Oil and grease, extractable from bottom material, extraction-gravimetric (O-5108-83)

Parameter	*Code*
Oil and grease, recoverable from bottom material (mg/kg as oil and grease)	00557

1. Application

1.1 This method is suitable for the determination of oil and grease in air-dried bottom materials containing at least 1,000 mg/kg.

1.2 This method may be used for the determination of oil and grease in wet bottom materials if the appropriate moisture correction is applied.

2. Summary of method

A sample is extracted twice with trichlorotrifluoroethane and the extract is evaporated at 20°C to leave a nonvolatile residue whose weight represents an estimate of the extractable organic matter in the sample.

3. Interferences

Organic solvents vary considerably in their ability to dissolve oil substances and other organic matter. Any method used to obtain an estimate of the amount of extractable organic matter must, of necessity, be highly empirical. Close attention to all operations of the analytical procedure are required to obtain reproducible results.

4. Apparatus

4.1 *Dish,* aluminum foil, 110 mm in diameter, 100-mL capacity.

4.2 *Funnel,* separatory, pear-shaped, 2-L capacity (Corning 6404, or equivalent).

5. Reagents

5.1 *Sodium sulfate,* anhydrous, granular.

5.2 *Sulfuric acid,* concentrated (sp. gr. 1.84).

5.3 *Sulfuric acid,* (1 + 1): Slowly and cautiously, with constant stirring and cooling, add 100 mL concentrated H_2SO_4 to 100 mL demineralized water.

5.4 *Trichlorotrifluoroethane solvent,* 1,1,2-Trichlorotrifluoroethane, b.p. 47.6°C, reagent grade.

6. Procedure

6.1 Weigh, to the nearest mg, approximately 1 g of air-dried sample material. Alternatively, a wet sample may be weighed if a correction is made for moisture content.

6.2 Quantitatively transfer the weighed sample to a 2-L-capacity separatory funnel. Add approximately 900 mL demineralized water and shake to mix. Prepare a 900-mL demineralized water blank and carry it through the sample-analysis procedure. Subtract the residual weight (blank) from the sample extract residual weight. If the weight of the blank exceeds 4.0 mg, a new bottle of solvent must be obtained to provide a blank of 4.0 mg or less.

6.3 Add 5 mL sulfuric acid (1 + 1). Shake to mix thoroughly.

6.4 Add 25 mL trichlorotrifluoroethane and shake vigorously for 2 min, stopping to vent the pressure as necessary.

6.5 Allow the layers to separate and then draw off the solvent and filter it through a small amount of anhydrous Na_2SO_4 placed on a small filter paper (Whatman No. 40, or equivalent) in a funnel. Collect the filtrate in a tared aluminum-foil dish.

6.6 Repeat steps 6.4 and 6.5, filtering the solvent through the same funnel and adding the filtrate to that already collected in the aluminum-foil dish.

6.7 Wash the filter paper with three 5-mL portions of solvent, collecting all washings in the aluminum-foil dish.

6.8 Evaporate the solvent collected in the aluminum-foil dish at room temperature (20˚C) in a well-ventilated fume hood.

6.9 Rinse the inside of the aluminum-foil dish with demineralized water to remove traces of sulfuric acid. Dry the dish in a desiccator to remove water.

6.10 Weigh the residue remaining in the dish after the water has evaporated.

7. Calculations

7.1 Determine the mg/kg extractable organic matter in the air-dried sample as follows:

$$\text{Organic matter, extractable, mg/kg} = \frac{R_X - R_b}{\text{sample weight in g}} \times 1{,}000,$$

where

R_x = weight of extracted residue, in mg, and

R_b = weight of solvent residue (blank), in mg.

NOTE: If wet bottom-material sample is used in preference to air-dried sample, a factor correcting for moisture content must be applied to above equation.

8. Report

8.1 Report organic matter, extractable, air-dried bottom material, concentrations as follows: less than 10,000 mg/kg, nearest 1,000 mg/kg; 10,000 mg/kg and above, two significant figures.

9. Precision

Precision data cannot be given for this determination because of the variable nature of the materials being extracted.