



Designation: E 76 – 88 (Reapproved 1998)

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Chemical Analysis of Nickel-Copper Alloys¹

This standard is issued under the fixed designation E 76; 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 procedures for the chemical analysis of nickel-copper alloys having chemical compositions within the following limits:²

Element	Concentration Range, %
Nickel	40 to 90
Cobalt	0.01 to 1.0
Copper	10 to 50
Iron	0.1 to 5.0
Manganese	0.1 to 2.5
Carbon	0.01 to 1.0
Silicon	0.01 to 5.0
Sulfur	0.001 to 0.1
Aluminum	0.01 to 4.0

1.2 The analytical procedures appear in the following order: (This standard contains more than one test method for some elements. In some cases, the use of multiple test methods is needed to cover the concentration range of the scope of the standard; in others, multiple test methods are supplied to allow for variations in availability of instruments and other facilities among laboratories.)

	Sections
Copper:	
Perchloric Acid-Electrolytic Test Method	9 to 13
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Nickel:	
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Cobalt:	
Alpha-Nitroso-beta-Naphthol Test Method	27 to 29
Nitroso-R-Salt-Photometric Test Method	30 to 37
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Thiocyanate-Photometric Test Method	41 to 48
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Persulfate-Arsenite Test Method	49 to 52
Periodate-Photometric Test Method	53 to 60
Carbon, Total, by the Direct Combustion Test Method	61 to 63
Silicon:	
Perchloric Acid Test Method	64 to 66

¹ 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 E01.08 on Ni and Co and High Temperature Alloys.

Current edition approved Dec. 30, 1988. Published February 1989. Originally published as E 76 – 50 T. Last previous edition E 76 – 83.

² For procedures for the chemical analysis of copper-nickel alloys containing 50 % and over of copper, see ASTM Test Methods E 75, for Chemical Analysis of Copper-Nickel and Copper-Nickel-Zinc Alloys, *Annual Book of ASTM Standards*, Vol 03.05.

Sulfuric Acid Test Method	Sections 67 to 68
Sulfur:	
Gravimetric Test Method	69 to 71
Direct Combustion-Iodate Test Method	72 to 75
Aluminum by the Mercury Cathode-Cupferron-8- Hydroxyquinoline Test Method	76 to 81

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 5.*

2. Referenced Documents

2.1 ASTM Standards:

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications³

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴

E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition⁴

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals⁴

E 88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition⁴

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 and Reagents

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,

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.05.

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

5. Hazards

5.1 For precautions to be observed in these test methods, reference shall be made to Practices E 50.

6. Photometric Practice

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

7. Sampling

7.1 Wrought products shall be sampled in accordance with Practice E 55. Cast products shall be sampled in accordance with Practice E 88.

8. Rounding Calculated Values

8.1 Calculated values shall be rounded to the desired number of places in accordance with the rounding method given in the Rounding-Off Procedure paragraphs of Practice E 29.

COPPER BY THE PERCHLORIC ACID-ELECTROLYTIC TEST METHOD

9. Apparatus

9.1 *Electrodes for Electroanalysis*—Apparatus No. 9.

10. Reagents

10.1 *Nitric Acid (nitrous acid-free)*—Boil a suitable amount of HNO₃ until it is colorless. Cool in running water. Prepare as required, just before use, taking care not to expose the product to direct sunlight.

10.2 *Perchloric-Nitric Acid Mixture*—To 200 mL of water, add 200 mL of HNO₃ and 600 mL of HClO₄.

11. Procedure for Alloys Containing Under 2 % Silicon

11.1 Transfer 1.0000 g of the sample to a 250-mL beaker. Add 20 mL of HClO₄-HNO₃ mixture. Cover and heat gently until action ceases. Insert a glass hook between the cover and beaker, and evaporate to copious white fumes. Heat to incipient boiling and remove the glass hook.

11.2 Boil gently for 10 min at such a rate that most of the HClO₄ is retained. Allow to cool somewhat, add 100 mL of warm water, and stir until the perchlorates dissolve.

11.3 Filter through a medium paper into a 300-mL tall-form beaker. Wash the residue with hot water (Note 1). Add 3 mL of HNO₃(HNO₂-free) to the filtrate.

NOTE 1—If the silicon content of the alloy is over 0.5 %, it may be determined in this residue as described in 65.4-65.8, except that if HF has been used in the preliminary treatment any residue present at this stage should be discarded.

11.4 *Electrolysis*—Dilute the solution to 150 mL. Electrolyze overnight, using platinum gauze electrodes and a current density of 0.5 A/dm². Remove the electrodes and wash them with water, taking the usual precautions against loss. Reserve the electrolyte and washings. Dip the cathode in two successive baths of methanol or ethanol, and dry in an oven at 110°C for 3 to 5 min. Cool, and weigh the deposit as metallic copper.

11.5 *Calculation*—Calculate the percentage of copper as follows:

$$\text{Copper, \%} = (A/B) \times 100 \quad (1)$$

where:

A = copper deposited, g, and

B = sample used, g.

11.6 *Reserved Electrolyte*—If there is any brown deposit on the anode, place it in the reserved electrolyte and stir until the deposit dissolves. If necessary, add 1 or 2 drops of H₂SO₃ to aid in dissolution of the deposit. Transfer the solution to a 250-mL volumetric flask, dilute to the mark, and mix thoroughly. Reserve for determinations of nickel, cobalt, iron, and manganese as described in Sections 21 and 25, 36, 47, and 59, respectively.

12. Procedure for Alloys Containing 2 % and Over of Silicon

12.1 Transfer 1.0000 g of the sample to a 400-mL beaker. Add 20 mL of HClO₄-HNO₃ mixture. Cover and heat gently until action ceases. Rinse and remove the cover. Add HF dropwise, while swirling the solution, until the dark residue dissolves and the solution is clear green. Evaporate slowly to copious white fumes, heating carefully in order to avoid losses by spraying. Heat to incipient boiling; then cover the beaker. Proceed as described in 11.2-11.6.

13. Precision and Bias

13.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

COPPER BY THE SULFURIC ACID-ELECTROLYTIC TEST METHOD

14. Apparatus

14.1 *Electrodes for Electroanalysis*—Apparatus No. 9.

15. Procedure for Alloys Containing Under 0.50 % Silicon

15.1 Transfer 1.0000 g of the sample to a 300-mL, tall-form beaker. Add 15 mL of HNO₃(1+1). Cover and heat gently until brown fumes have been expelled. Boil gently for 5 min. Add 100 mL of water and 10 mL of H₂SO₄(1+1). Proceed as described in 11.4-11.6.

16. Procedure for Alloys Containing Under 2 % Silicon

16.1 Transfer 1.0000 g of the sample to a 400-mL beaker. Add 20 mL of HNO₃(1+1), cover, and heat gently until action ceases. Add 15 mL of H₂SO₄(1+1), insert a glass hook between the cover and beaker, and evaporate slowly to dense white fumes. Continue heating for 10 min; then let stand until cool.

16.2 Add 100 mL of water and heat until the sulfates dissolve, stirring occasionally to prevent bumping. Proceed as described in 11.3-11.6.



17. Procedure for Alloys Containing 2 % and Over of Silicon

17.1 Transfer 1.0000 g of the sample to a 400-mL beaker. Add 20 mL of $\text{HNO}_3(1+1)$. Cover and heat gently until action ceases. Rinse and remove the cover. Add HF drop by drop, while swirling the solution, until the dark residue dissolves and the solution is clear green. Add 15 mL of $\text{H}_2\text{SO}_4(1+1)$. Evaporate slowly to dense white fumes, heating carefully in order to avoid losses by spraying. Continue heating for 10 min; then let stand until cool.

17.2 Add 100 mL of water and heat until the sulfates dissolve, stirring occasionally to prevent bumping. Proceed as described in 11.3-11.6.

18. Precision and Bias

18.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

NICKEL BY THE DIMETHYLGLYOXIME- GRAVIMETRIC TEST METHOD

19. Apparatus

19.1 *Filter Crucible*—Apparatus No. 2. A 30 or 50-mL, medium-porosity glass filtering crucible shall be prepared each time before use as follows:⁵ Remove the bulk of the precipitate from the previous analysis mechanically. Immerse the crucible in HCl and heat for 1 h. With the aid of suction, wash with water, then NH_4OH , and finally with water. Dry at 150°C , cool in a desiccator, and weigh.

20. Reagents

20.1 *Ethanol Solution of Dimethylglyoxime* (10 g/L)—Reagent No. 104.

20.2 *Tartaric Acid Solution* (250 g/L)—Reagent No. 116.

21. Procedure

21.1 Transfer 50.0 mL of the reserved electrolyte from the copper determination (11.6) to a 600-mL beaker, or, alternatively, treat 0.2000 g of the sample as described in Sections 11, 12, 15, 16, or 17, and transfer the electrolyte to a 600-mL beaker. Add 1 mL of HNO_3 and heat to boiling. Add 300 mL of water and 10 mL of tartaric acid solution. Neutralize with NH_4OH and add 1 mL in excess. Heat to 60 to 70°C .

21.2 Add 0.4 mL of dimethylglyoxime solution (10 g/L) for each milligram of nickel present, and add 5 to 10 mL in excess. Stir the mixture vigorously and allow to cool to room temperature, stirring occasionally. Filter, using a fritted-glass crucible of medium porosity prepared as described in Section 19. Wash with cool water.

21.3 Add 5 mL of dimethylglyoxime solution (10 g/L) to the filtrate and let stand overnight in order to make certain that the

nickel was completely precipitated.

21.4 Dry the precipitate at 150°C to constant weight. Cool in a desiccator and weigh as nickel dimethylglyoxime.

21.5 *Calculation*—Calculate the percentage of nickel as follows:

$$\text{Nickel, \%} = [(A \times 0.2032)/B] \times 100 \quad (2)$$

where:

A = nickel dimethylglyoxime, g, and

B = sample used, g.

22. Precision and Bias

22.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

NICKEL BY THE DIMETHYLGLYOXIME- ELECTROLYTIC TEST METHOD

23. Apparatus

23.1 *Electrodes for Electroanalysis*—Apparatus No. 9.

24. Reagents

24.1 *Ethanol Solution of Dimethylglyoxime* (40 g/L)—Reagent No. 105.

24.2 *Tartaric Acid Solution* (250 g/L)—Reagent No. 116.

25. Procedure

25.1 Transfer 150.0 mL of the electrolyte from the copper determination to a 600-mL beaker, or, alternatively, treat 0.6000 g of the sample as described in Sections 11, 12, 15, 16, or 17, and transfer the electrolyte to a 600-mL beaker.

25.2 Dilute to 350 mL. Add 1 mL of HNO_3 and heat to boiling. Allow to cool somewhat, and add 10 mL of tartaric acid solution. Neutralize with NH_4OH and add 1 mL in excess. Heat to 60 to 70°C .

25.3 Add 0.1 mL of hot dimethylglyoxime solution (40 g/L) for each milligram of nickel present, and add 5 to 10 mL in excess. Stir the mixture vigorously and allow to cool to room temperature, stirring occasionally. Filter, using a 15-cm medium paper, and wash with cool water.

25.4 Add 1 or 2 mL of dimethylglyoxime solution (40 g/L) to the filtrate and let stand overnight in order to make certain that the nickel was completely precipitated.

25.5 Transfer the paper and precipitate to the precipitation beaker. Add 40 mL of HNO_3 and 15 mL of HClO_4 . Cover the beaker and heat until the paper disintegrates. Insert three glass hooks between the cover and beaker, and evaporate to dryness, guarding against loss from spraying. Heat the residue at 250°C until HClO_4 has been driven off completely. Let stand until cool.

25.6 Add 40 mL of $\text{H}_2\text{SO}_4(1+3)$ and heat until the nickel salts have been dissolved. Filter through a small paper into a 250-mL electrolytic beaker, and wash the paper with hot water. Cool the filtrate, neutralize with NH_4OH , and add 25 mL in excess.

⁵ Minster, J. T., "The Determination of Nickel by Precipitation with Dimethylglyoxime," *The Analyst*. ANALA, Vol 71, 1946, p. 427.

25.7 Dilute to 125 mL and electrolyze overnight, using platinum gauze electrodes and a current density of 0.2 to 0.3 A/dm². Remove the electrodes and wash them with water, taking the usual precautions against loss. Dip the cathode in two successive baths of methanol or ethanol, and dry in an oven at 110 °C for 3 to 5 min. Cool, and weigh the deposit as metallic nickel.

NOTE 2—Small amounts of nickel (0.2 to 0.5 mg) will usually remain in the electrolyte and in very accurate work may be determined as follows: Add 5 mL of bromine water, followed by 10 mL of dimethylglyoxime solution (10 g/L). Add standard nickel solution (1 mL = 0.1 mg Ni) to a blank electrolyte, similarly treated, until the colors of the solutions match, allowing sufficient time between additions of nickel solution for the reddish brown color to develop.

25.8 *Calculation*—Calculate the percentage of nickel as follows:

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

where:

A = nickel deposited, g, and

B = sample used, g.

26. Precision and Bias

26.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

COBALT BY THE ALPHA-NITROSO-beta-NAPHTHOL TEST METHOD

27. Reagents

27.1 *Alpha-Nitroso-beta-Naphthol Solution* (70 g/L)—Reagent No. 112.

27.2 *Zinc Oxide Suspension*—Transfer 50 g of finely powdered ZnO to a 500-mL flask and add 300 mL of water. Stopper the flask and shake vigorously each time before using.

28. Procedure

28.1 Transfer 3.00 g of the sample to a 400-mL beaker. Add 20 mL of HNO₃, cover, and heat gently until action ceases. Rinse and remove the cover. Add HF drop by drop, while swirling the solution, until any residue dissolves and the solution is clear green. Convert to perchlorates in accordance with 28.2, or, optionally, to sulfates in accordance with 28.3.

28.2 *Conversion to Perchlorates*—Add 20 mL of HClO₄ and evaporate to copious white fumes. Heat to incipient boiling, cover the beaker, and boil gently for 10 min. Allow to cool and add 125 mL of water. Proceed in accordance with 28.4.

28.3 *Conversion to Sulfates*—Add 30 mL of H₂SO₄(1+1) and evaporate slowly to dense white fumes. Continue heating for 10 min; then let stand until cool. Add 125 mL of water and heat until the sulfates dissolve, stirring occasionally to prevent bumping. Proceed in accordance with 28.4.

28.4 Remove the copper by electrolysis.

28.5 Transfer the electrolyte to a 500-mL wide-mouth flask. Add NH₄OH until most of the free acid has been neutralized but enough remains to keep the solution clear. Add 1 mL of HNO₃ and heat to boiling. Remove from the source of heat and, while swirling the solution, add ZnO suspension until an excess is indicated by white particles on the bottom of the flask and a milky turbidity in the supernatant liquid. Filter the solution through a medium paper into a 600-mL beaker, and wash the precipitate twice with hot water. Reserve the filtrate.

28.6 Transfer the paper and precipitate to the precipitation flask. Add 5 mL of HCl and stir with a glass rod until the precipitate dissolves and the paper disintegrates. Add 100 mL of hot water and repeat the precipitation with ZnO. Filter the solution through a medium paper into the reserved filtrate. Wash the precipitate with hot water and discard it. Add 20 mL of HCl to the filtrate and adjust the volume to 400 mL.

28.7 Heat to 60°C, add 15 mL of *α*-nitroso-*β*-naphthol solution, and stir vigorously for 1 min. Allow the solution to cool to room temperature, stirring occasionally. Filter, using a medium paper, and wash thoroughly with hot HCl (1+19) and then with hot water.

NOTE 3—**Warning:** Unless all of the HClO₄ is washed from the paper, it will cause a violent reaction during the subsequent ignition, with attendant losses.

28.8 Ignite the precipitate in a 30-mL tall-form porcelain crucible. Allow to cool, add 1 to 2 g of NaHSO₄, and heat on a hot plate until dense white fumes appear. Fuse the oxides and let stand until cool. Tap gently to detach the melt and transfer it to a 400-mL beaker. Rinse the crucible thoroughly with hot water and add the rinsings to the beaker. Digest until the melt dissolves, add 10 mL of HCl, and dilute to 200 mL.

28.9 Reprecipitate the cobalt, filter, and wash as described in 28.7.

28.10 Ignite the precipitate in a tared porcelain crucible to constant weight at 750 to 850°C. Cool in a desiccator and weigh as Co₃O₄.

28.11 *Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

28.12 *Calculation*—Calculate the percentage of cobalt as follows:

$$\text{Cobalt, \%} = ([A - B] \times 0.7342)/C \times 100 \quad (4)$$

where:

A = Co₃O₄ from the sample, g,

B = correction for blank, g, and

C = sample used, g.

29. Precision and Bias

29.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

COBALT BY THE NITROSO-R-SALT- PHOTOMETRIC TEST METHOD

30. Summary of Test Method

30.1 Cobalt, in a hot solution buffered with sodium acetate, forms an orange-colored complex with nitroso-R-salt. The addition of a controlled amount of HNO_3 destroys interfering complexes and stabilizes the cobalt complex. Photometric measurement is made at approximately 520 nm.

31. Concentration Range

31.1 The recommended concentration range is from 0.005 to 0.10 mg of cobalt in 50 mL of solution, using a cell depth of 2 cm.⁶

32. Stability of Color

32.1 The color is stable for more than 2 h.

33. Interfering Elements

33.1 Under the conditions of this test method, the elements normally present in nickel-copper alloys do not interfere if the contents are under the maximum limits shown in 1.1.

34. Reagents

34.1 *Cobalt Standard Solution* (1 mL = 0.01 mg Co):

34.1.1 Transfer 0.1000 g of cobalt to a 1-L volumetric flask. Add 10 mL of $\text{HNO}_3(1+1)$, heat gently until action ceases, and then boil until free of brown fumes. Cool, dilute to the mark, and mix. Transfer 100 mL of this solution to a 1-L volumetric flask, dilute to the mark, and mix.

34.1.2 Alternatively, transfer 0.4770 g of $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ to a 1-L volumetric flask. Add 75 mL of water and 4 mL of $\text{H}_2\text{SO}_4(1+1)$. Swirl until the salt dissolves, dilute to the mark, and mix. Standardize the solution as follows: Transfer a 100-mL aliquot to a 400-mL beaker, add 10 mL of HCl, and dilute to 200 mL. Proceed as described in 28.7, 28.10, and 28.11. For use, dilute 100 mL of this solution to 1 L in a volumetric flask and mix.

34.2 *Nitroso-R-Salt Solution* (7.5 g/L)—Dissolve 0.75 g of nitroso-R-salt in water, filter, and dilute to 100 mL. Do not use solutions more than 1 week old.

34.3 *Sodium Acetate Solution* (500 g/L)—Dissolve 500 g of sodium acetate trihydrate in about 600 mL of water, filter, and dilute to 1 L.

35. Preparation of Calibration Curve

35.1 *Calibration Solutions*—Transfer 1.0, 2.0, 4.0, 6.0, 8.0, and 10.0 mL of cobalt solution (1 mL = 0.01 mg Co) to six 50-mL beakers. Dilute to 10 mL and proceed in accordance with 35.3.

35.2 *Reference Solution*—Transfer 10 mL of water to a 50-mL beaker and proceed in accordance with 35.3.

35.3 *Color Development*—Add 5 mL of sodium acetate solution, followed by 2.0 mL of nitroso-R-salt solution, swirl-

ing the solution after each addition (Note 4). Cover the beaker, heat to boiling, and maintain just under the boiling temperature for 1 to 2 min. Add 5.0 mL of $\text{HNO}_3(1+2)$ and boil gently for 1 to 2 min. Cool to room temperature. Transfer to a 50-mL volumetric flask, dilute to the mark, and mix.

NOTE 4—The pH of the solutions at this point should be about 5.5.

35.4 *Photometry*—Transfer a suitable portion of the reference solution to an absorption cell and adjust the photometer to the initial setting, using a light band centered at approximately 520 nm. While maintaining this photometer adjustment, take the photometric readings of the calibration solutions.

35.5 *Calibration Curve*—Plot the photometric readings of the calibration solutions against milligrams of cobalt per 50 mL of solution.

36. Procedure

36.1 *Sample Solution*—Transfer a 2.0 to 5.0-mL aliquot of the reserved electrolyte from the copper determination (11.6) to a 50-mL beaker. **Warning**—If HClO_4 is present, add 1 mL of $\text{H}_2\text{SO}_4(1+3)$. Evaporate to dryness. Add 10 mL of water and one drop of $\text{H}_2\text{SO}_4(1+3)$, cover, and heat until the salts dissolve. Develop the color as described in 35.3.

36.2 *Reference Solution*—Transfer 10 mL of water to a 50-mL beaker and proceed as described in 35.3.

36.3 *Photometry*—Take the photometric reading of the sample solution as described in 35.4.

36.4 *Reagent Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

NOTE 5—This correction is ignored in routine work.

36.5 *Background Color*—Transfer to a 50-mL beaker the same volume of the reserved electrolyte as was taken for the sample solution and treat it as described in 36.1. Continue as described in 35.3, but omit the addition of nitroso-R-salt. Take the photometric reading of this solution, using an equal aliquot of the reagent blank solution, similarly treated, as the reference solution (see Note 5).

36.6 *Calculation*—Convert the photometric readings of the sample, reagent blank, and background color solutions to milligrams of cobalt by means of the calibration curve. Calculate the percentage of cobalt as follows:

$$\text{Cobalt, \%} = (A - B - C)/(D \times 10) \quad (5)$$

where:

- A = cobalt found in the aliquot used, mg,
- B = reagent blank correction, mg of cobalt,
- C = background color correction, mg of cobalt, and
- D = sample represented in the aliquot used, g.

37. Precision and Bias

37.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

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



IRON BY THE STANNOUS CHLORIDE- POTASSIUM DICHROMATE TEST METHOD

38. Reagents

38.1 *Mercuric Chloride Solution (saturated)*—Dissolve 80 g of HgCl_2 in 1 L of hot water, cool to room temperature, and filter.

38.2 *Potassium Dichromate Standard Solution (0.02 N)*—See Reagent No. 10.

38.3 *Sodium Diphenylamine Sulfonate Indicator (2 g/L)*—Reagent No. 121.

38.4 *Stannous Chloride Solution (50 g/L)*—Dissolve 5 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 10 mL of HCl and dilute to 100 mL.

38.5 *Sulfuric-Phosphoric Acid Mixture*—Add 150 mL of H_2SO_4 and 150 mL of H_3PO_4 to 700 mL of cold water slowly, while stirring.

39. Procedure

39.1 *Silicon Under 0.5 %*—Transfer 1.000 g of the sample to a 250-mL beaker. Add 15 mL of HNO_3 , cover, and heat gently until action ceases. Boil until free of brown fumes. Proceed in accordance with 39.3.

39.2 *Silicon 0.5 % and Over*—Transfer 1.000 g of the sample to a 250-mL beaker. Add 15 mL of HNO_3 , cover, and heat gently until action ceases. Rinse and remove the cover. Add HF drop by drop, while swirling the solution, until the residue dissolves and the solution is clear green. Boil for 5 min. Proceed in accordance with 39.3.

39.3 Add 35 mL of hot water and 40 mL of NH_4OH . Boil for 1 min and filter, using a coarse paper. Wash twice with NH_4OH (1+19).

39.4 Sluice the precipitate into the precipitation beaker. Add 10 mL of HCl and heat until the precipitate dissolves. Add 20 mL of NH_4OH and boil for 1 min. Filter, using the original paper, and wash with hot water.

39.5 Dissolve the precipitate in 40 mL of hot HCl (1+3) (Note 6), receiving the solution in a 300-mL conical flask. Wash the paper free of iron with hot HCl (1+19). Heat the yellow solution to boiling. While swirling the boiling solution, add SnCl_2 solution dropwise until the yellow color is discharged, and add 1 or 2 drops in excess. Dilute to 100 mL and cool in running water.

NOTE 6—A few drops of SnCl_2 solution may be added to the HCl (1+3) to dissolve manganese oxides, if present.

39.6 Add 10 mL of HgCl_2 solution rapidly, while swirling the cool solution. Let stand 5 min, then add 15 mL of H_2SO_4 - H_3PO_4 mixture and 3 drops of sodium diphenylamine sulfonate indicator. Titrate with $\text{K}_2\text{Cr}_2\text{O}_7$ solution to a reddish-blue end point.

39.7 *Blank*—Make a blank determination, following the same procedure and using the same amounts of all reagents.

39.8 *Calculation*—Calculate the percentage of iron as follows:

$$\text{Iron, \%} = [(A - B)C \times 0.0559]/D \times 100 \quad (6)$$

where:

A = $\text{K}_2\text{Cr}_2\text{O}_7$ solution required to titrate the sample, mL,

B = $\text{K}_2\text{Cr}_2\text{O}_7$ solution required to titrate the blank, mL,

C = normality of the $\text{K}_2\text{Cr}_2\text{O}_7$ solution, and

D = sample used, g.

40. Precision and Bias

40.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify, by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

IRON BY THE THIOCYANATE-PHOTOMETRIC TEST METHOD

41. Summary of Test Method

41.1 Ferric iron forms a red-brown soluble complex with thiocyanate in acid solution. Photometric measurement is made at approximately 490 nm.

42. Concentration Range

42.1 The recommended range is from 0.015 to 0.25 mg of iron in 100 mL of solution, using a cell depth of 2 cm.⁵

43. Stability of Color

43.1 The color develops immediately and is stable for 30 min if protected from direct sunlight.

44. Interfering Elements

44.1 Under the conditions of this test method, the elements normally present in nickel-copper alloys do not interfere.

45. Reagents

45.1 *Ammonium Persulfate Solution (5 g $(\text{NH}_4)_2\text{S}_2\text{O}_8/\text{L}$)*—Prepare the required amount just before using.

45.2 *Iron Standard Solution (1 mL = 0.025 mg Fe)*—Transfer 0.1756 g of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ to a 400-mL beaker. Add 100 mL of water and 5 mL of HNO_3 . Boil for 5 min. Cool to room temperature. Transfer to a 1-L volumetric flask, dilute to the mark, and mix.

45.3 *Sodium Thiocyanate (200 g NaCNS/L)*—Dissolve 200 g of NaCNS in 500 mL of water, filter, and dilute to 1 L. Store in a dark place.

46. Preparation of Calibration Curve

46.1 *Calibration Solutions*—Transfer 2.0, 4.0, 6.0, 8.0, 10.0, and 12.0 mL of iron solution (1 mL = 0.025 mg Fe) to six 100-mL volumetric flasks. Dilute to 60 mL and proceed in accordance with 46.3.

46.2 *Reference Solution*—Transfer 60 mL of water to a 100-mL volumetric flask and proceed in accordance with 46.3.

46.3 *Color Development*—Add 10 mL of HCl (1+9), 10 mL of $(\text{NH}_4)_2\text{S}_2\text{O}_8$ solution, and 10 mL of NaCNS solution, swirling the solution after each addition. Dilute to the mark and mix.

46.4 *Photometry*—Transfer a suitable portion of the reference solution to an absorption cell and adjust the photometer to the initial setting, using a slight band centered at approximately 490 nm. While maintaining this photometer adjustment, take