International Standard 848

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Phosphoric acid for industrial use — Determination of calcium content — Titrimetric method

Acide phosphorique à usage industriel — Dosage du calcium — Méthode titrimétrique

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 848 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in February 1980.

It has been approved by the member bodies of the following countries :

Austria
Belgium
China
Czechoslovakia
Egypt, Arab Rep. of
France
Germany, F. R.
Hungary
India
Italy
Korea, Rep. of
Mexico
Philippines
Poland
Romania
South Africa, Rep. of
Switzerland
Thailand
United Kingdom
USSR

The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

This International Standard cancels and replaces ISO Recommendation R 848-1968, of which it constitutes a technical revision.

Printed in Switzerland

Phosphoric acid for industrial use — Determination of calcium content — Titrimetric method

1 Scope

This International Standard specifies a titrimetric method for the determination of the calcium content of phosphoric acid for industrial use.

2 Field of application

2.1 General case

The general method is applicable to phosphoric acid not containing polyphosphoric acids and to products having a calcium content equal to or greater than 0,020 % (*m/m*).

2.2 Special case

If it is not certain that polyphosphoric acids are absent, proceed as specified in clause 8 for the special case.

3 Principle

Precipitation of calcium as calcium oxalate at pH 4 ± 0,3.

Filtration of the precipitate, dissolution in hydrochloric acid and re-precipitation under the same operating conditions in order to purify the precipitate.

Oxidimetric titration in sulphuric acid solution by means of potassium permanganate.

4 Reagents

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

4.1 Sulphuric acid, ϱ approximately 1,84 g/ml, about 96 % (*m/m*) solution.

4.2 Hydrochloric acid, ϱ approximately 1,19 g/ml, about 37 % (*m/m*) solution.

4.3 Ammonia, ϱ approximately 0,91 g/ml, about 25 % (*m/m*) solution.

4.4 Ammonium chloride, saturated solution at room temperature (approximately 300 g/l).

4.5 Ammonium oxalate, 40 g/l solution.

4.6 Potassium permanganate, standard volumetric solution, $c(1/5\ KMnO_4) = 0,1$ mol/l.

5 Apparatus

Ordinary laboratory apparatus and

5.1 pH meter.

5.2 Witt's apparatus, with a 250 ml beaker (see the figure).

5.3 Funnel, of diameter approximately 60 mm, with a sintered glass disk of porosity grade P 10 (pore size index between 4 and 10 µm).

6 Procedure

6.1 Test portion

Weigh by difference, to the nearest 0,01 g, approximately 50 g of the test sample.

6.2 Determination

6.2.1 Preparation of test solution

Place the test portion (6.1) in a beaker of suitable capacity (for example 250 ml), add 50 ml of water and 20 ml of the hydrochloric acid solution (4.2); bring to the boil, cool and transfer the solution quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark, mix thoroughly and, if necessary, filter on a dry filter paper, collecting the filtrate in a dry container.

6.2.2 First precipitation of calcium oxalate

Transfer 100,0 ml of the filtrate (6.2.1) to a beaker of suitable capacity (for example 250 ml). Add 10 ml of the ammonium chloride solution (4.4) and 50 ml of the ammonium oxalate solution (4.5). Adjust the pH to 4 ± 0,3 by addition, drop by drop, of the ammonia solution (4.3), stirring continually during the operation and checking the pH by means of the pH meter (5.1).

Bring to the boil and continue boiling for 10 min, then allow the precipitate to settle for about 20 min. Decant the supernatant liquid onto the funnel (5.3) using the Witt's apparatus (5.2), leaving most of the precipitate in the beaker.

6.2.3 Dissolution of calcium oxalate

Place the beaker containing most of the precipitate in the Witt's apparatus. With no vacuum applied and the funnel in position on the apparatus, transfer to the funnel approximately 10 ml of water and then 1 ml of the hydrochloric acid solution (4.2).

After a few minutes, apply partial vacuum and collect the filtrate in the beaker.

Disconnect the apparatus from the pump and repeat the operation using 10 ml of water and 1 ml of the hydrochloric acid solution (4.2). Then carefully wash the funnel with water (70 to 80 ml in all), operating under vacuum.

Remove the beaker from the Witt's apparatus, heat to obtain complete dissolution of the calcium oxalate and allow to cool to room temperature.

6.2.4 Second precipitation and dissolution of calcium oxalate

Add 10 ml of the ammonium chloride solution (4.4) and 50 ml of the ammonium oxalate solution (4.5) to the cooled solution. Adjust the pH to 4 ± 0,3 by addition, drop by drop, of the ammonia solution (4.3), stirring continually during the operation and checking the pH by means of the pH meter (5.1).

Bring to the boil and continue boiling for 20 min, then allow the precipitate to settle for 20 min. Decant the supernatant liquid onto the funnel (5.3) using the Witt's apparatus, leaving most of the precipitate in the beaker. Then wash the precipitate contained in the beaker by decantation, using eight to ten 10 ml portions of water.

Place the beaker containing most of the precipitate in the Witt's apparatus. With no vacuum applied and the funnel in position on the apparatus, transfer to the funnel approximately 12 ml of a boiling solution containing 2 ml of the sulphuric acid solution (4.1).

After a few minutes, apply partial vacuum and collect the filtrate in the beaker.

Disconnect the apparatus from the pump and repeat the last acid treatment four times, then carefully wash the funnel with approximately 150 ml of water, operating under vacuum.

Remove the beaker from the Witt's apparatus and heat and stir its contents until the calcium oxalate is completely dissolved.

6.2.5 Titration of calcium oxalate

Adjust the temperature of the solution to 70 °C and titrate with the potassium permanganate solution (4.6) until a light-pink coloration, persisting for 1 min, appears.

At the end of the titration, the temperature of the solution shall not be lower than 60 °C.

7 Expression of results

The calcium content, expressed as Ca as a percentage by mass, is given by the formula

$$\frac{V \times 0{,}002\,005 \times r_D \times 100}{m}$$

$$= \frac{V \times r_D \times 0{,}200\,5}{m}$$

where

V is the volume, in millilitres, of the potassium permanganate solution (4.6) used for the titration (6.2.5);

r_D is the ratio of the volume of the test solution (6.2.1) to the volume of the aliquot portion taken for the determination (6.2.2);

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

0,002 005 is the mass, in grams, of calcium (Ca) corresponding to 1,00 ml of potassium permanganate solution, $c(1/5\ KMnO_4) = 0{,}100$ mol/l.

NOTE — If the standard volumetric solution used does not have exactly the strength indicated in the list of reagents, a suitable correction should be applied.

Express the result to three decimal places.

8 Special case — Phosphoric acid containing polyphosphoric acids

8.1 Principle

Preliminary hydrolysis of polyphosphoric acids by prolonged boiling in the presence of hydrochloric acid. Determination by the method specified above.

8.2 Reagents

See clause 4.

8.3 Apparatus

See clause 5.

8.4 Procedure

8.4.1 Test portion

See 6.1.

8.4.2 Preparation of test solution

Place the test portion (8.4.1) in a flask of capacity approximately 250 ml, add 10 ml of the hydrochloric acid solution (4.2) and boil for about 20 min.

Allow to cool and add 100 ml of water and 10 ml of the hydrochloric acid solution (4.2). Fit a reflux condenser and boil for 1 h.

Allow to cool and transfer the solution quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark, mix thoroughly and filter through a dry filter paper, collecting the filtrate in a dry container.

8.4.3 First precipitation of calcium oxalate

See 6.2.2.

8.4.4 Dissolution of calcium oxalate

See 6.2.3.

8.4.5 Second precipitation and dissolution of calcium oxalate

See 6.2.4.

8.4.6 Titration of calcium oxalate

See 6.2.5.

8.5 Expression of results

See clause 7.

9 Test report

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any usual features noted during the determination;

e) any operations not included in this International Standard or regarded as optional.

Dimensions in millimetres

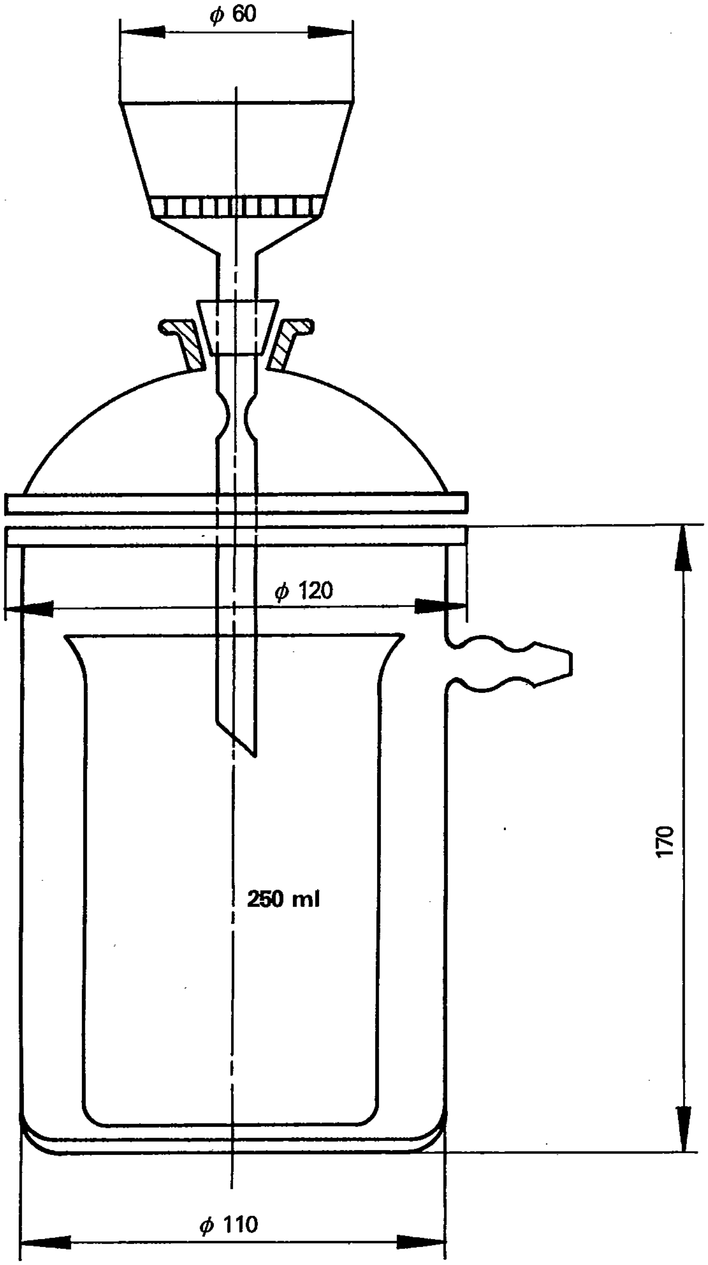

Figure — Witt's apparatus

Annex

ISO publications relating to phosphoric acid for industrial use

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. *

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

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

ISO 3707 — Determination of calcium content — Flame atomic absorption method. *

ISO 3708 — Determination of chloride content — Potentiometric method. *

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

ISO 4285 — Guide to sampling techniques.

ISO 6678 — Determination of lead content — Atomic absorption spectrometric method.

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* Also applicable to phosphoric acid for use in the foodstuffs industry.