Designation: E 106 – 83 (Reapproved 2004)

Standard Test Methods for Chemical Analysis of Copper-Beryllium Alloys[1]

This standard is issued under the fixed designation E 106; 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 copper-beryllium alloys having chemical compositions within the following limits:

Element	Concentration Range,%
Copper	97 to 98
Beryllium	0.4 to 2.05
Nickel	0.0 to 0.30
Cobalt	0.0 to 0.3
Iron	0.0 to 0.30

1.2 The analytical procedures appear in the following order:

	Sections
Copper by the Electrolytic Method	8-12
Beryllium:	
Phosphate Gravimetric Method	13-19
Aluminon (Photometric) Method	20-27
Nickel by the Dimethylglyoxime (Photometric) Method	28-36
Cobalt by the Nitroso-R-Salt (Photometric) Method	37-44
Iron by the Thiocyanate (Photometric) Method	45-52

2. Referenced Documents

2.1 *ASTM Standards:* [2]

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

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

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

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

E 76 Test Methods for Chemical Analysis of Nickel-Copper Alloys

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[3]

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 methods shall conform to Practice E 60.

5. Safety Precautions

5.1 For precautions to be observed in these methods, reference shall be made to Practices E 50. Both beryllium metal and its compounds may be toxic. Care should be exercised to prevent contact of beryllium-containing materials with the skin. The inhalation of any beryllium-containing substance, either as a volatile compound or as finely divided powder, should be especially avoided. Beryllium-containing residues (especially ignited oxide) should be carefully disposed of.

6. Sampling

6.1 Sampling shall conform to Practice E 55.

7. Rounding Off Calculated Values

7.1 Calculated values shall be rounded off to the desired number of places in accordance with the rounding-off method given in 3.4 and 3.5 of Practice E 29.

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[1] These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.05 on Cu, Pb, Zn, Cd, Sn, Be, their Alloys and Related Metals.

Current edition approved June 1, 2004. Published July 2004. Originally approved in 1954. Last previous edition approved in 1996 as E 106 – 83 (1996).

[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.

COPPER BY THE ELECTROLYTIC TEST METHOD

8. Apparatus

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

9. Reagents

9.1 *Sulfuric-Nitric Acid Mixture*—Add slowly, while stirring, 300 mL of H_2SO_4 to 750 mL of water. Cool and add 210 mL of HNO_3.

10. Procedure

10.1 Transfer 5.00 g of sample to a 300-mL electrolysis beaker. Add 42 mL of the H_2SO_4-HNO_3 mixture, cover, and allow to stand a few minutes until reaction has nearly ceased. Heat at 80 to 90°C until dissolution is complete and brown fumes have been expelled. Wash down the cover glass and the sides of the beaker and dilute to about 175 mL (enough to submerge the cathode when it is inserted).

10.2 Insert the electrodes, cover the solution with a pair of split watch glasses, and electrolyze at a current density of about 0.6 A/dm^2 for about 16 h. Wash down the cover glasses, sides of beaker, and electrode stems and continue electrolysis for about 15 min. If no copper plates on the newly exposed cathode surface, copper deposition may be considered completed.

10.3 Quickly withdraw the cathode from the electrolyte while directing a gentle stream of water from a wash bottle over its surface. Rinse the cathode in a water bath and then dip in two successive baths of ethanol or acetone. Dry in an oven at 110°C for 3 to 5 min, cool, and weigh. Reserve the spent electrolyte.

where:
A = grams of copper, and
B = grams of sample used.

10.4 *Reserved Electrolyte*—Evaporate the spent electrolyte to dense white fumes and fume for about 5 min to dehydrate silicic acid. Cool, add about 50 mL of water, and heat until all salts are in solution. Filter through a small, medium-texture paper, catching the filtrate in a 250-mL volumetric flask. Wash the beaker and paper thoroughly with hot H_2SO_4 (1 + 99), combining the washings with the filtrate. Cool the solution in the volumetric flask, dilute to the mark, and mix. Reserve for the determinations of beryllium, nickel, cobalt, and iron as described in Sections 17, 34, 43, and 51 respectively. If the filtrate is not to be used for the gravimetric determination of beryllium, the removal of silica is not necessary and the electrolyte may be diluted to volume directly.

11. Calculation

11.1 Calculate the percentage of copper as follows:

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

12. Precision and Bias

12.1 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.

BERYLLIUM BY THE PHOSPHATE GRAVIMETRIC TEST METHOD

13. Scope

13.1 This test method covers the determination of beryllium in concentrations from 0.1 to 3.0 %.

14. Summary of Test Method

14.1 Interfering elements are complexed with (ethylenedinitrilo) tetraacetate solution. Beryllium is precipitated as the phosphate, which is filtered, ignited, and weighed as beryllium pyrophosphate.

15. Interferences

15.1 The elements ordinarily present in beryllium-copper alloys do not interfere if their concentrations are under the maximum limits shown in 1.1.

16. Reagents

16.1 *Ammonium Acetate Solution* (500 g/L)—Dissolve 500 g of ammonium acetate in water, and dilute to 1 L.

16.2 *Ammonium Acetate Wash Solution*—Dilute 5 mL of the ammonium acetate solution to 1 L, and adjust the pH to 5.2 ± 0.05 with acetic acid.

NOTE 1—Use a pH meter for all pH adjustments.

16.3 *Ammonium Dihydrogen Phosphate* (100 g/L)—Dissolve 100 g of ammonium dihydrogen phosphate ($NH_4H_2PO_4$) in water and dilute to 1 L.

16.4 *Ammonium (Ethylenedinitrilo) Tetraacetate Solution* (28 g/L)—To 2.5 g of (ethylenedinitrilo) tetraacetic acid add 30 mL of water and a drop of methyl red solution. Neutralize with NH_4OH (1 + 1), and warm gently to dissolve the last traces of solid. Cool and dilute to 100 mL.

16.5 *Methyl Red Indicator Solution* (0.5 g/L ethanol)—Dissolve 0.05 g of methyl red in 100 mL of ethanol.

17. Procedure

17.1 Using a pipet, transfer 50 mL of the electrolyte reserved in 10.4 to a 400-mL beaker. Add 3 drops of HF and 10 mL of H_2SO_4(1 + 2), and evaporate to fumes. Cool to room temperature and add 100 mL of water. Heat to dissolve soluble salts and again cool to room temperature.

17.2 Add 10 mL of ammonium (ethylenedinitrilo) tetraacetate solution, and adjust the pH to 2.0 ± 0.05 (see Note 1) with NH_4OH (1 + 1). Boil 1 min and cool to room temperature. Add 10 mL of ammonium dihydrogen phosphate solution and adjust the pH to 5.2 ± 0.05 with ammonium acetate solution.

17.3 Heat to boiling cautiously to prevent bumping, and then maintain just below the boiling point until the precipitate becomes granular. Remove from the source of heat and allow to stand at least 12 h.

17.4 Filter using an 11-cm fine paper and wash six times with ammonium acetate wash solution. Discard the filtrate.

Dissolve the precipitate with 100 mL of hot HCl (1 + 4), collecting the solution in the original beaker.

17.5 Add 2 mL of ammonium (ethylenedinitrilo) tetraacetate solution, and adjust the pH to 2.0 ± 0.05 with NH_4OH (1 + 1). Cool, add 2 mL of ammonium dihydrogen phosphate solution, and adjust the pH to 5.2 ± 0.05 with ammonium acetate solution. Proceed as directed in 17.3.

17.6 Filter using an 11-cm fine paper and wash six times with ammonium acetate wash solution. Transfer the paper to a weighed platinum crucible. Place the crucible in a muffle furnace, and dry and char the paper by gradually increasing the temperature to 500°C. When all the carbon has been removed, raise the temperature to 1000°C and maintain at this temperature for 4 h. Cool in a desiccator and weigh.

18. Calculation

18.1 Calculate the percentage of beryllium as follows:

$$\text{Beryllium, \%} = (A \times 0.0939/B) \times 100 \tag{2}$$

where:

A = grams of beryllium pyrophosphate, and
B = grams of sample used

19. Precision and Bias

19.1 *Precision*—Eight laboratories cooperated in testing this method and obtained the data summarized in Table 1.

19.2 *Bias*—No certified reference materials suitable for testing this test method were available when the interlaboratory testing program was conducted. The user of this standard is encouraged to employ accepted reference materials, if available, to determine the accuracy of this test method as applied in a specific laboratory.

TABLE 1 Statistical Information

Test Specimen	Beryllium Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
(1) Beryllium copper, B-7	1.744	0.026	0.042
(2) Beryllium copper, C-7	0.460	0.020	0.046

BERYLLIUM BY THE ALUMINON (PHOTOMETRIC) TEST METHOD

20. Principle of Test Method

20.1 In a properly buffered solution, ammonium aurin tricarboxylate (aluminon) forms a red lake with beryllium. The addition of ethylenediamine tetraacetic acid (complexone) prevents the interference of aluminum, iron, copper, and similar elements. Photometric measurement is made at approximately 515 nm.

21. Concentration Range

21.1 The recommended concentration range is from 0.004 to 0.09 mg of beryllium in 100 mL of solution, using a cell depth[4] of 2 cm.

22. Stability of Color

22.1 The intensity of the color of the beryllium lake increases slowly on standing. Therefore, a uniform standing time must be adhered to.

23. Interfering Elements

23.1 Provision is made in the procedure for preventing, or compensating for, interference from metals present in amounts not exceeding the maximum limits given in 1.1

24. Reagents

24.1 *Aluminon-Buffer Composite Solution*—Add 500 g of ammonium acetate to 1 L of water in a 2-L beaker. Add 80 mL of glacial acetic acid and stir until dissolution is complete. Filter if necessary. Dissolve 1.000 g of a suitable grade of aluminon[5] (aurin tricarboxylic acid-ammonium salt) in 50 mL of water and add to the buffer solution. Dissolve 3 g of benzoic acid in 20 mL of methanol and add to the buffer solution while stirring. Dilute the mixture to 2 L. Add 10 g of gelatin[6] to 250 mL of water in a 400-mL beaker. Place the beaker in a boiling water bath and allow to remain, with frequent stirring, until the gelatin has dissolved completely. Pour the warm gelatin solution into 500 mL of distilled water, while stirring. Cool to room temperature, dilute to 1 L, and mix. Transfer the aluminon and gelatin solutions to a 4-L chemically resistant glass-stoppered bottle, mix well, and store in a cool, dark place.

24.2 *Complexone Solution*—See 16.4.

24.3 *Copper Chloride Solution* (1 mL = 2 mg Cu)—Dissolve 0.54 g of $CuCl_2 \cdot 2H_2O$ in water and dilute to 100 mL in a volumetric flask.

24.4 *Standard Beryllium Solution* (1 mL = 1.0 mg Be)—Dissolve 9.82 g of $BeSO_4 \cdot 4H_2O$ in 100 mL of HCl (1 + 3). Filter, if necessary, and dilute to 500 mL. Standardize as follows: Transfer 25 mL of the solution to a 250-mL beaker and proceed in accordance with Section 17.2-17.6 and 18.1.

24.5 *Standard Beryllium Solution* (1 mL = 0.01 mg Be)—Transfer 10 mL of the above solution to a 1-L volumetric flask, add 10 mL of HCl, dilute to the mark, and mix.

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[4] 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.

[5] Certain commercially available grades of aluminon have been found to be unsatisfactory for this purpose. It may be necessary to prepare a small portion of the composite reagent before use. The currently available (1954) product from Eastman Kodak appears to be satisfactory.

[6] Knox gelatin has been found satisfactory for this purpose.

25. Preparation of Calibration Curve

25.1 *Calibration Solutions*—Transfer 1.0, 2.0, 4.0, 5.0, 7.0, and 9.0 mL of beryllium solution (1 mL = 0.01 mg Be) to 100-mL volumetric flasks. Add 1 mL of $CuCl_2$ solution (1 mL = 2 mg Cu) to each flask and dilute to about 75 mL.

25.2 *Reference Solution*—Add 1 mL of $CuCl_2$ solution (1 mL = 2 mg Cu) to a 100-mL volumetric flask and dilute to about 75 mL.

25.3 *Color Development*—Add 2 mL of complexone solution and 15 mL of aluminon buffer composite solution to each flask, mixing well between additions. Dilute to the mark and mix without delay. Immediately transfer portions of the solutions to absorption cells and allow to stand away from direct sunlight for exactly 20 min from the time of addition of the aluminon buffer composite solution.

25.4 *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 515 nm. While maintaining this photometer adjustment, take the photometric readings of the calibration solutions.

25.5 *Calibration Curve*—Plot the photometric readings of the calibration solutions against milligrams of beryllium per 100 mL of solution.

26. Procedure

26.1 *Sample Solution*—Transfer 0.2000 g of the sample to a 500-mL volumetric flask. Add 5 mL of HCl and then, cautiously, 5 mL of H_2O_2(30 %). Cool if the reaction becomes too violent. When dissolution is complete, wash down the sides of the flask and boil gently for about 10 min to destroy excess H_2O_2. Cool, dilute to the mark, and mix. Transfer 5 mL of this solution to a 100-mL volumetric flask and dilute to about 75 mL.

26.2 *Reference Solution*—Proceed in accordance with 25.2.

26.3 *Color Development*—Proceed in accordance with 25.3.

26.4 *Photometry*—Proceed in accordance with 25.4.

26.5 *Calculation*—Convert the photometric reading of the sample solution to milligrams of beryllium by means of the calibration curve. Calculate the percentage of beryllium as follows:

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

where:

A = milligrams of beryllium found in the aliquot used, and

B = grams of sample represented in the aliquot used.

27. Precision and Bias

27.1 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 (PHOTOMETRIC) TEST METHOD

28. Summary of Test Method

28.1 Nickel after oxidation with bromine, forms a red-colored, soluble salt with dimethylglyoxime. Photometric measurement is made at approximately 530 nm.

29. Concentration Range

29.1 The recommended concentration range is from 0.02 to 0.40 mg of nickel per 100 mL of solution, using a cell depth[4] of 2 cm.

30. Stability of Color

30.1 The intensity of the color increases gradually for approximately 30 min and then starts to fade slowly.

31. Interferences

31.1 The elements ordinarily present in beryllium-copper alloys, except manganese, do not interfere if their concentrations are under the maximum limits shown in 1.1. Manganese, if not removed, will cause a positive error equal to about 1 % of the manganese content.

32. Reagents

32.1 *Alcohol Solution of Dimethylglyoxime* (10 g/L)—Reagent No. 104.

32.2 *Bromine Water (Saturated)*.

32.3 *Citric Acid Solution* (100 g/L)—Dissolve 100 g of citric acid in water and dilute to 1 L. Add 1 g of benzoic acid to prevent bacterial growth.

32.4 *Hydrogen Sulfide Solution*—Saturate sodium hydroxide (NaOH) solution (0.2 g/L) with hydrogen sulfide (H_2S). Prepare fresh as needed.

32.5 *Nickel, Standard Solution* (1 mL = 0.02 mg Ni)—Dissolve 0.200 g of nickel (purity, 99.9 % min) in 20 mL of HNO_3(1 + 1). Boil to expel oxides of nitrogen. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Transfer a 100-mL aliquot to a 1-L volumetric flask, dilute to volume, and mix.

33. Preparation of Calibration Curve

33.1 *Calibration Solutions*—Transfer 1.0, 2.0, 5.0, 10.0, 15.0, and 20.0 mL of nickel solution (1 mL = 0.02 mg Ni) to six 100-mL volumetric flasks. Add 5 mL of citric acid solution and dilute to approximately 50 mL. Proceed as directed in 33.3.

33.2 *Reference Solution*—Transfer 5 mL of citric acid solution to a 100-mL volumetric flask and dilute to approximately 50 mL. Proceed as directed in 33.3.

33.3 *Color Development*—Add 5 mL of bromine water. Add NH_4OH (1 + 1) dropwise, to just bleach the bromine, and then add 3 mL in excess. Cool rapidly and add 3 mL of dimethylglyoxime solution (Note 2). Dilute to volume, mix, and allow to stand for a definite period, preferably 10 min, after the addition of dimethylglyoxime solution.

NOTE 2—The addition of bromine water, NH_4OH, and dimethylglyoxime solution must be completed within a period of 5 min.

33.4 *Photometry*—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 530 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

33.5 *Calibration Curve*—Plot the photometric readings of the calibration solutions against milligrams of nickel per 100 mL of solution.

34. Procedure

34.1 *Test Solution*—Transfer a 5-mL aliquot of the solution reserved in accordance with 10.4 to a 100-mL volumetric flask. Add 5 mL of citric acid solution and dilute to about 50 mL.

34.2 *Reference Solution*—Carry a reagent blank through the entire procedure, using the same amount of all reagents, for use as a reference solution.

34.3 *Color Development*—Proceed as directed in 33.3.

34.4 *Photometry*—Take the photometric reading of the test solution as directed in 33.4.

35. Calculation

35.1 Convert the photometric reading of the test solution to milligrams of nickel by means of the calibration curve. Calculate the percentage of nickel as follows:

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

where:

A = milligrams of nickel found in 100 mL of the final solution, and

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

36. Precision and Bias

36.1 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

37. Summary of Test Method

37.1 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.

38. Concentration Range

38.1 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.[4]

39. Stability of Color

39.1 The color is stable for more than 2 h.

40. Interfering Elements

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

41. Reagents

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

41.1.1 Transfer 0.1000 g of cobalt to a 1-L volumetric flask. Add 10 mL of 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.

41.1.2 Alternatively, transfer 0.4770 g of $CoSO_4 \cdot 7H_2O$ to a 1-L volumetric flask. Add 75 mL of water and 4 mL of H_2SO_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 in accordance with 22.7, 22.10, and 22.11 of Methods E 76. For use, dilute 100 mL of this solution to 1 L in a volumetric flask and mix.

41.2 *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.

41.3 *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.

42. Preparation of Calibration Curve

42.1 *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 42.3.

42.2 *Reference Solution*—Transfer 10 mL of water to a 50-mL beaker and proceed in accordance with 42.3.

42.3 *Color Development*—Add 5 mL of sodium acetate solution, followed by 2.0 mL of nitroso-R-salt solution, swirling the solution after each addition (Note 3). Cover the beaker, heat to boiling, and maintain just under the boiling temperature for 1 to 2 min. Add 5.0 mL of 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 3—The pH of the solutions at this point should be about 5.5.

42.4 *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.

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

43. Procedure

43.1 *Sample Solution*—Transfer an appropriate aliquot of the solution reserved in accordance with 10.4 to a 50-mL beaker and dilute to about 10 mL. Develop the color as described in 42.3.

43.2 *Reference Solution*—Transfer 10 mL of water to a 50-mL beaker and proceed as described in 42.3.

43.3 *Photometry*—Take the photometric reading of the sample solution as described in 42.4.

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

NOTE 4—This correction is ignored in routine work.

43.5 *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 43.1. Continue as described in 42.3, 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 4).

43.6 *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 = milligrams of cobalt found in the aliquot used,
B = reagent blank correction, mg of cobalt,
C = background color correction, mg of cobalt, and
D = grams of sample represented in the aliquot used.

44. Precision and Bias

44.1 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

45. Summary of Test Method

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

46. Concentration Range

46.1 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.[4]

47. Stability of Color

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

48. Interfering Elements

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

49. Reagents

49.1 *Ammonium Persulfate Solution* (5 g $(NH_4)_2S_2O_8$/L)—Prepare the required amount just before using.

49.2 *Iron Standard Solution* (1 mL = 0.025 mg Fe)—Transfer 0.1756 g of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ 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.

49.3 *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.

50. Preparation of Calibration Curve

50.1 *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 50.3.

50.2 *Reference Solution*—Transfer 60 mL of water to a 100-mL volumetric flask and proceed in accordance with 50.3.

50.3 *Color Development*—Add 10 mL of HCl (1 + 9), 10-mL of $(NH_4)_2S_2O_8$ solution and 10 mL of NaCNS solution, swirling the solution after each addition. Dilute to the mark and mix.

50.4 *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 490 nm. While maintaining this photometer adjustment, take photometric readings of the calibration solutions, within 30 min after developing the color.

50.5 *Calibration Curve*—Plot the photometric readings of the calibration solutions against milligrams of iron per 100 mL of solution.

51. Procedure

51.1 *Sample Solution*—Transfer a 5-mL aliquot of the solution reserved in accordance with 10.5 to a 100-mL volumetric flask and dilute to about 60 mL. Develop the color as described in 50.3.

51.2 *Reference Solution*—Transfer to a 100-mL volumetric flask the same volume of the reserved electrolyte as was taken for the sample solution. Dilute to 55 to 60 mL and treat as described in 50.3, except to omit the addition of NaCNS solution.

51.3 *Photometry*—Take the photometric reading of the sample solution as described in 50.4.

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

51.5 *Calculation*—Convert the photometric readings of the sample and blank solutions to milligrams of iron by means of the calibration curve. Calculate the percentage of iron as follows:

$$\text{Iron, \%} = (A - B)/(C \times 10) \quad (6)$$

where:

A = milligrams of iron found in the aliquot used,
B = reagent blank correction, mg of iron, and
C = grams of sample represented in the aliquot used.

52. Precision and Bias

52.1 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.

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