



Standard Test Method for Gravimetric Determination of Phosphorus in Phosphorus- Copper Alloys or Phosphorus-Copper-Silver Alloys¹

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1. Scope

1.1 This test method covers the gravimetric determination of phosphorus in phosphorus-copper or phosphorus-copper-silver alloys containing 1 to 15 % phosphorus.

1.2 *This standard does not purport to address 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.*

2. Referenced Documents

2.1 ASTM Standards:

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications²

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals³

E 255 Practice for Sampling Copper and Copper Alloys for Determination of Chemical Composition³

3. Summary of Test Method

3.1 After dissolution of the sample in nitric acid, phosphorus is precipitated with ammoniacal magnesium chloride. Magnesium ammonium phosphate is separated by filtration and redissolved with dilute hydrochloric acid. Phosphorus is reprecipitated with ammoniacal magnesium chloride, then filtered, ignited, and weighed as magnesium pyrophosphate.

4. Significance and Use

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

5. Interferences

5.1 Silver is complexed as diamminesilver (I). Copper is complexed as tetraminecopper (II).

5.2 When present, tin interferes by forming insoluble stannic phosphate. Other elements which form insoluble phosphates or hydroxides in ammoniacal solution also interfere.

6. Apparatus and Reagents

6.1 *Magnesium Chloride Precipitant*—Dissolve 65 g of magnesium chloride (MgCl_2) and 140 g of ammonium chloride (NH_4Cl) in 500 mL water, add 375 mL of NH_4OH , dilute to 1 L with water, and mix.

6.2 *Potassium Chlorate Solution*—Dissolve 10 g of potassium chlorate (KClO_3) in 200 mL water.

7. Sampling

7.1 Select the sample so as to be representative of the material to be analyzed.

7.2 For procedures for sampling the material, refer to Practice E 255.

8. Procedure

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

Phosphorus, %	Sample Weight, g	Tolerance in Sample Weight, mg
0.25 to 2.0	2.0	0.4
2.0 to 10.0	1.0	0.3
10.0 to 15.0	0.5	0.2

8.2 Place the weighed sample in a 400-mL beaker. Add 20 mL of $\text{HNO}_3(1 + 1)$. Heat to dissolve the sample and boil to expel the oxides of nitrogen. Add 10 mL of potassium chlorate solution to complete the oxidation of the phosphorus to phosphate. Continue boiling and evaporate to between 5 and 10 mL then cool. If crystals appear, add a few millilitres of water to dissolve them. A faint cloudiness in the solution may indicate the presence of silver chloride.

8.3 Dilute to 125 mL and heat to boiling. Remove from heat and carefully add 25 mL of magnesium chloride precipitant followed by 50 mL of NH_4OH . Stir with a glass rod, leaving the rod in the beaker. Cool overnight to allow for complete precipitation.

¹ This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.03 on Precious Metals.

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

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