

# STANDARD OPERATING PROCEDURE

## For SM 2540 G

### Determination of Total, Fixed, and Volatile Solids In Solid and Semi-solid Samples

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SOP #: SM 2540 G

REVISION #: 0

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# MassDEP

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#### LIST OF REVISIONS

Rev. #	Date	Description of Revision	Page #
0	October 2000	None	
0	October 2000	Replaced old DEP Logo with state seal + MassDEP (December 2006)	Title page & header



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## **METHOD # SM 2540 G, Total/Volatile Solids, Approved for NPDES Monitoring**

### **1.0 SCOPE AND APPLICATION**

- 1.1 This method is applicable to the determination of total solids, and its fixed and volatile fractions in such solid and semisolid samples as river and lake sediment, sludges separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge de-watering processes.

### **2.0 SUMMARY OF METHOD**

- 2.1 A sample of 25 to 50 g is dried at 105°C to determine percent solid and is then ignited at 550°C for 1 h to determine fixed and volatile solids.

### **3.0 DEFINITIONS**

Reserved

### **4.0 INTERFERENCES**

- 4.1 The determination of both total and volatile solids in these materials is subject to negative error due to loss of ammonium carbonate and volatile organic matter during drying. Although this is true also for wastewater, the effect tends to be more pronounced with sediments, and especially with sludges and sludge cake. The mass of organic matter recovered from sludge and sediment requires a longer ignition time than that specified for raw wastewater, wastewater effluent, or polluted waters. Carefully observe specified ignition time and temperature to control losses of volatile inorganic salts if these are a problem. Make all weighings quickly because wet samples tend to lose weight by evaporation. After drying or ignition, residues are often very hygroscopic and rapidly absorb moisture from the air.

### **5.0 SAFETY**

- 5.1 Standard laboratory protective clothing is required. A face mask is worn for hazardous samples.

### **6.0 EQUIPMENT AND SUPPLIES**

- 6.1 Analytical Balance- capable of weighing to 0.1 mg.
- 6.2 Drying Oven- for operation at 103° to 105°C.
- 6.3 Evaporating Dishes- Porcelain.
- 6.4 Desiccator- provided with a desiccant containing a color indicator or moisture concentration.
- 6.5 Muffle furnace for operation at 550°C.

### **7.0 REAGENTS AND STANDARDS**

Reserved



## **8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE**

- 8.1 Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4°C up to time of analysis to minimize microbiological decomposition of solids.

## **9.0 QUALITY CONTROL**

- 9.1 Duplicates are analyzed with every batch (10% frequency).
- 9.2 The analytical balance is calibrated every day.

## **10.0 CALIBRATION AND STANDARDIZATION**

- 10.1 Calibration of Analytical Balance-
- 10.1.1 Press and hold Cal/Menu button until 'Cal' appears in display.
- 10.1.2 When blinking "100.0000" appears in display, place 100 g weight on pan.
- 10.1.3 Remove when blinking '0.0000' appears in display. The calibration is complete when 'Cal Done' appears in display.

## **11.0 PROCEDURE**

- 11.1 Preparation of evaporating dish - If only total solids are to be measured, heat clean dish to 103 to 105°C for 1 h. Store and cool dish in desiccator until needed. Weigh immediately before use. If volatile solids are to be measured, ignite a clean evaporating dish at 550°C for 1 h in a muffle furnace.
- 11.2 Sample Analysis
- 11.2.1 Fluid samples: If the sample contains enough moisture to flow more or less readily, stir to homogenize, place 25 to 50 g in a prepared evaporating dish and weigh. Evaporate to dryness at 94°C, dry at 103 to 105°C for 1 h, cool to balance temperature in an individual desiccator containing fresh desiccant, and weigh. Repeat heating, cooling, desiccating, and weighing procedure until the weight change is less than 4% or 50 mg whichever is less. Analyze at least 10% of all samples in duplicate. Duplicate determinations should agree within 5% of their average weight.
- 11.2.2 Solid samples : If the sample consists of discrete pieces of solid material (dewatered sludge for example), take cores from each piece with a No. 7 cork borer or pulverize the entire sample coarsely on a clean surface by hand, using rubber gloves. Place 25 to 50 g in a prepared evaporating dish and weigh. Place in oven at 103 to 105°C overnight. Cool to balance temperature in a desiccator and weigh. Repeat drying (1 h), cooling, weighing, and desiccating steps until weight change is less than 4% or 50 mg, which ever is less. Analyze at least 10% of all samples in duplicate. Duplicate determinations should agree within 5% of their average weight.
- 11.2.3 Fixed and Volatile Solids: Transfer dried residue from previous step. To a cool muffle furnace, heat furnace to 550°C and ignite for 1 h. (If the residue contains large amounts of organic matter, first ignite it over a gas burner and under an exhaust hood in the presence of adequate air to lessen losses due to reducing conditions and to avoid odors in the laboratory.) Cool in desiccator to balance temperature and weigh. Repeat igniting (30 min), cooling, desiccating, and weighing steps until the weight change is less than 4



% or 50 mg, whichever is less. Duplicate determinations should agree within 5% of their average weight.

## 12.0 DATA ANALYSIS AND CALCULATIONS

$$\% \text{ total solid} = \frac{(A - B) \times 100}{C - B}$$

$$\% \text{ volatile solid} = \frac{(A - D) \times 100}{A - B}$$

$$\% \text{ fixed solids} = \frac{(D - B) \times 100}{A - B}$$

where:

A = weight of dried residue + dish, mg,

B = weight of dish, mg,

C = weight of wet sample + dish, mg, and

D = weight of residue + dish after ignition, mg.

## 13.0 METHOD PERFORMANCE

Reserved

## 14.0 POLLUTION PREVENTION

14.1 There are no hazardous chemicals used in this method.

## 15.0 WASTE MANAGEMENT

15.1 WES laboratory waste management practices comply with all applicable federal, state, and local rules and regulations.

## 16.0 REFERENCES

16.1 American Public Health Association (APHA). 1995. *Standard Methods for the Examination of Water and Wastewater*, 19th Edition. APHA, American Water Works Association, and Water Environment Federation, Washington, D.C.

## 17.0 VALIDATION DATA

Reserved