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# INTERNATIONAL STANDARD



# 2997

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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  $\text{SO}_4$ , 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**,  $\rho$  approximately 1,71 g/ml, about 57 % (*m/m*) solution.

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

**3.3 Hydrochloric acid**,  $\rho$  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  $\mu\text{g}$  of  $\text{SO}_4$ .

**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,  $\rho$  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.**

## 4 APPARATUS

Ordinary laboratory apparatus and

**4.1 Microburette**, graduated in 0,01 ml.

**4.2 Apparatus for reduction and distillation** (see figure for a typical example), of which all the components are fitted together by means of ground glass joints.

## 5 PROCEDURE

### 5.1 Test portion

Weigh, to the nearest 0,000 1 g, not more than 10 g of the test sample containing between 50 and 200 µg of SO<sub>4</sub>.

NOTE — The test portion shall not contain more than 2 ml of water.

### 5.2 Check test

The purpose of this test is to check the gas-tightness and functioning of the apparatus (reduction of sulphur compounds and quantitative recovery of the hydrogen sulphide liberated).

Introduce 5 ml of the sodium hydroxide solution (3.9), 5 ml of the acetone (3.7) and 0,1 ml of the dithizone solution (3.8) into the test tube of the apparatus (4.2).

Mix and add, drop by drop, the mercury(II) acetate solution (3.6) until the colour changes from yellow to pink.

Introduce 2,00 ml of the standard reference sodium sulphate solution (3.5) into the clean and dry reduction flask. Fit the components of the apparatus (4.2) together as indicated in the figure, taking care to smear the ground joints lightly with a silicone grease and to tighten them with suitable spring-clips in order to ensure perfect gas-tightness. Pass a gentle current of nitrogen (3.10) through the inlet tube (2 to 3 bubbles per second). Then run 15 ml of the reduction solution (3.4) into the reduction flask through the dropping funnel, under a slight pressure of nitrogen.

Maintaining the current of nitrogen through the inlet tube, immerse the reduction flask partially (about 70 mm) in a boiling water bath for at least 30 min. Then allow the nitrogen to flow through the dropping funnel for 5 min.

The hydrogen sulphide liberated by the reaction is absorbed in the solution contained in the receiver, which becomes yellow.

NOTE — Development of a blue-green coloration indicates that a large quantity of hydrochloric acid has been entrained and the test must be repeated.

Disconnect the apparatus (4.2) and wash the connection tube with several millilitres of a mixture of equal volumes of water and the acetone (3.7), collecting the washings in the test tube. Titrate the solution obtained with the mercury(II) acetate solution (3.6) contained in the microburette (4.1) until a pink coloration is obtained.

The result of the check test is considered satisfactory if the volume of the standard volumetric mercury(II) acetate solution (3.6) used for the titration is between 1,90 and 2,10 ml, after subtraction of the blank test result (5.4). Otherwise, check the apparatus for leaks.

### 5.3 Determination

Place the test portion (5.1) in the reduction flask of the apparatus. Connect the different parts of the apparatus (4.2) to the reduction flask containing the test portion (5.1). Introduce 5 ml of the sodium hydroxide solution (3.9), 5 ml of the acetone (3.7) and 0,1 ml of the dithizone solution (3.8) into the test tube. Mix and add, drop by drop, the standard volumetric mercury(II) acetate solution (3.6) until the colour changes from yellow to pink.

Pass a current of nitrogen through the apparatus and run in, through the dropping funnel, 15 ml of the reduction solution (3.4) under a slight pressure of nitrogen.

Continue as described in 5.2 for the check test.

### 5.4 Blank test

Carry out a blank test under the same conditions as the determination (5.3) but using 2 ml of water instead of the test portion (5.1).

## 6 EXPRESSION OF RESULTS

The sulphate content, expressed as SO<sub>4</sub>, is given, as a percentage by mass, by the formula

$$\frac{(V_1 - V_0) \times 96 \times 100}{1\ 000\ 000\ m_0} = \frac{(V_1 - V_0) \times 0,009\ 6}{m_0}$$

where

$V_1$  is the volume, in millilitres, of the standard volumetric mercury(II) acetate solution (3.6) used for the determination;

$V_0$  is the volume, in millilitres, of the standard volumetric mercury(II) acetate solution (3.6) used for the blank test;

$m_0$  is the mass, in grams, of the test portion (5.1);

96 is the mass, in micrograms, of SO<sub>4</sub> corresponding to 1 ml of the standard volumetric mercury(II) acetate solution (3.6).

## 7 TEST REPORT

The test report shall include the following particulars :

- the reference of the method used;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard, or regarded as optional.

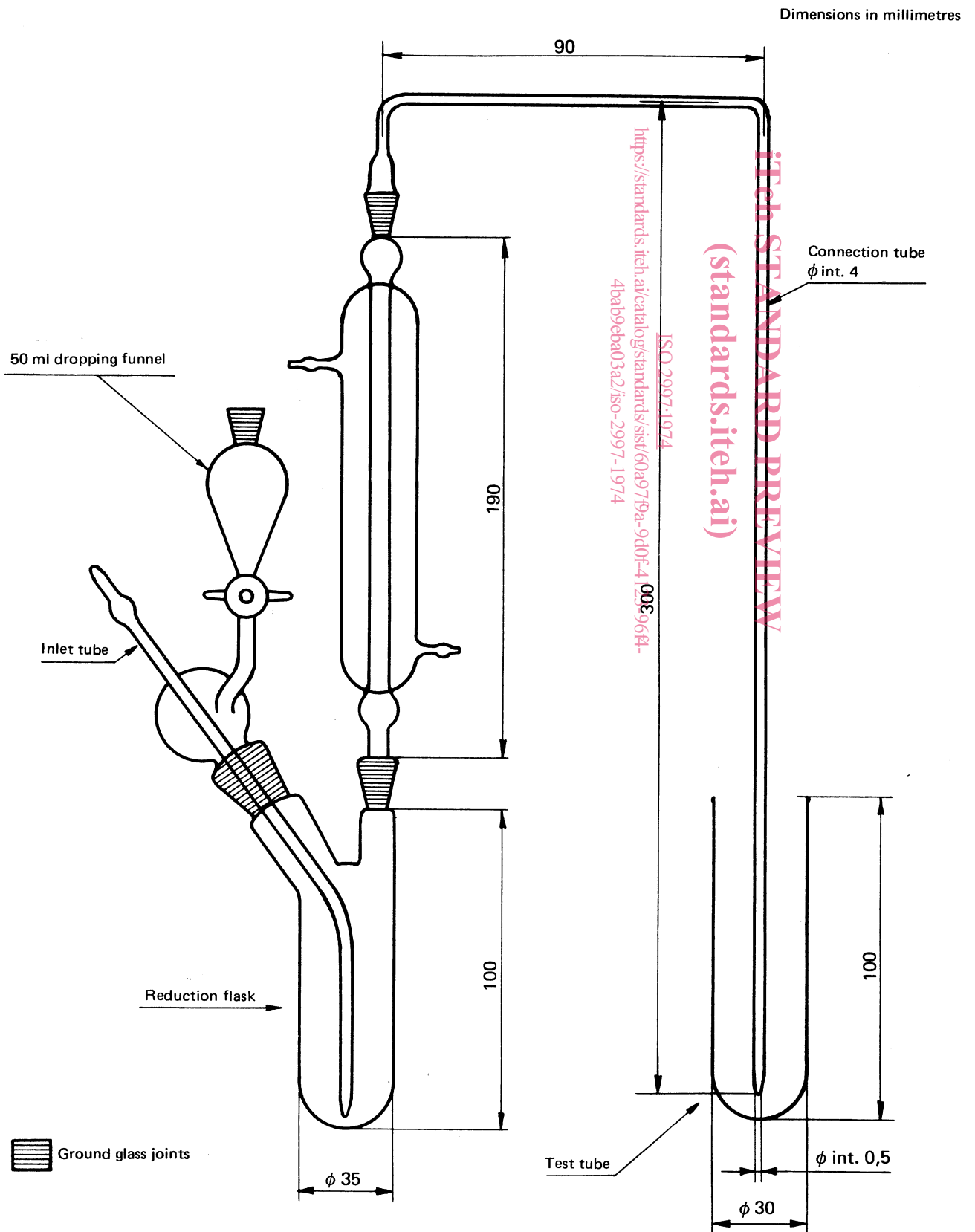


FIGURE – Typical apparatus for reduction and distillation

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