



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.

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.

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 $HNO_3(1 + 1)$. 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.

8.4 Set up a vacuum filter using a glass funnel and 12.5-cm low porosity, ashless filter paper⁴ with a 9-cm hardened ashless filter paper as a support.⁵ Moisten these with water and pack with enough filter pulp to fill the funnel about $\frac{3}{4}$ in. from the bottom. Transfer the solution and precipitate to the funnel and rinse the beaker three times with NH_4OH (1 + 19). Some of the precipitate may stick to the beaker and stirring rod. It is not necessary to remove it completely, because the beaker will be used for the second precipitation. Wash the filter paper and precipitate four times with 10-mL portions of NH_4OH (1 + 19).

8.5 Turn off the vacuum, set the funnel aside, empty the filter flask, and discard the filtrate and washings containing the copper and silver. Rinse the filter flask three times with water and reinsert the funnel containing the precipitate.

8.6 Turn the vacuum on and dissolve the magnesium ammonium phosphate precipitate through the filter with 20 mL of HCl (1 + 3). Wash with water. Then wash alternately with 10 mL of HCl (1 + 3) and water, two more times each. Rinse one additional time with water. Discard the filter paper.

8.7 Rinse the sides of the original 400 mL beaker and the stirring rod with 10 mL of HCl (1 + 3) to dissolve any precipitate. Transfer the filtrate from the filter flask to the beaker, rinse the flask thoroughly with water, and add the washings to the beaker.

8.8 Add 10 mL of magnesium chloride precipitant and 30 mL of NH_4OH to the solution in the beaker. If the precipitate does not start to form immediately, add another 10 mL of NH_4OH . Let the solution stand for 30 min at room temperature.

8.9 Set up a vacuum filter using a glass funnel and 12.5-cm low porosity, ashless filter paper³ with a 9-cm hardened ashless filter paper as a support.⁴ Moisten these with water and pack with enough filter pulp to fill the funnel about $\frac{3}{4}$ in. from the bottom. Transfer the solution and precipitate to the funnel. Use NH_4OH (1 + 19) to complete the transfer and rinse the beaker three times. Wash the precipitate three times with NH_4OH (1 + 19) and once with a methanol.

8.10 Transfer the filter paper with the precipitate to an ignited and tared porcelain crucible. Heat the crucible uncovered over a Meeker burner, gently at first to dry the paper, then strongly to burn off as much paper as possible. Transfer to a muffle furnace at 1700°F (927°C) and ignite for 1 h, or until the precipitate is pure white. Cool in a desiccator and weigh.

⁴ Whatman No. 42 filter paper, available from Whatman Lab Sales, Inc., P.O. Box 1359, Hillsboro, OR 97123 or equivalent has been found suitable for this purpose.

⁵ Whatman No. 541 or equivalent has been found suitable for this purpose.

TABLE 1 Statistical Information—Phosphorus

Test Material	Phosphorus Found, %	R1, E173	R2, E173
Copper Phosphorus Powder 2.50 % Phosphorus	2.37	.021	.039
Copper Phosphorus Powder 7.00 % Phosphorus	7.11	.075	.107
Copper Phosphorus Powder 15.0 % Phosphorus	14.19	.175	.273

NOTE 1—Ignition at the proper temperatures in excess oxygen such as that described above is necessary to completely convert the precipitate to magnesium pyrophosphate and to prevent any reduction of that compound.

9. Calculation

9.1 *Rounding Calculated Values*—Calculated values shall be rounded to the desired number of places in accordance with the rounding method given in Sections 4.4 and 4.5 of Practice E 29.

9.2 Calculate the percentage of phosphorus as follows:

$$P, \% = [(A \times 0.2783)/B] \times 100 \quad (1)$$

where:

A = magnesium pyrophosphate $\text{Mg}_2\text{P}_2\text{O}_7$, g, and

B = sample used, g.

10. Precision and Bias ⁶

10.1 *Precision*—Five laboratories, including two operators at one location, cooperated in testing the precision of this test method and obtained the data summarized in Table 1. The test method was tested and the data calculated according to Practice E 173. Care should be exercised in drawing inferences from the statistics because fewer than the required number of laboratories participated.

10.2 *Bias*—No estimate of the bias of this test method is available, because no certified reference materials were available at the time of the interlaboratory test. To verify the results of the procedure, analysts may prepare a test sample of known phosphorus content by combining a weighed amount of potassium dihydrogen phosphate and copper metal, test it as directed by the procedure, and compare the result with the theoretical percent phosphorus.

11. Keywords

11.1 phosphorus; phosphorus-copper alloys; phosphorus-copper-silver alloys

⁶ Supporting data are available from ASTM Headquarters. Request RR: E1-1000.

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