SLOVENSKI STANDARD
SIST EN 13196:2000

01-december-2000

Sadni in zelenjavni sokovi - Določ evanje celotnega ǵveplovega dioksida z destilacijo

Fruit and vegetable juices - Determination of total sulfur dioxide by distillation

Frucht- und Gemüsesäfte - Bestimmung des Gesamtschwefeldioxids durch Destillation

Jus de fruits et de légumes - Dosage du dioxyde de soufre total par distillation

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Ta slovenski standard je istoveten z: **EN 13196:2000**

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ICS:

67.160.20 Brezalkoholne pijač e Non-alcoholic beverages

SIST EN 13196:2000 **en**

2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 13196

March 2000

ICS 67.160.20

English version

Fruit and vegetable juices - Determination of total sulfur dioxide by distillation

Jus de fruits et de légumes - Dosage du dioxyde de soufre total par distillation

Frucht- und Gemüsesäfte - Bestimmung des Gesamtschwefeldioxids durch Destillation

This European Standard was approved by CEN on 20 January 2000.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. 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.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This European Standard has been prepared by 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of an identical text or by endorsement, at the latest by September 2000, and conflicting national standards shall be withdrawn at the latest by September 2000.

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

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1 Scope

This European Standard specifies a distillation method for the quantitative determination of total sulfur dioxide in fruit or vegetable juices and related products.

This method applied to onions, leek or cabbage products can lead to false positive results.

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 ISO 3696:1995, *Water for analytical laboratory use - Specification and test method (ISO 3696:1987).*

3 Symbols

For the purposes of this standard the following symbols apply :

- c Substance concentration;
- ρ Mass concentration;
- ϕ Volume fraction.

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4 Principle

The acidified fruit or vegetable juice to be analysed is heated in a distillation system. The sulfur dioxide liberated is swept out of the system with nitrogen or air and is bubbled through a neutralised solution of hydrogen peroxide where it is oxidised to sulfuric acid. The quantification of the sulfur dioxide is then carried out by titration against a standardised sodium hydroxide solution.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade and only water in accordance with at least grade 1 of EN ISO 3696:1995.

5.2 Phosphoric acid, ϕ (H_3PO_4) = 85 %.

5.3 Ethanol, ϕ (CH_3CH_2OH) = 50 %.

5.4 Mixed indicator solution

Dissolve 100 mg of methyl red and 50 mg of methylene blue in approximately 50 ml of ethanol (see 5.3) with stirring. After the indicators have dissolved make the solution up to 100 ml with ethanol.

5.5 Hydrogen peroxide solution, ϕ (H_2O_2) = 30 %.

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5.6 Hydrogen peroxide solution, ϕ (H_2O_2) = 0,9 %.

Dilute 3,00 ml of the hydrogen peroxide solution (see 5.5) to 100 ml in water.

5.7 Sodium hydroxide solution, c (NaOH) = 0,01 mol/l.

Dilute an appropriate standardized solution of sodium hydroxide solution (0,1 mol/l or 1,0 mol/l) to 0,01 mol/l in freshly boiled water. This solution should be prepared freshly every week or standardized against a known mass of potassium hydrogen phthalate using phenolphthalein as the indicator.

5.8 Purified nitrogen gas or air supply

6 Apparatus

Usual laboratory apparatus and, in particular, the following :

6.1 Distillation system

A distillation system similar to the one shown in Figure C.1 is used to carry out this determination. The condenser of this type is a critical feature of the method.

NOTE Sodium hydroxymethylsulfonate can be used to assess the reliability of the apparatus.

6.2 10 ml burette graduated in 0,02 ml units.

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7 Procedure

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7.1 Preparation of the test sample

Normally products shall not be pre-treated and their analysis by this method shall be on a volumetric basis, results being expressed per litre of sample. The analysis of concentrated samples 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 a high viscosity and/or a very high content of cells (for example pulp), determination on the basis of a weighed test sample is the usual procedure.

Mix cloudy samples well before dilution.

7.2 Test procedure

If a fruit or vegetable juice or related product preserved with sulfur dioxide (> 50mg/l) is analysed, place 20 ml of the test sample in a 100 ml round bottom flask. Connect this flask to the distillation system as shown in Figure C.1 in annex C. 5 ml of phosphoric acid (see 5.2) is placed in the dropping funnel.

If a fruit or vegetable juice or related product, unpreserved with sulfur dioxide (< 50 mg/l), is analyzed place 50 ml of the test sample in the 250 ml round bottom flask. Connect this flask to the distillation system as shown in Figure C.1 in annex C. 15 ml of phosphoric acid (see 5.2) is placed in the dropping funnel.

2 or 3 ml of the hydrogen peroxide solution (see 5.6) is added to the absorption receiver, shown as B in Figure C.1 in annex C. To this solution is added 2 drops of the mixed indicator solution (see 5.4) and the hydrogen peroxide solution is neutralised with the sodium hydroxide solution (see 5.7). This flask is then connected to the distillation system.

Allow the phosphoric acid (see 5.2) to enter the distillation flask from the dropping funnel. The distillation flask is then heated to boiling with a small flame (4 cm to 5 cm) positioned directly beneath the flask. The flask is heated over a suitable disk with a small hole cut in it (30 mm diameter) to stop the product from burning in the flask. A wire gauze should not be used.

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During the distillation process air or nitrogen gas passes through the apparatus to "sweep out" the sulfur dioxide from the distillation flask into the receiver vessel. A flow rate of approximately 40 l per hour shall be used. After the reflux has commenced heating shall be continued for 15 minutes.

After the distillation is complete the receiver is removed and the receiver tip is washed with water both internally and externally. The wash water is collected in the receiver flask. The sulfuric acid produced, by the oxidation of the sulfur dioxide, is then titrated against the sodium hydroxide solution (see 5.7) added from a burette (see 6.2) to give a green colour end point.

8 Calculation

The total level of sulfur dioxide ρ is given by the following equations and shall be expressed in milligrams per litre without decimals :

For 50 ml of test sample :

$$\rho = a \times 6,4 \quad (1)$$

For 20 ml of test sample :

$$\rho = a \times 16 \quad (2)$$

where

a is the volume (ml) of sodium hydroxide (see 5.7) used to restore the green colour to the solution in the receiver flask.

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NOTE 1 ml NaOH (c = 0,01 mol/l) is equivalent to 0,32 mg SO_2.

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9 Precision

Details of the inter-laboratory test on 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 results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit *r* in not more than 5 % of the cases.

The value is :

r = 0,8 mg/l.

9.2 Reproducibility

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

The value is :

R = 3,5 mg/l.

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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 known) ;

— the date of receipt ;

— the date of test ;

— test results and units in which they have been expressed ;

— whether the repeatability has been verified ;

— any particular points observed in 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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