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Sodium tripolyphosphate for industrial use — Estimation of tripolyphosphate content — Tris(ethylenediamine) cobalt(III) chloride gravimetric method

Tripolyphosphate de sodium à usage industriel — Évaluation de la teneur en tripolyphosphate — Méthode gravimétrique au chlorure de tris(éthylènediamine) cobalt(III)

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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 3000 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 :

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

The Member Body of the following country expressed disapproval of the document on technical grounds :

Austria

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INTERNATIONAL STANDARD ISO 3000-1974 (E)

Sodium tripolyphosphate for industrial use — Estimation of tripolyphosphate content — Tris(ethylenediamine) cobalt(III) chloride gravimetric method

1 SCOPE

This International Standard specifies a gravimetric method, using tris(ethylenediamine) cobalt(III) chloride, for the estimation of the tripolyphosphate content of sodium tripolyphosphate (*penta*sodium triphosphate) for industrial use.

2 FIELD OF APPLICATION

The method is not applicable in the presence of metaphosphate, but the presence of pyrophosphate at normal concentrations in commercial products (maximum of 6 %) does not interfere.

3 PRINCIPLE

Precipitation of tripolyphosphate as its complex with tris(ethylenediamine) cobalt(III) chloride at a pH of between 3 and 4.

Filtration, washing, drying and weighing of the precipitate.

4 REAGENTS

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

4.1 Hydrochloric acid, approximately 0,5 N solution.

4.2 Acetate buffer solution.

Prepare a solution containing 110,4 ± 0,5 g of glacial acetic acid, ρ approximately 1,05 g/ml, about 17,4 N solution, and 12,3 ± 0,1 g of sodium acetate trihydrate per litre.

The pH of the solution should be 3,6 ± 0,1. If it differs from this value, correct the pH by the addition of one or other of the components.

4.3 Tris(ethylenediamine) cobalt(III) chloride $[Co(NH_2{-}CH_2{-}CH_2{-}NH_2)_3]Cl_3$, 40 g/l solution.

4.3.1 *Preparation of the salt*

Weigh 260 ± 0,5 g of ethylenediamine monohydrate ($NH_2CH_2CH_2NH_2.H_2O$). Place in a 1 000 ml beaker, dissolve and partially neutralize by adding a solution comprising 535 ml of water and 85 ml of a hydrochloric acid solution, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution.

This is solution A.

Prepare a solution of 250 ± 0,5 g of cobalt(II) chloride hexahydrate ($CoCl_2.6H_2O$) in 750 ml of water in a 2 000 ml conical flask.

This is solution B.

Pour solution A into solution B with vigorous stirring.

Oxidize the cobalt by passing a current of air into the solution through a tube terminating in a fritted disc of porosity P90 (pore size index 40-90 μm). Continue to oxidize for 8 h.

Pour the solution into a 2 000 ml beaker and evaporate on a boiling water bath, passing a current of air over the surface of the liquid. Stop the evaporation when crystals form on the surface of the liquid. Cool the solution. Add 150 ml of hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution, stir mechanically and heat until completely dissolved. Continuing the stirring, re-precipitate the product by adding, very slowly, 300 ml of 95 % (*V/V*) ethanol. This addition should take about 1 h.

Cool the solution and filter through a sintered glass disc. Wash the crystals four times with the 95 % (*V/V*) ethanol. Purify the product by dissolving the crystals in 200 ml of water in a 1 000 ml beaker. Stir mechanically and heat to facilitate dissolution.

Filter the warm solution through a Buchner funnel of about 55 mm diameter, using a fine grade filter paper, collecting the filtrate in a 1 000 ml filtration flask. Wash the beaker and the Buchner funnel with about 100 ml of boiling water. Return the filtrate to the same beaker, mix and heat on a boiling water bath for 30 min.

Continuing the stirring, add, very slowly, over a period of 1 h, 300 ml of 95 % (*V/V*) ethanol. Cool and filter through a sintered glass disc. Wash the product four times with the 95 % (*V/V*) ethanol. Spread the crystals in thin layers in dishes and allow to dry in air for at least 12 h. Finally dry for several hours in the oven (5.4) controlled at 105 ± 5 °C.

By using the indicated quantities, about 300 g of dry crystals are obtained. These crystals are hygroscopic. They shall be stored in air-tight flasks and it is recommended that the portion used for preparing the solution (4.3.2) should be dried just before use.

4.3.2 *Preparation of the solution*

Weigh 4 ± 0,01 g of the salt (4.3.1), carefully dried in the oven (5.4) controlled at 105 ± 5 °C. Dissolve in water, dilute to 100 ml and mix.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Filter crucible, of about 30 ml capacity, with a sintered glass disc of 30 mm diameter and of porosity P40 (pore size index 16-40 μm).

5.2 Mechanical stirrer, consisting of a bent glass rod or other stirrer which allows solids to be washed off with a minimum of water.

5.3 pH meter, with glass electrode.

5.4 Oven, capable of being controlled at 105 ± 5 °C.

6 PROCEDURE

6.1 Test portion and preparation of the test solution

Weigh, to the nearest 0,001 g, 7 g of the test sample, place in a 1 000 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

6.2 Determination

If the solution (6.1) is cloudy, filter it through a dry filter paper, collecting the filtrate in a dry flask. Place 50,0 ml of the clear solution in a 250 ml beaker. Add the hydrochloric acid solution (4.1) until the pH reaches 3,6, checking with the pH meter (5.3), and then add 10 ml of the acetate buffer solution (4.2).

Bring the temperature to about 25 °C, place the mechanical stirrer (5.2) in the beaker and start the stirrer.

Run in the reagent solution (4.3) from a burette in 1 ml portions, until 10 ml in all have been added, stirring for 1 min after each addition. Stir for 15 min after adding the portion of the reagent solution (4.3) which gives the first precipitation. This usually occurs after the third or fourth addition. After stirring for 15 min, continue to run in the reagent solution (4.3), in portions of 1 ml, stirring for 1 min after each addition. After having added all 10 ml of the reagent solution (4.3), stir for a further 15 min.

Withdraw the stirrer from the beaker, taking care to recover all the precipitate sticking to it. This can be carried out by rubbing the stirrer with a glass rod fitted with a rubber policeman, keeping the end of the stirrer immersed in the liquid contained in the beaker. Remove the last crystals by means of a water jet (5 to 10 ml of water). Allow to stand for 1 h.

Filter, under vacuum, on the filter crucible (5.1), previously dried and weighed to the nearest 0,5 mg. To remove the precipitate remaining in the beaker, use the filtrate as many times as is necessary. For convenience, it is recommended that the filtrate be returned to a wash-bottle and that the precipitate sticking to the walls of the beaker be removed by means of a glass rod fitted with a rubber policeman.

When all the precipitate has been transferred to the filter, dry it under vacuum. Wash three times with 5 to 10 ml of water and suck dry after each such washing. The total volume of filtrate should not exceed 100 ml. Dry as far as possible in air, by aspiration.

Finally dry in the oven (5.4), controlled at 105 ± 5 °C, until constant mass, which generally requires 2 to 3 h. Weigh to the nearest 0,5 mg.

7 EXPRESSION OF RESULTS

The tripolyphosphate content, expressed as a percentage by mass of anhydrous sodium tripolyphosphate ($Na_5P_3O_{10}$), is given by the formula :

$$m_1 \times \frac{368}{530} \times \frac{1\,000}{50} \times \frac{100}{m_0} = \frac{1\,388{,}7\, m_1}{m_0}$$

where

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

m_1 is the mass, in grams, of the precipitate;

$\frac{368}{530}$ is the ratio between the masses of anhydrous sodium tripolyphosphate ($Na_5P_3O_{10}$) and the corresponding mass of the precipitate $[Co(NH_2-CH_2-CH_2-NH_2)_3]H_2P_3O_{10}.2H_2O$.

Calculate the result to the first decimal place.

8 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.