

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 847

PHOSPHORIC ACID FOR INDUSTRIAL USE
DETERMINATION OF SULPHATE CONTENT

Volumetric method

1st EDITION

October 1968

COPYRIGHT RESERVED

The copyright of ISO Recommendations and ISO Standards belongs to ISO Member Bodies. Reproduction of these documents, in any country, may be authorized therefore only by the national standards organization of that country, being a member of ISO.

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

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 847, *Phosphoric acid for industrial use – Determination of sulphate 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. 1108) 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	Israel	Spain
Belgium	Italy	Switzerland
Brazil	Japan	Thailand
Bulgaria	Korea, Dem. P.R. of	Turkey
Chile	Korea, Rep. of	U.A.R.
Czechoslovakia	New Zealand	U.S.S.R.
Germany	Poland	Yugoslavia
Hungary	Romania	
India	South Africa, Rep. of	

Two Member Bodies opposed the approval of the Draft :

France
United Kingdom

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.

PHOSPHORIC ACID FOR INDUSTRIAL USE
DETERMINATION OF SULPHATE CONTENT

Volumetric method

1. SCOPE

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

2. FIELD OF APPLICATION

The general method is applicable to the determination of the sulphate content in phosphoric acid for industrial use greater than 0.30 % (m/m) of H_2SO_4 content.

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

Separation of SO_4^{2-} ions as benzidine sulphate. Alkalimetric titration, in the presence of phenolphthalein, as indicator, of the sulphuric acid evolved by hydrolysis.

4. REAGENTS

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

4.1 *Potassium carbonate, anhydrous.*

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

4.3 Benzidine hydrochloride, 28 g/l solution.

Dissolve 28 g of benzidine hydrochloride in approximately 500 ml of warm water and then add 20 ml of the $d = 1.19$ hydrochloric acid solution. Cool, dilute to 1000 ml and filter if required.

Because of its carcinogenic nature, handle benzidine with care.

4.4 Sodium hydroxide, 0.1 N standard volumetric solution (see Note, section 7).

4.5 Phenolphthalein, 10 g/l ethanolic solution.

Dissolve 1 g of phenolphthalein in 95 % (v/v) ethanol and dilute to 100 ml with the same ethanol.

4.6 Wash solution

Make a suspension of 0.5 g of carefully washed benzidine sulphate in one litre of water. Shake mechanically for about 2 hours at room temperature and then filter.

5. APPARATUS

Ordinary laboratory apparatus.

6. PROCEDURE

6.1 Test portion

Weigh by difference, to the nearest 0.01 g, approximately 20 g of the test sample.

6.2 Preparation of sample solution

Place the test portion (6.1) in a beaker of suitable capacity (250 ml for example), add approximately 1 g of potassium carbonate (4.1) and boil for about 20 minutes.

Cool and allow to stand, then filter and wash the precipitate until it is acid-free, collecting quantitatively filtrate and washings in a 200 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid solution (4.2), dilute to the mark and mix thoroughly.

6.3 Separation of SO_4^{2-} ions

According to the expected sulphuric acid content to be determined, take aliquots of the sample solution (see clause 6.2) following the examples indicated in the table below.

H_2SO_4 expected content	Aliquots of the solution (6.2) to be taken	Water to be added
(m/m)	ml	ml
up to 2 %	50	10
2 to 4 %	25	35
above 4 %	10	50

Place 20 ml of the benzidine solution (4.3) into a beaker of suitable capacity and add the quantity of water indicated in the table. Add in a fine stream, with stirring, the aliquot of the sample solution indicated in the same table. Stir for several minutes and then allow to stand for about 15 minutes. Filter the supernatant liquid with the aid of a filter pump onto a Buchner funnel, using a double thickness of coarse grained filter paper.

Transfer the precipitate to the Buchner funnel using small portions of the filtered solution to assist the transfer; then wash both the beaker and the precipitate with small portions of the wash solution (4.6) until the washings are acid-free. About 150 ml of wash solution should be used.

To check that the precipitate is acid-free, add to 5 ml of the washings a drop of the phenolphthalein solution (4.5) and a drop of the sodium hydroxide solution (4.4); freedom from acidity is shown by a pink colouration.

6.4 Titration

Transfer the precipitate and the filter paper to the beaker previously used for the separation of the sulphate ions and wash the funnel with water. Disperse the precipitate in approximately 100 ml of water by means of a glass stirrer.

Add 100 ml of water and heat to a temperature of about 70 °C; stir during the operation. Add a few drops of the phenolphthalein solution (4.5) and titrate with the sodium hydroxide solution (4.4). As soon as a permanent pink colour appears, bring to the boil; if the colour disappears, carry on the titration with the sodium hydroxide solution (4.4) until the pink colour reappears. Check that the solution is not decolourized by boiling for 2 or 3 minutes.

7. EXPRESSION OF RESULTS

Sulphate content, expressed as sulphuric acid (H_2SO_4), 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 sodium hydroxide solution (4.4) used for the titration,
- A* is the mass, in grammes, of sulphuric acid corresponding to 1 ml of 0.1 N sodium hydroxide solution
(theoretical value : 1 ml $\hat{=}$ 0.0049 g of H_2SO_4 – see Note below),
- D* is the ratio of the volume of the sample solution (6.2) to the aliquot taken for the determination (6.3),
- E* is the mass, in grammes, of the test portion.

NOTE. – If the sodium hydroxide standard volumetric solution (4.4) 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.

Separation of sulphate ions and determination by the general method.

8.2 **Reagents.** See section 4.

8.3 **Apparatus.** See section 5.

8.4 Procedure

8.4.1 *Test portion.* See clause 6.1.

8.4.2 *Preparation of sample solution.* Place the test portion (8.4.1) in an approximately 250 ml flask, add 10 ml of the hydrochloric acid solution (4.2) and boil for about 20 minutes.

After cooling, add 100 ml of water and 10 ml of the hydrochloric acid solution (4.2). Boil under a reflux condenser for 1 hour. Cool and transfer quantitatively to a 200 ml one-mark volumetric flask. Dilute to the mark and mix thoroughly.

8.4.3 *Separation of SO₄²⁻ ions.* See clause 6.3.

8.4.4 *Titration.* See clause 6.4.

8.5 **Expression of results.** See section 7.

9. TEST REPORT

Give 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 ISO Recommendation or regarded as optional.
