

METHOD 9070

TOTAL RECOVERABLE OIL AND GREASE (GRAVIMETRIC, SEPARATORY FUNNEL EXTRACTION)

1.0 SCOPE AND APPLICATION

1.1 This method measures the fluorocarbon-113 extractable matter from surface and saline waters and industrial, domestic, and aqueous wastes. It is applicable to the determination of relatively nonvolatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related matter.

1.2 The method is not applicable to measurement of light hydrocarbons that volatilize at temperatures below 70°C. Petroleum fuels, from gasoline through No. 2 fuel oils, are completely or partially lost in the solvent removal operation.

1.3 Some crude oils and heavy fuel oils contain a significant percentage of residue-type materials that are not soluble in fluorocarbon-113. Accordingly, recoveries of these materials will be low.

1.4 The method covers the range from 5 to 1,000 mg/L of extractable material.

1.5 When determining the level of oil and grease in sludge samples, Method 9071 is to be employed.

2.0 SUMMARY OF METHOD

2.1 The 1-liter sample is acidified to a low pH (2) and serially extracted with fluorocarbon-113 in a separatory funnel. The solvent is evaporated from the extract and the residue is weighed.

3.0 INTERFERENCES

3.1 Matrix interferences will likely be coextracted from the sample. The extent of these interferences will vary from waste to waste, depending on the nature and diversity of the waste being analyzed.

4.0 APPARATUS AND MATERIALS

4.1 Separatory funnel: 2,000-mL, with Teflon stopcock.

4.2 Vacuum pump, or other source of vacuum.

4.3 Flask: Boiling, 125-mL (Corning No. 4100 or equivalent).

4.4 Distilling head: Claisen or equivalent.

4.5 Filter paper: Whatman No. 40, 11 cm.

5.0 REAGENTS

5.1 Hydrochloric acid, 1:1: Mix equal volumes of concentrated HCl and Type II water.

5.2 Fluorocarbon-113 (1,1,2-trichloro-1,2,2-trifluoroethane): Boiling point, 48°C.

5.3 Sodium sulfate: Anhydrous crystal.

5.4 ASTM Type II water (ASTM D1193): Water should be monitored for impurities.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 A representative sample should be collected in a 1-liter glass bottle. If analysis is to be delayed for more than a few hours, the sample is preserved by the addition of 5 mL HCl (5.1) at the time of collection and refrigerated at 4°C.

6.2 Collect a representative sample in a wide-mouth glass bottle that has been rinsed with the solvent to remove any detergent film and acidify in the sample bottle.

6.3 All samples must have been collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

6.4 Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentration over an extended period.

7.0 PROCEDURE

7.1 Mark the sample bottle at the water meniscus for later determination of sample volume. If the sample was not acidified at time of collection, add 5 mL HCl (5.1) to the sample bottle. After mixing the sample, check the pH by touching pH-sensitive paper to the cap to ensure that the pH is 2 or lower. Add more acid if necessary.

7.2 Pour the sample into a separatory funnel.

7.3 Tare a boiling flask (pre-dried in an oven at 103°C and stored in a desiccator). Use gloves when handling flask to avoid adding fingerprints.

7.4 Add 30 mL fluorocarbon-113 (Step 5.2) to the sample bottle and rotate the bottle to rinse the sides. Transfer the solvent into the separatory funnel. Extract by shaking vigorously for 2 min. Allow the layers to separate and filter the solvent layer through a funnel containing solvent-moistened filter paper.

NOTE: An emulsion that fails to dissipate can be broken by pouring about 1 g sodium sulfate (Step 5.3) into the filter paper cone and slowly draining the emulsion through the salt. Additional 1-g portions can be added to the cone as required.

7.5 Repeat Step 7.4 twice more, with additional portions of fresh solvent, combining all solvent in the boiling flask.

7.6 Rinse the tip of the separatory funnel, the filter paper, and then the funnel with a total of 10-20 mL solvent and collect the rinsings in the flask.

7.7 Connect the boiling flask to the distilling head and evaporate the solvent by immersing the lower half of the flask in water at 70°C. Collect the solvent for reuse. A solvent blank should accompany each set of samples.

7.8 When the temperature in the distilling head reaches 50°C or the flask appears dry, remove the distilling head. To remove solvent vapor, sweep out the flask for 15 sec with air by inserting a glass tube that is connected to a vacuum source. Immediately remove the flask from heat source and wipe the outside to remove excess moisture and fingerprints.

7.9 Cool the boiling flask in a desiccator for 30 min and weigh.

7.10 Calculation:

$$\text{mg/L total oil and grease} = \frac{R - B}{V}$$

where:

R = residue, gross weight of extraction flask minus the tare weight;

B = blank determination, residue of equivalent volume of extraction solvent, mg; and

V = volume of sample in liters, determined by refilling sample bottle to calibration line and correcting for acid addition, if necessary.

8.0 QUALITY CONTROL

8.1 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free water method blank that all glassware is free of organic contamination; if there is a change in reagents, a method blank should be processed as a safeguard against reagent contamination. The blank sample should be carried through all stages of the sample preparation and measurement.

8.2 Standard quality assurance practices should be used with this method. Laboratory replicates should be analyzed to validate the precision of

the analysis. Fortified samples should be carried through all stages of sample preparation and measurement; they should be analyzed to validate the sensitivity and accuracy of the analysis.

8.3 Comprehensive quality control procedures are specified for each target compound in the referring analytical method.

8.4 All quality control data should be maintained and available for easy reference or inspection.

8.5 Employ a minimum of one blank per sample batch to determine if contamination has occurred.

8.6 Verify calibration with an independently prepared check standard every 15 samples.

8.7 Run one spike duplicate sample for every 10 samples if possible. A duplicate sample is a sample brought through the whole sample preparation and analytical process.

8.8 The use of corn oil is recommended as a reference sample solution.

9.0 METHOD PERFORMANCE

9.1 The two oil and grease methods (Methods 9070 and 9071) in this manual were tested on sewage by a single laboratory. This method determined the oil and grease level in the sewage to be 12.6 mg/L. When 1-liter portions of the sewage were dosed with 14.0 mg of a mixture of No. 2 fuel oil and Wesson oil, the recovery was 93%, with a standard deviation of ± 0.9 mg/L.

10.0 REFERENCES

1. Blum, K.A., and M.J. Taras, "Determination of Emulsifying Oil in Industrial Wastewater," JWPCF Research Suppl., 40, R404 (1968).
2. Standard Methods for the Examination of Water and Wastewater, 14th ed., p. 515.

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