SLOVENSKI STANDARD
SIST EN 12137:1998

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Sadni in zelenjavni sokovi - Določevanje vinske kisline v grozdnih sokovih - Metoda tekočinske kromatografije

Fruit and vegetable juices - Determination of tartaric acid in grape juices - Method by high performance liquid chromatography

Frucht- und Gemüsesäfte - Bestimmung von Weinsäure in Traubensaft - Hochleistungsflüssigkeitschromatographisches Verfahren

Jus de fruits et de légumes - Dosage de l'acide tartrique dans les jus de raisin - Méthode de chromatographie liquide a haute performance

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67.160.20 Brezalkoholne pijače Non-alcoholic beverages

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English version

Fruit and vegetable juices - Determination of tartaric acid in grape juices - Method by high performance liquid chromatography

Jus de fruits et de légumes - Dosage de l'acide tartrique dans les jus de raisin - Méthode par chromatographie liquide à haute performance

Frucht- und Gemüsesäfte - Bestimmung von Weinsäure in Traubensaft - Hochleistungs-flüssigkeitschromatographisches Verfahren

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

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Contents

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of an identical text or by endorsement, at the latest by March 1998, and conflicting national standards shall be withdrawn at the latest by March 1998.

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.

1 Scope

This European Standard specifies a method for the determination of tartaric acid in grape juices by high performance liquid chromatography (HPLC).

2 Normative references

This European Standard incorporates by dated or undated references, 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 methods (ISO 3696:1987)
ISO 5725	1986	Precision of test methods - Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests

3 Symbols

For the purposes of this standard, the following symbol applies :

c substance concentration

4 Principle

The content of tartaric acid in grape juices is determined by HPLC (using ultraviolet-detection). The separation takes place on an ion-exclusion column.

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 Sulfuric acid, $c(H_2SO_4)$ = 0,005 mol/l

5.3 Standard tartaric acid solution ($C_4H_6O_6$)

A defined quantity of tartaric acid is dissolved in water. The concentration should be approximately 500 mg/l.

6 Apparatus

Usual laboratory apparatus and, in particular, the following :

6.1 High performance liquid chromatograph

6.2 Ultraviolet (UV) detector, capable of measuring at a wavelength of 210 nm.

6.3 Column oven, capable of maintaining a temperature of 40 °C.

6.4 Separation column : Ion-exclusion column made of sulfonated divinyl benzene-styrene copolymer in Hydrogen form, typical particules sizes 10 µm, (300 mm x 7,8 mm) with a cation H^+ precolumn.

6.5 Membrane filter, of pore size 0,45 µm.

7 Procedure

7.1 Preparation of the test sample

Normally products shall not be pre-treated, however dilution may be necessary 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 a high viscosity and/or a very high content of cells (for example pulp), a determination on the basis of a weighed test sample is the usual procedure.

Dilute grape juices at 1 to 20 volumes (concentrates 1 to 100) and directly use them for HPLC analysis after filtration through a membrane filter (6.5). (When frozen samples are used, ensure that there is no sediment in the sample before dilution).

7.2 Test procedure

7.2.1 Preparation of calibration solutions

Prepare the calibration solutions in the range of 100 mg/l to 500 mg/l of tartaric acid using suitable solutions of the standard tartaric acid solution (5.3). Use the solutions as described in 7.2.2.

7.2.2 HPLC analysis

Inject the calibration solutions (7.2.1) and the samples (7.1) in an HPLC system (6.1, 6.2, 6.3) with the following conditions :

Eluent :	0,005 mol/l sulfuric acid (5.2) ;
Column :	separation column (6.4) ;
Flow :	e.g. 0,6 ml/min (to avoid a high pressure the flow should be increased slowly from 0,2 ml/min to 0,6 ml/min during equilibration) ;
Wavelength :	210 nm ;
Injection volume :	typically 15 µl ;
Running time :	20 min ;
Retention time :	approximately 10 min for tartaric acid ;
Temperature :	40 °C.

8 Calculation

Calculate the content of tartaric acid in the sample using the calibration curve based on peak height.

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.

Report the tartaric acid concentration in grams per litre to one decimal place.

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 limit r in not more than 5 % of the cases.

The value is : $r = 0{,}14$ g/l

9.2 Reproducibility

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

The value is : R = 0,33 g/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 known) ;
- 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 in the course of the test ;
- any operations not specified in the method or regarded as optional, which might have affected the results.