.2Ni-0.3Cr-0.8 Mo (NBS 107b, 1.35 Si)	1.36	0.02	0.02
1. Cast iron 1.2Ni-0.3Cr-0.8 Mo (NIST 107b, 1.35 Si)	1.36	0.02	0.02
2. Cast iron (NBS 4i, 1.45 Si)	1.45	0.04	0.05
2. Cast iron (NIST 4i, 1.45 Si)	1.45	0.04	0.05
3. Cast iron 1.07Ni-0.32Cr (NBS 82a, 2.07 Si)	2.08	0.04	0.05
3. Cast iron 1.07Ni-0.32Cr (NIST 82a, 2.07 Si)	2.08	0.04	0.05
4. Cast iron (NBS 5k, 2.08 Si)	2.08	0.03	0.05
4. Cast iron (NIST 5k, 2.08 Si)	2.08	0.03	0.05
5. Cast iron, high (0.79) phosphorus (NBS 7g, 2.41 Si)	2.40	0.04	0.07
5. Cast iron, high (0.79) phosphorus (NIST 7g, 2.41 Si)	2.40	0.04	0.07
6. White cast iron (NBS 1176, 3.19 Si)	3.20	0.03	0.10
6. White cast iron (NIST 1176, 3.19 Si)	3.20	0.03	0.10
H_2SO_4 Dehydration			
1. Cast iron 1.2Ni-0.3Cr-0.8Mo (NBS 107b, 1.35 Si)	1.36	0.02	0.03
1. Cast iron 1.2Ni-0.3Cr-0.8Mo (NIST 107b, 1.35 Si)	1.36	0.02	0.03
2. Cast iron (NBS 4i, 1.45 Si)	1.45	0.04	0.06
2. Cast iron (NIST 4i, 1.45 Si)	1.45	0.04	0.06
3. Cast iron 1.07Ni-0.32Cr (NBS 82a, 2.07 Si)	2.08	0.04	0.04
3. Cast iron 1.07Ni-0.32Cr (NIST 82a, 2.07 Si)	2.08	0.04	0.04
4. Cast iron (NBS 5k, 2.08 Si)	2.08	0.04	0.05
4. Cast iron (NIST 5k, 2.08 Si)	2.08	0.04	0.05
5. Cast iron, high (0.79) phosphorus (NBS 7g, 2.41 Si)	2.41	0.03	0.05
5. Cast iron, high (0.79) phosphorus (NIST 7g, 2.41 Si)	2.41	0.03	0.05

48. Interferences

48.1 The elements ordinarily present do not interfere if their concentrationscompositions are under the maximum limits shown in 1.1.

49. Reagents ~~and Materials~~

49.1 The analyst should make certain by analyzing blanks and other checks that possible silicon contamination of reagents will not significantly bias the results.

49.2 *Perchloric Acid:*

49.2.1 Select a lot of $HClO_4$ that contains not more than 0.0002 % silicon for the analysis of samples containing silicon in the range from 0.02 % to 0.10 % and not more than 0.0004 % silicon for samples containing more than 0.10 % by determining duplicate values for silicon in accordance with 49.2.2-49.2.6.

49.2.2 Transfer 15 mL of $HClO_4$ (Note 5) to each of two 400-mL beakers. To one of the beakers transfer an additional 50 mL of $HClO_4$. Using a pipet, transfer 20 mL of Na_2SiO_3 solution (1 mL = 1.00 mg Si) to each of the beakers. Evaporate the solutions to fumes and heat for 15 min to 20 min at such a rate that $HClO_4$ refluxes on the sides of the beakers. Cool sufficiently, and add 100 mL of water (40 °C to 50 °C).

NOTE 5—The 15-mL addition of $HClO_4$ can be from the same lot as the one to be tested. Once a lot has been established as having less than 0.0002 % silicon, it should preferably be used for the 15-mL addition in all subsequent tests of other lots of acid.

49.2.3 Add paper pulp and filter immediately, using lowash 11-cm medium-porosity filter papers. Transfer the precipitates to the papers, and scrub the beakers thoroughly with a rubber-tipped rod. Wash the papers and precipitates alternately with 3-mL to 5-mL portions of hot HCl (1 + 19) and hot water, for a total of 6 times. Finally wash the papers twice with H_2SO_4 (1 + 49). Transfer the papers to platinum crucibles.

49.2.4 Dry the papers and heat at 600 °C until the carbon is removed. Finally ignite at 1100 °C to 1150 °C or to constant weight (at least 30 min). Cool in a desiccator and weigh.

49.2.5 Add enough H_2SO_4 (1 + 1) to moisten the SiO_2, and add 3 mL to 5 mL of HF. Evaporate to dryness and then heat at a gradually increasing rate until H_2SO_4 is removed. Ignite for 15 min at 1100 °C to 1150 °C, cool in a desiccator, and weigh.

49.2.6 Calculate the percent of silicon as follows:

$$\text{Silicon,\%} = [(A - B) - (C - D)] \times 0.4674/E \times 100 \quad (3)$$

where:

A = initial weight of crucible plus impure SiO_2 when 65 mL of $HClO_4$ was taken, g,
B = final weight of crucible plus impurities when 65 mL of $HClO_4$ was taken, g,
C = initial weight of crucible plus impure SiO_2 when 15 mL of $HClO_4$ was taken, g,
D = final weight of crucible plus impurities when 15 mL of $HClO_4$ was taken, g, and
E = nominal weight (80 g) of 50 mL of $HClO_4$.

49.3 *Sodium Silicate Solution*—~~Proceed as directed~~ Transfer 11.0 g of sodium silicate ($Na_2SiO_3 \cdot 9H_2$~~in 49.1 through 49.4 of Test Methods~~ O) to a 400-mL beaker. Add 150 mL of water and dissolve the salt. Filter through a medium paper, collecting the filtrate in a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle. Use this solution to determine the suitability of the $HClO_4$~~E350~~.

49.4 *Tartaric Acid Solution* (20.6 g/L)—Dissolve 20.6 g of tartaric acid ($C_4H_6O_6$) in water, dilute to 1 L, and filter.

49.5 *Water*—Use freshly prepared Type II water known to be free of silicon. Water distilled from glass, demineralized in columns containing silicon compounds, or stored for extended periods in glass, or combination thereof, has been known to pick up silicon.

50. Procedure

50.1 Select and weigh a sample in accordance with the following:

Silicon, %	Sample Weight, g	Tolerance in Sample Weight, mg	Dehydrating Acid, mL H_2SO_4 (1 + 4)	Dehydrating Acid, mL $HClO_4$
0.10 to 1.00	4.0	4	150	60
1.00 to 2.00	3.0	3	100	50
2.00 to 4.00	2.0	2	100	40
4.00 to 6.00	1.0	1	100	40

Transfer it to a 400-mL beaker or a 300-mL porcelain casserole.

50.2 If the sample type is other than white iron, proceed as directed in ~~50.2~~50.3 ~~or 50.3 of Test Methods E350~~; treat samples of white iron as directed in 50.2.1~~50.2.1.~~.

50.2.1 Crush the material in an iron mortar and use only particles passing through a No. 100 (150-μm) sieve. Add 30 mL of HNO_3 and 10 mL of HBr. When the dissolution reaction becomes passive, decant the bulk of the solution to a 400-mL beaker and crush the remaining insoluble matter in the original beaker with a glass rod. Add 20 mL of HNO_3 and 10 mL of HBr, and heat gently until dissolution is complete. Combine the two portions of the solution and add the amount of H_2SO_4 or $HClO_4$ specified in 50.1~~50.1~~.

50.2.2 ~~Proceed as directed in 50.2.2 or 50.3.2 of Test Methods E350.~~*Sulfuric Acid Dehydration:*

50.2.2.1 Evaporate until salts begin to separate; at this point evaporate the solution rapidly to the first appearance of fumes and fume strongly for 2 min to 3 min. Cool sufficiently, and add 100 mL of water (40 °C to 50 °C). Stir to dissolve the salts and heat, if necessary, but do not boil. Proceed immediately in accordance with 50.4.

50.2.3 *Perchloric Acid Dehydration:*

50.2.3.1 Evaporate the solution to fumes and heat for 15 min to 20 min at such a rate that the $HClO_4$ refluxes on the sides of the container. Cool sufficiently and add 100 mL of water (40 °C to 50 °C). Stir to dissolve the salts and heat to boiling. If the sample solution contains more than 100 mg of chromium, add, while stirring, 1 mL of tartaric acid solution for each 25 mg of chromium.

50.3 Add paper pulp and filter immediately, on a low-ash 11-cm medium-porosity filter paper. Collect the filtrate in a 600-mL beaker. Transfer the precipitate to the paper, and scrub the container thoroughly with a rubber-tipped rod. Wash the paper and precipitate alternately with 3-mL to 5-mL portions of hot HCl (1 + 19) and hot water until iron salts are removed but for not more than a total of ten washings. If the $HClO_4$ dehydration method was followed, wash the paper twice more with H_2SO_4 (1 + 49), but do not collect these washings in the filtrate; discard the washings. Transfer the paper to a platinum crucible and reserve.

50.4 Add 15 mL of HNO_3 to the filtrate, stir, and evaporate in accordance with either 50.2.2 or 50.2.3, depending upon the dehydrating acid used. Filter immediately, using a low-ash, 9-cm-100-porosity filter paper, and wash in accordance with 50.3.

50.5 Proceed as directed in 50.4 or 50.7 of Test Methods Transfer the paper and precipitate to the reserved platinum crucible. Dry the papers and then heat the crucible at 600 °C until the carbon is removed. Finally ignite at 1100 °C to 1150 °C to constant weight (at least 30 min). Cool in a desiccator and weigh.E350.

50.6 Add enough H_2SO_4 (1 + 1) to moisten the impure SiO_2, and add 3 mL to 5 mL of HF. Evaporate to dryness and then heat at a gradually increasing rate until H_2SO_4 is removed. Ignite at 1100 °C to 1150 °C for 15 min, cool in a desiccator, and weigh.

51. Calculation

51.1 Proceed as directed in Section 51 of Test MethodsCalculate the percent of silicon as E350.follows:

$$\text{Silicon},\% = [((A - B) \times 0.4674)/C] \times 100 \quad (4)$$

where:

A = initial weight of crucible and impure SiO_2, g,
B = final weight of crucible and residue, g, and
C = sample used, g.

52. Precision

52.1 Eleven laboratories cooperated in testing this method and obtained the data summarized in Table 43. Although samples covered by this method with silicon concentrationscompositions near the extreme limits of the scope were not available for testing, the precision data obtained for low-alloy steels by Test Methods E350 should apply at the lower limit.

COBALT BY THE ION-EXCHANGE-POTENTIOMETRIC TITRATION METHOD

53. Scope

53.1 This test method covers the determination of cobalt in concentrationscompositions from 2.0 % to 4.5 %.

54. Summary of Method

54.1 Cobalt is separated from interfering elements by selective elution from an anion-exchange column using hydrochloric acid. HCl. The cobalt is oxidized to the trivalent state with ferricyanide, and the excess ferricyanide is titrated potentiometrically with cobalt solution.

55. Interferences

55.1 The elements ordinarilynormally present do not interfere if their concentrationscompositions are under the maximum limits shown in 1.1.

56. Apparatus

56.1 *Ion-Exchange Column*, approximately 25 mm in diameter and 300 mm long, in length, tapered at one end, and provided with a stopcock to control the flow rate, and a second, lower stopcock to stop the flow. Apparatus No. 8A Jones Reductor, may be adapted to this method. A reservoir for the eluants may be added at the top of the column.

56.2 *Apparatus No. 3B,pH meter,* with a platinum and a saturated calomel electrode.

57. Reagents

57.1 *Ammonium Citrate Solution* (200 g/l)—Dissolve 200 g of di–ammonium hydrogen citrate in water and dilute to 1 L.

57.2 *Cobalt, Standard Solution* (1mL = 1.5 mg of C9)—Reagent No. 25B.Co).

57.2.1 *Preparation*—Dry a weighing bottle in an oven at 130 °C for 1 h, cool in a desiccator, and weigh. Transfer 3.945 g of cobalt sulfate ($CoSO_4$)[25] that has been heated at 550 °C for 1 h to the weighing bottle. Dry the bottle and contents at 130 °C for 1 h, cool in desiccator, stopper the bottle, and weigh. The difference in weight is the amount of $CoSO_4$ taken. Transfer the weighed $CoSO_4$ to a 400-mL beaker, rinse the weighing bottle with water, and transfer the rinsings to the beaker. Add 150 mL of water and 20 mL of HNO_3, and heat to dissolve the salts. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

57.2.2 *Standardization*—Calculate the cobalt concentration as follows:

$$\text{Cobalt, mg/mL} = \text{weight of } CoSO_4\text{, g, } \times 0.38026 \quad (5)$$

57.3 *Ion-Exchange Resin:*[6]

57.3.1 Use an anion exchange resin of the alkyl quaternary ammonium type (chloride form) consisting of spherical beads having a nominal crosslinkage of 8 %, and 200200-nominal to 400 nominal 400-nominal mesh size. To remove those beads greater than about 180-µm in diameter as well as the excessively fine beads, treat the resin as follows: Transfer a supply of the resin to a beaker, cover with water, and allow sufficient time (at least 30 min) for the beads to undergo maximum swelling. Place a No. 80 (180-µm) screen, 150 mm in diameter over a 2-L beaker. Prepare a thin slurry of the resin and pour it onto the screen. Wash the fine beads through the screen, using a small stream of water. Discard the beads retained on the screen, periodically, if necessary, to avoid undue clogging of the openings. When the bulk of the collected resin has settled, decant the water and transfer approximately 100 mL of resin to a 400-mL beaker. Add 200 mL of HCl (1 + 19), (1 + 19), stir vigorously, allow the resin to settle for 4 min to 6 min, decant 150 mL to 175 mL of the suspension, and discard. Repeat the treatment with HCl (1 + 19) (1 + 19) twice more, and reserve the coarser resin for the column preparation.

57.3.2 Prepare the column as follows: Place a 1010-mm to 20-mm layer of glass wool or poly(vinyl chloride)polyvinyl chloride plastic fiber in the bottom of the column, and add a sufficient amount of the prepared resin to fill the column to a height of approximately 140 mm. Place a 20-mm layer of glass wool or poly(vinyl chloride)polyvinyl chloride plastic fiber at the top of the resin bed to protect it from being carried into suspension when the solutions are added. While passing a minimum of 35 mL of HCl (7 + 5) (7 + 5) through the column, with the hydrostatic head 100 mm above the top of the resin bed, adjust the flow rate to not more than 3.0 mL per min. Drain to 10 mm to 20 mm above the top of the resin bed and then close the lower stopcock.

NOTE 6—The maximum limits of 0.125 g of cobalt and 0.500 g in the sample solution take into account the exchange capacity of the resin, the physical dimensions of the column, and the volume of eluants.

57.4 *Potassium Ferricyanide, Standard Solution* (1 mL = 3.0 mg of Co):

57.4.1 Dissolve 16.68 g of potassium ferricyanide ($K_3Fe(CN)_6$) in water and dilute to 1 L. Store the solution in a dark-colored bottle. Standardize the solution each day before use as follows: Transfer from a 50-mL buret approximately 20 mL of $K_3Fe(CN)_6$ solution to a 400-mL beaker. Record the buret reading to the nearest 0.01 mL. Add 25 mL of water, 10 mL of ammonium citrate solution, and 25 mL of NH_4OH. Cool to 5 °C to 10 C, 10 °C, and maintain this temperature during the titration. Transfer the beaker to the potentiometric titration apparatus. While stirring, titrate the $K_3Fe(CN)_6$ with the cobalt solution (1 mL = 1.5 mg Co) using a 50-mL buret. Titrate at a fairly rapid rate until the end point is approached, and then add the titrant in 1-drop increments through the end point. After the addition of each increment, record the buret reading and voltage when equilibrium is reached. Estimate the buret reading at the end point to the nearest 0.01 mL by interpolation.

57.4.2 Calculate the cobalt equivalent as follows (Note(Note 7 6):):

$$\text{Cobalt equivalent, mg/mL} = (A \times B)/C \quad (6)$$

$$\text{Cobalt equivalent, mg/mL} = (A \times B)/C \quad (6)$$

where:

A = cobalt standard solution required to titrate the potassium ferricyanide solution, mL,
B = cobalt standard solution, mg/mL, and
C = potassium ferricyanide solution, mL.

NOTE 7—Duplicate or triplicate values should be obtained for the cobalt equivalent. The values obtained should check within 1 part per thousand to 2 parts per thousand.

58. Procedure

58.1 Transfer a 0.50-g sample, weighed to the nearest 0.1 mg, to a 150-mL beaker. Add 20 mL of a mixture of 5 parts of HCl and 1 part of HNO_3 (Note 58). Cover the beaker and digest at 60 °C to 70 C 70 °C until the sample is decomposed. Rinse and remove the cover. Place a ribbed cover glass on the beaker, and evaporate the solution nearly to dryness, but do not bake. Cool, add 20 mL of HCl (7 + 5), (7 + 5), and digest at 60 °C to 70 C 70 °C until salts are dissolved (approximately 10 min).

NOTE 8—Other ratios and concentrations of acids, with or without the addition of 1 mL to 2 mL of HF, are used for the decomposition of special grades of alloys.

[6] Available from the Dow Chemical Co., Midland, MI. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.