Designation: D7575 – 10$^{\varepsilon 1}$

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.

ε[1] NOTE—Research report information was editorially added to Section 17 in March 2010.

1. Scope

1.1 This test method covers the determination of oil and grease in water 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

2.2 *EPA Standards*[3]

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.

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.

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.

[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.

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.

[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.

[6] The sole source of supply of the apparatus known to the committee at this time is Orono Spectral Solutions, P/N 1018SPE-CSD. 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.

[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.

8.5 *Acetone*—ACS, residue less than 1 mg/L.
8.6 *Hexadecane*—98 % minimum purity.
8.7 *Stearic Acid*—98 % minimum purity.

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 still causes the r^2 to be <0.97, it may be excluded but minimally a six point calibration is required. The high or low point of the calibration may be excluded to attain an r^2> 0.97, but the reporting range must be modified to reflect this change. If two points must be excluded to attain an r^2> 0.97 calibration must be repeated, and if this still is not achieved, calibration must be repeated with a new set of calibration standard devices.

12.3 If a laboratory has not performed the test before, or if there has been a major change in the measurement system, for example a new analyst or new instrument, perform a method detection limit and a precision and bias study to demonstrate laboratory capability.

12.4 *Method Detection Limit (MDL)*:

12.4.1 The MDL procedure that follows is a paraphrased version of the MDL procedure in the 40 CFR Part 136, Appendix B.

12.4.2 Analyze at least seven replicates of a sample at a concentration near three times the expected method detection limit (that is, 4 mg/L). A laboratory spike sample, matrix sample, or a sample similar to the desired matrix to be examined may be tested. The complete method must be followed, including the preservation and pretreatment steps.

12.4.3 The method detection limit (MDL) is determined as 3.143 times the standard deviation of measured concentration of the seven replicates. The recommended reporting limit is 10 times the standard deviation of the seven replicates. The mean (average) measured concentration must be 1–5 times the calculated method detection limit; if this criterion is not met repeat the detection limit study.

12.4.4 This study should be repeated until the desired method detection limit is achieved, with the lowest possible detection limit being the lowest calibration standard device level used in Section 12.2. However, the recommended reporting limit is still 10 times the standard deviation of the seven replicates.

12.5 *Initial Precision and Recovery*:

12.5.1 Analyze at least four replicates of a sample containing 40 mg/L of oil and grease. A laboratory spike sample, matrix sample, or a sample similar to the desired matrix to be examined may be tested. The complete method must be followed, including the preservation and pretreatment steps.

12.5.2 Calculate the mean (average) recovery and relative standard deviation (RSD) of the four replicates. The mean recovery must be within the range given in Table 2. This study should be repeated until the single operator precision and recovery are within the recommended limits. If a concentration other than the recommended concentration is used refer to Practice D5847 for information on applying the F test and t test in evaluating the acceptability of the mean recovery and RSD.

12.6 *Laboratory Control Sample (LCS)*:

12.6.1 To be sure the test method is in control, analyze an LCS prepared with the spiking solution at 40 mg/L in Type II reagent water. An LCS should be analyzed with each set of 20 samples or once a day.

12.6.2 The LCS recovery should be within the limits given in Table 2. If the result is not within this range, analysis of samples is halted until the problem is corrected and either all samples in the batch must be re-analyzed or the results must be qualified with a statement indicating they do not fall within the performance criteria of the test.

12.7 *Method Blank*:

12.7.1 Analyze a reagent water blank with each batch of 20 or fewer samples. The blank must be taken through all the method steps, including the preservation and pretreatment. The concentration of oil and grease found in the blank must be below the MDL determined in Section 12.4 or less than 10 % of the known concentration of the associated test samples.

12.7.2 If the concentration of oil and grease in the blank is found above this level, analysis of samples is halted until the contamination is eliminated and the blank shows no contamination above this level or the results must be qualified with a statement indicating they do not fall within the performance criteria of the test method.

12.8 *Matrix Spike (MS)*:

12.8.1 To check for interferences in the specific matrix being tested, perform an MS on at least one sample from each batch of 20 samples by spiking the sample with a known concentration of oil and grease using the spiking solution and following the analytical method including preservation and pretreatment. The spike must produce a sample that is 1.5 times the original concentration or 30 times the method detection limit as determined in Section 12.4, whichever is greater. If the spiked concentration plus the background concentration exceeds the highest calibration standard device used in Section 12.2, the sample must be diluted to near 40 mg/L.

12.8.2 If the MS sample is above the calibration range the matrix spike must be diluted.

12.8.3 Calculate the percent recovery according to Eq 2:

$$MSR = 100\,[(A(V_s + V) - BV_s] / (CV) \quad (2)$$

where:

A = concentration found in spiked sample,
B = concentration found in unspiked sample,
C = concentration of analyte in spiking solution,
V_s = volume of sample that is spiked,

TABLE 2 Interlaboratory Validation Study QC Acceptance Criteria

Analyte	Initial Precision and Recovery				Lab Control Sample		Matrix Spike	
	Test Concentration (mg/L)	Recovery (%)		Precision	Recovery (%)		Recovery (%)	
		Lower limit	Upper limit	Maximum RSD (%)	Lower limit	Upper limit	Lower limit	Upper limit
Oil and Grease	40	88	105	10.5	79	113	70	126

V = volume of spiking solution added, and
MSR = percent recovery.

12.8.4 The percent recovery of the spike shall fall within the limits given in Table 2. If the percent recovery is not within these limits, it is suggested that another aliquot from the same bottle is processed by the method. If neither the first nor second aliquots are within the limits, a second sample bottle should be spiked if available. If the matrix spike fails again, a matrix interference must be present. Under these circumstances, one of the following must be employed: the matrix interference must be removed, all samples in the batch must be reanalyzed by a test method not affected by the matrix interference, or the results must be qualified with a statement indicating that they do not fall within the performance criteria of the test method.

13. Preparation of Infrared Instrument

13.1 Preparation of the infrared instrument will vary by type of analysis instrument chosen. Follow the instructions of the instrument manufacturer.

NOTE 8—As an example, preparation of an FTIR instrument is described. At the start of each analysis day, take a new background file through a blank extractor. Depending on ambient laboratory conditions, it may be helpful to take another new background file as necessary as environmental conditions change. It is not necessary to take the background through the same extractor that will be used for the extraction.

14. Procedure

14.1 *Calibration verification*—Verify the calibration each day the test is to be performed by re-analyzing the CSD at or near 40 mg/L. If the verification value varies from the value on the CSD label by more than 5 %, recalibrate the instrument as described in Section 12.2.

14.2 *Sample pretreatment*—If the sample to be tested is a grab sample, vigorously shake the sample bottle to sufficiently mix the sample. The sample bottle may require more shaking if collected seven or more days prior to testing. It is up to the operator to ensure the sample is homogeneous prior to proceeding with the measurement.

NOTE 9—It has been found that vigorously shaking a sample bottle for 20 minutes with a paint shaker or an appropriate laboratory shaker with sample container holder accessory is sufficient to homogenize a sample that has been stationary for the maximum allowed holding time of 28 days in the vast majority of cases. A sample processed very soon after taking the sample or created in the lab by spiking immediately before testing may require minimal shaking. If an inspection of the sample clearly indicates this amount of shaking has not sufficiently homogenized the sample (that is, stratification in the sample container), further homogenization by the same method or a different method may be required. In general it has been found that contact mixing (that is, ultrasonic homogenization, stand mixer, etc.) of the sample does not cause any interferences for the method. If the sample is known to be problematic, it may be ideal to collect the sample in a wide mouth jar to facilitate contact mixing.

14.3 *Extraction*:

14.3.1 Within one minute of completing 14.2, draw the sample into a syringe.

14.3.2 Within 5 minutes of drawing the sample into the syringe, attach an extractor to the syringe.

14.3.3 Within 5 minutes of attaching the extractor to the syringe, flow 10 mL of the sample through the extractor at a rate of 5 mL/min. Ensure air bubbles do not interfere with the flow of sample through the extractor by holding the syringe and extractor vertically with flow upwards during the entire course of the extraction. A syringe pump or similar device may be used.

NOTE 10—If the amount of particulate in the sample clogs the extractor and precludes processing a 10 mL sample, repeat the procedure of Section 14 except using a smaller volume of sample; alternatively dilute the sample and re-run the analysis, making sure to account for the dilution in the final calculation of sample oil and grease concentration. It is recommended that a new MDL study be performed to determine the MDL and lower limit of the reporting range for processing a volume other than 10 mL.

14.4 *Drying*:

14.4.1 Dry the extractor using the drying system until sufficient water has been removed so that an IR absorbance measurement can be performed.

NOTE 11—Drying time will vary from sample to sample. In some cases, the extractor could be sufficiently dry after one minute of drying. In others, a longer drying time may be necessary. Drying may be complete when the color of the sample on the membrane has lightened in color, indicating most of the water is gone. Also, sufficient dryness may be indicated when compressed air directed to flow through the membrane flows at some rate. If a spectrometer is used, the dryness can be determined from the spectrum by spectral analysis, with examples shown the Appendix. An iterative drying/measurement (as in Section 14.5) procedure could be used to confirm dryness. Instrument software may contain automated dryness confirmation; if so, follow the instrument manufacturer's instructions. The humidity of the air or other gas leaving the extractor may indicate level of dryness, whereby the humidity will be high prior to dryness and approach the humidity level of the feed gas when the extractor is sufficiently dry.

14.5 *Infrared Absorbance Measurement*:

14.5.1 Measure and record the infrared absorbance at the peak of the extract according to Practices E168 for the peak near 2920 cm^{-1} (3.42 micron) or other absorbance bands as discussed in Section 12.2. The instrument may include automatic measurement and calculation software; if so follow instrument manufacturer instructions for using the software.

15. Calculation or Interpretation of Results

15.1 Determine the concentration of the sample manually according to the calibration determined in Section 12.2. If a volume different than 10 mL was used adjust the concentration according to:

$$C_S = C_i \times V_C / V_P \quad (3)$$

where C_i is the concentration determined according to the calibration curve, V_C is the volume of sample for which the calibration standard devices were intended to be used, V_P is the volume of sample processed, and C_S is the concentration of the sample. Alternatively, automatic calculation software may be included with the infrared instrument; in this case, follow the manufacturer's instructions for using the software.

16. Report

16.1 Determine the results in units of mg/L in a water sample. Calculate the concentration in the sample using the calibration curve calculated in Section 12.2. All data that does not meet the specifications in the test method must be appropriately qualified.

17. Precision and Bias [8]

17.1 Initially, an independent laboratory study was performed to determine preliminary method values of precision and bias across a range of real-world matrices. Data are shown in Tables 3-7. Results of this test method were compared to average results from testing the same matrices by EPA Method 1664. It was found that this test method and EPA Method 1664 results were comparable at 95 % confidence. This single laboratory study helped define procedures and initial statements of precision and bias that would shape the test plan for the next phase of testing—an interlaboratory study.

17.2 This test method was then tested by twelve independent laboratories during an interlaboratory validation study.

17.3 The test design of the study meets the requirements of Practice D2777 for the analytes listed in this test method with one exception. Due to the cost and logistics of performing the analysis, each matrix tested contained one set of Youden pair concentrations. In accordance with Section 1.5 of Practice D2777, an exception from the requirement for using three Youden pairs within each matrix was granted by the Technical Operations Committee of D19 on the recommendation of the Results Advisor in order to enable evaluation of the method based on more than one matrix. The exception specified that a single Youden pair be used for each matrix and that the range of concentrations represented by all three Youden pairs thus formed cover the range of the test method.

17.4 The precision and bias data (Table 1 and Table 2) for this method are based on this testing. A summary of this interlaboratory study data is provided in Tables 8-11.

17.5 It is the user's responsibility to ensure the validity of precision and bias outside of the interlaboratory validation study ranges and matrices.

NOTE 12—EPA Method 1664 data shown in Tables 5-7 and Table 11 are results using the liquid-liquid extraction process.

TABLE 4 Single Laboratory Study: Initial Precision and Recovery

Matrix: Reagent Water
Fortification Level: 40 mg/L

Measured Result mg/L as Oil and Grease			
Replicate #1	Replicate #2	Replicate #3	Replicate #4
34.8	36.2	33.3	39.1
Percent Recovery (%) as Oil and Grease			
87 %	91 %	83 %	98 %

Average Recovery of Four Replicates: 35.9 mg/L, 90 %
Standard Deviation of Recovery: 2.5 mg/L, 6 % RSD

18. Keywords

18.1 dispersive infrared; filtometric infrared; FTIR; grease; infrared transmission; oil; solid phase extraction; spectroscopy; water

[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1183.

TABLE 3 Method Detection Limit and Minimum Level

Matrix: Reagent Water
Fortification Level: 4 mg/L

Measured Result mg/L as Oil and Grease						
Replicate #1	Replicate #2	Replicate #3	Replicate #4	Replicate #5	Replicate #6	Replicate #7
4.9	4.3	4.1	3.7	4.3	4.8	4.4

Average of Seven Replicates: 4.36
Standard Deviation of Seven Replicates: 0.41
MDL (Student's t 3.143 × Standard Deviation) = 1.29 mg/L
Recommended lower reporting limit (SD × 10, rounded to nearest whole number) = 4.1 rounded to 5 mg/L