



Designation: D8143 – 24

Standard Test Method for Determination of the EU-11 List of PAH Compounds in Carbon Black¹

This standard is issued under the fixed designation D8143; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers the qualitative and quantitative determination of the EU-11 list of polycyclic aromatic hydrocarbons (PAH) on carbon black. The EU-11 list of PAH compounds is given in [Table 1](#). The procedure involves Soxhlet extraction with toluene followed by extract analysis using gas chromatography with mass spectrometry (GC/MS). This method is not intended to test for U.S. Food and Drug Administration (FDA 21 CFR 178.3297) compliance of carbon blacks used for indirect food contact applications.

1.2 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 European Commission Regulation:

[Commission Regulation \(EU\) 1272/2013 amending annex XVII to Regulation \(EC\) 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorization and Restriction of Chemicals \(REACH\) as regards polycyclic aromatic hydrocarbons \(2013\) OJ L328/6](#)

[Commission Regulation \(EU\) 2020/2096 of 15 December 2020 amending Annex XVII to Regulation \(EC\) No 1907/](#)

[2006 of the European Parliament and of the Council concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals \(REACH\), as regards carcinogenic, mutagenic, or reproductive toxicant \(CMR\) substances](#)

[Commission Regulation \(EU\) 2021/2204 of 13 December 2021 amending Annex XVII to Regulation \(EC\) No 1907/2006 of the European Parliament and of the Council concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals \(REACH\), as regards carcinogenic, mutagenic, or reproductive toxicant \(CMR\) substances](#)

2.2 Federal Standard:²

[21 CFR 178.3297 Indirect Food Additives: Adjuvants, Productions Aids, and Sanitizers, Colorants for Polymers](#)

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *polycyclic aromatic hydrocarbon, PAH, n*—organic compound(s) that consist of chemical structures of alternating single and double bonds of a ring like nature (for example, benzene), with no heteroatom or other substituents; frequently the rings are fused to make larger chemical structures.

3.1.1.1 *Discussion*—PAH compounds naturally occur in oil, coal, and tar deposits; are produced by the incomplete combustion of hydrocarbons; and occur in many other products and processes.

3.1.2 *EU-11 PAH compound list, n*—a list of eight polycyclic aromatic hydrocarbons that are specified by the Commission Regulation (EU) 1272/2013 (December 6, 2013)³ amending Annex XVII to Regulation (EC) 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorization and Restriction of Chemicals (REACH) as regards polycyclic aromatic hydrocarbons (2013) OJ L328/69. The additional three benzopyrene polycyclic aromatic hydrocarbons also covered in this standard are

¹ This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.66 on Environment, Health, and Safety.

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² Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, <http://www.access.gpo.gov>.

³ Commission Regulation (EU) No 1272/2013 of 6 December 2013. Available at <http://eur-lex.europa.eu/eli/reg/2013/1272/oj>.

TABLE 1 EU-11 PAH Compounds and Internal Standards

Native PAH	CAS#	Deuterated Internal Standards for Quantitation	CAS#
Benz[a]anthracene	56-55-3	Chrysene-d12	1719-03-5
Chrysene	218-01-9	Benzo[a]pyrene-d12	63466-71-7
Benzo[b]fluoranthene	205-99-2	1,12-Benzoperylene-d12	93951-66-7
Benzo[j]fluoranthene	205-82-3	Perylene-d12	1520-96-3
Benzo[k]fluoranthene	207-08-9	Dibenzo(a,i)pyrene-d14	158776-07-9
Benzo[e]pyrene	192-97-2		
Benzo[a]pyrene	50-32-8		
Dibenz[a,h]anthracene	53-70-3		
Dibenzo[a,i]pyrene	189-55-9		
Dibenzo[a,h]pyrene	189-64-0		
Dibenzo[a,i]pyrene	191-30-0		

referenced in entry 28 Appendix 2 of Annex XVII to Regulation (EC) No 1907/2006 of the European Parliament and of the Council concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH).

3.1.2.1 *Discussion*—Regulation (EU) No. 1272/2013 sets PAH concentration limits on finished products specified by their application. The regulation does not refer directly to carbon black, though in many instances and for formulation purposes there is a need to know the quantitative, extractable PAH content present in carbon black that will become part of the finished product. In addition, no specific test method has been defined by the regulation for testing finished products or raw materials used in the finished products.

4. Summary of Test Method

4.1 A portion of carbon black is Soxhlet-extracted with toluene for 48 h. The extract is concentrated and subsequently analyzed for specific PAH compounds by gas chromatography with mass spectrometry (GC/MS). Each PAH compound is quantified individually using internal and external standards.

5. Significance and Use

5.1 This test procedure is used to determine the individual concentrations of the PAH compounds in the EU-11 list extracted from carbon black by the means of a Soxhlet extraction apparatus with toluene.

6. Apparatus

6.1 *Soxhlet Extractor with Reflux Condenser*, 50 or 100 cm³ capacity.

6.2 *Extraction Thimbles*, glass, or cellulose, 50 to 70 cm³ capacity. For instance, glass extraction thimble of 35 mm diameter by 90 mm height with coarse porosity (70 to 100 µm like Ace Glass Size C, Porosity B, Code-14).

6.3 *Heating Mantle*, compatible with boiling flask described in 6.4.

6.4 *Boiling Flasks for Soxhlet*, for example, 250 cm³.

6.5 *Glass Beads*, 180 to 250 µm (60/80 mesh) if glass thimbles are used. Need to be very clean or toluene extracted.

6.6 *Glass Boiling Beads*, approximately 3 mm.

6.7 *Glass Wool*, need to be clean or toluene extracted.

6.8 *Rotary Evaporator*, with temperature-controlled water bath, automatic pressure regulation, and solvent-proof mem-

brane vacuum pump. Other evaporators may be used if they produce acceptable results.

6.9 *Nitrogen Blow-Down Apparatus*, equipped with a controlled water bath and nitrogen pressure control.

6.10 *Pear-Shaped Flasks for Rotary Evaporator*, for example, 25, 50, and 100 cm³.

6.11 *GC/MS*, with autosampler.

6.11.1 *Mass Spectrometer (MS)*, with electron impact capability and Selected Ion Monitoring (SIM) mode.

6.11.2 *GC Capillary Column*, to obtain proper separation of the eleven PAH compounds, a GC column specially designed for their separation is needed. One such column is DB-EUPAH (Agilent Technologies/agilent.com). This column has been shown to be able to separate benzo(b)fluoranthene, benzo(j)fluoranthene, and benzo(k)fluoranthene. Other similar columns or column phases may be used if they provide acceptable peak resolutions.

6.11.3 *Deactivated Straight Borosilicate Liner with Small Piece of Deactivated Glass Wool*—This liner may be used if peak resolution is satisfactory.

6.11.4 Alternative liner is a split/splitless nondeactivated liner with glass wool (4 mm internal diameter, straight liner). This shall be deactivated with a silanizing agent before use. Another alternative is a split/splitless liner with fluorocarbon liner seals. Such a liner will already contain conditioned silanized glass wool. Other liners can be used if they produce acceptable results.

6.11.5 *Gold-Plated Seal*, for GC injector port or similar nonreactive seal.

6.11.6 *GC/MS Amber Autosampler Vials*, with polytetrafluoroethylene (PTFE)-coated caps.

6.12 *Adjustable Micropipettes*, for example 1000, 200, and 20 µL, or similar analytical-grade volume measuring equipment.

6.13 *Microliter Syringes of Different Volumes*, for example, 10 and 100 µL.

6.14 *Volumetric Flasks*, for example 10 and 100 cm³.

6.15 *Analytical Balance*, with an accuracy of 0.1 mg.

6.16 *Furnace*, capable of temperature regulation of 500 ± 25°C, used to burn off organic contamination from glass surfaces.

TABLE 2 PAH Compound Target Ions for Quantitation, Typical Relative Retention Times, and Corresponding Internal Standards

Group ^A	Peak #	Analyte PAH	Target ion for SIM quantitation	Relative Retention Time ^B to corresponding internal standard	Corresponding deuterated internal standard (IS) compound	Target ion of internal standard for SIM quantitation
1	1	Benzo(a)anthracene	228	0.997	Chrysene-d12	240
	2	Chrysene	228	1.006		
2	3	Benzo(b)fluoranthene	252	0.944	Benzo(a)pyrene-d12 ^C	264
	4	Benzo(j)fluoranthene	252	0.944		
	5	Benzo(k)fluoranthene	252	0.948		
	6	Benzo(e)pyrene	252	0.991		
	7	Benzo(a)pyrene	252	1.000		
3	8	Dibenz(a,h)anthracene	278	0.989	1,12-Benzperylene-d12 OR	288
	9	Dibenzo(a,i)pyrene	302	1.169		
	10	Dibenzo(a,h)pyrene	302	1.479	Dibenzo(a,i)pyrene-d14 ^D	316
	11	Dibenzo(a,l)pyrene	302	1.263		

^A PAH compounds and their corresponding internal standard can be grouped together for Selected Ion Monitoring (SIM). For example, SIM Group 1 would monitor ions 228 and 240 during their respective GC retention time window.

^B Relative Retention Time (RRT) will be determined by the individual compounds, GC column, GC oven programming, and other GC conditions.

^C If desired, Perylene-d12 (target ion for SIM quantitation of 264) can be used as the internal standard for Benzo(b)fluoranthene, Benzo(j)fluoranthene, Benzo(k)fluoranthene, and Benzo(e)pyrene.

^D Dibenzo(a,i)pyrene-d14 and 1,12-Benzperylene-d12 are optional internal standards for the Dibenzopyrenes.

6.17 *Manometer*, capable of pressure readings in the range of 5 ± 0.3 kPa for controlling vacuum evaporators.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Stock solutions or mixtures of the native PAH standards and of the internal standard (deuterated PAH compounds) can be purchased as premade solutions or prepared from solid materials (Table 1). Refer to Table 2 to know which internal standard compound is recommended for each PAH analyte compound.

7.2.1 A concentration range of 80 to 100 $\mu\text{g}/\text{cm}^3$ for each of the native PAH analytes is recommended for a stock solution mixture.

7.2.2 A similar concentration range of 80 to 100 $\mu\text{g}/\text{cm}^3$ for each of the deuterated internal standard compounds is recommended for the internal standard (IS) stock solution.

7.2.3 All purchased standard materials shall be 98 % pure or better and certified with respect to their purity and concentration by the manufacturer. Follow the manufacturer's recommendation on how to store the standard solutions and their recommended expiration date. Typically, the stock solutions are protected from light and stored at room temperature. They should be checked for signs of degradation or evaporation. The standard stock solutions shall be replaced/recertified on a

yearly basis or sooner if comparisons with quality control (QC) samples indicate a problem.

7.3 Toluene, suitable for high resolution gas chromatography analysis (99.99 % pure).

7.4 Cyclohexane, suitable for high resolution gas chromatography analysis (99.99 % pure).

7.5 Helium, GC/MS purity grade.

7.6 Nitrogen, analytical purity grade.

7.7 Silica gel columns for Solid Phase Extraction (SPE) removal of polar compounds.

7.7.1 Silica SPE Cartridges, single-use application, having a volume capacity of 1 to 10 cm^3 .

7.7.1.1 *Preparation of Silica SPE Cartridge*—Follow the manufacturer's instruction for preparation and use. The typical method of preparation is to wet the cartridge with approximately 10 cm^3 of the elution solvent. Discard the wetting solvent.

7.7.2 Alternative SPE Method: Silica Gel/13 % H_2O Gravity Column:

7.7.2.1 *Preparation of a Silica Gel/13 % H_2O Adsorbent*—Pour 200 g silica gel (high purity grade, type 60, particle size 0.063 to 0.200 mm) into a screw cap glass bottle and add 30 g of deionized water in small portions (for example, 2 cm^3) using a suitable pipette. After addition of each portion of water, the silica gel bottle is shaken to evenly distribute the wetted silica gel. No aggregation should occur during this process. If so, shaking must be continued until a homogeneous material is obtained. Finally, the closed bottle is shaken for 8 h by means of an overhead shaker. If properly closed and stored, the adsorbent should be viable for at least 6 months.

7.7.2.2 *Preparation of a Silica Gel/13 % H_2O Gravity Column*—Insert a glass wool plug into the bottom of a pipette tip (for example, 8 to 10 mm inner diameter and 5 cm^3 capacity). Place 1 g of the silica gel/13 % H_2O in the column and tap the column gently to settle the silica gel.

⁴ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.