
Vpliv materialov na pitno vodo - Identifikacija GC-MS vodno lužljivih organskih snovi

Influence of materials on water intended for human consumption - GC-MS identification of water leachable organic substances

Identifizierung mittels GC-MS von durch Wasser auslaugbaren organischen Substanzen aus Materialien für den Kontakt mit Trinkwasser

Influence sur l'eau des matériaux en contact avec l'eau destinée à la consommation humaine - Identification par CG-SM de substances organiques lixiviables à l'eau

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Ta slovenski standard je istoveten z: EN 15768:2015

ICS:

| | | |
|-----------|---------------------------------------|---|
| 13.060.20 | Pitna voda | Drinking water |
| 67.250 | Materiali in predmeti v stiku z živil | Materials and articles in contact with foodstuffs |
| 71.040.50 | Fizikalnokemijske analitske metode | Physicochemical methods of analysis |

SIST EN 15768:2015**en,de**

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EUROPEAN STANDARD

EN 15768

NORME EUROPÉENNE

EUROPÄISCHE NORM

January 2015

ICS 13.060.20; 67.250; 71.040.50

English Version

Influence of materials on water intended for human consumption - GC-MS identification of water leachable organic substances

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EN 15768:2015 (E)**Foreword**

This document (EN 15768:2015) has been prepared by Technical Committee CEN/TC 164 "Water supply", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by July 2015 and conflicting national standards shall be withdrawn at the latest by July 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] 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 European Free Trade Association.

This document has been prepared on the basis of co-normative research project EVK1-CT 2000-00052 and work funded by DG Enterprise and Industry (Grant Agreement S12.403892).

This document describes methods of identification only, and should not be used or quoted as a specification.

References to this document should indicate that the methods of identification used are in accordance with EN 15678:2015.

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Introduction

Organic substances that migrate from products containing such substances have, when used in contact with water intended for human consumption, the potential to cause health concerns for consumers. The potential health effects of these chemicals are assessed in three stages as follows:

- a) preparation of migration waters by exposing a portion of the material to water under controlled conditions;
- b) analysis of the migration waters;
- c) assessment of the identities and concentrations of the substance detected.

The analysis of organic substances present in migration waters can involve two different types of analytical methods as follows:

- d) a screening method, which allows a variety of substances to be detected and a semi-quantitative assessment to be made of their concentrations;
- e) accurate quantitative methods for the determination of specific target substances known to be present in the chemical formulations of the materials.

This standard describes the analytical procedures based upon gas chromatography and mass spectrometry (GC-MS) used to screen migration waters for organic substances derived from finished products such as pipes, protective coatings, membranes, etc. This method is suitable for migration waters from all products that can potentially release organic chemicals into water when they are used in contact with water intended for human consumption, and which are the subject of an application for approval by the national regulatory body. It may be one of several methods that form part of the overall approval process. The method may also be used as part of an approval audit process. The method does not provide accurate quantitative results and other analytical methods are recommended for accurate quantitative determination of specific target substances.

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EN 15768:2015 (E)

1 Scope

This European Standard describes a method for detecting and identifying organic chemicals that are amenable to GC-MS analysis using the procedures described and which can migrate from a product into water intended for human consumption. This European Standard does not provide all the necessary tools to completely identify all the substances that are detected. A method of semi-quantitatively estimating the concentrations of the organic substances detected is also provided, however, concentrations should only be seen as indicative.

NOTE The method to be used for the preparation of migration waters is specified by separate ENs, as noted below.

2 Normative references

EN 12873-1, *Influence of materials on water intended for human consumption - Influence due to migration - Part 1: Test method for factory-made products made from or incorporating organic or glassy (porcelain/vitreous enamel) materials*

EN 12873-2, *Influence of materials on water intended for human consumption - Influence due to migration - Part 2: Test method for non-metallic and non-cementitious site-applied materials*

EN 12873-3, *Influence of materials on water intended for human consumption - Influence due to migration - Part 3: Test method for ion exchange and adsorbent resins*

EN 12873-4, *Influence of materials on water intended for human consumption - Influence due to migration - Part 4: Test method for water treatment membranes*

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

3 Terms and definitions

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For the purposes of this document, the following terms and definitions apply.

3.1

u

atomic mass unit, defined as 1/12 of the mass of a single atom of carbon-12 in the gas phase (i.e. unbound), at rest and in its ground state

3.2

asymmetry factor

A_s

measure of the absorption of a compound during gas chromatographic analysis

Note 1 to entry: The asymmetry factor (*A_s*) can be derived from Formula (1).

$$A_s = \frac{(a + b)}{2b} \quad (1)$$

where

- a* is the distance from the leading edge of the peak at the point on the baseline where the perpendicular dropped from the peak maximum crosses it;
- b* is the corresponding distance from the trailing edge of the peak.

Locate the apex of the peaks that require their asymmetry values calculated. For each peak, drop a perpendicular line down at a right-angle to the baseline.

Note 2 to entry: Some manufacturer's GC-MS software packages allow the calculation of peak asymmetries to be produced automatically. Check the criteria used for the measurement, as some packages perform the calculation at 5 % of the peak height.

3.3

electron impact ionization

ionization by a beam of electrons

3.4

GC-MS

analytical instrument comprising a gas chromatograph (GC) linked to a mass spectrometer (MS)

3.5

GC-MS general survey analysis

acquisition of a series of mass spectra (up to several thousand) during the course of a gas chromatographic run, by operating the mass spectrometer in a continuous cyclic scanning mode over a wide m/z range

3.6

injection standard

organic substance added to the final solvent extract prior to analysis

Note 1 to entry: An injection standard is added to allow the recoveries of the internal standards to be calculated, by providing a means of normalizing the GC-MS responses for solvent extracts and GC-MS standards.

3.7

internal standard

organic substance added to the migration water at a known concentration prior to the commencement of the analysis

Note 1 to entry: Internal standards are added for the following reasons (a) to demonstrate that the analysis has been undertaken successfully, and (b) to provide a reference to allow other substances detected to be quantified. Ideally, the internal standards should not be present in the migration waters being analysed; for this reason, isotopically labelled standards are used.

3.8

test water

water used to prepare the migration water, as specified in EN 12873-1, EN 12873-2, EN 12873-3 or EN 12873-4 depending on the material being tested

3.9

laboratory blank

water, as specified in EN ISO 3696, known to contain negligible levels of contamination, to which internal standards (3.7) have been added and which is then analysed in the same way as the migration water

Note 1 to entry: Laboratory blanks are used to check for potential contamination of either migration waters or solvent extracts which may occur within the laboratory during the course of the analysis.

3.10

migration water

aqueous solution that results from leaving test water in contact with the test material under the specified test conditions

3.11

procedural blank

aqueous solution that results from leaving test water in contact with tanks or containers identical to those used to prepare the migration water in the absence of the test material

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Note 1 to entry: Procedural blanks are used to check for potential contamination of migration waters that can arise during the migration procedure. For example, substances can migrate from the tanks or containers used into the test water, or aerial contamination can occur if volatile substances are present in the laboratory atmosphere. Further details are provided in the European Standards regarding the preparation of migration waters.

3.12**mass spectrometric resolution**

measure of the capability of a mass spectrometer to correctly distinguish two mass spectral peaks, having sequential mass to charge (m/z) values, as separate peaks

Note 1 to entry: If $z = 1$, this is generally denoted by $m_2 / (m_2 - m_1)$, where m_2 has the higher m/z value and m_1 has the lower m/z value.

Note 2 to entry: Mass spectrometer set up so that the resolution is 650 will satisfactorily resolve and assign the correct masses to mass spectral peaks at m/z 649 and m/z 650. Although the mass spectrometric resolution of a mass spectrometer using a magnetic field for mass resolution remains constant throughout the mass range scanned, a quadrupole mass spectrometer is usually operated so that unit mass resolution is achieved over the mass range scanned (i.e. at m/z 200 the resolution is 200, whereas at m/z 500 the resolution is 500).

3.13 **m/z**

mass (m)-to-charge (z) ratio of an ion

Note 1 to entry: As most ions produced by electron impact ionization are singly charged, this ratio usually corresponds to the mass of an ion. However, exceptionally, ions can possess multiple charges.

3.14**solvent extract**

solution containing substances partitioned from the migration waters into the extraction solvent (in this case dichloromethane)

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3.15**total ion current****TIC**

sum of all the separate ion currents carried by the individual ions contributing to a single mass spectrum

3.16**TIC chromatogram**

graphical representation of the TIC versus time

3.17**Linear Retention Index**

$$\text{Linear Retention Index} = (n \times 100) + 100 \times \left(\frac{(RT_x - RT_n)}{(RT_{n+1} - RT_n)} \right) \quad (2)$$

where

n is the number of carbon atoms in n-alkane prior to substance of interest;

RT_n is the retention time (min) of n-alkane;

RT_{n+1} is the retention time (min) of next n-alkane;

RT_x is the retention time (min) of substance of interest

3.18**threshold value**

concentration below which detection and/or identification is uncertain

4 Principle

A mixture of isotopically-labelled internal standards is added to each of the migration waters to be analysed immediately before the extraction solvent dichloromethane is added. After solvent extraction, the extract is concentrated, an injection standard added, and the resultant concentrated extract is then analysed by GC-MS to determine the identity of any organic chemicals that may be present above a pre-set threshold value.

The mass spectrometer is used in a repetitive full-scan mode operating with positive electron impact ionization and the mass spectra produced are recorded by, and stored on, the GC-MS data system.

Wherever possible, each substance detected above the threshold value is identified, and semi-quantified by reference of its TIC to the TIC responses obtained for the internal standards.

NOTE The procedure to be used to identify organic substances from their mass spectra is given in Annex B.

5 Reagents**5.1 General**

Only reagents of analytical grade shall be used, except where specified otherwise. All reagents shall be of sufficient purity to ensure that they do not give rise to interferences during the GC-MS analysis.

NOTE Contamination can arise from various sources, e.g. the laboratory atmosphere, glassware, plastics and rubber materials. The use of procedural blanks and laboratory blanks assists in detecting and identifying the source of any contamination and are used to correct results for the effects of contamination.

5.2 Required reagents

5.2.1 Reagent water, having a conductivity of < 2 mS/m, a total organic carbon content of $< 0,2$ mg/l carbon, and free from organic contaminants which can interfere with the GC-MS analysis of the extracts. Suitable water can be prepared by reverse osmosis, de-ionization or distillation.

5.2.2 Hydrochloric acid, concentrated (density 1,18 g/ml).

5.2.3 Hydrochloric acid solution (6 mol/l), prepared as follows:

- slowly add $(0,5 \pm 0,01)$ l of concentrated hydrochloric acid (5.2.2) to $(0,5 \pm 0,01)$ l of reagent water (5.2.1);
- this solution should be replaced on a six monthly basis.

NOTE Care is needed in preparing this solution which can generate heat.

5.2.4 Sulfuric acid, concentrated (density 1,84 g/ml).

5.2.5 Sulfuric acid solution (0,5 mol/l), prepared as follows:

- slowly add $(14,0 \pm 0,5)$ ml of sulfuric acid (5.2.4) to (300 ± 5) ml of reagent water (5.2.1) and make up to (500 ± 5) ml with reagent water;
- this solution should be replaced on a yearly basis.

NOTE Care is needed in preparing this solution which can generate heat.

EN 15768:2015 (E)**5.2.6 Sodium hydroxide solution (0,5 mol/l), prepared as follows:**

- dissolve (20,0 ± 0,1) g of sodium hydroxide pellets in reagent water (5.2.1) and make up to 1 l;
- replace this solution on a two weekly basis.

NOTE Care is needed in preparing this solution which can generate heat.

5.2.7 Dichloromethane, glass distilled grade, batches should not be used until they have been verified as free from significant contamination, see Note 2.

NOTE 1 Other grades can be suitable but it is necessary to demonstrate that any impurities present do not interfere with the detection of substances of interest or the internal standards, or introduce unacceptable contamination, see Note 2.

NOTE 2 Generally any peak in the solvent after contaminants have been concentrated by evaporation (200 ml of solvent reduced to 500 µl) with an estimated concentration greater than 1 µg/l is likely to interfere with the method.

5.2.8 Acetone, glass distilled grade, batches should not be used until they have been verified as free from significant contamination, see Note 2.

NOTE 1 Other grades can be suitable but it is necessary to demonstrate that any impurities present do not interfere with the detection of substances of interest or the internal standards, or introduce unacceptable contamination, see Note 2.

NOTE 2 Generally any peak in the solvent after the acetone has been diluted 5 times with dichloromethane with an estimated concentration greater than 1 µg/l is likely to interfere with the method.

5.2.9 Ascorbic acid solution, prepared as follows:

- dissolve (4,0 ± 0,1) g of ascorbic acid in reagent water (5.2.1) and make up to 1 l. Before use, extract the ascorbic acid solution with two 100 ml aliquots of dichloromethane, discarding the solvent afterwards. Transfer the ascorbic acid solution to a suitable screw-capped bottle. The solution is stable for use for one month after preparation.

5.2.10 Sodium sulphate (anhydrous), prepared as follows:

- remove any organic contaminants by heating at (500 ± 50) °C for ≥ 4 h, and store so that rehydration is minimized and re-contamination cannot occur.

5.2.11 Internal standards

Use the following isotopically-labelled (≥98 atom % D) substances:

- d₆-benzene;
- d₂₁-2,6-di-*t*-butyl-4-methylphenol (d₂₁-BHT);
- d₅-chlorobenzene;
- d₃₄-hexadecane;
- d₈-naphthalene;
- d₁₀-phenanthrene;
- d₅-phenol;