
Sadni in zelenjavni sokovi - Določevanje razmerja stabilnih vodikovih izotopov (2H/1H) v vodi iz sadnih sokov - Metoda masne spektrometrije razmerja izotopov

Fruit and vegetable juices - Determination of the stable hydrogen isotope ratio (2H/1H) of water from fruit juices - Method using isotope ratio mass spectrometry

Frucht- und Gemüsesäfte - Bestimmung des Verhältnisses der stabilen Wasserstoff-Isotope (2H/1H) im Wasseranteilen von Fruchtsäften - Verfahren unter Verwendung der Isotopenverhältnis-Massenspektrometrie

Jus de fruits et de légumes - Détermination du rapport des isotopes stables en hydrogene (2H/1H) dans l'eau des jus de fruits - Méthode utilisant la spectrométrie de masse des rapports isotopiques

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ICS:

67.160.20 Brezalkoholne pijače Non-alcoholic beverages

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EUROPEAN PRESTANDARD

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English version

**Fruit and vegetable juices - Determination of the
stable hydrogen isotope ratio ($^2\text{H}/^1\text{H}$) of water
from fruit juices - Method using isotope ratio mass
spectrometry**

Jus de fruits et de légumes - Détermination du rapport des isotopes stables en hydrogène ($^2\text{H}/^1\text{H}$) dans l'eau des jus de fruits - Méthode utilisant la spectrométrie de masse des rapports isotopiques

Frucht- und Gemüsesäfte - Bestimmung des Verhältnisses der stabilen Wasserstoff-Isotope ($^2\text{H}/^1\text{H}$) im Wasseranteil von Fruchtsäften - Verfahren unter Verwendung der Isotopenverhältnis-Massenspektrometrie

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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 hydrogen isotope ratio ($^2\text{H}/^1\text{H}$) 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 :

$(^2\text{H}/^1\text{H})$	Isotopic ratio of deuterium to hydrogen of mass 1 for a considered sample ;
$\delta^2\text{H}$	Deuterium (^2H) content expressed in parts per thousand (‰) ;
SMOW	Standard Mean Ocean Water ;
SLAP	Standard Light Antarctic Precipitation ;
GISP	Greenland Ice Sheet Precipitation ;
g	Acceleration due to gravity at the surface of the earth (9,81 m/s ²) ;

4 Principle

The deuterium (^2H) content of water is determined by applying isotope mass spectrometric measurements of the intensities of the ions having a mass of 2 ($^1\text{H}_2$) and of 3 ($^1\text{H}^2\text{H}$) on hydrogen gas obtained by the reduction of water.

5 Reagents

5.1 Nuclear pure Uranium 238 [1], [2] or zinc alloy [3] which is suitable for water reduction.

Uranium and zinc shall be pre-treated before use as follows. Wash with hydrochloric or nitric acid, and then wash with water. Dry and degass metal carefully. For zinc, the degassing should take approximately 30 min to 40 min at 180 °C.

WARNING : Uranium 238, when heated at 800 °C, releases toxic vapours. Do not stand near the oven (6.2).

5.2 SMOW and SLAP reference waters (obtainable from the International Atomic Energy Agency, (IAEA)¹⁾) [4].

5.3 Hydrogen gas of analytical quality.

6 Apparatus

Usual laboratory apparatus and, in particular, the following :

6.1 Isotope ratio mass spectrometer, with the ability to determine the ²H content of hydrogen gas at natural abundance with an internal precision of 0,1 ‰ or better (expressed in relative δ value (see 8)). The internal precision is here defined as the difference between two measurements of the same hydrogen sample. This instrument should comprise :

- a double collector allowing simultaneous collection of the ions having respective masses of 2 and 3 ;

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- a dual inlet system for the introduction of the gases into the mass spectrometer which enables the repeated, alternating measurement of a reference hydrogen gas and the hydrogen gas resulting from the reduction of a water sample ;

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- an electronic compensation system for calculation and elimination of the amount of ¹H₃⁺ ions having been formed in the mass spectrometer and being measured together with ¹H ²H at mass 3.

6.2 Apparatus for the reduction of water samples and the collection of hydrogen gas for the isotopic analysis comprising :

- an oven containing 20 g to 40 g of uranium (5.1) maintained at a temperature of 800°C ; or a reduction container made of special glass and filled with 0,3 g of suitable zinc alloy (5.1) which can be heated to 450 °C to 455 °C for 30 min to 60 min.

- when using uranium a system for trapping the hydrogen gas released by the reduction of the water. A TOEPLER pump [5] is ideally suited for this application or a trapping system may be chosen, using uranium at 60 °C [6]. This enables the hydrogen gas to be collected in a sample bottle which can then be connected up to the inlet system of the mass spectrometer.

6.3 Sample bottles, which can be adequately sealed under vacuum.

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

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

6.5 Centrifuge, capable of producing a centrifugal acceleration of 1 400 g at the base of the centrifuge tube (6.6).

NOTE : The rotational frequency required to give correct centrifugal acceleration can be calculated from the following equation :

$$a = 11,18 \times r \times (n / 1000)^2 \quad (1)$$

where :

- a is the centrifugal acceleration
- r is the radius of the centrifuge in centimetres, measured from the mid point (the centrifuge axis) to the bottom of the centrifuge tube when swung out ;
- n is the rotational frequency per minute.

6.6 Centrifuge tubes

7 Procedure

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7.1 Sample preparation

Centrifuge (6.5) 25 ml of the fruit juice for 10 min at 1 400 g.

7.2 Distillation in a closed vessel

Perform a microdistillation to separate the fruit juice water from organic compounds (sugar, acids, lipids) with quantitative recovery of the water (reaction of the raw juices with zinc or uranium will not produce hydrogen gas with the same deuterium content as the water of the fruit juice). For the details of the microdistillation process, see figure in B1.

A distillation under static vacuum may also be performed by using a special piece of distillation equipment called Rittenberg "trousers" on 2 ml to 3 ml of juice. This process always gives a quantitative recovery of the water.

7.3 Reduction

Use 10 μ l of sample (7.2) and a reduction time of at least 15 min when employing uranium (5.1). Use between 5 μ l to 10 μ l of sample (7.2) and a reduction time of 30 min to 60 min when employing the zinc (5.1) method.
The hydrogen gas obtained is collected in sample bottles (6.3).

7.4 Determination

Connect the sample bottles (6.3) to the mass spectrometer (6.1) and measure the relative difference in parts per thousand of deuterium content between the sample hydrogen gas and a reference hydrogen gas (5.3) using the following equation :

$$\delta_{\text{sample}} = \left[\frac{(^2\text{H}/^1\text{H})_{\text{sample}}}{(^2\text{H}/^1\text{H})_{\text{reference}}} - 1 \right] \times 1000 \quad (2)$$

7.5 Calibration

In accordance with 7.3 and 7.4 carry out the same determination on the two reference waters (SMOW and SLAP (5.2)) and determine $\delta^2\text{H}$ SMOW and $\delta^2\text{H}$ SLAP with respect to the reference hydrogen gas

8 Calculation

Express the deuterium content of the water of the fruit juice with respect to the SMOW reference water by the following equation :

$$\delta^2\text{H}_{\text{sample}} = \frac{\delta_{\text{sample}} - \delta_{\text{SMOW}}}{\delta_{\text{SMOW}} - \delta_{\text{SLAP}}} \times 428 \quad (3)$$

The accepted value for the SLAP reference water is $\delta^2\text{H} = -428 \text{‰ SMOW}$ [4].

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9 Precision

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

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 values are :

Orange juice	$r = 3,69 \text{‰}$;
Apple juice	$r = 3,95 \text{‰}$
GISP ²⁾ water	$r = 3,47 \text{‰}$;
Laboratory water ³⁾	$r = 3,21 \text{‰}$.

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.

²⁾ GISP is a secondary international IAEA standard

³⁾ Laboratory water is the ordinary water used by the laboratory