SLOVENSKI STANDARD
SIST EN 16857:2017

01-julij-2017

Živila - Ugotavljanje benzena v brezalkoholnih pijačah, drugih pijačah in hrani za dojenčke na osnovi zelenjave s "headspace" plinsko kromatografijo z masno spektrometrijo (HS-GC-MS)

Foodstuffs - Determination of benzene in soft drinks, other beverages and vegetable-based infant foods by headspace gas chromatography mass spectrometry (HS-GC-MS)

Lebensmittel - Bestimmung von Benzol in Erfrischungsgetränken, anderen Getränken und in Babynahrung auf Gemüsebasis mit Headspace-Gaschromatographie/Massenspektrometrie (HS-GC-MS)

Produits alimentaires - Détermination de la teneur en benzène dans les boissons non alcoolisées, les autres boissons et les aliments pour nourrissons à base de légumes par chromatographie en phase gazeuse avec espace de tête couplée à la spectrométrie de masse (ET CG-SM)

Ta slovenski standard je istoveten z: EN 16857:2017

ICS:

67.160.20	Brezalkoholne pijače	Non-alcoholic beverages
67.230	Predpakirana in pripravljena hrana	Prepackaged and prepared foods
71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

SIST EN 16857:2017 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16857

May 2017

ICS 67.160.20; 67.230

English Version

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This European Standard was approved by CEN on 20 February 2017.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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European foreword

This document (EN 16857:2017) 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 November 2017, and conflicting national standards shall be withdrawn at the latest by November 2017.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the Europan Free Trade Association.

WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to take appropriate measures for ensuring the safety and health of the personnel prior to application of the standard and to fulfil statutory requirements for this purpose. Benzene has been classified by IARC as carcinogenic to humans (see [1]).

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of benzene in soft drinks, other beverages and vegetable-based infant foods, by headspace gas chromatography mass spectrometry (HS-GC-MS). The method has been validated in an interlaboratory study via the analysis of spiked samples of carbonated soft drink, still fruit-based drink, carbonated fruit-based drink, vegetable and fruit juice containing carrot, infant food vegetable based and infant food containing meat, ranging from 1,9 µg/kg to 18,6 µg/kg. However, linearity of the instrument response was proven for the concentration range from 0,5 µg/kg to 20 µg/kg. The limit of quantification (LOQ) depends on the instrument but can generally be expected to be in the range from 0,5 µg/kg to 1,0 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

The sample is homogenized, a test portion is heated in a closed system with isotopically-labelled benzene added as internal standard. A portion of the headspace is injected into a GC-MS system for identification and quantification. The injection is performed with a split-splitless injection port. The chromatographic separation is obtained on a mid-polarity capillary GC column. The benzene is ionized at 70 eV, recorded in selected ion monitoring (SIM) mode, and quantified by comparison with the stable isotope labelled analogue.

4 Reagents

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696:1995, unless otherwise specified. Prepare standard solutions preferably gravimetrically. Use an analytical balance (5.1) for the preparation of both native and stable isotope labelled benzene standard solutions.

4.1 Benzene, C_6H_6, purity is ≥ 99,0 % (CAS 71-43-2).

While not necessary, it is recommended to store at 4 °C to 6 °C to prevent evaporation. Bring to room temperature before use.

4.2 Benzene-*d_6*, purity is ≥ 99, 6 atom % D (CAS 1076-43-3).

While not necessary, it is recommended to store at 4 °C to 6 °C to prevent evaporation. Bring to room temperature before use.

4.3 Helium purified compressed gas, purity equivalent to 99,995 % or better.

4.4 Methanol, suitable for headspace GC analysis (CAS 67-56-1).

4.5 Preparation of stock and standard solutions

4.5.1 General

Prepare all standard solutions preferably gravimetrically. Record the tare masses of all recipients and the masses after each preparation step and use for calculation of the mass concentrations of standard solutions.

Volumetric preparation of standard solutions may also be applied provided that the volumetric glassware used complies with EN ISO 1042 (see [2]).

4.5.2 Benzene stock solution, mass concentration ρ approximately 2,20 mg/ml

Pipette 20 ml ± 0,1 ml of methanol into a 20 ml headspace vial (5.17) and seal the vial with a crimp cap. Weigh the sealed vial to the nearest 0,1 mg and record the weight as W_1. Using a microsyringe, transfer 50 µl of benzene (4.1) through the septum of the vial containing the methanol and shake vigorously or vortex to mix. Reweigh the sealed vial and record the weight (W_2) to the nearest 0,1 mg. Subtract W_1 from W_2 to determine the weight of benzene transferred (W_3). Calculate the mass concentration of stock solution from W_3 divided by the total volume (20,05 ml).

Alternatively, commercially available certified standard solutions may be used if available. Store the benzene stock solution at 4 °C to 6 °C but allow it to attain ambient temperature before use. Discard after 2 weeks. Once the septum on the stock solution has been pierced, the cap shall be replaced.

4.5.3 Benzene intermediate standard solution, ρ approximately 54 µg/ml

Using a microsyringe, transfer 500 µl of benzene stock solution (4.5.2) to a sealed headspace vial containing 20 ml ± 0,1 ml of methanol and shake vigorously. Prepare fresh daily. Calculate the exact mass concentration from the stock solution (4.5.2).

4.5.4 Benzene standard solution, ρ approximately 0,5 µg/ml

Using a microsyringe, transfer 200 µl of benzene intermediate standard solution (4.5.3) to a sealed headspace vial containing 20 ml ± 0,1 ml of water and shake vigorously. Prepare fresh daily. Calculate the exact concentration from the stock solution (4.5.2). Use this standard solution to prepare a 6-point calibration from 0,5 µg/kg to 20 µg/kg (see 4.6).

4.5.5 Benzene-d_6 internal standard (IS) stock solution, ρ approximately 2,36 mg/ml

Pipette 20 ml ± 0,1 ml of methanol into a 20 ml headspace vial (5.17) and seal the vial with a crimp cap. Weigh the sealed vial to the nearest 0,1 mg and record the weight as W_1. Using a microsyringe, transfer 50 µl of benzene-d_6 (4.2) through the septum of the vial containing the methanol and shake vigorously or vortex. Reweigh the sealed vial and record the weight (W_2) to the nearest 0,1 mg. Subtract W_1 from W_2 to determine the weight of benzene-d_6 transferred (W_3). Calculate the mass concentration of IS stock solution from W_3 divided by the total volume (20,05 ml).

Alternatively, commercially available certified standard solutions may be used if available. Store the benzene-d_6 IS stock solution at 4 °C to 6 °C but allow it to attain ambient temperature before use. Discard after 2 weeks. Once the septum on the IS stock solution has been pierced, the cap shall be replaced.

4.5.6 Benzene-d_6 internal standard (IS) solution, ρ approximately 4,2 µg/ml

Using a microsyringe, transfer 36 µl of benzene-d_6 IS stock solution (4.5.5) to a sealed headspace vial containing 20 ml ± 0,1 ml of water and shake vigorously. Prepare fresh daily. Calculate the exact mass concentration from the benzene-d_6 IS stock solution (4.5.5).

4.6 Preparation of the calibration solutions

Prepare calibration solutions of approximately 0 µg/kg, 0,5 µg/kg, 1 µg/kg, 2,5 µg/kg, 5 µg/kg, 10 µg/kg and 20 µg/kg benzene and approximately 10 µg/kg IS according to the following scheme.

Add 10 g ± 0,05 g of water to each of seven 20 ml headspace vials and transfer using a positive displacement pipette benzene standard solution (4.5.4), IS solution (4.5.6)and water according to the values given in Table 1. To avoid loss of benzene, seal the vials immediately following addition of the entire benzene and benzene-d_6 solutions and water.

Table 1 — Preparation of calibration solutions

Mass of water g	Volume of 0,5 µg/ml benzene (4.5.4) µl	Volume of 4,2 µg/ml benzene-d_6 (4.5.6) µl	Volume of water µl	Benzene mass concentration µg/kg	Benzene d_6 mass concentration µg/kg
10	0	25	400	0	10,1
10	10	25	390	0,5	10,1
10	20	25	380	1,0	10,1
10	50	25	350	2,5	10,1
10	100	25	300	5,0	10,1
10	200	25	200	10	10,1
10	400	25	0	20	10,1

5 Apparatus

WARNING — All glassware shall be meticulously cleaned (except disposable glassware).

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

5.1 Analytical balance, capable of weighing to the nearest of 0,0 001 g.

5.2 Top pan balance, capable of weighing to the nearest 0,001 g.

5.3 Headspace gas chromatography – mass spectrometry (HS-GC-MS) apparatus, comprising the following:

5.3.1 Headspace sampling and injection system, suitable for incubation temperatures of up to 60 °C and incubation time of 30 min. The headspace sampling needle should be suitable for maintenance at 100 °C and the transfer line at 105 °C.

5.3.2 Injection system, split-splitless injector, suitable for temperatures up to 250 °C.

A programmed temperature vaporizing (PTV) injector may be used as an alternative to a split/splitless injector. However, split/splitless injection was proven in the method validation study by collaborative trial suitable for the scope of this standard.

5.4 GC oven, suitable for temperatures up to 240 °C and capable of temperature programming.

5.5 Sample carousel, suitable for use with 20 ml glass headspace vials and caps (**5.17**).

5.6 GC capillary column, DB-VRX[1)] 6 %-cyanopropylphenyl-94 %-dimethylpolysiloxane length of 60 m, internal diameter of 0,25 mm, df = 1,4 µm (β = 44), or any column with comparable separation characteristics.

5.7 Interface to the mass spectrometer, with a temperature control device, suitable for temperatures up to 250 °C.

5.8 Mass spectrometer with the following characteristics:

— electron ionization source;

— ionization energy of 70 eV;

— mass resolution of at least 1 at each prescribed m/z value;

— temperature control devices for the ion source (230 °C) and the GC-MS interface (225 °C). Optionally a temperature control device for the quadrupole (150 °C);

— tuning stability of at least 48 h (allowing for the analysis of a sequence of samples and standards);

— response linearity range of at least two orders of magnitude.

5.9 Computer based instrument control system, capable of programming headspace sampling, gas chromatographic and mass spectrometric acquisition depending upon run time.

5.10 Data processing system, computer based.

5.11 Crimper, for sealing vial caps (5.17).

5.12 De-crimper, for removing vial caps (5.17).

5.13 Oven, forced-air maintained at 90 °C ± 1 °C.

5.14 Pipettes, glass, 10 ml and 20 ml capacity.

5.15 Pipettes, calibrated positive displacement, 10 µl to 400 µl capacity.

5.16 Syringes, calibrated precision glass, 50 µl, 100 µl, 250 µl and 500 µl capacity.

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1) This 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. Equivalent products may be used if they can be shown to lead to the same results.