BS EN 17844:2023

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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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National foreword

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The UK participation in its preparation was entrusted to Technical Committee B/557, Construction products - Assessment of dangerous substances.

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

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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

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

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

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Full document details: BS EN 17844:2023

Contents

Page

European foreword 4
Introduction 5
1 Scope 6
2 Normative references 6
3 Terms and definitions 6
4 Abbreviations 9
5 Sample preparation 10
6 Principle 10
6.1 Flow chart 10
6.2 Sample pre-treatment 10
6.3 Extraction and sample pre-treatment 10
6.3.1 Determination of PAH 10
6.3.2 Determination of BTEX 10
6.4 Gas chromatography determination 10
6.4.1 Determination of PAH 10
6.4.2 Determination of BTEX 11
7 Reagents 11
8 Equipment and devices 14
9 Sampling 15
10 Preservation and pre-treatment of samples 15
10.1 Sealant and roofing material 15
10.2 Other construction products 16
11 Method 16
11.1 Choice of method 16
11.2 Method for determining PAH 16
11.3 Method for determining BTEX 19
11.4 Calibration 20
11.5 Identification 20
11.5.1 Criteria 20
11.5.2 Gas chromatography criterion 20
11.5.3 Mass spectrometry criterion 21
11.6 HPLC analysis 21
12 Calculation of results 22
12.1 Calculation of individual PAH contents 22
12.2 Calculation of PAH contents with correction for recovery 22
12.3 Calculation of individual BTEX contents 23
12.4 Rounding 23
12.5 Calculation of resolution of the different compounds 23
12.6 Recovery 24

Documents
Document Sharing Hub
Full document details: BS EN 17844:2023

13 Test performance 24
14 Test report 25
Annex A (normative) List of PAH and BTEX 27
Annex B (informative) Chromatography tube for clean-up of an extract 28
Annex C (informative) Validation results for content of PAH and BTEX in construction products 29
C.1 General 29
C.2 Precision data for content of PAHs and BTEX in construction products 29
Annex D (informative) Example settings that can serve as a starting point for the analysis and detection of PAH and BTEX 34
D.1 Example settings for the analysis and detection of PAH 34
D.2 Example settings for the analysis and detection of BTEX 35
Annex E (informative) Flowchart for determining PAH and BTEX 37
Bibliography 39

Documents
Document Sharing Hub
Full document details: BS EN 17844:2023

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.

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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.

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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 at el., 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/

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Full document details: BS EN 17844:2023

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]

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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]

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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

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Full document details: BS EN 17844:2023

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.

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The hexane extract of clean-up method PAH 1 (7.19), that is reprocessed for the gas chromatographic determination with mass selective detection, may, after additional clean-up with aluminium oxide and conversion to acetonitrile, further be tested with HPLC for the determination of the chrysene content.

6.4.2 Determination of BTEX

A known quantity of the final extract is placed in the purge vessel after dilution with water. In the case of online systems, the sample is transferred into a suitable sample bottle and injected by the equipment into the purge vessel. A known quantity of the injection standard is then placed in the purge vessel. After injection via an injection technique such as purge-and-trap, the compounds to be measured are separated on a slightly polar or medium polar column and detected with mass spectrometry. Calibration and quantification take place by measurement of an external standard.

NOTE The method described here is fully in accordance with EN ISO 15009.

7 Reagents

Use only reagents of at least analytical grade. These substances shall be free of interfering compounds. Check this beforehand by carrying out a blank determination (see 11.2.2 and 11.3.2).

WARNING —The use of this document may involve hazardous materials, operations and equipment. It is the responsibility of the user of this document to establish appropriate measures to ensure the safety and health of personnel prior to application of the document and to fulfil statutory and regulatory requirements for this purpose.

7.1 Methanol [CAS 67-56-1].

7.2 Hexane [CAS 110-54-3].

7.3 Surrogate standard.

Preferably choose compounds, of which the undeuterated form is similar to a target analyte in the sample. The following may be used:

— anthracene-d_{10} [CAS 1719-06-8] for determining PAH;

— chrysene-d_{12} [CAS 1719-03-5] for determining PAH;

— naphthalene-d_8 [CAS 1146-65-2] for determining PAH;

— ethylbenzene-d_{10} [CAS 25837-05-2] for determining BTEX.

7.4 Injection standard.

Choose compounds for the injection standard that are physically and chemically similar to the compounds to be analysed, but not present in the sample. The following may be used:

— triphenylene-d_{12} [CAS 17777-56-9] for determining PAH;

— trifluorotoluene [CAS 98-08-8] for determining BTEX;

— 6-methylchrysene [CAS 1705-85-7] for determining chrysene with HPLC.

6-methylchrysene is used specifically for any HPLC analysis following the GC-MS analysis (11.6).

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