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AMERICAN SOCIETY FOR TESTING AND MATERIALS  
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# Standard Test Methods for Chemical Analysis of Electronic Nickel<sup>1</sup>

This standard is issued under the fixed designation E 107; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover photometric procedures for the chemical analysis of nickel, intended primarily for use in electronic devices, having a chemical composition within the following limits:

Element	Concentration Range, %
Nickel	94 to 100
Copper	0.005 to 0.3
Iron	0.0035 to 0.3
Cobalt	0.05 to 1.0
Manganese	0.02 to 0.5
Titanium	0.0005 to 0.5
Silicon	0.001 to 0.3
Aluminum	0.01 to 0.35
Carbon	0.001 to 0.10
Hydrogen	0.0001 to 0.01
Nitrogen	0.0001 to 0.01
Oxygen	0.001 to 0.10
Tungsten	3.0 to 5.0
Magnesium	0.005 to 0.2

1.2 The techniques and procedures covered in these test methods have been chosen so as to keep the consumption of sample to a minimum.

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

	Sections
Copper by the Hydrobromic Acid (Photometric) Method	8 to 15
Iron by the Thiocyanate (Photometric) Method	16 to 24
Cobalt by the Nitroso-R-Salt (Photometric) Method	25 to 32
Manganese by the Periodate (Photometric) Method	33 to 40
Titanium by the Tiron (Photometric) Method	41 to 48
Silicon by the Molybdenum Blue (Photometric) Method	49 to 56
Aluminum by the Aluminon (Photometric) Method	57 to 64
Carbon by the Low-Pressure Combustion Method	65 to 73
Hydrogen, Nitrogen, and Oxygen by the Vacuum Fusion Method	74 to 78
Copper by the Neocuproine (Photometric) Method	79 to 87
Tungsten by the Acid Digestion-Cinchonine (Gravimetric) Method	88 to 92

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.08 on Ni and Co and High Temperature Alloys. Current edition approved Dec. 30, 1988. Published February 1989. Originally published as E 107 – 54 T. Last previous edition E 107 – 83.

These test methods were developed in cooperation with ASTM Committee F-1 on Electronics.

Magnesium by the 8-Hydroxyquinoline (Photometric) Method

93 to 102

1.4 *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.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications<sup>2</sup>
- E 39 Methods for Chemical Analysis of Nickel<sup>3</sup>
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals<sup>3</sup>
- E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition<sup>3</sup>
- E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals<sup>3</sup>

## 3. Significance and Use

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

## 4. Photometric Practice, Apparatus, and Reagents

4.1 *Photometers and Photometric Practice*—Photometers and photometric practice prescribed in these test methods shall conform to Practice E 60.

4.2 Apparatus other than photometers, 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 the use of certain reagents in these test methods, reference shall be made to Practices E 50.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.05.

## 6. Sampling

6.1 The sample shall be selected so as to be representative of the material to be analyzed.

6.2 For the determination of carbon, hydrogen, nitrogen, and oxygen, wrought products shall be sampled in accordance with Practice E 55, with the exception that for the determination of hydrogen, nitrogen, and oxygen solid pieces are preferred to millings or drillings, if representative, so as to minimize the effect of surface area.

## 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 Section 3.4 and 3.5 of Practice E 29.

### COPPER BY THE HYDROBROMIC ACID (PHOTOMETRIC) TEST METHOD

## 8. Summary of Test Method

8.1 Cupric copper in a mixture of HBr and  $\text{H}_3\text{PO}_4$  forms a red-violet colored complex. Photometric measurement is made at approximately 600 nm.

NOTE 1—By calibrating the system using a light band centered at approximately 600 nm, this test method can be used to determine higher concentrations of copper, if necessary. Under these conditions, the range is from 0.15 to 3.0 mg of copper in 25 mL of solution, using a cell depth of 2 cm.

## 9. Concentration Range

9.1 The recommended concentration range is from 0.05 to 0.8 mg of copper per 25 mL of solution, using a cell depth<sup>4</sup> of 2 cm.

## 10. Stability of Color

10.1 The color develops immediately and is stable for several days.

## 11. Interfering Elements

11.1 Gold and the platinum group metals interfere if present. Provision is made in this test method to eliminate other interfering elements that might be present in nickel.

## 12. Reagents

12.1 *Copper, Standard Solution* (1 mL = 0.1 mg Cu)—Dissolve 0.1000 g of high-purity copper (99.9 % Cu and over) in 3 mL of  $\text{HNO}_3$  by heating gently in a 125-mL conical flask. Add 10 mL of  $\text{HClO}_4$  and heat to copious fumes to expel  $\text{HNO}_3$ . Cool and add 10 mL of water. Transfer to a 1-L volumetric flask, dilute to the mark, and mix.

12.2 *Hydrobromic Acid - Bromine Mixture*—Add 1 volume of bromine to 16 volumes of HBr and mix.

12.3 *Hydrogen Peroxide* (3 %)—Dilute 1 mL of  $\text{H}_2\text{O}_2$  (30 %) to 10 mL with water. Prepare fresh before use.

12.4 *Test Lead*—Finely granulated test lead containing less than 0.0001 % of copper and less than 0.001 % of iron or nickel.

<sup>4</sup> These procedures have 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 amount of sample and reagents used.

## 13. Preparation of Calibration Curve

### 13.1 Calibration Solutions:

13.1.1 Transfer 0.5, 1.0, 2.0, 3.0, 4.0, 6.0, and 8.0 mL of copper solution (1 mL = 0.1 mg Cu) to 125-mL conical flasks. Add 3 mL of  $\text{HClO}_4$  and dilute to 40 mL.

13.1.2 Add 1 g of test lead, cover, and boil at a moderate rate for 15 min to displace all the copper. Cool somewhat, remove the solution by decantation, and wash once with water, decanting thoroughly. Heat the flask gently to remove moisture.

13.1.3 Add 10 mL of HBr –  $\text{Br}_2$  mixture to the flask, cover, and heat gently to dissolve the metal. Boil to expel the excess bromine. Cool to room temperature. Transfer 10 mL of  $\text{H}_3\text{PO}_4$  and 1 drop (0.05 mL) of HBr –  $\text{Br}_2$  mixture to a dry 25-mL volumetric flask (Note 2). Transfer the sample solution to a volumetric flask, washing with a few millilitres of HBr. Dilute to the mark with HBr and mix.

NOTE 2—Partial reduction of the copper to the cuprous state may occur when a bromine-free HBr solution of cupric copper is boiled. For this reason, it is necessary to add a small amount of bromine to oxidize any cuprous copper before photometric measurement is made. Bromine in small amounts does not absorb appreciably at 600 nm.

13.2 *Reference Solution*—Transfer 40 mL of water and 3 mL of  $\text{HClO}_4$  to a 125-mL flask and proceed as directed in 13.1.2 and 13.1.3.

13.3 *Photometry*—Transfer a suitable portion of the reference solution to an absorption cell and adjust the photometer to the initial setting, using a light band centered at approximately 600 nm. While maintaining this photometer adjustment, take the photometric readings of the calibration solutions.

13.4 *Calibration Curve*—Plot the photometric readings of the calibration solutions against milligrams of copper per 25 mL of solution.

## 14. Procedure

### 14.1 Sample Solution:

14.1.1 Transfer 1.000 g of the sample to a 125-mL conical flask and add 10 mL of  $\text{HNO}_3$ (1 + 1). Cover and warm gently to dissolve the sample.

14.1.2 When dissolution is as complete as possible, add 6 mL of  $\text{HClO}_4$  and heat while swirling over an open flame until the volume of the solution has been reduced to about 3 mL (Note 3). Cool, add 10 mL of water plus 2 drops of  $\text{H}_2\text{O}_3$ (3 %), and heat to boiling (Note 4). Dilute to 40 mL with water. If necessary, filter through a fine paper into a 150-mL flask or beaker (Note 5). Wash once or twice with water and discard the paper and precipitate.

NOTE 3—It is essential that the fuming operation completely remove the  $\text{HNO}_3$ , yet care should be used to avoid evaporation of too much  $\text{HClO}_4$ , lest an insoluble nickel oxide be formed.

NOTE 4—The  $\text{H}_2\text{O}_2$  is added to destroy any  $\text{MnO}_2$  or  $\text{HMnO}_4$  that might be present.

NOTE 5—If more than a small amount of tungsten is present, as indicated by a colored precipitate at this point, the subsequent method for the determination of iron should not be used, as the precipitate tends to hold iron. No apparent difficulties are encountered in the determination of copper, cobalt, or manganese.

14.1.3 Add 1 g of test lead to the flask or beaker. Cover and boil gently for 15 min to collect the copper on the lead. Decant

the filtrate. Quickly wash the container and test lead twice by decantation with water. Cool the decanted solution and wash water to room temperature, transfer to a 100-mL volumetric flask, dilute to the mark, and mix. Reserve this solution for the determination of cobalt, iron, and manganese (see Note 5).

14.1.4 Add 10 mL of HBr – Br<sub>2</sub> mixture to the lead remaining in the flask or beaker, cover, and heat gently to dissolve. Proceed as directed in 13.1.3.

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

14.3 *Photometry*—Take the photometric reading of the sample solution as described in 13.3.

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

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

where:

A = copper found, g, and

B = sample used, g.

## 15. Precision and Bias

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

## IRON BY THE THIOCYANATE (PHOTOMETRIC) TEST METHOD

### 16. Summary of Test Method

16.1 Ferric iron forms a red-brown soluble complex with thiocyanate in acid solution. Photometric measurement is made at approximately 470 nm.

### 17. Concentration Range

17.1 The recommended concentration range is from 0.007 to 0.14 mg of iron in 50 mL of solution, using a cell depth<sup>3</sup> of 2 cm.

### 18. Stability of Color

18.1 The color develops immediately and is reasonably stable for 30 min in the presence of H<sub>2</sub>O<sub>2</sub>.

### 19. Interfering Elements

19.1 The elements ordinarily present in electronic nickel do not interfere with this test method. This test method, however, is not applicable to alloys containing appreciable amounts of tungsten (see Note 5).

### 20. Reagents

20.1 *Ammonium Thiocyanate Solution* (115 g/L)—Dissolve 115 g of NH<sub>4</sub>CNS in 300 mL of water. Filter and dilute to 1 L. Store in a dark bottle.

20.2 *Hydrogen Peroxide Solution* (1.5 %)—Dilute 1 mL of H<sub>2</sub>O<sub>2</sub>(30 %) to 20 mL with water. Prepare fresh before use.

20.3 *Iron, Standard Solution* (1 mL = 0.025 mg Fe)—Dissolve 0.1756 g of Fe(NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O in 10 mL of HNO<sub>3</sub>(1 + 1). Heat to gentle boiling to expel brown fumes. Cool, dilute to 1 L in a volumetric flask, and mix.

20.4 *Nickel Nitrate*, Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O.

## 21. Preparation of Calibration Curve A

21.1 *Calibration Solutions*:

21.1.1 Transfer 5.0 g of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O to each of two 125-mL conical flasks. Add 3 mL of water and warm to dissolve most of the sample. Add 6 mL of HClO<sub>4</sub> and heat over an open flame until the volume of the solution has been reduced to 3 mL. Add 10 mL of water and heat to boiling. Combine the two solutions, transfer to a 200-mL volumetric flask, dilute to the mark, and mix.

21.1.2 Transfer 5.0-mL portions of the nickel solution to seven 50-mL beakers, and add 0.5, 1.0, 2.0, 3.0, 4.0, 5.0, and 6.0 mL of iron solution (1 mL = 0.025 mg Fe).

21.1.3 Add 5 mL of HNO<sub>3</sub>(1 + 3) to each beaker and boil for 1 min to expel brown fumes. Cool, transfer to 50-mL volumetric flasks, and dilute to approximately 35 mL. Add 1 mL of H<sub>2</sub>O<sub>2</sub> solution and then add 10 mL of NH<sub>4</sub>CNS solution. Dilute to the mark and mix.

21.2 *Reference Solution*—Transfer a 5-mL aliquot of the nickel solution (21.1.1) to a 50-mL beaker and proceed as directed in 21.1.3.

21.3 *Photometry*—Transfer a suitable portion of the reference solution to an absorption cell and adjust the photometer to the initial setting, using a light band centered at approximately 470 nm. While maintaining this photometer adjustment, take the photometric readings of the calibration solutions.

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

## 22. Preparation of Calibration Curve B

22.1 Repeat the preparation of a calibration curve, as directed in 21.1.2 to 21.4, except to use 20-mL portions of a nickel solution prepared as directed in 21.1.1.

NOTE 6—The presence of varying amounts of nickel affects the ferric thiocyanate color, and necessitates the preparation of two calibration curves to cover the analytical range indicated in 1.1.

## 23. Procedure

23.1 *Sample Solution*—Depending on the iron content of the sample, transfer a 5.0 or 20.0-mL aliquot portion of the solution reserved as directed in 14.1.3 to a 50-mL beaker and continue as described in 21.1.3.

23.2 *Reference Solution*—Transfer a corresponding aliquot of the reagent blank solution reserved from the copper determination to a beaker and carry through all the steps of the procedure for use as a reference solution.

23.3 *Photometry*—Take the photometric reading of the sample solution as described in 21.3.

23.4 *Background Color*—Transfer a corresponding aliquot of the sample solution to a 50-mL beaker, and continue as directed in 21.1.3 but omit the addition of NH<sub>4</sub>CNS solution.