SLOVENSKI STANDARD
SIST EN 1376:1998

01-november-1998

Živila - Določevanje saharina v namiznih sladilih - Spektrometrijska metoda

Foodstuffs - Determination of saccharin in table top sweetener preparations - Spectrometric method

Lebensmittel - Bestimmung von Saccharin in Tafelsüßen - Spektralphotometrisches Verfahren

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Produits alimentaires - Dosage de la saccharine dans les édulcorants de table - Méthode spectrométrique

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Ta slovenski standard je istoveten z: EN 1376:1996

ICS:

67.180.10 Sladkor in sladkorni izdelki Sugar and sugar products

SIST EN 1376:1998 **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 1376

September 1996

ICS 67.180.10

Descriptors: food products, intense sweeteners, chemical analysis, determination of content, spectrometric analysis

English version

Foodstuffs - Determination of saccharin in table top sweetener preparations - Spectrometric method

Produits alimentaires - Dosage de la saccharine dans les édulcorants de table - Méthode spectrométrique

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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

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 Standard has been prepared by the Technical Commitee CEN/TC 275 "Food analysis - Horizontal methods" the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by March 1997, and conflicting national standards shall be withdrawn at the latest by March 1997.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of 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 and the United Kingdom.

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1 Scope

This European Standard specifies a spectrometric method for the determination of sodium saccharin and saccharin content in solid table top sweetener preparations prepared from cyclamate/saccharin or saccharin.

An inter-laboratory test has been carried out on sweetener tablets [1].

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
Water for analytical laboratory use - Specification and test methods

3 Principle

Preparation of the sample test solution by dissolving table top sweetener preparation in sodium hydroxide solution. Photometric determination of the sodium saccharin content at the absorption maximum of about 265 nm.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water of at least grade 3 as defined in EN ISO 3696.

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4.1 Sodium saccharin standard substance with a known content of at least 98 % in dry matter (105 °C ± 2 °C, to constant mass). The loss in mass on drying shall not exceed 15 % (see 6.2).

NOTE: For further information on identification and purity, see [2].

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4.2 Sodium hydroxide solution, c (NaOH) = 0,1 mol/l [1])

4.3 Sodium saccharin stock solution, ρ ($NaC_7H_4NO_3S$) ≈ 1 g/l [2])

Finely grind an amount of at least 1,5 g of the sodium saccharin standard substance (4.1), then, without delay, dissolve about 280 mg (m_1) of the undried ground sodium saccharin standard substance (equivalent to about 250 mg of anhydrous sodium saccharin or 223 mg of saccharin as free imide. For conversion factors, see 7.2) weighed to the nearest 0,1 mg, in sodium hydroxide solution (4.2) in a 250 ml volumetric flask and dilute to the mark with sodium hydroxide solution (4.2).

Reserve the remaining finely ground sodium saccharin standard substance for the determination of loss in mass on drying (see 6.2). The determination of loss in mass on drying is carried out immediately.

Calculate the mass concentration, ρ, of anhydrous sodium saccharin, in milligrams per litre of the stock solution, using the following equation:

$$\rho = \frac{m_1 \times (100 - L_D) \times 4}{100} \qquad \text{... (1)}$$

where:

m_1 is the mass of undried sodium saccharin standard substance used for the stock solution, in milligrams;

L_D is the loss in mass on drying, in %.

[1]) c is the substance concentration
[2]) ρ is the mass concentration

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4.4 Sodium saccharin standard solutions

Pipette 2,0 ml, 5,0 ml, 10,0 ml and 15,0 ml portions of the sodium saccharin stock solution (4.3) into separate 100 ml volumetric flasks and dilute to the mark with sodium hydroxide solution (4.2). 1 l of these solutions contains about 20 mg, 50 mg, 100 mg and 150 mg, respectively, of anhydrous sodium saccharin.

NOTE: Additional solutions with concentrations within the linear range may be prepared for the calibration graph.

5 Apparatus and equipment

Usual laboratory apparatus and, in particular, the following.

5.1 Spectrometer, suitable for measurements in the ultraviolet (UV) range.

5.2 Quartz cuvettes with an optical path length of 1 cm.

6 Procedure

6.1 Determination of average tablet mass

Determine the mass of at least 20 sweetener tablets to the nearest 0,1 mg and calculate the average mass (m_2) of one tablet.

NOTE: For improved accuracy the use of 100 tablets is recommended.

6.2 Determination of loss in mass on drying of standard substance

Weigh about 1,0 g to the nearest 0,1 mg, of the reserved finely ground sodium saccharin standard substance used for the preparation of the stock solution (4.3), dry this portion to constant mass at 105 °C ± 2 °C and determine the loss in mass on drying in percent (L_D) by weighing.

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6.3 Preparation of the sample test solution

Dissolve an amount (m_0) of finely ground table top sweetener preparation equivalent to about 35 mg of sodium saccharin weighed to the nearest 0,1 mg in the sodium hydroxide solution (4.2) in a 50 ml volumetric flask and dilute to the mark.

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Pipette 20,0 ml of this solution into a 100 ml volumetric flask and dilute to the mark with sodium hydroxide solution (4.2).

6.4 Determination

6.4.1 Measure the absorption spectrum of the standard sodium saccharin solution (4.4) containing about 100 mg of anhydrous sodium saccharin in 1000 ml between wavelengths of 230 nm and 300 nm in quartz cuvettes (5.2) with sodium hydroxide solution (4.2) as reference and determine the wavelength of the absorption maximum (about 265 nm).

6.4.2 Prepare the calibration graph by measuring the absorptions of the standard sodium saccharin solutions (4.4) at the absorption maximum determined in 6.4.1.

6.4.3 Measure the absorption spectrum of the sample test solution as described in 6.4.1 and determine the absorption at the absorption maximum determined in 6.4.1.

If the shape of the curve obtained for the sample test solution differs from that of the standard solution, it is probable that an interfering substance is present. In this case, the method is not applicable.

Verify the applicability by determining the absorptions 15 nm above and below the wavelength of the absorption maximum (about 265 nm). Absorption ratios between these values and the maximum absorption shall not differ from those obtained when using the sodium saccharin standard solutions (4.4).

Examples for absorption spectra of sodium saccharin are given in Annex A.

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7 Expression of results

7.1 General

Plot the absorption values of the sodium saccharin standard solutions (4.4) on millimetre graph paper against the sodium saccharin concentrations in milligrams per litre.

The calibration graph should be linear.

Read off the sodium saccharin concentration, x, in milligrams per litre, corresponding to the absorption of the sample test solution from the calibration graph.

An alternative calculative evaluation using the regression graph may be used.

7.2 Calculation

7.2.1 Calculate the mass fraction, w_1, of anhydrous sodium saccharin, in milligrams per kilogram table top sweetener preparation, using the following equation:

$$w_1 = \frac{x \times 0{,}25 \times 10^6}{m_0} \quad \text{... (2)}$$

where:

x is the concentration of anhydrous sodium saccharin in the sample test solution, read off from the calibration graph, in milligrams per litre;

m_0 is the initial sample mass, in milligrams.

7.2.2 Calculate the mass fraction, w_2, of anhydrous sodium saccharin, in milligrams per tablet, using the following equation:

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$$w_2 = \frac{x \times m_2 \times 0{,}25}{m_0} \quad \text{... (3)}$$

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where:

m_2 is the average tablet mass (6.1), in milligrams;

m_0, x see equation (2).

7.2.3 Calculate the mass fraction, w_3, of sodium saccharin dihydrate, in milligrams per kilogram table top sweetener preparation, using the following equation:

$$w_3 = w_1 \times 1{,}175 \quad \text{... (4)}$$

7.2.4 Calculate the mass fraction, w_4, of saccharin, in milligrams per kilogram table top sweetener preparation, using the following equation:

$$w_4 = w_1 \times 0{,}893 \quad \text{... (5)}$$

7.2.5 Report the result according to current legislation by one of the values obtained in 7.2.1 to 7.2.4 after rounding to one decimal place.

8 Precision

Details of the inter-laboratory test of the precision of the method according to ISO 5725 : 1986 [3] are summarized in annex B. The values derived from the inter-laboratory test may not be applicable to analyte concentration ranges and matrices other than given in annex B.

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8.1 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 = 0,42 mg/100 g

8.2 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 = 0,85 mg/100 g

9 Test report

The test report shall contain at least the following data:

- all information necessary for the identification of the sample;
- a reference to this European Standard or to the method used;
- the results and the units in which the results have been expressed;
- if 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.

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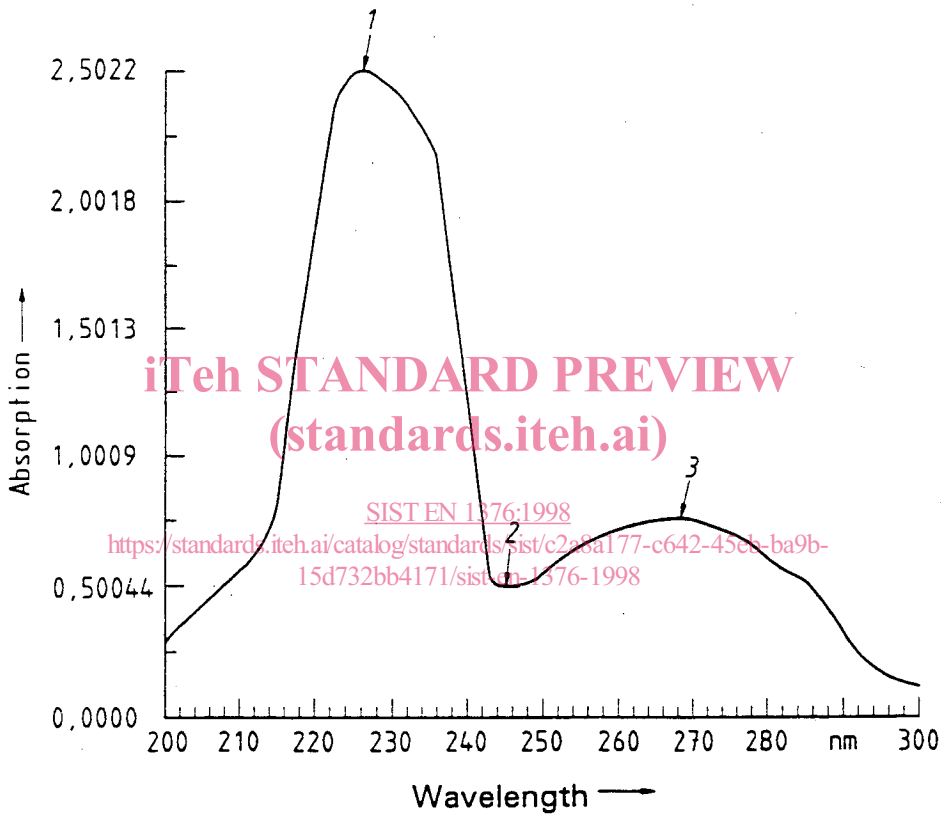
Annex A (informative)

Examples for absorption spectra of a sodium saccharin standard solution

Sample:	Sodium-Saccharin	Function:	Absorption
Solvent:	NaOH; 0,1 mol/l	Wavelength range:	200 nm to 300 nm
Concentration:	100 mg/l		



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Recognized maxima or minima:

1: Wavelength = 226 nm Absorption = 2,502197
2: Wavelength = 246 nm Absorption = 0,498856
3: Wavelength = 270 nm Absorption = 0,750565

Figure A.1: Absorption spectrum of a sodium saccharin standard solution