



SLOVENSKI STANDARD

SIST ENV 12141:1998

01-junij-1998

Sadni in zelenjavni sokovi - Določevanje razmerja stabilnih kisikovih izotopov (18O/16O) v vodi iz sadnih sokov - Metoda masne spektrometrije razmerja izotopov

Fruit and vegetable juices - Determination of the stable oxygen isotope (18O/16O) of water from fruit juices - Method using isotope ratio mass spectrometry

Frucht- und Gemüsesäfte - Bestimmung des Verhältnisses der stabilen Sauerstoff-Isotope (18O/16O) im Wasseranteil von Fruchtsäften - Verfahren unter Verwendung der Isotopenverhältnis-Massenspektrometrie

Jus de fruits et de légumes - Détermination du rapport des isotopes stables en oxygene (18O/16O) dans l'eau des jus de fruits - Méthode utilisant la spectrométrie de masse des rapports isotopiques

Ta slovenski standard je istoveten z: ENV 12141:1996

ICS:

67.160.20 Brezalkoholne pijače Non-alcoholic beverages

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en

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EUROPEAN PRESTANDARD

ENV 12141

PRÉNORME EUROPÉENNE

EUROPÄISCHE VORNORM

October 1996

ICS 67.160.20

Descriptors: food products, beverages, fruit and vegetable juices, chemical analysis, determination, ratios, isotopes, oxygen, mass spectrometry

English version

**Fruit and vegetable juices - Determination of the
stable oxygen isotope ratio ($^{18}\text{O}/^{16}\text{O}$) of water
from fruit juices - Method using isotope ratio mass
spectrometry**

Jus de fruits et de légumes - Détermination du
rapport des isotopes stables en oxygène
($^{18}\text{O}/^{16}\text{O}$) dans l'eau des jus de fruits
Méthode utilisant la spectrométrie de masse des
rapports isotopiques

Frucht- und Gemüsesäfte - Bestimmung des
Verhältnisses der stabilen Sauerstoff-Isotope
($^{18}\text{O}/^{16}\text{O}$) im Wasseranteil von Fruchtsäften -
Verfahren unter Verwendung der
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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

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are required to announce the existence of this European Prestandard : Austria, Belgium, 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 Prestandard specifies a method for the determination of the stable oxygen isotope ratio ($^{18}\text{O}/^{16}\text{O}$) of water from fruit juices by isotope ratio mass spectrometry (IRMS).

2 Normative references

This European Prestandard 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 Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

ISO 5725:1986 Precision of the test methods-Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.

3 Symbols and abbreviations

For the purposes of this Standard, the following symbols and abbreviations apply :

$(^{18}\text{O}/^{16}\text{O})$	Isotope ratio of oxygen 18 to oxygen 16 for a considered sample ;
$\delta^{18}\text{O}$	Oxygen 18 (^{18}O) content expressed in parts per thousand (‰);
SMOW	Standard Mean Ocean Water ;
SLAP	Standard Light Antarctic Precipitation ;
GISP	Greenland Ice Sheet Precipitation.

4 Principle

The measurement of $^{18}\text{O}/^{16}\text{O}$ ratios are obtained on carbon dioxide equilibrated with water according to the isotopic exchange reaction [1] :



The $^{18}\text{O}/^{16}\text{O}$ isotopic ratios are determined by mass spectrometry by measuring the ratio of the intensities of ions having a mass of 46 ($^{12}\text{C } ^{16}\text{O } ^{18}\text{O}$) to those with a mass of 44 ($^{12}\text{C } ^{16}\text{O}_2$).

5 Reagents

5.1 Carbon dioxide of analytical grade

5.2 **SMOW and SLAP reference waters** (obtainable from the International Atomic Energy Agency (IAEA)¹⁾), [2].

6 Apparatus

Usual laboratory apparatus and, in particular, the following :

6.1 **Isotope ratio mass spectrometer**, with the ability to determine ¹⁸O content of CO₂ gas natural abundance with an internal precision of 0,05 ‰ or better (expressed in relative δ value (see 8)). The internal precision is here defined as the difference between two measurements of the same CO₂ sample. This instrument should comprise :

- either a triple collector capable of simultaneous collections of the ions with the respective masses of 44, 45 and 46, or a double collector capable of simultaneous collection of ions with respective masses of 44 and 46 ;
- an inlet system capable of introducing carbon dioxide into the mass spectrometer after equilibration with both sample and reference liquids.

6.2 **Sample bottles**, which can be adequately sealed under vacuum and having identical sample volumes of at least 10 ml, and not more than 30 ml (water sample size of 2 ml and 5 ml, respectively), preferably made of borosilicate glass.

6.3 **Pumping system**, capable of obtaining a vacuum of 0,13 Pa during the sample preparation.

6.4 **Thermostatically controlled water bath**, capable of being maintained at 25 °C ± 0,5 °C.

7 Procedure

7.1 Sample preparation

Products shall not normally require any pre-treatment.

¹⁾ International Atomic Energy Agency
P.O. Box 100
A - 1400 Wien
AUSTRIA

7.2 CO₂ equilibration step

Pour into one of the calibrated sample bottles at least 2 ml and not more than 5 ml of the raw fruit juice (not centrifuged or filtered), depending on the size of sample bottles (6.2), then chill to - 80 °C.

Evacuate the air contained in the sample bottle under vacuum using the pump unit (6.3). Then introduce into the sample bottle the carbon dioxide (5.1) at a pressure of approximately 80 000 Pa. Hermetically close the sample bottle and then place it in the thermostatically controlled water bath (6.4). The isotopic equilibrium is reached overnight (approximately 15 h), but this period can be shortened to 6h by shaking the samples. After equilibration, introduce the carbon dioxide contained in the sample bottle into the mass spectrometer (6.1) through a cryogenic trap maintained at - 80 °C or a chemical trap capable of removing water vapour without isotopic fractionation of the carbon dioxide.

7.3 Determination

Using the mass spectrometer (6.1), measure the relative difference δ in parts per thousand (‰) between the ratio of the intensities of the ions having a mass of 46 (¹²C¹⁶O¹⁸O) to those with a mass of 44 (¹²C¹⁶O₂) obtained on the carbon dioxide equilibrated with the sample (I_{46}/I_{44})_{sample} and the reference carbon dioxide (5.1) (I_{46}/I_{44})_{reference} using the following equation :

$$\delta_{\text{sample}} = \left[\frac{(I_{46}/I_{44})_{\text{sample}}}{(I_{46}/I_{44})_{\text{reference}}} - 1 \right] \times 1\,000 \quad (2)$$

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7.4 Calibration

Carry out the same determination under the same conditions (quantity of water, of carbon dioxide, temperature) on standard SMOW and SLAP reference waters (5.2), and then measure δ_{SMOW} and δ_{SLAP} .

8 Calculation

The same carbon dioxide gas used to equilibrate the samples, SLAP and SMOW reference waters is also used as the reference gas in the mass spectrometer. It follows that all the corrections that can be applied cancel themselves out from the equation and the oxygen 18 (¹⁸O) content of the sample with respect to the SMOW reference water is :

$$\delta^{18}\text{O} = \frac{\delta_{\text{sample}} - \delta_{\text{SMOW}}}{\delta_{\text{SMOW}} - \delta_{\text{SLAP}}} \times 55,5 \quad (3)$$

The accepted value for the SLAP reference water is $\delta^{18}\text{O} = - 55,5 \text{ ‰ SMOW}$ [2].

9 Precision

Details of the interlaboratory 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 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 values are :

Orange juice $r = 0,22 \text{ ‰}$;

Apple juice $r = 0,45 \text{ ‰}$;

GISP²⁾ water $r = 0,23 \text{ ‰}$.

9.2 Reproducibility

The absolute differences 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 values are :

Orange juice $R = 0,33 \text{ ‰}$;

Apple juice $R = 0,58 \text{ ‰}$;

GISP²⁾ water $R = 0,34 \text{ ‰}$.

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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
- the test results and units in which they have been expressed ;
- whether the repeatability of the method has been verified ;

²⁾ GISP is a secondary international IAEA standard