Designation: D6209 − 13

Standard Test Method for Determination of Gaseous and Particulate Polycyclic Aromatic Hydrocarbons in Ambient Air (Collection on Sorbent-Backed Filters with Gas Chromatographic/Mass Spectrometric Analysis)[1]

This standard is issued under the fixed designation D6209; 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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method[2] specifies sampling, cleanup, and analysis procedures for the determination of polycyclic aromatic hydrocarbons (PAH) in ambient air.

1.2 This test method is designed to collect both gas-phase and particulate-phase PAH and to determine them collectively.

1.3 This test method is a high-volume sampling (100 to 250 L/min) method capable of detecting PAH at sub-nanograms per cubic metre (ng/m^3) concentrations with sampling volumes up to 350 m^3 of air.

1.4 This test method has been validated for sampling periods up to 24 h.

1.5 Precision and bias under normal conditions can be expected to be ±35 to 50 %.

1.6 This test method describes a sampling and analysis procedure for PAH that involves collection from air on a combination fine-particle filter and sorbent trap and subsequent analysis by gas chromatography/mass spectrometry (GC/MS).

1.7 The range of this test method is approximately 0.05 to 1000 ng/m^3 of air sampled.

1.8 The values stated in SI units shall be regarded as standard.

1.9 *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 and health practices and determine the applicability of regulatory limitations prior to use.* See also Section 8 for additional safety precautions.

1.10 *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 *ASTM Standards:*[3]

D1356 Terminology Relating to Sampling and Analysis of Atmospheres
D1357 Practice for Planning the Sampling of the Ambient Atmosphere
D3631 Test Methods for Measuring Surface Atmospheric Pressure
E1 Specification for ASTM Liquid-in-Glass Thermometers
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1356.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *sampling efficiency (SE), n*—ability of the sampler to trap and retain PAH. The percent SE is the percentage of the analyte of interest collected and retained by the sampling medium when it is introduced into the air sampler and the sampler is operated under normal conditions for a period of time equal to or greater than that required for the intended use.

3.2.2 *dynamic retention efficiency, n*—ability of the sampling medium to retain a given PAH that has been added to the sorbent trap in a spiking solution when air is drawn through the

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[1] This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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[2] This test method is based on U. S. Environmental Protection Agency Compendum Method TO-13, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Report No. EPA/600-4-89/018, June 1988, available from the National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161, Order No. PB90-11989/AS.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Formulae and Physical Properties of Selective PAH

Compound (Common Name)	Formula	Molecular Weight	Melting Point, °C	Boiling Point,[A] °C	Vapor Pressure, kPa at 25°C
Naphthalene	$C_{10}H_8$	128.18	80.2	218	1.1×10^{-2}
Acenaphthylene	$C_{12}H_8$	152.20	92-93	265-280	3.9×10^{-3}
Acenaphthene	$C_{12}H_{10}$	154.20	90-96	278-279	2.1×10^{-2}
Fluorene	$C_{13}H_{10}$	166.23	116-118	293-295	8.7×10^{-5}
9-Fluorenone	$C_{13}H_8O$	180.21	84	341.5	ca.10^{-5}
Anthracene	$C_{14}H_{10}$	178.24	216-219	340	3.6×10^{-6}
Phenanthrene	$C_{14}H_{10}$	178.24	96-101	339-340	2.3×10^{-5}
Fluoranthene	$C_{16}H_{10}$	202.26	107-111	375-393	6.5×10^{-7}
Pyrene	$C_{16}H_{10}$	202.26	150-156	360-404	3.1×10^{-6}
Cyclopental[*cd*]pyrene	$C_{18}H_{10}$	226.28	ca. 275?	—	ca. 10^{-7}
Benz[*a*]anthracene	$C_{18}H_{12}$	228.30	157-167	435	1.5×10^{-8}
Chrysene	$C_{18}H_{12}$	228.30	252-256	441-448	5.7×10^{-10}
Retene	$C_{18}H_{18}$	234.34	101	390	ca. 10^{-6}
Benzo[*b*]fluoranthene	$C_{20}H_{12}$	252.32	167-168	481	6.7×10^{-8}
Benzo[*k*]fluoranthene	$C_{20}H_{12}$	252.32	198-217	480-481	2.1×10^{-8}
Perylene	$C_{20}H_{12}$	252.32	273-278	500-503	7.0×10^{-10}
Benzo[*a*]pyrene	$C_{20}H_{12}$	252.32	177-179	493-496	7.3×10^{-10}
Benzo[*e*]pyrene	$C_{20}H_{12}$	252.32	178-179	493	7.4×10^{-10}
Benzo[*ghi*]perylene	$C_{22}H_{12}$	276.34	275.278	525	1.3×10^{-11}
Indeno[1,2,3-*cd*]pyrene	$C_{22}H_{12}$	276.34	162-163	—	ca. 10^{-11}
Dibenz[*ah*]anthracene	$C_{22}H_{14}$	278.35	266-270	524	1.3×10^{-11}
Coronene	$C_{24}H_{12}$	300.36	438-440	525	2.0×10^{-13}

[A] Many of these compounds sublime.

sampler under normal conditions for a period of time equal to or greater than that required for the intended use.

4. Summary of Test Method

4.1 *Sampling:*

4.1.1 An air sample is collected directly from the ambient atmosphere by pulling air at approximately 225 L/min through a fine particulate filter followed by a vapor trap containing polyurethane foam (PUF) or styrene/divinylbenzene polymer resin (XAD-2).[4] Sampling times may be varied from 1 to 24 h, depending on monitoring needs and the detection limits required, so as not to exceed a total sample volume of 350 m^3.

4.2 *Analysis:*

4.2.1 After sampling a fixed volume of air, the particle filter and sorbent cartridge are extracted together in a Soxhlet extractor. The sample extract is concentrated by means of a Kuderna-Danish concentrator (or other validated method), followed by a further concentration under a nitrogen stream, if necessary, and an aliquot is analyzed by gas chromatography/mass spectrometry. The results derived represent the combined gas-phase and particulate-phase air concentrations of each PAH analyzed.

5. Significance and Use

5.1 Polycyclic aromatic hydrocarbons (PAH) as defined by this test method are compounds made up of two or more fused aromatic rings.

5.2 Several PAH are considered to be probable human carcinogens.

5.3 PAH are emitted in the atmosphere primarily through wood or fossil fuel combustion.

5.4 Two- and three-ring PAH are typically present in urban air at concentrations ranging from 10 to several hundred nanograms per cubic metre (ng/m^3); those with four or more rings are usually found at concentrations of a few ng/m^3 or lower.

5.5 PAH span a broad spectrum of vapor pressures (for example, from 1.1×10^{-2} kPa for naphthalene to 2×10^{-13} kPa for coronene at 25°C). Table 1 lists some PAH that are frequently found in ambient air. Those with vapor pressures above about 10^{-8} kPa will be present in the ambient air substantially distributed between the gas and particulate phases. This test method will permit the collection of both phases. However, particulate-phase PAH will tend to be lost from the particulate filter during sampling due to desorption and volatilization.

5.5.1 The distribution between phases depends on ambient temperature, humidity, types and concentrations of PAH and particulate matter, and residence time in the air. PAH, especially those having vapor pressures above 10^{-8} kPa, may vaporize from particulate filters during sampling. Consequently, a back-up vapor trap must be used for efficient sampling.

5.6 Separate analyses of the filter and vapor trap will not reflect the original atmospheric phase distributions and should be discouraged.

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[4] XAD is a trademark of Rohm and Haas Co., Philadelphia, PA; it is available in the United States solely from Supelco, Inc., Bellefonte, PA. If you are aware of equivalent styrene/divinylbenzene polymer resins, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee[1], which you may attend.