INTERNATIONAL STANDARD 3709

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Phosphoric acid for industrial use (including foodstuffs) — Determination of oxides of nitrogen content — 3,4-Xylenol spectrophotometric method

Acide phosphorique à usage industriel (y compris les industries alimentaires) — Dosage des oxydes d'azote — Méthode spectrophotométrique au xylénol-3,4

First edition — 1976-11-15

UDC 661.634 : 543.272.32

Ref. No. ISO 3709-1976 (E)

Descriptors : chemical compounds, phosphoric acids, food industry, chemical analysis, determination of content, nitrogen oxides, spectrophotometric analysis, xylenol.

Price based on 4 pages

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.

International Standard ISO 3709 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and was circulated to the Member Bodies in February 1975.

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

Austria
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No Member Body expressed disapproval of the document.

Printed in Switzerland

Phosphoric acid for industrial use (including foodstuffs) – Determination of oxides of nitrogen content – 3,4-Xylenol spectrophotometric method

1 SCOPE

This International Standard specifies a 3,4-xylenol spectrophotometric method for the determination of the oxides of nitrogen content of phosphoric acid for industrial use (including foodstuffs).

2 FIELD OF APPLICATION

The method is applicable to products having an oxides of nitrogen content, expressed as nitrogen (N), equal to or greater than 0,2 mg/kg.

3 PRINCIPLE

Oxidation of nitrous nitrogen to nitric nitrogen by potassium permanganate.

Reaction of nitric nitrogen with 3,4-xylenol under specified conditions to form a nitrate derivative. Distillation of the nitrate derivative and absorption in sodium hydroxide solution. Spectrophotometric measurement of the yellow-coloured nitrophenol at a wavelength of about 435 nm.

4 REAGENTS

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

4.1 Mercury(II) acetate [$Hg(CH_3COO)_2$].

4.2 Sulphuric acid, approximately 80 % (*m/m*) solution, free from nitrogen oxides.

Carefully add, while stirring, 750 ml of sulphuric acid, ρ approximately 1,84 g/ml, free from nitrogen, to 250 ml of water. To ensure complete elimination of nitrogen oxides from the sulphuric acid used for the dilution, proceed as follows : cautiously add 800 ml of sulphuric acid, ρ 1,84 g/ml, to about 200 ml of water and heat until white fumes are liberated. Cool and repeat the dilution and heating twice.

4.3 3,4-Xylenol, 50 g/l solution in acetic acid.

Dissolve 5 g of 3,4-xylenol in glacial acetic acid, ρ approximately 1,05 g/ml, about 17,4 N solution, and dilute to 100 ml with the same acid.

Store this solution below 5 °C.

4.4 Potassium permanganate, approximately 0,1 N solution.

4.5 Sodium hydroxide, approximately 2 N solution.

4.6 Hydrogen peroxide, 1 g/l solution.

4.7 Potassium nitrate, standard solution corresponding to 0,500 g of nitrogen per litre.

Weigh, to the nearest 0,001 g, 3,609 g of potassium nitrate, previously dried for 2 h at 120 °C and cooled in a desiccator. Transfer to a 1 000 ml one-mark volumetric flask, dissolve in a little water, dilute to the mark and mix.

1 ml of this standard solution contains 500 μg of N.

4.8 Potassium nitrate, standard solution corresponding to 0,005 g of nitrogen per litre.

Take 10,0 ml of the standard potassium nitrate solution (4.7), transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 5 μg of N.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Weighing pipette, of capacity approximately 50 ml, with ground glass stoppers.

5.2 Water bath, capable of being controlled at 35 ± 1 °C.

5.3 Distillation apparatus, with 24/29 conical ground glass joints (see ISO 383), comprising, for example, the following parts (see figure) :

5.3.1 distillation flask, of capacity 250 ml;

5.3.2 sloping recovery bend, at 75°;

5.3.3 Liebig condenser, water circulation, effective length approximately 450 mm, connecting to the sloping recovery bend (5.3.2).

5.4 Spectrophotometer.

6 PROCEDURE

6.1 Test portion

Fill the weighing pipette (5.1) with the test sample and take a test portion of approximately 15 g, weighing by difference to the nearest 0,01 g. Place the test portion in the distillation flask (5.3.1).

6.2 Blank test

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

6.3 Preparation of the calibration graph

6.3.1 *Preparation of standard colorimetric solutions* for spectrophotometric measurements in cells of optical path length 4 or 5 cm.

Into a series of seven of the distillation flasks (5.3.1), place respectively the volumes of the standard potassium nitrate solution (4.8) shown in the following table :

Standard potassium nitrate solution (4.8)	**Corresponding mass of N**
ml	µg
0*	0
0,4	2
1,0	5
2,0	10
3,0	15
4,0	20
5,0	25

* Compensation solution.

Dilute, with water, the contents of each flask to 5 ml and treat each as follows.

Place the distillation flask (5.3.1) in a bath of water and ice, and introduce into the flask 0,200 g of the mercury(II) acetate (4.1)[1]. Then add 15 ml of the sulphuric acid solution (4.2) very slowly, in small portions and with stirring, so that the temperature never exceeds 35 °C. Add, drop by drop, the volume of the potassium permanganate solution (4.4) necessary to give a pink coloration which is stable for a few minutes, and than decolorize the solution by adding, drop by drop, the hydrogen peroxide solution (4.6). Withdraw the flask from the bath, add 1 ml of the 3,4-xylenol solution (4.3), stir and place the flask in the water bath (5.2), controlled at 35 ± 1 °C. Stir from time to time. After 30 min, cautiously add, while stirring, 100 ml of water to the flask.

6.3.2 *Distillation*

Connect the flask to the distillation apparatus (5.3), heat to boiling and collect, over a period of approximately 15 min, 30 ml of distillate in a 50 ml one-mark volumetric flask containing 10 ml of the sodium hydroxide solution (4.5). Stop the circulation of condenser water and distil a few more millilitres. Cool the flask containing the distillate to ambient temperature, dilute to the mark and mix.

6.3.3 *Spectrophotometric measurements*

After 15 min, carry out the measurements with the spectrophotometer (5.4) at the wavelength of maximum absorption (wavelength about 435 nm), after having adjusted the apparatus to zero absorbance against the compensation solution.

6.3.4 *Plotting of the graph*

Plot a graph having, for example, the masses, in micrograms, of nitrogen (N) contained in 50 ml of the standard solutions as abscissae, and the corresponding values of absorbance as ordinates.

6.4 Determination

6.4.1 *Preparation of the test solution*

Place the distillation flask (5.3.1) containing the test portion (6.1) in a bath of water and ice and add 0,200 g of the mercury(II) acetate (4.1). Then add 15 ml of the sulphuric acid solution (4.2) very slowly, in small portions and with stirring, so that the temperature never exceeds 35 °C. Add, drop by drop, the volume of the potassium permanganate solution (4.4) necessary to give a pink coloration which is stable for a few minutes. Then decolorize the solution by adding, drop by drop, the hydrogen peroxide solution (4.6). Withdraw the flask from the bath, add 1 ml of the 3,4-xylenol solution (4.3), stir and place the flask in the water bath (5.2), controlled at 35 ± 1 °C. Stir from time to time. After 30 min, cautiously add, while stirring, 100 ml of water to the flask.

6.4.2 *Distillation*

Connect the flask to the distillation apparatus (5.3) and distil as specified in 6.3.2.

6.4.3 *Spectrophotometric measurement*

Carry out the spectrophotometric measurement of the solution as specified in 6.3.3, after having adjusted the apparatus to zero absorbance against the blank test solution (6.2).

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[1] The addition of mercury(II) acetate has been found necessary to avoid interference from chlorides.

7 EXPRESSION OF RESULTS

By means of the calibration graph (6.3.4), determine the mass, in micrograms, of nitrogen corresponding to the value of the spectrophotometric measurement.

The nitrogen content, expressed as milligrams of N per kilogram, is given by the formula

$$\frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in micrograms, of nitrogen found in the test 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.

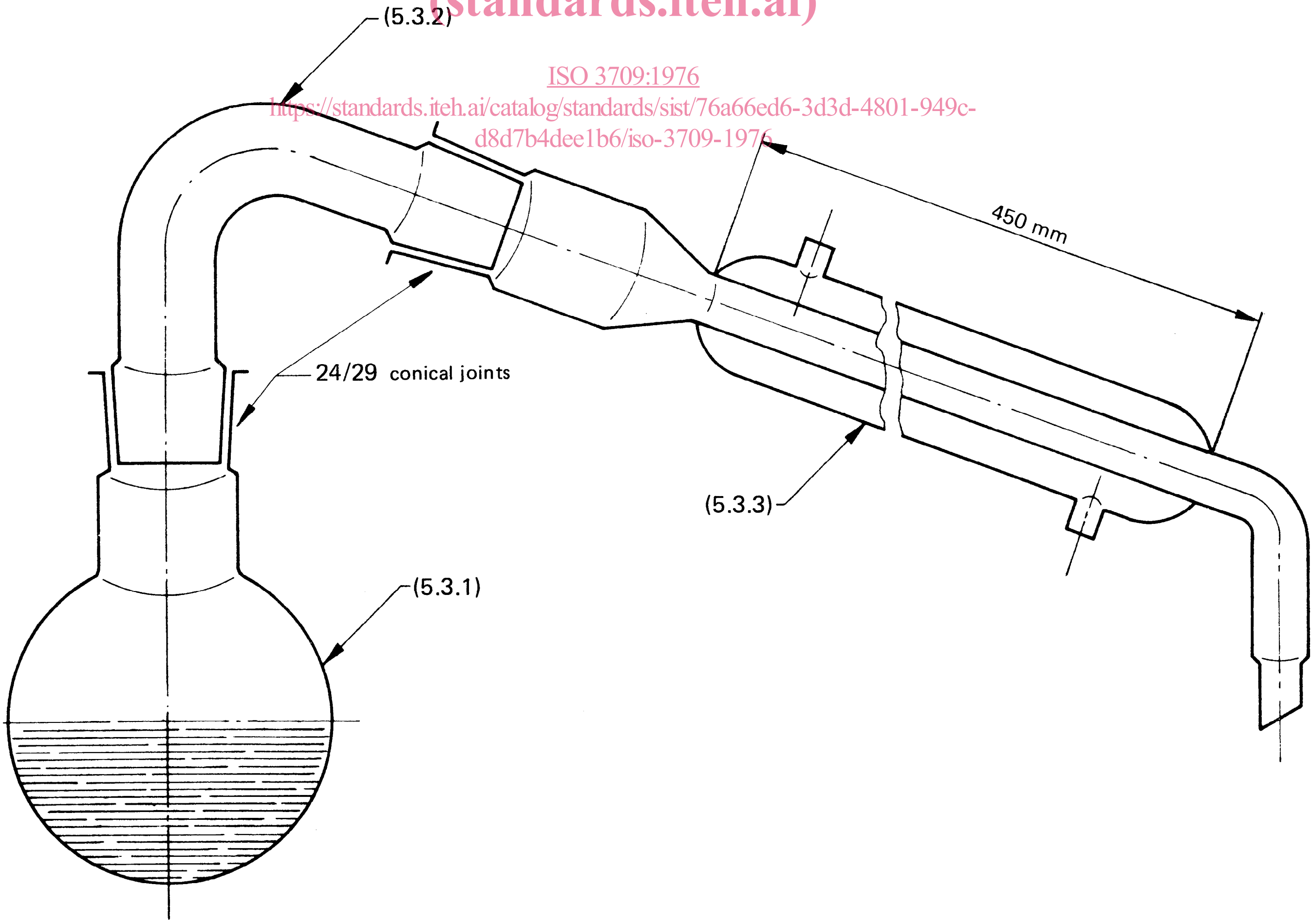

FIGURE – **Typical distillation apparatus**

ANNEX

ISO PUBLICATIONS RELATING TO PHOSPHORIC ACID FOR INDUSTRIAL USE

PHOSPHORIC ACID

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.

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