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INTERNATIONAL STANDARD



5375

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

*Phosphates condensés à usage industriel (y compris les industries alimentaires) – Dosage des oxydes d'azote –  
Méthode spectrophotométrique au xylénol-3,4*

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First edition – 1979-02-15

[ISO 5375:1979](#)

<https://standards.iteh.ai/catalog/standards/sist/f91dfe45-3aec-4ccb-8cd5-0bcflf5bee03/iso-5375-1979>



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UDC 661.635 : 546.17 : 543.42

Ref. No. ISO 5375-1979 (E)

**Descriptors** : orthophosphates, food additives, food industry, chemical analysis, determination of content, nitrogen oxides, spectrophotometric analysis.

## 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 5375 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in June 1977.

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

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

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).

# Condensed phosphates 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 condensed phosphates 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 2 mg/kg.

## 3 PRINCIPLE

Oxidation of nitrous nitrogen of a test portion 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 a 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 [ $\text{Hg}(\text{CH}_3\text{COO})_2$ ].

### 4.2 Sulphuric acid, approximately 80 % (*m/m*) solution.

Eliminate oxides of nitrogen from 800 ml of sulphuric acid,  $\rho$  approximately 1,84 g/ml, by adding it cautiously to about 200 ml of water and heating until white fumes are evolved. Cool, add the fumed acid to a further 200 ml of water and again heat to fuming. Repeat the dilution and fuming once more.

Cool and add 750 ml of this sulphuric acid, while stirring, to 250 ml of water.

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

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

Store this solution below 5 °C.

### 4.4 Potassium permanganate, approximately 16 g/l solution.

### 4.5 Sodium hydroxide, approximately 80 g/l 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 it 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.

Transfer 10,0 ml of the standard potassium nitrate solution (4.7) 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 Water bath, capable of being controlled at $35 \pm 1$ °C.

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

#### 5.2.1 Distillation flask, of capacity 250 ml.

#### 5.2.2 Sloping recovery bend, at 75°.

#### 5.2.3 Liebig condenser, water circulation, effective length approximately 450 mm, connecting to the sloping recovery bend (5.2.2).

### 5.3 Spectrophotometer.

**6 PROCEDURE**

**WARNING** — The residual solutions of distillation contain mercury salts. For the removal and recovery of mercury, in order to prevent pollution of waste water, treat the solutions as follow :

- collect the residual solutions in a container of convenient capacity;
- precipitate the mercury in an alkaline medium with excess sodium sulphide;
- oxidize this excess by hydrogen peroxide to prevent the mercury from re-dissolving in the form of polysulphides;
- decant and filter the mercury-free solution before disposal to waste;
- transfer the insoluble residues to a storage container with a view to subsequent recovery of the mercury by specialist firms.

**6.1 Test portion**

Weigh, to the nearest 0,001 g, about 1 g of the test sample.

**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 all 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 (5.2.1), place 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 (5.2.1) in a bath of water and ice, and introduce into the flask 0,200 g of the mercury(II) acetate (4.1)<sup>1)</sup>. 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 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 (5.1), 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 (5.2), 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 (5.3) 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 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**

**6.4.1.1** If the test portion (6.1) contains from 2 to 25 µg of oxides of nitrogen, expressed as nitrogen (N), introduce it directly into the distillation flask (5.2.1) and add 5 ml of water. Proceed in accordance with 6.4.1.3.

**6.4.1.2** If the test portion (6.1) contains more than 25 µg of oxides of nitrogen, expressed as nitrogen (N), dissolve it in water, transfer the solution quantitatively to a volumetric flask of convenient capacity, dilute to the mark and mix. This solution should contain between 2 and 25 µg of nitrogen (N) per 5 ml.

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

Place 5,0 ml of this solution into the distillation flask (5.2.1). Proceed in accordance with 6.4.1.3.

**6.4.1.3** Place the distillation flask and its contents 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 (5.1), 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 (5.2) and distil as specified in 6.3.2.

#### 6.4.3 Spectrophotometric measurements

Carry out the spectrophotometric measurement of the test solution (6.4.2) and of the blank test solution (6.2) as specified in 6.3.3, after having adjusted the apparatus to zero absorbance against water.

### 7 EXPRESSION OF RESULTS

By means of the calibration graph (6.3.4), determine the masses, in micrograms, of nitrogen corresponding to the values of the spectrophotometric measurements (6.4.3).

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

$$\frac{m_1 - m_2}{m_0} \times D$$

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;

$m_2$  is the mass, in micrograms, of nitrogen found in the blank test solution.

$D$  is the ratio of the volume, in millilitres, of the test solution to the volume, in millilitres, of the aliquot portion taken for the determination. (If the determination has been carried out on the whole of the test solution,  $D$  is equal to 1.)

### 8 TEST REPORT

The test report shall include the following particulars :

- an identification of the sample;
- the reference of the method used;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard, or regarded as optional.

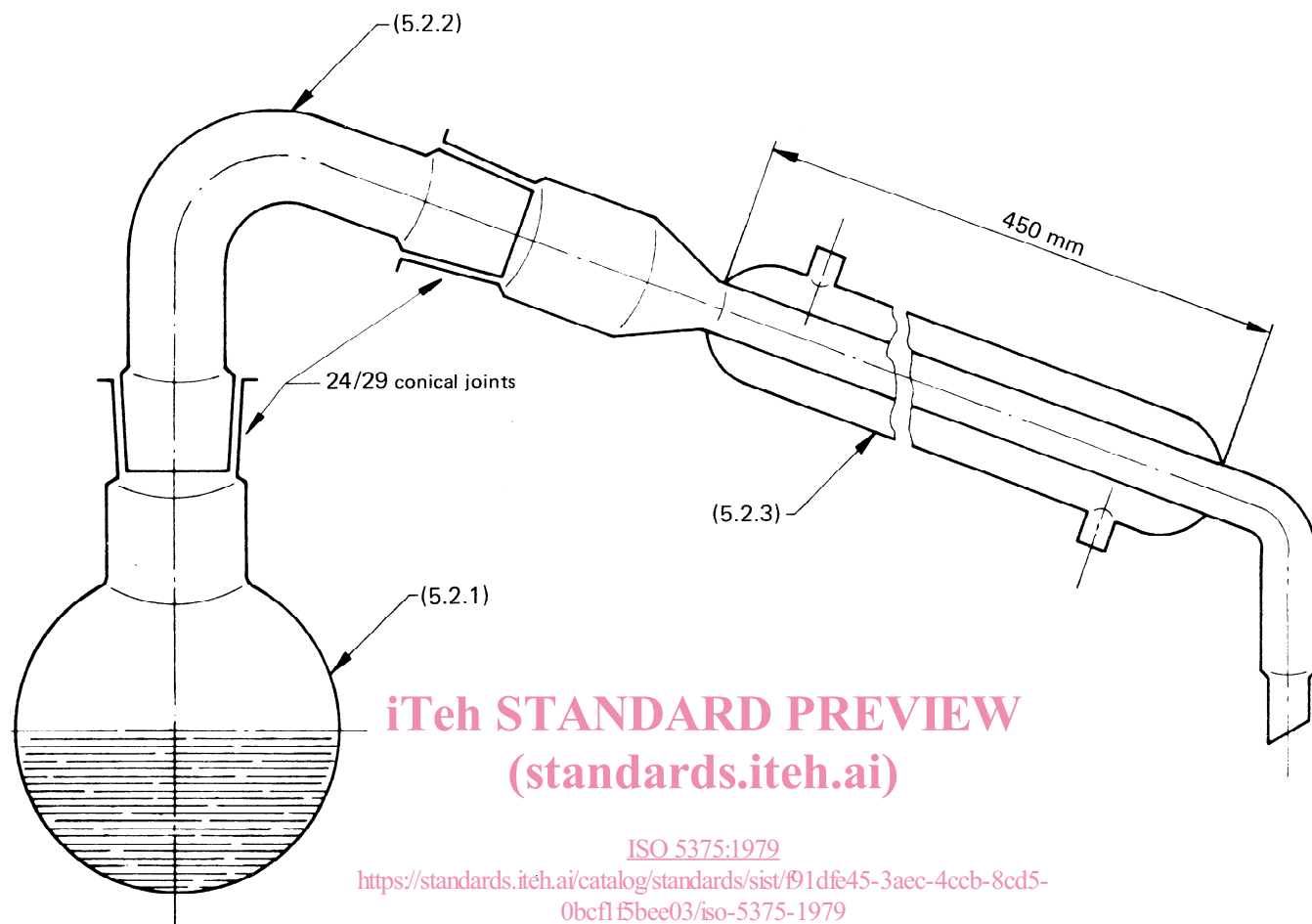


FIGURE – Typical distillation apparatus

ANNEX

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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