



# Standard Test Methods for Chemical Analysis of Ferrochrome-Silicon<sup>1</sup>

This standard is issued under the fixed designation E 364; 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 ferrochrome-silicon having chemical compositions within the following limits:<sup>2</sup>

Element	Concentration, %
Aluminum	0.50 max
Antimony	0.005 max
Arsenic	0.005 max
Bismuth	0.005 max
Boron	0.005 max
Carbon	0.15 max
Chromium	34.0 to 47.0
Cobalt	0.10 max
Columbium	0.050 max
Copper	0.050 max
Lead	0.005 max
Manganese	0.75 max
Molybdenum	0.050 max
Nickel	0.50 max
Nitrogen	0.050 max
Phosphorus	0.030 max
Silicon	30.0 to 45.0
Silver	0.005 max
Sulfur	0.030 max
Tantalum	0.050 max
Tin	0.005 max
Titanium	0.50 max
Vanadium	0.50 max
Zinc	0.005 max
Zirconium	0.050 max

1.2 The test methods appear in the following order:

	Sections
Arsenic by the Molybdenum Blue Photometric Method	8-18
Chromium by the Acid Dissolution Titrimetric Method	19-25
Silicon by the Sodium Peroxide Fusion-Perchloric Acid Dehydration Method	26-33

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.* For precautions to be observed in these test methods, refer to Practices E 50, and to precautions included in the individual methods.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee E-1 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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<sup>2</sup> These test methods are intended for use in determining the composition of ferrochrome-silicon as specified in Specification A 482.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- A 482 Specification for Ferrochrome-Silicon<sup>3</sup>
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>4</sup>
- E 32 Practices for Sampling Ferroalloys and Steel Additives for Determination of Chemical Composition<sup>5</sup>
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals<sup>5</sup>
- E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals<sup>5</sup>
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals<sup>5</sup>
- E 360 Test Methods for Chemical Analysis of Silicon and Ferrosilicon<sup>6</sup>

## 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 solutions, and certain 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 that 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. Sampling

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

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 01.02.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 03.05.

<sup>6</sup> *Annual Book of ASTM Standards*, Vol 03.06.

## 6. Rounding Off Calculated Values

6.1 Calculated values shall be rounded off to the desired number of places as directed in the Rounding Off and Special Case Rounding Off procedures in the Rounding-Off Method Section of Practice E 29.

## 7. Interlaboratory Studies

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

### ARSENIC BY THE MOLYBDENUM BLUE PHOTOMETRIC METHOD

## 8. Scope

8.1 This method covers the determination of arsenic in ferrochrome-silicon in concentrations from 0.001 to 0.005 %.

8.2 The limits of the scope have been set at 0.001 to 0.005 % because test materials containing other arsenic concentrations were unavailable for testing. However, recognizing that the procedure should give satisfactory results at lower and higher concentrations, the calibration and procedure section cover the range from 0.001 to 0.1 %.

8.2.1 Users of this method are cautioned that its use on samples outside of the 0.001 to 0.005 % range is not supported by interlaboratory testing.

## 9. Summary of Method

9.1 Arsenic is first separated by distillation as the trivalent chloride. Ammonium molybdate is added to form arsenomolybdate ion which is then reduced by hydrazine sulfate to form the molybdenum blue complex. Photometric measurement is made at approximately 850 nm.

## 10. Concentration Range

10.1 The recommended concentration range is 0.01 to 0.15 mg of arsenic per 50 mL of solution using a 1-cm cell.

NOTE 1—This 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 amount of sample and reagents used.

## 11. Stability of Color

11.1 The color is stable for at least 2 h.

## 12. Interferences

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

## 13. Apparatus

13.1 *Distillation Apparatus*, Fig. 1, Methods E 360.

13.2 *Zirconium Crucibles*, 30-mL capacity.

## 14. Reagents

14.1 *Ammonium Bromide* ( $\text{NH}_4\text{Br}$ ).

14.2 *Ammonium Molybdate Solution* (10 g/L)—Dissolve 2.5 g of ammonium heptamolybdate tetrahydrate ( $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ ) in 40 mL of warm water. Add 128 mL of  $\text{H}_2\text{SO}_4$  (1 + 3), dilute to 250 mL, and mix.

14.3 *Ammonium Molybdate-Hydrazine Sulfate Solution*—Dilute 100 mL of ammonium molybdate solution to 900 mL, add 10 mL of hydrazine sulfate solution, dilute to 1 L, and mix. Do not use a solution that has stood more than 1 h.

14.4 *Arsenic, Standard Solution A* (1 mL = 0.10 mg As)—Transfer 0.1320 g of arsenic trioxide ( $\text{As}_2\text{O}_3$ ) to a 1-L volumetric flask, dissolve in 100 mL of HCl, cool, dilute to volume, and mix.

14.5 *Arsenic, Standard Solution B* (1 mL = 0.01 mg As)—Using a pipet, transfer 100 mL of arsenic standard solution A (1 mL = 0.10 mg As) to a 1-L volumetric flask, dilute to volume, and mix.

14.6 *Hydrazine Sulfate* —( $(\text{NH}_2)_2\cdot\text{H}_2\text{SO}_4$ ).

14.7 *Hydrazine Sulfate Solution* (1.5 g/L)—Dissolve 1.5 g of hydrazine sulfate ( $(\text{NH}_2)_2\cdot\text{H}_2\text{SO}_4$ ) in water, dilute to 1 L, and mix. Do not use a solution that has stood more than 1 day.

14.8 *Sodium Carbonate* ( $\text{Na}_2\text{CO}_3$ ).

14.9 *Sodium Peroxide* ( $\text{Na}_2\text{O}_2$ ).

## 15. Preparation of Calibration Curve

15.1 *Calibration Solutions*:

15.1.1 Using pipets, transfer 1, 2, 5, 10, and 15 mL of arsenic standard solution B (1 mL = 0.01 mg As) to 125-mL Erlenmeyer flasks.

15.1.2 Add 10 mL of  $\text{HNO}_3$  and evaporate the solution to dryness on a hot plate. Bake for 30 min at 150 to 180°C. Remove from the hot plate. Add 45 mL of ammonium molybdate-hydrazine sulfate solution to each flask, warm gently to dissolve the residue, and transfer the solution to a 50-mL volumetric flask. Proceed as directed in 15.3.

15.2 *Reference Solution*—Transfer 10 mL of  $\text{HNO}_3$  to a 125-mL Erlenmeyer flask and evaporate the solution to dryness on a hot plate. Bake for 30 min at 150 to 180°C. Remove from the hot plate. Add 45 mL of ammonium molybdate-hydrazine sulfate solution to the flask, warm gently to dissolve the residue, transfer to a 50-mL volumetric flask and proceed as directed in 15.3.

15.3 *Color Development*—Heat the flask in a boiling water bath for 15 min. Remove the flask, cool to room temperature, dilute to volume with ammonium molybdate-hydrazine sulfate solution, and mix.

15.4 *Photometry*:

15.4.1 *Multiple-Cell Photometer*—Measure the cell correction with water using absorption cells with a 1-cm light path and a light band centered at approximately 850 nm. Using the test cell, take the photometric readings of the calibration solutions using the solution prepared in 15.2 as a reference.

15.4.2 *Single-Cell Photometer*—Transfer a suitable portion of the reference solution (15.2) to an absorption cell with a 1-cm light path and adjust the photometer to the initial setting, using a light band centered at approximately 850 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

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

## 16. Procedure

16.1 *Test Solution*:

16.1.1 Select and weigh a sample to the nearest 0.1 mg in accordance with the following:

Arsenic, %	Sample Weight, g
0.001 to 0.015	0.500
0.01 to 0.04	0.250
0.035 to 0.10	0.125

Transfer the sample to a 30-mL zirconium crucible containing 10 g of Na<sub>2</sub>O<sub>2</sub> and 2 g of Na<sub>2</sub>CO<sub>3</sub> or 8 g of Na<sub>2</sub>O<sub>2</sub> plus 2 g of Na<sub>2</sub>CO<sub>3</sub>.

16.1.2 Mix thoroughly with a metal spatula (Note 2). Fuse carefully over a free flame by holding the crucible with a pair of tongs and slowly revolving it around the outer edge of the flame until the contents have melted down quietly; raise the temperature gradually to avoid spattering. When the contents are molten, give the crucible a rotary motion to stir up any unattached particles of the alloy adhering to the bottom or sides. Finally, increase the temperature until the crucible is bright red for 1 min. Cool the crucible to room temperature. Transfer the crucible to an 800-mL beaker containing 60 mL of H<sub>2</sub>SO<sub>4</sub>(1 + 1) and 200 mL of water. Dissolve the melt; remove and rinse the crucible.

NOTE 2—**Precaution:** Use proper safety practices and equipment when performing sodium peroxide fusions.

16.1.3 If manganese dioxide is present, add H<sub>2</sub>SO<sub>3</sub> dropwise until the solution clears.

16.1.4 Heat to boiling, and cool. While stirring vigorously, add NH<sub>4</sub>OH until the solution is alkaline to litmus, and then add 3 to 5 mL in excess. Heat to boiling, remove from the heat, and allow the precipitate to settle. Filter on a coarse filter paper and wash five times with hot water. Discard the filtrate. Remove the filter paper, carefully open it, and place it on the inside wall of the original 800-mL beaker. Wash the precipitate from the paper using a fine stream of water. Pass 25 mL of HCl over the paper, and wash well with water but do not exceed a total volume of 40 mL. Discard the paper. Warm gently until the precipitate dissolves.

16.1.5 Transfer the solution to the distillation flask, add 1 g of NH<sub>4</sub>Br and 0.75 g of hydrazine sulfate. Add 20 mL of HNO<sub>3</sub>(1 + 1) to the receiving flask, and place the flask in an 800-mL beaker containing cold water. Assemble the apparatus (Fig. 1, Methods E 360), heat the distillation flask, and distill into the receiving flask.

16.1.6 Distill until the volume is reduced to 10 mL or until oxides of nitrogen are noted in the distillation flask. Remove the distillation flask from the heat source. Place the receiving flask on a hot plate and evaporate the solution to dryness. Bake for 30 min at 150 to 180°C. Add 45 mL of ammonium molybdate-hydrazine sulfate solution to the flask, warm gently to dissolve the residue, and transfer the solution to a 50-mL volumetric flask. Proceed as directed in 16.3.

16.2 *Reference Solution*—Carry a reagent blank through the entire procedure using the same amounts of all reagents with the sample omitted, for use as a reference solution.

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

16.4 *Photometry*—Take the photometric reading of the test solution as directed in 15.4.

## 17. Calculation

17.1 Convert the net photometric reading of the test solution

to milligrams of arsenic by means of the calibration curve. Calculate the percentage of arsenic as follows:

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

where:

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

## 18. Precision and Bias

18.1 *Precision*—Nine laboratories cooperated in testing this method and obtained the data summarized in Table 1. Samples with arsenic concentrations near the upper limit of the scope were not available for testing.

18.2 *Bias*—The accuracy of the method could not be evaluated because adequate certified standard reference materials were unavailable at the time of testing. The user is cautioned to verify by the use of certified reference materials, if available, that the accuracy of this method is adequate for the contemplated use.

## CHROMIUM BY THE ACID DISSOLUTION TITRIMETRIC METHOD

### 19. Scope

19.1 This method covers the determination of chromium in ferrochrome-silicon in concentrations from 30 to 60 %.

### 20. Summary of Method

20.1 The alloy is dissolved in sulfuric, nitric, and hydrofluoric acids, and the excess of the latter is complexed with boric acid. The chromium and manganese ions are oxidized to dichromate and permanganate ions, respectively, by ammonium peroxydisulfate with silver nitrate as a catalyst. After adding HCl to reduce permanganate ions, the dichromate ions are reduced by adding an excess of standard ferrous ammonium sulfate (salt or solution). The excess ferrous ions are titrated with standard potassium permanganate solution.

### 21. Interferences

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

### 22. Reagents

22.1 *Ammonium Peroxydisulfate* —((NH<sub>4</sub>)<sub>2</sub>-S<sub>2</sub>O<sub>8</sub>).

22.2 *Boric Acid* (H<sub>3</sub>BO<sub>3</sub>).

22.3 *Ferrous Ammonium Sulfate Salt*— Fine well-mixed, free-flowing crystals of ferrous ammonium sulfate hexahydrate [Fe(NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub> · 6H<sub>2</sub>O] are required. Standardize as follows: Transfer 0.9806 g of National Bureau of Standards standard potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) (equivalent to 200 mL of 0.1 N solution) to a 600-mL beaker. Add 300 mL of water, 30 mL of H<sub>2</sub>SO<sub>4</sub> (1 + 1), and 8.000 g of the ferrous ammonium sulfate. Stir until completely dissolved. Add 6 drops of 1,10-phenanthroline indicator solution. Using a 50-mL buret,

TABLE 1 Statistical Information—Arsenic

Ferroalloy Type	Arsenic Found, %	Repeatability (R <sub>1</sub> , E 173)	Reproducibility (R <sub>2</sub> , E 173)
Cr40-Si42-C0.05	0.0018	0.0003	0.0003

titrate the ferrous ions with 0.1 N  $\text{KMnO}_4$  solution to the color change from red to green. Record the buret reading to the nearest 0.02 mL. Calculate the volume of 0.1 N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution equivalent to 1 g of the ferrous ammonium sulfate as follows:

$$A = [200 + (B \times C/0.1000)]/8 \quad (2)$$

where:

- $A$  = millilitres of 0.1 N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution equivalent to 1 g of ferrous ammonium sulfate,  
 $B$  = millilitres of 0.1 N  $\text{KMnO}_4$  solution used, and  
 $C$  = normality of  $\text{KMnO}_4$  solution used.

NOTE 3—The ferrous ammonium sulfate is stable for at least 1 week; store in an air-tight container.

22.4 *Ferrous Ammonium Sulfate, Standard Solution* (0.25 N) (Note 4)—Dissolve 89.6 g of ferrous ammonium sulfate hexahydrate  $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$  in 500 mL of cold  $\text{H}_2\text{SO}_4(5 + 95)$ , and dilute to 1 L with  $\text{H}_2\text{SO}_4(5 + 95)$ . Use a solution that has been standardized within the previous 8 h as follows: Transfer 0.9806 g of National Bureau of Standards standard potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) (equivalent to 200 mL of 0.1 N solution) to an 800-mL beaker. Add 300 mL of water and 30 mL of  $\text{H}_2\text{SO}_4(1 + 1)$ , and stir until completely dissolved. Add a slight excess of the ferrous ammonium sulfate solution from a calibrated 100-mL buret, and record the buret reading to the nearest 0.02 mL. Add 6 drops of 1,10-phenanthroline indicator solution and titrate the ferrous ions with 0.1 N  $\text{KMnO}_4$  solution to the color change from red to green. Calculate the volume of 0.1 N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution equivalent to 1 mL of ferrous ammonium sulfate solution as follows:

$$A = [200 + (B \times C/0.1000)]/D \quad (3)$$

where:

- $A$  = millilitres of 0.1 N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution equivalent to 1 mL of ferrous ammonium sulfate solution,  
 $B$  = millilitres of 0.1 N  $\text{KMnO}_4$  solution used,  
 $C$  = normality of  $\text{KMnO}_4$  solution used, and  
 $D$  = millilitres of 0.25 N ferrous ammonium sulfate solution used.

NOTE 4—Ferrous ammonium sulfate salt is preferred to the standard ferrous ammonium sulfate solution.

22.5 *1,10-Phenanthroline Ferrous Complex Indicator Solution*—(0.25 M)—Reagent No. 122.

22.6 *Potassium Permanganate Standard Solution* (0.1 N)—Reagent No. 13.

22.7 *Potassium Permanganate Solution* (20 g/L)—Reagent No. 134.

22.8 *Silver Nitrate Solution* (8 g/L)—Reagent No. 133.

## 23. Procedure

23.1 Transfer a 0.50-g sample, weighed to the nearest 0.1 mg, to a 150-mL poly(tetrafluoroethylene) beaker. Add 20 mL of  $\text{H}_2\text{SO}_4(1 + 1)$  and 5 mL of  $\text{HNO}_3$ , and cover the beaker with a plastic cover. Add 2 mL of HF and if the reaction does not begin, warm until dissolution begins. Add an additional 6 mL of HF in 2-mL increments and finally heat at 90°C for 5 min. Dilute to 100 mL and transfer the solution to an 800-mL beaker containing 3 g of  $\text{H}_3\text{BO}_3$ . Dilute to 400 mL and add 5 mL of

$\text{H}_3\text{PO}_4$ , 5 mL of  $\text{AgNO}_3$  solution, 4 drops of  $\text{KMnO}_4$  solution (20 g/L), and 15 g of  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ . Boil for 15 min. If the permanganate color has not developed after 5 min of boiling, remove from the heat, cool to 70 to 80°C, cautiously add an additional 5 g of  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , and boil for 10 min. Add 5 mL of HCl (1 + 3) and boil for 5 min after any  $\text{MnO}_2$  has dissolved and the  $\text{KMnO}_4$  color has completely disappeared. Cool to room temperature and dilute to approximately 350 mL.

23.2 Select and weigh a portion of the standard ferrous ammonium sulfate salt (Note 5) to the nearest 0.1 mg in accordance with the following:

Chromium, %	Ferrous Ammonium Sulfate, g
34 to 40	5.0
40 to 45	5.5
45 to 50	6.0
50 to 55	6.5
55 to 60	7.0

Add the salt to the test solution and stir until it has completely dissolved. Add 6 drops of 1,10-phenanthroline indicator solution. Using a 50-mL buret, titrate the excess ferrous ions with 0.1 N  $\text{KMnO}_4$  standard solution to the color change from red to green. Record the buret reading to the nearest 0.02 mL.

NOTE 5—A measured amount of ferrous ammonium sulfate solution in excess of that required for the reduction may be used instead of the salt, if desired. (See Note 3 and Note 4.)

## 24. Calculation

24.1 When ferrous ammonium sulfate salt is used, calculate the percentage of chromium as follows:

$$\text{Chromium, \%} = [(A \times B) - (C \times D)/0.1000]/E \times 0.1733 \quad (4)$$

where:

- $A$  = millilitres of 0.1 N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution equivalent to 1 g of ferrous ammonium sulfate (see 22.3),  
 $B$  = ferrous ammonium sulfate used, g,  
 $C$  = millilitres of 0.1 N  $\text{KMnO}_4$  solution used,  
 $D$  = normality of  $\text{KMnO}_4$ , and  
 $E$  = sample used, g.

24.2 When ferrous ammonium sulfate solution is used, calculate the percentage of chromium as follows:

$$\text{Chromium, \%} = [(A \times B) - (C \times D)/0.1000]/E \times 0.1733 \quad (5)$$

where:

- $A$  = millilitres of 0.1 N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution equivalent to 1 mL of ferrous ammonium sulfate solution (see 22.4),  
 $B$  = millilitres of ferrous ammonium sulfate solution used,  
 $C$  = millilitres of 0.1 N  $\text{KMnO}_4$  solution used,  
 $D$  = normality of  $\text{KMnO}_4$  solution used, and  
 $E$  = sample used, g.

## 25. Precision and Bias <sup>7</sup>

25.1 *Precision*—Nine laboratories cooperated in testing this method and obtained the data summarized in Table 2.

25.2 *Bias*—The accuracy of this method could not be evaluated because adequate certified standard reference materials were unavailable at the time of testing. The user is

<sup>7</sup> Supporting data are available from ASTM Headquarters. Request RR: E03 - 1004.



**TABLE 2 Statistical Information—Chromium**

Test Specimen	Chromium Found, %	Repeatability (R <sub>1</sub> , E 173)	Reproducibility (R <sub>2</sub> , E 173)
Ferrochrome-silicon, No. 4	34.79	0.18	0.61
Ferrochrome-silicon, No. 5	55.23	0.18	0.38

cautioned to verify by the use of certified reference materials, if available, that the accuracy of this method is adequate for the contemplated use.

### SILICON BY THE SODIUM PEROXIDE FUSION-PERCHLORIC ACID DEHYDRATION METHOD

#### 26. Scope

26.1 This method covers the determination of silicon in ferrochrome-silicon in concentrations from 38 to 45 %.

#### 27. Summary of Method

27.1 The sample is fused with sodium peroxide and leached with water. Silicic acid is dehydrated by fuming with perchloric acid. The solution is filtered and the residue is ignited and weighed. The silica in the residue is volatilized with hydrofluoric acid. The residue is ignited and reweighed. The loss in weight is used to calculate the silicon content of the sample.

#### 28. Interferences

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

#### 29. Apparatus

29.1 *Crucible*, 40 mL, made of silicon-free iron, zirconium, nickel, or vitreous carbon.

NOTE 6—A crucible made from No. 20 gauge ingot iron 0.965 mm (0.038 in.) in thickness is suitable for this purpose.

#### 30. Reagents

30.1 *Silver Nitrate* (10 g/L)—Dissolve 10 g of silver nitrate (AgNO<sub>3</sub>) in water and dilute to 1 L.

30.2 *Sodium Carbonate* (Na<sub>2</sub>CO<sub>3</sub>), anhydrous powder.

30.3 *Sodium Peroxide* (Na<sub>2</sub>O<sub>2</sub>), 35 mesh or finer.

#### 31. Procedure

31.1 Transfer a 0.5-g sample, weighed to the nearest 0.2 mg to a crucible containing a mixture of 10 g of Na<sub>2</sub>O<sub>2</sub> and 3 g of Na<sub>2</sub>CO<sub>3</sub>. Mix thoroughly. Carry a blank test through the same procedure, using the same quantities of reagents.

31.2 Heat the crucible and contents on a hot plate at 350 to 400°C until the melt darkens (Note 7). Carefully fuse over a low flame by holding the crucible with a pair of tongs and slowly revolve it around the outer edge of the flame until the contents have melted down. When the contents are completely molten, rotate the crucible carefully to stir up any particles of sample on the bottom or sides, keeping the crucible and contents at a low, red heat (Note 8). Increase the temperature to bright redness for 5 min. Allow the crucible to cool almost to room temperature.

NOTE 7—**Caution:** Use proper safety practices and equipment when performing sodium peroxide fusions.

NOTE 8—**Caution:** If the reaction proceeds violently with spattering of the contents because of too rapid heating, the use of insufficient Na<sub>2</sub>CO<sub>3</sub>, or the lack of thorough mixing, appreciable loss may occur and the work should be repeated.

31.3 Dissolve the fusion as directed in 31.3.1 or 31.3.2.

31.3.1 *Alternative 1*—Transfer the crucible and contents to a 600-mL beaker (polytetrafluoroethylene, stainless steel, or high purity nickel) containing 200 mL of water. Cover with a watch glass. When effervescence has ceased, remove and rinse the crucible with hot water. Cautiously, with stirring, transfer the solution to a 600- or 800-mL glass beaker containing 30 mL of HCl. Add 100 mL of HClO<sub>4</sub>, and proceed as directed in 31.4.

31.3.2 *Alternative 2*—Cover the crucible and tap it on a hard surface to loosen the melt. Transfer the melt to a clean 600-mL glass beaker. Add 100 mL of HClO<sub>4</sub> to the beaker and cover with a watch glass. Fill the crucible with hot water and, after effervescence has ceased in the beaker, add the contents of the crucible to the beaker. Transfer any residue from the crucible to the beaker using a rubber policeman and a minimum amount of water. Proceed as directed in 31.4.

31.4 Place the beaker on a hot plate and heat to fumes of HClO<sub>4</sub>. Continue heating until the residue begins to crystallize. Remove the beaker from the hot plate and allow to cool. Carefully add 20 mL of HCl down the wall of the beaker. Stir and dilute to 250 mL with hot water. Stir well and allow to settle.

31.5 Filter the solution through a 12.5-cm ashless, medium-porosity filter paper. Place in a 75-mm fluted glass funnel, collecting the filtrate in a 600-mL beaker. Scrub the original beaker thoroughly with a rubber-tipped rod. Wash the paper and precipitate with hot HCl (1 + 19) until the yellow color of the iron salts disappears, then finally wash several times with hot water until the chloride ions disappear (verified by means of a spot test with silver nitrate solution) (Note 9).

NOTE 9—Thorough washing of the filter is necessary to remove any trace of HClO<sub>4</sub> which would cause the paper to flame up during ignition.

31.6 Transfer the filtrate and washings to the beaker used for initial dehydration. Evaporate to a volume of about 250 mL. Add 20 mL of HClO<sub>4</sub> and carry out a second dehydration following the procedure described in 31.4. Filter and wash the precipitate as directed in 31.5, but use cold water instead of hot water.

31.7 Transfer the two filter papers to a 40-mL platinum crucible. Add 4 drops of ammonium hydroxide (Note 10). Heat gently at a maximum temperature of 400°C on a gas burner or other suitable means until the papers are dry. Partially cover the crucible with a platinum lid and continue heating the crucible and contents until the carbon is completely charred (Note 11). Cool and add 1 mL of H<sub>2</sub>SO<sub>4</sub> (1 + 1). Evaporate to dryness on a sand bath or other suitable means. Transfer covered crucible to a muffle furnace and heat at 1100°C to constant weight. Cool in a desiccator and weigh.

NOTE 10—The addition of the ammonium hydroxide reduces the hazard from the reaction of perchlorates during ignition which may cause spattering of the silica from the crucible.

NOTE 11—**Caution:** Exercise great care in igniting the papers since the current of air produced by a burning filter paper is sufficient to carry SiO<sub>2</sub> out of the crucible.

31.8 Moisten the impure silica with a few drops of water. Add approximately 10 mL of HF plus 2 to 3 drops of concentrated H<sub>2</sub>SO<sub>4</sub>. Evaporate until fumes cease to be evolved and then cool.

31.9 Repeat the procedure described in 31.8, but decrease the volume of HF to 2 mL. Heat in a muffle furnace at 1100°C to constant weight. Cool in a desiccator and weigh.

### 32. Calculation

32.1 Calculate the percentage silicon as follows:

$$\text{Silicon, \%} = \frac{(A - B) - (C - D)}{E} \times 0.4674 \times 100 \quad (6)$$

where:

*A* = weight of crucible plus impure silica, g,

*B* = weight of crucible plus impurities, g,

*C* = weight of crucible plus impure silica in blank test, g,

*D* = weight of crucible plus impurities in blank test, g, and

*E* = weight of sample, g.

### 33. Precision and Bias <sup>8</sup>

33.1 *Precision*—Under the auspices of ISO Committee

TC-132/SC 2, eleven laboratories cooperated in testing this method on test specimen JK 26 (FeCrSi) and nine laboratories cooperated on test specimen BCS 305/1 (FeSi). Each laboratory analyzed the sample on 6 separate days. Repeatability (*R*<sub>1</sub>) and reproducibility (*R*<sub>2</sub>) have been calculated by an analysis of variance described in Practice E 173. The results of this study are summarized in Table 3. Although the silicon (Si) concentration of the FeSi sample exceeds the scope of the standard, it was included as additional evidence to demonstrate the precision obtained with this method.

33.2 *Bias*—No information on the accuracy of this method is known. The accuracy may be judged, however, by comparing accepted reference values with the corresponding arithmetic average obtained by inter-laboratory testing.

### 34. Keywords

34.1 acid dehydration; arsenic content; chemical composition; chromium content; ferrochrome-silicon; fusion; photometric; silicon content; titrimetric

**TABLE 3 Statistical Information—Silicon**

Test Specimen	Silicon Found, %	Repeatability ( <i>R</i> <sub>1</sub> , E 173, <i>M</i> = 1)	Reproducibility ( <i>R</i> <sub>2</sub> , E 173, <i>M</i> = 1)
FeCrSi (JK 26) (45.5 % Si)	45.24	0.469	0.793
FeSi (BCS 305/1) (75.0 % Si)	74.93	0.350	0.945

<sup>8</sup> Supporting data are available from ASTM Headquarters. Request RR: E03 – 1057.

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