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Standard Test Methods for Chemical Analysis of Silver Brazing Alloys[1]

This standard is issued under the fixed designation E 56; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the chemical analysis of silver brazing alloys having chemical compositions within the following limits:

Element	Concentration Range, %
Silver	10 to 90
Copper	15 to 70
Zinc	15 to 30
Cadmium	3 to 25

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

	Sections
Silver by the Silver Chloride (Gravimetric) Method	8 to 13
Copper by the Electrolytic Method	14 to 19
Zinc by the Ethylenediamine Tetraacetic Acid (Volumetric) Method	20 to 28
Cadmium by the Ethylenediamine Tetraacetic Acid (Volumetric) Method	29 to 35

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements see Section 5.

2. Referenced Documents

2.1 *ASTM Standards:*

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance to Specification[2]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[3]

E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition[3]

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

4. Apparatus and Reagents

4.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure. The apparatus, standard solutions, and certain other reagents used in more than one procedure are referred to by number, and shall conform to the requirements prescribed in Practices E 50.

5. Hazards

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

6. Sampling

6.1 Wrought products shall be sampled in accordance with Practice E 55.

7. Rounding Calculated Values

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

SILVER BY THE SILVER CHLORIDE (GRAVIMETRIC) TEST METHOD

8. Scope

8.1 This test method covers the determination of silver in silver brazing alloys in the range from 10 to 90 %.

9. Summary of Test Method

9.1 This test method for the determination of silver by the silver chloride method is based on the precipitation of silver from a nitrate solution with hydrochloric acid.

10. Apparatus

10.1 *Fritted-Glass Crucibles* of 30-mL capacity, medium porosity (Apparatus No. 2).

11. Procedure

11.1 Transfer 1 g of the sample, weighed to the nearest 0.1 mg, to a 150-mL beaker. Add 10 mL of HNO_3 (1+1). Heat gently to dissolve and boil to remove the brown fumes. Cool to room temperature and dilute to 50 mL.

11.2 Add 20 mL of HCl (1+9) slowly with constant stirring. Let stand for about 1 h.

11.3 Decant the solution through a weighed fritted-glass crucible. Wash the precipitate twice with warm HNO_3 (1+99) and decant through the crucible. Transfer the precipitate to the crucible and wash twice with hot water. Transfer the filtrate and washings to a 400-mL beaker and reserve for the determination of copper.

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[1] These test methods are under the jurisdiction of ASTM Committee E-3 on Chemical Analysis of Metals and are the direct responsibility of Subcommittee E03.05 on Nonferrous Metals.

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

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

11.4 Dry the crucible and precipitate at 110°C for 2 h or until constant weight is attained. Cool in a desiccator to room temperature and reweigh.

12. Calculation

12.1 Calculate the percentage of silver as follows:

$$\text{Silver, \%} = [(A \times 0.7526)/B] \times 100$$

where:
A = silver chloride, g, and
B = sample used, g.

13. Precision and Bias

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

COPPER BY THE ELECTROLYTIC TEST METHOD

14. Scope

14.1 This test method covers the determination of copper in silver brazing alloys in the range from 15 to 70 %.

15. Summary of Test Method

15.1 After removal of silver as silver chloride, the copper in the filtrate is determined by electrolysis in the presence of cadmium and zinc.

16. Apparatus

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

17. Procedure

17.1 Add 5 mL of H_2SO_4 to the reserved filtrate (11.3). Evaporate the solution to fumes and fume strongly for 1 min. Cool the solution to room temperature, dilute to 250 mL with water, and add 10 mL of HNO_3.

17.2 Weigh the cathode, adjust the electrodes in the solution, and cover the beaker with a pair of split watch glasses. Electrolyze at a current of 0.5 A/dm^2 until the solution becomes colorless. Rinse the watch glasses and the sides of the beaker with water and add sufficient water to raise the solution level 5 to 10 mm. Continue the electrolysis until deposition of copper is complete as indicated by failure of copper to plate on the newly exposed cathode surface when the solution level is raised.

17.3 When deposition of copper is complete, remove the solution quickly while rinsing the electrodes with water and without interrupting the current. Reserve the electrolyte and rinsings for the determination of cadmium and zinc.

17.4 Rinse the cathode in two successive baths of ethanol or methanol. Dry in an oven at 110 °C for 3 to 5 min, cool, and reweigh the cathode. The difference in weight is metallic copper.

18. Calculation

18.1 Calculate the percentage of copper as follows:

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

where:
A = copper, g, and
B = sample used, g.

19. Precision and Bias

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

ZINC BY THE ETHYLENEDIAMINE TETRAACETIC ACID (VOLUMETRIC) TEST METHOD

20. Scope

20.1 This test method covers the determination of zinc in silver brazing alloys in the range from 15 to 30 %.

21. Summary of Test Method

21.1 In a solution containing both zinc and cadmium, cadmium is removed by precipitation with sodium diethyldithiocarbamate. The complexed zinc salt is separated by filtration and determined by titration with disodium ethylenediamine tetraacetate.

22. Concentration Range

22.1 The recommended concentration of zinc is between 50 and 150 mg.

23. Interferences

23.1 Lead, bismuth, and thallium interfere; however, these metals are rarely present in silver brazing alloys except as minor impurities.

24. Apparatus

24.1 *Fritted-Glass Crucibles* of 30-mL capacity, medium porosity (Apparatus No. 2).

25. Reagents

25.1 *Buffer Solution*—Dissolve 54 g of ammonium chloride (NH_4Cl) in 300 mL of water, add 350 mL of NH_4OH, and dilute to 1 L. This solution has a pH of 10.

25.2 *Disodium Ethylenediamine Tetraacetate (EDTA), Standard Solution* (0.05 *M*)—Dissolve 18.6 g of the salt in 600 mL of water with heat. Cool to room temperature, add 0.1 g of magnesium chloride ($MgCl_2 \cdot 6H_2O$) and dilute to 1 L with water. Standardize the solution as follows:

25.2.1 Transfer to an 800-mL beaker an aliquot of the standard zinc solution approximately equal in zinc content to the aliquot of the sample. Continue as directed in 26.2 through 26.5. Calculate the equivalent of the EDTA solution in terms of grams of zinc per millilitre of solution.

25.3 *Eriochrome Black-T Indicator Solution*—Dissolve 0.4 g of Eriochrome Black-T (1hydroxy-2-naphthylazo-5-nitro-2-naphthol-6-sulfonic acid sodium salt) in 20 mL of ethanol, add 30 mL of triethanolamine (2-2′-2″ nitrilotriethanol), and store in a polyethylene dropping bottle.

25.4 *Formaldehyde* (1+9)—Dilute 100 mL of formaldehyde (37 %) with 900 mL of water.