Designation: D7575 – 11

Standard Test Method for Solvent-Free Membrane Recoverable Oil and Grease by Infrared Determination[1]

This standard is issued under the fixed designation D7575; 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 oil and grease in produced and waste water samples over the concentration range outlined in Table 1 that can be extracted with an infrared-amenable membrane and measured by infrared transmission through the membrane.

1.2 This method defines oil and grease in water as that which is extractable in the test method and measured by infrared transmission.

1.3 The method detection limit (MDL) and recommended reporting range are listed in Table 1.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

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
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
E168 Practices for General Techniques of Infrared Quantitative Analysis
E178 Practice for Dealing With Outlying Observations

TABLE 1 MDL and Reporting Range

Analyte	MDL[A] (mg/L)	Reporting Range[A] (mg/L)
Oil and Grease	1.0	5–200

[A] MDL and recommended reporting range determined by Section 12.4, which follows the Code of Federal Regulations, 40 CFR Part 136, Appendix B; limits should be determined by each operator.

2.2 *EPA Standards*[3]

EPA Method 1664 Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-polar Material) By Extraction and Gravimetry
40 CFR
49 CFR

3. Terminology

3.1 *Definitions:* 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 *oil and grease*, *n*—"membrane-recoverable oil and grease" is a method-defined analyte; that is, the definition of membrane-recoverable oil and grease is dependent on the procedure used.

3.2.1.1 *Discussion*—The nature of the oils or greases (or both), and the presence of recoverable non-oily matter in the sample will influence the material measured and interpretation of results.

3.2.2 *extractor*, *n*—a device that contains an infrared-amenable oil-and-grease solid-phase-extraction-membrane and directs water flow through the membrane under applied pressure.

4. Summary of Test Method

4.1 This is a performance-based method and modifications are allowed to improve performance.

4.2 A sample of water is processed through an extractor.

[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 United States Environmental Protection Agency (EPA), Ariel Rios Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

4.3 The extractor is then sufficiently dried of water so as to allow infrared analysis.

4.4 The extractor is examined by an infrared analyzer for an oil and grease measurement.

4.5 Calibrations and data are processed manually or with appropriate software.

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 health, environmental, safety, and aesthetic effects.

5.2 Regulations and standards have been established that require monitoring of oil and grease in water and wastewater.[4]

NOTE 1—Different oil and grease materials may have different infrared absorptivities. Certain materials, such as synthetic silicone-based or perfluorinated oils, may have absoptivities inconsistent with those of naturally occurring oil and grease materials. Caution should be taken when testing matrices suspected of containing proportions of these materials. In such cases, laboratory spike samples, laboratory check samples, equivalency testing, or combinations thereof, using these materials in question may be appropriate.

6. Interferences

6.1 Method interferences may be caused by contaminants in instrumentation, reagents, glassware and other apparatus producing artifacts. Routine laboratory method blanks will demonstrate all these materials are free from interferences.

6.2 Matrix interference may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences can vary considerably from sample to sample.

6.3 In cases of samples which contain a relatively large amount of particulate or biological material, processing the standard 10 mL amount of sample may not be possible. Note 2 and Note 10 discuss how to deal with processing such samples.

NOTE 2—It is important to note that the capture of solid matter on the extractor does not preclude IR measurement; in the majority of cases there is sufficient IR throughput to still perform the measurement as described herein. This is the case with most metal-oxide materials (that is, clay or sand) and biological material (that is, algae or cellulose). There may of course be samples encountered wherein the solid matter is not sufficiently IR transmitting; one example may be a sample containing a large concentration of metal particulate. In these instances a different measurement technique may be necessary.

7. Apparatus

7.1 *Extractor*—Device which contains an infrared-amenable oil and grease solid phase extraction membrane, includes a connection to a syringe, such as a Luer connection, and is designed for pressurized flow of water through the membrane.[5]

7.2 *Calibration Standard Devices Set*—Calibration standards have the same or similar outward appearance as the extractor. Each set contains devices with a specified amount of oil and grease; set should include seven devices that cover the reporting range.[6]

7.3 *Syringe*—A one-time use plastic syringe with low-extractable components and connection to attach to the extractor, capable of flowing the sample volume to be processed.

7.4 *Infrared Instrument*—Infrared absorption measurement instrument; the instrument may be spectroscopic, dispersive, radiometric or filtometric based. The method was validated and the detection limit was determined with an MB3000 FTIR spectrometer manufactured by ABB according to 12.4; the detection limit and reporting range may vary with the instrument chosen to perform the analysis; the user should perform a detection limit study as described in 12.4 to determine the method detection limit and reporting range when using the chosen instrument.

7.5 *Homogenizer*—A device capable of sufficiently homogenizing a collected sample, if a grab sample is collected and stored prior to testing; examples are a paint can shaker or table shaker (optional).

7.6 *Fluid Flow Device*—A device capable of forcing the fluid through the extractor, such as a syringe pump (optional).

7.7 *Drying System*—A system capable of drying the extractor sufficiently for infrared analysis without compromising analyte retention; an example is a clean, compressed air line at 80 psi (552 kPa).

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, where such specifications are available.[7] 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water that meets the purity specifications of Type II water, presented in Specification D1193.

8.3 *Hydrochloric Acid*—Concentration of 12.1 M.

8.4 *Sulfuric Acid*—Concentration of 18.4 M; optional replacement for hydrochloric acid for preservation.

8.5 *Acetone*—ACS, residue less than 1 mg/L.

8.6 *Hexadecane*—98 % minimum purity.

8.7 *Stearic Acid*—98 % minimum purity.

[4] 40 CFR 136

[5] The sole source of supply of the apparatus known to the committee at this time is Orono Spectral Solutions, P/N 1018SPE (US Patent Application number 12/324,688). 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, which you may attend.

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[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual 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.

9. Hazards

9.1 Normal laboratory safety applies to this method. Analysts should wear safety glasses, gloves, and lab coats when working with acids. Analysts should review the Material Safety Data Sheets (MSDS) for all reagents used in this method. Additional hazards may be presented by the particular sample being tested so proper care must be taken.

10. Sampling

10.1 Fill the sample container. Do not fill the container to the brim; sufficient headspace is required to allow vigorous homogenization. Do not rinse the sample container with the sample to be analyzed. Do not allow the sample to overflow the container during collection. Preventing overflow may not be possible in all sampling situations; however, measures should be taken to minimize overflow at all times.

NOTE 3—About 5–10 % of volume headspace has been found to be suitable for homogenization.

10.2 Add a sufficient quantity of either sulfuric (see Section 8.4) or hydrochloric acid (see Section 8.3) to a pH of 2. If analysis is to be delayed for more than four hours, refrigerate to 6°C or less, without freezing, from the time of collection until extraction. The amount of acid required will be dependent upon the pH and buffer capacity of the sample at the time of collection. If the amount of acid required is not known, make the pH measurement on a separate sample that will not be analyzed. Introduction of pH paper to an actual sample or sample cap may remove some oil from the sample. To more accurately calculate the final oil and grease concentration the following equation can be used:

$$C_S = C_i \times (V_S + V_A) / V_S \quad (1)$$

where C_i is the measured concentration, V_S is the sample volume, V_A is the volume of acid added to the sample, and C_S is the sample concentration before the acid was added.

10.3 If the sample is to be shipped by commercial carrier, U.S. Department of Transportation regulations (see 49 CFR part 172) limit the pH to a minimum of 1.96 if HCl is used and 1.15 if H_2SO_4 is used. (see 40 CFR Part 136, Table II Footnote 3).

NOTE 4—For those circumstances requiring the collection of multiple aliquots of one sample, each aliquot is to be collected in either of the following ways: *(1)* collect simultaneously in parallel, if possible, or *(2)* collect as grab samples in rapid succession, filling ⅓ of each container at a time and continuing until all containers are 90–95 % full, consistent with Note 3.

11. Preparation of Apparatus

11.1 *Hexadecane and Stearic Acid (1+1) Spiking Solution*—Place 400 mg ±4 mg hexadecane and 400 mg ±4 mg stearic acid in a 100-mL volumetric flask and fill to the bottom of the neck, not to the mark, with acetone.

NOTE 5—The solution may require warming for complete dissolution of stearic acid.

11.2 After the hexadecane and stearic acid has dissolved, allow to cool to room temperature and add acetone to the mark. Stopper the volumetric flask or transfer the solution to a 100–150 mL vial with fluoropolymer-lined cap. Mark the solution level on the vial and store in the dark at room temperature.

11.3 Immediately prior to the first use, verify the level on the vial and bring to volume with acetone, if required. Warm to redissolve all visible precipitate, if required. If there is doubt of the concentration, remove 10.0 ± 0.1 mL with a volumetric pipet, place in a tared weighing pan, and evaporate to dryness in a fume hood. The weight must be 80 ± 1 mg. If not, prepare a fresh solution (Section 11.1).

11.4 The spiking solutions should be checked frequently for signs of degradation or evaporation using the test in Section 11.3.

11.5 If necessary, this solution can be made more or less concentrated to suit the concentration needed for the matrix spike. A fresh spiking solution should be prepared weekly or bi-weekly.

12. Calibration and Standardization

12.1 To ensure analytical values obtained using this test method are valid and accurate within the confidence limits of the test, the instrument manufacturer's instructions and the following procedures must be followed when performing the test method.

NOTE 6—Instruments other than FTIR spectrometers may have different procedures that should be followed according to the manufacturer's instructions.

12.2 Calibration is carried out using the set of calibration standard devices (CSD).

12.2.1 Take a background reference file through the CSD labeled "Background" according to the instrument manufacturer's instructions.

12.2.2 Scan each of the other CSDs according to the instrument manufacturer's instructions.

12.2.3 Measure and record the absorbance of the peak centered near 2920 cm^{-1}(3.42 micron) according to Practices E168. The instrument may include automatic measurement software; if so follow instrument manufacturer's instructions for using the software.

NOTE 7—Other peaks associated with the methylene moiety may also be used; detection limits will be affected so the operator should follow Section 12.4 to determine the detection limit for the absorbance peaks chosen.

12.2.4 Linear calibration may be used if the coefficient of determination, r^2, is >0.95 for the analyte. If one of the calibration standards other than the high or low point causes the r^2 to be <0.95 this point must be reanalyzed. If the point still causes the r^2 to be <0.95, it may be excluded but minimally a six point calibration is required. The high or low point of the calibration may be excluded but the reporting range must be modified to reflect this change. If two points must be excluded to attain an r^2>0.95, calibration must be repeated, and if this still is not achieved, calibration must be repeated with a new set of calibration standard devices.

12.2.5 Quadratic calibration may be used if the coefficient of determination, r^2, is >0.97 for the analyte. If one of the calibration standards other than the high or low point causes the r^2 to be <0.97 this point must be reanalyzed. If the point