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Standard Test Method for Oil and Grease and Petroleum Hydrocarbons in Water [1]

This standard is issued under the fixed designation D 3921; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of fluorocarbon-extractable substances as an estimation of the combined oil and grease and the petroleum hydrocarbon contents of a sample of water or waste water in the range from 0.5 to 100 mg/L. It is the user's responsibility to assume the validity of the standard for untested types of water.

1.2 This test method defines oil and grease in water and waste water as that matter which is extractable in the test method and measured by infrared absorption. Similarly, this test method defines petroleum hydrocarbons in water and waste water as that oil and grease which is not adsorbed by silica gel in the test method and that is measured by infrared absorption.

1.3 Low-boiling organic materials are lost by evaporation during the manipulative transfers. However, these evaporative losses are generally much lower than those experienced with gravimetric procedures that require solvent evaporation before the residue is weighed.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[2]

D 3325 Practice for Preservation of Waterborne Oil Samples[3]

D 3370 Practices for Sampling Water from Closed Conduits[2]

D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water[2]

D 4210 Practice for Interlaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data[2]

D 5789 Practice for Writing Quality Control Specifications for Standard Test Methods for Organic Constituents[3]

E 168 Practices for General Techniques of Infrared Quantitative Analysis[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129 and Practices E 168.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *oil and grease*—the organic matter extracted from water or waste water and measured by this test method.

3.2.2 *petroleum hydrocarbons*—the oil and grease remaining in solution after contact with silica gel and measured by this test method.

4. Summary of Test Method

4.1 The acidified sample of water or waste water is extracted serially with three 30-mL volumes of 1, 1, 2-trichloro-1, 2, 2-trifluoroethane (referred to in this test method as solvent). [5] The extract is diluted to 100 mL and a portion is examined by infrared spectroscopy [6] to measure the amount of oil and grease removed from the original sample. A major portion of the remaining extract is contacted with silica gel to remove polar substances, thereby providing a solution of petroleum hydrocarbons. This treated extract is then similarly examined by infrared spectroscopy.

5. Significance and Use

5.1 The presence of oil and grease in domestic and industrial waste water is of concern to the public because of its deleterious aesthetic effect and its impact on aquatic life. Regulations and standards have been established that require monitoring of oil and grease in water and waste water. This test method provides an analytical procedure to measure oil and grease in water and waste water.

6. Interferences

6.1 Since the constituents oil and grease and petroleum

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[1] This test method is under the jurisdiction of ASTM Committee D-19 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] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] Gruenfeld, M., "Extraction of Dispersed Oils from Water for Quantitative Analysis by Infrared Spectrophotometry," *Environmental Science and Technology*, Vol 7, 1973, pp. 636–639.

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

hydrocarbons are defined as the results of the test procedure, interferences are precluded by definition. Interpretation of test results on the basis of chemical structure, pollution potential, or treatability should be approached with caution, however, because of the diversity of substances measured by this procedure.

6.2 Organic solvents and certain other organic compounds not considered as oil and grease on the basis of chemical structure may be extracted and measured as oil and grease. Of those measured, certain ones may be adsorbed by silica gel while others may not. Those which are not adsorbed are measured as petroleum hydrocarbons.

7. Apparatus

7.1 *Cell(s)*, quartz, 10-mm path length, two required for double-beam operation, one required for single-beam operation, or built-in cell for nondispersive infrared analyzer operation.

7.2 *Filter Paper*, ashless, quantitative, general-purpose, 11-cm or equivalent.

7.3 *Glass Bottle*, approximately 1000-mL, with screw cap having a TFE-fluorocarbon liner.

7.4 *Graduated Cylinder*, 1000-mL.

7.5 *Infrared Spectrometer*, double-beam dispersive, single-beam dispersive, Fourier transform, or nondispersive infrared analyzer.

7.6 *Magnetic Stirrer*, with small TFE-fluorocarbon stirring bar.

7.7 *Separatory Funnel*, 2000-mL, with TFE-fluorocarbon stopcock (one for each sample analyzed during any one period of time).

7.8 *Volumetric Flask*, 100-mL (minimum of six required for calibration plus one for each sample analyzed during any one period of time).

8. Reagents

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, [7] where 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.

8.2 *Purity of Water*— Unless otherwise indicated, references to water (not sample water) shall be understood to mean reagent water conforming to Specification D 1193, Type II.

8.3 *Calibration Oil and Grease*, similar in composition to oil and grease determined by this test method for possible use as calibration material.

8.4 *Cetane (n-Hexadecane)*, 99 % minimum purity, for possible use in calibration mixture.

8.5 *Isooctane (2,2,4-Trimethylpentane)*, 99 % minimum purity, for possible use in calibration mixture.

8.6 *Silica Gel* [8], 100 to 200 mesh, which has been deactivated with 2 % water.

8.7 *Sodium Bisulfate* ($NaHSO_4$), monohydrate.

8.8 *Sodium Sulfate* (Na_2SO_4), anhydrous, granular.

8.9 *Solvent* —1, 1, 2-trichloro-1, 2, 2- trifluoroethane. [9]

NOTE 1—Frequently, this solvent will extract plasticizer from the liner of its shipping container. Check for such contamination by evaporating 100 mL of solvent in a steam bath and weighing its residue. If this value exceeds 0.1 mg, purify the solvent by distillation and check the overhead material for residue. Store the purified solvent in clean, glass bottles having TFE-Fluorocarbon cap liners. Purification of this solvent as a matter of course is highly desirable.

8.10 *Sulfuric Acid (1 + 1)*—Slowly and carefully add 1 volume of sulfuric acid (H_2SO_4, sp gr 1.84) to 1 volume of water, stirring and cooling the solution during the addition.

9. Sampling

9.1 Collect the sample in accordance with the principles described in Practices D 3370, using a glass bottle equipped with a screw cap having a TFE-fluorocarbon liner.

9.2 A sample of about 750 mL is required for this test. Use the entire sample since no portion should be removed for other tests.

9.3 Preserve the sample with a sufficient quantity of either sulfuric acid (see 8.10) or sodium bisulfate (see 8.7) to attain a pH of 2 or lower. The amount of reagent required will be dependent upon the pH of the sample at the time of collection and upon its buffer capacity.

10. Calibration

NOTE 2—A choice of two calibration species is available to the analyst. The preferred material is a sample of the same oil and grease that is known to be present in the sample of water or waste water awaiting analysis. The other material is a mixture of *iso*octane and cetane. This latter blend is to be used when the same (as described) material is not available.

10.1 If the blend of *iso*octane and cetane is to be used for calibration, prepare a calibration mixture by pipetting 15 mL of *iso*octane and 15 mL of cetane into a glass-stoppered bottle. Mix the contents well and maintain the integrity of the mixture by keeping the container tightly sealed except when a portion is withdrawn for blending.

10.2 *Calibration Solution Blend A*—Place about 20 mL of solvent into a 100-mL volumetric flask, stopper, and weigh. To this flask quickly add about 1 mL of either the calibration oil and grease or the calibration mixture of *iso*octane and cetane. Obtain its exact weight by difference. Fill to the mark with solvent and mix the liquid well by shaking the flask. Calculate the exact concentration of the calibrating material in solution in terms of mg/100 mL. If the calibration oil and grease is used, proceed to 10.3. If the calibration mixture is used, multiply this calculated concentration (about 730 mg/100 mL) by 1.4 (refer

[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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[8] Silica Gel, Davison Chemical Grade 923 has been found to be satisfactory for this purpose. Other available types from the same or different suppliers may be suitable.

[9] This solvent is available also as Freon 113, Freon TF, Freon PCA, Genetron 113, Genesolve D, and as other names.

to Note 3). This new concentration value (about 1022 mg/100 mL) is to be used for Blend A throughout the remainder of this test method.

NOTE 3—Dating back to at least 1951,[10] for many years a mixture of *iso*octane, cetane, and benzene was accepted as a standard for calibration. Concern regarding the hazards of exposure to benzene, which acts here only as a diluent having no contribution at 2930 cm^{-1} (3.41 µm), has prompted elimination of this chemical as a component for calibration. To maintain relevance between current and future analytical data with those of the past, it is necessary to compensate for differences in concentration and in density between the former and the present calibration standards. The factor of 1.4 accomplishes this because the weight ratio of combined *iso*octane plus cetane in the new two-way mixture to that in the older three-way mixture is 1.000 to 0.714, or 1.40. Henceforth, all concentrations involving the calibration mixture will be based upon the converted value obtained in 10.2.

10.3 *Calibration Solution Blend B*—Dilute 4 mL of Blend A with solvent in a 100-mL volumetric flask (about 41 mg/100 mL).

10.4 *Calibration Solution Blend C*—Dilute 3 mL of Blend A with solvent in a 100-mL volumetric flask (about 31 mg/100 mL).

10.5 *Calibration Solution Blend D*—Dilute 50 mL of Blend B with solvent in a 100-mL volumetric flask (about 20 mg/100 mL).

10.6 *Calibration Solution Blend E*—Dilute 30 mL of Blend C with solvent in a 100-mL volumetric flask (about 9 mg/100 mL).

10.7 *Calibration Solution Blend F*—Dilute 10 mL of Blend E with solvent in a 100-mL volumetric flask (about 0.9 mg/100 mL).

NOTE 4—During the calibration events which follow, the cell used for the blends must be thoroughly cleaned with fresh solvent and then dried prior to the addition of a new blend. Take care to avoid insertion of the cell stopper so tightly that the cell could burst from expansion of its contents as it resides in the light beam. It is desirable to flush the cell compartment of the spectrometer with nitrogen or dry air to prevent chemical reaction of solvent fumes with components of the instrument. For double-beam operation, either block the light beam from the reference cell containing solvent or remove the reference cell from the instrument during the intervals between scans in order to protect the solvent from unnecessary warming. However, place the reference cell in the reference beam during all scans. For single-beam operation, use the same cell throughout the calibration procedure. Rely upon sole recommendations of the manufacturer for single-beam and nondispersive infrared analyzers since variations in design make it impractical to offer instructions for their use with this method. Also, in relation to nondispersive infrared operation, reference to scanning or running, or both, should be interpreted to mean obtaining a reading or a plot of the 2930-cm^{-1} (3.41-µm) band.

10.8 Fill the reference cell (for double-beam operation) and the sample cell with solvent and scan from 3200 cm^{-1} (3.13 µm) to 2700 cm^{-1} (3.70 µm). A nearly horizontal, straight line should be obtained. If it is not, check cells for cleanliness, matching, etc. Drain and clean the sample cell. Obtain spectral data for the solvent at this time for single-beam and nondispersive infrared instruments, also. After running, drain, and clean the sample cell.

10.9 Fill the sample cell with Blend B. Scan as in 10.8; drain, and clean the sample cell.

10.10 Fill the sample cell with Blend C. Scan as in 10.8; drain, and clean the sample cell.

10.11 Fill the sample cell with Blend D. Scan as in 10.8; drain, and clean the sample cell.

10.12 Fill the sample cell with Blend E. Scan as in 10.8; drain, and clean the sample cell.

10.13 Fill the sample cell with Blend F. Scan as in 10.8; drain, and clean the sample cell.

10.14 For each double-beam spectrum obtained in 10.9 through 10.13, draw a baseline similar to that found in Fig. 1. Obtain the net absorbance for the peak that occurs near 2930 cm^{-1} (3.41 µm). Obtain net values for single-beam and nondispersive infrared runs as recommended.

NOTE 5—For infrared instruments having computer capability, data may be obtained automatically or as described in 10.14. However, all data must be obtained consistently by one means or the other, not a combination of the two.

10.15 On linear graph paper, plot the new absorbance values, found in 10.14 or as permitted in Note 5, versus the respective mg/100 mL values for each of the blends examined. The points should lie very nearly in a straight line. Draw the best-fitting straight line through the points and keep this calibration graph for use with the test samples. Alternatively, determine the equation of the best-fitting straight line calculated by a linear regression technique. Record this equation for use with the test samples.

11. Procedure

NOTE 6—This procedure applies to all samples regardless of the type of infrared instrumentation used for measurement. Thus, to comply with this test method, no extraction is to be attempted in a nondispersive infrared analyzer or any other instrument capable of automatic or semiautomatic extraction.

11.1 *Extraction*:

11.1.1 Mix the sample by shaking the original sample bottle. Check the pH of the liquid by touching pH-sensitive paper to the cap. If necessary, add sufficient sulfuric acid or sodium bisulfate to attain a pH of 2 or less.

11.1.2 Add 30 mL of solvent to the sample in the original sample bottle. Recap immediately and shake the bottle vigorously for 2 min. Allow the bottle to stand until the contents settle and bubbles disappear. Remove the cap carefully to release any pressure build-up and immediately transfer the contents of the bottle to a clean separatory funnel. Wash down the transfer funnel with clean solvent, stopper the separatory funnel, and recap the bottle. Allow the contents of the separatory funnel to settle. Transfer the bottom layer into a clean 100-mL volumetric flask through filter paper and about 1 g of sodium sulfate that have been prewetted with solvent to remove any organic material which could contaminate the sample.

NOTE 7—Use of the sodium sulfate is absolutely necessary to protect the activity level of the silica gel that will be used later.

11.1.3 Add another 30 mL of solvent to the original sample bottle, recap, and shake the container to obtain good contact between the liquid and all inner surfaces. Transfer this new

[10] Simard, R. G., Hasegawa, I., Bandaruk, W., and Headington, C. E., "Infrared Spectrophotometric Determination of Oil and Phenols in Water", *Analytical Chemistry*, Vol 23, 1951, pp. 1384–1387.

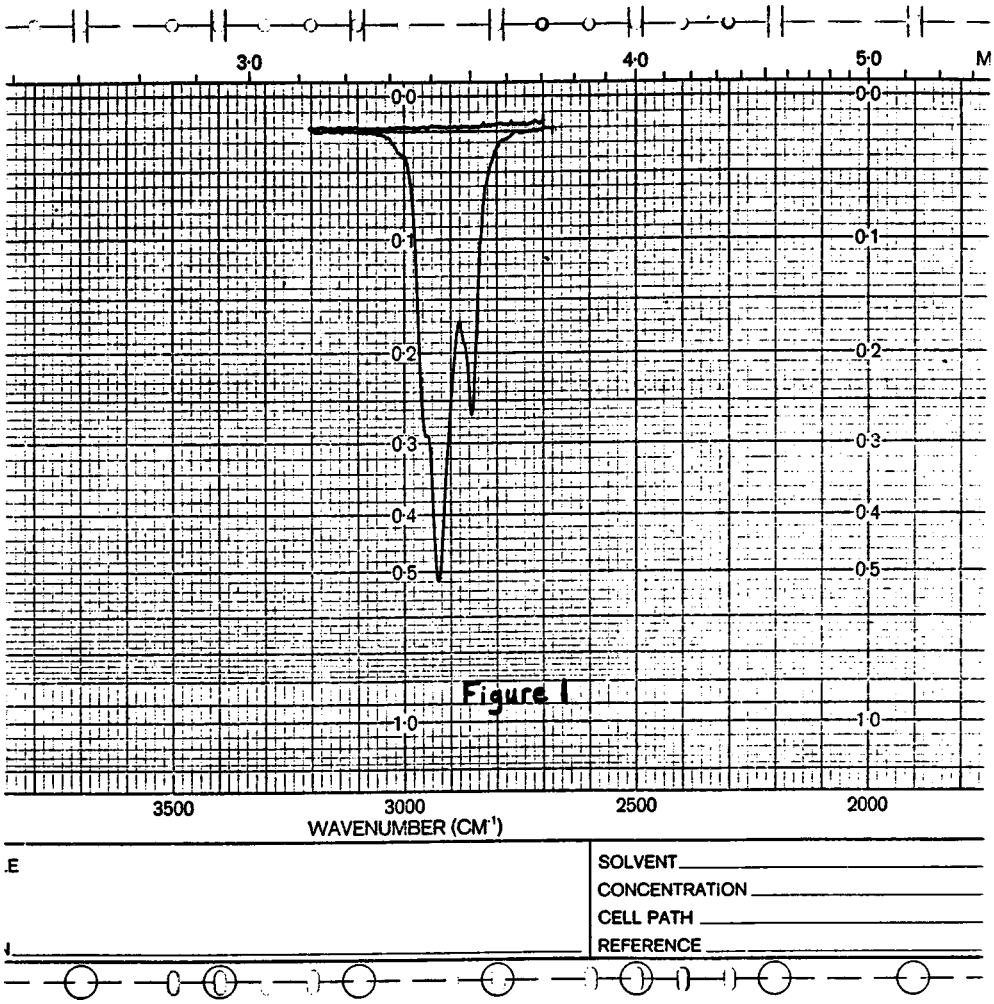

Figure 1

FIG. 1 Absorbance Values

wash to the separatory funnel, replace the stopper, and shake the mixture vigorously for 2 min. After allowing the contents to settle, partially remove the glass stopper to release any pressure. Transfer the bottom layer through the same filter assembly into the same 100-mL volumetric flask as in 11.1.2.

NOTE 8—If sodium sulfate cakes when contacted with the extract, flush with a few millilitres of solvent which is then added to the 100-mL volumetric flask, remove the solid with a clean spatula, and add about 1 g of fresh sodium sulfate to the filter. Wash the fresh salt with solvent and discard this liquid before resuming regular filtration.

11.1.4 Repeat 11.1.3 with a final 30 mL of solvent. Break any emulsions in the separatory funnel as much as possible before draining the extract. Do not allow any emulsion to enter the 100-mL volumetric flask.

NOTE 9—Certain types of samples, such as those containing a large amount of detergent, form a thick emulsion resembling mayonnaise during the extraction process. If such an emulsion cannot be broken by any attempted means, the test method is not applicable to the problem sample. Do not attempt to proceed since accurate, quantitative results for the test are not obtainable.

11.1.5 Wash down the filter assembly with fresh solvent and bring the level of the liquid to the mark in the flask. Shake the flask to completely mix the extract. Carefully release any pressure build-up.

11.1.6 Drain the remaining contents of the separatory funnel into a 1000-mL graduated cylinder and record the volume for later calculations in 12.3 and 12.4.

11.2 *First Infrared Absorbance Measurement*—Measure the infrared absorbance of the extract in a manner identical to that used for the calibration blends in Note 4, 10.8, 10.9, 10.14 and Note 5 by substituting the extract for the blend in 10.9. If the *gross* absorbance exceeds 0.8 (16 % transmittance), dilute one part of the extract to ten parts total with solvent and scan the diluted extract. Keep a record of each dilution for use in 12.3.

11.3 *Silica Gel Treatment*:

11.3.1 Remove sufficient extract from the 100-mL volumetric flask to lower the level of the liquid to about 20 mm below the base of the neck of the flask. Add about 3 g of silica gel to the liquid; finally, insert a magnetic stirring bar.

NOTE 10—Silica gel, as obtained from the supplier, may exist in any degree of activation. This test method requires the use of 2 % deactivated material. To prepare fresh silica gel for use, first weigh a quantity of the material in a clean, tared container having a screw cap lined with either aluminum or TFE-fluorocarbon. Add solvent to make a slurry and mix well. After the gel has settled, remove as much of the liquid as practical. Place the container in a clean oven at 100°C or less to remove the remainder of the solvent. Following this, raise the temperature to 150°C and heat the gel for at least 2 h. Remove the container from the oven, immediately seal with the screw cap, and store in a dry atmosphere for

several hours as it cools slowly. Later, add water equal to 2 % of the weight of the gel and, again, immediately seal with the screw cap. Shake the container to increase contact between the water and the gel. Allow the container to stand for several hours to reach equilibrium. Do not expose the gel to the atmosphere any more than necessary when removing portions for use.

11.3.2 Place the flask on a magnetic stirrer and stir the extract for 10 min at a rate sufficient to cause continuous convection of the silica gel but not so great as to cause splashing or a vortex down to the stirring bar.

11.3.3 Allow the silica gel to settle completely.

NOTE 11—It is important that no silica gel enter the sample cell. This is particularly true when a nondispersive infrared analyzer is used.

11.4 *Second Infrared Absorbance Measurement*—Measure the infrared absorbance of the treated extract in a manner identical to that used in 11.2. Keep a record of each dilution for use in 12.4.

12. Calculation

12.1 Determine the amount of oil and grease in the *untreated* extract by entering either the calibration graph or the straight-line equation of 10.15 with the absorbance found in 11.2.

12.2 Determine the amount of petroleum hydrocarbons in the *treated* extract by entering either the calibration graph or the straight-line equation of 10.15 with the absorbance found in 11.4.

12.3 To the nearest 0.1 mg/L, calculate the concentration of *oil and grease* in the original water or waste water sample as follows:

$$\text{oil and grease, mg/L} = \frac{R \times D}{V}$$

where:

R = amount of oil and grease in 100 mL of untreated extract, as found in 12.1, mg,
D = dilution factor, if any, as utilized in 11.2, where
D = volume of diluted extract/volume of undiluted extract; and
V = volume of extracted water found in 11.1.6, L.

12.4 To the nearest 0.1 mg/L, calculate the concentration of *petroleum hydrocarbons* in the original water or waste water sample as follows:

$$\text{Petroleum hydrocarbons, mg/L} = \frac{R \times D}{V}$$

where:

R = amount of petroleum hydrocarbons in 100 mL of treated extract, as found in 12.2, mg,
D = dilution factor, if any, as utilized in 11.4,
D = volume of diluted extract/volume of undiluted extract;and
V = volume of extracted water found in 11.1.6, L.

13. Precision and Bias [11]

13.1 To permit acquisition of precision data for this test method, a group of 18 samples representing 6 concentration levels of *oil and grease* in triplicate was analyzed by a single operator in each of 18 different laboratories. One sample of the highest concentration level was considered suspect due to a handling difficulty and, therefore, not included in the statistical data. A total of 244 data points was accepted for *oil and grease* and 233 for *petroleum hydrocarbons*. All statistical data regarding this test were obtained in accordance to Practice D 2777.

13.2 The precision obtained in the analytical results for this test method reflects several factors, including the accuracy with which samples can be prepared, the handling of samples, the extraction step, the operator technique, the level of material, the instrumental error, as well as unknown effects. Along with the 18 unknown samples that originated in a petroleum refinery discharge water air-flotation unit, a control sample of known *oil and grease* content at the 16.4-mg/L level was analyzed by 16 laboratories. The overall precision, S_t, found by analysis of this known control sample is 1.5 mg/L; bias is + 2.4 %. These values reflect only the errors inherent in sample preparation, sample handling, and infrared instrumental portions of the method.

13.3 The overall precision for the *oil and grease* section of the test method in the concentration range of 0.6 to 66 mg/L may be expressed by the equation:

$$S_t = 0.167\,x + 0.333$$

where:

S_t = overall precision, and
x = concentration of *oil and grease* determined, mg/L.

The single-operator precision of this section may be expressed by the equation:

$$S_o = 0.122\,x + 0.148$$

where:

S_o = single-operator precision, and
x = concentration of *oil and grease* determined, mg/L.

13.4 The overall precision for the *petroleum hydrocarbons* section of the test method in the concentration range of 0.3 to 51 mg/L may be expressed by the equation:

$$S_t = 0.160\,x + 0.329$$

where:

S_t = overall precision, and
x = concentration of petroleum hydrocarbons determined, mg/L.

The single-operator precision of this section may be expressed by the equation:

$$S_o = 0.141\,x + 0.048$$

where:

S_o = single-operator precision, and
x = concentration of petroleum hydrocarbons determined, mg/L.

13.5 The data presented in 13.3 and 13.4 may not apply to other types of water.

13.6 The unknown samples were not assayed independently since to do so would require use of the same technique as that used in this study. Thus, the bias introduced by the test method is indeterminable.

[11] Supporting data for the precision and bias statements are available from ASTM Headquarters. Request RR: D19-1066.

14. Quality Assurance/Quality Control

14.1 Minimum quality control requirements are an initial demonstration of proficiency, plus analysis of method blanks and quality control samples. Recovery spikes and duplicates may be required for specific programs. For a general discussion of quality control and good laboratory practices, see Practice D 5789, Guide D 3856 and Practice D 4210.

14.2 *Method Blank*— Before processing any samples, the analyst shall demonstrate that all glassware and reagent interferences are under control. Each time a set of samples is extracted or reagents are changed, analyze a method blank. The blank result shall be low enough that it will not unduly influence the data (that is, <5 mg/L).

14.3 *Initial Demonstration of Proficiency*:

14.3.1 Select a representative spike concentration; 20 mg/L is recommended. Use a material as described in Section 10. The spike material should be from a different source than the material used for calibration. Add spike concentrate to at least seven 1-L aliquots of water, and analyze each aliquot according to the procedures in Sections 10 and 11. Calculate the mean and standard deviation of these values and compare to the acceptable range of precision and bias found in Table 1.

14.3.2 This study should be repeated until the single-operator precision and the mean value are within acceptable limits. Refer to Practice D 5789 to develop limits for spikes at other concentrations.

14.3.3 The analyst is permitted to modify the procedure, use alternate solvents, or use alternate extraction procedures, such as solid phase extraction, or both, to improve the procedure or lower analytical costs. Tetrachlorethene (perchloroethylene) has been recommended as an alternative solvent. Any time such modifications are made, the initial demonstration of proficiency must be successfully repeated.

TABLE 1 Acceptable Range of Precision and Bias

Spike Concentration mg/L	Proficiency		QC Check
	Maximum Acceptable Standard Deviation	Acceptance Range for Mean Recovery	Acceptance Range for QC Check
20 (O and G)	4.78 mg/L	6.9–33.1 mg/L	9–31 mg/L
20 (Pet HC)	5.29 mg/L	7.5–32.5	9.4–30.6

14.4 *Ongoing Quality Control Sample*—To ensure that the test method is in control, analyze a single quality control sample (prepared as in 14.3.1) containing 20 mg/L of oil and grease daily or with each batch of up to 20 samples. The value obtained should be within the range listed in Table 1 if the test is in control.

14.5 *Duplicates and Matrix Spikes*—Due to the inherent variability of oil and grease sampling and samples, results from duplicates and matrix spikes may be inconsistent or inconclusive. However, some programs may require analysis of these QC samples. Collect additional 1-L sample bottles for each duplicate and matrix spike sample to be analyzed should they be desired. Refer to Practice D 5789 for guidelines on reporting and evaluating these results.

15. Keywords

15.1 oil and grease; petroleum hydrocarbons

APPENDIX

(Nonmandatory Information)

X1. EXAMPLE CALCULATIONS FOR QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) STATISTICS

X1.1 This example[12] shows the calculation of control limits for oil and grease. The limits for petroleum hydrocarbons were calculated in the same manner. Eighteen operators analyzed five concentration levels in triplicate. The degrees of freedom (dof) for the study was 36:

$$(\text{operators} \times \text{replicates}) - (\text{operators}) = (18 \times 3) - 18 = 36$$

At 20 mg/L, the single operator precision S_O is 2.59 mg/L, and the overall precision S_T is 3.67 mg/L (using the formulas for precision and bias in Section 13).

X1.2 Calculation of Precision and Bias Criteria for the Initial Demonstration of Proficiency

X1.2.1 *Precision*—The value of F for 6×36 dof = 3.40. The maximum acceptable standard deviation is:

$$2.59 \text{ mg/L} \times \sqrt{3.40} = 4.78$$

X1.2.2 *Bias*—The Student's t for 6 dof is 3.71. The acceptance limits for a 20 mg/L test concentration is:

$$20 \pm [3.71 \text{ mg/L} \times \sqrt{(S_t)^2 - ((S_o)^2/7)}] = 20 \pm 13.1 \text{ mg/L}$$

or 6.9 to 33.1 mg/L.

X1.3 Calculation of Bias Criteria for Quality Control Samples

X1.3.1 The acceptance criteria for the verification of control at the representative concentration is calculated as:

$$X \pm 3S_t$$

or

$$20 \pm 3(3.67) \text{ mg/L} = 20 \pm 11 \text{ mg/L}$$

This yields an acceptable range of 9 to 31 mg/L.

[12] Reference statistics are from the Interlaboratory Method Study and calculations are based on Practice D 5789.

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