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Phosphoric acid for industrial use — Determination of higher soluble sulfate contents — Titrimetric method after reduction

Acide phosphorique à usage industriel — Dosage des sulfates solubles à teneur élevée — Méthode titrimétrique après réduction

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Foreword

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INTERNATIONAL STANDARD **ISO 7058-1983 (E)**

Phosphoric acid for industrial use — Determination of higher soluble sulfate contents — Titrimetric method after reduction

1 Scope and field of application

This International Standard specifies a method, by titrimetry after reduction, of the soluble sulfate content of technical phosphoric acid for industrial use, for example wet-process acid.

The method is applicable to products having sulfate contents, expressed as SO_4^{2-}, between 0,2 and 6 % (*m*/*m*).

NOTE — Any sulfides and any compounds which will be reduced to sulfide by the reduction procedure specified, will be included in the result.

2 References

ISO 4285, *Phosphoric acid for industrial use — Guide to sampling techniques.*

ISO 6228, *Chemical products for industrial use — General method for determination of traces of sulfur compounds, as sulfates, by reduction and titrimetry.*

3 Principle

Reduction of the sulfate present in a test portion to sulfide by a mixture of hydriodic and phosphinic (hyposphorous) acids. Absorption of the hydrogen sulfide evolved in an excess of cadmium acetate solution. Oxidation of the cadmium sulfide formed by an excess of standard volumetric potassium iodate/iodide solution, and titration of the excess iodine with standard volumetric sodium thiosulfate solution, using starch as indicator.

4 Reagents

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

4.1 Nitrogen, free from oxygen, in a cylinder provided with a pressure-release valve.

4.2 Hydrochloric acid, $c(HCl) \approx 4$ mol/l.

4.3 Hydriodic acid-phosphinic acid ($HI + H_3PO_2$), reducing solution.

See ISO 6228, reagent 4.4.

4.4 Cadmium acetate, ammoniacal solution.

Weigh, to the nearest 0,01 g, 16,4 g of cadmium acetate dihydrate [$Cd(CH_3COO)_2 \cdot 2H_2O$], place in a 1 000 ml one-mark volumetric flask and dissolve in 100 to 200 ml of water. Add 600 ml of ammonia solution, ϱ 0,88 g/ml, dilute to the mark and mix.

4.5 Potassium iodate/iodide, standard volumetric solution, $c(1/6\ KIO_3) = 0,1$ mol/l.

Weigh, to the nearest 0,000 1 g, 3,567 g of potassium iodate, previously dried at 120 °C and allowed to cool in a desiccator, and place in a 1 000 ml one-mark volumetric flask. Add approximately 40 g of iodate-free potassium iodide and 5 ml of a 40 g/l sodium hydroxide solution. Dissolve in water, dilute to the mark and mix.

4.6 Sodium thiosulfate, standard volumetric solution, $c(Na_2S_2O_3 \cdot 5H_2O) = 0,1$ mol/l.

4.6.1 Preparation

Dissolve 25 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3.5H_2O$) in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, add 1 ml of chloroform, dilute to the mark with water and mix.

4.6.2 Standardization

To 100 ml of water contained in a 250 ml conical flask provided with a ground glass stopper, add 10,0 ml of the potassium iodate/iodide standard volumetric solution (4.5) and 40 ml of the hydrochloric acid (4.2).

Stopper the flask immediately and mix the solution. Using the burette (5.1), titrate with the sodium thiosulfate solution (4.6.1) to a pale straw colour.

Add approximately 1 ml of the starch solution (4.7) and continue the titration until the blue colour is just discharged.

4.6.3 Calculation of the concentration

The concentration *c*, expressed in moles per litre, of the sodium thiosulfate solution is given by the formula

$$\frac{0,1 \times 10}{V} = \frac{1}{V}$$

where

V is the volume, in millilitres, of the sodium thiosulfate solution used for the titration;

0,1 is the concentration, in moles per litre, of the potassium iodate/iodide solution (4.5);

10 is the volume, in millilitres, of the potassium iodate/iodide solution (4.5) taken for the standardization (4.6.2).

4.7 Starch, indicator solution.

Triturate 1,0 g of soluble starch with 5 ml of water and, whilst stirring, pour the mixture into 100 ml of boiling water. Boil for a few minutes and cool.

Discard the solution after 2 weeks.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Burette, of capacity 25 ml, graduated in 0,1 ml.

5.2 Apparatus for reduction and entrainement (see figure 1 of ISO 6228 for a typical example), the receiving flask being replaced by a conical flask of capacity 250 ml, provided with a ground glass stopper.

6 Sampling

Sampling shall be carried out in accordance with the requirements of ISO 4285.

7 Procedure

7.1 Test portion

If the determination of the content of sulfates in solution is required, remove the suspended solids from the laboratory sample (see clause 6) by filtering either through a suitable filter paper or through a sintered glass filter of porosity grade P 4 (pore size index 1,6 to 4 µm), or by centrifuging at approximately 20 000 min^{-1} and decanting.

If the determination of the total sulfates content is required, do not remove the suspended solids, and follow the indications given in the appropriate clauses of ISO 4285.

Weigh by difference, to the nearest 0,001 g, a test portion of not more than 1 ml and containing not more than 90 mg of sulfate. Transfer it quantitatively to the reduction flask of the apparatus (5.2).

7.2 Determination

Assemble the apparatus, immersing the reduction flask in a boiling water bath to a depth of at least 70 mm.

Introduce 100 ml of water and 20 ml of the cadmium acetate solution (4.4) into the 250 ml conical flask (see 5.2) and place it in position so that the connecting tube dips into the cadmium acetate solution to receive the hydrogen sulfide from the reduction flask.

When the water in the bath is again boiling, add 10 ml of the reduction solution (4.3) to the reduction flask and pass a stream of the nitrogen (4.1) through the apparatus to sweep the hydrogen sulfide into the cadmium acetate solution.

Adjust the flow to obtain an almost continuous flow of 3 to 5 bubbles per second in the flask. Maintaining the nitrogen flow, heat for 25 min, after the hydrogen sulfide begins to be absorbed in the flask (as shown by the solution turning yellow).

Then pass a stream of the nitrogen through the dropping funnel for about 5 min.

Dismantle the apparatus and wash the connecting tube, inside and out, with a few millilitres of water, collecting the washings in the conical flask.

For acids containing not more than 3 % of sulfate, add 10,0 ml of the standard volumetric iodate/iodide solution (4.5) to the flask. For acids containing more than 3 % of sulfate, add 20,0 ml of the iodate/iodide solution. Add quickly 40 ml of the hydrochloric acid (4.2). Stopper the flask immediately and shake it for 1 min.

Titrate the excess of iodine with the standard volumetric sodium thiosulfate solution (4.6), using the burette (5.1).

8 Expression of results

The sulfate content, expressed as a percentage by mass as SO_4, is given by the formula

$$\frac{(V_1 \times 0{,}1 - V_2 \times c) \times 100 \times 0{,}048}{m_0}$$

where

V_1 is the volume, in millilitres, of the standard volumetric potassium iodate/iodide solution (4.5) added;

V_2 is the volume, in millilitres, of the standard volumetric sodium thiosulfate solution (4.6) used for the titration;

c is the concentration, in moles per litre, of the sodium thiosulfate solution (4.6);

m_0 is the mass, in grams, of the test portion (7.1);

0,1 is the concentration, in moles per litre, of the potassium iodate/iodide solution (4.5);

0,048 is the mass, in grams, of SO_4 corresponding to 1,00 ml of sodium thiosulfate solution, $c(Na_2S_2O_3.5H_2O) = 1\,000$ mol/l.

NOTE — If the concentration of the standard volumetric potassium iodate/iodide solution is not exactly as specified in the list of reagents, an appropriate correction should be made.

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 unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

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 sulfate 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.

ISO 7058 — Determination of higher soluble sulfate contents — Titrimetric method after reduction.

ISO 7099 — Determination of hydrogen sulfide content — Titrimetric method.*

ISO 7100 — Determination of vanadium content — Phosphotungsten vanadate spectrometric method.*

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[1] LUKE, C. L., *Ind. Eng. Chem. Anal.*, **15**, 1943.

[2] ARCHER, E. E., *Analyst*, **81**, 1956.

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