Designation: E2143 – 01 (Reapproved 2021)

Standard Test Method for Using Field-Portable Fiber Optics Synchronous Fluorescence Spectrometer for Quantification of Field Samples for Aromatic and Polycyclic Aromatic Hydrocarbons[1]

This standard is issued under the fixed designation E2143; 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 covers a rapid method for the screening of environmental samples for aromatic hydrocarbons (AHs) and polycyclic aromatic hydrocarbons (PAHs). The screening takes place in the field and provides immediate feedback on limits of contamination by substances containing AHs and PAHs. Quantification is obtained by the use of appropriately characterized, site-specific calibration curves. Remote sensing by use of optical fibers is useful for accessing difficult to reach areas or potentially dangerous materials or situations. When contamination of field personnel by dangerous materials is a possibility, use of remote sensors may minimize or eliminate the likelihood of such contamination taking place.

1.2 This test method is applicable to AHs and PAHs present in samples extracted from soils or in water. This test method is applicable for field screening or, with an appropriate calibration, quantification of total AHs and PAHs.

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

1.4 *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.*

1.5 *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:*[2]

D1129 Terminology Relating to Water
D4489 Practices for Sampling of Waterborne Oils
D5412 Test Method for Quantification of Complex Polycyclic Aromatic Hydrocarbon Mixtures or Petroleum Oils in Water
E131 Terminology Relating to Molecular Spectroscopy
E388 Test Method for Wavelength Accuracy and Spectral Bandwidth of Fluorescence Spectrometers
E578 Test Method for Linearity of Fluorescence Measuring Systems
E579 Test Method for Limit of Detection of Fluorescence of Quinine Sulfate in Solution

3. Terminology

3.1 For definitions of terms used in this test method refer to Terminology D1129 and E131.

4. Summary of Test Method

4.1 This test method consists of extracting the AHs and PAHs from soil samples or preparation of water samples followed by synchronous fluorescence analysis with a field-portable instrument. The samples require serial dilutions of samples to establish a linear response. These measurements are made using standard fluorescence cuvettes. While some optimization of selectivity can be accomplished by varying the wavelength difference between excitation and emission monochromators, generally spectra generated from petroleum contaminants with a wavelength difference such as 6 nm or 18 nm provide good results and no preliminary spectra are required (see Test Method D5412).

[1] This test method is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and Separation Science and is the direct responsibility of Subcommittee E13.09 on Fiber Optics, Waveguides, and Optical Sensors.

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[2] 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.

4.2 Different soils have varying partition coefficients. Therefore, representative samples of a subset of the extracts or the water samples should be analyzed by gas chromatography (GC) or other appropriate methods. The purpose is to establish a site-specific calibration curve to be used for quantification of total AHs and PAHs in the environmental samples of interest.

4.3 When desirable, determination of AHs and PAHs may be made remotely using an optical fiber.

5. Significance and Use

5.1 This technique is designed for on-site rapid screening and characterization of environmental soil and water samples resulting in significant cost savings for environmental remediation projects. Remote analysis can be made with optical fibers when situations warrant or demand use of this option.

5.2 Quantification of total AHs and PAHs in these environmental samples is accomplished by having a subset of the samples analyzed by an alternate technique and generating a site-specific calibration curve.

5.3 Synchronous fluorescence provides sufficient spectral information to characterize the AHs and PAHs present as benzene, toluene, ethylbenzene and xylene(s) (BTEX), the aromatic portion of total petroleum hydrocarbons (TPH), or large aromatic ring systems up to at least seven fused rings, such as might be found in creosote.

6. Interferences

6.1 The synchronous fluorescence spectrum can be distorted or quantification may be affected if there is a contaminant present that produces a synchronous peak in the same vicinity as the material of interest. Often spectroquality solvents contain impurities that produce background signals. Solvent blanks should be used to verify a low fluorescence background so the background can be subtracted from the sample's spectrum.

6.2 There are naturally occurring compounds that fluoresce, which may interfere with the detection of petroleum compounds, present in the sample. Humic acid from leaf mold is an example of such a compound. Its strongest emission occurs in the near ultraviolet range.

6.3 Absorption of the exciting light by the sample itself (self-filtering effect) produces erroneous results. Analysis of serial dilutions of the sample detects this effect and ensures an accurate analysis is made. Once linearity is established, then integration of the spectrum produces accurate results.

6.4 Certain solvents used for extraction of the soil samples could quench or absorb the fluorescence and raise the limit of detection. Care should be taken to avoid halogenated solvents or solvents containing other quenchers. The user of this test method should bear this in mind when selecting an appropriate solvent.

NOTE 1—Storage of samples in improper containers, such as plastics other than polytetrafluoroethylene (or TFE-fluorocarbon), may result in contamination.

NOTE 2—This test method is normally used without an internal standard due to possible interference by the internal standard.

6.5 Certain optical fibers may generate a fluorescence background. These should be avoided whenever possible. If they must be used, a background spectrum should be generated and subtracted from any samples measured.

7. Apparatus

7.1 *Fluorescence Spectrometer*—An instrument recording in the spectral range of at least 250 nm to 650 nm is required for both excitation and emission spectrum measurements and capable of scanning both monochromators at a constant speed with a constant wavelength offset between them for synchronous scanning. The bandwidth of the monochromators should be less than one half the wavelength offset between the monochromators or smaller. The spectrometer should be capable of remote sensing via optic fiber. The detector should be a photomultiplier tube or a device with similar sensitivity and response time. Occasionally field work requires the spectrometer to be battery powered. The instrument should meet the specifications in Table 1.

7.2 *Excitation Source*—A pulsed (9.9 W) Xenon lamp or other source having sufficient intensity throughout the ultraviolet and visible regions can be used.

7.3 *Cuvette Sample Holder*—Sample holders should be fabricated to hold commercially available, fluorescence-free, fused silica cuvettes.

7.4 *Optical Fiber Holder*—A stage that allows correct positioning of the optical fiber with respect to the emission and excitation monochromators. The device may also be used to optically match each fiber and the respective monochromator.

7.5 *Computer System*—The instrument should be interfaced to a computer system that is compatible with the instrument and has suitable software for spectral data manipulation.

7.6 *Cuvette*—A standard 12 mm by 12 mm by 31 mm fluorescence-free fused silica cuvette. Four sides of the cuvette should be polished.

7.7 *Optical Fiber*—Fused silica fiber (preferably a high hydroxide) is required for transmission of the ultraviolet wavelengths required for accurate spectroscopic analysis. In general, this material has good thermal characteristics, can be obtained with low fluorescence background, and is readily available commercially.

TABLE 1 Desirable Performance Standards of a Field Portable Fluorescence Spectrometer

Characteristic	Desirable Range	Typical
Monochromator		
Bandwidth	1 nm–5 nm	3 nm
Wavelength accuracy	±0.5nm–2 nm	±1.0 nm
Reproducibility	±0.1 %–1 %	±0.2 %
Interface		
Data collection	computerized	laptop PC
Instrument control	control and data	
Source		
Broad band	200 nm–1000 nm	Xenon lamp
Low-power consumption	5 W–75 W	10 W