SLOVENSKI STANDARD
SIST EN ISO 3946:1998

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Škrobi in škrobni derivati - Določevanje celotnega fosforja - Spektrofotometrijska metoda (ISO 3946:1982)

Starches and derived products - Determination of total phosphorus content - Spectrophotometric method (ISO 3946:1982)

Stärke und Stärkederivate - Bestimmung des Gesamtphosphorgehaltes - Spektralphotometrisches Verfahren (ISO 3946:1982)

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Amidons, fécules et produits dérivés - Détermination de la teneur en phosphore total - Méthode spectrophotométrique (ISO 3946:1982)

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Ta slovenski standard je istoveten z: **EN ISO 3946:1994**

ICS:

67.180.20	Škrob in izdelki iz njega	Starch and derived products

SIST EN ISO 3946:1998 **en**

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EUROPEAN STANDARD

NORME EUROPÉENNE

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EN ISO 3946

August 1994

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Descriptors: starches, chemical analysis, determination of content, phosphorus, spectrophotometric analysis

English version

Starches and derived products - Determination of total phosphorus content - Spectrophotometric method (ISO 3946:1982)

Amidons, fécules et produits dérivés - Détermination de la teneur en phosphore total - Méthode spectrophotométrique (ISO 3946:1982)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart,36 B-1050 Brussels

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Foreword

The text of the International Standard ISO 3946:1982, prepared by ISO/TC 93 "Starch", was submitted to the formal vote and was approved by CEN as EN ISO 3946:1994 on 1994-08-22 without any modifications.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 1995, and conflicting national standards shall be withdrawn at the latest by February 1995.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

Endorsement notice

The text of the International Standard ISO 3946:1982 was approved by CEN as a European Standard without any modification.

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SIST EN ISO 3946:1998

International Standard 3946

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

Starches and derived products — Determination of total phosphorus content — Spectrophotometric method

Amidons, fécules et produits dérivés — Détermination de la teneur en phosphore total — Méthode spectrophotométrique

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Descriptors : starches, chemical analysis, determination of content, phosphorus, spectrophotometric analysis.

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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.

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International Standard ISO 3946 was developed by Technical Committee ISO/TC 93, *Starch (including derivatives and by-products)*, and was circulated to the member bodies in February 1982.

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

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Austria	Netherlands	USA
Egypt, Arab Rep. of	Poland	USSR
France	Portugal	
Germany, F.R.	South Africa, Rep. of	

No member body expressed disapproval of the document.

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Starches and derived products — Determination of total phosphorus content — Spectrophotometric method

1 Scope and field of application

This International Standard specifies a spectrophotometric method for the determination of the total phosphorus content of starch, including derivatives and by-products, in which the expected content, calculated as phosphorus (P), does not exceed 5 % (*m/m*).

2 Definition

total phosphorus content : The quantity of phosphorus determined in accordance with the conditions specified in this International Standard and expressed as phosphorus (P) as a percentage by mass of the product as received.

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3 Principle

Destruction of the organic substances by digestion with a sulpho-nitric mixture and conversion of phosphates to orthophosphates.

Formation, by means of a reducing agent, of a phosphomolybdate know as molybdenum blue.

Spectrophotometric measurement of the intensity of the blue colour at a wavelength of 825 nm.

4 Reagents

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

4.1 Sulpho-nitric reagent.

Prepared by mixing 1 part by volume of sulphuric acid, ϱ_{20} 1,84 g/ml, 96 % (*m/m*) solution, and 1 part by volume of nitric acid, ϱ_{20} 1,38 g/ml, 65 % (*m/m*) solution.

4.2 Nitric acid, ϱ_{20} 1,38 g/ml, 65 % (*m/m*) solution.

4.3 Ascorbic acid, 50 g/l solution.

Keep this solution in a refrigerator for a maximum of 48 h.

4.4 Ammonium molybdate solution, prepared as follows.

In a 1 l flask, dissolve 10,6 g of ammonium molybdate tetrahydrate [$(NH_4)_6Mo_7O_{24}.4H_2O$] in 500 ml of water.

Add 500 ml of 10 mol/l sulphuric acid solution, mix and allow to cool to ambient temperature.

4.5 Sodium hydroxide, 10 mol/l solution.

4.6 Phosphorus, standard solutions.

4.6.1 Stock solution, corresponding to 100 mg of P per litre.

Weigh, to the nearest 0,5 mg, 0,439 3 g of anhydrous potassium dihydrogenorthophosphate and dissolve in water. Transfer quantitatively into a 1 000 ml one-mark volumetric flask.

Dilute to the mark with water and mix.

1 ml of this standard solution contains 100 µg of P.

NOTE — The potassium dihydrogenorthophosphate shall be dried before use for 1 h, in a drying oven controlled at 105 ± 2 °C, and then allowed to cool in a desiccator.

4.6.2 Standard solution, corresponding to 4 mg of P per litre.

Using a pipette, take 10 ml of the stock solution (4.6.1) and place it in a 250 ml one-mark volumetric flask.

Dilute to the mark with water and mix.

1 ml of this standard solution contains 4 µg of P.

5 Apparatus

NOTE — Ensure that the detergents used for cleaning glassware do not contain phosphorus.

Ordinary laboratory apparatus, and in particular

5.1 One-mark volumetric flasks, of capacities 50 - 100 - 200 - 250 and 500 ml, complying with the requirements of ISO 1042.

SIST EN ISO 3946:1998

5.2 **Conical flasks**, of capacity 50 ml.

5.3 **Digestion flasks**, of capacity 100 ml.

5.4 **Pipettes**, of capacities 1 - 2 - 5 - 10 - 15 and 25 ml, complying with the requirements of ISO 648.

5.5 **Circulation-type cooling bath**, of temperature between 15 and 25 °C.

5.6 **Boiling water bath**.

5.7 **Hot-plate**.

5.8 **Desiccator**, containing an effective desiccant.

5.9 **Spectrophotometer**, fitted with cells of optical path length 1,0 cm, capable of measuring at a wavelength of 825 nm.

5.10 **Analytical balance**.

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6 Procedure

Carry out the preparation of the calibration curve and the determination within 2 h.

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6.1 Preparation of the calibration graph

Take a series of seven 50 ml conical flasks (5.2). Using a pipette (5.4), introduce into six of them 1,0 - 2,0 - 3,0 - 4,0 - 5,0 and 10,0 ml of the standard phosphorus solution (4.6.2), corresponding to 4 - 8 - 12 - 16 - 20 and 40 µg of P.

Add water to each of the seven flasks so that the total volume is approximately 30 ml. Mix.

Using a pipette, add to each of the flasks, in the following order, 4 ml of the ammonium molybdate solution (4.4) and 2 ml of the ascorbic acid solution (4.3). Mix after each addition.

Place the seven flasks in the boiling water bath (5.6) for 10 min.

Cool to ambient temperature by immersing the flasks in the cooling bath (5.5). Transfer the contents of the flasks quantitatively to the 50 ml one-mark volumetric flasks (5.1). Dilute to the marks with water and mix.

Using the spectrophotometer (5.9), determine the absorbance at 825 nm of each of the six solutions, using the solution from the flask without the standard solution as the reference. Plot the calibration curve giving the number of micrograms of phosphorus as a function of the absorbance.

6.2 Preparation of the test sample

Mix the sample thoroughly.

6.3 Test portion

Weigh, to the nearest 0,2 mg, 0,5 g of the test sample (6.2). This mass corresponds to an absorbance range between 0,1 and 0,7; if this is not the case, adjust the test portion accordingly (see the table).

6.4 Digestion

Transfer the test portion (6.3) into a digestion flask (5.3). Add 15 ml of the sulpho-nitric mixture (4.1) and mix well. Place the flask on the hot-plate (5.7). Heat gradually until the liquid is boiling gently in the flask. Continue boiling until the brown vapour is replaced by white vapour and the liquid has become clear.

A persistent dark colour can be eliminated by adding the nitric acid solution (4.2), drop by drop, whilst continuing the digestion.

Allow to cool, then add 10 ml of water and eliminate the excess nitric acid solution by heating until the flask is again filled with white vapour.

6.5 Preparation of the test solution

Cool the mixture (6.4) again and add 45 ml of water. Raise the pH to 7 with the sodium hydroxide solution (4.5). Transfer the contents of the digestion flask to a one-mark volumetric flask of suitable volume (5.1). Dilute to the mark with water. Mix thoroughly.

6.6 Determination

Take an aliquot portion (see the table) and introduce it into a 50 ml conical flask.

Using a pipette, add, in the following order, 4 ml of the ammonium molybdate solution (4.4) and 2 ml of the ascorbic acid solution (4.3). Mix after each addition.

Place the flask in the boiling water bath (5.6) for 10 min.

Cool to ambient temperature by immersing the flask in the cooling bath (5.5). Transfer quantitatively to a 50 ml one-mark volumetric flask (5.1). Dilute to the mark with water and mix.

Using the spectrophotometer, determine the absorbance at 825 nm of this solution.

Read from the calibration curve (see 6.1) the corresponding number of micrograms of phosphorus.

6.7 Blank test

Carry out a blank test in parallel with the determination, replacing the test portion by water.

6.8 Number of determinations

Carry out two determinations on the same test sample (6.2).