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ISO RECOMMENDATION

R 848

PHOSPHORIC ACID FOR INDUSTRIAL USE

DETERMINATION OF CALCIUM CONTENT

VOLUMETRIC METHOD

1st EDITION

October 1968

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 848, *Phosphoric acid for industrial use – Determination of calcium content – Volumetric method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question by the Technical Committee began in 1960 and led, in 1966, to the adoption of a Draft ISO Recommendation.

In December 1966, this Draft ISO Recommendation (No. 1109) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria
Belgium
Brazil
Bulgaria
Chile
Czechoslovakia
France
Germany
Hungary
India
Israel
Italy
Japan
Korea, Dem. P. R. of
Korea, Rep. of
Netherlands
New Zealand
Poland
Romania
South Africa, Rep. of
Spain
Switzerland
Thailand
Turkey
U.A.R.
United Kingdom
U.S.S.R.
Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in October 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 848 October 1968

PHOSPHORIC ACID FOR INDUSTRIAL USE

DETERMINATION OF CALCIUM CONTENT

VOLUMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a volumetric method for the determination of the calcium content in phosphoric acid for industrial use.

2. FIELD OF APPLICATION

The general method is applicable to the determination of the calcium content in phosphoric acid for industrial use.

2.1 Special case

Phosphoric acid for industrial use containing polyphosphoric acids (see section 8).

NOTE. – In case there is no certainty of absence of polyphosphoric acids, carry out the determination according to the procedure of the special case directly.

3. PRINCIPLE

Precipitation of calcium as calcium oxalate at pH 4 ± 0.3.

Filtration of the precipitate and purification by dissolving in hydrochloric acid and re-precipitation under the same operating conditions.

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

4. REAGENTS

Distilled water or water of equivalent purity should be used in the test.

4.1 *Sulphuric acid*, approximately $d = 1.84$, 96 % (m/m) or 36 N solution.

4.2 *Hydrochloric acid*, approximately $d = 1.19$, 37 % (m/m) or 12 N solution.

4.3 *Ammonium hydroxide*, approximately $d = 0.91$, 25 % (m/m) or 15 N solution.

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

4.5 *Ammonium oxalate*, 40 g/l solution.
Dissolve 4 g of ammonium oxalate monohydrate in water and dilute to 100 ml.

4.6 *Potassium permanganate*, 0.1 N standard volumetric solution (see Note, section 7).

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *pH-meter*, fitted with a glass electrode.

5.2 *Witt's apparatus*, with a 250 ml beaker (see Figure opposite).

5.3 *Funnel*, approximately 60 mm in diameter, with sintered glass disk of porosity grade between 5 and 15 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 sample solution*. Place the test portion (6.1) in a beaker of suitable capacity (250 ml, for example), add 50 ml of water and 20 ml of the hydrochloric acid solution (4.2); bring to the boil, cool and transfer quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark, mix thoroughly and filter, if necessary, on a dry filter collecting the filtrate into 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 (250 ml, for example).

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 ammonium hydroxide solution (4.3), stirring during the operation and checking the pH by means of the pH-meter (5.1).

Bring to the boil and continue boiling for 10 minutes, then let the precipitate settle for about 20 minutes.

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.

Dimensions in millimetres

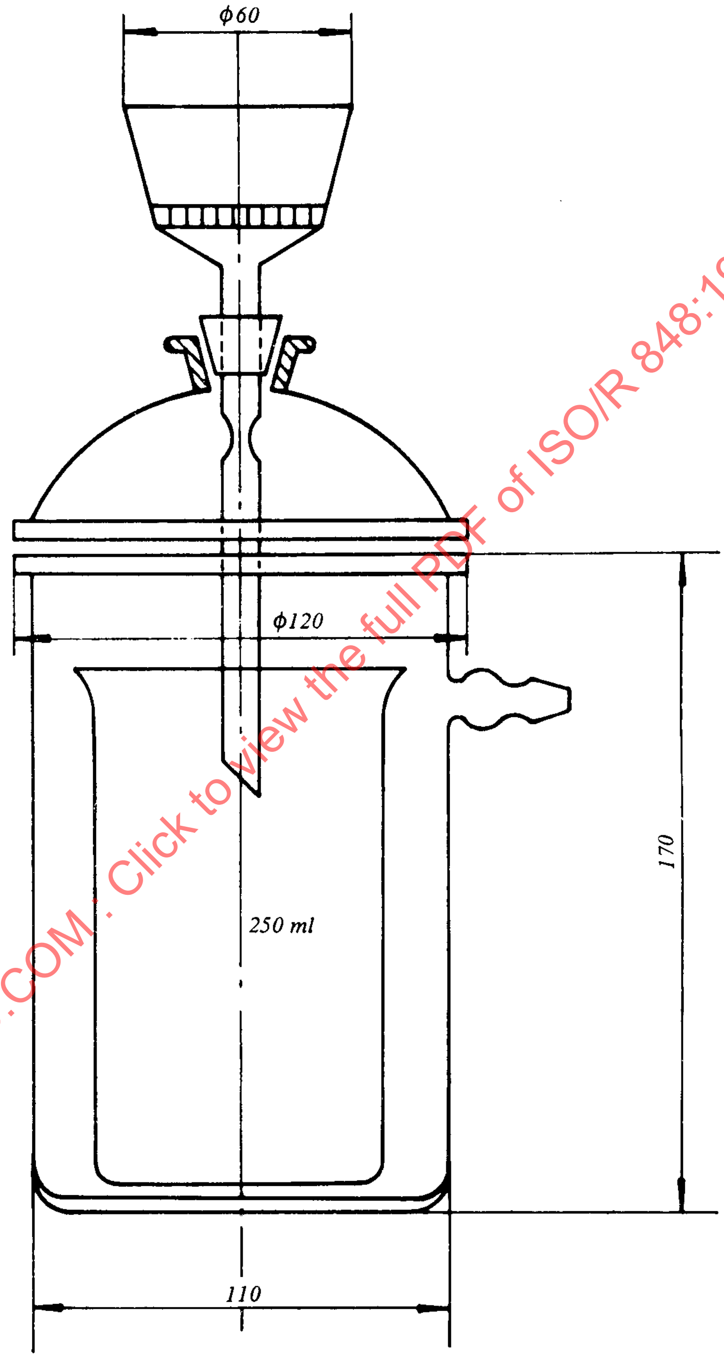

FIGURE – **Witt's apparatus**

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 ammonium hydroxide solution (4.3), stirring during the operation and checking the pH by means of the pH-meter (5.1). Bring to the boil and continue boiling for 20 minutes, then let the precipitate settle for 20 minutes.

Decant the supernatant liquid onto the funnel (5.3) using the Witt's apparatus, leaving most of the precipitate in the beaker. Then wash by decantation the precipitate contained in the beaker, using eight to ten 10 ml portions of water.

Place the beaker containing most of the precipitate into 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 hot 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 operation four times, then carefully wash the funnel with approximately 150 ml of water, operating under vacuum. Remove the beaker from the Witt's apparatus; heat and stir its contents until the calcium oxalate is completely dissolved.

6.2.5 *Titration of calcium oxalate.* Cool the solution to a temperature of approximately 70 °C and titrate with the potassium permanganate solution (4.6) until a light-pink colouration appears; the colouration should be stable for 1 minute.

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

7. EXPRESSION OF RESULTS

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

$$\frac{V \times A \times D}{E} \times 100$$

where

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

A is the mass, in grammes, of calcium corresponding to 1 ml of 0.1 N potassium permanganate solution (theoretical value : 1 ml = 0.002 005 g of Ca See Note below);

D is the ratio of the volume of sample solution (6.2.1) to the aliquot taken for the determination (6.2.2).

E is the mass, in grammes, of the test portion.

NOTE. – If the potassium permanganate standard volumetric solution (4.6) is not of exactly the strength indicated in the list of reagents, a suitable correction factor should be employed in calculating the results.

8. SPECIAL CASE

Phosphoric acid for industrial use containing polyphosphoric acids.

8.1 Principle

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

8.2 Reagents

See section 4.

8.3 Apparatus

See section 5.