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**Gradbeni proizvodi - Ocenjevanje sproščanja nevarnih snovi - Določanje policikličnih aromatskih ogljikovodikov (PAH) ter benzena, toluena, etilbenzena in ksilena (BTEX) - Metoda plinske kromatografije z masno spektrometrijo**

Construction products - Assessment of release of dangerous substances - Determination of the content of polycyclic aromatic hydrocarbons (PAH) and of benzene, toluene, ethylbenzene and xylenes (BTEX) - Gas chromatographic method with mass spectrometric detection

Bauprodukte - Bewertung der Freisetzung von gefährlichen Stoffen - Bestimmung des Gehalts an polyzyklischen aromatischen Kohlenwasserstoffen (PAK) und an Benzol, Toluol, Ethylbenzol und Xylol (BTEX) - Gas-chromatographisches Verfahren mit massenspektrometrischer Detektion

Produits de construction - Évaluation de l'émission de substances dangereuses - Détermination de la teneur en hydrocarbures aromatiques polycycliques (HAP) et en benzène, toluène, éthylbenzène et xylènes (BTEX) - Chromatographie en phase gazeuse avec détection par spectrométrie de masse

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Construction products: Assessment of release of dangerous substances - Determination of the content of polycyclic aromatic hydrocarbons (PAH) and of benzene, toluene, ethylbenzene and xylenes (BTEX) - Gas chromatographic method with mass spectrometric detection

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This European Standard was approved by CEN on 14 August 2023.

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**EN 17844:2023 (E)****European foreword**

This document (EN 17844:2023) has been prepared by Technical Committee CEN/TC 351 “Construction products: Assessment of release of dangerous substances”, the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by May 2024, and conflicting national standards shall be withdrawn at the latest by May 2024.

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 Standardization Request given to CEN by the European Commission and the European Free Trade Association.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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## Introduction

This document deals with the determination of the content of polycyclic aromatic hydrocarbons (PAH) and of benzene, toluene, ethylbenzene and xylenes (BTEX) with gas chromatography with mass spectrometric detection (GC-MS). NEN 7331 has been used as basis.

This document is intended to be used for construction products and is suitable for determining:

- the full suite PAH, including the EPA-PAH series ([EPA 8100]);
- six BTEX.

In some cases, additional analysis with high performance liquid chromatography (HPLC) can be necessary to determine a number of compounds.

The methods described have been subjected to robustness validation [Van De Weghe et al., 2018]. The detectability limit of the methods for individual compounds in construction products for PAH is 0,5 mg/kg to 1,5 mg/kg and for BTEX 0,1 mg/kg.

This document is part of a modular horizontal approach and belongs to the analytical step. An overview of all modules which belong to a chain of measurement and the manner how modules are selected is given in CEN/TR 16220.

In the growing amount of product and sector-oriented test methods it was recognized that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that can be used for all materials and products or for all materials and products with certain specifications.

In this context a horizontal modular approach is adopted in CEN/TC 351. “Horizontal” means that the methods can be used for a wide range of materials and products with certain properties. “Modular” means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole “chain of measurement” (from sampling to analyses). A beneficial feature of this approach is that “modules” can be replaced by better ones without jeopardizing the standard “chain”.

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics, it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the entire test procedure.

**EN 17844:2023 (E)****1 Scope**

This document describes two methods for determining the content of polycyclic aromatic hydrocarbons (PAH) and one method for determining the content of benzene, toluene, ethylbenzene and xylenes (BTEX) with gas chromatography with mass spectrometric detection (GC-MS).

See Annex A (normative) for lists of PAH and BTEX that can be determined with this document.

This document is intended to be used for construction products.

In a number of cases additional analysis with high performance liquid chromatography (HPLC) can be necessary to determine a number of compounds. To determine PAH multiple liquid-liquid extraction is used to remove interfering compounds, e.g. maltenes. The tests that led to this document were carried out on different types of roofing material, bitumen and bituminous binders as well as asphalt including one tar containing asphalt (see [Van De Weghe et al., 2018] and [García-Ruiz et al., 2020]).

The detectability limit of the methods for individual compounds in roofing material, asphalt and tar containing asphalt for PAH is 0,5 mg/kg to 1,5 mg/kg and for BTEX 0,1 mg/kg.

**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 58, *Bitumen and bituminous binders — Sampling bituminous binders*

EN 12594, *Bitumen and bituminous binders — Preparation of test samples*

EN 16687:2023, *Construction products: Assessment of release of dangerous substances — Terminology*

EN 17087, *Construction products: Assessment of release of dangerous substances — Preparation of test portions from the laboratory sample for testing of release and analysis of content*

EN ISO 15009, *Soil quality — Gas chromatographic determination of the content of volatile aromatic hydrocarbons, naphthalene and volatile halogenated hydrocarbons — Purge-and-trap method with thermal desorption (ISO 15009)*

ISO 20595, *Water quality — Determination of selected highly volatile organic compounds in water — Method using gas chromatography and mass spectrometry by static headspace technique (HS-GC-MS)*

**3 Terms and definitions**

For the purposes of this document, the terms and definitions given in EN 16687:2023 and the following apply.

ISO and IEC maintain terminology 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/>



### 3.1

#### **asphalt**

homogenous mixture typically of coarse and fine aggregates, filler aggregate and bituminous binder which is used in the construction of a pavement

Note 1 to entry: Asphalt can include one or more additives to enhance the laying characteristics, performance or appearance of the mixture.

[SOURCE: EN 13108-1:2016, 3.1.8]

### 3.2

#### **bitumen**

virtually not volatile, adhesive and waterproofing material derived from crude petroleum, or present in natural asphalt, which is completely or nearly completely soluble in toluene, and very viscous or nearly solid at ambient temperatures

Note 1 to entry: Some grades of bitumen are used in both paving and industrial applications, e.g. certain penetration-graded bitumens are used for industrial purposes such as in the manufacture of roofing felts and other waterproofing membranes.

[SOURCE: EN 12597:2014, 2.2]

### 3.3

#### **blank value**

test result obtained by carrying out the test procedure in the absence of a test portion

[SOURCE: EN 16687:2023, 3.3.1.10; modified – Note 1 to entry removed]

### 3.4

#### **clean-up**

purification of a crude extract to remove interfering compounds

[SOURCE: EN 16687:2023, 3.2.2.27; modified – Note 1 to entry removed]

### 3.5

#### **external standard**

known quantity of the target analytes that is measured in the same series as the solution to be measured and is used for identification and quantification

[SOURCE: EN 16687:2023, 3.2.2.20]

### 3.6

#### **extract**

solution resulting from extraction of a sample with a solvent

[SOURCE: EN 16687:2023, 3.2.2.13]

### 3.7

#### **extraction**

dissolution of substances in a solvent for subsequent chemical analysis

[SOURCE: EN 16687:2023, 3.2.2.14; modified – Note 1 to entry removed ]

**EN 17844:2023 (E)****3.8****final extract**

solution that is obtained after clean-up of the Soxhlet extract through a purification stage

[SOURCE: EN 16687:2023, 3.2.2.18]

**3.9****injection standard**

known quantity of a substance (where applicable deuterated) not present in the sample, that after the clean-up step is added to the analytical portion

[SOURCE: EN 16687:2023, 3.2.2.19]

**3.10****laboratory sample**

sample or subsample(s) sent to or received by the laboratory

[SOURCE: EN 16687:2023, 3.2.2.1; modified – Notes to entry removed]

**3.11****method detection limit****MDL**

lowest analyte concentration that can be detected with a specified analytical method including sample preparation with a defined statistical probability

[SOURCE: EN 16687:2023, 3.3.1.12; modified – Note 1 to entry removed]

**3.12****product matrix**

main composition of the product dictating the manner of sample preparation and the type of digestion or extraction for later chemical analysis

[SOURCE: EN 16687:2023, 3.1.1.2; modified – Note 1 to entry removed]

**3.13****sample**

portion of material selected from a larger quantity of material

[SOURCE: EN 16687:2023, 3.2.1.5; modified – Notes to entry removed]

**3.14****Soxhlet extract**

solution that is obtained after extraction of a solid subsample by the Soxhlet technique for determining organic compounds

[SOURCE: EN 16687:2023, 3.2.2.17]

**3.15****Soxhlet extraction**

chemical pre-treatment of a solid subsample, where the organic compounds to be determined are dissolved by the Soxhlet technique

[SOURCE: EN 16687:2023, 3.2.2.16]

**3.16****surrogate standard**

known quantity of a substance (where applicable isotope labelled) not present in the sample, which is added to the Soxhlet extract in order to determine the recovery

[SOURCE: EN 16687:2023, 3.2.2.26]

**3.17****test portion**

analytical portion

amount of the test sample taken for testing/analysis purposes, usually of known dimension, mass or volume

[SOURCE: EN 16687:2023, 3.2.2.3; modified – Examples removed]

**3.18****test sample**

analytical sample

sample, prepared from the laboratory sample, from which test portions are removed for testing or for analysis

[SOURCE: EN 16687:2023, 3.2.2.2]

**4 Abbreviations**

For the purposes of this document, the following abbreviations apply.

BTEX Alkylated benzenes: sum of benzene, toluene, ethylbenzene and xylenes  
NOTE 1 A list of BTEX is in Table A.2.

DTL Detection limit

GC Gas chromatography

HPLC High performance liquid chromatography  
NOTE 2 High pressure liquid chromatography is an (outdated) synonym.

HRGC High-resolution gas chromatography

LLE Liquid-liquid extraction, also known as solvent extraction and partitioning

LOD Limit of detection

MQL Method quantification limit

MS Mass spectrometry;  
Mass selective detection

PAH Polycyclic aromatic hydrocarbon(s)  
NOTE 3 A list of PAHs is in Table A.1.

PLE Pressurized liquid extraction

SIM Single ion monitoring

SLE Solid-liquid extraction

**EN 17844:2023 (E)****5 Sample preparation**

To obtain test samples for extraction (and analysis) guidance on sample preparation as specified in EN 17087 shall be applied. The sample shall be analysed for the total content of substances of interest.

Precautions shall be taken before and during transport of the laboratory sample as well as during the time in which the samples are preserved in the laboratory before being analysed, to avoid alteration of the sample (see CEN/TR 16220).

Extracts are susceptible to change due to physical or chemical reactions which can take place between the time of extraction and the analysis.

It is therefore essential to take the necessary precautions to minimize these reactions and in the case of many parameters to analyse the extract with a minimum of delay. The maximum delay is given in the respective analytical standards.

**6 Principle****6.1 Flow chart**

The method for determination of PAH and BTEX is summarized schematically in Annex E (informative).

**6.2 Sample pre-treatment**

The quantity of construction product that is processed is reduced prior to grinding by quartering according to EN 17087. This procedure is repeated until a suitable quantity of subsample is obtained. The construction product is reduced cryogenically prior to analysis according to EN 17087. For some samples such as for very open asphalt concrete, cryogenic pre-reduction with a jaw crusher may be performed.

If bitumen, bituminous products or bitumen containing materials are tested according to this document, a representative subsample shall be taken as described in EN 58 and EN 12594. Pre-treatment may be chosen here according to the methods described above, e.g. for bitumen and bituminous binders, but for some materials direct sampling is also permitted.

**6.3 Extraction and sample pre-treatment****6.3.1 Determination of PAH**

A known quantity of the homogenized sample is extracted with petroleum ether (7.14) for about 16 h with a Soxhlet extraction set-up. Part of the extract is purified according to one of the two methods described and the final extract is then analysed for PAH with GC-MS.

**6.3.2 Determination of BTEX**

A known quantity of the homogenized sample is extracted with methanol (7.1) for about 24 h. The final extract is analysed after dilution with water with the purge-and-trap method and GC-MS or headspace-GC-MS.

**6.4 Gas chromatography determination****6.4.1 Determination of PAH**

For the separation a column with a slightly polar stationary phase is used.

NOTE Examples are also given in 11.2.8 and in Annex D (informative).

Injection takes place via a non-discriminating technique, such as on-column injection or splitless injection. Detection takes place using mass spectrometry. Calibration and quantification take place by measurement of an external standard.