BRITISH STANDARD

BS 1748-8: 1960

Method for

The determination of phosphorus in copper alloys (photometric method)

Co-operating organizations

The Non-ferrous Metals Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments, and scientific and industrial organizations:

Admiralty*
Aluminium Development Association
Aluminium Industry Council
Association of Bronze and Brass Founders*
Association of Consulting Engineers, Incorporated
Association of Non-ferrous Metal Stockists
British Bronze and Brass Ingot Manufacturers' Association*
British Electrical and Allied Manufacturers' Association
British Non-ferrous Metals Federation
British Non-ferrous Metals Research Association*
Cable Makers' Association
Copper Development Association
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Institution of Mechanical Engineers (Automobile Division)
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Tin Research Institute
War Office
Zinc Development Association
Individual manufacturers

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the sub-committee entrusted with the preparation of this British Standard:

D.S.I.R. — Laboratory of the Government Chemist
Individual firms

This British Standard, having been approved by the Non-ferrous Metals Industry Standards Committee and endorsed by the Chairman of the Engineering Divisional Council, was published under the authority of the General Council on 20 July 1960

The following BSI references relate to the work on this standard:
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Amendments issued since publication

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Contents

Foreword

This standard makes reference to the following British Standards:

BS 1400, *Copper alloy ingots and castings.*

BS 1499, *Sampling non-ferrous metals.*

This method for the determination of phosphorus is one of a series of methods for the analysis of copper alloys, which will form a complete British Standard issued in several parts. The methods are intended primarily for the analysis of the copper alloys included in BS 1400, "*Copper alloy ingots and castings*", and those being included in the series of schedules of wrought copper and copper alloys, BS 2870 to BS 2875, of which BS 2871 has already been published, and the remainder are in course of preparation.

The methods have been found to give reliable and reproducible results and, while in some instances they may appear to be lengthy, it should be realized that they are put forward as referee methods to be used in cases of dispute.

In the preparation of the calibration graphs an attempt has been made to define concentrations, but because these are linked with the particular instrument used for determining optical density they should be used only as a guide.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 3 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Introduction

a) *Principle*. The sample is dissolved in a mixture of nitric and hydrochloric acids, and phosphorus is converted into orthophosphoric acid. Phosphovanadomolybdic acid is subsequently formed and the yellow colour is measured photometrically.

b) *Range*. Up to 1.20 per cent phosphorus.

c) *Reproducibility*. Experiments have been carried out independently by a number of analysts using the method recommended in this standard. The degree of reproducibility that can be expected is shown by the following analysis of the results obtained:

Phosphorus content	Standard deviation
per cent	per cent
Below 0.10	0.004
0.5	0.01
1.20	0.02

d) *Application*. The method is applicable to all types of copper alloys provided a suitable compensating solution is used when elements are present which would otherwise interfere in the optical density determination. The percentage of arsenic normally present in the specified alloys does not interfere.

Apparatus

a) Grade A graduated glassware shall be used throughout.

b) Any instrument suitable for measuring the optical density of the solution at a wave-length of 435 mμ may be used.

If a filter photometer is used the filter combination should conform as closely as possible to the above wave-length. The following conditions have been found suitable:

Ilford 601 with Chance H503.

4 cm and 1 cm cells.

Solutions required

All reagents shall be of the highest purity obtainable, and distilled water shall be used throughout. Solutions shall be freshly prepared and, where necessary, filtered.

Standard potassium dihydrogen phosphate (1 ml ≡ 0.20 mg of phosphorus). Dissolve 0.8787 g of potassium dihydrogen phosphate (previously dried at 105 °C) in about 250 ml of water. Add 100 ml of nitric acid (20 per cent v/v) and dilute to 1 litre in a graduated flask.

Ammonium molybdate (10 per cent w/v). Dissolve 100 g of ammonium molybdate ($(NH_4)_6Mo_7O_{24}$ $4H_2O$) in about 600 ml of warm water, cool, and dilute to 1 litre. Filter through a fine textured paper immediately before using.

Ammonium vanadate (0.25 per cent w/v). Dissolve 2.5 g of ammonium metavanadate in about 500 ml of hot water. Add 20 ml of nitric acid (50 per cent v/v), cool, and dilute to 1 litre.

Hydrogen peroxide (3 per cent). Dilute 10 ml of hydrogen peroxide (100 vol) to 100 ml. Store in a dark bottle in a cool place.

Nitric acid (50 per cent v/v). Dilute 50 ml of nitric acid (sp. gr. 1.42) to 100 ml.

Nitric acid (20 per cent v/v). Dilute 20 ml of nitric acid (sp. gr. 1.42) to 100 ml.

Nitric-hydrochloric acid mixture. To 500 ml of water add 320 ml of nitric acid (sp. gr. 1.42) and 120 ml of hydrochloric acid (sp. gr. 1.16–1.18). Cool and dilute to 1 litre.

Sampling

Recommended methods of obtaining a suitable sample for the analytical procedure given below are described in BS 1499, "*Sampling non-ferrous metals*".

Calibration

Weigh 2.00 g of pure copper (containing less than 0.0002 per cent phosphorus) into each of a number of 150 ml beakers. Use a further beaker in each case for a blank determination and proceed as described below.

Graph	Phosphorus content	Cell size	Volume of standard phosphate solution	Sample weight subsequently used
	per cent	cm	ml	g
1 (*a*)	Up to 0.03	4	0.5 1.0 2.0 3.0 4.0	2
(*b*)	0.10–0.30			0.2
2 (*a*)	0.02–0.12	1	2.0 4.0 6.0 8.0 10.0 12.0 15.0	2
(*b*)	0.20–1.20			0.2

i) *Preparation of Graphs* 1*a and* 2*a*

Add 30 ml of the nitric-hydrochloric acid mixture to each beaker, cover and heat gently until the copper has dissolved. Make additions of the standard potassium dihydrogen phosphate solution as indicated in the table (Note 1). Add 1 ml of hydrogen peroxide (3 per cent) and boil gently for 5 minutes. Avoid vigorous or prolonged boiling, since excessive loss of acid will affect the subsequent colour development. Cool slightly and add 10.0 ml of ammonium vanadate solution (0.25 per cent). Cool to room temperature and add 10.0 ml of ammonium molybdate solution (10 per cent). Transfer to a 100 ml graduated flask, dilute to the mark and mix well. Allow to stand for 5 minutes at 20 ± 1 °C, then measure the optical densities using conditions as specified under "Apparatus" (see also Note 2). Use 4 cm cells for Graph 1a and 1 cm cells for Graph 2a. Use the blank solution in the compensating cell.

ii) *Preparation of Graphs* 1*b and* 2*b*

Add 45 ml of the nitric-hydrochloric acid mixture to each beaker, cover and heat gently until the copper has dissolved. Cool and dilute to 100 ml in a graduated flask. Transfer 10.0 ml of each solution (equivalent to 0.2 g sample) separately to 150 ml beakers. Make additions of the standard potassium dihydrogen phosphate solution as indicated in the table (Note 1). Add 10 ml of the nitric-hydrochloric acid mixture, 1 ml of hydrogen peroxide (3 per cent) and continue as described under "Calibration (i)" above. Use 4 cm cells for Graph 1b and 1 cm cells for Graph 2b. Use the blank solution in the compensating cell.

In each case plot the values obtained against percentage phosphorus (based on the sample weights subsequently used) and prepare calibration graphs.

Procedure

i) *Phosphorus content up to* 0.12 *per cent*

Dissolve two separate 2.000 g weighings of the sample in 30 ml (Note 1) of nitric-hydrochloric acid mixture. Mark the solutions "(T)" and "(C)".

At the same time, dissolve two separate 2.00 g weighings of pure copper in 30 ml of nitric-hydrochloric acid mixture. Mark the solutions "Blank (T)" and "Blank (C)". Proceed with each as described under "Calibration", but omit the addition of ammonium molybdate solution (10 per cent) to solutions (C) and Blank (C).

Measure the optical densities using the conditions as specified under "Apparatus" (Note 2), using the solution (C) in the compensating cell.

Deduct the blank reading and obtain the percentage phosphorus in the sample by reference to the appropriate calibration graph.

ii) *Phosphorus content* 0.10 to 1.20 *per cent*

Dissolve 2.000 g of the sample in 45 ml (Note 1) of the nitric-hydrochloric acid mixture. At the same time, dissolve 2.000 g of pure copper in 45 ml of the nitric-hydrochloric acid mixture to provide a blank. Boil each solution for 1 minute to remove nitrous fumes, cool and dilute each to 100 ml in graduated flasks.

Transfer two separate 10 ml aliquots of the sample solution (equivalent to 0.20 g sample) to 150 ml beakers and mark the solutions "(T)" and "(C)".

Transfer two separate 10 ml aliquots of the blank solution to 150 ml beakers and mark the solutions "Blank (T)" and "Blank (C)".

Add to each solution 10 ml of the nitric-hydrochloric acid mixture, then 1 ml of hydrogen peroxide (3 per cent). Proceed with each as described under "Calibration", but omit the addition of ammonium molybdate solution (10 per cent) to solutions (C) and Blank (C).

Measure the optical densities using the conditions as specified under "Apparatus" (Note 2), using the solution (C) in the compensating cell.

Deduct the blank reading and obtain the percentage phosphorus in the sample by reference to the appropriate calibration graph.

NOTE 1 These recommended volumes should be found suitable, using the type of instrument referred to under "Apparatus". Preparation of these graphs will depend on the type of instrument used to determine optical density and it may therefore be necessary to reduce these volumes, the weight of copper and the weight of sample subsequently used. The excess of nitro-hydrochloric acid mixture, however, must be maintained throughout.

NOTE 2 In the examination of sample solutions optical densities should preferably be between 0.15 and 0.85, although this range may be extended slightly in the preparation of calibration graphs.

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