BRITISH STANDARD

BS 4258-8:
1978
ISO 3707:1976

Methods of test for

Phosphoric acid (orthophosphoric acid) for industrial use —

Part 8: Determination of calcium content (flame atomic absorption method)

[ISO title: Phosphoric acid for industrial use (including foodstuffs) — Determination of calcium content — Flame atomic absorption method]

UDC 661.634.2:546.185 – 325:543.422.062:546.41

Cooperating organizations

The Chemicals Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Association of Fatty Acid Distillers
British Tar Industry Association
Chemical Industries Association*
Chemical Society, Analytical Division
Department of Health and Social Security
Department of Industry, Chemicals and Textiles Division
Department of Industry, Laboratory of the Government Chemist*
Fertiliser Manufacturers' Association Ltd.*
Hydrocarbon Solvents Association
Ministry of Agriculture, Fisheries and Food
Ministry of Defence
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Paintmakers' Association of Great Britain Ltd.
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association
Standardization of Tar Products Tests Committee

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Pharmacopoeia Commission
Campden Food Preservation Research Association
Flour Milling and Baking Research Association
Institute of Metal Finishing
Institution of Water Engineers and Scientists
National Association of Soft Drinks Manufacturers
Society of Chemical Industry
Textile Institute
Individual expert

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

Amd. No.	Date of issue	Comments

Contents

Foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee in order to provide methods for the analysis of phosphoric acid.

For some years the United Kingdom has participated in the work of preparing methods of test applicable to phosphoric acid for industrial use, organized by Subcommittee 6 (formerly Working Group 7), Phosphoric acid and condensed phosphates, of Technical Committee 47, Chemistry, of the International Organization for Standardization (ISO). As international agreement is reached on the methods, it is proposed to publish them as Parts of this British Standard.

This Part is identical with ISO 3707 "*Phosphoric acid for industrial use (including foodstuffs) — Determination of calcium content — Flame atomic absorption method*".

Terminology and conventions. The text of the International Standard has been accepted as suitable for publication, without deviation, as a British Standard. Certain terminology and conventions are used, however, that are not identical with those used in British Standards. Attention is therefore drawn to the following.

Where the words "International Standard" relating to this publication appear, they should be interpreted as "British Standard".

The comma has been used throughout as a decimal marker. In British Standards it is current practice to use a full point (a full stop on the baseline) as the decimal marker.

Cross references. The related ISO publications listed in the Annex correspond to Parts of this British Standard as follows.

ISO publication	Part of BS 4258
ISO/R 848[a]	Part 1 *Determination of calcium content*
ISO/R 849[a]	Part 2 *Determination of iron content*
ISO 2997	Part 3 *Determination of sulphate content*
ISO 3360	Part 4 *Determination of fluorine content*
ISO 3361	Part 5 *Determination of silica content*
ISO 3359	Part 6 *Determination of arsenic content*
ISO 3706	Part 7 *Determination of phosphorus (V) oxide content*
ISO 3707	Part 8 *Determination of calcium content* (flame atomic absorption method)
ISO 3708	Part 9 *Determination of chloride content*
ISO 3709	Part 10 *Determination of oxides of nitrogen content*
ISO 4285	Part 11 *Guide to sampling techniques*[b]

[a] At the time of publication of this British Standard, ISO/R 847, ISO/R 848 and ISO/R 849 have not been transformed into International Standards, although they are listed as such in the Annex.

[b] In course of preparation.

There is no British Standard corresponding to ISO/R 847[1)] and it is not intended that one will be published as the method uses as a reagent the known carcinogen benzidine and ISO/TC 47/SC 6 has recommended that it be withdrawn.

[1)] At the time of publication of this British Standard, ISO/R 847, ISO/R 848 and ISO/R 849 have not been transformed into International Standards, although they are listed as such in the Annex.

Textual error. When adopting the text of the International Standard, the error given below was noticed. This has been corrected in this British Standard.

Clause reference	Textual change
Annex	In ISO 3707, the title of ISO 4285 was incorrectly given as "*Sampling technique*".

Additional information. This standard specifies methods of test only and should not be used as a specification defining limits of purity. Reference to the standard should be in the form of words indicating that the methods of test used comply with the requirements of BS 4258.

WARNING. Attention is drawn to **5.1**. It is essential that the atomic absorption spectrophotometer is equipped with a burner designed to operate with an acetylene/dinitrogen monoxide flame, as serious hazards will result if the wrong type of burner is used with this gas mixture.

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.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 4, an inside back cover 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.

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1 Scope

This International Standard specifies a flame atomic absorption method for the determination of the calcium content of phosphoric acid for industrial use (including foodstuffs).

2 Field of application

The method is applicable to products having a calcium content higher than 50 mg/kg but, by preparation of a suitable calibration graph, the range can be extended down to 10 mg/kg.

Soluble SiO_2 present in the test solution at levels lower than 5 μg/ml does not interfere.

2.1 Special case

Presence of soluble SiO_2 at levels higher than 5 μg/ml of test solution (under study).

3 Principle

Addition, to a hydrochloric acid solution of the test portion, of sodium ions to promote and to stabilize the emission of calcium, and of lanthanum ions to suppress the interference of aluminium. Aspiration of the solution into an acetylene-dinitrogen monoxide flame and determination of the calcium content by photometric measurement of the absorption of the 422,7 nm line emitted by a hollow-cathode calcium lamp.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water doubly distilled in borosilicate glass apparatus with ground glass joints, or water of equivalent purity.

4.1 *Phosphoric acid*, 40 g/l solution free from calcium.

Weigh, to the nearest 0,1 g, 29 g of phosphorus(V) oxide (P_2O_5) and spread out in a shallow layer in a suitable dish. Allow the dish to stand in a closed vessel containing water (for example a desiccator containing water in place of a desiccant), in order to effect the initial hydration. Then dissolve the hydrated oxide in 1 000 ml of water.

4.2 *Hydrochloric acid*, approximately 6 N solution.

4.3 *Sodium chloride and lanthanum chloride*, combined solution.

Dissolve 25,5 g of sodium chloride and 10 g of lanthanum chloride heptahydrate ($LaCl_3.7H_2O$) in water and dilute to 100 ml.

1 ml of this solution contains approximately 100 mg of Na and 100 mg of lanthanum chloride heptahydrate.

4.4 *Calcium*, standard solution, corresponding to 1,000 g of Ca per litre.

Weigh, to the nearest 0,000 1 g, 2,497 2 g of calcium carbonate, previously dried at 250 °C for 2 h and cooled in a desiccator. Place in a beaker of convenient capacity (for example 600 ml) and dissolve carefully in 30 ml of the hydrochloric acid solution (**4.2**). Dilute the solution and transfer quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 1,000 mg of Ca.

Store this solution in a bottle of material free from calcium.

4.5 *Calcium*, standard solution, corresponding to 0,050 g of Ca per litre.

Transfer 50,0 ml of the standard calcium solution (**4.4**) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 50 μg of Ca.

Prepare this solution just before use.

5 Apparatus

Ordinary laboratory apparatus, of material free from calcium, and

5.1 *Atomic absorption spectrophotometer*, fitted with a burner fed with acetylene and dinitrogen monoxide.

5.2 *Hollow-cathode calcium lamp*

6 Procedure

6.1 Test portion

Weigh, by difference, to the nearest 0,001 g, about 5 g of the test sample.

6.2 Preparation of the calibration graph

6.2.1 *Preparation of the standard matching solutions*

Into each of a series of five 100 ml one-mark volumetric flasks, place a quantity of the phosphoric acid solution (**4.1**) such that it contains the same quantity of P_2O_5 as the test portion (**6.1**). Add 1 ml of the combined solution (**4.3**) and 4 ml of the hydrochloric acid solution (**4.2**) and then, respectively, the volumes of the standard calcium solution (**4.5**) shown in the following table.

Standard calcium solution (4.5)	Corresponding mass of Ca
ml	μg
0[a]	0
1,0	50
2,0	100
4,0	200
6,0	300

[a] Blank test on reagents of the calibration graph.

Dilute the contents of each flask to the mark and mix.

NOTE If the test solution contains less than 50 μg of calcium (Ca) in 100 ml, prepare a more dilute standard calcium solution by diluting 10,0 ml of the standard calcium solution (**4.5**) to 100 ml. Use this weaker solution to prepare a calibration graph covering the range 0 to 50 μg of Ca in 100 ml. The bracketing measurements (**6.3.2.2**) should then be carried out between two standard matching solutions differing by 5 μg of Ca in 100 ml.

6.2.2 *Spectrophotometric measurements*

6.2.2.1 *ADJUSTMENT OF THE APPARATUS FITTED WITH THE HOLLOW CATHODE CALCIUM LAMP (5.2)*

Switch on the current to the apparatus (**5.1**) a sufficient time in advance to ensure stabilization. Adjust the wavelength to about 422,7 nm and adjust the sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the pressure of the acetylene and of the dinitrogen monoxide according to the characteristics of the aspirator burner. Adjust the aspiration rate to between 2 and 4 ml/min.

6.2.2.2 *MEASUREMENTS*

Aspirate the series of standard matching solutions (**6.2.1**) in the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration graph.

Aspirate water through the burner after each measurement.

6.2.3 *Plotting the calibration graph*

Plot a graph having, for example, the numbers of micrograms of Ca contained in 100 ml of the standard matching solutions as abscissae and the corresponding values of the absorbances, reduced by the value for the standard matching solution No. 0, as ordinates.

6.3 Determination

6.3.1 *Preparation of the test solution*

Transfer the test portion (**6.1**) to a 500 ml one-mark volumetric flask and dilute to about 250 ml. Add 20 ml of the hydrochloric acid solution (**4.2**), and 5 ml of the combined solution (**4.3**). Dilute to the mark and mix.

If the calcium content is between 50 and 200 mg/kg, carry out the measurements directly on the test solution thus obtained.

If the calcium content is higher, carry out further dilutions as indicated in the following table.

Expected Ca content	Aliquot portion of the test solution (6.3.1) to be taken	Combined solution (4.3) to be added	Final volume of the solution
mg/kg	ml	ml	ml
200 to 500	50	0,50	100
500 to 1 000	25	0,75	100
1 000 to 1 500	20	0,80	100
1 500 to 2 000	10	0,90	100

6.3.2 *Spectrophotometric measurements*

6.3.2.1 *PRELIMINARY MEASUREMENT*

Carry out a preliminary measurement on the test solution (**6.3.1**) following the procedure specified in **6.2.2.2**, at the same time as the spectrophotometric measurements are carried out on the standard matching solutions (**6.2.1**).

From the calibration graph (**6.2.3**), calculate the approximate concentration of Ca, in micrograms per 100 ml of the test solution (**6.3.1**).

6.3.2.2 *BRACKETING MEASUREMENT*

Carry out a second measurement on the test solution (**6.3.1**) by bracketing between two standard matching solutions differing by only 25 μg of Ca in 100 ml.

To prepare these standard matching solutions, follow the procedure specified in **6.2.1**, using, however, suitable quantities of the standard calcium solution (**4.5**).

7 Expression of results

The concentration C of calcium, expressed as micrograms of Ca per 100 ml of the test solution, is given by the formula

$$C = C_1 + (C_2 - C_1)\,\frac{A_0 - A_1}{A_2 - A_1}$$

where

C_1 is the concentration, in micrograms per 100 ml, of the weaker bracketing solution;

A_1 is the corresponding value of the absorbance;

C_2 is the concentration, in micrograms per 100 ml, of the stronger bracketing solution;

A_2 is the corresponding value of the absorbance;

A_0 is the value of the absorbance corresponding to the test solution (**6.3.1**).

The calcium (Ca) content, expressed in milligrams per kilogram, is given by the formula

$$\frac{C}{m} \times \frac{500}{100} \times D = \frac{C}{m} \times 5 \times D$$

where

C is the concentration of Ca, expressed as micrograms per 100 ml of the test solution;

m is the mass, in grams, of the test portion (**6.1**);

D is the dilution ratio (see the table in **6.3.1**);

5 is the ratio of the volume of the test solution (**6.3.1**) to the volume of the calibration solutions (**6.2.1**).

8 Test report

The test report shall include the following particulars:

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

Annex ISO Publications relating to phosphoric acid for industrial use

ISO 847, *Determination of sulphate content — Titrimetric method.*

ISO 848, *Determination of calcium content — Titrimetric method.*

ISO 849, *Determination of iron content — 2,2′-Bipyridyl spectrophotometric method.*

ISO 2997, *Determination of sulphate content — Method by reduction and titrimetry.*

ISO 3359, *Determination of arsenic content — Silver diethyldithiocarbamate photometric method.*

ISO 3360, *Determination of fluorine content — Alizarin complexone and lanthanum nitrate photometric method*[2)].

ISO 3361, *Determination of soluble silica content — Reduced molybdosilicate spectrophotometric method.*

ISO 3706, *Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method*[2)].

ISO 3707, *Determination of calcium content — Flame atomic absorption method*[2)].

ISO 3708, *Determination of chloride content — Potentiometric method*[2)].

ISO 3709, *Determination of nitrogen oxides content — 3,4-Xylenol spectrophotometric method*[2)].

ISO 4285, *Guide to sampling techniques.*

[2)] Also applicable to phosphoric acid for use in the foodstuffs industry.

Publications referred to

See foreword

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