



# Standard Test Method for Oil and Grease (Fluorocarbon Extractable Substances) by Gravimetric Determination<sup>1</sup>

This standard is issued under the fixed designation D 4281; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## INTRODUCTION

In the determination of oil and grease, an absolute quantity of a specific substance is not measured. Rather, groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in a specified fluorocarbon solvent. Oil and grease may therefore, be said to include hydrocarbons, fatty acids, soaps, fats, waxes, oil, and any other material that is extracted by the solvent from an acidified sample of the test. It is important that this limitation be clearly understood. Unlike some constituents—which represent distinct chemical elements, ions, compounds, or groups of compounds, oils and greases are defined by the method used for their determination.

## 1. Scope

1.1 These test methods cover the estimation of oil and grease in water and wastewater by a gravimetric determination of fluorocarbon extractable substances from an acidified sample. Also included in this estimation of oil and grease are any other compounds soluble in chlorofluorocarbon-113 and non-volatile under the conditions of test.

1.2 The following two test methods are included:

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Test Method A—Liquid-Liquid Extraction	10-15
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1.3 These test methods are applicable to natural waters and domestic wastewaters. They are also suitable for most industrial wastewaters, although certain wastes may yield low results because of the presence of either excessive concentrations of natural greases or synthetic or modified compounds that are not well recovered by these test methods.

1.4 These test methods measure those groups of substance called oil and grease, whether soluble or insoluble, polar or non-polar, or biological or mineral in origin, to the extent these substances partition from the matrix into chlorofluorocarbon-113 and are not volatilized under conditions of the test. (See the Introduction and Section 3 of these test methods.)

1.5 Because these test methods include an acidification step prior to extraction, soluble metallic soaps are hydrolyzed and recovered as fatty acids in the extraction step.

1.6 These test methods were fully validated by collaborative testing in reagent Type IV water. The information on precision may not apply to other waters.

NOTE 1—Refer to companion procedures (Test Method D 3921).

1.7 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.8 *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.* For specific hazard statements, see 12.1, 13.2, 20.1, and 20.5.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- D 1129 Terminology Relating to Water<sup>2</sup>
- D 1192 Specification for Equipment for Sampling Water and Steam in Closed Conduits<sup>2</sup>
- D 1193 Specification for Reagent Water<sup>2</sup>
- D 3370 Practices for Sampling Water from Closed Conduits<sup>2</sup>
- D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water and Waste Water<sup>2</sup>

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

D 3921 Test Method for Oil and Grease and Petroleum Hydrocarbons in Water<sup>3</sup>

D 4210 Practice for Interlaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data<sup>2</sup>

D 5789 Practice for Writing Quality Control Specifications for Standard Test Methods for Organic Constituents<sup>3</sup>

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *oil and grease*—the organic matter and other materials extracted by the solvent from water and wastewater and measured by these test methods.

### 4. Summary of Test Method

4.1 In this test method, an acidified water sample is extracted with fluorocarbon solvent in a separatory funnel.

4.2 In the gravimetric portion of the procedure, the fluorocarbon solvent containing the extracted materials is evaporated and the residue is determined gravimetrically.

### 5. Significance and Use

5.1 A knowledge of the quantity of oil and grease present in a waste is helpful in overcoming difficulties in wastewater treatment plant operation, in determining plant efficiencies, and in controlling the subsequent discharge of these materials to receiving streams.

5.2 When oils and greases are discharged in wastewater or treated effluents, they often cause surface films and shoreline deposits.

### 6. Interferences

6.1 This test method is entirely empirical, and duplicate results can be obtained only by strict adherence to all details. By definition, any material recovered is called oil and grease including such things as elemental sulfur and certain organic dyes. In addition, heavier residuals of petroleum may contain a significant portion of material insoluble in the solvent used.

6.2 The rate and time of extraction in the Soxhlet apparatus must be exactly as directed because of varying solubilities of different oils and greases. In addition, the length of time required for drying and cooling the extracted material cannot be varied. There may be a gradual increase in weight, presumably due to the absorption of oxygen, or a gradual loss of weight due to volatilization.

6.3 Modern industry uses a number of long-chain carbon compounds as lubricants and emulsifiers, as well as for other purposes. Often the composition of these materials differs from that of natural oils and greases, and may render them more soluble in water or more easily emulsified than the natural products. As a result, they behave as oils and greases in

treatment processes and the receiving water. The procedures described here may fail to provide complete recovery of such products.<sup>4, 5</sup>

6.4 Organic based boiler and cooling water chemicals such as polymeric dispersants and chelants, antifoams, filming and neutralizing amines, and oxygen scavengers may be recovered as oil and grease when these test methods are applied to such waters.

6.5 Low-boiling fractions are lost in the solvent-removal steps of the gravimetric procedures. Even lubrication oil fractions evaporate at a significant rate at the temperature necessary for removal of the last traces of the extraction solvent. Kerosine is still more volatile and gasoline cannot be determined with any reliability by a gravimetric method.

6.6 Suspended solids may interfere by contributing to emulsions.

### 7. Apparatus

7.1 *Drying Oven*, 103°C.

7.2 *Evaporating Flask*, 250-mL capacity. A flat-bottom boiling flask with standard taper fitting is recommended to facilitate solvent recycling.

7.3 *Distillation Apparatus*, water-cooled condenser, side arm, and receiver, all of appropriate standard taper fitting.

7.4 *Separatory Funnels*, 2-L funnels with TFE-fluorocarbon stopcocks.

7.5 *Steam Bath*.

7.6 *Desiccator*.

### 8. Purity of Reagents

8.1 Reagent grade chemicals shall be used in all 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.<sup>6</sup> 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 Type IV reagent water conforming to Specification D 1193.

### 9. Sampling

9.1 Collect the samples in accordance with Specification D 1192 or Practices D 3370, when applicable.

9.2 Collect grab samples only directly into the sample bottle. Oils and greases may be lost on any sampling equipment used. Do not rinse the sample bottles with the sample

<sup>4</sup> Chanin, G., E. H. Chow, R. B. Alexander, and J. F. Powers, "Scum Analysis: A New Solution to a Difficult Problem," WWSWA-5,6-49-68.

<sup>5</sup> Taras, M. J. and K. A. Blum, "Determination of Emulsifying Oil in Industrial Wastewater," JWPPA-40-R404-68.

<sup>6</sup> *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.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.02.

prior to filling as the oils may adhere to the inside of the bottle and lead to high results.

9.3 Because a portion of the collected oils and greases may adhere to the sample bottle, analyze all of the sample collected as one specimen. One-litre samples are normally specified, although larger or smaller samples may be taken depending on the concentration of extractables expected to be present.

9.4 Leave an air space above the liquid in the sample bottle to facilitate handling during analysis.

9.5 Collect all samples in glass bottles with nonorganic or insoluble caps, or both.

9.6 Acidify the sample to < pH 2 with HCl (1 + 1) or H<sub>2</sub>SO<sub>4</sub>(1 + 1) to maintain the integrity of the sample.

NOTE 2—For Clean Water Act purposes only H<sub>2</sub>SO<sub>4</sub> should be used and the sample should be cooled to 4°C and analyzed within 28 days.<sup>7</sup>

## TEST METHOD A—LIQUID-LIQUID EXTRACTION

### 10. Scope

10.1 This test method covers the estimation of oil and grease from 4 to 100 mg/L in water and wastewater by a gravimetric determination of fluorocarbon extractable substances from an acidified sample.

10.2 This test method is generally applicable to samples containing extractable substances.

### 11. Reagents and Materials

11.1 *Acetone* ((CH<sub>3</sub>)<sub>2</sub>CO), technical grade.

11.2 *Filter Paper*, any high flow, low-retention grade, or optionally phase separating paper.<sup>8</sup>

11.3 *Fluorocarbon Solvent* (Chlorofluorocarbon-113 or 1,1,2-Trichloro-1,2,2-Trifluoroethane)<sup>9</sup> must be shown to contain no significant residue on evaporation. Redistill if necessary.

11.4 *Hydrochloric Acid* (HCl), sp gr 1.19.

11.5 *Sodium Bisulfate*—(NaHSO<sub>4</sub>·H<sub>2</sub>O).

11.6 *Sodium Chloride* (NaCl).

11.7 *Sodium Sulfate* (Na<sub>2</sub>SO<sub>4</sub>), anhydrous.

11.8 *Sulfuric Acid* (H<sub>2</sub>SO<sub>4</sub>), sp gr 1.84.

### 12. Procedure

12.1 Tare, to the nearest tenth of a milligram, a boiling flask that has been dried at 103°C in an oven for 1 h and cooled in a desiccator to room temperature. **Caution**—Always handle the flask with metal tongs or weighing gloves to avoid deposition of body oils.

NOTE 3—Run a reagent and materials blank to show that they contain no significant residue with respect to the precision of the test method at the level measured.

<sup>7</sup> See Title 40, Code of Federal Regulations, Part 136 (40 CFR 136), Table 2, Required Containers, Preservation Techniques, and Holding Times, by the U.S. Environmental Protection Agency, available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20401.

<sup>8</sup> Whatman IPS Phase Separating Paper or an equivalent has been shown to be satisfactory; this material should be rinsed with solvent before use to eliminate silicone residue contamination.

<sup>9</sup> Freon 113 (E. I. DuPont de Nemours, Inc.) or equivalent has been shown to be satisfactory; this solvent is available as Freon TF, Freon PCA, Genetron 113, and Genesolve D.

NOTE 4—Frequently, solvent will extract plasticizer from plastic tubing that is used to transfer from one container to another and from shipping container liner. Check for contamination by evaporating 180 mL of solvent on a steam bath and weighing the residue. The solvent should leave no measurable residue greater than 0.1 mg. If this value is exceeded, distill the solvent and check the distillate for residue.

12.2 Mark the sample bottle at the water meniscus for later determination of sample volume. Pour the acidified sample into a separatory funnel.

12.3 Add 60 mL of fluorocarbon solvent to the sample bottle, cap, and shake the bottle well. Pour the solvent into the separatory funnel and extract the sample by shaking vigorously for 2 min. Invert the separatory funnel and vent with stopcock to relieve pressure buildup during the extraction. After the layers have separated, drain the solvent layer through filter paper held by a small funnel into the tared boiling flask. If emulsion problems are anticipated, add 1 g Na<sub>2</sub>SO<sub>4</sub> to the filter paper cone and slowly drain the solvent through the crystals. Add more Na<sub>2</sub>SO<sub>4</sub> if necessary.

NOTE 5—Solvent phase separation paper<sup>8</sup> helps to keep water out.

12.4 If a clear solvent layer cannot be obtained due to emulsion with water, add up to 100 g of NaCl to separatory funnel. Shake to dissolve the salt. Frequently this will break the emulsion. If the emulsion cannot be broken, this type of sample must be analyzed by the Soxhlet extraction test method.

12.5 Repeat the bottle rinse and extraction with two additional 60-mL portions of solvent, combining all solvent in the flask. Rinse the filter with 20 mL of solvent, into the flask.

12.6 Proceed to 13.1.

### 13. Procedure, Gravimetric for Test Methods A and B

13.1 Evaporate the solvent from the boiling flask (12.5, 20.8) on a hot water bath or steam bath. (Recovering solvent using a condenser system is recommended.)

13.2 When only a few millilitres of solvent remain (under 10 mL of solvent is not recommended), leave the flask on the steam bath and draw air through the flask using vacuum for 5 min to remove the last traces of solvent or residual water. Carefully wipe the exterior of the flask with a lint free cloth and a small amount of acetone to remove any water adhering to the flask. **Warning**—Wear protective gloves (polyethylene or similar solvent-resistant material) to prevent the acetone from coming in contact with the skin. Use acetone in a fume hood.

13.3 Place in a desiccator for 1 h, remove, and weigh immediately, to the nearest tenth of a milligram.

13.4 Measure the original sample volume by filling the sample bottle to the mark with water at 20°C and measure the volume of water with a graduated cylinder, to the nearest 5 mL.

NOTE 6—It is recommended that the chlorofluorocarbon-113 solvent be recycled to reduce costs and minimize discharges to the atmosphere. To recycle the solvent, attach a water cooled condenser with a side arm take off. The recovered solvent can usually be reused for this test without redistillation.

### 14. Calculation

14.1 Calculate the results of the determination, in milligrams per litre as follows:

$$\text{Extractable Residue, mg/L} = \frac{(B - A)}{C} \times 1000$$

**TABLE 1 Recovery and Precision Data (Test Method A)**

Amount Added, mg/L	Amount Found, mg/L	Bias, ± %	Statistically Significant (95 % Confidence Level)	Single Operator Precision $S_o$ , mg/L	Overall Precision $S_T$ , mg/L
4.4	5.3	+ 19.4	yes	1.4	1.7
13.1	13.9	+ 6.0	yes	0.9	1.8
21.9	22.3	+ 1.8	no	1.9	2.3
65.7	62.4	-5.0	yes	2.2	4.4

where:

$A$  = tare weight of boiling flask, mg,

$B$  = weight of boiling flask after removal of extraction solvent, mg, and

$C$  = volume of sample, L.

Report results to the nearest milligram per litre.

### 15. Precision and Bias <sup>10</sup>

15.1 Nine operators from nine laboratories determined four concentration levels of oil and grease in reagent water, Type IV, over three days.

15.2 Recoveries of known amounts of oil and grease in a series of prepared standards were as shown in Table 1.

15.3 It should be recognized that these data may not apply to different water matrices.

## TEXT METHOD B—SOXHLET EXTRACTION

### 16. Scope

16.1 This test method covers the estimation of oil and grease from 20 to 200 mg/L in water and wastewater by a gravimetric determination of fluorocarbon extractable substances from an acidified sample.

16.2 This test method should be used with waters where emulsion problems do not permit separatory funnel extraction techniques. This test method should not be used where a significant second phase of oily material is present in the sample, due to the difficulty in recovering all the oily phase for gravimetric analysis.

### 17. Summary of Test Methods

17.1 In this test method, an acidified water sample is filtered through diatomaceous earth on filter paper. The cake is dried and extracted in a Soxhlet apparatus for 4 h with fluorocarbon solvent.

17.2 In the gravimetric portion of the procedure, the fluorocarbon solvent containing the extracted materials is evaporated and the residue is determined gravimetrically.

### 18. Apparatus

18.1 *Buchner Funnel*, porcelain, 120-mm.

18.2 *Drying Oven*, 103°C.

18.3 *Extraction Apparatus*, consisting of Soxhlet extractor, Allihn condenser, and a boiling flask.

18.3.1 *Allihn Condenser*, bottom standard taper joint 45/50. <sup>11</sup>

18.3.2 *Boiling Flask*, flat bottom, standard taper joint 24/40. <sup>12</sup>

18.3.3 *Soxhlet Extractor*, 85 mL, top standard taper joint 45/50, bottom standard taper joint 24/40. <sup>13</sup>

18.4 *Heating Source*, capable of heating the Soxhlet apparatus to achieve 20 solvent cycles per hour. Most commercially available hot plates or heating mantles are adequate.

18.5 *Hot Water Bath or Steam Bath*.

18.6 *Vacuum Pump*, or other source of vacuum.

18.7 *Desiccator*.

### 19. Reagents and Materials

19.1 *Acetone* ((CH<sub>3</sub>)<sub>2</sub>CO), technical grade.

19.2 *Diatomaceous Silica Filter Aid Suspension* <sup>14</sup>—Add a 10-g filter aid to water and dilute to 1 L.

19.3 *Extraction Thimble*, 33 by 94 mm cellulose. <sup>15</sup>

19.4 *Filter Paper*, 11-cm circles. <sup>16</sup>

19.5 *Fluorocarbon Solvent*—(1,1,2-Trichloro-1,2,2-trifluoroethane) <sup>17</sup> must be shown to contain no significant residue on evaporation. Redistill if necessary.

19.6 *Glass Beads*, 2 to 3 mm or TFE-fluorocarbon boiling stones.

19.7 *Hydrochloric Acid* (HCl), sp gr 1.19. Dilute to 1 + 1 with Type IV reagent water.

19.8 *Muslin Cloth Disks*, 7.5 cm, unbleached and rinsed with solvent.

19.9 *Sulfuric Acid* (H<sub>2</sub>SO<sub>4</sub>), sp gr 1.84. Dilute to 1 + 1 with Type IV reagent water.

19.10 *Desiccant*, active, indicating, silica gel or equivalent.

### 20. Procedure

20.1 Tare a boiling flask that has been dried at 103°C in an oven for 1 h and cooled to room temperature in a desiccator. **Caution**—Always handle the flask with metal tongs or weighing gloves to avoid deposition of body oils.

NOTE 7—A reagent and materials blank should be run to show that they contain no significant residue, with respect to the precision of the method at the level measured. If the blank is significant, review procedure details and eliminate source of contamination.

20.2 Mark the sample bottle at the water meniscus for later determination of sample volume.

20.3 Prepare the filtration apparatus, consisting of muslin cloth disk, overlaid with filter paper, in a Buchner funnel. Wet the paper and muslin with water and apply a vacuum to firmly seat the filter paper. Make sure the filter paper is smooth over the surface of the funnel. Pass 100 mL of filter aid suspension through the prepared filter and wash with 1 L of water. By

<sup>11</sup> Corning No. 3840 or equivalent has been shown to be satisfactory.

<sup>12</sup> Corning No. 4100 or equivalent has been shown to be satisfactory.

<sup>13</sup> Corning No. 3740 or equivalent has been shown to be satisfactory.

<sup>14</sup> Hyflo-Super-Cel (Johns-Manville Corp.) or equivalent has been shown to be satisfactory.

<sup>15</sup> Whatman No. 1317275 or equivalent has been shown to be satisfactory.

<sup>16</sup> Whatman No. 40 or equivalent has been shown to be satisfactory.

<sup>17</sup> Freon 113 (E. I. DuPont de Nemours, Inc.) or equivalent has been shown to be satisfactory.

<sup>10</sup> Supporting data for precision and bias statements are available from ASTM Headquarters. Request RR: D-19-1089.

careful addition of the wash water, the filter aid can be washed away from the edges of the filter paper. This facilitates removal of the filter paper later without spilling the filter aid.

20.4 Filter the entire acidified sample (see 9.6) through the prepared filter.

NOTE 8—If the entire sample collected cannot be filtered through one prepared filter, the sample can be filtered and extracted in separate portions, and the individual weights combined for the final result. Release the vacuum when the entire sample has passed through the filter. Using forceps, remove the filter paper, taking care not to lose any of the filter aid. Fold the paper several times to completely enclose the filter aid. Place the folded filter in the extraction thimble.

20.5 Wipe the inside of the sample container and Buchner funnel with pieces of filter paper soaked in fluorocarbon solvent, taking care not to remove all films caused by oil and grease and to collect all solid material. Add the pieces of paper to the extraction thimble. **Warning**—Wear rubber gloves to prevent fluorocarbon solvent from coming in contact with skin. Alternatively, a portion of the solvent that will be used in the extraction apparatus (see 20.7) can be used to rinse the sampling bottle and cap and these washings added directly to the Soxhlet boiling flask.

20.6 Dry the thimble in an oven at 103°C for 30 min.

20.7 Fill the thimble with glass beads, and insert into the assembled Soxhlet apparatus. Add fluorocarbon solvent to the tared boiling flask containing a few glass beads or teflon boiling stones and attach to Soxhlet apparatus. Apply heat and extract at 20 cycles per hour for four hours timed from the beginning of the first cycle.

NOTE 9—Initially the Soxhlet apparatus should be rinsed with fluorocarbon solvent to insure its cleanliness. In addition, some sort of loose cap can be kept over the top of the condenser to keep out any dust. Great care must be taken to insure that no glass beads spill into the extractor chamber as they can become lodged in the syphon tube.

20.8 After refluxing for 4 h, cool and remove the extraction thimble. Pour any solvent remaining in the extraction chamber into the boiling flask. The glass beads and extraction thimble can be reused as long as there are no holes in the thimble and the exterior of the thimble is free of dust.

## 21. Procedure, Gravimetric

21.1 See Section 13.

## 22. Calculation

22.1 See Section 14.

## 23. Precision and Bias<sup>18</sup>

23.1 Six operators from four laboratories determined three concentration levels of oil and grease in reagent water, Type IV, over three days. These data were acceptable under D2777 – 72, which was in place at the time this test method was validated.

23.2 Recoveries of known amounts of oil and grease in a series of prepared standards were as shown in Table 2.

23.3 It should be recognized that these data may not apply to different water matrices.

**TABLE 2 Recovery and Precision Data (Test Method B)**

Amount Added, mg	Amount Found, mg	± % Bias	Statistically Significant (95 % Confidence Level)	Single Operator Precision S <sub>O</sub> , mg	Overall Precision S <sub>T</sub> , mg
20.7	18.8	−9.0	yes	1.3	1.6
73.6	64.4	−12.6	yes	4.8	5.8
151.0	135.0	−10.6	yes	3.3	5.9

## 24. Quality Assurance/Quality Control

24.1 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.

24.2 *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 date (that is, <5 mg/L).

### 24.3 Initial Demonstration of Proficiency:

24.3.1 Select a representative spike concentration; a level used in the interlaboratory study is recommended (Method A 21.9 mg/L; Method B 20.7 mg/L). Use a spike material that is a sample of the same oil and grease that is expected to be present in actual samples. Add spike concentrate to at least seven 1-L aliquots of water, and analyze each aliquot according to the procedures in Section 12 (Method A) or Section 20 (Method B). Calculate the mean and standard deviation of these values and compare to the acceptable range of precision and bias found in Table 3.

24.3.2 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.

24.3.3 The analyst is permitted to modify the procedure, use alternate solvents, or alternate extraction procedures, such as solid phase extraction, or both, to improve the procedure or lower analytical costs. Hexane has been recommended as an alternative solvent; analysts should note that hexane is lighter than water, and the water phase must be drained to collect the hexane layer. Any time such modifications are made, the initial demonstration of proficiency shall be successfully repeated.

24.4 *Ongoing Quality Control Sample*—To ensure that the test method is in control, analyze a single quality control sample (prepared as in 24.3.1) containing 21.9 mg/L (or selected level) 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 3 if the test is in control.

24.5 *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

<sup>18</sup> Supporting data for precision and bias statements are available from ASTM Headquarters. Request RR: D-19-1090.

**TABLE 3 Criteria for Quality Control Requirements**

Spike Concentration mg/L	Proficiency Demonstration		QC Check
	Maximum Acceptable Standard Deviation	Acceptance Range for Mean Recovery	Acceptance Range for QC Check
21.9 (Method A)	3.8 mg/L	13.8–30.0 mg/L	15.0–28.8 mg/L
20.7 (Method B)	2.8 mg/L	15.0–26.4 mg/L	15.9–25.5 mg/L

be desired. Refer to Practice D 5789 for guidelines on reporting and evaluating these results.

## 25. Keywords

25.1 gravimetric determination; oil and grease

## APPENDIX

### (Nonmandatory Information)

#### X1. EXAMPLE CALCULATIONS FOR QUALITY ASSURANCE/QUALITY CONTROL STATISTICS

Reference statistics are from the interlaboratory method study, and calculations are based on Practice D 5789.

X1.1 This example shows the calculation of control limits for Test Method A. The limits for Method B were calculated in the same manner. Nine operators analyzed four concentration levels in triplicate. The degrees of freedom (dof) for the test level of 21.9 mg/L was 18:

$$((\text{operators} \times \text{replicates}) - (\text{operators})) = (9 \times 3) - 9 = 18$$

At this level, the single operator precision  $S_O$  was 1.9 mg/L, and the overall precision  $S_T$  was 2.3 mg/L.

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

X1.2.1 *Precision*—The value of  $F$  for  $6 \times 18$  dof = 4.01. The maximum acceptable standard deviation is:

$$1.9 \text{ mg/L} \times \sqrt{4.01} = 3.8$$

X1.2.2 *Bias*—The student's  $t$  for 6 dof is 3.71. The acceptance limits for a 21.9 mg/L test concentration is:

$$21.9 \pm [3.71 \text{ mg/L} \times \sqrt{(S_T)^2 - ((S_O)^2/7)}] = 21.9 \pm 8.1 \text{ mg/L}$$

or  
13.8 to 30 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

$$21.9 \pm 3 \text{ mg/L} = 21.9 \pm 6.9 \text{ mg/L}$$

This yields an acceptable range of 15.0 to 28.8 mg/L.

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