SLOVENSKI STANDARD
SIST EN 1377:1998

01-november-1998

Živila - Določevanje acesulfama K v namiznih sladilih - Spektrometrijska metoda

Foodstuffs - Determination of acesulfame K in table top sweetener preparations - Spectrometric method

Lebensmittel - Bestimmung von Acesulfam-K in Tafelsüßen - Spektralphotometrisches Verfahren

Produits alimentaires - Dosage de l'acésulfame K dans les édulcorants de table - Méthode spectrométrique

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ICS:

67.180.10 Sladkor in sladkorni izdelki Sugar and sugar products

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English version

Foodstuffs - Determination of acesulfame K in table top sweetener preparations - Spectrometric method

Produits alimentaires - Dosage de l'acésulfame K dans les édulcorants de table - Méthode spectrométrique

Lebensmittel - Bestimmung von Acesulfam-K in Tafelsüßen - Spektralphotometrisches Verfahren

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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

Contents

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of an identical text or by endorsement, at the latest by March 1997, and conflicting national standards shall be withdrawn at the latest by March 1997.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

1 Scope

This European Standard specifies a spectrometric method for the determination of acesulfame K in solid table top sweetener preparations containing it.

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 water. Photometric determination of the acesulfame K content at the absorption maximum of about 227 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.

4.1 Acesulfame K standard substance, with a known content of at least 99 % in dry matter.

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

4.2 Acesulfame K stock solution ρ ($C_4H_4NO_4SK$) $\approx$ 0,8 g/l [1])

Dissolve about 400,0 mg of the acesulfame K standard substance (4.1) weighed to the nearest 0,1 mg, in water in a 500 ml volumetric flask, and dilute to the mark with water.

4.3 Acesulfame K standard solution ρ ($C_4H_4NO_4SK$) $\approx$ 8 mg/l

Pipette 5,00 ml of the acesulfame K stock solution (4.2) into a 500 ml volumetric flask and dilute to the mark with water.

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.

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[1]) ρ is the mass concentration

6.2 Preparation of the sample test solution

Dissolve an amount of finely ground table top sweetener preparation (m_0), equivalent to about 400 mg of acesulfame K or corresponding to 20 times the average mass of a tablet (6.1), transfer to a 500 ml volumetric flask with water, dissolve it in water and dilute to the mark with water.

Allow any undissolved constituents to settle and, if necessary, filter the solution, discarding the first 100 ml of the filtrate. Then pipette 5,00 ml of the solution into a 500 ml volumetric flask and dilute to the mark with water.

6.3 Determination

6.3.1 Measure the absorption spectrum of the standard acesulfame K solution between 200 nm and 280 nm in quartz cuvettes (5.2) with water as reference and determine the absorption (A_1) at the wavelength of the absorption maximum (about 227 nm).

Check the linearity range with a series of solutions of suitable concentrations (calibration graph).

6.3.2 Measure the absorption spectrum of the sample test solution as described in 6.3.1 and determine the absorption (A_2) at the absorption maximum as determined in 6.3.1.

If the shape of the absorption curve obtained for the sample test solution (6.2) 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 12 nm above and 12 nm below the wavelength of the absorption maximum (about 227 nm). Absorption ratios between these values and the maximum absorption shall not differ from those obtained when using the acesulfame K standard solution (4.3).

An example for an absorption spectrum of acesulfame K is given in Annex A.

7 Expression of results

7.1 Calculate the mass fraction, w_1, of acesulfame K, in milligrams per kilogram, using the following equation:

$$w_1 = \frac{A_2 \times m_1 \times F \times 10^6}{A_1 \times m_0} \quad \text{... (1)}$$

where:

A_2 is the absorption of the sample test solution (6.3.2);

A_1 is the absorption of the acesulfame K standard solution (6.3.1);

m_0 is the initial sample mass (6.2), in milligrams;

m_1 is the mass of the acesulfame K standard substance in 500 ml standard solution (4.3) in milligrams, (here: 4,0 mg);

F is the dilution factor, (here: 100).

7.2 Calculate the mass fraction, w_2 of acesulfame K, in milligrams per tablet, using the following equation:

$$w_2 = \frac{A_2 \times m_2 \times F \times m_1}{A_1 \times m_0} \quad \text{... (2)}$$

where:

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

A_2, F, m_1, A_1, m_0 see equation (1).

If the calculation is based on a calibration graph, an alternative calculative evaluation using the regression graph may be used.

Report the result 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 summa-rized 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.

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 = 3,2 mg/100 mg for commercially available acesulfame-K tablets.

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 = 3,7 mg/100 mg for commercially available acesulfame-K tablets.

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.

Annex A (informative)

Figure

Sample:	Acesulfame K	Function:	Absorption
Solvent:	Water	Wavelength range:	200 nm to 300 nm
Concentration:	8,0 mg/l		

Recognized maximum:

1: Wavelength = 228 nm Absorption = 0,484833

Figure A.1: Absorption spectrum of an acesulfame K standard solution in water

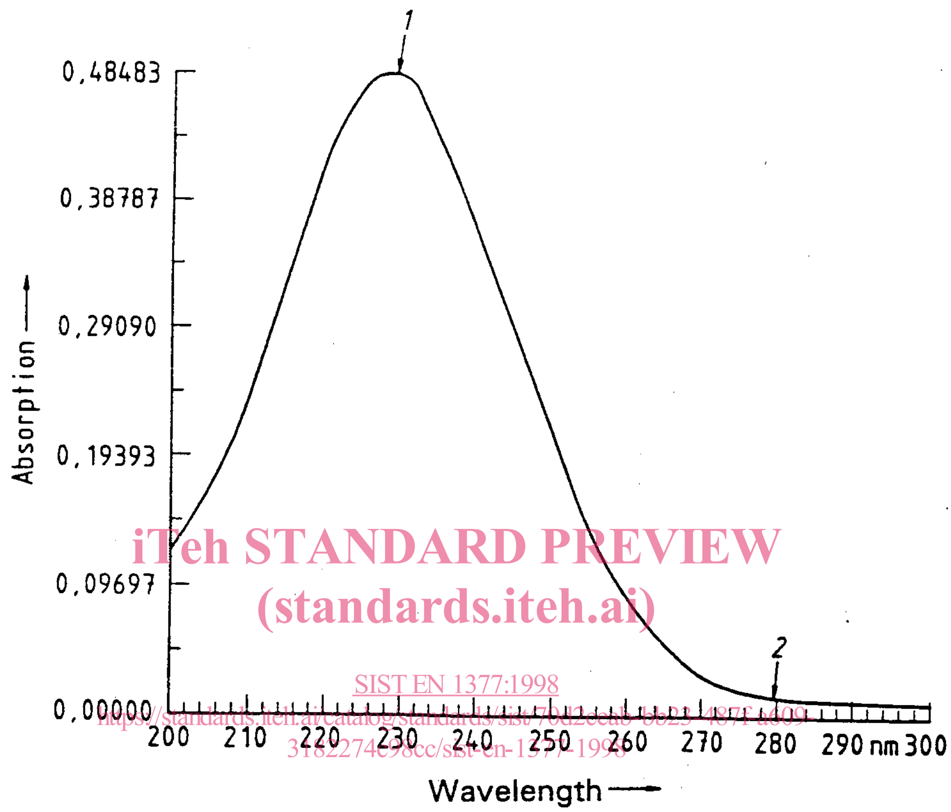
CORRECTED 1996-09-25

Annex A (informative)

Figure

Sample: Acesulfame K
Solvent: Water
Concentration: 8,0 mg/l

Function: Absorption
Wavelength range: 200 nm to 300 nm



Recognized maximum:

1: Wavelength = 228 nm   Absorption = 0,484833

Figure A.1: Absorption spectrum of an acesulfame K standard solution in water