SVENSK STANDARD
SS-EN 16852:2017

Fastställd/Approved: 2017-06-07
Publicerad/Published: 2017-06-08
Utgåva/Edition: 1
Språk/Language: engelska/English
ICS: 67.160.10

Livsmedel – Etylkarbamat i spritdrycker framställda av stenfrukter, fruktrester och andra alkoholhaltiga drycker, bestämning med GC-MS metod

Foodstuffs – Determination of ethyl carbamate in stone fruit spirits, fruit marc spirits and other spirit drinks – GC-MS method

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 16852

May 2017

ICS 67.160.10

English Version

Foodstuffs - Determination of ethyl carbamate in stone fruit spirits, fruit marc spirits and other spirit drinks - GC-MS method

Produits alimentaires - Détermination de la teneur en carbamate d'éthyle dans les eaux-de-vie de fruits à noyaux, les eaux-de-vie de marc de fruits et les autres boissons alcoolisées - Méthode par CG-SM

Lebensmittel - Bestimmung von Ethylcarbamat in Steinobstbränden, Obstbränden und anderen Spirituosen - GC-MS-Verfahren

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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European foreword

This document (EN 16852:2017) 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of an identical text or by endorsement, at the latest by November 2017, and conflicting national standards shall be withdrawn at the latest by November 2017.

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1 Scope

This European Standard specifies a gas chromatographic method using mass spectrometric detection for the determination of ethyl carbamate (EC) in stone fruit spirits, fruit marc spirits and other spirit drinks.

The method has been validated in an interlaboratory study for stone fruit spirits and fruit liqueurs, at levels ranging from 0,253 mg/l to 1,11 mg/l. However, linearity of the instrument response was proven for the concentration ranges 0,10 mg/l to 4,0 mg/l (simplified method) and 0,025 mg/l to 3,0 mg/l (procedure including sample clean-up), respectively.

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

Stone fruit spirits with a content of total dry extract of less than 10 g/l are injected directly into the gas chromatography mass spectrometry (GC-MS) system after the adjustment of the alcoholic strength of the beverage when indicated and addition of internal standard (ISTD). Sugared brandies, liqueurs and other spirit drinks with a higher total dry extract are first transferred onto a solid phase extraction (SPE) cartridge and the ethyl carbamate is eluted with a mixture of cyclohexane and ethyl acetate.

Stone fruit spirits can contain precursors of ethyl carbamate which get transformed into EC under the influence of sunlight, e.g. during the shelf life of a spirit. To obtain the actual content of EC in the sample, light-protected glass ware (e.g. brown glass) shall be used during the analysis.

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 (High Performance Liquid Chromatography) analysis.

Ethyl carbamate has been classified by IARC as probably carcinogenic to humans (see [1]).

4.1 Ethanol absolute.

4.2 Ethanol solutions.

4.2.1 Ethanol solution, volume fraction φ = 65 %.

Pipet 65 ml of ethanol (4.1) into a 100 ml volumetric flask and dilute to the mark with water.

4.2.2 Ethanol solution, φ = 35 %.

Pipet 35 ml of ethanol (4.1) into a 100 ml volumetric flask and dilute to the mark with water.

4.3 Cyclohexane.

4.4 Ethyl acetate.

4.5 Eluant, mixture of one part per volume of cyclohexane and one part per volume of ethyl acetate.

4.6 n-Pentane.

4.7 Solid phase material.

Approximately 20 g of a chemically inert, wide-pore and highly pure diatomaceous earth based solid phase, e.g. Extrelut® NT[1)]

4.8 Sodium chloride.

4.9 Ethyl carbamate, > 99 %.

4.10 Butyl carbamate.

4.11 d_5-Ethyl carbamate, > 99 %.

4.12 Ethyl carbamate stock solution, mass concentration ρ(EC) = 1 000 mg/l.

Weigh, to the nearest 0,01 mg, 50 mg of ethyl carbamate (4.9) into a 50 ml volumetric flask and dilute to the mark with ethanol (4.1).

4.13 Ethyl carbamate working solution 1, ρ(EC) = 100 mg/l.

Pipet 5 ml of ethyl carbamate stock solution (4.12) into a 50 ml volumetric flask and dilute to the mark with ethanol (4.1).

4.14 Ethyl carbamate working solution 2, ρ(EC) = 10 mg/l.

Pipet 5 ml of ethyl carbamate working solution 1 (4.13) into a 50 ml volumetric flask and dilute to the mark with ethanol (4.1).

4.15 Butyl carbamate stock solution, ρ(BC) = 1 000 mg/l.

Weigh, to the nearest 0,01 mg, 50 mg of butyl carbamate (4.10) into a 50 ml volumetric flask and dilute to the mark with ethanol (4.1).

4.16 Butyl carbamate working solution, ρ(BC) = 40 mg/l.

Pipet 2 ml of butyl carbamate stock solution (4.15) into a 50 ml volumetric flask. Add 33 ml of ethanol (4.1) and dilute to the mark with water.

4.17 d_5-Ethyl carbamate stock solution, ρ(d5-EC) = 1 000 mg/l.

Weigh, to the nearest 0,01 mg, 50 mg of d_5-ethyl carbamate (4.11) into a 50 ml volumetric flask and dilute to the mark with ethanol (4.1).

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4.18 d_5-Ethyl carbamate working solution, ρ(d5-EC) = 40 mg/l.

Pipet 2 ml of d_5-ethyl carbamate stock solution (4.17) into a 50 ml volumetric flask. Add 33 ml of ethanol (4.1) and dilute to the mark with water.

4.19 Ethyl carbamate standard solutions for simplified method, dry extract < 10 g/l

The injection of samples with a dry extract exceeding 10 g/l can lead to a rapid loss of sensitivity of the GC-MS system, depending on the type of liner and GC column used. For samples with higher dry extract content a sample clean up is recommended (see 6.2).

Prepare the ethyl carbamate standard solutions for the simplified method according to Table 1. In principle it is possible to prepare the ethyl carbamate standard solutions to an alcoholic strength with a volume fraction of 40 % or 70 % depending on the GC system.

For example, for an ethyl carbamate standard solution with a volume fraction of 70 %, pipet the amount of ethyl carbamate working solution (4.13 or 4.14) stated in Table 1 into a 10 ml volumetric flask and add the corresponding amount of ethanol (4.1). Fill the volumetric flask to the mark with an ethanol solution with a volume fraction of 65 % (4.2.1).

For an ethyl carbamate standard solution with a volume fraction of 40 %, use an ethanol solution with a volume fraction of 35 % (4.2.2) to fill up the volumetric flasks.

Table 1 — Preparation of the ethyl carbamate standard solutions for simplified method

Standard solution	**EC working solution 2 (4.14)** µl	**EC working solution 1 (4.13)** µl	**Ethanol (4.1)** µl	**ρ ethyl carbamate** mg/l
C0	0	-	500	0,00
C1	100	-	400	0,10
C2	200	-	300	0,20
C3	500	-	0	0,50
C4	-	80	420	0,80
C5	-	100	400	1,00
C6	-	200	300	2,00
C7	-	300	200	3,00
C8	-	400	100	4,00

Fill the 10 ml volumetric flasks to the mark and add 0,25 ml of butyl carbamate working solution (4.16) into each of them with a pipette (to give a total volume of 10,25 ml).

It is very important that the volumes of standard solutions and sample solutions (before an optional adjustment of the alcoholic strength, see 6.1) are equal. Usually, 10 ml are used.

Mix the solution thoroughly and transfer an aliquot into a GC vial and seal the vial with a septum cap.

4.20 Ethyl carbamate standard solutions for method using sample clean up

Pipet the amount of ethyl carbamate working solution as given in Table 2 into a GC vial and add the corresponding amount of ethanol (4.1) and 100 µl of d_5-ethyl carbamate working solution (4.18). Seal the GC vial with a septum cap and mix the content thoroughly.

The mass concentration of ethyl carbamate given in mg/l in Table 2 is related to a sample amount of 20 ml.

Table 2 — Preparation of ethyl carbamate standard solutions for method using sample clean up

Standard solution	**EC working solution 2 (4.14)** µl	**EC working solution 1 (4.13)** µl	**Ethanol (4.1)** µl	***ρ* ethyl carbamate** mg/l for a sample of 20 ml
C0	0	-	900	0,00
C1	50	-	850	0,025
C2	200	-	700	0,10
C3	500	-	400	0,25
C4	-	100	800	0,50
C5	-	150	750	0,75
C6	-	200	700	1,00
C7	-	400	500	2,00
C8	-	600	300	3,00

NOTE Data provided by the participants of the interlaboratory study has shown that it is possible to use different internal standards besides butylcarbamate (BC) as described above for the simplified method (e.g. d_5-ethyl carbamate, propyl carbamate (PC) or methyl carbamate (MC)).

5 Apparatus

Usual laboratory equipment and, in particular, the following. One-mark volumetric flasks should comply with EN ISO 1042:1999 [2].

5.1 Glass vials for GC analysis, brown glass with septum caps.

5.2 Columns for solid phase extraction (SPE), volume of 20 ml.

5.3 Rotary evaporator, with a water bath or a suitable concentration evaporator.

5.4 Gas chromatograph with mass spectrometric detector (GC-MS), capable of single ion monitoring (SIM), preferably equipped with a programmed temperature vaporizing (PTV) injector.

PTV injection allows injecting larger extract volumes compared to split/splitless (SSL) injection. This provides larger signal intensities and could be advantageous with regards to the limit of detection (LOD). However, split/splitless injection was proven suitable for the scope of this European Standard during the method validation study by collaborative trial.

5.5 Capillary column for gas chromatography, polyethylene glycol (PEG) stationary phase, e.g. DB WAX[2)], 30 m × 0,25 mm internal diameter × 0,25 µm film thickness.

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