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Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

Tripolyphosphate et pyrophosphate de sodium à usage industriel — Dosage de l'oxyde de phosphore(V) total — Méthode gravimétrique au phosphomolybdate de quinoléine

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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.

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

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Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a gravimetric method using quinoline phosphomolybdate for the determination of the total phosphorus(V) oxide content of sodium tripolyphosphate (pentasodium triphosphate) and sodium pyrophosphate (tetrasodium diphosphate) for industrial use.

2 PRINCIPLE

Hydrolysis of a test portion by prolonged boiling in the presence of hydrochloric acid.

Precipitation of phosphates in the form of quinoline phosphomolybdate in the presence of acetone. Filtration, washing, drying and weighing of the precipitate.

3 REAGENTS

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

3.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) or approximately 12 N solution.

3.2 Citromolybdate reagent.

3.2.1 Dissolve 70 g of sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) in 150 ml of water.

3.2.2 Dissolve 60 g of citric acid monohydrate ($C_6H_8O_7.H_2O$) in 150 ml of water and add 85 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) or approximately 14 N solution.

3.2.3 Add, with mixing, solution 3.2.1 to solution 3.2.2.

3.2.4 Add 35 ml of nitric acid solution, ρ approximately 1,40 g/ml, about 68 % (*m/m*) or approximately 14 N solution, followed by 5 ml of pure quinoline, recently distilled, to 100 ml of water.

3.2.5 Pour solution 3.2.4 into solution 3.2.3 and mix. Allow to stand for at least 12 h (for example, overnight) and filter through a sintered glass filter of porosity grade P 10 (pore size index 4 to 10 µm).

Store this solution protected from light in a well-stoppered flask.

3.2.6 Add 280 ml of acetone to solution 3.2.5 and dilute to 1 000 ml with water.

Do not keep this solution for more than 1 week. Store under the same conditions as solution 3.2.5.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Filter crucible, with sintered glass disk, porosity grade P 10 (pore size index 4 to 10 µm).

4.2 Electric oven, capable of being controlled at 250 ± 10 °C.

5 PROCEDURE

WARNING — **Acetone is very flammable and must be handled only inside a well-ventilated fume cupboard, in the absence of flames.**

5.1 Test portion

Weigh, to the nearest 0,000 2 g, 1 g of the test sample, taking care to avoid any gain or loss of moisture.

5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same quantities of all the reagents as used in the determination.

5.3 Determination

5.3.1 *Preparation of the test solution*

Place the test portion (5.1) in a 250 ml flask with ground neck, dissolve in about 50 ml of water, add 20 ml of the hydrochloric acid solution (3.1), fit a reflux condenser on the flask and boil for at least 20 min. Cool, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use and filter if necessary.

NOTE — If hydrochloric acid of the concentration indicated in the list of reagents is not available, solutions of lower concentration may be used, by adding the corresponding stoichiometric quantity of HCl.

5.3.2 *Precipitation and filtration*

Take 25,0 ml of the test solution (5.3.1) and place in a 400 ml beaker. Dilute to 100 ml and add 100 ml of the citromolybdate reagent (3.2).

Cover the beaker with a clock glass, warm inside a fume cupboard, on a hot-plate, until its contents reach 75 ± 5 °C and maintain at this temperature for about 30 s. (Do not use a flame and do not mix, either during the addition of the reagent or during the heating, so as to avoid the formation of clots.) Allow to cool to ambient temperature, stirring three or four times with a glass rod during cooling.

Heat the filter crucible (4.1) in the oven (4.2), controlled at 250 ± 10 °C, for 15 min, allow to cool in a desiccator containing silica gel in good condition and weigh to the nearest 0,000 1 g. Decant the liquid through the crucible and wash the precipitate six times, by decantation, using about 30 ml of water each time. Transfer all the precipitate to the filter crucible by means of a jet of water from a wash bottle. Then wash the precipitate four times, removing each portion of wash water with the aid of a vacuum pump.

5.3.3 *Drying and weighing*

Place the filter crucible in the oven (4.2), controlled at 250 ± 10 °C, for 15 min starting from the stabilization of temperature. Allow to cool for not more than 30 min in the desiccator containing silica gel in good condition and weigh to the nearest 0,000 1 g.

6 EXPRESSION OF RESULTS

The total phosphorus(V) oxide (P_2O_5) content, expressed as a percentage by mass, is given by the formula :

$$\frac{0{,}032\,07 \times \frac{1\,000}{25} \times 100\,(m_1 - m_2)}{m_0} = \frac{128{,}28 \times (m_1 - m_2)}{m_0}$$

where

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

m_1 is the mass, in grams, of precipitate obtained during the determination (5.3.3);

m_2 is the mass, in grams, of precipitate obtained during the blank test (5.2);

$\frac{1\,000}{25}$ is the ratio between the volume of the test solution and the aliquot portion taken for the determination;

0,032 07 is the factor for conversion of quinoline phosphomolybdate to phosphorus(V) oxide.

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

ISO PUBLICATIONS RELATING TO CONDENSED SODIUM PHOSPHATES FOR INDUSTRIAL USE

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 phosphomolybdate gravimetric method.*

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.