INTERNATIONAL STANDARD ISO 5374

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Condensed phosphates for industrial use (including foodstuffs) — Determination of chloride content — Potentiometric method

Phosphates condensés à usage industriel (y compris les industries alimentaires) — Dosage des chlorures — Méthode potentiométrique

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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 5374 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in June 1977.

It has been approved by the member bodies of the following countries :

Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Israel	Switzerland
Bulgaria	Italy	Turkey
Chile	Kenya	United Kingdom
Czechoslovakia	Korea, Rep. of	U.S.S.R.
Egypt, Arab Rep. of	Netherlands	Yugoslavia
France	Poland	
Germany, F.R.	Portugal	

No member body expressed disapproval of the document.

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

Printed in Switzerland

Condensed phosphates for industrial use (including foodstuffs) — Determination of chloride content — Potentiometric method

1 SCOPE

This International Standard specifies a potentiometric method for the determination of the chloride content of condensed phosphates for industrial use (including foodstuffs).

2 FIELD OF APPLICATION

The method is applicable to products having chloride contents, expressed as chlorine (Cl), equal to or greater than 30 mg/kg.

3 PRINCIPLE

Potentiometric titration of the chloride ions (Cl^-) with standard volumetric silver nitrate solution in a nitric acid/acetone/water medium, at a temperature below 20 °C, using a silver measurement electrode and a calomel reference electrode or a pair of silver-mercury(I) sulphate electrodes.

NOTE — A manual procedure is described, but automatic devices can obviously be used.

4 REAGENTS

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

4.1 Acetone.

4.2 Nitric acid, approximately 1,40 g/ml, about 68 % (*m*/*m*) solution.

4.3 Silver nitrate, approximately 0,1 N solution.

Dissolve 8,5 g of silver nitrate in water in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Store this solution in a brown glass bottle.

4.4 Silver nitrate, approximately 0,01 N solution.

Take 50 ml of the silver nitrate solution (4.3), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

4.5 Potassium chloride, 0,1 N standard reference solution.

Weigh, to the nearest 0,000 1 g, 3,727 6 g of potassium chloride, previously dried for 1 h at about 130 °C and cooled in a desiccator, dissolve in a little water, transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

This solution shall not be kept for more than 1 month.

4.6 Potassium chloride, 0,01 N standard reference solution.

Take 50,0 ml of the standard reference potassium chloride solution (4.5), place in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

4.7 Potassium nitrate solution, saturated at ambient temperature.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Potentiometric titration apparatus, comprising

5.1.1 Potentiometer, sensitivity 2 mV, covering the range − 500 to + 500 mV.

5.1.2 Calomel electrode, fitted with a safety reservoir, filled with saturated potassium chloride solution.

5.1.3 Bridge, containing potassium nitrate solution (4.7), connected to the calomel electrode (5.1.2) and fitted with porous diaphragms at the ends.

NOTE — This bridge is not necessary if the silver-mercury(I) sulphate pair of electrodes is used.

5.1.4 Silver electrode.

5.2 Magnetic stirrer, with a polytetrafluorethylene (PTFE)-coated rod.

5.3 Microburette, with fine-pointed tip, graduated in 0,01 ml divisions.

6 PROCEDURE

6.1 Calibration of the silver nitrate solution (4.4)

6.1.1 *Titration*

Take 5,00 ml and 10,00 ml of the standard reference potassium chloride solution (4.6) and place in two low-form beakers of convenient capacity (for example 400 ml). Add to each beaker 100 ml of water, 200 ml of the acetone (4.1) and about 2 ml of the nitric acid solution (4.2). Carry out the following titration on the contents of each beaker.

Introduce the rod of the magnetic stirrer (5.2) into the beaker and place the beaker in a container of convenient capacity (for example, a basin of diameter about 20 cm) containing water and crushed ice. Place the container and beaker on the magnetic stirrer (5.2) and set the stirrer in motion.

Place a thermometer in the beaker and maintain the temperature below 20 °C during the titration by the occasional addition of crushed ice to the container.

Immerse the silver electrode (5.1.4) and the free end of the bridge (5.1.3) in the solution, connect the electrodes to the potentiometer (5.1.1) and, after having verified the zero of the apparatus, note the value of the starting potential.

Add 4 ml of the silver nitrate solution (4.4) to the beaker containing 5,00 ml of the standard reference potassium chloride solution, and add 9 ml of the silver nitrate solution to that containing 10,00 ml of the potassium chloride solution. Continue the addition of the silver nitrate solution, in 0,1 ml portions, to each beaker. After each addition, wait for the potential to become stable.

Note the volumes added and the corresponding values of the potential in the first two columns of a table.

In a third column of the table, note the successive increments ($\Delta_1 E$) of the potential E. In a fourth column, note the differences ($\Delta_2 E$), positive or negative, between the potential increments ($\Delta_1 E$).

The end of the titration corresponds to the addition of the 0,1 ml portion (V_1) of the silver nitrate solution (4.4) which gives the maximum value of $\Delta_1 E$.

In order to calculate the exact volume, V_{EQ}, of the silver nitrate solution (4.4) corresponding to the end of the reaction, use the formula

$$V_{EQ} = V_0 + V_1 \times \frac{b}{B}$$

where

V_0 is the volume, in millilitres, of the silver nitrate solution (4.4) immediately lower than the volume which gives the maximum increment of $\Delta_1 E$;

V_1 is the volume, in millilitres, of the silver nitrate solution (4.4) corresponding to the last portion added (0,1 ml);

b is the last value of $\Delta_2 E$ which is positive;

B is the sum of the absolute values of the final positive value of $\Delta_2 E$ and the first negative value of $\Delta_2 E$ (see example in annex A).

6.1.2 *Calculation of concentration of the solution*

The concentration (T) of the silver nitrate solution (4.4), expressed as a normality, is given by the formula

$$T = T_0 \times \frac{5}{V_2 - V_3}$$

where

T_0 is the strength, expressed as a normality, of the standard reference potassium chloride solution (4.6);

V_2 is the value, in millilitres, of V_{EQ} corresponding to the titration of 10 ml of the standard reference potassium chloride solution (4.6);

V_3 is the value, in millilitres, of V_{EQ} corresponding to the titration of 5 ml of the standard reference potassium chloride solution (4.6);

5 is the difference, in millilitres, between the two volumes of the standard reference potassium chloride solution (4.6) taken.

6.1.3 *Calculation of the blank test result*

The result of the blank test on the reagents, V_4, is given, in millilitres, by the formula

$$V_4 = 2V_3 - V_2$$

where V_2 and V_3 have the same meaning as in 6.1.2.

6.2 Determination

6.2.1 *Test portion*

Into a low-form beaker of convenient capacity (for example 400 ml), weigh, to the nearest 0,001 g, a mass of test sample between 1 and 10 g, such that the test portion does not contain more than 1 500 µg of chlorine.

6.2.2 *Preparation of the test solution*

Dissolve the test portion (6.2.1) in 100 ml of water. Introduce the rod of the magnetic stirrer (5.2) into the beaker and place the beaker in a container of convenient capacity (for example, a basin of diameter about 20 cm) containing water and crushed ice. Place the container and beaker on the magnetic stirrer (5.2) and set the stirrer in motion.

Place a thermometer in the beaker and maintain the temperature below 20 °C during the neutralization of the solution and the successive titration (6.2.3). Neutralize the solution to approximately pH 3 with the nitric acid solution (4.2), checking with indicator paper, and add 2 ml in excess of this acid and 200 ml of the acetone (4.1).

6.2.3 *Titration*

Immerse the silver electrode (5.1.4) in the test solution (6.2.2) and proceed as specified in the fourth paragraph of 6.1.1 and, omitting the initial addition of the silver nitrate solution (i.e. 4 ml or 9 ml), complete the titration as specified in the fifth paragraph.

7 EXPRESSION OF RESULTS

The chloride content, expressed in milligrams of chlorine (Cl) per kilogram, is given by the formula

$$\frac{354{,}5\ T\ (V_5 - V_4)}{m}$$

where

T is the concentration, expressed as a normality, of the silver nitrate solution determined according to 6.1.2;

V_4 is the result, in millilitres, of the blank test (6.1.3);

V_5 is the value, in millilitres, of V_{EQ} corresponding to the determination (6.2.3);

m is the mass, in grams, of the test portion (6.2.1);

354,5 is the mass, in micrograms, of chlorine corresponding to 1 ml of exactly 0,01 N silver nitrate solution.

8 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 regarded as optional.

ANNEX A

EXAMPLE

Volume of silver nitrate solution V	Potential E	$\Delta_1 E$	$\Delta_2 E$
ml	mV		
4,80	176		
		35	
4,90	211		+ 37
		72	
5,00	283		− 49
		23	
5,10	306		− 10
		13	
5,20	319		

$$V_{EQ} = 4{,}9 + 0{,}1 \times \frac{37}{37 + 49} = 4{,}943$$

ANNEX B

ISO PUBLICATIONS RELATING TO CONDENSED PHOSPHATES FOR INDUSTRIAL USE (INCLUDING FOODSTUFFS)

ISO 5372 — Determination of arsenic content — Silver diethyldithiocarbamate photometric method.

ISO 5373 — Determination of calcium content — Flame atomic absorption method.

ISO 5374 — Determination of chloride content — Potentiometric method.

ISO 5375 — Determination of oxides of nitrogen content — 3,4-Xylenol spectrophotometric method.

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