



Standard Test Methods for Chemical Analysis of Molybdenum¹

This standard is issued under the fixed designation E 315; 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 approval. 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 molybdenum and molybdenum alloys having chemical compositions within the following limits:

Element	Concentration Range, %
Carbon	0.010 to 0.040
Iron	0.020 max
Molybdenum	99.25 to 99.99
Nickel	0.010 max
Nitrogen	0.0010 max
Oxygen	0.0030 max
Silicon	0.010 max
Titanium	0.005 to 0.55
Zirconium	0.06 to 0.12

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

	Sections
Nickel by the Persulfate-Dimethylglyoxime Photometric Method	9-18
Iron by the 1,10-Phenanthroline Photometric Method	19-29
Silicon by the Molybdenum Blue-Extraction Photometric Method	30-40

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 the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary information is given in Section 5.

2. Referenced Documents

2.1 ASTM Standards:²

- B 384 Specification for Molybdenum and Molybdenum Alloy Forgings
- B 385 Specification for Molybdenum and Molybdenum Alloy Billets for Reforging
- B 386 Specification for Molybdenum and Molybdenum Alloy Plate, Sheet, Strip, and Foil

¹ These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.06 on Titanium, Zirconium, Wolfram, Molybdenum, Tantalum, Niobium, Hafnium.

Current edition approved May 1, 2004. Published June 2004. Originally approved in 1971. Last previous edition approved in 1999 as E 315 – 88 (1999).

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.

- B 387 Specification for Molybdenum and Molybdenum Alloy Bar, Rod, and Wire
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications
- E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
- E 60 Practice for Molecular Absorption and Spectrometric Methods for Chemical Analysis of Metals, Ores, and Related Materials
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals
- E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Significance and Use

3.1 These test 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 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.

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 sections, 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, except the photometers shall conform to the requirements prescribed in Practice E 60.

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

5. Hazards

5.1 For precautions to be observed in the use of certain reagents in these test methods, refer to Practices E 50.

6. Sampling

6.1 For procedures for sampling the material, refer to Specifications B 384, B 385, B 386, and B 387.

7. Rounding Calculated Values

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

8. Interlaboratory Studies

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

NICKEL BY THE PERSULFATE-DIMETHYLGLYOXIME PHOTOMETRIC TEST METHOD

9. Scope

9.1 This test method covers the determination of nickel in concentrations from 1 to 400 ppm. The range may be extended by suitably varying the sample weight, aliquot size, amount of reagents, and cell depth.

10. Summary of Test Method

10.1 A red soluble salt of trivalent nickel and dimethylglyoxime is formed. Photometric measurement is made at approximately 445 nm.

11. Concentration Range

11.1 The recommended concentration ranges are from 1.0 to 2.0 μg and from 10 to 100 μg of nickel for each 50 mL of solution, using cell depths of 5 and 2 cm respectively.

NOTE 1—This test method has been written for cells having 5 and 2-cm light paths. 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 develops in 5 min and is stable for 30 min.

13. Interferences

13.1 Provision has been made for the correction of any interfering elements present.

14. Reagents

14.1 *Ammonium Persulfate Solution* (150 g/L)—Dissolve 15 g of ammonium persulfate $[(\text{NH}_4)_2\text{S}_2\text{O}_8]$ in water and dilute to 100 mL. Prepare fresh as needed.

14.2 *Citric Acid Solution* (100 g/L)—Dissolve 10 g of citric acid in water and dilute to 100 mL.

14.3 *Nickel, Standard Solution A* (1 mL = 10 μg Ni)—Dissolve 0.1000 g of nickel (purity 99.9 % min) in 10 mL of water and 5 mL of HNO_3 in a 150-mL beaker. When dissolution is complete boil to remove oxides of nitrogen. Cool to room temperature, transfer to a 1-L volumetric flask, dilute to volume, and mix. Transfer a 50-mL aliquot of this solution to a 500-mL volumetric flask, dilute to volume, and mix.

14.4 *Nickel, Standard Solution B* (1 mL = 1 μg Ni)—Transfer a 10-mL aliquot of nickel solution A (1 mL = 10 μg Ni) to a 100-mL volumetric flask, dilute to volume, and mix.

14.5 *Sodium Dimethylglyoximate Solution* (30 g/L)—Dissolve 30 g of sodium dimethylglyoxime in water and dilute to 1 L.

15. Preparation of Calibration Curves

15.1 *Calibration Solutions*:

15.1.1 Transfer 1.00, 3.00, 4.00, 7.00, and 10.00 mL of nickel solution A (1 mL = 10 μg Ni) to five 50-mL volumetric flasks, dilute to 20 mL, and proceed as directed in 15.3.

15.1.2 Transfer 1.00, 5.00, 10.00, 15.00, and 20.00 mL of nickel solution B (1 mL = 1 μg Ni) to five 50-mL volumetric flasks, dilute to 20 mL, and proceed as directed in 15.3.

15.2 *Reference Solution*—Transfer 20 mL of water to a 50-mL volumetric flask and proceed as directed in 15.3.

15.3 *Color Development*—Add 4 mL of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ solution, 5 mL of citric acid solution, and 10 mL of NH_4OH , and cool to room temperature. Add 1 mL of sodium dimethylglyoximate solution, dilute to volume, and mix.

15.4 *Photometry*—Transfer a suitable portion of the reference solution to an absorption cell with a 2 or a 5-cm light path, as appropriate (see 11.1), and adjust the photometer to the initial setting, using a light band centered at approximately 445 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

15.5 *Calibration Curves*—Plot the photometric readings of the calibration solutions against micrograms of nickel per 50 mL of solution.

16. Procedure

16.1 *Test Solution*:

16.1.1 Select a sample weight in accordance with the following table:

Nickel, ppm	Sample Weight, g
1 to 50	1.00
45 to 120	0.50
115 to 400	0.25

16.1.2 Weigh duplicate samples of the size selected to the nearest 1 mg (Note 2), and transfer to 100-mL beakers. Add 10 mL of HCl and heat. Add HNO_3 dropwise until all the metal has decomposed (Note 3). Remove from the hot plate and cool.

NOTE 2—Duplicate samples are taken in order to have one available to determine the sample blank. For the higher concentrations of nickel, a larger sample may be dissolved and diluted to volume; duplicate aliquots may then be taken from a single sample.

NOTE 3—If any molybdenic oxide precipitates, add more HCl dropwise and boil gently until the molybdenum is completely in solution.

16.1.3 Transfer the solutions to 50-mL volumetric flasks and dilute to about 20 mL.

16.2 *Reference Solution*—Distilled water.

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

16.4 *Sample Blank Solution*—Treat the duplicate as directed in 15.3 but omit the addition of the sodium dimethylglyoximate solution.

16.5 *Color Development*—Proceed as directed in 15.3.

16.6 *Photometry*—Take the photometric readings of the reagent blank solution, the sample blank solution, and the test solution as directed in 15.4.

17. Calculation

17.1 Convert the photometric readings of the test solution to micrograms of nickel, and of the reagent blank and sample

blank solutions to the equivalent micrograms of nickel from the appropriate calibration curve. Calculate the parts per million of nickel as follows:

$$\text{Nickel, ppm} = [A - (B + C)]/D \quad (1)$$

where:

- A = micrograms of nickel found in 50 mL of the final solution,
- B = reagent blank correction, in equivalent micrograms of nickel,
- C = sample blank correction, in equivalent micrograms of nickel, and
- D = grams of sample represented in 50 mL of the final solution.

18. Precision and Bias

18.1 *Precision*—Data on this test method were obtained by nine cooperators. Repeatability, standard deviation, and coefficient of reproducibility were graphically calculated as directed in Practices E 173, and were found to be as listed in Table 1.

18.2 *Bias*—Data on bias is not available because of the lack of standard samples. However, one cooperator prepared synthetic samples containing the equivalent of 53.9 ppm of nickel and recoveries within 0.8 ppm were obtained. Another cooperator added 25 µg of nickel, equivalent to 25 ppm, to four 1-g samples of Material A. Nickel recoveries (25 ppm plus 4 ppm present) ranged between 28 and 30 ppm.

IRON BY THE 1,10-PHENANTHROLINE PHOTOMETRIC TEST METHOD

19. Scope

19.1 This test method covers the determination of iron in concentrations from 50 to 750 ppm.

20. Summary of Test Method

20.1 The iron is reduced with hydroxylamine hydrochloride and converted to the 1,10-phenanthroline complex. Photometric measurement is made at approximately 510 nm.

21. Concentration Range

21.1 The recommended concentration range is from 50 to 300 µg of iron for each 100 mL of solution, using a 2-cm cell.

NOTE 4—This test method has been written for cells 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.

22. Stability of Color

22.1 The color develops fully after heating at 60 to 70°C for 30 min and is stable for 15 to 20 h.

TABLE 1 Statistical Information^A

Test Material	Mean, ppm Ni	Repeatability, ppm Ni	Standard Deviation, ppm Ni	Coefficient of Reproducibility, %
A	3.9	0.737	0.226	18.9
B	114	11.04	3.45	9.7

^AIn accordance with Practice E 173 (discontinued 1997), the Reproducibility R_2 of E 173 corresponds to the Reproducibility Index R of E 1601. The Repeatability R_1 of E 173 corresponds to the Repeatability Index r of E 1601.

23. Interferences

23.1 Nickel, copper, and cobalt interfere if present in the final test solution in amounts greater than 800 µg, 1600 µg, and 800 µg, respectively. Reduced molybdenum produces a positive error and provision has been made in the method to eliminate this interference.

24. Apparatus

24.1 *Plastic (TFE-Fluorocarbon) Beakers and Covers*, 50-mL capacity.

NOTE 5—Platinum beakers and covers may be used.

25. Reagents

25.1 *Ammonium Citrate-Sodium Borate Solution*—Dissolve 200 g of ammonium citrate in 800 mL of water, and dilute to 1 L. Dissolve 85 g of sodium tetraborate, ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) in 900 mL of hot water, cool, and dilute to 1 L. Transfer the two solutions to a 2-L bottle, and mix.

25.2 *Hydroxylamine Hydrochloride* (10 g/L)—Dissolve 1 g of hydroxylamine hydrochloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$) in water and dilute to 100 mL. Do not use a solution that has stood longer than 8 h.

25.3 *Iron, Standard Solution* (1 mL = 100 µg Fe)—Dissolve 0.1000 g of iron (purity: 99.8 % min)³ in 60 mL of HCl (1 + 1). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

25.4 *1,10-Phenanthroline Solution* (2 g/L)—Dissolve 0.4 g of 1,10-phenanthroline monohydrate in 150 mL of warm water and dilute to 200 mL.

25.5 *Reagent Mixture*—Transfer 10 mL of HF and 8 mL of HNO_3 to a 50-mL plastic beaker (Note 5) and evaporate to about 10 mL. Add about 30 mL of water and transfer to a 500-mL volumetric flask containing 360 mL of ammonium citrate-sodium borate solution. Cool, dilute to volume, and mix.

26. Preparation of Calibration Curve

26.1 *Calibration Solution*—Using pipets, transfer 0.5, 1, 2, and 3 mL of iron solution (1 mL = 100 µg Fe) to 100-mL volumetric flasks containing 50 mL of reagent mixture, and dilute to about 75 mL. Proceed as directed in 26.3.

26.2 *Reference Solution*—Transfer 50 mL of reagent mixture to a 100-mL volumetric flask and dilute to about 75 mL.

26.3 *Color Development*—Using pipets, transfer 5 mL of $\text{NH}_2\text{OH} \cdot \text{HCl}$ solution and 10 mL of 1,10-phenanthroline solution. Dilute to about 90 mL and heat in a water bath at 60 to 70°C for 30 min. Cool, dilute to volume, and mix.

26.4 *Photometry*:

26.4.1 *Multiple-Cell Photometer*—Measure the cell correction using absorption cells with a 2-cm light path (see Note 4) and a light band centered at approximately 510 nm. Using the test cell, take the photometric readings of the calibration solutions.

26.4.2 *Single-Cell Photometer*—Transfer a suitable portion of the reference solution to an absorption cell with a 2-cm light

³National Institute of Standards and Technology Sample 55 has been found satisfactory for this purpose.

path (see Note 4) and adjust the photometer to the initial setting, using a light band centered at approximately 510 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

26.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against micrograms of iron per 100 mL of solution.

27. Procedure

27.1 Test Solution:

27.1.1 Transfer a 2.0-g sample, weighed to the nearest 1.0 mg, to a 50-mL plastic beaker (Note 5).

27.1.2 Add 5 mL of HF and place the cover on the beaker.

27.1.3 Carefully add 4 mL of HNO₃ and warm until dissolution is complete.

27.1.4 Evaporate to about 5 mL and cool.

27.1.5 Transfer to a 250-mL volumetric flask containing 180 mL of ammonium citrate-sodium borate solution. Dilute to volume, and mix.

27.1.6 Using a pipet, transfer two 50-mL portions of solution to two 100-mL volumetric flasks. Treat one portion as directed in 26.2. Treat the other portion as directed in 27.5.

27.2 *Reference Solution*—Distilled water.

27.3 *Background Color Solution*—Proceed as directed in 26.3 omitting the addition of the 1,10-phenanthroline solution.

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

27.5 *Color Development*—Proceed as directed in 26.3.

27.6 *Photometry*—Take the photometric readings of the test solution, background color solution, and the reagent blank solution as directed in 26.4.

28. Calculation

28.1 Convert the photometric readings of the test solution to micrograms of iron and the photometric readings of the background color solution and reagent blank solution to the equivalent micrograms of iron by means of the calibration curve. Calculate the parts per million of iron as follows:

$$\text{Iron, ppm} = [A - (B + C)]/D \quad (2)$$

where:

A = micrograms of iron found in 100 mL of final test solution,

B = micrograms of iron equivalent found in 100 mL of the background color solution,

C = micrograms of iron equivalent found in 100 mL of the reagent blank solution, and

D = grams of sample represented in 100 mL of final test solution

29. Precision and Bias

29.1 *Precision*—Nine laboratories cooperated in testing this test method and obtained the data summarized in Table 2. Samples with iron concentration near the limits of the scope were not available for testing. Data were obtained on Bar 2 in anticipation that the test method would be applicable at this level.

TABLE 2 Statistical Information^A

Test Material	Iron Found, ppm	Repeatability (<i>R</i> ₁ , E173) <i>r</i> , E1601)	Reproducibility (<i>R</i> ₂ , E173) <i>R</i> , E1601)
Bar 2	24.6	9.7	12.7
Bar 186	124.8	10.5	8.4

^AIn accordance with Practice E 173 (discontinued 1997), the Reproducibility *R*₂ of E 173 corresponds to the Reproducibility Index *R* of E 1601. The Repeatability *R*₁ of E 173 corresponds to the Repeatability Index *r* of E 1601.

29.2 *Bias*—No information on the bias of this test method is available.

SILICON BY THE MOLYBDENUM BLUE-EXTRACTION PHOTOMETRIC TEST METHOD

30. Scope

30.1 This test method covers the determination of silicon in concentrations from 0.0010 to 0.0125 %.

31. Summary of Test Method

31.1 After dissolution of the sample in sulfuric and nitric acids, hydrofluoric acid is added, followed by the addition of boric acid. The solution is oxidized with potassium permanganate. The pH of the solution is adjusted to 1.7 to 1.8. The molybdisilicic acid is extracted with 1-butanol and reduced with stannous chloride to a heteropoly blue complex. Photometric measurement is made at approximately 635 nm.

32. Concentration Range

32.1 The recommended concentration range is from 0.005 to 0.030 mg of silicon per 50 mL of solution, using a 2-cm cell.

NOTE 6—This test method has been written for cells 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.

33. Stability of Color

33.1 The color is stable for at least 1 h.

34. Interferences

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

35. Apparatus

35.1 It is recommended that plastic ware be used wherever it is feasible, especially when in contact with hot liquids or alkaline solutions. If the use of glassware is unavoidable, the period of contact with the hot or alkaline solution should be kept to a minimum.

36. Reagents

36.1 *Ammonium Hydroxide Solution*—Bubble ammonia gas, using plastic tubing, into cooled distilled water (use an ice bath) contained in a polyethylene bottle until the solution is saturated.

36.2 *Ammonium Molybdate Solution (50 g/L)*—Dissolve 10 g of ammonium molybdate tetrahydrate (NH₄)₆Mo₇O₂₄·4H₂O in 150 mL of water, and dilute to 200 mL.

36.3 *Boric Acid Solution* (50 g/L)—Dissolve 50 g of boric acid (H_3BO_3) in 900 mL of warm water, cool, and dilute to 1 L.

36.4 *Mixed Acid Solution*—Add 15 mL of H_2SO_4 and 15 mL of HNO_3 to 120 mL of water.

36.5 *Potassium Permanganate Solution* (0.6 g/L)—Dissolve 0.15 g of potassium permanganate (KMnO_4) in 200 mL of water, and dilute to 250 mL.

36.6 *Silicon, Standard Solution* (1 mL = 0.002 mg Si)—Ignite some SiO_2 in a platinum crucible for 2 to 3 h at 1000°C and cool in a desiccator. Fuse 0.1070 g of anhydrous silicon dioxide (SiO_2) with 2 g of sodium carbonate (Na_2CO_3) in a platinum crucible, cool, dissolve the melt in about 200 mL of water in a plastic beaker, and cool. Transfer to a 250-mL volumetric flask, dilute to volume, and mix. Transfer the solution immediately to a polyethylene bottle. Using a pipet, transfer 10 mL to a 1-L volumetric flask, dilute to volume, and mix. Transfer the solution immediately to a polyethylene bottle.

36.7 *Stannous Chloride Solution* (20 g/L)—Dissolve 2 g of stannous chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) and 1 mg of cupric chloride dihydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) in 100 mL of HCl and store in a glass-stoppered bottle. Do not use a solution that has stood for more than 1 week.

36.8 *Sulfuric Acid* (1 + 99) *Saturated with 1-Butanol*—Saturate H_2SO_4 by shaking with 1-butanol in a separatory funnel.

36.9 *Tartaric Acid Solution* (200 g/L)—Dissolve 50 g of tartaric acid in 200 mL of water, and dilute to 250 mL.

36.10 *Water*—The distilled water used in the preparation of the reagents and in the procedure must be of very high quality. If traces of silicon are present, the blank will be too high.

37. Preparation of Calibration Curve

37.1 Calibration Solutions:

37.1.1 Using pipets, transfer 2, 5, 7, 10, and 15 mL of silicon solution (1 mL = 0.002 mg Si) to 250-mL plastic beakers. Add 15 mL of mixed acid solution and 0.5 mL of HF.

37.1.2 Add 40 mL of H_3BO_3 solution. Add KMnO_4 solution dropwise until a permanent pink tint is obtained. Add 8 mL of ammonium molybdate solution, and mix. Using a pH meter, adjust the pH to 1.7 to 1.8 with NH_4OH solution. Cover the beaker with a plastic cover and heat on a steam bath for 10 min. Cool in a water bath at 20°C for 10 min. Add 4 mL of tartaric acid solution while mixing.

37.1.3 Transfer the solution to a 250-mL separatory funnel. Rinse the beaker twice with water and add the rinsings to the funnel. Adjust the solution, if necessary, to approximately 90 mL, add 14 mL of H_2SO_4 (1 + 1) and 40 mL of 1-butanol and shake the solution vigorously for 1 min. When the layers have separated drain and discard the acid layer. Scrub the 1-butanol layer with 20 mL of H_2SO_4 (1 + 99) saturated with 1-butanol to the organic phase in the separatory funnel, shake for 30 s, allow the layers to separate, drain, and discard the aqueous phase. Repeat the washings two more times. Transfer the solution to a dry 50-mL beaker (Note 7) and then to a dry 50-mL volumetric flask. Rinse the funnel and beaker twice with 1 to 2 mL of 1-butanol and transfer each rinse to the volumetric flask. Proceed as directed in 37.3.

NOTE 7—The solution is first transferred to a dry beaker in order to remove most of the water droplets before transferring it to the volumetric flask.

37.2 *Reference Solution*—Transfer 5 mL of water to a 250-mL plastic beaker. Add 15 mL of mixed acid solution and 0.5 mL of HF. Proceed as directed in 37.1.2 and 37.1.3.

37.3 *Color Development*—Add 1.0 mL of SnCl_2 solution to the volumetric flask, dilute to volume with 1-butanol, and mix.

37.4 Photometry:

37.4.1 *Multiple-Cell Photometer*—Measure the cell correction using absorption cells with a 2-cm light path and a light band centered at approximately 635 nm. Using the test cell, take the photometric readings of the calibration solutions.

37.4.2 *Single Cell Photometer*—Transfer a suitable portion of the reference solution to an absorption cell with a 2-cm light path and adjust the photometer to the initial setting, using a light band centered at approximately 635 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

37.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against milligrams of silicon per 50 mL of solution.

38. Procedure

38.1 *Test Solution*—Transfer a 0.200-g sample, weighed to the nearest 0.5 mg, to a 150-mL plastic beaker and cover with a plastic cover. Add 3 mL of H_2SO_4 (1 + 1) and 3 mL of HNO_3 (1 + 1). When dissolution is complete, rinse the cover and eliminate the oxides of nitrogen by heating in a beaker of boiling water for 1 h. Cool to room temperature and add 0.5 mL of HF and 1.5 mL of HNO_3 . Proceed as directed in 37.1.2 and 37.1.3 but omit the addition of the 8 mL of ammonium molybdate solution.

38.2 *Reference Solution*—Carry a reagent blank through the entire procedure, using the same amounts of all reagents with the sample omitted, but using the 8 mL of ammonium molybdate solution.

38.3 *Color Development*—Proceed as directed in 37.3.

38.4 *Photometry*—Take the photometric reading of the test solution as directed in 37.4.

39. Calculation

39.1 Convert the net photometric reading of the test solution to milligrams of silicon by means of the calibration curve. Calculate the percent of silicon as follows:

$$\text{Silicon, \%} = A/(B \times 10) \quad (3)$$

where:

A = silicon found in 50 mL of final test solution, mg, and
B = sample represented in 50 mL of final test solution, g.

40. Precision and Bias

40.1 *Precision*—Six laboratories cooperated in testing this test method. Two laboratories submitted data from two different analysts in order to have eight sets of results for Bar No. 2. Three laboratories submitted data from two different analysts giving a total of nine sets of results for Bar No. 186. The data obtained are summarized in Table 3.

TABLE 3 Statistical Information^A

Test Material	Silicon Found, %	Repeatability (R_1 , E173) r , E1601)	Reproducibility (R_2 , E173) r , E1601)
Bar No. 2	0.00180	0.00036	0.00102
Bar No. 186	0.01055	0.00074	0.00103

^AIn accordance with Practice E 173 (discontinued 1997), the Reproducibility R_2 of E 173 corresponds to the Reproducibility Index R of E 1601. The Repeatability R_1 of E 173 corresponds to the Repeatability Index r of E 1601.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).

40.2 *Bias*—No information on the bias of this test method is available.

41. Keywords

41.1 chemical analysis; iron content; molybdenum; nickel content; photometric; silicon content; spectrophotometric