

Standard Methods for the Examination of Water and Wastewater

5520 OIL AND GREASE*#(1)

5520 A. 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 an organic extracting solvent. "Oil and grease" is defined as any material recovered as a substance soluble in the solvent. It includes other material extracted by the solvent from an acidified sample (such as sulfur compounds, certain organic dyes, and chlorophyll) and not volatilized during the test. The 12th edition of *Standard Methods* prescribed the use of petroleum ether as the solvent for natural and treated waters and *n*-hexane for polluted waters. The 13th edition added trichlorotrifluoroethane as an optional solvent for all sample types. In the 14th through the 17th editions, only trichlorotrifluoroethane was specified. However, because of environmental problems associated with chlorofluorocarbons, an alternative solvent (80% *n*-hexane and 20% methyl-*tert*-butyl ether) was included for gravimetric methods in the 19th edition. In the 20th edition, trichlorotrifluoroethane has been dropped from all gravimetric procedures (retained for 5520C, an infrared method), and replaced by *n*-hexane. Solvent-recovery techniques are included and solvent recycling is strongly recommended.

It is important to understand that, unlike some constituents that represent distinct chemical elements, ions, compounds, or groups of compounds, oils and greases are defined by the method used for their determination. In a detailed study involving many complex organic matrices, it was shown that either *n*-hexane or 80/ 20 *n*-hexane/methyl-*tert*-butyl ether gave results that were not statistically different from results obtained with trichlorotrifluoroethane.¹ Although 5520B allows either solvent system for extraction of wastewaters, note that for certain regulatory purposes U.S. EPA currently recommends only *n*-hexane.²

The methods presented here are suitable for biological lipids and mineral hydrocarbons. They also may be suitable for most industrial wastewaters or treated effluents containing these materials, although sample complexity may result in either low or high results because of lack of analytical specificity. The method is not applicable to measurement of low-boiling fractions that volatilize at temperatures below 85°C.

1. Significance

Certain constituents measured by the oil and grease analysis may influence wastewater treatment systems. If present in excessive amounts, they may interfere with aerobic and anaerobic biological processes and lead to decreased wastewater treatment efficiency. When discharged in wastewater or treated effluents, they may cause surface films and shoreline deposits leading to environmental degradation.

A knowledge of the quantity of oil and grease present is helpful in proper design and operation of wastewater treatment systems and also may call attention to certain treatment

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difficulties.

In the absence of specially modified industrial products, oil and grease is composed primarily of fatty matter from animal and vegetable sources and from hydrocarbons of petroleum origin. The portion of oil and grease from each of these two major sources can be determined with Method 5520F. A knowledge of the relative composition of a sample minimizes the difficulty in determining the major source of the material and simplifies the correction of oil and grease problems in wastewater treatment plant operation and stream pollution abatement.

2. Selection of Method

For liquid samples, three methods are presented: the partition-gravimetric method (B), the partition-infrared method (C), and the Soxhlet method (D). Method C is designed for samples that might contain volatile hydrocarbons that otherwise would be lost in the solvent-removal operations of the gravimetric procedure. Method D is the method of choice when relatively polar, heavy petroleum fractions are present, or when the levels of nonvolatile greases may challenge the solubility limit of the solvent. For low levels of oil and grease (<10 mg/L), Method C is the method of choice because gravimetric methods do not provide the needed precision.

Method E is a modification of the Soxhlet method and is suitable for sludges and similar materials. Method F can be used in conjunction with Methods B, C, D, or E to obtain a hydrocarbon measurement in addition to, or instead of, the oil and grease measurement. This method makes use of silica gel to separate hydrocarbons from the total oil and grease on the basis of polarity.

3. Sample Collection, Preservation, and Storage

Collect a representative grab sample in a wide-mouth glass bottle that has been washed with soap, rinsed with water, and finally rinsed with solvent to remove any residues that might interfere with the analysis. As an alternative to solvent rinsing, cap bottle with aluminum foil and bake at 200 to 250°C for at least 1 h. Use PTFE-lined caps for sample bottles; clean liners as above, but limit temperature to 110 to 200°C. Collect a separate sample for an oil and grease determination. Do not overfill the sample container and do not subdivide the sample in the laboratory. Collect replicate samples for replicate analyses or known-addition QA checks. Collect replicates either in rapid succession, in parallel, or in one large container with mechanical stirring (in the latter case, siphon individual portions). Typically, collect wastewater samples of approximately 1 L. If sample concentration is expected to be greater than 1000 mg extractable material/L, collect proportionately smaller volumes. If analysis is to be delayed for more than 2 h, acidify to pH 2 or lower with either 1:1 HCl or 1:1 H₂SO₄ and refrigerate. When information is required about average grease concentration over an extended period, examine individual portions collected at prescribed time intervals to eliminate losses of grease on sampling equipment during collection of a composite sample.

In sampling sludges, take every possible precaution to obtain a representative sample. When analysis cannot be made within 2 h, preserve samples with 1 mL conc HCl/80 g

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sample and refrigerate. Never preserve samples with CHCl_3 or sodium benzoate.

4. Interferences

a. Organic solvents have the ability to dissolve not only oil and grease but also other organic substances. Any filterable solvent-soluble substances (e.g., elemental sulfur, complex aromatic compounds, hydrocarbon derivatives of chlorine, sulfur, and nitrogen, and certain organic dyes) that are extracted and recovered are defined as oil and grease. No known solvent will dissolve selectively only oil and grease. Heavier residuals of petroleum may contain a significant portion of materials that are not solvent-extractable. The method is entirely empirical; duplicate results with a high degree of precision can be obtained only by strict adherence to all details.

b. For Methods 5520B, D, E, and F, solvent removal results in the loss of short-chain hydrocarbons and simple aromatics by volatilization. Significant portions of petroleum distillates from gasoline through No. 2 fuel oil are lost in this process. Adhere strictly to sample drying time, to standardize gradual loss of weight due to volatilization. For Methods 5520B, D, E, and F, during the cooling of the distillation flask and extracted material, a gradual increase in weight may be observed, presumably due to the absorption of water if a desiccator is not used. For Method 5520C use of an infrared detector offers a degree of selectivity to overcome some coextracted interferences (§ 4*a*). For Methods 5520D and E, use exactly the specified rate and time of extraction in the Soxhlet apparatus because of varying solubilities of different greases. For Method 5520F, the more polar hydrocarbons, such as complex aromatic compounds and hydrocarbon derivatives of chlorine, sulfur, and nitrogen, may be adsorbed by the silica gel. Extracted compounds other than hydrocarbons and fatty matter also interfere.

c. Alternative techniques may be needed for some samples if intractable emulsions form that cannot be broken by centrifugation. Such samples may include effluents from pulp/paper processing and zeolite manufacturing. Determine such modifications on a case-by-case basis.

d. Some sample matrices can increase the amount of water partitioned into the organic extraction fluid. When the extraction solvent from this type of sample is dried with sodium sulfate, the drying capacity of the sodium sulfate can be exceeded, thus allowing sodium sulfate to dissolve and pass into the tared flask. After drying, sodium sulfate crystals will be visible in the flask. The sodium sulfate that passes into the flask becomes a positive interference in gravimetric methods. If crystals are observed in the tared flask after drying, redissolve any oil and grease with 30 mL of extraction solvent and drain the solvent through a funnel containing a solvent-rinsed filter paper into a clean, tared flask. Rinse the first flask twice more, combining all solvent in the new flask, and treat as an extracted sample.

e. Silica gel fines may give positive interferences in 5520F if they pass through the filter. Use filters with smaller pores if this occurs with a particular batch of silica gel.

5. References

1. U.S. ENVIRONMENTAL PROTECTION AGENCY. 1995. Report of the Method 1664 Validation Studies. EPA-821-R-95-036, U.S. Environmental Protection Agency, Washington, D.C.

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2. U.S. ENVIRONMENTAL PROTECTION AGENCY. 1995. Method 1664. EPA-821-B-94-004B, U.S. Environmental Protection Agency, Washington, D.C.

5520 B. Partition-Gravimetric Method

1. General Discussion

Dissolved or emulsified oil and grease is extracted from water by intimate contact with an extracting solvent. Some extractables, especially unsaturated fats and fatty acids, oxidize readily; hence, special precautions regarding temperature and solvent vapor displacement are included to minimize this effect. Organic solvents shaken with some samples may form an emulsion that is very difficult to break. This method includes a means for handling such emulsions. Recovery of solvents is discussed. Solvent recovery can reduce both vapor emissions to the atmosphere and costs.

2. Apparatus

- a. *Separatory funnel*, 2-L, with TFE*#(2) stopcock.
- b. *Distilling flask*, 125-mL.
- c. *Liquid funnel*, glass.
- d. *Filter paper*, 11-cm diam. †#(3)
- e. *Centrifuge*, capable of spinning at least four 100-mL glass centrifuge tubes at 2400 rpm or more.
- f. *Centrifuge tubes*, 100-mL, glass.
- g. *Water bath*, capable of maintaining 85°C.
- h. *Vacuum pump* or other source of vacuum.
- i. *Distilling adapter* with drip tip. Setup of distillate recovery apparatus is shown in Figure 5520:1. Alternatively, use commercially available solvent recovery equipment.
- j. *Ice bath*.
- k. *Waste receptacle*, for used solvent.
- l. *Desiccator*.

3. Reagents

- a. *Hydrochloric or sulfuric acid*, 1:1: Mix equal volumes of either acid and reagent water.
- b. *n-Hexane*, boiling point 69°C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.
- c. *Methyl-tert-butyl ether (MTBE)*, boiling point 55°C to 56°C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.

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- d. *Sodium sulfate*, Na₂SO₄, anhydrous crystal.
- e. *Solvent mixture*, 80% *n*-hexane/20% MTBE, v/v.

4. Procedure

When a sample is brought into the laboratory, either mark sample bottle at the water meniscus or weigh the bottle, for later determination of sample volume. If sample has not been acidified previously (see Section 5520A.3), acidify with either 1:1 HCl or 1:1 H₂SO₄ to pH 2 or lower (generally, 5 mL is sufficient for 1 L sample). Using liquid funnel, transfer sample to a separatory funnel. Carefully rinse sample bottle with 30 mL extracting solvent (either 100% *n*-hexane, ¶ 3b, or solvent mixture, ¶ 3e) and add solvent washings to separatory funnel. Shake vigorously for 2 min. Let layers separate. Drain aqueous layer and small amount of organic layer into original sample container. Drain solvent layer through a funnel containing a filter paper and 10 g Na₂SO₄, both of which have been solvent-rinsed, into a clean, tared distilling flask. If a clear solvent layer cannot be obtained and an emulsion of more than about 5 mL exists, drain emulsion and solvent layers into a glass centrifuge tube and centrifuge for 5 min at approximately 2400 rpm. Transfer centrifuged material to an appropriate separatory funnel and drain solvent layer through a funnel with a filter paper and 10 g Na₂SO₄, both of which have been prerinsed, into a clean, tared distilling flask.

Recombine aqueous layers and any remaining emulsion or solids in separatory funnel. For samples with <5 mL of emulsion, drain only the clear solvent through a funnel with pre-moistened filter paper and 10 g Na₂SO₄. Recombine aqueous layers and any remaining emulsion or solids in separatory funnel. Extract twice more with 30 mL solvent each time, but first rinse sample container with each solvent portion. Repeat centrifugation step if emulsion persists in subsequent extraction steps. Combine extracts in tared distilling flask, and include in flask a final rinsing of filter and Na₂SO₄ with an additional 10 to 20 mL solvent. Distill solvent from flask in a water bath at 85°C for either solvent system. To maximize solvent recovery, fit distillation flask with a distillation adapter equipped with a drip tip and collect solvent in an ice-bath-cooled receiver (Figure 5520:1). When visible solvent condensation stops, remove flask from water bath. Cover water bath and dry flasks on top of cover, with water bath still at 85°C, for 15 min. Draw air through flask with an applied vacuum for the final 1 min. Cool in desiccator for at least 30 min and weigh. To determine initial sample volume, either fill sample bottle to mark with water and then pour water into a 1-L graduated cylinder, or weigh empty container and cap and calculate the sample volume by difference from the initial weight (assuming a sample density of 1.00).

5. Calculation

If the organic solvent is free of residue, the gain in weight of the tared distilling flask is due to oil and grease. Total gain in weight, *A*, of tared flask, less calculated residue from solvent blank, *B*, is the amount of oil and grease in the sample:

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$$\text{mg oil and grease/L} = \frac{(A - B) \times 1000}{\text{mL sample}}$$

6. Precision and Bias

Method B with 80:20 hexane/MTBE mixture was tested by a single laboratory on a raw wastewater sample. The oil and grease concentration was 22.4 mg/L. When samples were dosed with 30 mg Fisher Heavy Mineral Oil, recovery of added oil was 84.2% with a standard deviation of 1.2 mg/L. Method B was tested with *n*-hexane as solvent. The method detection limit was determined to be 1.4 mg/L.¹ When reagent water was fortified with hexadecane and stearic acid each at approximately 20 mg/L, initial precision and recovery limit standards were 10% and 83 to 101%, respectively. Acceptable recovery limits for laboratory-fortified matrix/laboratory-fortified matrix duplicate and ongoing laboratory control standards are 79 to 114%, with a relative percent difference limit of 18%.

7. References

1. U.S. ENVIRONMENTAL PROTECTION AGENCY. 1995. Report of the Method 1664 Validation Studies. EPA-821-R-95-036, U.S. Environmental Protection Agency, Washington, D.C.
2. U.S. ENVIRONMENTAL PROTECTION AGENCY. 1995. Method 1664. EPA-821-B-94-004B, U.S. Environmental Protection Agency, Washington, D.C.

8. Bibliography

KIRSCHMAN, H.D. & R. POMEROY. 1949. Determination of oil in oil field waste waters. *Anal. Chem.* 21:793.

5520 C. Partition-Infrared Method

1. General Discussion

a. Principle: The use of trichlorotrifluoroethane as extraction solvent allows absorbance of the carbon-hydrogen bond in the infrared to be used to measure oil and grease. Elimination of the evaporation step permits infrared detection of many relatively volatile hydrocarbons. Thus, the lighter petroleum distillates, with the exception of gasoline, may be measured accurately. With adequate instrumentation, as little as 0.2 mg oil and grease/L can be measured.

b. Definitions: A “known oil” is defined as a sample of oil and/or grease that represents the only material of that type used or manufactured in the processes represented by a wastewater. An “unknown oil” is defined as one for which a representative sample of the oil or grease is not available for preparation of a standard.

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2. Apparatus

- a. *Separatory funnel*, 2-L, with TFE*#(4) stopcock.
- b. *Volumetric flask*, 100-mL.
- c. *Liquid funnel*, glass.
- d. *Filter paper*, 11-cm diam. †#(5)
- e. *Centrifuge*, capable of spinning at least four 100-mL glass centrifuge tubes at 2400 rpm or more.
- f. *Centrifuge tubes*, 100-mL, glass.
- g. *Infrared spectrophotometer*, double-beam, recording.
- h. *Cells*, near-infrared silica.

3. Reagents

- a. *Hydrochloric acid*, HCl, 1 + 1.
- b. *Trichlorotrifluoroethane* (1,1,2-trichloro-1,2,2-trifluoro ethane), boiling point 47°C. The solvent should leave no measurable residue on evaporation; distill if necessary. Do not use any plastic tubing to transfer solvent between containers.
- c. *Sodium sulfate*, Na₂SO₄, anhydrous, crystal.
- d. *Reference oil*: Prepare a mixture, by volume, of 37.5% isooctane, 37.5% hexadecane, and 25.0% benzene. Store in sealed container to prevent evaporation.

4. Procedure

Refer to Section 5520B.4 for sample handling and for method of dealing with sample emulsions. After carefully transferring sample to a separatory funnel, rinse sample bottle with 30 mL trichlorotrifluoroethane and add solvent washings to funnel. Shake vigorously for 2 min. Let layers separate. Drain all but a very small portion of the lower trichlorotrifluoroethane layer through a funnel containing a filter paper and 10 g Na₂SO₄, both of which have been solvent-rinsed, into a clean, 100-mL volumetric flask. If a clear solvent layer cannot be obtained and an emulsion of more than about 5 mL exists, see Section 5520B.4. Extract twice more with 30 mL solvent each time, but first rinse sample container with each solvent portion. Repeat centrifugation step if emulsion persists in subsequent extraction steps. Combine extracts in volumetric flask, and include in flask a final rinsing of filter and Na₂SO₄ with an additional 10 to 20 mL solvent. Adjust final volume to 100 mL with solvent.

Prepare a stock solution of known oil by rapidly transferring about 1 mL (0.5 to 1.0 g) of the oil or grease to a tared 100-mL volumetric flask. Stopper flask and weigh to nearest milligram. Add solvent to dissolve and dilute to mark. If the oil identity is unknown (Section 5520C.1b) use the reference oil (Section 5520C.3d) as the standard. Using volumetric techniques, prepare a series of standards over the range of interest. Select a pair of matched near-infrared silica cells. A 1-cm-path-length cell is appropriate for a working range of about 4 to 40 mg. Scan standards and samples from 3200 cm⁻¹ to 2700 cm⁻¹ with solvent in the

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reference beam and record results on absorbance paper. Measure absorbances of samples and standards by constructing a straight base line over the scan range and measuring absorbance of the peak maximum at 2930 cm^{-1} and subtracting baseline absorbance at that point. If the absorbance exceeds 0.8 for a sample, select a shorter path length or dilute as required. Use scans of standards to prepare a calibration curve.

5. Calculation

$$\text{mg oil and grease/L} = \frac{A \times 1000}{\text{mL sample}}$$

where:

A = mg of oil or grease in extract as determined from calibration curve.

6. Precision and Bias

Method C was used by a single laboratory to test a wastewater sample. By this method the oil and grease concentration was 17.5 mg/L. When 1-L sample portions were dosed with 14.0 mg of a mixture of No. 2 fuel oil and Wesson oil, the recovery of added oils was 99% with a standard deviation of 1.4 mg.

7. Bibliography

GRUENFELD, M. 1973. Extraction of dispersed oils from water for quantitative analysis by infrared spectrophotometry. *Environ. Sci. Technol.* 7:636.

5520 D. Soxhlet Extraction Method

1. General Discussion

Soluble metallic soaps are hydrolyzed by acidification. Any oils and solid or viscous grease present are separated from the liquid samples by filtration. After extraction in a Soxhlet apparatus with solvent, the residue remaining after solvent evaporation is weighed to determine the oil and grease content. Compounds volatilized at or below 103°C will be lost when the filter is dried.

2. Apparatus

- a. *Extraction apparatus*, Soxhlet, with 125-mL extraction flask.
- b. *Extraction thimble*, paper, solvent-extracted.
- c. *Electric heating mantle*.
- d. *Vacuum pump* or other source of vacuum.
- e. *Vacuum filtration apparatus*.
- f. *Buchner funnel*, 12-cm.

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- g. *Filter paper*, 11-cm diam.*#(6)
- h. *Muslin cloth disks*, 11-cm diam, solvent-extracted.
- i. *Glass beads or glass wool*, solvent-extracted.
- j. *Water bath*, capable of maintaining 85°C.
- k. *Distilling adapter* with drip tip. See Section 5520B.2i and Figure 5520:1.
- l. *Ice bath*.
- m. *Waste receptacle*, for used solvent.
- n. *Desiccator*.

3. Reagents

- a. *Hydrochloric acid*, HCl, 1 + 1.
- b. *n-Hexane*: See Section 5520B.3b.
- c. *Methyl-tert-butyl ether (MTBE)*: See Section 5520B.3c.
- d. *Diatomaceous-silica filter aid suspension*, †#(7) 10 g/L distilled water.
- e. *Solvent mixture*, 80% n-hexane/20% MTBE, v/v.

4. Procedure

When sample is brought into the laboratory, either mark sample bottle at the meniscus or weigh bottle for later determination of volume. If sample has not been acidified previously (see Section 5520A.3), acidify with 1:1 HCl or 1:1 H₂SO₄ to pH 2 or lower (generally, 5 mL is sufficient). Prepare filter consisting of a muslin cloth disk overlaid with filter paper. Wet paper and muslin and press down edges of paper. Using vacuum, pass 100 mL filter aid suspension through prepared filter and wash with 1 L distilled water. Apply vacuum until no more water passes filter. Filter acidified sample. Apply vacuum until no more water passes through filter. Using forceps, transfer entire filter to a watch glass. Add material adhering to edges of muslin cloth disk. Wipe sides and bottom of collecting vessel and Buchner funnel with pieces of filter paper soaked in extraction solvent, taking care to remove all films caused by grease and to collect all solid material. Add pieces of filter paper to material on watch glass. Roll all filter material containing sample and fit into an extraction thimble. Add any pieces of material remaining on watch glass. Wipe watch glass with a filter paper soaked in extraction solvent and place in extraction thimble. Dry filled thimble in a hot-air oven at 103°C for 30 min. Fill thimble with glass wool or small glass beads. Weigh extraction flask and add 100 mL extraction solvent (*n*-hexane, ¶ 3b, or solvent mixture, ¶ 3e). Extract oil and grease in a Soxhlet apparatus, at a rate of 20 cycles/h for 4 h. Time from first cycle. For stripping and recovery of solvent, cooling extraction flask before weighing, and determining initial sample volume, see Section 5520B.4.

5. Calculation

See Section 5520B.5.

6. Precision and Bias

In analyses of synthetic samples containing various amounts of Crisco and Shell S.A.E.

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No. 20 oil, an average recovery of 98.7% was obtained, with a standard deviation of 1.86%. Ten replicates each of two wastewater samples yielded standard deviations of 0.76 mg and 0.48 mg.

7. Bibliography

- HATFIELD, W.D. & G.E. SYMONS. 1945. The determination of grease in sewage. *Sewage Works J.* 17:16.
- GILCREAS, F.W., W.W. SANDERSON & R.P. ELMER. 1953. Two new methods for the determination of grease in sewage. *Sewage Ind. Wastes* 25:1379.
- ULLMANN, W.W. & W.W. SANDERSON. 1959. A further study of methods for the determination of grease in sewage. *Sewage Ind. Wastes* 31:8.

5520 E. Extraction Method for Sludge Samples

1. General Discussion

Drying acidified sludge by heating leads to low results. Magnesium sulfate monohydrate is capable of combining with 75% of its own weight in water in forming $MgSO_4 \cdot 7H_2O$ and is used to dry sludge. After drying, the oil and grease can be extracted with an organic solvent.

2. Apparatus

- a. *Beaker*, 150-mL, glass.
- b. *Mortar and pestle*, porcelain.
- c. *Extraction apparatus*, Soxhlet.
- d. *Extraction thimble*, paper, solvent-extracted.
- e. *Glass beads or glass wool*, solvent-extracted.
- f. *Electric heating mantle*.
- g. *Vacuum pump* or other source of vacuum.
- h. *Liquid funnel*, glass.
- i. *Grease-free cotton*: Extract nonabsorbent cotton with solvent.
- j. *Water bath*, capable of maintaining 85°C.
- k. *Distilling adapter* with drip tip. See Section 5520.2i and Figure 5520:1.
- l. *Ice bath*.
- m. *Waste receptacle*, for used solvent.
- n. *Desiccator*.

3. Reagents

- a. *Hydrochloric acid*, HCl, conc.
- b. *n-Hexane*: See Section 5520B.3b.
- c. *Methyl-tert-butyl ether (MTBE)*: See Section 5520B.3c.

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d. Magnesium sulfate monohydrate: Prepare $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ by drying a thin layer overnight in an oven at 150°C .

e. Solvent mixture, 80% *n*-hexane/20% MTBE, v/v.

4. Procedure

When sample is brought into the laboratory, if it has not been acidified previously (Section 5520A.3), add 1 mL conc HCl/80 g sample. In a 150-mL beaker weigh out a sample of wet sludge, 20 ± 0.5 g, for which the dry-solids content is known. Acidify to pH 2.0 or lower (generally, 0.3 mL conc HCl is sufficient). Add 25 g $\text{MgSO}_4 \cdot \text{H}_2\text{O}$. Stir to a smooth paste and spread on sides of beaker to facilitate subsequent sample removal. Let stand until solidified, 15 to 30 min. Remove solids and grind in a porcelain mortar. Add powder to a paper extraction thimble. Wipe beaker and mortar with small pieces of filter paper moistened with solvent and add to thimble. Fill thimble with glass wool or small glass beads. Tare extraction flask, and add 100 mL extraction solvent (¶ 3*b* or ¶ 3*e*). Extract in a Soxhlet apparatus at a rate of 20 cycles/h for 4 h. If any turbidity or suspended matter is present in the extraction flask, remove by filtering through grease-free cotton into another weighed flask. Rinse flask and cotton with solvent. For solvent stripping and recovery, and cooling the extraction flask before weighing, see Section 5520B.4.

5. Calculation

Oil and grease as % of dry solids

$$= \frac{\text{gain in weight of flask, g} \times 100}{\text{weight of wet solids, g} \times \text{dry solids fraction}}$$

6. Precision

The examination of six replicate samples of sludge yielded a standard deviation of 4.6%.

5520 F. Hydrocarbons

1. General Discussion

Silica gel has the ability to adsorb polar materials. If a solution of hydrocarbons and fatty materials in a nonpolar solvent is mixed with silica gel, the fatty acids are removed selectively from solution. The materials not eliminated by silica gel adsorption are designated hydrocarbons by this test.

2. Apparatus

- a. Magnetic stirrer.*
- b. Magnetic stirring bars,* TFE-coated.
- c. Liquid funnel,* glass.
- d. Filter paper,* 11-cm diam.*#(8)

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e. *Desiccator*.

3. Reagents

a. *n-Hexane*: See Section 5520B.3b.

b. *Trichlorotrifluoroethane*: See Section 5520C.3b.

c. *Silica gel*, 100 to 200 mesh. †#(9) Dry at 110°C for 24 h and store in a tightly sealed container.

4. Procedure

Use the oil and grease extracted by Method B, C, D, or E for this test. When only hydrocarbons are of interest, introduce this procedure in any of the previous methods before final measurement. When hydrocarbons are to be determined after total oil and grease has been measured, redissolve the extracted oil and grease in trichlorofluoroethane (Method C) or 100 mL *n*-hexane. To 100 mL solvent add 3.0 g silica gel/100 mg total oil and grease, up to a total of 30.0 g silica gel (1000 mg total oil and grease). For samples with more than 1000 mg total oil and grease use a measured volume of the 100 mL solvent dissolved sample, add appropriate amount of silica gel for amount of total oil and grease in the sample portion, and bring volume to 100 mL. Stopper container and stir on a magnetic stirrer for 5 min. For infrared measurement of hydrocarbons no further treatment is required before measurement as described in Method C. For gravimetric determinations, filter solution through filter paper pre-moistened with solvent, wash silica gel and filter paper with 10 mL solvent, and combine with filtrate. For solvent stripping and recovery, and for cooling extraction flask before weighing, see Section 5520B.4.

5. Calculation

Calculate hydrocarbon concentration, in milligrams per liter, as in oil and grease (Method B, C, D, or E).

6. Precision and Bias

The following data, obtained on synthetic samples, are indicative for natural animal, vegetable, and mineral products, but cannot be applied to the specialized industrial products previously discussed.

For hydrocarbon determinations on 10 synthetic solvent extracts containing known amounts of a wide variety of petroleum products, average recovery was 97.2%. Similar synthetic extracts of Wesson oil, olive oil, Crisco, and butter gave 0.0% recovery as hydrocarbons measured by infrared analysis.

Using reagent water fortified with approximately 20 mg/L each of hexadecane and stearic acid, initial hydrocarbon recovery limits based on hexadecane of 83 to 116% were developed, with a precision limit of 13%. Laboratory-fortified matrix/laboratory-fortified matrix duplicate gave recovery limits of 66 to 114% with a relative percent difference of 24%.

7. Bibliography

U.S. ENVIRONMENTAL PROTECTION AGENCY. 1995. Report of the Method 1664 Validation Studies. EPA-821-R-95-036, U.S. Environmental Protection Agency, Washington, D.C.

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Endnotes

1 (Popup - Footnote)

* APPROVED BY STANDARD METHODS COMMITTEE, 1997.

2 (Popup - Footnote)

* Teflon or equivalent.

3 (Popup - Footnote)

† Whatman No. 40 or equivalent.

4 (Popup - Footnote)

* Teflon or equivalent.

5 (Popup - Footnote)

† Whatman No. 40 or equivalent.

6 (Popup - Footnote)

* Whatman No. 40 or equivalent.

7 (Popup - Footnote)

† Hyflo Super-Cel, Manville Corp., or equivalent.

8 (Popup - Footnote)

* Whatman No. 40 or equivalent.

9 (Popup - Footnote)

† Davidson Grade 923 or equivalent.