
Tekstilije in tekstilni izdelki - Določevanje policikličnih aromatskih ogljikovodikov (PAH), metoda s kromatografskimi tehnikami

Textiles and textile products - Determination of Polycyclic Aromatic Hydrocarbons (PAH), method using gas chromatography

Textilien und textile Erzeugnisse - Kritische Stoffe, die potentiell in Bestandteilen von Materialien textiler Erzeugnisse vorhanden sind - Bestimmung von polycyclischen aromatischen Kohlenwasserstoffen (PAK), Verfahren mit chromatographischen Methoden

Textiles et produits textiles - Détermination des hydrocarbures aromatiques polycycliques (HAP), méthode par chromatographie en phase gazeuse

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents		Page
European foreword.....		3
Introduction		4
1	Scope.....	5
2	Normative references.....	5
3	Terms and definitions	5
4	Principle	5
5	Reagents	6
6	Apparatus.....	8
7	Sample Preparation.....	8
8	Procedure.....	9
9	Expression of results.....	9
10	Test report.....	10
Bibliography.....		11

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European foreword

This document (EN 17132:2019) has been prepared by Technical Committee CEN/TC 248 “Textiles and textile products”, the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by January 2020, and conflicting national standards shall be withdrawn at the latest by January 2020.

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This document is adapted from CEN ISO/TS 16190 [1], prepared by Technical Committee CEN/TC 309, “Footwear”, in collaboration with ISO Technical Committee ISO/TC 216, “Footwear”, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement). The adaptation is based on the extension of the scope to textiles and textile products.

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Introduction

In the European Union, according to Regulation (EC) No 1907/2006 (REACH), Annex XVII, Entry 50, articles such as clothing, footwear and gloves are not to be placed on the market for supply to the general public if any of their rubber or plastic components that come into direct, as well as prolonged or short-term repetitive, contact with the human skin or the oral cavity, under normal or reasonably foreseeable conditions of use, contain more than 1 mg/kg (0,000 1 % by weight of this component) of any of the following PAHs.

Toys and childcare articles, are not to be placed on the market if any of their rubber or plastic components that come into direct as well as prolonged or short-term repetitive contact with the human skin or the oral cavity, under normal or reasonably foreseeable conditions of use, contain more than 0,5 mg/kg (0,000 05 % by weight of this component) of any of the following PAHs.

Further information can be found in the ECHA guideline [3].

According to Regulation (EU) 2018/1513 amending Annex XVII to Regulation (EC) No 1907/2006 (REACH) by Entry 72, (a) clothing or related accessories; (b) textiles other than clothing which, under normal or reasonably foreseeable conditions of use, come into contact with human skin to an extent similar to clothing; (c) footwear are not to be placed on the market after 1 November 2020, if they contain more than 1 mg/kg (0,000 1 % by weight of this component) of any of the following PAHs.

This restriction does not apply to: (a) clothing, related accessories or footwear, or parts of clothing, related accessories or footwear, made exclusively of natural leather, fur or hide; (b) non-textile fasteners and non-textile decorative attachments; (c) second-hand clothing, related accessories, textiles other than clothing or footwear; (d) wall-to-wall carpets and textile floor coverings for indoor use, rugs and runners. It also does not apply to clothing, related accessories, textiles other than clothing, or footwear within the scope of Regulation (EU) 2016/425 or Regulation (EU) 2017/745. The restriction on textiles other than clothing does not apply to disposable textiles. "Disposable textiles" means textiles that are designed to be used only once or for a limited time and are not intended for subsequent use for the same or a similar purpose.

Regulated PAHs: Benzo[a]pyrene (BaP), Benzo[e]pyrene (BeP), Benzo[a]anthracene (BaA), Chrysen (CHR), Benzo[b]fluoranthene (BbFA), Benzo[j]fluoranthene (BjFA), (g) Benzo[k]fluoranthene (BkFA), Dibenzo[a,h]anthracene (DBAhA).

The PAH Benzo(g,h,i)perylene was added to the REACH Candidate List of SVHC on 27 June 2018.

In addition, the following PAHs are under consideration for addition to the REACH Regulation.

Indeno(1,2,3-cd)pyrene, Benzo(j,k)fluorene (fluoranthrene), Napthalene, Anthracene, Pyrene, Benzo(g,h,i)perylene, Acenaphthalene, Acenaphthene

WARNING — The use of this document involves hazardous materials. It does not purport to address all of the safety or environmental problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel and the environment prior to application of the document and fulfil statutory and regulatory requirements for this purpose.

1 Scope

This document specifies a method to determine the amounts of polycyclic aromatic hydrocarbons (PAH) in components of textile products. This method has been elaborated to achieve a limit of quantification of 0,1 mg/kg.

NOTE A list of relevant materials can be found in CEN/TR 16741 [2].

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 ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use (ISO 4787)*

EN ISO 5089, *Textiles — Preparation of laboratory test samples and test specimens for chemical testing (ISO 5089)*

ISO 28540:2011, *Water quality — Determination of 16 polycyclic aromatic hydrocarbons (PAH) in water — Method using gas chromatography with mass spectrometric detection (GC-MS)*

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:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

The test sample is extracted using toluene at 60 °C in an ultrasonic bath for 1 h. An aliquot is then analysed using a gas chromatograph with mass selective detector.

EN 17132:2019 (E)

5 Reagents

WARNING — Polycyclic Aromatic Hydrocarbons can cause cancer, are suspected of damaging an unborn child, can cause damage to organs through prolonged or repeated exposure, and can be fatal if swallowed and/or enter airways. Appropriate measures shall be taken to ensure safety.

Unless otherwise specified, analytical grade chemicals shall be used.

5.1 Toluene, CAS¹ number: 108-88-3.

5.2 PAH

The following 18 PAH are relevant:

Naphthalene	CAS number: 91-20-3
Acenaphthylene	CAS number: 208-96-8
Acenaphthene	CAS number: 83-32-9
Fluorene	CAS number: 86-73-7
Phenanthrene	CAS number: 85-01-8
Anthracene	CAS number: 120-12-7
Fluoranthene	CAS number: 206-44-0
Pyrene	CAS number: 129-00-0
Benzo[a]anthracene	CAS number: 56-55-3
Benzo[e]pyrene	CAS number: 192-97-2
Benzo[j]fluoranthene	CAS number: 205-82-3
Chrysene	CAS number: 218-01-9
Benzo[b]fluoranthene	CAS number: 205-99-2
Benzo[k]fluoranthene	CAS number: 207-08-9
Benzo[a]pyrene	CAS number: 50-32-8
Indeno[1,2,3-cd]pyrene	CAS number: 193-39-5
Dibenzo[a,h]anthracene	CAS number: 53-70-3
Benzo[g,h,i]perylene	CAS number: 191-24-2

5.3 PAH standard solution(s) (100 µg/ml)

18 different components specified in 5.2, either as commercially available certified mixes or as individual components in solution.

5.3.1 Target PAHs – Stock solution (0,5 µg/ml)

Put 9 ml of toluene (5.1) in a 10 ml volumetric flask (6.6), add 50 µl of PAH stock solution (5.3) and then fill up to the calibration mark with toluene (5.1).

¹ CAS: Chemical Abstract Service.

5.3.2 Target PAHs – Working (Calibration) solutions

Prepare at least 4 calibration solutions of PAH, including internal standard in toluene at suitable concentrations for the analysis e.g. PAH concentrations 0,005 µg/ml, 0,025 µg/ml, 0,100 µg/ml, 0,500 µg/ml, IS concentration 0,050 µg/ml. Put the required volume of PAH standard solution (5.3.1) and 0,1 ml of internal standard solution (5.4.2) into a 10 ml volumetric flask (6.6) and fill up to the mark with toluene (5.1).

5.4 Internal standards (examples of), commercially available as certified stock solution

Naphthalene-d8,	CAS number: 1146-65-2
Pyrene-d10,	CAS number: 1718-52-1
Perylene-d12,	CAS number: 1520-96-3
Anthracene-d10,	CAS number: 1719-06-8
Phenanthrene-d10,	CAS number: 1517-22-2
Triphenylbenzene,	CAS number: 612-71-5
Benzo[a]pyrene-d12,	CAS number: 63466-71-7

NOTE The following list shows examples of correspondence between PAH and internal standards.

Name	Internal standard
Naphthalene	Naphthalene-d8
Acenaphthylene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Acenaphthene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Fluorene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Phenanthrene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Anthracene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Fluoranthene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Pyrene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Benzo[a]anthracene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Benzo[e]pyrene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Benzo[j]fluoranthene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Chrysene	Pyrene-d10 or Anthracene-d10 or Phenanthrene-d10
Benzo[b]fluoranthene	Benzo[a]pyrene-d12 or Perylene-d12 or Triphenylbenzene
Benzo[k]fluoranthene	Benzo[a]pyrene-d12 or Perylene-d12 or Triphenylbenzene
Benzo[a]pyrene	Benzo[a]pyrene-d12 or Perylene-d12 or Triphenylbenzene
Indeno[1,2,3-cd]pyrene	Benzo[a]pyrene-d12 or Perylene-d12 or Triphenylbenzene
Dibenzo[a,h]anthracene	Benzo[a]pyrene-d12 or Perylene-d12 or Triphenylbenzene
Benzo[g,h,i]perylene	Benzo[a]pyrene-d12 or Perylene-d12 or Triphenylbenzene