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Phosphoric acid for industrial use (including foodstuffs) – Determination of chloride content – Potentiometric method

Acide phosphorique à usage industriel (y compris les industries alimentaires — Dosage des chlorures — Méthode potentiométrique

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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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Phosphoric acid 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 phosphoric acid 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 0,001 % (*m/m*).

NOTE — The lower limit of applicability can be extended downwards by using weaker standard reference and volumetric solutions.

3 PRINCIPLE

Potentiometric titration of the chloride (Cl^-) ions with silver nitrate solution in a nitric acid-acetone-water medium, using a silver measurement electrode and a calomel reference electrode.

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, ρ 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 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 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.

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.

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).

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 respectively 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 20 ml of water, 200 ml of the acetone (4.1) and 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 and 9 ml respectively of the silver nitrate solution (4.4) from the microburette (5.3) and continue the addition in 0,1 ml portions. After each addition, await the stabilization of the potential.

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 strength of the solution*

The strength (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 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.

6.1.4 *Automatic titration*

The procedure described is manual but can equally be applied using an automatic titration device which enables the graphs of the following relations to be drawn directly :

$$E = f(V)$$

$$\frac{\Delta E}{\Delta V} = f(V)$$

and, for certain devices,

$$\frac{\Delta^2 E}{\Delta V^2} = f(V)$$

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 30 g, depending on the expected chloride content and such that the test portion does not contain more than 1 500 µg of chlorine.

6.2.2 *Titration*

Dilute the test portion (6.2.1) to 20 ml with water and add 200 ml of the acetone (4.1) and 2 ml of the nitric acid solution (4.2).

Continue in accordance with the instructions in 6.1, from the second paragraph.

7 EXPRESSION OF RESULTS

The chloride content, expressed as a percentage by mass of chlorine (Cl), is given by the formula

$$(V_5 - V_4) \times T \times 0{,}035\,45 \times \frac{100}{m} = \frac{3{,}545\, T\,(V_5 - V_4)}{m}$$

where

T is the strength, 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 (6.2.2);

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

0,035 45 is the mass, in grams, of chlorine corresponding to 1 ml of exactly 1 N silver nitrate solution.

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 A

EXAMPLE

Volume of silver nitrate solution (4.4) V	Potential E	Δ_1E	Δ_2E
ml	mV		
0,80	176		
		35	
0,90	211		+ 37
		72	
1,00	283		– 49
		23	
1,10	306		– 10
		13	
1,20	319		

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

ANNEX B

ISO PUBLICATIONS RELATING TO PHOSPHORIC ACID FOR INDUSTRIAL USE

ISO 847 – Determination of sulphate content – Titrimetric method.

ISO 848 – Determination of calcium content – Titrimetric method.

ISO 849 – Determination of iron content – 2,2-Bipyridyl photometric method.

ISO 2997 – Determination of sulphate 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 – Sampling technique.

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