

METHOD 9071A

OIL AND GREASE EXTRACTION METHOD FOR SLUDGE AND SEDIMENT SAMPLES

1.0 SCOPE AND APPLICATION

1.1 Method 9071 is used to quantify low concentrations of oil and grease (10 mg/L) by chemically drying a wet sludge sample and then extracting via the Soxhlet apparatus. It is also used to recover oil and grease levels in sediment and soil samples.

1.2 Method 9071 is used when relatively polar, heavy petroleum fractions are present, or when the levels of nonvolatile greases challenge the solubility limit of the solvent.

1.3 Specifically, Method 9071 is suitable for biological lipids, mineral hydrocarbons, and some industrial wastewaters.

1.4 Method 9071 is not recommended for measurement of low-boiling fractions that volatilize at temperatures below 70°C.

2.0 SUMMARY OF METHOD

2.1 A 20-g sample of wet sludge with a known dry-solids content is acidified to pH 2.0 with 0.3 mL concentrated HCl.

2.2 Magnesium sulfate monohydrate will combine with 75% of its own weight in water in forming $MgSO_4 \cdot 7H_2O$ and is used to dry the acidified sludge sample.

2.3 Anhydrous sodium sulfate is used to dry samples of soil and sediment.

2.4 After drying, the oil and grease are extracted with trichlorotrifluoroethane (Fluorocarbon-113)¹ using the Soxhlet apparatus.

3.0 INTERFERENCES

3.1 The method is entirely empirical, and duplicate results can be obtained only by strict adherence to all details of the processes.

3.2 The rate and time of extraction in the Soxhlet apparatus must be exactly as directed because of varying solubilities of the different greases.

3.3 The length of time required for drying and cooling extracted material must be constant.

3.4 A gradual increase in weight may result due to the absorption of oxygen; a gradual loss of weight may result due to volatilization.

¹Replacement solvent will be specified in a forthcoming regulation.

4.0 APPARATUS AND MATERIALS

- 4.1 Soxhlet extraction apparatus.
- 4.2 Analytical balance.
- 4.3 Vacuum pump or some other vacuum source.
- 4.4 Extraction thimble: Filter paper.
- 4.5 Glass wool or small glass beads to fill thimble.
- 4.6 Grease-free cotton: Extract nonabsorbent cotton with solvent.
- 4.7 Beaker: 150-mL.
- 4.8 pH Indicator to determine acidity.
- 4.9 Porcelain mortar.
- 4.10 Extraction flask: 150-mL.
- 4.11 Distilling apparatus: Waterbath at 70°C.
- 4.12 Desiccator.

5.0 REAGENTS

5.1 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. 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.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Concentrated hydrochloric acid (HCl).

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

5.5 Sodium sulfate, granular, anhydrous (Na_2SO_4): Purify by heating at 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

5.6 Trichlorotrifluoroethane (1,1,2-trichloro-1,2,2-trifluoroethane): Boiling point, 47°C. The solvent should leave no measurable residue on evaporation; distill if necessary.²

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Transfers of the solvent trichlorotrifluoroethane should not involve any plastic tubing in the assembly.

6.2 Sample transfer implements: Implements are required to transfer portions of solid, semisolid, and liquid wastes from sample containers to laboratory glassware. Liquids may be transferred using a glass hypodermic syringe. Solids may be transferred using a spatula, spoon, or coring device.

6.3 Any turbidity or suspended solids in the extraction flask should be removed by filtering through grease-free cotton or glass wool.

7.0 PROCEDURE

7.1 Determination of Sample Dry Weight Fraction

Weigh 5-10 g of the sample into a tared crucible. Determine the dry weight fraction of the sample by drying overnight at 105°C.

NOTE: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

Allow to cool in a desiccator before weighing:

$$\text{dry weight fraction} = \frac{\text{g of dry sample}}{\text{g of sample}}$$

7.2 Sample Handling

7.2.1 Sludge Samples

7.2.1.1 Weigh out 20 ± 0.5 g of wet sludge with a known dry-weight fraction (Section 7.1). Place in a 150-mL beaker.

7.2.1.2 Acidify to a pH of 2 with approximately 0.3 mL concentrated HCl.

7.2.1.3 Add 25 g prepared $\text{Mg}_2\text{SO}_4 \cdot \text{H}_2\text{O}$ and stir to a smooth paste.

7.2.1.4 Spread paste on sides of beaker to facilitate evaporation. Let stand about 15-30 min or until substance is solidified.

²Replacement solvent will be specified in a forthcoming regulation.

7.2.1.5 Remove solids and grind to fine powder in a mortar.

7.2.1.6 Add the powder to the paper extraction thimble.

7.2.1.7 Wipe beaker and mortar with pieces of filter paper moistened with solvent and add to thimble.

7.2.1.8 Fill thimble with glass wool (or glass beads).

7.2.2 Sediment/Soil Samples

7.2.2.1 Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2.2 Blend 10 g of the solid sample of known dry weight fraction with 10 g of anhydrous sodium sulfate, and place in an extraction thimble. The extraction thimble must drain freely for the duration of the extraction period.

7.3 Extraction

7.3.1 Extract in Soxhlet apparatus using trichlorotrifluorocarbon at a rate of 20 cycles/hr for 4 hr.

7.3.2 Using grease-free cotton, filter the extract into a pre-weighed 250-mL boiling flask. Use gloves to avoid adding fingerprints to the flask.

7.3.3 Rinse flask and cotton with solvent.

7.3.4 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 analytical batch of samples.

7.3.5 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 the heat source and wipe the outside to remove excess moisture and fingerprints.

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

7.3.7 Calculate oil and grease as a percentage of the total dry solids. Generally:

$$\% \text{ of oil and grease} = \frac{\text{gain in weight of flask (g)} \times 100}{\text{wt. of wet solids (g)} \times \text{dry weight fraction}}$$

8.0 QUALITY CONTROL

8.1 All quality control data should be maintained and available for easy reference and inspection. Refer to Chapter One for additional quality control guidelines.

8.2 Employ a minimum of one blank per analytical batch or twenty samples, whichever is more frequent, to determine if contamination has occurred.

8.3 Run one matrix duplicate and matrix spike sample every twenty samples or analytical batch, whichever is more frequent. Matrix duplicates and spikes are brought through the whole sample preparation and analytical process.

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

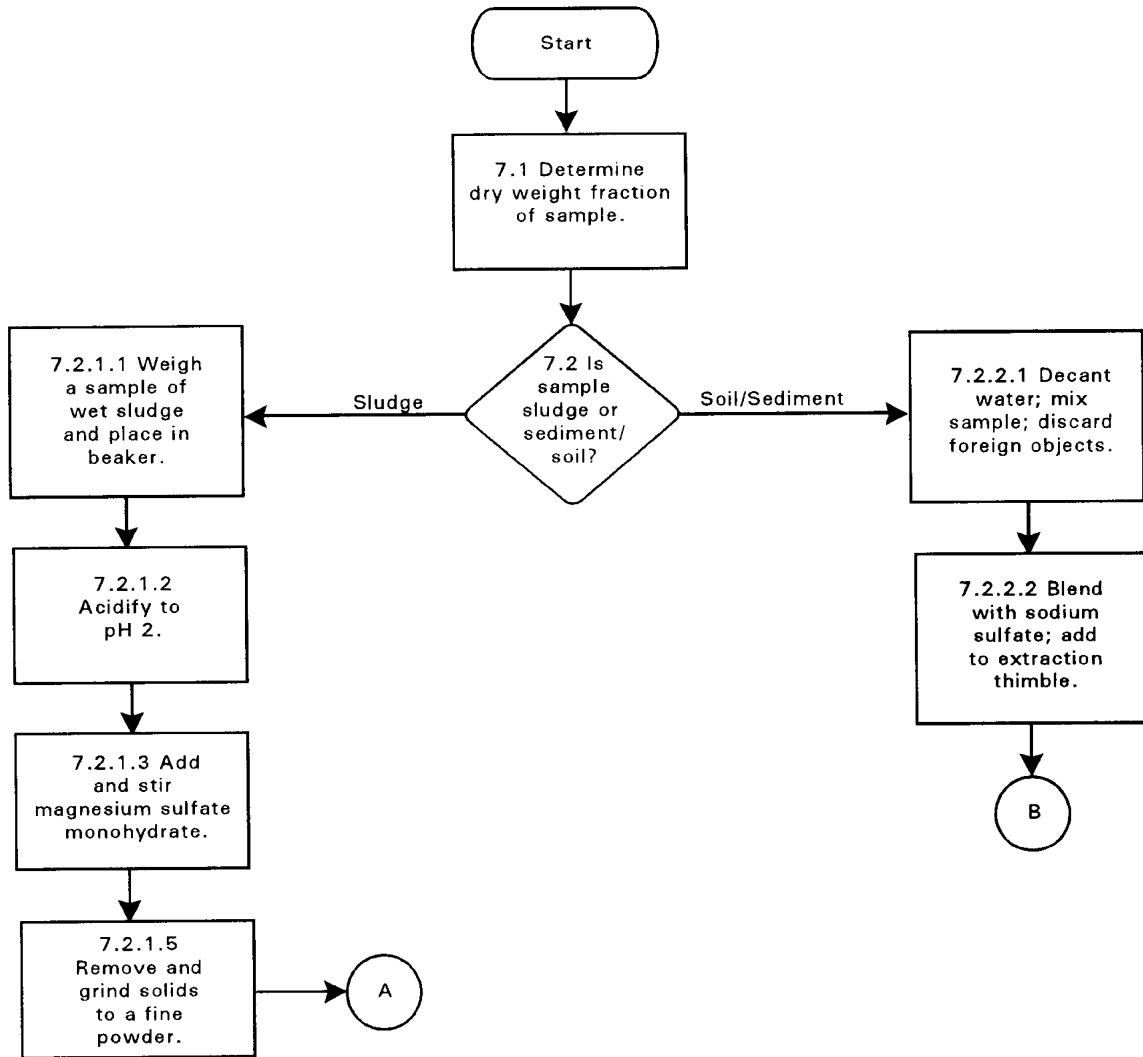
9.0 METHOD PERFORMANCE

9.1 Two oil and grease methods (Methods 9070 and 9071) were tested on sewage by a single laboratory. 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, Method 502A (1975).

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