



Designation: D8478 – 23

Standard Test Method for N-Hexane Recoverable Total Oil and Grease and Total Petroleum Hydrocarbons in Water by ATR-Infrared Determination¹

This standard is issued under the fixed designation D8478; 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, and total petroleum hydrocarbons in produced water and wastewater by an infrared (IR) determination of n-hexane extractable substances from the sample. Included in this estimation of total oil and grease are any other compounds soluble in the n-hexane.

1.2 This test method defines total oil and grease in produced water and wastewater as that which is extractable in the test method and measured by IR absorption from $3.34\text{ }\mu\text{m}$ to $3.54\text{ }\mu\text{m}$ (2825 cm^{-1} to 2994 cm^{-1}). Similarly, this test method defines total petroleum hydrocarbons in produced water and wastewater as that oil and grease which is not adsorbed by silica gel in the test method, and is measured by IR absorption from $3.34\text{ }\mu\text{m}$ to $3.54\text{ }\mu\text{m}$ (2825 cm^{-1} to 2994 cm^{-1}). Alternative methods for total oil and grease or total petroleum hydrocarbons, or both, can produce differing results.

1.3 This method covers the range of 5 mg/L to 175 mg/L for total oil and grease and the range of 5 mg/L to 50 mg/L for total petroleum hydrocarbons. The range may be extended to a lower or higher level by extraction of a larger or smaller sample volume collected separately.

1.4 This test method uses horizontal attenuated total reflectance (HATR) with a cubic zirconia crystal.

1.5 This test method is intended as a field test only and should be treated as such. This method is not intended to replace laboratory-based regulatory methods currently in use.

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

1.7 *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. See Guide D3856 for more information.*

1.8 *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
- 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
- D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water
- 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 CFR Publications:³

- 40 CFR Part 136 Guidelines Establishing Test Procedures for the Analysis of Pollutants
- 49 CFR Part 172 Hazardous Materials Table, Special

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.

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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.

³ Available from U.S. Government Publishing Office (GPO), 732 N. Capitol St., NW, Washington, DC 20401, <http://www.gpo.gov>.

Provisions, Hazardous Materials Communications, Emergency Response Information, Training Requirements, and Security Plans

2.3 EPA Standards:⁴

Method 1664, Revision A N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGTHEM; Non-polar Material) by Extraction and Gravimetry

3. Terminology

3.1 Definitions:

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

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *horizontal attenuated total reflection (HATR) crystal*, *n*—horizontally oriented infrared transmitting crystal shaped so that light will pass through the filter while reflecting off the top and bottom crystal face; as light strikes the top crystal the light will be attenuated by any sample placed on the top crystal at specific wavelengths corresponding to the molecular shape and chemistry of the sample; this attenuation can be used to generate an infrared spectrum of the sample.

3.2.2 *total oil and grease (TOG)*, *n*—organic material that can be extracted from produced water or wastewater by this test method and which can be measured by infrared absorption in the region from 3.34 μm to 3.54 μm .

3.2.3 *total petroleum hydrocarbon (TPH)*, *n*—non-polar organic material that can be extracted from produced water or wastewater, and which can be measured by infrared absorption in the region from 3.34 μm to 3.54 μm .

4. Summary of Test Method

4.1 A sample of produced water or wastewater between 100 mL and 250 mL is extracted with a proportional amount of n-hexane at a ratio of 10:1 (sample to n-hexane). A portion of the extract is evaporated on the HATR crystal and examined by infrared spectroscopy (IR) for a total oil and grease measurement.⁵ If a total petroleum hydrocarbons measurement is required, a portion of the extract is contacted with silica gel to remove polar substances, thereby producing a solution containing non-polar material. The non-polar material is measured by infrared spectroscopy.

5. Significance and Use

5.1 The presence and concentration of oil and grease 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 TOG and TPH in produced water and wastewater.

6. Interferences

6.1 Soaps, detergents, surfactants, and other materials can form emulsions that can reduce the amount of oil and grease 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 on the basis of chemical structure can be extracted and measured as total oil and grease. Of those measured, certain ones will be adsorbed by silica gel while others will not. Those not adsorbed are measured as total petroleum hydrocarbons.

6.3 Sulfur present in the sample can extract into n-hexane and cause a positive bias in the results. See **Annex A2** for testing details.

7. Apparatus

7.1 *Filter Paper*, ashless, quantitative, general-purpose, 11 cm diameter, 8 μm pore size.

7.2 *Glass Sample Bottle*, minimum 125 mL, with screw cap having a fluoropolymer liner.

7.3 *Glass Graduated Cylinder*, 100 mL

7.4 *Glass Funnel*.

7.5 *Volumetric Flasks*, glass, various.

7.6 *Polytetrafluoroethylene (PTFE) Spritz Bottle*, one-piece wash bottle for rinsing.

7.7 *Syringes*, 100 μL , 25 mL.

7.8 *Glass Separatory-Funnel*, 500 mL, with fluoropolymer stopcock and stopper (optional).

7.9 *Volumetric Pipettes*, glass, various (optional).

7.10 *Analytical Balance* (optional).

7.11 *Infrared Analyzer*, scanning or non-scanning, capable of measuring absorption from 3.34 μm to 3.54 μm (2825 cm^{-1} to 2994 cm^{-1}). In order for the analyzer to comply, the standard deviation of 10 measurements with no sample present shall not exceed 0.1 mAU.

7.11.1 *HATR Crystal*. The apparatus shall use a cubic zirconia HATR crystal which contains a trough that prevents the n-hexane from spreading off the crystal.

8. Reagents and Materials

8.1 *Purity of Reagents*—Spectroscopic grade (preferred) or reagent grade chemicals shall be used in tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the committee on analytical reagents of the American Chemical Society, where such specifications are available.⁶ Other grades may be used, provided it is first

⁴ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

⁵ Consult the manufacturer's operation manual for the specific instructions related to the infrared spectrometer or analyzer to be used.

⁶ *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.