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

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[1] These test methods are under the jurisdiction of ASTM Committee ~~E-1~~ 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.

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1. Scope

1.1 These test methods cover the chemical analysis of copper alloys having chemical compositions within the following limits:[2]

Element	Concentration, %
Aluminum	12.0 max
Antimony	1.0 max
Arsenic	1.0 max
Cadmium	1.5 max
Cobalt	1.0 max
Copper	40.0 min
Iron	6.0 max
Lead	27.0 max
Manganese	6.0 max
Nickel	50.0 max
Phosphorus	1.0 max
Silicon	5.0 max
Sulfur	0.1 max
Tin	20.0 max
Zinc	50.0 max

1.2 The test methods appear in the following order:

	Sections
~~Aluminum by the Carbamate Extraction-(Ethylenedinitrilo) Tetraacetate Titrimetric Test Method~~	~~70-77~~
Aluminum by the Carbamate Extraction-(Ethylenedinitrilo) Tetraacetate Titrimetric Test Method [2 to 12 %]	70-77
Copper by the Combined Electrodeposition Gravimetric and Oxalyldihydrazide Photometric Test Method [50 %, minimum]	9-17
~~Iron by the 1,10-Phenanthroline Photometric Test Method [0.003 to 1.25 %]~~	~~18-27~~
Iron by the 1,10-Phenanthroline Photometric Test Method [0.003 to 1.25 %]	18-27
~~Lead by the Atomic Absorption Test Method~~	~~89-99~~
Lead by the Atomic Absorption Test Method [0.002 to 15 %]	89-99
~~Lead by the (Ethylenedinitrilo)tetraacetic Acid (EDTA) Titrimetric Test Method [2.0 to 30.0 %]~~	~~28-35~~
Lead by the (Ethylenedinitrilo)tetraacetic Acid (EDTA) Titrimetric Test Method [2.0 to 30.0 %]	28-35
~~Nickel by the Dimethylglyoxime Extraction Photometric Test Method [0.03 to 5.0 %]~~	~~36-45~~
Nickel by the Dimethylglyoxime Extraction Photometric Test Method [0.03 to 5.0 %]	36-45
~~Nickel by the Dimethylglyoxime Gravimetric Test Method [4 to 50 %]~~	~~54-61~~

[2] The actual limits of application of each test method are presented in 1.2.

Nickel by the Dimethylglyoxime Gravimetric Test Method [4 to 50 %]	54-61
~~Silver in Silver-Bearing Copper by the Atomic Absorption Test Method~~	~~100-111~~
Silver in Silver-Bearing Copper by the Atomic Absorption Test Method [0.01 to 0.12 %]	100-111
~~Tin by the Iodatimetric Titration Test Method [0.5 to 20 %]~~	~~62-69~~
Tin by the Iodatimetric Titration Test Method [0.5 to 20 %]	62-69
~~Tin by the Phenylfluorone Photometric Test Method %]~~	~~112-122~~
Tin by the Phenylfluorone Photometric Test Method [0.01 to 1.0 %]	112-122
~~Zinc by Atomic Spectrometry~~	~~78-88~~
Zinc by Atomic Spectrometry [0.2 to 2 %]	78-88
~~Zinc by the (Ethylenedinitrilo)tetraacetic Acid (EDTA) Titrimetric Test Method [2 to 40 %]~~	~~46-53~~
Zinc by the (Ethylenedinitrilo)tetraacetic Acid (EDTA) Titrimetric Test Method [2 to 40 %]	46-53

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 hazard statements are given in Section 5, Note 4, and Section 106.~~

2. Referenced Documents

2.1 *ASTM Standards:*

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]

E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[4]

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[4]

E 255 Practice for Sampling Copper and Copper Alloys for Determination of Chemical Composition[4]

E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry[4]

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method[4]

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended as referee methods to test such materials for compliance with compositional specifications. It is assumed that all who use these 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. ~~S~~Hazards

5.1 Specific hazard statement~~y P~~s are given in Section 5, Note 4, a~~u~~nd Section~~s~~ 106.

5.~~1~~2 For other 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 Practice E 255. However, this method does not supersede any sampling requirements specified in a specific ASTM material specification.

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~~ were evaluated in accordance with Practice E 173 unless otherwise noted in the precision section. E 173 has been replaced by Practice E 1601. The Reproducibility Index R_2 corresponds to the Reproducibility Index R

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

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

of Practice E 1601. Likewise the Repeatability Index R_1 of E 173 corresponds to Repeatablility Index r of Practice E 1601.

COPPER BY THE COMBINED ELECTRODEPOSITION GRAVIMETRIC AND OXALYLDIHYDRAZIDE PHOTOMETRIC TEST METHOD

9. Scope

9.1 This test method covers the determination of copper in concentrations greater than 50 %.

10. Summary of Test Method

10.1 After dissolution of the sample in nitric and hydrofluoric acids, the oxides of nitrogen are reduced with hydrogen peroxide, and the copper deposited electrolytically. Loss of platinum from the anode is minimized by the addition of lead. The copper oxalyldihydrazide complex is formed with the copper remaining in the electrolyte. Photometric measurement is made at approximately 540 nm.

11. Interferences

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

12. Apparatus

12.1 ~~*Apparatus No. 9*—Gauze cathodes are recommended where rapid electrolysis is used.~~

~~12.2~~ *Polytetrafluoroethylene or Polypropylene Beakers*, 250-mL capacity.

12.~~3~~2 *Polytetrafluoroethylene or Polypropylene Split Covers*.

12.3 *Electrodes for Electroanalysis* —Platinum electrodes of the stationary type are recommended as described in 12.3.1 and 12.3.2, but strict adherence to the exact size and shape of the electrodes is not mandatory. When agitation of the electrolyte is permissible in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed. The surface of the platinum electrodes should be smooth, clean and bright to promote uniform deposition and good adherence. Sandblasting is not recommended.

12.3.1 *Cathodes*—Platinum cathodes may be formed either from plain or perforated sheets or from wire gauze, and may be either open or closed cylinders. Gauze cathodes are recommended, and shall be made preferably from 50-mesh gauze woven from wire approximately 0.21 mm (0.0085 in.) in diameter. The cathode should be stiffened by doubling the gauze for about 3 mm at the top and the bottom of the cylinder or by reinforcing the gauze at the top and bottom with a platinum band or ring. The cylinder should be approximately 30 mm in diameter and 50 mm in height. The stem should be made from a platinum alloy wire such as platinum-iridium, platinum-rhodium, or platinum-ruthenium, having a diameter of approximately 1.30 mm. It should be flattened and welded the entire length of the gauze. The over-all height of the cathode should be approximately 130 mm. A cathode of these dimensions will have a surface area of 135 cm^2 exclusive of the stem.

12.3.2 *Anodes*—Platinum anodes may be of the spiral type when anodic deposits are not being determined, or if the deposits are small (as in the electrolytic determination of lead when it is present in amounts not over 0.2 %). When used in analyses where both cathodic and anodic plates are to be determined, the anodes should be of wire gauze. Spiral anodes should be made from 1.00-mm or larger platinum wire formed into a spiral of seven turns having a height of approximately 50 mm and a diameter of 12 mm, the over-all height being approximately 130 mm. A spiral anode of this description will have a surface area of 9 cm^2. Platinum gauze anodes should be made of the same material and of the same general design as platinum gauze cathodes. The anode cylinder should be approximately 12 mm in diameter and 50 mm in height and the over-all height of the anode should be approximately 130 mm. A gauze anode of these dimensions will have a surface area of 54 cm^2. Both areas are exclusive of the stem.

12.3.3 Gauze cathodes are recommended where rapid electrolysis is used.

13. Reagents

13.1 *Ammonium Chloride Solution* (0.02 g/L)—Dissolve 0.02 g of ammonium chloride (NH_4Cl) in water and dilute to 1 L.

13.2 *Hydrogen Peroxide* (3 %)—Dilute 100 mL of 30 % hydrogen peroxide to 1 L.

13.3 *Lead Nitrate Solution* (10 g/L) —Dissolve 10.0 g of lead nitrate ($Pb(NO_3)_2$) in water and dilute to 1 L.

14. Procedure

14.1 Transfer a 2.000-g sample, weighed to the nearest 0.1 mg, to a 250-mL poly(tetrafluoroethylene) or polypropylene beaker, add 2 mL of HF, and 30 mL of HNO_3 (1 + 1). Cover with a cover glass and allow to stand for a few minutes until the reaction has nearly ceased. Warm but do not heat over 80°C. When dissolution is complete, add 25 mL of 3 % H_2O_2 and 3 mL of $Pb(NO_3)_2$ solution. Rinse the cover glass and dilute to approximately 150 mL with NH_4Cl solution.

14.2 With the electrolyzing current off, position the anode and the accurately weighed cathode in the solution so that the gauze is completely immersed. Cover the beaker with a split plastic cover.

14.3 Start the electrolysis and increase the voltage until the ammeter indicates a current which is equivalent to about 1.0 A/dm^2 and electrolyze overnight. Alternatively electrolyze at a current density of 4 A/dm^2 for 1.5 h. (The more rapid procedure requires the use of gauze electrodes).

14.4 Slowly withdraw the electrodes (or lower the beaker) with the current still flowing, and rinse with a stream of water from a wash bottle. Quickly remove the cathode, rinse it in water, and then dip into two successive baths of ethanol or methanol. Dry in an oven at 110°C for 3 to 5 min.

14.5 Return the voltage to zero, and turn off the switch. Reserve the electrolyte.

14.6 Allow the electrode to cool to room temperature, and weigh.

15. Calculation

15.1 Calculate the percentage of copper as follows:

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

where:

A = deposited copper, g,
B = copper in the electrolyte as calculated in 16.10, g, and
C = sample used, g.

16. Photometric Determination of the Residual Copper in the Electrolyte

16.1 *Interferences*—The elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.

16.2 *Concentration Range*—The recommended concentration range is from 0.0025 to 0.07 mg of copper per 50 mL of solution, using a 2-cm cell.

NOTE 1—This procedure 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.

16.3 *Stability of Color*—The color fully develops in 20 min, and is stable for 1 h.

16.4 *Reagents*:

16.4.1 *Acetaldehyde Solution* (40 %)—Dilute 400 mL of acetaldehyde to 1 L with water.

16.4.2 *Boric Acid Solution*~~—Reagent No. 136.~~ (50 g/L) —Dissolve 50 g of boric acid (H_3BO_3) in hot water, cool, and dilute to 1 L.

16.4.3 *Citric Acid Solution* (200 g/L) —Dissolve 200 g of citric acid in water and dilute to 1 L.

16.4.4 *Copper, Standard Solution A* (1 mL = 1.0 mg Cu)—Transfer a 1.000-g sample of electrolytic copper (purity: 99.9 % minimum) to a 250-mL beaker and add 10 mL of HNO_3 (1 + 1). Evaporate till nearly to dryness. Add 5 mL of water to dissolve the residue. Transfer to a 1-L volumetric flask, dilute to volume, and mix.

16.4.5 *Copper, Standard Solution B* (1 mL = 0.010 mg Cu)—Using a pipet, transfer 10 mL of copper solution A (1 mL = 1.0 mg Cu) to a 1-L volumetric flask, dilute to volume and mix.

16.4.6 *Oxalyldihydrazide Solution* (2.5 g/L)—Dissolve 2.5 g of oxalyldihydrazide in warm water and dilute to 1 L.

16.5 *Preparation of Calibration Curve*:

16.5.1 *Calibration Solutions*:

16.5.1.1 Transfer 25 mL of boric acid solution to a 250-mL volumetric flask and then add a solution containing 150 mL of water, 2 mL of HF, and 30 mL of HNO_3 (1 + 1). Dilute to volume, and mix.

16.5.1.2 Transfer 10 mL of this solution to each of four 50-mL volumetric flasks. Using pipets, transfer 1, 3, 5, and 7 mL of copper solution B (1 mL = 0.010 mg Cu) to the flasks. Proceed as directed in 16.5.3.

16.5.2 *Reference Solution*—Add 10 mL of boric acid solution prepared as directed in 16.5.1.1 to a 50-mL volumetric flask and proceed as directed in 16.5.3.

16.5.3 *Color Development*—Add in order, and with mixing after each addition, 5 mL of citric acid solution, 6 mL of NH_4OH, 10 mL of acetaldehyde solution, and 10 mL of oxalyldihydrazide solution. Cool, dilute to volume, and mix. Allow to stand for 30 min and proceed as directed in 16.5.4.

16.5.4 *Photometry*:

16.5.4.1 *Multiple-Cell Photometer*—Measure the cell correction using absorption cells with a 2-cm light path and a light band centered at approximately 540 nm. Using the test cell, take the photometric readings of the calibration solutions.

16.5.4.2 *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 540 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

16.5.5 *Calibration Curve*—Plot the net photometric-readings of the calibration solutions against milligrams of copper per 50 mL of solution.

16.6 *Test Solution*—Transfer the reserved electrolyte to a 250-mL volumetric flask containing 25 mL of boric acid solution, dilute to volume, and mix. Using a pipet, transfer 10 mL to a 50-mL volumetric flask (Note 2). Proceed as directed in 16.8.

NOTE 2—If the solution shows a permanganate color, add sodium nitrite solution (20 g/L) dropwise until the color is discharged, and then proceed as directed in 16.8.

16.7 *Reference Solution*—Proceed as directed in 16.5.2.

16.8 *Color Development*—Proceed as directed in 16.5.3.

16.9 *Photometry*—Take the photometric reading of the test solution as directed in 16.5.4.

16.10 *Calculation*—Convert the net photometric reading of the test solution to milligrams of copper by means of the calibration curve. Calculate the grams of copper in the total electrolyte as follows:

$$\text{Copper, g} = (A \times 25)/1000 \quad (2)$$

where:

A = copper found in 50 mL of the final test solution, mg.

17. Precision and Bias

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

TABLE 1 Statistical Information

Test Specimen	Copper Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
~~1. Bronze ounce metal (NBS 124d, 83.60 Cu)~~	~~83.56~~	~~0.09~~	~~0.13~~
1. Bronze ounce metal (NIST 124d, 83.60 Cu)	83.56	0.09	0.13
2. AAB 521	91.98	0.03	0.08
3. AAB 655	95.38	0.09	0.14
4. AAB 681	57.60	0.10	0.09
5. AAB 715	68.95	0.08	0.21

17.2 *Bias*—The accuracy of this method has been deemed satisfactory based upon the data for the standard reference material in Table 1. Users are encouraged to use this or similar reference materials to verify that the method is performing accurately in their laboratories.

IRON BY THE 1,10-PHENANTHROLINE PHOTOMETRIC TEST METHOD

18. Scope

18.1 This test method covers the determination of iron in concentrations from 0.003 to 1.25 %.

19. Summary of Test Method

19.1 The sample is dissolved in hydrochloric acid and hydrogen peroxide, and the excess oxidant removed by evaporation. The iron is extracted with methyl isobutyl ketone-benzene mixture. The iron is extracted from the organic phase into a hydroxylamine hydrochloride solution and the red-colored 1,10-phenanthroline complex is formed. Photometric measurement is made at approximately 510 nm.

20. Concentration Range

20.1 The recommended concentration range is from 0.005 to 0.125 mg of iron per 50 mL of solution, using a 2-cm cell.

NOTE 3—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.

21. Stability of Color

21.1 The color develops within 5 min and is stable for at least 4 h.

22. Interferences

22.1 Elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.

23. Reagents

23.1 *Hydroxylamine Hydrochloride Solution* (10 g/L)~~—Prepare a solution as directed for Reagent No. 131, but dilute to~~—Dissolve 5.0 g of hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$ in 500 mL of water. Prepare fresh as needed.

23.2 *Iron, Standard Solution A* (1 mL = 0.125 mg Fe)~~—Prepare a solution as directed for Reagent No. 4, but use 0.1250~~—Transfer 0.125 g ~~instead~~ of iron (purity: 99.9 % min) to a 100 mL beaker. Add 10 mL of HCl (1 + 1) and 1 mL of bromine water. Boil gently until the ~~specified weight.~~ excess bromine is removed. Add 20 mL of HCl, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

23.3 *Iron, Standard Solution B* (1 mL = 0.00625 mg Fe)—Using a pipet, transfer 50 mL of iron solution A to a 1-L volumetric flask, dilute to volume with HCl (1 + 49), and mix.

23.4 *Methyl Isobutyl Ketone-Benzene Mixture*—Mix 200 mL of methyl isobutyl ketone (MIBK) and 100 mL of benzene.

23.5 *1,10-Phenanthroline-Ammonium Acetate Buffer Solution* —Dissolve 1.0 g of 1,10-phenanthroline monohydrate in 5 mL of HCl in a 600-mL beaker. Add 215 mL of acetic acid, and, while cooling, carefully add 265 mL of NH_4OH. Cool to room temperature. Using a pH meter, check the pH; if it is not between 6.0 and 6.5, adjust it to that range by adding acetic acid or NH_4OH as required. Dilute to 500 mL.

24. Preparation of Calibration Curve

24.1 *Calibration Solutions*:

24.1.1 Using pipets, transfer 1, 2, 5, 10, 15, and 20 mL of iron solution B (1 mL = 0.00625 mg Fe) to 50-mL volumetric flasks. Dilute to 20 mL.

24.1.2 Add 20 mL of $NH_2OH{\cdot}HCl$ solution, mix, and allow to stand 1 min. Proceed as directed in 24.3.

24.2 *Reference Solution*—Transfer 20 mL of water to a 50-mL volumetric flask and proceed as directed in 24.1.2.

24.3 *Color Development*—Add 5 mL of 1,10-phenanthroline-ammonium acetate buffer solution, dilute to volume, and mix. Allow to stand at least 5 min but not more than 4 h.

24.4 *Photometry*:

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

24.4.2 *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 510 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

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

25. Procedure

25.1 *Test Solution*:

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

Iron, %	Sample Weight, g	Tolerance in Sample Weight, mg	Aliquot Volume, mL
0.003 to 0.02	2.0	2.0	25
0.02 to 0.10	1.0	1.0	10
0.05 to 0.20	0.5	0.5	10
0.10 to 0.40	0.5	0.5	5
0.25 to 1.25	0.2	0.5	5

Transfer it to a 400-mL beaker, or to a poly(tetrafluoroethylene) beaker if HF is to be used.

25.1.2 Carry a reagent blank through the entire procedure, using the same amounts of all reagents but with the sample omitted.

25.1.3 Add 12 mL of HCl (7 + 3) per gram of sample, and then H_2O_2 as needed to completely dissolve the alloy. Add HF as needed to decompose high-silicon alloys. When dissolution is complete, add 10 mL of concentrated HCl per gram of sample and heat carefully to decompose excess peroxide. Cool to room temperature, transfer to a 100-mL volumetric flask, dilute to volume with HCl (1 + 1), and mix.

25.1.4 Using a pipet, transfer an aliquot in accordance with 25.1.1 to a 125-mL conical separatory funnel. Add HCl (1 + 1), as required, to adjust the volume to 25 mL.

25.1.5 Add 20 mL of MIBK-benzene mixture to the separatory funnel and shake 1 min. Allow the phases to separate, discard the aqueous phase, wash the organic phase 3 times with 3 to 5-mL portions of HCl (1 + 1) to remove copper, and discard the washings. Extract the iron from the organic phase by shaking vigorously 30 s with 10 mL of $NH_2OH{\cdot}HCl$ solution. Transfer the aqueous phase to a 50-mL volumetric flask. Repeat the extraction with a second 10-mL portion of $NH_2OH{\cdot}HCl$ solution, and transfer the extract to the 50-mL flask.

25.2 *Reference Solution*—Use the reagent blank solution prepared as directed in 25.1.2.

25.3 *Color Development*—Proceed as directed in 24.3.

25.4 *Photometry*—Proceed as directed in 24.4.

26. Calculation

26.1 Convert the net photometric reading of the test solution to milligrams of iron by means of the calibration curve. Calculate the percentage of iron as follows:

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

where:
A = iron found in 50 mL of the final test solution, mg, and
B = sample represented in 50 mL of the final test solution, g.

27. Precision and Bias

27.1 *Precision*—Seven laboratories cooperated in testing this method, submitting nine pairs of values, and obtained the data summarized in Table 2.

27.2 *Bias*—The accuracy of this method has been deemed satisfactory based upon the data for the standard reference materials in Table 2. Users are encouraged to use these or similar reference materials to verify that the method is performing accurately in their laboratories.

TABLE 2 Statistical Information

Test Specimen	Iron Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
~~1. Cast bronze (NBS 52c, 0.004 Fe)~~	~~0.0034~~	~~0.0005~~	~~0.0010~~
1. Cast bronze (NIST 52c, 0.004 Fe)	0.0034	0.0005	0.0010
~~2. Ounce metal (NBS 124d, 0.18 Fe)~~	~~0.187~~	~~0.012~~	~~0.017~~
2. Ounce metal (NIST 124d, 0.18 Fe)	0.187	0.012	0.017
3. Cupro Nickel, 30 Ni	0.60	0.015	0.044
~~4. Silicon bronze (NBS 158a, 1.23 Fe)~~	~~1.24~~	~~0.019~~	~~0.037~~
4. Silicon bronze (NIST 158a, 1.23 Fe)	1.24	0.019	0.037

LEAD BY THE (ETHYLENEDINITRILO)TETRAACETIC ACID (EDTA) TITRIMETRIC TEST METHOD

28. Scope

28.1 This test method covers the determination of lead in concentrations from 2.0 to 30.0 %.

29. Summary of Test Method

29.1 Lead diethyldithiocarbamate is extracted with chloroform from an alkaline tartrate-cyanide solution. After the removal of organic material, lead is titrated with disodium (ethylenedinitrilo) tetraacetic acid (EDTA) solution.

30. Interferences

30.1 Elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.

31. Apparatus

31.1 *Separatory Funnels*, 250-mL capacity.
31.2 *Magnetic Stirrer and Poly(tetrafluoroethylene)-Covered Magnetic Stirring Bar*.

32. Reagents

32.1 *Ascorbic Acid*.
32.2 *Chloroform* ($CHCl_3$).
32.3 *Disodium (Ethylenedinitrilo) tetraacetic Acid (EDTA), Standard Solution* (0.025 *M*~~)—Prepare a solution as directed for Reagent No. 22, using~~)—Dissolve 9.3 g ~~instead~~ of disodium (ethyl~~e spn~~edinitrilo) tetraacetate dihydrate in water, transfer to a 1-L volmetric flask, dilute to volume, and mix. The solution is stable for several months when stored in plastic or borosilicate g~~h~~lass bottles. Standardize as follows: Using a pipet, transfer 25 mL of lead solution (1 mL = 6.0 mg Pb) to a 250-mL beaker and dilute to 100 mL. Proceed as directed in 33.7. Calculate the lead equivalent of the solution as follows:

$$\text{Lead equivalent, g/mL} = A/B \tag{4}$$

where:
A = weight of lead, g, and
B = EDTA solution required for titration of the lead solution, mL.
32.4 *Fluoboric Acid* (37 to 40 %).

32.5 *Hexamethylenetetramine*.

32.6 *Lead, Standard Solution* (1 mL = 6.0 mg Pb)—Transfer 1.500 g of lead (purity 99.9 % minimum) to a 150-mL beaker. Add 10 mL of HNO_3 (1 + 1) and heat until dissolution is complete. Boil to remove oxides of nitrogen, cool, transfer to a 250-mL volumetric flask, dilute to volume, and mix.

32.7 *Sodium Cyanide Solution* (200 g/L)—Dissolve 200 g of sodium cyanide (NaCN) in water and dilute to 1 L. Store in a plastic bottle.

NOTE 4—**Caution:** The preparation, storage, and use of NaCN solutions require care and attention. Avoid inhalation of fumes and exposure of skin to the chemical and its solutions. Work in a well-ventilated hood. Refer to Section 6 of Practices E 50.

32.8 *Sodium Diethyldithiocarbamate Solution* (100 g/L)—Dissolve 10 g of sodium diethyldithiocarbamate in water and dilute to 100 mL. Do not use a solution that has stood more than 24 h.

32.9 *Sodium Hydroxide Solution* (250 g/L)—Dissolve 250 g of sodium hydroxide (NaOH) in water and dilute to 1 L. Store in a plastic bottle.

32.10 *Sodium Tartrate Solution* (250 g/L)—Dissolve 250 g of sodium tartrate dihydrate in water and dilute to 1 L.

32.11 *Xylenol Orange Indicator Solution* (1 g/L)—Dissolve 0.050 g of xylenol orange powder in a mixture of 25 mL of water and 25 mL of ethanol.

33. Procedure

33.1 Select a sample in accordance with the following:

Lead, %	Sample Weight, g
2.0 to 20.0	1.00
20.0 to 30.0	0.60

Weigh the sample to the nearest 0.5 mg, and transfer it to a 250-mL beaker.

33.2 Add 5 mL of HBF_4 and then 10 mL of HNO_3 (1 + 1). Cover the beaker and heat until dissolution is complete. Boil until oxides of nitrogen have been expelled, and cool.

33.3 Wash the cover and walls of the beaker. Add 25 mL of sodium tartrate solution, 25 mL of NaOH solution, and 25 mL of NaCN solution (**Caution,** Note 4), mixing after each addition. Cool to room temperature.

33.4 Transfer to a 250-mL separatory funnel. Add 15 mL of sodium diethyldithiocarbamate solution and 15 mL of $CHCl_3$, and shake for 30 s. Allow the layers to separate; draw off the lower organic layer into a 250-mL beaker, retaining the aqueous layer. Add 5 mL more of diethyldithiocarbamate solution to the separatory funnel and mix. If no precipitate forms, proceed as directed in 33.5. If a precipitate does form, add 5 mL of diethyldithiocarbamate solution and 10 mL of $CHCl_3$, shake for 30 s, and draw off the organic layer into the 250-mL beaker containing the extract.

33.5 Extract twice with additional 10-mL portions of $CHCl_3$, adding the extracts to the extracts in the 250-mL beaker.

33.6 Add 10 mL of HCl (1 + 1) to the combined extracts, and place on a hot plate. Cover the beaker with a raised cover glass, and evaporate the solution to a volume of 2 to 3 mL. Wash the cover and walls of the beaker, dilute to 100 mL, and heat to dissolve salts.

33.7 Place the beaker on a magnetic stirrer and stir (Note 5). Add 10 to 20 mg of ascorbic acid and 3 or 4 drops of xylenol orange solution. Add enough hexamethylenetetramine to color the solution purple. Add 4 or 5 drops of NaCN solution (**Caution,** Note 4) and titrate with the EDTA solution. When a yellow color begins to appear, stop the titration and add 2 to 3 g of hexamethylenetetramine and a drop of xylenol orange solution. Titrate dropwise until the color changes from purplish-red to yellow.

NOTE 5—The titration may be performed in either a hot or cold solution.

34. Calculation

34.1 Calculate the percentage of lead as follows:

$$\text{Lead, \%} = [(C \times D)/E] \times 100 \quad (5)$$

where:

C = standard EDTA solution used, mL,
D = equivalent of EDTA solution, g/mL, and
E = sample used, g.

35. Precision and Bias

35.1 *Precision*—Due to limited data, a precision statement conforming to the requirements of Practices E 173 cannot be furnished. However, in a cooperative program conducted by six laboratories, the between-laboratory range was 3.13 to 3.20 % lead on a sample averaging 3.16 %, and 14.05 to 14.23 % on a sample averaging 14.15 %.

35.2 *Bias*—No information on the accuracy of this method is known, because at the time it was tested, no standard reference

materials were available. Users are encouraged to employ suitable reference materials, if available, to verify the accuracy of the method in their laboratories.

NICKEL BY THE DIMETHYLGLYOXIME-EXTRACTION PHOTOMETRIC TEST METHOD

36. Scope

36.1 This test method covers the determination of nickel in concentrations from 0.03 to 5.0 %.

37. Summary of Test Method

37.1 A dimethylglyoxime complex of nickel is formed in the presence of copper, and extracted with chloroform. Photometric measurement is made at approximately 405 nm.

38. Concentration Range

38.1 The recommended concentration range is 0.015 to 0.3 mg of nickel per 20 mL of solution, using a 2-cm cell.

NOTE 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.

39. Stability of Color

39.1 The color is stable for at least 2 h.

40. Interferences

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

41. Reagents

41.1 *Chloroform* ($CHCl_3$).

41.2 *Complexing Solution*—Mix 240 mL of sodium tartrate solution, 90 mL of NaOH solution, 480 mL of sodium acetate solution, and 200 mL of $Na_2S_2O_3$ solution.

41.3 *Dimethylglyoxime Solution* (10 g/L in alcohol)—~~R~~Dissolve~~a~~ 10 g of dimethylglyoxime in ehtanol, methanol, or No. ~~104.~~ 30 specially denatured alcohol and dilute to 1 L with alcohol. Filter before using. This solution keeps almost indefinitely.

41.4 *Hydroxylamine Hydrochloride Solution* (10 g/L)—Dissolve 10 g of hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) in water, and dilute to 1 L. Adjust the pH to 7.0 with NH_4OH.

41.5 *Nickel, Standard Solution A* (1 mL = 1.0 mg Ni)—Dissolve 1.000 g of nickel metal (purity, 99.8 % min) in 10 mL of HNO_3. When dissolution is complete, boil gently to expel oxides of nitrogen, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

41.6 *Nickel, Standard Solution B* (1 mL = 0.2 mg Ni)—Using a pipet, transfer 100 mL of nickel solution A (1 mL = 1.0 mg Ni) to a 500-mL volumetric flask, dilute to volume, and mix.

41.7 *Sodium Acetate Solution* (200 g/L)~~—Reagent No. 127, but use~~—Dissolve 200 g ~~instead~~ of ~~the specified weight.~~ sodium acetate trihydrate ($CH_3COONa{\cdot}3H_2O$) in about 600 mL of water, filter, and dilute to 1 L.

41.8 *Sodium Hydroxide Solution* (1 N)—Dissolve 40 g of sodium hydroxide (NaOH) in water, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a plastic bottle.

41.9 *Sodium Sulfate*, anhydrous (Na_2SO_4).

41.10 *Sodium Tartrate Solution* (100 g/L)—Dissolve 100 g of sodium tartrate dihydrate in water, and dilute to 1 L.

41.11 *Sodium Thiosulfate Solution* (200 g/L)—Dissolve 200 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3{\cdot}\ 5H_2O$) in water, and dilute to 1 L.

42. Preparation of Calibration Curve

42.1 *Calibration Solutions*:

42.1.1 Transfer 1.000 g of copper (purity, 99.99 % min) to each of five 250-mL beakers, add 20 mL of HCl (1 + 1), and add 10 mL of H_2O_2 solution in small portions. When dissolution is complete, boil for 1 min to destroy excess peroxide, and cool.

42.1.2 Using pipets, transfer 2, 5, 10, 20, and 30 mL of nickel solution B (1 mL = 0.2 mg Ni) to the beakers. Transfer the solutions to 500-mL volumetric flasks, dilute to volume, and mix.

42.1.3 Using a pipet, transfer 25 mL to a 250-mL conical separatory funnel. Add 5 mL of $NH_2OH{\cdot}HCl$ solution and 50 mL of complexing solution, shaking after each addition. Using indicator paper, check the pH, which should be between 6.5 and 7.2. If necessary, adjust the pH with HCl (1 + 1) or dilute NaOH solution.

42.2 *Reference Solution*—Transfer 1.000 g of copper (purity, 99.99 % min) to a 250-mL beaker and proceed as directed in 41.1, omitting the addition of nickel solution.

42.3 *Color Development*:

42.3.1 Add 3 mL of dimethylglyoxime solution, and shake for 1 min. Using a pipet, transfer 20 mL of $CHCl_3$ to the solution, and shake again for 40 s. Allow the phases to separate.

42.3.2 Transfer the yellow-colored chloroform phase to a 25-mL Erlenmeyer flask fitted with a ground-glass stopper and containing about 1 g of Na_2SO_4. Shake to stir the Na_2SO_4 into the $CHCl_3$. Decant the clear $CHCl_3$ solution into an absorption cell, and cover immediately to prevent loss of solvent.

42.4 *Photometry*:

42.4.1 *Multiple-Cell Photometer*—Measure the cell correc
tion using absorption cells with a 2-cm light path and a light band centered at approximately 405 nm. Using the test cell, take the photometric readings of the calibration solutions.

42.4.2 *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 405 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

42.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against milligrams of nickel per 20 mL of solution.

43. Procedure

43.1 *Test Solution*:

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

Nickel, %	Sample Weight, g	Tolerance in Sample Weight, mg	Weight of Copper, g	Aliquot Volume, mL
0.03 to 0.6	1.0	1.0	...	25
0.55 to 1.5	0.4	0.5	0.6	25
1.45 to 3.5	0.4	0.5	0.6	10
3.45 ± 5.0	0.25	0.2	0.75	10

Transfer it to a 250-mL beaker. Add to the beaker the weight of copper (purity, 99.99 % min) indicated in the table.

43.1.2 Add 20 mL of HCl (1 + 1), and add 10 mL of H_2O_2 solution in small portions. Cool until the violent reaction has ceased. When dissolution is complete, boil for approximately 1 min to destroy excess peroxide. Cool, transfer to a 500-mL volumetric flask, dilute to volume, and mix.

43.1.3 Proceed as directed in 42.1.3, using an aliquot volume in accordance with 43.1.1. If a 10-mL aliquot is used, add 3 mL of HCl (1 + 9) to the aliquot in the separatory funnel.

43.2 *Reference Solution*—Proceed as directed in 42.2.

43.3 *Color Development*—Proceed as directed in 42.3.

43.4 *Photometry*—Proceed as directed in 42.4.

44. Calculation

44.1 Convert the net photometric readings of the test solution to milligrams of nickel by means of the calibration curve. Calculate the percentage of nickel as follows:

$$\text{Nickel, \%} = A/(B \times 10) \tag{6}$$

where:

A = nickel found in 20 mL of the final test solution, mg, and
B = sample represented in 20 mL of the final test solution, g.

45. Precision and Bias

45.1 *Precision*—Eight laboratories cooperated in testing this test method and obtained the data summarized in Table 3.

45.2 *Bias*—The accuracy of this method has been deemed satisfactory based upon the data for the standard reference materials in Table 3. Users are encouraged to use these or similar reference materials to verify that the method is performing accurately in their laboratories.

ZINC BY THE ETHYLENEDIAMINE TETRAACETATE (TITRIMETRIC) TEST METHOD

46. Scope

46.1 This test method covers the determination of zinc in the range from 2 to 40 %.

47. Summary of Test Method

47.1 The zinc is converted to the zinc thiocyanate complex and extracted with methyl isobutyl ketone. The zinc is then stripped from the organic phase as the ammonia complex, which is further treated with potassium cyanide to complex bivalent metals as well as the zinc. Finally, the zinc is released from the cyanide complex by means of formaldehyde and titrated with disodium (ethylenedinitrilo) tetraacetic acid (EDTA) solution.

TABLE 3 Statistical Information

Test Specimen	Nickel Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
1. 816-12	0.107	0.010	0.028
~~2. Sheet Brass (NBS 37c, 0.53 Ni)~~	~~0.531~~	~~0.010~~	~~0.036~~
2. Sheet Brass (NIST 37c, 0.53 Ni)	0.531	0.010	0.036
~~3. Ounce Metal (NBS 124d, 0.99 Ni)~~	~~0.997~~	~~0.021~~	~~0.037~~
3. Ounce Metal (NIST 124d, 0.99 Ni)	0.997	0.021	0.037
4. 844-J	4.90	0.071	0.33

48. Interferences

48.1 None of the elements ordinarily present interfere. The extraction procedure also affords a separation of the zinc from cadmium.

49. Apparatus

49.1 *Electrodes for Electroanalysis*—~~Apparatus No. 9.~~ Platinum anode and cathode described in 12.3.

49.2 *Separatory Funnels*, conical, 500-mL capacity.

49.3 *Magnetic Stirrer*, with poly(tetrafluoroethylene)-covered magnetic stirring bar.

50. Reagents

50.1 *Ammonium Chloride Solution* (0.02 g/L)—Dissolve 0.20 g of ammonium chloride (NH_4Cl) in water and dilute to 10 L.

50.2 *Ammonium Fluoride Solution* (200 g/L)—Dissolve 200 g of ammonium fluoride (NH_4F) in water and dilute to 1 L. Store in a polyethylene bottle.

50.3 *Ammonium Thiocyanate Solution* (500 g/L)—Dissolve 500 g of ammonium thiocyanate (NH_4SCN) in water and dilute to 1 L. Filter, if necessary, and store in a polyethylene bottle.

50.4 *Ascorbic Acid*, powdered.

50.5 *Buffer Solution* (*pH 10*)—Dissolve 54 g of ammonium chloride (NH_4Cl) in 200 mL of water. Add 350 mL of NH_4OH and dilute to 1 L. Store in a polyethylene bottle.

50.6 *Disodium* —(*Ethylenedinitrilo*) *Tetraacetic Acid* (*EDTA*), *Standard Solution* (0.05 *M*)*:*

~~50.6.1 Prepare a solution as directed for Reagent No. 22, using~~

50.6.1 Dissolve 18.6125 g ~~instead~~ of disodium (ethyle~~sp~~nedinitrilo) tetraacetate dihydrate in water, transfer to a 1-L volumetric flask, dilute to volume, and mix. The solution is stable for several months when stored in plastic or borosilicate g~~h~~lass bottles.

50.6.2 *Standardization*—Dissolve 0.1 g of zinc in 10 mL of HNO_3 (1 + 1) in a 400-mL beaker. Dilute the solution to 150 mL and proceed as directed in 51.4-51.7.

$$\text{Zinc equivalent, mg/mL} = (A \times 1000)/(B - C) \tag{7}$$

where:

A = grams of zinc,
B = final buret reading, mL, and
C = initial buret reading, mL.

50.7 *Eriochrome Black-T Indicator Solution*—Dissolve 0.4 g of the sodium salt to 1-(1-hydroxy-2 naphtholazo)-5 nitro-2 naphthol-4 sulfonic acid in a mixture of 20 mL of ethanol and 30 mL of triethanolamine. Store in a tightly closed polyethylene dropping bottle. Do not use a solution that has stood for more than 3 months.

50.8 *Formaldehyde Solution* (37 %).

50.9 *Hydrogen Peroxide Solution* (3 %)—Dilute 100 mL of 30 % H_2O_2 to 1 L.

50.10 *Indicator Ion Solution* (0.05 *M* $MgCl_2$ Solution)—Dissolve 1.02 g of magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) in water and dilute to 100 mL.

50.11 *Lead Nitrate Solution* (10 g/L) —Dissolve 10 g of lead nitrate ($Pb(NO_3)_2$) in water and dilute to 1 L.

50.12 *Methyl Isobutyl Ketone*.

50.13 *Potassium Cyanide Solution* (100 g/L)—Dissolve 100 g of potassium cyanide (KCN) in water and dilute to 1 L. Store in a polyethylene bottle.

NOTE 7—**Caution:** The preparation, storage, and use of KCN solutions require care and attention. Avoid inhalation of fumes and exposure of the skin to the chemical and its solutions. Do not allow solutions containing cyanide to come in contact with strongly acidic solutions: Work in a well-ventilated hood. (Refer to Section 6 of Practices E 50.

50.14 *Sodium Hydroxide Solution* (200 g/L)—Dissolve 200 g of sodium hydroxide (NaOH) in water, cool, and dilute to 1 L. Store the solution in a polyethylene bottle.

50.15 *Thiocyanate Wash Solution*—Dissolve 100 g of sodium chloride (NaCl) in 600 mL of water. Add 10 mL of the NH_4SCN solution, and mix. Add 10 mL of HCl and dilute to 1 L.

50.16 *Zinc Metal* (purity: 99.9 % min)—Do not use finely divided powder, or surface oxidized material.

51. Procedure

51.1 Transfer a 2.00-g sample, weighed to the nearest 1 mg, to a 250-mL polytetrafluoroethylene or polypropylene beaker and add 2 mL of HF followed by 30 mL of HNO_3 (1 + 1). Cover the beaker with a plastic cover and allow the sample to dissolve. Do not place the beaker on a hot plate unless the temperature is less than 80°C. When dissolution is complete, add 25 mL of H_2O_2 solution and 3 mL of $Pb(NO_3)_2$ solution. Rinse the plastic cover glass and dilute to approximately 150 mL with NH_4Cl solution.

51.2 Insert the electrodes into the solution and cover the beaker with a pair of split cover glasses. Electrolyze for 2 h at a current density of 4 A/dm^2 using gauze electrodes. When deposition is complete, slowly withdraw the electrodes (or lower the beaker) with the current still flowing and rinse them with a stream of water from a wash bottle. Reserve the electrolyte.

51.3 Depending on the amount of zinc present, transfer the whole electrolyte or an aliquot portion, containing not more than 100 mg of zinc, to a 400-mL beaker. If an aliquot of the sample is to be taken, add 25 mL of saturated boric acid (H_3BO_3) solution to the volumetric flask, add the electrolyte, dilute to volume, and mix. Dilute the aliquot to 150 mL and proceed as directed in 51.4. If the entire electrolyte is to be used, proceed directly with the neutralization.

51.4 Neutralize with NaOH solution using litmus paper as an indicator; then add 10 mL of HCl (1 + 1), and cool.

51.5 Transfer to a 500-mL separatory funnel and dilute to about 250 mL. Add 30 mL of NH_4SCN solution, and 20 mL of NH_4F solution, and mix. Add 50 mL of methyl isobutyl ketone and shake vigorously for 1 min. Allow the layers to separate; then draw off the lower aqueous layer into a second separatory funnel. Retain the organic layer. Add an additional 50 mL of methyl isobutyl ketone to the second funnel and shake for 1 min. Allow the layers to separate. Draw off and discard the aqueous layer. Add the organic layer to that retained in the first separatory funnel. To the combined extracts, add 40 mL of thiocyanate wash solution, shake, and allow the layers to separate. Draw off and discard the aqueous layer.

51.6 To the organic layer add 20 mL buffer solution, and 30 mL of water, and shake to strip the zinc from the organic phase. Allow the layers to separate, and drain off the lower ammoniacal layer into a 600-mL beaker. Repeat the extraction of zinc with another 20 mL of buffer solution and 30 mL of water, followed by a final wash with 50 mL of water, combining all the aqueous extracts in the 600-mL beaker. Discard the organic layer.

51.7 Dilute to about 300 mL. Place a poly (tetrafluoroethylene)-covered stirring bar into the beaker, add 20 mL of KCN solution, and then add 10 to 20 mg of ascorbic acid powder. Add 1.0 mL of indicator ion solution and about 5 drops of eriochrome black-T indicator. Transfer the beaker to the magnetic stirring apparatus and titrate with EDTA solution to a pure blue end point. Record the initial buret reading. Cautiously add formaldehyde solution, 1 to 2 mL at a time, until the color has changed again to wine red. Titrate with EDTA solution to a pure blue end point. Make further additions of formaldehyde and each time titrate to the blue end point to ensure that all the zinc has been released. Avoid adding excessive amounts of formaldehyde. Record the final buret reading.

52. Calculation

52.1 Calculate the percentage of zinc as follows:

$$\text{Zinc, \%} = (A - B)C/(D \times 10) \tag{8}$$

where:

A = final buret reading, mL,
B = initial buret reading, mL,
C = zinc equivalent of standard EDTA solution, mg/mL, and
D = grams of sample represented in portion of electrolyte taken.

53. Precision and Bias

53.1 *Precision*—Eight laboratories cooperated in testing this method and obtained the data shown in Table 4.

53.2 *Bias*—The accuracy of this method has been deemed satisfactory based upon the data for the standard reference materials in Table 4. Users are encouraged to use these or similar reference materials to verify that the method is performing accurately in their laboratories.

NICKEL BY THE DIMETHYLGLYOXIME GRAVIMETRIC TEST METHOD

54. Scope

54.1 This test method covers the determination of nickel in concentrations from 4 to 50 %.

55. Summary of Test Method

55.1 After dissolution of the sample, the nickel is precipitated from an alkaline citrate solution with sodium dimethylglyoximate; this precipitate is subsequently weighed as nickel dimethylglyoxime.

TABLE 4 Statistical Information

Test Specimen	Zinc Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
~~1. Ounce Metal (NBS 124d, 5.06 Zn)~~	~~5.08~~	~~0.02~~	~~0.18~~
1. Ounce Metal (NIST 124d, 5.06 Zn)	5.08	0.02	0.18
~~2. Sheet Brass (NBS 37c, 27.85 Zn)~~	~~27.87~~	~~0.13~~	~~0.27~~
2. Sheet Brass (NIST 37c, 27.85 Zn)	27.87	0.13	0.27
3. AAB Alloy 681	40.84	0.23	0.40

56. Interferences

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

57. Apparatus

57.1 *Electrodes for Electroanalysis*—~~Apparatus No. 9.~~ Platinum anode and cathode described in 12.3.

57.2 *Filtering Crucibles*~~—Apparatus No. 2, Gooch~~—Gooch crucible (35- mL) fitted with a glass microfibre pad, or fritted glass crucible (30- mL) of medium porosity.

58. Reagents

58.1 *Citric Acid* (250 g/L)~~—Prepare a solution as directed for Reagent No. 129, but use~~—Dissolve 250 g ~~instead~~ of ~~the specified weight.~~ citric acid in water and dilute to 1 L. The addition of 1 g of benzoic acid per litre will prevent bacterial growth.

58.2 *Sodium Dimethylglyoximate Solution* (25 g/L)—Dissolve 25 g of sodium dimethylglyoximate [$(CH_3)_2C_2$-$(NONa)_2 \cdot 8H_2O$], in water and dilute to 1 L. Do not use a solution that has stood more than 24 h.

58.3 *Sulfamic Acid Solution* (100 g/L)—Dissolve 100 g of sulfamic acid [$H(NH_2)SO_3$]3] in water and dilute to 1 L.

59. Procedures

59.1 Transfer a sample, weighed to the nearest 0.1 mg, which contains between 40 and 150 mg of nickel, to a 250-mL beaker. Dissolve the sample in 25 mL of HNO_3 (1 + 1) and when dissolution is complete, boil gently to expel oxides of nitrogen. Add 50 mL of hot water and, if the solution is clear, proceed as described in 59.4. If enough tin is present at this point to cause turbidity, proceed as directed to 59.2 and 59.3.

59.2 Maintain the temperature of the solution at about 80°C for 1 h, or until the precipitate has coagulated. Add paper pulp and filter through a fine paper into a 250-mL beaker to remove the metastannic acid. Wash several times with hot HNO_3 (1 + 99), and reserve the filtrate and washings.

59.3 Transfer the filter paper and precipitate to the original beaker, add 15 to 20 mL of HNO_3 and 10 to 15 mL of $HClO_4$. Heat to copious white fumes and boil to destroy organic matter. Cool, wash the cover glass and sides of the beaker, and add 15 mL of HBr. Heat to copious white fumes to volatilize the tin. If the solution is not clear, repeat the treatment with HBr. Evaporate the solution to near dryness, cool, and dissolve the residue in a few millilitres of water. Combine with the filtrate reserved in 59.2.

59.4 Add 1 drop of HCl (1 + 99) and 5 mL of sulfamic acid solution. Insert the electrodes into the solution, cover with a pair of split cover glasses, and electrolyze overnight at a current density of 0.5 A/dm^2, or for a short period at a current density of 4 A/dm^2 while stirring. After the blue color due to copper has disappeared, wash the cover glasses, electrodes, and the sides of the beaker, and continue the electrolysis until deposition of the copper is complete, as indicated by failure to plate on a new surface when the level of the solution is raised.

NOTE 8—Rotating electrodes must be used at the higher current densities. The more rapid procedure requires the use of gauze electrodes.

59.5 When deposition of the copper is complete, with the current still flowing, lower the beaker slowly while washing the electrodes with water. Reserve the electrolyte.

59.6 Add 5 mL of HNO_3 and 10 mL of $HClO_4$ to the reserved electrolyte and evaporate to copious white fumes. Cool, add 100 mL of water, and heat to dissolve the soluble salts. If insoluble matter is present, filter the solution through a medium paper into a 600-mL beaker. If there is no insoluble matter, transfer the solution to a 600-mL beaker.

59.7 Add 10 mL of citric acid solution. Add NH_4OH until the blue color is formed, and then add 1 mL in excess. Dilute to 400 mL and heat to 60 to 70°C.

59.8 Add 0.4 mL of sodium dimethylglyoximate solution for each milligram of nickel, plus 10 mL in excess. Stir the mixture vigorously and allow to cool to room temperature while stirring occasionally. Filter on a Gooch or fritted glass crucible of medium porosity which has been dried at 150°C for 1 h and weighed. Wash with water 10 to 12 times. Add 5 mL of sodium dimethylglyoximate solution to the filtrate and let stand overnight to make certain that the separation of the nickel is complete.

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

60. Calculation

60.1 Calculate the percentage of nickel as follows:

$$\text{Nickel, \%} = [(A \times 0.2032)/B] \times 100 \quad (9)$$

where:
A = nickel dimethylglyoxime, g, and
B = sample used, g.

61. Precision and Bias

61.1 *Precision*—Eight laboratories cooperated in testing this test method, submitting eight pairs of values, and obtained the data summarized in Table 5. Although samples with the nickel concentration near the upper limit of the scope were not available for testing, the precision data obtained for the other specimens should apply.

61.2 *Bias*—No information on the accuracy of this method is known, because at the time it was tested, no standard reference materials were available. Users are encouraged to employ suitable reference materials, if available, to verify the accuracy of the method in their laboratories.

TABLE 5 Statistical Information

Test Specimen	Nickel Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
1. Cupro-nickel	29.74	0.12	0.14
2. Nickel-aluminum bronze	5.00	0.05	0.04

TIN BY THE IODATIMETRIC TITRATION TEST METHOD

62. Scope

62.1 This test method covers the determination of tin in concentrations from 0.5 to 20 %.

63. Summary of Test Method

63.1 After dissolution of the sample in hydrochloric and nitric acids, iron is added as a collector and tin is separated from copper by double precipitation with ammonium hydroxide. The precipitate is dissolved in hydrochloric acid and the tin is reduced with iron and nickel and titrated with a standard potassium iodate solution in an inert atmosphere. Starch is used to indicate the end point.

64. Interferences

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

65. Apparatus

65.1 *Apparatus for Reduction of Tin*—~~A~~ When tin is to be reduced to the stannous state and determined by titration with standard iodine or iodate solution, air must be excluded during the reduction and titration to prevent oxidation of the stannous tin. This exclusion of air is usually accomplished by keeping the solution under a blanket of gaseous CO_2 and may be accomplished in a variety of ways. One of the simplest methods is by means of the apparatus shown in Fig. 1 in which the reduction of the tin solution is made in a flask capped with a rubber stopper containing an L-shape siphon tube. When reduction is complete, the end of the siphon is dipped into a saturated solution of $NaHCO_3$ and set aside to cool. ~~7A~~ When cool, the stopper is removed and the solution titrated.

66. Reagents

66.1 *Ammonium Chloride Solution* (10 g/L)—Dissolve 10 g of ammonium chloride (NH_4Cl) in water and dilute to 1 L.

66.2 *Ferric Chloride Solution* (50 g/L)—Dissolve 50 g of ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) in 5 mL of HCl and 995 mL of water.

66.3 *Iron*, metal powder, containing less than 0.001 % tin.

66.4 *Nickel*, sheet, containing less than 0.001 % tin and having a total (exposed) surface area of at least 65 cm^2.

66.5 *Potassium Iodate, Standard Solution* (0.05 *N*~~)—Prepare a solution as directed for Reagent No. 12, but use~~)—Dry the crystals of potassium iodate (KIO_3) at 180ºC to constant weight. Dissolve 1.785 g ~~instead~~ of the ~~specified weight.~~ KIO_3 in 200 mL of water containing 1 g of sodium hydroxide (NaOH) and 10 g of potassium iodide (KI). When dissolution is complete, transfer to a 1-L volumetric flask, dilute to volume, and mix. Standardize the solution as follows:

[redacted]

FIG. 1

66.5.1 Using a pipet, transfer 50 mL of the tin solution (1 mL = 0.001 g Sn) to a 500-mL Erlenmeyer flask. Add 75 mL of HCl, 200 mL of water, and 2 to 3 g of iron powder. Insert a roll of sheet nickel. Stopper the flask as described ~~under Apparatus No. 7A.~~ in 65.1. Boil the solution gently for 45 min.

66.5.2 Cool to about 10°C while maintaining an atmosphere of CO_2 as described under ~~Apparatus No. 7A.~~ the apparatus for the reduction of tin (Fig. 1).

66.5.3 Add 5 mL of starch solution and titrate with KIO_3 solution until a blue color persists.

66.5.4 Determine a blank using the same amounts of all reagents but with the tin omitted. Calculate the tin equivalent of the KIO_3 solution as follows:

$$\text{Tin equivalent, g Sn/mL} = A/(B - C) \tag{10}$$

where:
A = tin titrated, g,
B = KIO_3 solution required to titrate tin, mL, and
C = KIO_3 solution required to titrate the blank, mL.

66.6 *Potassium Iodide Solution* (100 g/L) ~~—Reagent No. 142.~~ —Dissolve 10 g of potassium iodide (KI) in water, and dilute to 100 mL. Prepare fresh as needed.

66.7 *Starch Solution* (10 g/L) ~~—Reagent No. 110.~~ —Add about 5 mL of water gradually to 1 g of soluble (or arrowroot) starch, with stirring, until a paste is formed, and add this to 100 mL of boiling water. Cool, add 5 g of potassium iodide (KI), and stir until the KI is dissolved. Prepare fresh as needed.

66.8 *Tin, Standard Solution* (1 mL = 0.001 g Sn) ~~—R~~Transfer 1.0000 g of tin (purity, 99.9 % min) to a 400-mL beaker, and cover. Add 300 mL of HCl (1 + 1) and warm gent~~N~~ly until the metal is dissolved. If dissolution is difficult, add 0.5 to 1~~9~~.0 g of potassium chlorate ($KClO_3$). Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

67. Procedure

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

Tin, %	Sample Weight, g	Tolerance in Sample Weight, mg
0.5 to 2.5	2	4
2.5 to 10.0	1	2
10.0 to 20.0	0.5	1

Transfer it to a 400-mL beaker.

67.2 Carry a reagent blank through the entire procedure using the same amount of all reagents, but with the sample omitted.

67.3 Add 5 mL of HCl and 20 mL of HNO_3 (1 + 1), plus an additional 5 mL of HCl and 5 mL of HNO_3 (1 + 1) for each gram of sample. Heat until dissolution is complete and then boil the solution for 2 to 3 min. Add 100 mL of water and 10 mL of $FeCl_3$ solution.

67.4 Add NH_4OH (1 + 1) until the salts, which initially form, have been dissolved and the solution becomes clear dark blue. Heat to boiling to coagulate the precipitate. Filter on a 12.5-cm coarse paper and wash the beaker and paper alternately five times each with hot slightly ammoniacal NH_4Cl solution and water. Discard the filtrate. Place the original beaker beneath the funnel and dissolve the precipitate with hot HCl (1 + 1). Wash the paper several times with hot water and reserve the filter paper. Precipitate the iron and tin as before and heat to boiling to coagulate the precipitate. Wash the reserved filter paper three times with hot NH_4OH (1 + 99), collecting the washings in a 400-mL beaker, and then filter the hot solution containing the precipitated hydroxides of iron and tin into the 400-mL beaker containing the NH_4OH washings. Wash alternately five times each with hot, slightly ammoniacal NH_4Cl solution and water. Discard the filtrate. Transfer the paper and precipitate to a 500-mL Erlenmeyer flask.

67.5 Add 75 mL of HCl and gently swirl the flask to partially disintegrate the paper and to dissolve the precipitate. Add 200 mL of water and 2 to 3 g of iron powder. Insert a roll of sheet nickel. Stopper the flask as described under Apparatus No. 7A. Boil the solution gently for 45 min.

67.6 Cool the solution to about 10°C while maintaining an atmosphere of CO_2 as described under Apparatus No. 7A. Add 5 mL of KI solution and 5 mL of starch solution and titrate with KIO_3 solution until a blue color persists.

68. Calculation

68.1 Calculate the percentage of tin as follows:

$$\text{Tin, \%} = \frac{(A - B) \times C}{D} \times 100 \tag{11}$$

where:
A = KIO_3 solution required to titrate the tin in the sample, mL,
B = KIO_3 solution required to titrate the blank, mL,

C = the tin equivalent of the KIO_3 solution, and
D = sample used, g.

69. Precision and Bias

69.1 *Precision*—Eight laboratories cooperated in testing this test method and obtained the data summarized in Table 6. Although samples with tin concentration near the upper limit of the scope were not available for testing, the precision data obtained for the other specimens should apply.

69.2 *Bias*—No information on the accuracy of this method is known, because at the time it was tested, no standard reference materials were available. Users are encouraged to employ suitable reference materials, if available, to verify the accuracy of the method in their laboratories.

TABLE 6 Statistical Information

Test Specimen	Tin Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
1. Tin bronze AAB521	7.51	0.14	0.23
2. Yellow brass AAB681	0.93	0.04	0.04
3. Yellow brass AAB681 + 20 % Pb	0.93	0.03	0.05

ALUMINUM BY THE CARBAMATE EXTRACTION-(ETHYLENEDINITRILO) TETRAACETATE TITRIMETRIC TEST METHOD

70. Scope

70.1 This test method covers the determination of aluminum in concentrations from 2 to 12 %.

71. Summary of Test Method

71.1 A diethyldithiocarbamate extraction at pH 5.5 removes antimony, cadmium, copper, iron, lead, manganese, nickel, tin, and zinc. Aluminum is chelated with an excess of a standard solution of disodium (ethylenedinitrilo) tetraacetate (EDTA) and then determined by back-titration with standard zinc solution.

72. Interferences

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

73. Apparatus

73.1 *Separatory Funnels*, 250-mL capacity with poly(tetrafluoroethylene) stopcocks.

74. Reagents

74.1 *Buffer Solution*—Dissolve 250 g of ammonium acetate (CH_3COONH_4) in 600 mL of water and add 30 mL of glacial acetic acid (CH_3COOH). Dilute to 1 L and mix. Add 10 mL of buffer solution to 100 mL of water. Using a pH meter, check the pH of the solution which should be between 5.3 and 5.6. If it is not in the range, add sufficient CH_3COOH or NH_4OH to provide the desired pH.

74.2 *Diethyldithiocarbamate (DDC) Solution* (100 g/L)—Dissolve 100 g of diethyldithiocarbamic acid, disodium salt, in water, dilute to 1 L, and mix. Do not use a solution that has stood more than 24 h.

74.3 *(Disodium) (Ethylenedinitrilo) Tetraacetate Dihydrate (EDTA), Standard Solution* (0.05 *M*)~~—Prepare a solution as directed for Reagent No. 22, but use~~—Dissolve 18.613 g of disodium (ethylenedinitrilo) Tetra~~g~~ace~~n~~tate dihydrate in water, transfer to a 1-L volumetric flask, dilute to volume, and mix. The solution is stable for several months when s~~p~~tored in plastic or borosi~~f~~licate~~d wei~~ g~~h~~lass bottles. Standardize the solution as follows:

74.3.1 Using a pipet, transfer 25 mL of the EDTA solution to a 400-mL beaker. Add 150 mL of water and 30 mL of the buffer solution. Add 6 to 8 drops of xylenol orange indicator solution and titrate with standard zinc solution until the color changes from yellow to orange or pink.

74.3.2 Calculate the volume of EDTA standard solution equivalent to 1 mL of zinc standard solution as follows:

$$EDTA \text{ equivalent} = (A/B) \quad (12)$$

where:
A = EDTA solution, mL, and
B = zinc solution (0.0500 *M*), mL.

74.3.3 Calculate the molarity of the EDTA solution as follows:

$$\text{Molarity, } EDTA \text{ solution} = C \times 0.002 \quad (13)$$

where:
C = millilitres of zinc standard solution required to titrate 25.00 mL of EDTA standard solution.

74.4 *Sodium Tartrate Solution* (250 g/L)—Dissolve 250 g of sodium tartrate ($Na_2C_4H_4O_6 \cdot 2H_2O$) in water. Dilute to 1 L, and mix.

74.5 *Xylenol Orange Indicator Solution* (2 g/L)—Dissolve 0.100 g of xylenol orange tetrasodium salt in 50 mL of water. Store in and dispense from a polyethylene dropping bottle.

74.6 *Zinc, Standard Solution* (0.0500 *M*)— Transfer 3.2690 g of zinc (purity: 99.9 % min) to a 1 L borosilicate volumetric flask. Add 50 mL of water and 20 mL of HCl and heat to dissolve. Cool, dilute to volume, and mix.

75. Procedure

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

Aluminum, %	Sample Weight, g	Tolerance in Sample Weight, mg
2.0 to 3.0	1.0	3.0
2.9 to 4.3	0.7	2.0
4.0 to 6.0	0.5	1.0
5.0 to 7.5	0.4	0.5
7.0 to 10.0	0.3	0.5
8.0 to 12.0	0.25	0.5

75.2 Transfer the sample to a 250-mL beaker, and add 5 mL of HCl (1 + 1), plus an additional 1 mL for each 0.1 g of sample over 0.25 g. Add H_2O_2 in 1-mL portions until the sample has been completely dissolved. Cover the beaker with a ribbed cover glass.

75.3 Boil gently and evaporate the excess acid until the color turns from a clear green to a brown-green. Cool. Remove and rinse the cover glass.

75.4 Add 50 mL of water and 5 mL of sodium tartrate solution. With swirling, add NH_4OH dropwise until the color changes from a clear green to a turquoise-green color (pH approximately 5.5). Add 30 mL of the buffer solution, and mix.

75.5 Transfer the solution to a 500-mL separatory funnel and dilute to approximately 125 to 150 mL. Add 8 mL of the DDC solution for each 0.1 g of sample used. Add 50 mL of $CHCl_3$. Shake the separatory funnel vigorously for 30 s and allow the phases to separate. Draw off the organic phase and discard, being careful to avoid any losses of the aqueous solution in this operation and the subsequent phase separations.

75.6 Add 5 mL of the DDC solution and 25 mL of $CHCl_3$ to the separatory funnel. Shake the separatory funnel vigorously for 30 s and allow the phases to separate. Add an additional 2 to 3 mL of the DDC solution to ensure that an excess of DDC has been added. (If a precipitate appears, shake again, add 5 mL of the DDC solution, shake vigorously for 30 s and allow the phases to separate. Repeat this extraction until no further precipitation occurs. Draw off and discard the organic phase.

75.7 Add 25 mL of the $CHCl_3$, and shake the separatory funnel for 15 s. Allow the phases to separate. Draw off and discard the organic phase. Repeat this step.

75.8 Transfer the aqueous layer quantitatively to a 500-mL Erlenmeyer flask. Rinse the separatory funnel with distilled water and transfer the rinsings to the flask.

75.9 Using a pipet, add 25 mL of EDTA solution, and mix. Boil gently for 3 to 5 min to completely decompose any residual DDC and to chelate the aluminum. Cool to below 20°C.

75.10 Add 6 to 8 drops of xylenol orange indicator solution, and mix.

75.10.1 If the solution is red, add CH_3COOH dropwise until the color just turns from red to yellow. Proceed as directed in 74.5.11.

75.10.2 If the solution is yellow, add NH_4OH dropwise just to the transition color from yellow to red. Then add acetic acid dropwise until the color just turns from red to yellow.

75.11 Titrate the excess EDTA with the standard zinc solution (0.0500 *M*) to the first color change from yellow to orange or pink.

76. Calculation

76.1 Calculate the percentage of aluminum as follows:

$$\text{Aluminum, \%} = \frac{[25.00 - (A \times B)] \times C \times 2.698}{D} \quad (14)$$

where:
A = zinc solution required to titrate the excess EDTA in 74.11, mL,
B = EDTA equivalent to 1.00 millilitre of zinc standard solution, mL, 73.3.2,

C = molarity of EDTA solution, and
D = sample used, g.

77. Precision and Bias

77.1 *Precision*—Eight laboratories cooperated in testing this test method and obtained the data summarized in Table 7.

77.2 *Bias*—No information on the accuracy of this method is known, because at the time it was tested, no standard reference materials were available. Users are encouraged to employ suitable reference materials, if available, to verify the accuracy of the method in their laboratories.

ZINC BY ATOMIC SPECTROMETRY TEST METHOD

78. Scope

78.1 This test method covers the determination of zinc in concentrations from 0.02 to 2 %.

79. Summary of Test Method

79.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption by the sample solution of the zinc resonance line energy of 2138 Å is measured and compared with the absorption of calibration solutions containing known amounts of zinc.

80. Concentration Range

80.1 The concentration range of zinc must be determined experimentally because the optimum range will depend on the characteristics of the instrument used. Determine the appropriate concentration range as directed in 80.1.1-80.1.5.

80.1.1 Prepare a dilute standard solution as directed in 83.3.

80.1.2 Prepare the instrument for use as directed in 86.1. Measure the instrument response while aspirating a reference solution, the lowest, and the two highest calibration solutions. Apply the sensitivity test and curve linearity test as directed in 82.1.1 and 82.1.2, respectively.

80.1.3 If the criteria of sensitivity and of curve linearity are met, the initial concentration range may be considered acceptable. Proceed as directed in 80.1.5.

80.1.4 If the minimum response is not obtained, prepare a dilute standard solution to provide a higher concentration range and repeat 80.1.1 and 80.1.2. If the linearity criterion is not met, prepare a dilute standard solution to provide a concentration range lower than that of the original standard solution and repeat 80.1.1 and 80.1.2. If a concentration range cannot be found for which both criteria are met, the instrument's performance must be improved before this method is used.

80.1.5 Perform the stability test as directed in 82.1.3. If the minimum requirements are not met with the selected calibration solutions, do not use this method until the desired stability is obtained.

81. Interferences

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

82. Apparatus

82.1 *Atomic Absorption Spectrophotometer*—Determine that the atomic absorption spectrophotometer is satisfactory for use in this test method by proceeding as directed in 82.1.1-82.1.3.

NOTE 9—Optimum settings for the operating parameters of the atomic absorption spectrophotometer vary with the instrument used; use the 2138 Å zinc line, a band pass of approximately 5 Å, and a lean air-acetylene flame.

82.1.1 *Sensitivity*—The difference between the readings of the two highest of eight equally spaced calibration solutions must be sufficient to permit an estimation equivalent to one fifth of the difference between the concentrations of the two solutions.

82.1.2 *Curve Linearity*—The difference between the readings of the two highest of eight equally spaced calibration solutions must be more than 0.7 times the difference between the reference solution and the lowest of the calibration solutions. Absorbance values are to be used in this calculation.

TABLE 7 Statistical Information

Test Specimen	Aluminum Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
High tensile brass (BCS 179/1, 2.54 Al)	2.52	0.05	0.08
Manganese bronze, high tensile	5.29	0.04	0.08
Nickel-aluminum bronze	11.58	0.05	0.18

82.1.3 *Minimum Stability*—Obtain the readings of the reference solution and the highest calibration solution. Repeat at least twice with no change in parameters. The variability of the readings of the highest calibration solution and of the reference solution must be less than 3.0 % and 1.5 %, respectively, as calculated as follows:

$$V_C = \frac{100}{\bar{C}} \times \sqrt{\frac{\Sigma(C - \bar{C})^2}{n - 1}} \tag{15}$$

$$V_O = \frac{100}{\bar{C}} \times \sqrt{\frac{\Sigma(O - \bar{O})^2}{n - 1}} \tag{16}$$

where:

V_C	=	percent variability of the highest calibration readings,
$\bar{C}$	=	average absorbance value for the highest calibration solution,
$\Sigma (C - \bar{C})^2$	=	sum of the squares of the *n* differences between the absorbance readings of the highest calibration solution and their average,
$\bar{O}$	=	average absorbance value of the reference solution,
V_O	=	percent variability of the readings on the reference solution relative to $\bar{C}$,
$\Sigma (O - \bar{O})^2$	=	sum of the squares of the *n* difference between the absorbance readings of the reference solution and their average, and
n	=	number of determinations.

83. Reagents

83.1 *Dissolving Solution*—Add 250 mL of HNO_3 to 500 mL of water, mix, add 250 mL of HCl, and mix. Store in a plastic bottle.

NOTE 10—All plastic bottles used in the method must be well rinsed with the dissolving solution before use.

83.2 *Zinc, Standard Solution A* (1 mL = 1.00 mg Zn)—Dissolve 1.00 g of zinc metal (purity: 99.95 % min) in a covered 600-mL beaker with 50 mL of dissolving solution. Boil gently to remove gases, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a plastic bottle.

83.3 *Zinc, Standard Solution B* (1 mL = 0.004 mg Zn)—Using a pipet, transfer 4 mL of Zinc Solution A to a 1-L volumetric flask, add 10 mL of dissolving solution, dilute to volume, and mix. Store in a plastic bottle. Do not use a solution that has stood more than 24 h.

84. Calibration

84.1 *Calibration Solutions*—Using a 50-mL buret, transfer 4, 8, 12, 16, 20, 24, 28, and 32 mL of Zinc Solution B to 100-mL volumetric flasks. Add 2 mL of dissolving solution to each flask, dilute to volume, and mix.

84.2 *Reference Solution*—Add 2 mL of dissolving solution to a 100-mL volumetric flask, dilute to volume, and mix.

84.3 Determine the suitability of the selected concentration range and apparatus as directed in Section 82.

85. Procedure

85.1 *Test Solution*:

85.1.1 Transfer a 1.00-g sample to a 400-mL beaker, cover, and add 20 mL of dissolving solution. Allow the initial reaction to subside. Heat gently to remove gases and to complete the dissolution. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a plastic bottle.

85.1.2 Select the appropriate aliquot in accordance with the following, and, using a pipet, transfer it to a 100-mL volumetric flask, add 2 mL of dissolving solution, dilute to volume, and mix.

Zinc Concentration, %	Aliquot
0.02 to 0.1	use as prepared
0.1 to 0.25	50 mL
0.25 to 1.0	10 mL
1.0 to 2.0	5 mL

86. Measurements

86.1 *Instrument Adjustments*:

86.1.1 Set the parameters to the values suggested in 82.1 and light the burner.

86.1.2 Aspirate the highest calibration solution and optimize all adjustments to obtain maximum absorption.

86.1.3 Aspirate the reference solution to ensure stability and set the initial reading above, but near, zero.

86.2 Aspirate the test solution to determine its place in the order of increasing concentration of the calibration solutions.

86.3 Aspirate the reference solution and adjust to the base reading. Aspirate the reference solution, the test solution, and calibration solutions, in the order of increasing readings.

86.4 Repeat 86.3 at least twice.

87. Calculation

87.1 Calculate the variability of the readings for the reference solution and the highest calibration solution as directed in 82.1.3 to determine whether they are less than 1.5 and 3.0 %, respectively. If they are not, disregard the data, readjust the instrument, and proceed again as directed in 86.2.

87.2 If necessary, convert the average of the readings for each calibration solution to absorbance.

87.3 Prepare a calibration curve by plotting the average absorbance values for the calibration solutions against milligrams of zinc/100 mL.

87.4 Convert the absorbance value of the test solution to milligrams of zinc per 100 mL by means of the calibration curve.

87.5 Calculate the percentage of zinc as follows:

$$\text{Zinc, \%} = \frac{A}{B} \times 100 \tag{17}$$

where:

A = zinc/100 mL of the final test solution, mg, and
B = sample represented in 100 mL of the test solution taken for analysis, mg.

88. Precision and Bias

88.1 *Precision*—Eight laboratories cooperated in the testing of this test method and obtained the data summarized in Table ~~8. 88.2 No information on the bias~~ 8. Supporting data are available from ASTM Headquarters. Request RR:E03-1012.

88.2 *Bias*—The accuracy of this ~~test~~ method ~~is available. The bias of~~ has been deemed satisfactory based upon the ~~test method may be judged, however, by comparing accepted~~ data for the standard reference ~~values, if available, with~~ materials in Table 8. Users are encouraged to use these or similar reference materials to verify that the ~~corresponding arithmetic average obtained by interlaboratory testing.~~ method is performing accurately in their laboratories.

TABLE 8 Statistical Information

Test Specimen	Zinc Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
1. 70-30 cupro-nickel alloy	0.965	0.0306	0.0391
2. Aluminum-bronze alloy (78 Cu-9 Al-5 Fe-5 Ni)	0.034	0.0009	0.003
~~3. NBS 52c, 2.12 % zinc~~	~~2.10~~	~~0.025~~	~~0.078~~
3. NIST 52c, 2.12 % zinc	2.10	0.025	0.078
~~4. NBS 158a, 2.08 % zinc~~	~~2.09~~	~~0.039~~	~~0.108~~
4. NIST 158a, 2.08 % zinc	2.09	0.039	0.108
5. 70-30 cupro-nickel alloy	0.129	0.007	0.017

LEAD BY THE ATOMIC ABSORPTION TEST METHOD

89. Scope

89.1 This test method covers the determination of lead in concentrations from 0.002 to 15 %.

90. Summary of Test Method

90.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption by the sample solution of the lead resonance line energy at 2833 Å is measured and compared with the absorption of calibration solutions containing known amounts of lead.

91. Concentration Range

91.1 If the optimum concentration range is not known; determine it as directed in Guide E 1024, here in after referred to as the operational guide. A sensitivity of 0.5 µg/mL of 0.0044 absorbance is widely obtained.

92. Interferences

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

93. Apparatus

93.1 *Atomic Absorption Spectrophotometer*—Determine that the instrument is suitable for use as prescribed in the operational guide. The percent of variability for the highest calibration solution (V_c) should be less than 1.0 %.

93.1.1 *Operating Parameters*:

Wavelength	2833 Å (Note 11)
Bandpass	about 5 Å
Gas mixture	air-acetylene
Flame type	lean

NOTE 11—For very low concentrations of lead, the resonance line energy at 2170Å may be used provided the criteria set forth in 93.1 are met.

94. Reagents

94.1 *Fluoboric Acid* (37 to 40 %).

94.2 *Lead, Standard Solution A* (1 mL = 1.00 mg Pb)—Dissolve 1.000 g of lead metal (purity: 99.9 % min) in a covered 150-mL beaker with 15 mL of HNO_3 (1 + 2). Transfer to a 1-L volumetric flask, add 100 mL of HNO_3 (1 + 2), dilute to volume, and mix. Store in a plastic bottle.

94.3 *Lead, Standard Solution B* (1 mL = 0.200 mg Pb)—Using a pipet, transfer 50 mL of Standard Lead Solution A to a 250-mL volumetric flask. Dilute to volume and mix.

95. Calibration

95.1 Select a convenient value, Q µg/mL, less than the maximum obtained in 91.1. Using pipets, transfer into individual 100-mL volumetric flasks 0.1Q, 0.2Q, 0.3Q, 0.4Q, and 0.5Q mL of Standard Lead Solution B. Add 5 mL of HNO_3 (1 + 2) to each flask, dilute to volume, and mix.

95.2 *Reference Solution*—Add 5 mL of HNO_3 (1 + 2) to a 100-mL volumetric flask, dilute to volume, and mix.

95.3 Determine the suitability of the selected concentration range and apparatus as directed in the operational guide.

96. Procedure

96.1 *Test Solution*:

96.1.1 Transfer a 1-g sample, weighed to the nearest 1 mg, to a 150-mL beaker, add 5 mL of HBF_4 and 15 mL of HNO_3 (1 + 2), and cover. Allow the initial reaction to subside. Heat gently to complete the dissolution and remove gases. Cool, transfer to a 100-mL volumetric flask, dilute to volume, and mix.

96.1.2 Select an appropriate aliquot (nominal values) in accordance with the following, and, using a pipet, transfer it to a 100-mL volumetric flask, add 5 mL HNO_3 (1 + 2), dilute to volume, and mix.

Lead Concentration, %	Aliquot
0.002 to 0.60	use as prepared
0.50 to 3.00	20 mL
2.0 to 12.0	5 mL

97. Measurements

97.1 Optimize the response of the instrument (adjustments) and take preliminary readings; complete the analysis and calculate the concentration of lead in the test solution as in the graphical, ratio, or single point procedures, as described in the operational guide. For low levels of lead, expanded scale readout is advisable.

98. Calculation

98.1 Calculate the percentage of lead as follows:

$$\text{Lead, \%} = \frac{A}{B} \times 100 \tag{18}$$

where:

A = lead/100 mL of the final test solution, mg, and

B = sample represented in 100 mL of the test solution taken for analysis, mg.

99. Precision and Bias

99.1 *Precision*—Due to limited data, a precision statement conforming to the requirements of Practices E 173 cannot be furnished. However, in a cooperative program conducted by six laboratories, the results reported in Table 9 were obtained. Supporting data are available from ASTM Headquarters. Request RR: E03-1013.

99.2 *Bias*—No information on the ~~bias~~ accruacy of this ~~test~~ method is ~~available. The bias of~~ known, because at the ~~test method may be judged, however, by comparing accepted~~ time it was tested, no standard reference materials were available. Users are encouraged to employ suitable reference materials, if ~~available, with~~ available to verify the ~~corresponding arithmetic average obtained by interlaboratory testing.~~ accuracy of the method in their laboratories.

SILVER IN SILVER-BEARING COPPER BY THE ATOMIC ABSORPTION TEST METHOD

100. Scope

100.1 This test method covers the determination of silver in concentrations from 0.01 to 0.12 %.

TABLE 9 Statistical Information

Material	Average of Lead, %	Lowest Value of Lead Obtained, %	% Pb, Highest Value of Lead Obtained, %
Copper (C102)	0.0037	0.0028	0.0052
Bronze (C544)	4.22	4.14	4.35
Bronze (C922)	2.00	1.93	2.06
Bronze (C939)	14.43	14.27	14.67

101. Summary of Test Method

101.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption by the sample solution of the silver resonance line energy at 328.1 nm is measured and compared with the absorption by calibration solutions containing known amounts of silver.

102. Concentration Range

102.1 If the optimum concentration range is not known determine it as directed in the Operational Guide.

103. Interferences

103.1 Elements normally present in silver-bearing copper do not interfere. Contamination of the calibration or test solutions by halides may lead to the loss of silver. (Note that copper that has been soldered may contain a flux residue with a chloride constituent).

104. Apparatus

104.1 *Atomic Absorption Spectrophotometer*—Determine that the instrument is suitable for use as prescribed in the Operational Guide. The percent variability for the highest calibration solution should be less than 1.0.

104.1.1 *Operating Parameters*:

Wavelength	328.1 nm
Gas mixture	air – acetylene
Flame type	lean

105. Reagents

105.1 *Mercury Solution* (3 g/L)—Dissolve 3 g of mercury in 10 mL of HNO_3 (1 + 1). Warm gently to remove fumes, cool, add 25 mL HNO_3, and dilute to 1 L. Store in a plastic bottle.

105.2 *Silver, Standard Solution A* (1 mL = 0.2 mg Ag)—Dissolve 0.3150 g of silver nitrate ($AgNO_3$) (purity, 99.7 % min) in water. Transfer to a 1-L volumetric flask, add 100 mL of mercury solution (3 g/L) and 25 mL of HNO_3. Dilute to volume and mix. Store in a tightly sealed plastic bottle in a dark place. The solution is stable for one year.

105.3 *Silver, Standard Solution B* (1 mL = 0.02 mg Ag)—Using a pipet, transfer 25 mL of silver Solution A to a 250-mL volumetric flask. Dilute to volume and mix. Prepare fresh prior to use.

106. ~~Safety Precautions~~ Hazards

106.1 *Caution*—Mercury is a health hazard. Handling and disposal should be done in a safe manner.

107. Calibration

107.1 *Calibration Solutions*—Select a convenient value, *Q* µg/mL, less than the range maximum obtained in 101.1. Using pipets, transfer *Q, 2Q, 3Q, 4Q,* and 5*Q* mL of silver Solution B to 100-mL volumetric flasks. Add 10 mL of mercury solution (3 g/L) and 5 mL of HNO_3(1 + 1) to each flask, dilute to volume, and mix. Store away from the light.

107.2 *Reference Solution*—Add 10 mL of mercury solution (3 g/L) and 5 mL HNO_3 (1 + 1) to a 100-mL volumetric flask. Dilute to the mark and mix.

107.3 Determine the suitability of the selected concentration range and apparatus as directed in the Operational Guide.

108. Procedure

108.1 *Test Solution*—Accurately weigh a sample (0.5 g or less) to contain up to 100 *Q* µg Ag. Transfer to a 150-mL beaker, add 8 mL of HNO_3 (1 + 1), and allow to dissolve. Warm gently to remove gasses, cool, and transfer to a 100-mL volumetric flask. Add 10 mL of mercury solution (3 g/L), dilute to volume, and mix.

109. Measurements

109.1 Optimize the response of the instrument and take preliminary readings; complete the analysis and calculate the concentration of silver in the test solution as described in the Operational Guide.

110. Calculation

110.1 Calculate the percentage of silver as follows:

$$\text{Silver, \%} = \frac{A}{B} \times 100 \tag{19}$$

where:
A = silver per 100 mL of the final test solution, mg, and
B = sample represented in 100 mL of the test solution taken for analysis, mg.

110.2 If required, convert to troy ounces per short ton:

$$\text{Silver, \%} \times 291.7 = \text{silver, oz/ton} \tag{20}$$

111. Precision and Bias

111.1 *Precision*—Seven laboratories cooperated in testing this test method and obtained 9 sets of data summarized in Table 10. Supporting data are available from ASTM Headquarters. Request RR: E03-1032.

111.2 *Bias*—No information on the ~~bias~~ accuracy of this ~~test~~ method is known, because at the time it was tested, no standard reference materials were available. Users are encouraged to employ suitable reference materials, if available, to verify the accuracy of the method in their laboratories.

TABLE 10 Statistical Information

Test Specimen (Alloy Number)	Silver Found, %	Repeatability (R_1, E 173)	Reproducibility (R_2, E 173)
1. C11600	0.0959	0.0039	0.0077
2. C11400	0.0383	0.0020	0.0038

TIN BY THE PHENYLFLUORONE PHOTOMETRIC TEST METHOD

112. Scope

112.1 This test method covers the determination of tin in concentrations from 0.01 to 1.0 %.

113. Summary of Method

113.1 The sample is dissolved in fluoboric and nitric acids. Phenylfluorone in a perchloric acid-sodium citrate-buffered solution reacts with the tin to form an orange-red complex. Photometric measurement is made at approximately 510 nm.

114. Concentration Range

114.1 The recommended concentration range is from 0.02 to 0.16 mg of tin in 100 mL of solution, using a 1-cm cell.

NOTE 12—This test method has been written for cells having a 1-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

115. Stability of Color

115.1 Photometric measurement is made after 75 ± 10 min.

116. Interferences

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

117. Apparatus

117.1 *pH Meter*~~—Apparatus No. 3A.~~ .

118. Reagents

118.1 *Ascorbic Acid, Powder*.

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

118.3 *Copper, High-Purity*—Use copper containing less than 0.0001 % tin.

118.4 *Fluoboric Acid (HBF_4)* (49 to 50 %).

118.5 *Gelatin Solution* (10 g/L)—While stirring, add 1 g of gelatin to 100 mL of boiling water. Do not use a solution that has stood more than 1 day.

118.6 *Perchloric Acid Solution* (9 + 31) —Slowly add 225 mL of $HClO_4$ to a 1-L volumetric flask containing 500 mL of water, cool, dilute to volume, and mix.

118.7 *Phenylfluorone Solution* (0.2 g/L)—Dissolve 0.200 g of phenylfluorone (2,6,7-trihydroxy-9-phenylisoxanthene3-one)[5] in a mixture of 600 mL of 2-ethoxy-ethanol (ethylene glycol monoethyl ether), 30 mL of water, and 30 mL of $HClO_4$, while stirring with a magnetic stirrer. Dilute to 1 L with water. Do not use a solution that has stood more than 1 week.

118.8 *Potassium Permanganate Solution* (20 g/L)~~—Reagent No. 134.~~ —Dissolve 20 g of potassium permanganate ($KmnO_4$) in water and dilute to 1 L.

118.9 *Sodium Citrate Solution* (100 g/L)—Dissolve 100 g of sodium citrate dihydrate in water and dilute to 1 L.

118.10 *Sodium Hydroxide Solution* (250 g/L)~~—Prepare a solution as directed for Reagent No. 146, but use~~ —Dissolve 250 g ~~instead~~ of ~~the specified weight.~~ sodium hydroxide (NaOH) in about 100 mL of water. When dissolution is complete, cool, and dilute to 1 L. Store in a plastic bottle.

118.11 *Tin, Standard Solution A* (1 mL = 1.0 mg Sn)—Dissolve 1.000 g of tin (purity: 99.9 % min) in a mixture of 25 mL of HNO_3, 25 mL of HCl, and 25 mL of HBF_4. When dissolution is complete, boil gently to expel oxides of nitrogen, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

118.12 *Tin, Standard Solution* (1 mL = 0.005 mg Sn)—Using a pipet, transfer 5 mL of tin Solution A (1 mL = 1.0 mg of Sn) to a 1-L volumetric flask, add 25 mL of HBF_4 (1 + 9), dilute to volume, and mix. Do not use a solution that has stood more than 1 week.

118.13 *Tin, Standard Solution C* (1 mL = 0.010 mg Sn)—Using a pipet, transfer 10 mL of tin Solution A (1 mL = 1.0 mg Sn) to a 1-L volumetric flask, add 25 mL of HBF_4 (1 + 9), dilute to volume, and mix. Do not use a solution that has stood more than 1 week.

119. Preparation of Calibration Curve

119.1 *Calibration Solutions*—Using pipets, transfer 5, 10, and 15 mL of tin Solution B (1 mL = 0.005 mg Sn), and 10 and 15 mL of tin solution C (1 mL = 0.010 mg Sn) to 150-mL beakers. Proceed as directed in 119.3.

119.2 *Reference Solution*—Transfer 15 mL of water to a 150-mL beaker. Proceed as directed in 119.3.

119.3 *Color Development*—Adjust the volume to 15 mL with water and add 15 mL of sodium citrate solution. Using a pH meter, adjust the pH to 12 ± 0.25 with NaOH solution. Using a pipet, add 10 mL of $HClO_4$ (9 + 31) and, while stirring, add $KMnO_4$ solution until a pink color persists. Add 25 mL of H_3BO_3 solution and 5 mL of gelatin solution, 25 to 50 mg of ascorbic acid, and 10 mL of phenylfluorone solution. Transfer the solution to a 100-mL volumetric flask, dilute to volume, and mix. Allow to stand 75 ± 10 min.

119.4 *Photometry*:

119.4.1 *Multiple-Cell Photometer*—Measure the cell correction using absorption cells with a 1-cm path and a light band centered at approximately 510 nm. Using the test cell, take photometric readings of the calibration solutions.

NOTE 13—Test absorption cells must be cleaned after each reading in a solution of dilute nitric acid (1 + 4) to remove the colored complex that may adhere to the windows.

119.4.2 *Single-Cell Photometer*—Transfer a suitable portion of the reference solution 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 510 nm. While maintaining this adjustment, take photometric readings of the calibration solutions (Note ~~24).~~ 13).

119.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against milligrams of tin per 100 mL of solution.

120. Procedure

120.1 *Test Solution*:

~~*Annual Book of ASTM Standards*, Vol 03.06.~~

[5] Eastman No. 6346 has been found satisfactory for this purpose.

TABLE 11 Statistical Information

Test Specimen	Tin Found, %	Repeatability (R_1 E 173)	Reproducibility (R_2 E 173)
~~1. Copper-zinc-nickel (NBS 157a)~~	~~0.023~~	~~0.002~~	~~0.004~~
1. Copper-zinc-nickel (NIST 157a 0.021 Sn)	0.023	0.002	0.004
2. Tin-copper alloy (Kennecott 1784)	0.201	0.013	0.031
3. High tensile brass (BCS 179/1)	0.528	0.016	0.085
~~4. Silicon bronze (NBS 158a)~~	~~0.962~~	~~0.024~~	~~0.120~~
4. Silicon bronze (NIST 158a 0.96 Sn)	0.962	0.024	0.120
5. Anaconda brass (AABC 681)	0.952	0.042	0.114

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

Tin, %	Sample Weight, g	Tolerance in Sample Weight, mg	Dilution, mL	Aliquot Volume, mL
0.01 to 0.035	2.00	1.0	100	15
0.03 to 0.10	1.00	1.0	100	15
0.09 to 0.16	1.00	1.0	100	10
0.15 to 0.32	0.50	0.5	100	10
0.30 to 0.64	0.25	0.25	100	10
0.60 to 1.00	0.25	0.25	100	5

Transfer it to a 100-mL borosilicate volumetric flask.

120.1.2 Add 10 mL of HBF_4 (1 + 9) and 10 mL of HNO_3 (1 + 1). When dissolution is complete, heat the solution to boiling and continue to boil until oxides of nitrogen are removed. Cool, dilute to volume, and mix.

120.1.3 Using a pipet, transfer 5 to 15 mL portions, as specified in 120.1.1, to 150-mL beakers. Proceed as directed in 120.3.

120.2 *Reference Solution*—Transfer a weight of copper taken in 120.1.1 to a 100-mL borosilicate volumetric flask. Treat an aliquot the same size as that of the test solution as directed in 120.1.2. Proceed as directed in 120.3.

120.3 *Color Development*—Proceed as directed in 119.3.

120.4 *Photometry*—Proceed as directed in 119.4.

121. Calculation

121.1 Convert the net photometric reading of the test solution to milligrams of tin by means of the calibration curve. Calculate the percentage of tin as follows:

$$\text{Tin, \%} = \frac{A}{B \times 10} \tag{21}$$

where:

A = tin found in 100 mL of the final test solution, mg, and
B = sample represented in 100 mL of the final test solution, g.

122. Precision and Bias

122.1 *Precision*—Eight laboratories cooperated in testing this test method with one laboratory reporting a second pair of values. The data are summarized in Table ~~11.~~

~~122.2 No information on the bias~~ 11.

122.2 *Bias*—The accuracy of this ~~test~~ method ~~is known. The bias of this test method may be judged by comparing accepted~~ has been deemed satisfactory based upon the data for the standard reference ~~v~~ material in Table 11. Users are encouraged to us ~~w~~e these or similar reference materials to verify that the ~~corresponding arithmetic average obtained by interlaboratory testing.~~ method is performing accurately in their laboratories.

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