



BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of EN 16852:2017.

The UK participation in its preparation was entrusted to Technical Committee AW/275, Food analysis - Horizontal methods.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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 GC-SM

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

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Contents

Page

European foreword.....	3
1 Scope	4
2 Normative references.....	4
3 Principle	4
4 Reagents	4
5 Apparatus.....	7
6 Procedure.....	8
7 Gas chromatography and mass spectrometry (GC-MS).....	9
8 Calculation	9
9 Precision.....	10
10 Test report.....	11
Annex A (informative) Typical chromatograms	12
Annex B (informative) Precision data	16
Bibliography	17

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of an identical text or by endorsement, at the latest by November 2017, and conflicting national standards shall be withdrawn at the latest by November 2017.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 $\varphi = 65\%$.

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, $\varphi = 35\%$.

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® NT¹⁾

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 $\rho(\text{EC}) = 1\,000\text{ mg/l}$.**

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, $\rho(\text{EC}) = 100\text{ mg/l}$.

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, $\rho(\text{EC}) = 10\text{ mg/l}$.

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, $\rho(\text{BC}) = 1\,000\text{ mg/l}$.

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, $\rho(\text{BC}) = 40\text{ mg/l}$.

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, $\rho(\text{d}_5\text{-EC}) = 1\,000\text{ mg/l}$.

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).

¹⁾ This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of these products. Equivalent products may be used if they can be shown to lead to the same results.