



SLOVENSKI STANDARD
SIST EN 1136:1996

01-avgust-1996

Sadni in zelenjavni sokovi - Ugotavljanje vsebnosti fosforja - Spektrometrijska metoda

Fruit and vegetable juices - Determination of phosphorus content - Spectrometric method

Frucht- und Gemüsesäfte - Bestimmung des Phosphorgehaltes - Spektralphotometrisches Verfahren

Jus de fruits et de légumes - Détermination de la teneur en phosphore - Méthode spectrométrique

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Ta slovenski standard je istoveten z: EN 1136:1994

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ICS:

67.160.20 Brezalkoholne pijače Non-alcoholic beverages

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en

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EUROPEAN STANDARD

EN 1136

NORME EUROPÉENNE

EUROPÄISCHE NORM

October 1994

UDC 663.81/.82:620.1:543.42:661.632

Descriptors: food products, beverages, fruit and vegetable juices, chemical analysis, determination of content, phosphore, spectrophotometric analysis

English version

Fruit and vegetable juices - Determination of phosphorus content - Spectrometric method

Jus de fruits et de légumes - Détermination de la teneur en phosphore - Méthode spectrométrique

Frucht- und Gemüsesäfte - Bestimmung des Phosphorgehaltes - Spektralphotometrisches Verfahren

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

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

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

Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 174 "Fruit and vegetable juices - Methods of analysis", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by April 1995, and conflicting national standards shall be withdrawn at the latest by April 1995.

Annexes designated "informative" are given only for information. In this standard annexes A and B are informative.

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.

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1 Scope

This european standard specifies a method for the determination of the phosphorus content of fruit and vegetable juices and related products.

2 Normative references

This european standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this european standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 1135:1994	Fruit and vegetable juices - Determination of ash
ISO 5725:1986	Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests
ISO 3696:1987	Water for analytical laboratory use - Specification and test methods

3 Symbols

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For the purposes of this standard, the following symbols apply:

c	Substance concentration ;	SIST EN 1136:1996
r	Mass concentration.	https://standards.iteh.ai/catalog/standards/sist/12117bd7-1806-4f65-b573-1b6fe564a3cb/sist-en-1136-1996

4 Principle

Phosphate and molybdate react in acid solution to form a phosphomolybdic complex. In a mixture of molybdic and phosphomolybdic acids, only the molybdenum in the form of phosphomolybdate is reduced to molybdenum-blue, by suitable reducing agents (ascorbic acid). The absorbance of the molybdenum-blue, which is directly proportional to the phosphate content, is measured spectrometrically. Phosphate, $(\text{PO}_4)^{3-}$ is expressed as phosphorus (P).

5 Reagents

5.1 General

Use only reagents of recognised analytical grade and only water of a least grade 2 according to ISO 3696:1987.

5.2 Hydrochloric acid, $c(\text{HCl}) = 2 \text{ mol/l}$.

5.3 Sulfuric acid, $c(\text{H}_2\text{SO}_4) = 1 \text{ mol/l}$.

5.4 L-Ascorbic acid, (Vitamin C) $C(\text{C}_6\text{H}_8\text{O}_6) = 0,02 \text{ mol/l} = 0,353 \text{ g/100 ml}$.

This solution shall be freshly prepared.

5.5 Ammonium molybdate solution

Dissolve approximately 2 g ammonium molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \times 4\text{H}_2\text{O}$) in about 60 ml warm water (60 °C). Cool to about 20 °C and transfer to a 100 ml graduated flask and dilute to 100 ml with water.

5.6 Standard phosphorus solution, $\rho(\text{P}) = 1,0 \text{ g/l}$.

Dissolve 11,5627 g $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$ in water and dilute to 1 l with water.

6 Apparatus

Usual laboratory apparatus and, in particular, the following:

6.1 Platinum dish, approximately 80 mm diameter.

6.2 Muffle furnace, capable of maintaining a temperature of $525 \text{ °C} \pm 25 \text{ °C}$.

6.3 Water bath

6.4 Spectrometer, capable of measuring at 720 nm.

6.5 Cuvettes of 10 mm optical path length, and which have no significant absorption at 720 nm.

7 Procedure

7.1 Preparation of the test sample

Normally products shall not be pretreated and their analysis by this method shall be on a volumetric basis, results being expressed per litre of sample. The analysis of concentrated products may also be carried out on a volumetric basis, after dilution to a known relative density. In this case, the relative density shall be indicated. Based on a weighed sample and taking the dilution factor for analysis into account, the results may also be expressed per kilogram of product. In products with high viscosity and/or very high content of cells (for example pulp), determination on the basis of a weighed test sample is the usual procedure.

7.2 Test procedure

7.2.1 Preparation of the standard curve solutions

Prepare the standard curve solutions so that concentration of the final test solutions measured spectrometrically (see 7.2.3) shall contain phosphorus at concentrations within the range of 0,1 mg/l to 1,5 mg/l of P using suitable dilutions of the standard phosphorus solution (5.6). The solutions are treated as described in section 7.2.3.

7.2.2 Preparation of the test sample for spectrometric analysis

Dissolve the white ash from 25 ml of test sample which has been ashed according to EN 1135 in 2ml to 3 ml hydrochloric acid (5.2) and transfer the solution to a 50 ml volumetric flask and dilute to the mark.

7.2.3 Spectrometric analysis

Pipette a suitable volume (recommended volumes : for grape and orange juices 2 ml; for apple juice 5 ml) of test sample prepared as in clause 7.2.2, or standard curve solution, into a 100 ml volumetric flask. Dilute with water to about 50 ml then add 20 ml sulfuric acid (5.3.), 4 ml ammonium molybdate solution (5.5) and 2 ml L-ascorbic acid solution (5.4).

In some samples of vegetable juices the high content of nitrate present interferes with the reduction of the phosphomolybdate complex and so the amount of ascorbic acid solution (5.4 ; 2 ml) may be insufficient. In such cases use a higher concentration of ascorbic acid (e.g. 10 g/l).

Place the flask (not stoppered) in a boiling water-bath for 15 minutes, then cool to room temperature and make the volume up to 100 ml. Measure the absorbance of the solution in the spectrometer at 720 nm against water. The absorbance of this solution remains constant for up to 3 h and the Beer-Lambert law is obeyed for concentrations between 0,1 mg/l and 1,5 mg/l of P.

8 Calculation

Plot the curve of absorbance against P concentration for the standard solutions. Calculate the phosphorus content, expressed as milligrams of phosphorus per litre, from the following expression:

$$\rho(\text{P}) = \frac{200 \times a}{V}$$

where :

- a is the phosphorus content in milligrams per litre of the final solution prepared as in (7.2.3), corresponding to the absorbance of the same final solution as measured in (7.2.3) and read from the standard curve ;
- V is the volume of test sample used, in millilitres.

Take into account the dilution factor and the relation of the value to mass or volume. If a concentrated product has been diluted to single strength, report the relative density of the single strength sample.

Express the phosphorus content as milligrams of phosphorus per litre, to one decimal place.

NOTE : The phosphate content may be calculated from the phosphorus content $\rho(P)$ by multiplying $\rho(P)$ by the factor 3,066.

9 Precision

Details of the interlaboratory test on the precision of the method are summarized in annex B. The values derived from the interlaboratory test may not be applicable to analyte concentration ranges and matrices other than given in annex B.

9.1 Repeatability

The absolute difference between two single test results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value r in not more than 5 % of the cases.

The values are :

clear juices : $r = 4$ mg/l
cloudy juices : $r = 8$ mg/l

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9.2 Reproducibility

The absolute difference between two single test results on identical test material reported by two laboratories will exceed the reproducibility value R in not more than 5 % of the cases.

The values are :

clear juices : $R = 10$ mg/l
cloudy juices : $R = 21$ mg/l

10 Test report

The test report shall contain the following data :

- all information necessary for the identification of the sample (kind of sample, origin of sample, designation) ;
- a reference to this european standard ;
- the date and type of sampling procedure (if possible) ;
- the date of receipt ;

- the date of test ;
- the test results and units in which they have been expressed ;
- whether the repeatability of the method has been verified ;
- any particular points observed during the course of the test ;
- any operations not specified in the method or regarded as optional, which might have affected the results.

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