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Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Determination of orthophosphate content — Photometric method using the reduced molybdophosphate

Tripolyphosphate et pyrophosphate de sodium à usage industriel — Dosage de l'orthophosphate — Méthode photométrique au molybdophosphate réduit

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 2998 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
Austria
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Switzerland
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Turkey
United Kingdom
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Yugoslavia

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

No Member Body expressed disapproval of the document.

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Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Determination of orthophosphate content — Photometric method using the reduced molybdophosphate

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method, using the reduced molybdophosphate, for the determination of orthophosphate content of sodium tripolyphosphate (pentasodium triphosphate) and sodium pyrophosphate (tetrasodium pyrophosphate) for industrial use. The method is applicable to products with orthophosphate contents, expressed as P_2O_5, between 0,05 % and 1,0 % (*m/m*).

2 PRINCIPLE

Separation of orthophosphate in the form of ammonium molybdophosphate and immediate absorption in a mixture of 2-methylpropan-1-ol and toluene so as to suppress possible error due to the hydrolysis of phosphates other than orthophosphate.

Reduction in an ethanolic sulphuric acid medium of the molybdophosphate contained in the organic phase by a solution of tin(II) chloride and photometric measurement of the blue complex at a wavelength of about 630 nm.

3 REAGENTS

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

3.1 Sulphuric acid, approximately 0,7 N ethanolic solution.

Add, with care, 20 ml of concentrated sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, to about 980 ml of 99,5 % (*V/V*) ethanol.

Store this solution in a dark coloured stoppered flask.

3.2 Sulphuric acid, approximately 10 N solution.

3.3 Ammonium molybdate, 50 g/l acid solution.

Dissolve 50 g of ammonium molybdate tetrahydrate $[(NH_4)_6Mo_7O_{24}.4H_2O]$ in approximately 400 ml of the sulphuric acid solution (3.2), dilute to 1 000 ml with the same sulphuric acid solution (3.2) and mix.

3.4 Tin(II) chloride, 2 g/l solution in a mixture of glycerol and ethanol.

Dissolve 0,2 g of tin(II) chloride dihydrate ($SnCl_2.2H_2O$) in 100 ml of a mixture of equal volumes of glycerol and 99,5 % (*V/V*) ethanol.

This solution should not be kept for more than 1 week.

3.5 Solvent mixture

Mix equal volumes of 2-methylpropan-1-ol (*iso*butanol) and toluene.

3.6 Standard phosphorus(V) oxide solution corresponding to 1,00 g of P_2O_5 per litre.

Weigh, to the nearest 0,001 g, 1,917 g of potassium dihydrogenphosphate (KH_2PO_4), previously dried at 110 °C and cooled in a desiccator. Dissolve in water and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

1 ml of this standard solution contains 1,00 mg of P_2O_5.

3.7 Standard phosphorus(V) oxide solution corresponding to 0,010 g of P_2O_5 per litre.

Place 10,0 ml of the standard phosphorus(V) oxide solution (3.6) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,010 mg of P_2O_5.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Spectrophotometer, or

4.2 Photoelectric absorptiometer

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,000 1 g, 0,500 g of the test sample.

5.2 Blank test

Carry out a blank test at the same time as the determination and under the same conditions, using the same quantities of all the reagents used for the determination.

5.3 Preparation of the test solution

Place the test portion (5.1) in a 500 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

5.4 Preparation of the calibration curve

5.4.1 *Preparation of standard matching solutions*

Into a series of eight 100 ml test tubes with ground glass stoppers, introduce the volumes of the standard phosphorus(V) oxide solution (3.7) indicated in the following table :

Standard phosphorus(V) oxide solution (3.7)	Corresponding mass of P_2O_5	Path length of photometric cells[2]
ml	mg	cm
0[1]	0,000	1 and 5
1,0	0,010	5
3,0	0,030	5
5,0	0,050	5
8,0	0,080	1 and 5
12,0	0,120	1
16,0	0,160	1
20,0	0,200	1

[1] Compensation solution.

[2] Cells with path lengths appropriate to the instrument in use may be used.

First add, to each test tube, the quantity of water necessary to bring the volume to 20 ml and then 25 ml of the solvent mixture (3.5) and 5 ml of the ammonium molybdate solution (3.3). Immediately mix by vigorous shaking for 15 s. Allow to stand, assisting the separation by gently tapping the walls of each tube.

Take 5,0 ml of the supernatant liquid (organic layer) from each tube and introduce into a series of eight 50 ml one-mark volumetric flasks. Dilute to about 45 ml with the ethanolic sulphuric acid solution (3.1). Add 1 ml of the tin(II) chloride solution (3.4) to each flask, dilute to the marks with the ethanolic sulphuric acid solution (3.1) and mix.

5.4.2 *Photometric measurements*

After allowing to stand for 10 min, carry out the photometric measurements using the spectrophotometer (4.1), at a wavelength of about 630 nm, or the photoelectric absorptiometer (4.2), fitted with appropriate filters, after having adjusted the instrument to zero absorbance against the compensation solution.

5.4.3 *Preparation of the calibration chart*

Draw two graphs corresponding to the cells used, having, for example, the numbers of milligrams of P_2O_5 contained in 50 ml of standard matching solutions as abscissae and the corresponding values of absorbance as ordinates.

5.5 Determination

5.5.1 *Colour development*

Place 25 ml of the solvent mixture (3.5) and 5 ml of the ammonium molybdate solution (3.3) into a 100 ml test tube with ground glass stopper. Add 20 ml of the test solution (5.3), which should contain not more than 0,200 mg of P_2O_5 in the form of orthophosphate, and immediately mix by vigorous shaking for 15 s. Allow to stand and assist the separation by gently tapping the wall of the test tube. Take 5,0 ml of the supernatant liquid (organic phase) and introduce into a 50 ml one-mark volumetric flask. Dilute to about 45 ml with the ethanolic sulphuric acid solution (3.1). Add 1 ml of the tin(II) chloride solution (3.4) to the flask, dilute to the mark with the ethanolic sulphuric acid solution (3.1) and mix.

5.5.2 *Photometric measurement*

Carry out the photometric measurement according to the procedure described in 5.4.2, using photometric cells of appropriate path lengths, after having adjusted the apparatus to zero absorbance against the blank test solution (5.2).

6 EXPRESSION OF RESULTS

By means of the calibration chart corresponding to the photometric cell used (see 5.4.3), determine the mass of P_2O_5 corresponding to the value of the photometric measurement on the aliquot portion of the test solution taken for the determination.

The orthophosphate content, expressed as P_2O_5, is given, as a percentage by mass, by the formula

$$m_1 \times \frac{1}{1\,000} \times \frac{500}{20} \times \frac{100}{m_0} = 2{,}5 \frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in milligrams, of P_2O_5 found in the aliquot portion of the test solution taken for the determination.

7 TEST REPORT

The test report shall include 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 International Standard, or regarded as optional.

ANNEX

This document forms one of a series on methods of test for sodium phosphates for industrial use. The complete list of ISO publications already prepared or in course of preparation is as follows :

Sodium pyrophosphate (tetrasodium diphosphate)

ISO 2999 – *Estimation of pyrophosphate content – Potentiometric method.*

Sodium tripolyphosphate (pentasodium triphosphate)

ISO/R 850 – *Determination of matter insoluble in water.*

ISO/R 851 – *Measurement of pH – Potentiometric method.*

ISO 3000 – *Estimation of tripolyphosphate content – Tris(ethylenediamine) cobalt(III) chloride gravimetric method.*

Sodium pyrophosphate and sodium tripolyphosphate (tetrasodium diphosphate and pentasodium triphosphate)

ISO/R 852 – *Determination of iron content – 2,2'-Bipyridyl spectrophotometric method.*

ISO/R 853 – *Determination of loss on ignition.*

ISO 2996 – *Determination of particle size distribution by mechanical sieving.*

ISO 2998 – *Determination of orthophosphate content – Photometric method using the reduced molybdophosphate.*

ISO 3357 – *Determination of total phosphorus(V) oxide content – Quinoline molybdophosphate gravimetric method.*1)

ISO 3358 – *Separation by column chromatography and determination of the different phosphate forms.*1)

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1) At present at the stage of draft.