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# International Standard ISO 7100

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# Phosphoric acid for industrial use (including foodstuffs) — Determination of vanadium content — Phosphotungsten vanadate spectrometric method

*Acide phosphorique à usage industriel (y compris les industries alimentaires) — Dosage du vanadium — Méthode spectrométrique au phosphotungsto-vanadate*

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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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No member body expressed disapproval of the document.

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# Phosphoric acid for industrial use (including foodstuffs) — Determination of vanadium content — Phosphotungsten vanadate spectrometric method

## 1 Scope and field of application

This International Standard specifies a phosphotungsten vanadate spectrometric method for the determination of the vanadium content of phosphoric acid for industrial use (including foodstuffs).

The method is applicable to products having a vanadium content equal to or greater than 3 mg/kg.

## 2 Reference

ISO 4285, *Phosphoric acid for industrial use — Guide to sampling techniques.*

## 3 Principle

Formation of a yellow complex by reaction of the vanadium present in a test portion with the phosphoric acid and sodium tungstate in acid medium. Spectrometric measurement of the absorbance at a wavelength of about 425 nm.

## 4 Reagents

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

**4.1 Sulphuric acid,** 245 g/l solution.

**4.2 Phosphoric acid,** 490 g/l solution, corresponding to 355 g of $P_2O_5$ per litre.

Weigh to the nearest 0,1 g, 177,5 g of phosphorus(V) oxide ($P_2O_5$) and spread out in a shallow layer in a suitable dish. Allow the dish to stand in a closed vessel containing water (for example a desiccator containing water in place of the desiccant), in order to effect the initial hydration. Then dissolve the hydrated phosphorus(V) oxide in 500 ml of water.

**4.3 Potassium permanganate,** 3,15 g/l solution.

**4.4 Sodium tungstate** ($Na_2WO_4$), 73,5 g/l solution.

Dissolve 82,5 g of sodium tungstate dihydrate ($Na_2WO_4 \cdot 2H_2O$) in 1 litre of water.

**4.5 Vanadium,** standard solution corresponding to 1,000 g of V per litre.

Weigh, to the nearest 0,000 1 g, 2,393 5 g of sodium metavanadate ($NaVO_3$) and dissolve in water. Transfer quantitatively the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 1 mg of V.

**4.6 Vanadium,** standard solution corresponding to 10 mg of V per litre.

Take 10,0 ml of the standard vanadium solution (4.5), transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 µg of V.

**4.7 Sodium nitride** ($Na_3N$), 10 g/l solution.

## 5 Apparatus

Ordinary laboratory apparatus, and

**5.1 Spectrometer,** with selector for continuous variation of wavelength, or

**5.2 Spectrometer,** fitted with filters allowing a maximum transmission at about 425 nm.

## 6 Procedure

### 6.1 Test portion

Weigh by difference, to the nearest 0,001 g, a quantity of the test sample (see ISO 4285) containing not more than 18 g of $P_2O_5$ and not more than 1 000 µg of vanadium.

### 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 as used for the determination, diluting to the same volume and taking an aliquot portion having the same volume as that used in the determination.

## 6.3 Preparation of the calibration graph

### 6.3.1 Preparation of the standard colorimetric solutions

for spectrometric measurements carried out using cells of 4 or 5 cm optical path length

Into a series of six 100 ml beakers, place the volumes of the standard vanadium solution (4.6) indicated in the following table.

| Vanadium standard solution (4.6) | Corresponding mass of V |
|---|---|
| ml | µg |
| 0* | 0 |
| 2,0 | 20 |
| 4,0 | 40 |
| 6,0 | 60 |
| 8,0 | 80 |
| 10,0 | 100 |

* Blank test on the reagents for calibration.

Add to each beaker 5 ml of the phosphoric acid solution (4.2) and 5 ml of the sulphuric acid solution (4.1). Dilute to about 30 ml and heat to boiling. Keep the solutions boiling and add the potassium permanganate solution (4.3), drop by drop, to each beaker until the solution shows a violet coloration, stable for 1 min. Remove the excess of permanganate by adding, drop by drop, the sodium nitride solution (4.7), waiting for 10 to 15 s after each addition until disappearance of the pink coloration. Then add 5 ml of the sodium tungstate solution (4.4) and heat again to boiling for 1 min. Allow to cool to ambient temperature.

Transfer each solution quantitatively to a separate 50 ml one-mark volumetric flask, dilute each to the mark and mix. Allow to stand for 1 h.

### 6.3.2 Spectrometric measurements

Carry out the spectrometric measurements at a wavelength of about 425 nm, after having adjusted the instrument (5.1 or 5.2) to zero absorbance against water.

NOTE — The wavelength of 425 nm is not that of maximum absorption and it is therefore necessary to adjust the wavelength always on the same value, both for the determination and for the calibration.

### 6.3.3 Plotting of the calibration graph

Deduct the absorbance of the blank test on the reagents for calibration from that of each standard colorimetric solution and plot a graph, having, for example, the vanadium contents, in micrograms per 50 ml of standard colorimetric solution, as abscissae and the corresponding values of absorbance as ordinates.

## 6.4 Determination

### 6.4.1 Preparation of the test solution and colour development

Place the test portion (6.1) in a 100 ml one-mark volumetric flask, dilute to the mark and mix. Take an aliquot volume of 10 ml (which should contain not more than 1,8 g of $P_2O_5$ and not more than 100 µg of vanadium) and place it in a 100 ml beaker. If necessary, adjust the amount of $P_2O_5$ in it to that in the standard colorimetric solutions (6.3.1), namely 1,8 g, by adding a calculated quantity of the phosphoric acid solution (4.2). Add 5 ml of the sulphuric acid solution (4.1), dilute to about 30 ml and heat the solution to boiling.

Continue the procedure, following the procedure specified in 6.3.1, starting from the sentence "Keep the solutions boiling and add the potassium permanganate solution (4.3) ...".

### 6.4.2 Spectrometric measurements

Carry out the measurements of the absorbance of the test solution (6.4.1) and that of the blank test solution (6.2), after having adjusted the instrument (5.1 or 5.2) to zero absorbance against water, following the procedure specified in 6.3.2.

## 7 Expression of results

By means of the calibration graph (6.3.3), determine the masses, in micrograms, of vanadium corresponding to the values of the absorbances measured on the test solution and on that of the blank test.

The vanadium content, expressed in milligrams of V per kilogram, is given by the formula

$$\frac{m_1 - m_2}{m_0} \times 10$$

where

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

$m_1$ is the mass, in micrograms, of vanadium corresponding to the value of the absorbance of the test solution (6.4.1);

$m_2$ is the mass, in micrograms, of vanadium corresponding to the value of the absorbance of the blank test solution (6.2);

10 is the ratio between the volume, in millilitres, of the test solution and the volume, in millilitres, of the aliquot portion taken for the determination (see 6.4.1).

## 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 the International Standard or in the International Standard to which reference is made, or regarded as optional.