BRITISH STANDARD

BS 4258-7: 1978

Methods of test for

Phosphoric acid (orthophosphoric acid) for industrial use —

Part 7: Determination of total phosphorus(V) oxide content: quinoline phosphomolybdate gravimetric method

UDC 661.634.2:546.185 – 325:543.21.062:546.185 – 31

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
National Sulphuric Acid Association
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

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Executive Board on 31 January 1978

The following BSI references relate to the work on this standard:
Committee reference CIC/25
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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.

Part 7 is based on ISO 3706 "*Phosphoric acid for industrial use (including foodstuffs) — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method*", with the inclusion of some technical changes in the text; these are given below. The relevant passages are indicated by a vertical line in the margin.

Clause reference	Textual change
4.1 and **4.2.4**	In ISO 3706, the relative density for hydrochloric acid is given as approximately 1,19 g/ml, about 38 % (*m*/*m*) solution.
4.2.2	In ISO 3706, the relative density for nitric acid is given as approximately 1,40 g/ml, about 68 % (*m*/*m*) solution.
6.3	In the table in ISO 3706, the heading of column 2 is "aliquot part of solution A (**6.1**) to be taken".
7.1.2	In ISO 3706, the equation is incorrectly printed as $$(m_1 - m_2) \times 0{,}032\,07 \times \frac{500}{50} \times D \times \frac{100}{m_0 \times \frac{m_4}{m_0 + m_3}}$$ $$= \frac{32{,}07 \times D \times (m_1 - m_2) \times (m_0 + m_3)}{m_0 \times m_4}$$ In the definition of m_4, the word "homogenized" is incorrectly included before "assay sample". NOTE The corrected versions of the formula and the definition of m_4, as printed in this British Standard, have been proposed by BSI as a technical correction to the ISO text.
Annex	In ISO 3706, the title of ISO 4285 was incorrectly given as "Sampling technique".

For printing purposes, the text of the International Standard has been used as the basis for the British Standard. The decimal comma has been used throughout; in British Standards it is current practice to use a full point 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 publication	Part of BS 4258
ISO 3709	Part 10 *Determination of oxides of nitrogen content*
ISO 4285[b]	Part 11[c] *Guide to sampling techniques*

[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] The footnote on page 1 referring to ISO 4285 states that this is at the drafting stage; this is now published.
[c] 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/TC47/SC 6 has recommended that it be withdrawn.

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.

With reference to clause **4**, water complying with the requirements of BS 3978 is suitable.

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.

[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.

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1 Scope

This British Standard specifies a gravimetric method using quinoline phosphomolybdate for the determination of the total phosphorus(V) oxide content of phosphoric acid for industrial use (including foodstuffs).

2 Field of application

The method is applicable to phosphoric acids, whether or not they are homogeneous and whether or not they contain polyphosphoric acids.

3 Principle

Preliminary hydrolysis of the polyphosphoric acids by boiling in the presence of hydrochloric acid. Precipitation of the phosphoric acid in the form of quinoline phosphomolybdate in the presence of acetone. Filtration, washing, drying and weighing of the precipitate.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 *Hydrochloric acid*, ρ approximately 1,18 g/ml, about 36 % (*m*/*m*) or approximately 12 N solution.

4.2 *Citromolybdate reagent*

4.2.1 Dissolve 70 g of sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) in 150 ml of water.

4.2.2 Dissolve 60 g of citric acid monohydrate ($C_6H_8O_7.H_2O$) in 150 ml of water and add 85 ml of nitric acid solution, ρ approximately 1,42 g/ml, about 70 % (*m*/*m*) or approximately 14 N solution.

4.2.3 Add, while stirring, solution **4.2.1** to solution **4.2.2**.

4.2.4 Add 35 ml of nitric acid solution, ρ approximately 1,42 g/ml, about 70 % (*m*/*m*) or approximately 14 N solution, then 5 ml of recently distilled quinoline to 100 ml of water.

4.2.5 Add solution (**4.2.4**) to solution (**4.2.3**) and mix.

Allow to stand for at least 12 h and filter through the filter crucible (**5.1**).

Store this solution protected from light, in a well-stoppered flask.

4.2.6 Add 280 ml of acetone to solution (**4.2.5**) and dilute to 1 000 ml with water.

Do not keep this solution for more than 1 week. Store under the same conditions as solution (**4.2.5**).

5 Apparatus

Ordinary laboratory apparatus and

5.1 *Filter crucible*, with sintered glass disk, of porosity P10 (pore size index between 4 and 10 μm).

5.2 *Electric oven*, capable of being controlled at 250 ± 10 °C.

6 Procedure

6.1 Test portion and preparation of the test solution

6.1.1 *Homogeneous phosphoric acid* (or phosphoric acid containing a precipitate which readily forms a suspension)

Weigh by difference, to the nearest 0,000 2 g, 5 ± 0,2 g of the test sample, in such a way that there is no gain or loss of moisture.

Transfer the test portion to a flask of about 250 ml capacity, add 10 ml of the hydrochloric acid solution (**4.1**), cover with a clock-glass and boil for about 10 min. Cool, add about 100 ml of water and 10 ml of the hydrochloric acid solution (**4.1**). Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer 50,0 ml of this solution to a 500 ml one-mark volumetric flask, dilute to the mark and mix (solution A).

Prepare this dilution at the time of use.

6.1.2 *Non-homogeneous phosphoric acid*, containing a precipitate which does not readily form a suspension

Weigh by difference, to the nearest 0,05 %, the whole of the assay sample[2)], in such a way that there is no gain or loss of moisture.

Transfer the sample to a polyethylene or polypropylene flask of suitable capacity, and homogenize by dilution or any other method which does not involve any loss of the constituents.

[2)] The definition of the term "assay sample" will be given in ISO 4285, *Phosphoric acid for industrial use — Sampling technique. (At present* at the stage of draft.)
If the assay sample is not available, a test sample may be taken from the laboratory sample, which must first be thoroughly mixed so as to render it completely homogeneous. In this case, weigh, to the nearest 0,05 %, about 25 g and treat it as described for the assay sample.

After homogenizing, weigh by difference, to the nearest 0,05 %, a test portion containing about 5 g of the original phosphoric acid, place in a 250 ml beaker and add 10 ml of the hydrochloric acid solution (**4.1**) cover with a clock-glass and boil for about 10 min. Cool, add about 100 ml of water and 10 ml of the hydrochloric acid solution (**4.1**). Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer 50,0 ml of this solution to a 500 ml one-mark volumetric flask, dilute to the mark and mix (solution A).

Prepare this dilution at the time of use and filter if necessary.

NOTE Homogenization may generally be achieved by simple dilution with water (or dilute hydrochloric acid solution), with prolonged stirring in a stoppered flask, the quantity of liquid added being weighed to the nearest 0,05 %.
When this treatment is insufficient, a more effective treatment should be carried out depending on the particular case (treatment with hot water with stirring in a stoppered flask, for example).
Finally, in the case where an insoluble deposit remains which adheres to the walls of the vessel containing the assay sample or which cannot be dispersed, it should be recovered, weighed and analysed separately.

6.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used in the determination.

6.3 Determination

Depending on the expected phosphorus(V) oxide content, transfer to a 400 ml beaker the aliquot portion of solution A (**6.1**) shown in the following table.

Expected P_2O_5 content	Aliquot portion of solution A (6.1) to be taken
% (*m*/*m*)	ml
Less than 30	50
From 30 to 50	25
Greater than 50	20

Dilute to about 100 ml and add 100 ml of the citro-molybdate reagent (**4.2**). Cover the beaker with a clock-glass and warm, inside a fume cupboard, on a hot-plate, until its contents reach 75 ± 5 °C and maintain at this temperature for about 30 s. (Do not use a flame and do not mix, either during the addition of the reagent or during the heating, so as to avoid the formation of clots.) Allow to cool to ambient temperature, stirring three or four times with a glass rod during cooling.

Heat the filter crucible (**5.1**) in the oven (**5.2**) controlled at 250 ± 10 °C, and leave for 15 min starting from the stabilization of the temperature. Allow to cool in a desiccator containing silica gel in good condition and weigh to the nearest 0,000 1 g.

Decant the liquid through the filter crucible (**5.1**) and wash the precipitate six times, by decantation, using about 30 ml of water each time. Transfer the precipitate quantitatively to the filter crucible (**5.1**) with the aid of a jet of water from a wash-bottle. Then wash the precipitate four times, removing each portion of wash water by suction.

Place the filter crucible in the oven (**5.2**) maintained at 250 ± 10 °C and leave for 15 min starting from the stabilization of the temperature. Allow to cool for not more than 30 min in a desiccator containing silica gel in good condition and weigh to the nearest 0,000 1 g.

7 Expression of results

7.1 Method of calculation and formulae

The total phosphorus(V) oxide content, expressed as a percentage by mass of P_2O_5, is given by the formulae:

7.1.1 ***Homogeneous phosphoric acid*** (see **6.1.1**)

$$(m_1 - m_2) \times 0{,}032\,07 \times \frac{500}{50} \times D \times \frac{100}{m_0} = \frac{32{,}07 \times D \times (m_1 - m_2)}{m_0}$$

7.1.2 ***Non-homogeneous phosphoric acid*** (see **6.1.2**)

$$(m_1 - m_2) \times 0{,}032\,07 \times \frac{500}{50} \times D \times \frac{100}{m_0 \times \frac{m_4}{m_4 + m_3}} = \frac{32{,}07 \times D \times (m_1 - m_2) \times (m_4 + m_3)}{m_0 \times m_4}$$

where

m_0 is the mass, in grams, of the test portion (**6.1**);

m_1 is the mass, in grams, of precipitate obtained with the aliquot portion of the test solution (**6.3**);

m_2 is the mass, in grams, of precipitate obtained with the corresponding aliquot portion of the blank test solution (**6.2**);

m_3 is the total mass, in grams, of water or other reagents added for the homogenization (see **6.1.2**, note);

m_4 is the mass, in grams, of assay sample taken (see **6.1.2**);

D is the ratio of the volume of the test solution (solution A) (**6.1**) to the volume of the aliquot portion taken for the determination (**6.3**);

0,032 07 is the factor for conversion of quinoline phosphomolybdate to phosphorus(V) oxide.

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 British Standard or regarded as optional.

Annex Publications relating to phosphoric acid and sodium phosphates 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 photometric 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*[3)].

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

ISO 3706, *Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method*[3)].

ISO 3707, *Determination of calcium content — Flame atomic absorption method*[3)].

ISO 3708, *Determination of chloride content — Potentiometric method*[3)].

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

ISO 4285, *Guide to sampling techniques.*

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

Publications referred to

See foreword

BSI — British Standards Institution

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London
W4 4AL