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.

Designation: D7363 – 13a (Reapproved 2021)$^{\varepsilon 1}$

Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode[1,2]

This standard is issued under the fixed designation D7363; 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.

ε^1 NOTE—Reapproved with editorial changes in November 2021.

1. Scope

1.1 The U.S. Environmental Protection Agency (USEPA) narcosis model for benthic organisms in sediments contaminated with polycyclic aromatic hydrocarbons (PAHs) is based on the concentrations of dissolved PAHs in the interstitial water or "pore water" in sediment. This test method covers the separation of pore water from PAH-impacted sediment samples, the removal of colloids, and the subsequent measurement of dissolved concentrations of the required 10 parent PAHs and 14 groups of alkylated daughter PAHs in the pore water samples. The "24 PAHs" are determined using solid-phase microextraction (SPME) followed by Gas Chromatography/Mass Spectrometry (GC/MS) analysis in selected ion monitoring (SIM) mode. Isotopically labeled analogs of the target compounds are introduced prior to the extraction, and are used as quantification references.

1.2 Lower molecular weight PAHs are more water soluble than higher molecular weight PAHs. Therefore, USEPA-regulated PAH concentrations in pore water samples vary widely due to differing saturation water solubilities that range from 0.2 µg/L for indeno[1,2,3-cd]pyrene to 31 000 µg/L for naphthalene. This method can accommodate the measurement of microgram per litre concentrations for low molecular weight PAHs and nanogram per litre concentrations for high molecular weight PAHs.

1.3 The USEPA narcosis model predicts toxicity to benthic organisms if the sum of the toxic units (ΣTU_c) calculated for all "34 PAHs" measured in a pore water sample is greater than or equal to 1. For this reason, the performance limit required for the individual PAH measurements was defined as the concentration of an individual PAH that would yield 1⁄34 of a toxic unit (TU). However, the focus of this method is the 10 parent PAHs and 14 groups of alkylated PAHs (Table 1) that contribute 95 % of the toxic units based on the analysis of 120 background and impacted sediment pore water samples.[3] The primary reasons for eliminating the rest of the 5-6 ring parent PAHs are: *(1)* these PAHs contribute insignificantly to the pore water TU, and *(2)* these PAHs exhibit extremely low saturation solubilities that will make the detection of these compounds difficult in pore water. This method can achieve the required detection limits, which range from approximately 0.01 µg/L, for high molecular weight PAHs, to approximately 3 µg/L for low molecular weight PAHs.

1.4 The test method may also be applied to the determination of additional PAH compounds (for example, 5- and 6-ring PAHs as described in Hawthorne et al.).[4] However, it is the responsibility of the user of this standard to establish the validity of the test method for the determination of PAHs other than those referenced in 1.1 and Table 1.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the*

[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

Current edition approved Nov. 1, 2021. Published December 2021. Originally approved in 2007. Last previous edition approved in 2013 as D7363 – 13a. DOI: 10.1520/D7363-13AR21E01.

[2] Standard methods under the jurisdiction of ASTM Committee D19 may be published for a limited time preliminary to the completion of full collaborative study validation. Such standards are deemed to have met all other D19 qualifying requirements but have not completed the required validation studies to fully characterize the performance of the test method across multiple laboratories and matrices. Preliminary publication is done to make current technology accessible to users of standards, and to solicit additional input from the user community.

[3] Hawthorne, S. B., Grabanski, C. B., and Miller, D. J., "Measured Partitioning Coefficients for Parent and Algae Polycyclic Aromatic Hydrocarbons in 114 Historically Contaminated Sediments: Part I, Koc Values," *Environmental Toxicology and Chemistry*, Vol 25, 2006, pp. 2901–2911.

[4] Hawthorne, S. B., Grabanski, C. B., Miller, D. J., and Kreitinger, J. P., "Solid Phase Microextraction Measurement of Parent and Akyl Polycyclic Aromatic Hydrocarbons in Milliliter Sediment Pore Water Samples and Determination of K_{DOC} Values," *Environmental Science Technology*, Vol 39, 2005, pp. 2795–2803.

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