
Karakterizacija odpadkov - Določevanje izbranih alkoholov z nizkim vreliščem s plinsko kromatografijo s statično "headspace" tehniko in s plamensko ionizacijskim detektorjem (HS-GC-FID)

Characterization of waste - Determination of selected low boiling point alcohols using gas chromatography with flame ionization detection after static head-space extraction (HS-GC-FID)

Charakterisierung von Abfällen - Bestimmung von ausgewählten, niedrigsiedenden Alkoholen mittels Gaschromatographie mit Flammenionisationsdetektor nach statischer Headspace Extraktion (HS-GC-FID)

Caractérisation des déchets - Détermination d'une sélection d'alcools à bas point d'ébullition par chromatographie en phase gazeuse avec détection par ionisation de flamme après extraction statique par espace de tête (HS-GC-FID)

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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (CEN/TS 17847:2022) has been prepared by Technical Committee CEN/TC 444 “Environmental characterization of solid matrices”, the secretariat of which is held by NEN.

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CEN/TS 17847:2022 (E)**Introduction**

Directive 96/82/EC, as amended, requires transit/sorting/regrouping or waste treatment facilities to assess the hazardous nature of their waste, in particular, from the point of view of the following categories of hazards:

- very toxic or toxic to humans if swallowed, if there is contact with the skin or by inhalation, for example;
- very toxic or toxic to the aquatic environment.

Based on knowledge of the composition of elements and substances of a waste, its hazard potential can then be determined from the classification rules established for mixtures in waste legislation.

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1 Scope

This document specifies a method for quantitative determination of the concentration of selected alcohols with low boiling point in liquid waste and pasty waste by gas chromatography with flame ionization detection after static headspace extraction.

Under the conditions specified in this document, a limit of application of 20 mg/kg, expressed on dry matter for pasty waste and expressed on raw waste for liquid waste, can be achieved.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15934:2012, *Sludge, treated biowaste, soil and waste - Calculation of dry matter fraction after determination of dry residue or water content*

EN 16179:2012, *Sludge, treated biowaste and soil - Guidance for sample pretreatment*

EN 15002:2015, *Characterization of waste - Preparation of test portions from the laboratory sample*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <https://www.electropedia.org/>

4 Principle

This method describes a gas chromatography (GC) method using a flame ionization detection (FID) after an equilibrium-based static headspace preparation.

A specified quantity of extractant solution is added to a test portion of raw sample before any pretreatment in a hermetically sealed vial with sufficient space for desorption of analytes into the gas phase. The vial is heated in a thermostat at 80 °C until desorption equilibrium is reached.

A defined portion of the gas phase is injected into the gas chromatograph using two analytical columns with different stationary phases, and the analytes are detected by a flame ionization detector. The amount of each analyte is calculated using a calibration function established with external standards. An internal standard is added in the extractant solution, for the retention time verification.

The list of analytes is given in Table 1.

Table 1 — Analytes, with CAS RN and boiling point

Substance	CAS RN	Boiling point (°C)
Methanol (Me-OH)	67-56-1	64,5
Ethanol (Et-OH)	64-17-5	78,5
<i>iso</i> -Propanol (<i>i</i> -Pr-OH)	67-63-0	82,4
<i>tert</i> -Butanol (<i>t</i> -Bu-OH)	75-65-0	83,0
<i>n</i> -Propanol (Pr-OH)	71-23-8	97,1
<i>iso</i> -Butanol (<i>i</i> -Bu-OH)	78-83-1	108,0
<i>n</i> -Butanol (Bu-OH)	71-36-3	118,0

NOTE The method can be applied to other low boiling point analytes not specified in the scope provided suitability is proven by proper in-house validation.

5 Interferences

5.1 Interferences with sampling and extraction

Use sampling glass containers. During storage of the samples, losses of alcohols can occur due to volatilization. Sampling directly in head-space vials minimizes handling of samples and analytes loss.

Samples can be contaminated by diffusion of volatile organic compounds through the septum seal of the sample vial during shipment and storage. A transport blank, prepared from extractant solution and sample container and carried through sampling and handling protocols, serves as a check on such contamination.

5.2 Interferences with GC

Substances that co-elute with analytes can interfere with the determination. These interferences can lead to insufficient signal resolution and can, depending on their magnitude, affect accuracy and precision of the analytical results. Two analytical columns with different stationary phases (6.1.5) are used to confirm the identification of the selected alcohols.

6 Apparatus and reagents

6.1 Apparatus

Usual laboratory glassware.

All glassware and material that come into contact with the sample or extract shall be thoroughly cleaned.

6.1.1 Analytical balance, with a weighing accuracy of 0,1 mg

6.1.2 Gas chromatograph, with head-space autosampler

6.1.3 Flame ionization detector

6.1.4 Glass head-space vials, 10 to 20 ml, according to the type of headspace equipment, with hermetic crimp or screw cap and septum (silicone /polytetrafluoroethylene (PTFE))

6.1.5 Chromatographic columns, e.g. DB-Wax 60 m × 0,25 mm × 0,25 μm, CP-SIL8-CB (see Annex A)

6.1.6 Automatic syringe or bottle top dispenser

6.2 Reagents

All reagents shall be of recognized analytical grade. The purity of the reagents used shall be checked by running a blank test. The blank shall be less than 50 % of the lowest reporting limit.

6.2.1 Ultra pure water

6.2.2 Reference substances, as in Table 1

6.2.3 Dimethylformamide, C₃H₇NO

6.2.4 Sodium chloride, NaCl

6.2.5 Carrier gas, for gas chromatography, according manufacturer's indications

6.2.6 Silica sand, analytical grade

6.2.7 Stock solutions

6.2.7.1 Internal standard stock solution (50 g/l) for retention time verification

Prepare internal standard stock solution by dissolving exactly ca. 5 g of dimethylformamide (6.2.3) in 100 ml water (6.2.1) using a volumetric flask.

6.2.7.2 Extractant solution with internal standard (1000 mg/l)

Prepare extractant solution by dissolving 360 mg of sodium chloride (6.2.4) in ca. 800 ml of water (6.2.1) in a 1000 ml volumetric flask. Add 20 ml of internal standard stock solution (6.2.7.1). Make up the volume to 1 000 ml.

6.2.7.3 Calibration stock solutions

Prepare five or more calibration stock solutions by dissolving reference substances (6.2.2) in extractant solution (6.2.7.2) containing the internal standard at 1000 mg/l. The concentrations of the calibration stock solutions shall be such that the addition of the extractant solution volume of each to the headspace vials (6.1.4) will bracket the analytical range.

6.3 Sampling, storage and pretreatment

6.3.1 Sampling

6.3.1.1 Sampling for dry matter via water content determination, for pasty waste

Take a suitable sample to determine the dry matter via water content determination according to method b) of EN 15934:2012.

6.3.1.2 Sampling for selected alcohols content determination using vials pre-filled with extractant

For the preparation of samples, prepare a sufficient number of sampling vials, by pre-filling headspace vials (6.1.4) with extractant solution (6.2.7.2). Identify each vial, weigh to the nearest 0,01 g and record mass, m_v .

Transfer a mass of material to be analysed using an appropriate device into a preweighed pre-filled vial with a volume (ml)/mass (g) ratio around 10 keeping a sufficient headspace in the vial (6.1.4) with around 50 % of the total volume. Make sure that the sample is completely covered with extractant

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solution (6.2.7.2). Prevent leakages by cleaning the top of the vessel before sealing. Then close the cap with the PTFE coated septum.

The samples should be taken from undisturbed material using an appropriate sample cutter of known volume, e.g. a modified disposable plastic syringe with the tip cut off. The sample should be collected immediately after exposing a fresh surface of the material to be analysed. The mass of the sample and the volume (ml) /mass (g) ratio shall be calculated after sampling, according to the formula given at 7.3 and recorded.

At least one blank sample on every site shall be prepared in the field by opening the pre-filled vial for the same time period as necessary for the filling with sample. Add silica sand (6.2.6) and close the vial. If blank values are unusually high (more than 50 % of the lowest value of interest) the reason for these high blanks shall be investigated.

The sampling vials shall be kept dark (before and after sampling) and at $5^{\circ}\text{C} \pm 3^{\circ}\text{C}$ throughout the whole transportation.

All replicates shall be collected from each sampling point to allow for potential re-test. For low level analysis by direct vapor partitioning, 3 or 4 replicate samples shall be collected, while duplicate samples may be sufficient for high level analysis.

6.3.2 Storage

The samples taken in vials pre-filled with extractant solution (6.3.1.2) shall be stored in the dark, at $5^{\circ}\text{C} \pm 3^{\circ}\text{C}$ and shall be analysed within 8 days following the sampling.

6.3.3 Pretreatment

According to subclause 10.3.2.1 of EN 16179:2012 or Clause 4 and subclause 5.1 of EN 15002:2015, no sample pretreatment is carried out.

7 Procedure**7.1 Determination of dry matter via water content for pasty waste**

Determine dry matter *DM* via water content according to method b) of EN 15934 on the dedicated sample (6.3.1.1).

7.2 Calibration

Five or more calibration standards are prepared by placing a mass (in g) of silica sand (6.2.6), similar to the mass of materials to be analysed (6.3.1.2) in a head-space vial (6.1.4), adding the same volume of each calibration solution (6.2.7.3) to keep the volume (ml)/mass (g) ratio at 10, and sealing the vial.

The calibration standards are analysed in the same manner as the samples (7.4).

7.3 Mass of sample

Weigh the vial with cap to the nearest 0,01 g. Calculate the test sample mass according to Formula (1):

$$m_s = m_t - m_v \quad (1)$$

where

m_s is the mass of the sample, in g;

m_t is the total mass of the vial containing sample and extractant, with cap, in g;

m_v is the mass of the vial sample pre-filled with extractant, with cap, in g.