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Phosphoric acid for industrial use — Determination of sulphate content — Method by reduction and titrimetry

Acide phosphorique à usage industriel — Dosage des sulfates — Méthode par réduction et titrimétrie

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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 2997 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in December 1972.

It has been approved by the Member Bodies of the following countries :

Australia	India	Spain
Belgium	Israel	Sweden
Brazil	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
France	New Zealand	Turkey
Germany	Poland	United Kingdom
Hungary	South Africa, Rep. of	Yugoslavia

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

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Austria
Bulgaria

Phosphoric acid for industrial use – Determination of sulphate content – Method by reduction and titrimetry

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method, by reduction and titrimetry, for the determination of the sulphate content of phosphoric (orthophosphoric) acid for industrial use and is applicable to sulphate contents, expressed as SO_4 , equal to or greater than 0,000 5 % (m/m).

2 PRINCIPLE

Reduction of the sulphate to sulphide by a mixture of hydriodic acid and hypophosphorous acid.

Absorption of the hydrogen sulphide evolved in a mixture of acetone and sodium hydroxide solution. Titration with a standard volumetric solution of mercury(II) acetate in the presence of dithizone as indicator.

3 REAGENTS

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

3.1 Hydriodic acid, ρ approximately 1,71 g/ml, about 57 % (m/m) solution.

3.2 Hypophosphorous acid [$\text{H}(\text{H}_2\text{PO}_2)$], ρ approximately 1,21 g/ml, about 50 % (m/m) solution.

3.3 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (m/m) solution.

3.4 Reduction solution.

Into a 1 000 ml flask, fitted with a ground glass socket capable of accepting a reflux condenser, introduce, in the following order, under a current of nitrogen (3.10), 100 ml of the hydriodic acid solution (3.1), 25 ml of the hypophosphorous acid solution (3.2) and 100 ml of the hydrochloric acid solution (3.3). Fit the reflux condenser to the flask and, while bubbling a gentle current of nitrogen through the mixture, boil under reflux for 4 h.

Then cool to room temperature, maintaining the current of nitrogen.

Store the solution in a dark glass flask, previously purged with nitrogen, fitted with a ground glass stopper.

3.5 Sodium sulphate, 0,001 M standard reference solution.

Weigh, to the nearest 0,000 1 g, 0,142 0 g of anhydrous sodium sulphate, previously dried at 110 °C and cooled in a desiccator. Introduce into a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

1 ml of this solution corresponds to 96 μg of SO_4 .

3.6 Mercury(II) acetate, 0,001 M standard volumetric solution.

Weigh, to the nearest 0,000 1 g, 0,318 7 g of mercury(II) acetate [$\text{Hg}(\text{CH}_3\text{COO})_2$]. Introduce into a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

Alternatively, 0,001 M standard volumetric mercury(II) nitrate solution may be used.

3.6.1 Mercury(II) nitrate, 0,001 M standard volumetric solution.

Weigh $10,85 \pm 0,01$ g of mercury(II) oxide (HgO), place in a beaker of suitable capacity (for example 100 ml) and dissolve in 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (m/m) solution.

Dilute the solution, transfer it quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. (The solution thus obtained is 0,05 M, corresponding to 0,1 N.)

Take 20,00 ml of this solution, introduce into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this final solution just before use.

NOTES

1 In most laboratories, an exactly 0,1 N standard volumetric mercury(II) nitrate solution (corresponding to a 0,05 M solution) will be available, this solution being commonly used in the mercurimetric determination of chlorides.

2 The strengths of solutions 3.6 and 3.6.1 prepared as described above are sufficiently exact, taking into consideration the small quantities of sulphates to be determined. Standardization is therefore unnecessary.

3.7 Acetone.

3.8 Dithizone, 0,5 g/l solution in acetone.

3.9 Sodium hydroxide, approximately N solution.

3.10 Nitrogen.