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: D7678 – 17 (Reapproved 2022)

Standard Test Method for Total Oil and Grease (TOG) and Total Petroleum Hydrocarbons (TPH) in Water and Wastewater with Solvent Extraction using Mid-IR Laser Spectroscopy[1]

This standard is issued under the fixed designation D7678; 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 the determination of total oil and grease (TOG) and total petroleum hydrocarbons (TPH) in water and waste water that are extractable by this test method from an acidified sample with a cyclic aliphatic hydrocarbon (for example cyclohexane, cyclopentane) and measured by IR absorption in the region from 1370 cm^{-1} to 1380 cm^{-1} (7.25 µm to 7.30 µm) using a mid-IR laser spectrometer. Polar substances are removed by clean-up with Florisil.[2]

1.2 This test method also considers the volatile fraction of petroleum hydrocarbons, which is lost by gravimetric methods that require solvent evaporation prior to weighing, as well as by solvent-less IR methods that require drying of the employed solid phase material prior to measurement. Similarly, a more complete fraction of extracted petroleum hydrocarbons are accessible by this test method as compared to GC methods that use a time window for quantification, as petroleum hydrocarbons eluting outside these windows are quantified too.

1.3 This test method covers the range of 0.1 mg/L to 1000 mg/L and may be extended to a lower or higher level by extraction of a larger or smaller sample volume collected separately.

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

1.5 *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.*

1.6 *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]

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D3370 Practices for Sampling Water from Flowing Process Streams

D3921 Test Method For Oil and Grease and Petroleum Hydrocarbons in Water (Withdrawn 2013)[4]

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

E168 Practices for General Techniques of Infrared Quantitative Analysis

2.2 *ISO Standards:*[5]

ISO 9377-2 Determination of Hydrocarbon Oil Index

2.3 *Code of Federal Regulations (CFR) Publications:*[6]

40 CFR Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants

49 CFR Part 172 Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, Training Requirements, and Security Plans

[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 Oct. 15, 2022. Published October 2022. Originally approved in 2011. Last previous edition approved in 2017 as D7678 – 17. DOI: 10.1520/D7678-17R22.

[2] Florisil is a trademark by U.S. Silica Company, Frederick, MD.

[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.

[4] The last approved version of this historical standard is referenced on www.astm.org.

[5] Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

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

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. United States

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129 and Practices E168.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *grease, n*—difference between measured value for TOG and TPH of a sample.

3.2.2 *total oil and grease (TOG), n*—material that can be extracted from water or wastewater by this test method and which can be measured by infrared absorption in the region from 1370 cm^{-1} to 1380 cm^{-1}.

3.2.3 *total petroleum hydrocarbons (TPH), n*—material that can be extracted from water or wastewater by this test method, which remains in the extract after treatment with Na_2SO_4 and Florisil, and which can be measured by infrared absorption in the region from 1370 cm^{-1} to 1380 cm^{-1}.

4. Summary of Test Method

4.1 Hydrocarbons from an acidified 900-mL sample of water or wastewater are extracted with 50 mL of solvent. The material, which is measured directly after extraction by mid-infrared laser spectroscopy is referred to as total oil and grease (TOG). The material, which is measured after the extract is treated with sodium sulfate and Florisil to remove traces of water as well as polar substances, and thereby producing a solution containing non-polar material, and which is measured by mid-infrared laser spectroscopy is referred to as total petroleum hydrocarbons (TPH). The difference between TOG and TPH provides the amount of grease extracted from the sample.

5. Significance and Use

5.1 The presence and concentration of total oil and grease as well as total petroleum hydrocoarbons, in domestic and industrial wastewater is of concern to the public because of its deleterious aesthetic effect and its impact on aquatic life.

5.2 Regulations and standards have been established that require monitoring of total oil and grease as well as total petroleum hydrocarbons in water and wastewater.

6. Interferences

6.1 Soaps, detergents, surfactants, and other materials may form emulsions that could reduce the amount of TOG and TPH extracted from a sample. This test method contains procedures that can assist the analyst in breaking such emulsions.

6.2 Organic compounds and other materials not considered as oil and grease or petroleum hydrocarbons on the basis of chemical structure (for example, halogenated hydrocarbons) may be extracted and measured by this test method.

7. Apparatus

7.1 All glassware that will come in contact with the sample shall be thoroughly cleaned, rinsed with distilled water and dried at 130 °C. Prior to starting this procedure, the glassware shall be rinsed with solvent and dried.

7.2 *Cell(s)*, calcium fluoride, two required for double-beam operation, one required for single-beam operation. The path length of the cells should be chosen for optimum signal to noise ratio at the measurement wavelengths. This will be 2 mm to 3 mm in case of quantum cascade laser based spectrometers. Longer path lengths may be appropriate, if more powerful lasers become available.

7.3 *Mid-infrared laser spectrometer* with an optical power >20 mW within the spectral region from 1370 cm^{-1} to 1380 cm^{-1} (7.25 µm to 7.30 µm). Either double-beam or single-beam and capable of measuring in the spectral region from 1370 cm^{-1} to 1380 cm^{-1} (7.25 µm to 7.30 µm) or single-beam instrument tunable from 1370 cm^{-1} to 1400 cm^{-1} (7.14 µm to 7.30 µm).[7]

7.4 *Wide mouth sample bottle*, made from glass, suggested 1 L, either with screw cap having a fluoropolymer liner or a wide-necked glass flask with a ground neck and with either glass or fluoropolymer stopper. The sampling bottle shall allow direct extraction from the bottle.

7.5 *Micro-separator* (for example, see Fig. X1.1), or other suitable device for phase separation.

7.6 *Clean-up columns*, made from glass, with frit of sinter porosity 2 (for example, see Fig. X1.2) or equivalent ready-made clean-up column containing Na_2SO_4 and Florisil (for TPH measurements).

7.7 *Glass bottle*, 50 mL to 100 mL, with glass or fluoropolymer stopper, or crimped cap with lined PTFE septum.

7.8 *Magnetic stirrer*, with PTFE stir bar (optional).

7.9 *Volumetric flasks*, glass, various (50 mL, 100 mL, and 200 mL).

7.10 PTFE *wash bottle*.

7.11 *Volumetric pipettes*, glass, various (10 mL, 20 mL, and 50 mL).

7.12 *Analytical balance*.

7.13 *Graduated glass syringes*, 5 µL and 25 µL.

7.14 *Benchtop shaker*.

7.15 *Glass stirring rod* (optional).

7.16 A *1.00-mL serological glass pipet* graduated in 0.01-mL increments and a *5.00-mL serological glass pipet* graduated in 0.1-mL increments, or equivalent (optional).

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specification of the Committee on Analytical Reagents of the American Chemical Society, if such specifications are available. 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] The sole source of supply of the Oil in Water analyzers known to the committee at this time is QuantaRed Technologies GmbH. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.