SLOVENSKI STANDARD
SIST EN 16620:2015

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Analize živil - Določevanje furana v kavi in proizvodih iz kave s "headspace" plinsko kromatografijo in masno spektrometrijo (HS GC-MS)

Food analysis - Determination of furan in coffee and coffee products by headspace gas chromatography and mass spectrometry (HS GC-MS)

Lebensmittelanalytik - Bestimmung von Furan in Kaffee und Kaffee-Erzeugnissen mit Headspace-Gaschromatographie und Massenspektrometrie (HS GC-MS)

Analyse des produits alimentaires - Dosage du furane dans le café et les produits à base de café par chromatographie en phase gazeuse avec espace de tête couplée à la spectrométrie de masse (ET-CPG-SM)

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67.140.20	Kava in kavni nadomestki	Coffee and coffee substitutes

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Food analysis - Determination of furan in coffee and coffee products by headspace gas chromatography and mass spectrometry (HS GC-MS)

Analyse des produits alimentaires - Dosage du furane dans le café et les produits à base de café par chromatographie en phase gazeuse avec espace de tête couplée à la spectrométrie de masse (ET-CPG-SM)

Lebensmittelanalytik - Bestimmung von Furan in Kaffee und Kaffee-Erzeugnissen mit Headspace-Gaschromatographie und Massenspektrometrie (HS GC-MS)

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

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Foreword

This document (EN 16620:2015) has been prepared by Technical Committee 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of an identical text or by endorsement, at the latest by October 2015 and conflicting national standards shall be withdrawn at the latest by October 2015.

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1 Scope

This European Standard specifies a method for the determination of furan in coffee and coffee products with headspace-gas chromatography-mass spectrometry (HS-GC-MS), see [1] and [2]. Coffee products in the scope of this method are extracts which have been spray-dried, agglomerated or freeze-dried. The method has been validated in an interlaboratory study via the analysis of naturally contaminated samples of spray-dried coffee, freeze-dried coffee and ground roasted coffee ranging from 264 µg/kg to 2 840 µg/kg.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

3 Principle

Coffee and coffee products are weighed into headspace vials, diluted with water and incubated at 50 °C for 30 min. The furan content in the headspace is measured with HS-GC-MS. Quantification is done by using a standard addition curve, taking into account deuterated furan (*d4*-furan) as internal standard.

4 Reagents

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696:1995, unless otherwise specified. Solvents shall be of quality for HPLC analysis.

4.1 Furan, (CAS 110-00-9), mass concentration ρ = 0,936 g/ml.

4.2 *d4*-Furan, (CAS 6142-90-1), ρ = 0,991 g/ml.

4.3 Methanol.

4.4 Potassium hydroxide, aqueous solution, ρ = 250 g/l.

4.5 Stock solutions

4.5.1 Furan stock solution, ρ approximately 2,50 mg/ml.

Place 20 ml of methanol in a headspace vial and seal the vial. Weigh the sealed vial. Using a chilled syringe, transfer 50 µl of furan through the septum of the vial into the methanol. Reweigh the sealed vial. Mix the contents well. The solution is stable for 2 weeks at 4 °C. Pierced headspace vials shall be re-capped.

4.5.2 *d4*-Furan stock solution, ρ approximately 2,50 mg/ml.

Place 20 ml of methanol in a headspace vial and seal the vial. Weigh the sealed vial. Using a chilled syringe, transfer 50 µl of *d4*-furan through the septum of the vial into the methanol. Reweigh the sealed vial. Mix the contents well. The solution is stable for 2 weeks at 4 °C. Pierced headspace vials shall be re-capped.

4.6 Standard solutions

4.6.1 General

The amount of added solutions is calculated by differential weighing considering the densities. The solutions and additional dilutions should be kept cooled when working. Adapt the concentration of standard and internal standard solutions to the expected values in the samples. If necessary, prepare additional dilutions according to the working range. All furan and *d4*-furan solutions should be prepared in different rooms. Use separate syringes without plastic material.

4.6.2 **Furan standard solution 1**, *ρ* approximately 25 µg/ml.

Place 10 ml of water in a headspace vial and seal the vial. Weigh the sealed vial. Using a chilled syringe, transfer 100 µl of furan stock solution (4.5.1) through the septum of the vial into the water. Reweigh the sealed vial. Mix the contents well. Prepare the solution daily.

4.6.3 ***d4*-Furan standard solution 1**, *ρ* approximately 25 µg/ml.

Place 10 ml water in a headspace vial and seal the vial. Weigh the sealed vial. Using a chilled syringe, transfer 100 µl of *d4*-furan stock solution (4.5.2) through the septum of the vial into the water. Reweigh the sealed vial. Mix the contents well. Prepare the solution daily.

4.6.4 **Furan standard solution 2**, *ρ* approximately 250 ng/ml.

Place 10 ml water in a headspace vial and seal the vial. Weigh the sealed vial. Using a chilled syringe, transfer 100 µl of furan standard solution 1 (4.6.2) through the septum of the vial into the water. Reweigh the sealed vial. Mix the contents well. Prepare the solution daily.

4.6.5 ***d4*-Furan standard solution 2**, *ρ* approximately 250 ng/ml.

Place 10 ml water in a headspace vial and seal the vial. Weigh the sealed vial. Using a chilled syringe, transfer 100 µl of *d4*-furan standard solution 1 (4.6.3) through the septum of the vial into the water. Reweigh the sealed vial. Mix the contents well. Prepare the solution daily.

5 Apparatus

Usual laboratory glassware and equipment and, in particular, the following:

5.1 Positive displacement pipette.

5.2 Microlitre syringes.

5.3 Hand crimper and de-crimper.

5.4 Homogenizer or mill.

5.5 Test tube shaker.

5.6 Gas chromatograph for capillary gas chromatography, with split/splitless injector and headspace autosampler.

5.6.1 Fused silica capillary column, suitable for analysing volatile organic compounds e.g. non-polar polysiloxane with 5 % phenyl. The following column has been shown to be suitable: J&W DB-5 from Agilent[1)] length of 60 m, internal diameter (i.d.) of 0,32 mm and film thickness (d_f) of 1 µm.

5.6.2 PLOT column for separation of very volatile compounds, e.g. HP® Plot Q from Agilent[1)], length of 15 m, i.d. of 0,32 mm and d_f of 20 µm coupled to a restriction column HP-5MS from Agilent[1)], length of 30 m, i.d. of 0,25 mm and d_f of 0,25 µm. This column is an alternative for the column given under 5.6.1.

5.6.3 Rt®Q-Bond.[2)]

5.6.4 Carrier gas, helium.

5.6.5 Mass spectrometer with capability of single ion monitoring, coupled to gas chromatograph.

5.7 Headspace vials, 20 ml, with aluminium crimp seals and PTFE-faced silicone septa.

6 Procedure

6.1 Sample preparation

The samples shall be stored refrigerated in unopened original packages. Immediately prior to analysis the original packages should be opened and weighed with minimal delay to avoid losses of furan. Powdered samples should be weighed without further treatment. Coffee beans should be milled under conditions that do not raise the temperature and weighed. Although not tested in the interlaboratory study, the use of cooled milling equipment is recommended for milling in order to avoid losses of furan. Milling tests minimizing losses of furan should be performed ahead of the sample preparation.

6.2 Estimation of furan content

The furan content in a sample is quantified by means of standard addition.

The amount of furan and/or *d4*-furan which is added to the test portions is based on a rough estimation (x0) in relation to the initial sample weight.

This rough estimation (x_0) is done as follows: to 0,5 g to 5 g of the sample (normally 1 g) water is added (normally 5 ml), an amount of *d4*-furan (internal standard) in ng is added (approximately corresponding to the expected amount of furan in the sample) and is analysed with HS-GC-MS as described in 6.4.

Using the peak area ratio furan/*d4*-furan (m/z = 68/72, see 7.2) and the amount of added internal standard in ng the furan content in relation to the initial weight of the sample solution can be estimated (equivalent to a simple isotope dilution analysis).

In order to avoid the forming of furan from precursors, the pH value is adjusted to ca. pH = 10. For this 5 drops to 6 drops of potassium hydroxide solution (4.4) are added into the headspace vial.

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1) These are examples of suitable products available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of these products.

2) Rt®-Q-Bond is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product.

6.3 Standard addition analysis

In 9 headspace vials each 0,5 g to 5 g of the sample (normally 1 g) are weighed in, water is added (normally 5 ml) and *d4*-furan standard solution in relation to the order of magnitude of the estimated amount of furan (see 6.2) into the solution. Three headspace vials are capped immediately. The others are fortified with 0,5, 1,0 and 2,0 times of estimated furan content. The fortifying volume should not exceed 10 % of the total volume. For each new sample the concentration of the furan- and *d4*-furan standard solution (see 4.6) shall be adapted.

For an estimated furan content in the sample of 2,5 mg/kg (2,5 µg/g) and an initial weight of 1 g, the furan standard solution 1 and *d4*-furan standard solution 1 (see 4.6) can be used. Table 1 gives an example of a standard addition for an estimated furan content of 2,5 µg/g.

Table 1 — Example of a standard addition for an estimated furan content x_0 of 2,5 µg/g

Sample solution	*d4*-Furan standard solution µl	*d4*-Furan standard solution µg	Furan standard solution µl	Furan standard solution µg
P_{11} (0 x_0)	100	2,5	-	-
P_{12} (0 x_0)	100	2,5	-	-
P_{13} (0 x_0)	100	2,5	-	-
P_{21} (0,5 x_0)	100	2,5	50	1,25
P_{22} (0,5 x_0)	100	2,5	50	1,25
P_{31} (1,0 x_0)	100	2,5	100	2,5
P_{32} (1,0 x_0)	100	2,5	100	2,5
P_{41} (2,0 x_0)	100	2,5	200	5,0
P_{42} (2,0 x_0)	100	2,5	200	5,0

With the above fortifying procedure the *d4*-furan is added to the sample solutions according to the estimated furan content of the sample (here 2,5 µg/kg).

If necessary more than 2 or 3 headspace vials can be prepared for each fortifying level.

6.4 Determination with headspace-GC-MS

The following parameters should be kept:

— Incubation temperature: 50 °C

— Incubation time: 30 min

Headspace parameters:

The following headspace parameters are examples and shall be adopted to the used device:

— Needle temperature: 52 °C to 55 °C

— Shaker speed: 300 min^{-1}