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**METHOD 1683**

**Specific Oxygen Uptake Rate in Biosolids**

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## Disclaimer

This draft method has been reviewed and approved for publication by the Analytical Methods Staff within the Engineering and Analysis Division of the U.S. Environmental Protection Agency. Mention of trade names or commercial products does not constitute endorsement or recommendation for use. EPA plans further validation of this draft method. The method may be revised following validation to reflect results of the study. This method version contains minor editorial changes to the February 1999 version.

EPA welcomes suggestions for improvement of this method. Suggestions and questions concerning this method or its application should be addressed to:

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Note: This method is intended to be performance based. The laboratory is permitted to modify or omit any steps or procedure, provided that *all* performance requirements in this method are met. The laboratory may *not* omit any quality control analyses. The terms “shall”, “must”, and “may not” indicate steps and procedures required for producing reliable results. The terms “should” and “may” indicate optional steps that may be modified or omitted if the laboratory can demonstrate that the modified method produces results equivalent or superior to results produced by this method.

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# Method 1683

## Specific Oxygen Uptake Rate in Biosolids

### 1.0 Scope and Application

- 1.1** This method is applicable to the determination of the specific oxygen uptake rate (SOUR) (Section 18.14) in biosolids (Section 18.2) treated in an aerobic process.
- 1.2** This method is for use in the Environmental Protection Agency's (EPA's) data gathering and monitoring programs under the Clean Water Act, the Solid Waste Disposal Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation, and Liability Act, and the Safe Drinking Water Act. To confirm approval of this method for use in compliance monitoring programs, consult the appropriate sections of the Code of Federal Regulations (Reference 16.1).
- 1.3** This method was developed by integrating the analytical procedures contained in Standard Method 2710 B (Reference 16.2) with the recommendations described in the EPA Document "Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge" (Reference 16.3) and the quality control (QC) procedures necessary to ensure the validity of analytical results. This method is associated with EPA Method 1691: *Municipal Biosolids Sampling Guidance* (Reference 16.4).
- 1.4** This method may not be applicable to sewage sludge (Section 18.11) with higher than two percent solids or to sewage sludge digested at temperatures lower than 10°C or higher than 30°C.
- 1.5** Microorganisms in sewage sludge use oxygen as they consume organic matter. The level of microbial activity in sludge is indicated by the microorganisms' oxygen uptake rate. High oxygen uptake rates indicate high microbial activity and high organic matter content; low oxygen uptake rates indicate low microbial activity and low organic matter content.
- 1.6** This method is performance-based. The laboratory is permitted to omit any step or modify any procedure, provided that all performance requirements in this method are met. Requirements for establishing method equivalency are given in Section 9.1.1.
- 1.7** Each laboratory that uses this method must demonstrate the ability to generate acceptable results based on the performance criteria given in Section 17.

### 2.0 Summary of Method

- 2.1** The sample is maintained at the temperature of the digester from which it was drawn, from the time of collection until the end of analysis. The sample is kept under aeration if the test cannot be run expeditiously.
- 2.2** The total solids content (Section 18.15) of the sample is determined on an aliquot (Appendix A).
- 2.3** The sample is mixed, and the concentration of dissolved oxygen (DO) (Section 18.3) in the sample is increased by aeration. An aliquot of the sample is placed in a biological oxygen demand (BOD) bottle and kept well mixed. Using an oxygen-sensing probe, a manometer, or respirometer, DO is recorded periodically over a 15-minute period, or until DO becomes rate-limiting (Section 18.10).

- 2.4** The oxygen consumption rate (Section 18.9) is calculated as the absolute value of the slope of the linear portion of the DO versus time curve when an oxygen probe is used, or from calculations as specified by the manufacturer of the manometer or respirometer. The SOUR is obtained by dividing the oxygen consumption rate by total solids. The SOUR for the temperature at which the aerobic digestion is occurring in the treatment works is corrected to 20°C, as a standardization for reporting purposes.

### **3.0 Definitions**

Definitions for terms used in this method are given in the glossary (Section 18).

### **4.0 Interferences**

- 4.1** SOUR determination is sensitive to the time lag between sample collection and analysis. To minimize changes in the sample due to microbial activity over time, samples should be analyzed as soon as possible after collection. The sample should be transported to the laboratory expeditiously and kept under aeration if the SOUR test cannot be run immediately. The sample must be kept at the temperature of the digester from which it was drawn.
- 4.2** This method may not be applicable to samples with total solids above two percent, samples that remain heterogenous after mixing, and samples that contain solids that do not remain in suspension and settle to the bottom of the container. In these cases, aeration and mixing are difficult, and the method yields inconsistent results because samples can not be kept well mixed during analysis. Although adequate mixing is essential, the samples must not be blended with a homogenizing instrument. Homogenization is likely to change sample characteristics and the rate of oxygen uptake significantly.
- 4.3** The results of the determination of SOUR are extremely sensitive to temperature variations. It is essential that the SOUR test be performed at the temperature of the digester from which the sample was drawn, because dissolved oxygen concentration and microbial activity are influenced by temperature. Poor precision is obtained when duplicate determinations are not made at the same temperature.
- 4.4** The SOUR for the sample reflects the temperature of the digester from which it was drawn. This value is corrected to 20°C by using Equation 3. The correction is valid only for digester temperatures between 10°C and 30°C.
- 4.5** The fixed solids (dissolved inert mineral solids) (Section 18.4) in the wastewater in which the sludge particles are suspended do not exert an oxygen demand and should not be part of the total solids in the SOUR determination. Ordinarily, the fixed solids are such a small part of the total solids that they can be ignored. If it is suspected that there is a high level of fixed solids in the wastewater (a ratio of fixed to total solids greater than 0.15), the volatile solids content (Section 18.16) should be determined with Method 1684 (Reference 16.5) and be used instead of total solids in the calculation of SOUR (Equation 2).
- 4.6** Aeration of the sample to at least 5 mg/L DO is critical. Low DO at the start of the test may limit oxygen uptake and will be indicated by a decreasing rate of oxygen consumption as the test progresses. Such data must be rejected as being unrepresentative of SOUR and the test must be repeated with higher initial DO levels. The sample should be aerated again following procedures in Section 11.4.

- 4.7** For samples suspected to contain high levels of oxidizable materials:
- 4.7.1** It is important not to oversupply samples with oxygen during initial sample aeration. Prolonged aeration may lead to oxidation of significant portions of the organic matter in the sample and may alter the characteristics of the sample. Procedures in Section 11.4 should be followed closely. After each attempt at sample aeration, DO should be checked. Once the sample has reached a DO concentration of at least 5 mg/L, the sample should be analyzed immediately.
- 4.7.2** High levels of oxidizable materials will result in high levels of microbial activity and rapid rates of oxygen consumption. Measurements of DO concentration should be taken at frequent intervals, especially in the first two minutes of the analysis (Section 11.5).
- 4.8** For calculation of the sample's oxygen uptake rate, only the linear portion of the DO versus time curve should be used. The curved portion of the line signifies that DO may have become rate-limiting, and the corresponding values must not be used to determine the oxygen uptake rate.
- 4.9** Air bubbles trapped among the solids in an aerated sample can cause falsely high DO readings. Care should be taken to aerate the sample in a manner that does not cause air bubbles to become entrained in the sample.
- 4.10** Oxygen probes may not be accurate below 1 mg DO/L. Any data that are recorded at or below 1 mg/L should not be used to calculate oxygen uptake rate.

## 5.0 Safety

- 5.1** This method does not address all safety issues associated with its use. Each chemical and environmental sample should be regarded as a potential health hazard and exposure should be minimized. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in the chemical analysis. Additional information on laboratory safety can be found in Reference 16.2 and Reference 16.6.
- 5.2** All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causative agents.

## 6.0 Equipment and Supplies

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**NOTE:** *Brand names, suppliers, and part numbers are for illustrative purposes only, and no endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here. Meeting the performance requirements of this method is the responsibility of the sampling team and laboratory.*

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- 6.1** Oxygen-consumption rate measurement device, either:
- 6.1.1** Probe with an oxygen-sensitive membrane electrode (polarographic or galvanic) OR

**6.1.2** Manometer or respirometer with appropriate reporting capabilities and sample capacity of at least 300 mL. The instrument should have an oxygen supply capacity greater than the oxygen consumption rate of the sample, or at least 150 mg/L·h.

**6.2** Stopwatch or other suitable timing device.

**6.3** Thermometer ( $\pm 0.5^{\circ}\text{C}$ ).

**6.4** Glass or plastic bottles of a suitable size (at least 800-mL) for sample collection.

**6.5** Biological oxygen demand (BOD) bottles, 300-mL capacity.

**6.6** Rubber gloves.

**6.7** Magnetic stirring device such as a PTFE-coated stir bar and stirring plate capable of keeping the sample well mixed during analysis.

## **7.0 Reagents and Standards**

**7.1** Reagent water —Deionized, distilled water. Autoclave at  $121^{\circ}\text{C}$  for 20 minutes to sterilize.

**7.2** Sand —Rinse sand. Bake at  $400^{\circ}\text{C}$  for eight hours to sterilize.

## **8.0 Sample Collection, Preservation, and Storage**

**8.1** Samples are collected in glass or plastic bottles, provided that the material in the sample does not adhere to container walls. A volume of at least 700 mL should be collected. Sampling should be done in accordance with Reference 16.3 and Reference 16.4.

**8.2** Samples must be analyzed as soon as possible after sample collection. Samples must be maintained at the temperature of the digester from which they were drawn from the time of collection until the end of analysis.

## **9.0 Quality Control**

**9.1** Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program *generally* include an initial demonstration of laboratory capability (Section 9.2). In the absence of an appropriate standard for SOUR, the characterization of the method detection limit (MDL) and initial precision and recovery (IPR) is not valid. The QC program for this method consists of the ongoing analysis of laboratory reagent blanks and duplicates (Sections 9.3 and 9.4) as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data generated. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

**9.1.1** In recognition of advances that are occurring in analytical technology, the analyst is permitted certain options to improve accuracy or lower the costs of measurements, provided that all performance specifications are met. If an analytical technique other than the techniques described in this method is used, that technique must have a specificity

equal to or better than the specificity of the techniques in this method for standard oxygen uptake rate in the sample of interest. Specificity is defined as producing results equivalent to the results produced by this method for laboratory blank samples (Sections 9.3 and 18.5) and, where applicable, duplicate environmental samples (Section 9.4).

- 9.1.1.1** The laboratory is required to maintain records of modifications made to this method. These records include the following, at a minimum:
- 9.1.1.1.1** The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modification.
  - 9.1.1.1.2** The analyte measured (SOUR).
  - 9.1.1.1.3** A narrative stating reason(s) for the modification.
  - 9.1.1.1.4** Results from all quality control (QC) tests comparing the modified method to this method, including:
    - (a) Calibration (Section 10);
    - (b) Analysis of blanks (Section 9.3); and
    - (c) Analysis of duplicates (Section 9.4).
  - 9.1.1.1.5** Data that will allow an independent reviewer to validate each determination by tracing the instrument output (weight, DO, or other data) to the final result. These data are to include:
    - (a) Sample numbers and other identifiers;
    - (b) Sample preparation dates;
    - (c) Analysis dates and times;
    - (d) Analysis sequence/run chronology;
    - (e) Sample weight or volume;
    - (f) Sample temperature (°C);
    - (g) Copies of logbooks, printer tapes, and other recordings of raw data; and
    - (h) Data system outputs and other data to link the raw data to the results reported.
- 9.1.2** Analyses of laboratory blanks are required to demonstrate freedom from contamination. The procedure and criteria for blank analyses are described in Section 9.3.
- 9.1.3** Analyses of duplicate samples are required to demonstrate method accuracy and precision. The procedure and criteria for duplicate analyses are described in Section 9.4.
- 9.2** Initial demonstration of laboratory capability—The initial demonstration of laboratory capability is generally used to characterize laboratory performance and method detection limits.
- 9.2.1** Method detection limit (MDL)—In the absence of an appropriate standard for SOUR, the characterization of the MDL is not valid for this method.

- 9.2.2** Initial precision and recovery (IPR)—In the absence of an appropriate standard for SOUR, the characterization of IPR is not valid for this method.
- 9.3** Laboratory blanks
- 9.3.1** Prepare and analyze a laboratory blank with each analytical batch (Section 18.1). The blank must be subjected to the same procedural steps as a sample, and will consist of a 5 g aliquot of sterile sand (Section 7.2) in 500 mL of sterile reagent water (Section 7.1).
- 9.3.2** Acceptance criteria for blank analyses will be determined during method validation studies.
- 9.4** Duplicate analyses
- 9.4.1** One sample per analytical batch must be analyzed in duplicate. The duplicate analyses must be performed within the same analytical batch (Section 18.1).
- 9.4.2** The relative percent difference (RPD) between duplicate samples analyzed for SOUR must be determined using Equation 1.

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Equation 1

$$RPD = 200 \times \frac{(|D1 - D2|)}{(D1 + D2)}$$

Where:

*D1* = SOUR in the environmental sample (mg/g)/h

*D2* = SOUR in the duplicate sample (mg/g)/h

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- 9.4.3** Acceptance criteria for duplicate analyses will be determined during method validation studies.
- 9.5** The following QC analyses are not applicable to this method and are therefore not required: method detection limit, initial precision and recovery, ongoing precision and recovery, matrix spike, matrix spike duplicate, calibration verification, and quality control samples.

## 10.0 Calibration and Standardization

- 10.1** Oxygen probe—Follow manufacturer's calibration procedure exactly. Determine DO concentration of a sample of reagent water using the membrane electrode method (Reference 16.2). Calibrate the membrane electrodes with the sample of reagent water of known DO concentration. When performing multiple analyses, calibration must be checked before each analysis against the sample of reagent water of known DO concentration.
- 10.2** Manometer or respirometer—Calibrate the meter following manufacturer's calibration procedure exactly.

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## 11.0 Procedure

11.1 Filter the sample through a 1/4-inch screen to remove large objects.

11.2 Determine total solids (Appendix A).

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**NOTE:** *This method may not be applicable to samples with total solids high enough to prevent adequate mixing during the analysis (Section 4.2).*

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11.3 Monitor the temperature of the sample during analysis to ensure that it remains constant. The sample should have been kept at the temperature of the digester from which it was drawn from the time of collection until analysis.

11.4 Increase dissolved oxygen (DO) concentration of the sample by shaking it vigorously in a partially filled bottle or by bubbling air through it for five minutes. Check the DO concentration of the sample.

11.4.1 If DO is 5 mg/L or greater, the sample is sufficiently aerated. Proceed immediately with the analysis.

11.4.2 If DO is below 5 mg/L, repeat Section 11.4.

11.4.2.1 If the DO has not increased by more than 0.1 mg/L, the sample is sufficiently aerated. Proceed immediately with the analysis.

11.4.2.2 If the DO has increased by more than 0.1 mg/L, continue repeating Section 11.4 until the criterion in Section 11.4.1 or 11.4.2.1 is met.

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**NOTE:** *For samples suspected to contain high levels of oxidizable materials, it is important not to oversupply samples with oxygen during sample aeration. Procedures in Section 11.4 should be followed closely. After each attempt at sample aeration, DO should be checked. Once the requirements in Sections 11.4.1 through 11.4.2.2 have been met, the sample should be analyzed immediately to minimize oxidation of available materials. High levels of oxidizable materials will result in high levels of microbial activity in a well-aerated sample.*

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11.5 Measurement of oxygen uptake rate

11.5.1 Oxygen probe

11.5.1.1 Fill one 300-mL biochemical oxygen demand (BOD) bottle containing a magnetic stirring bar to overflowing with an appropriate volume of a representative sample of the aerated sludge to be tested. Containers should allow for complete mixing and oxygen availability.

11.5.1.2 Immediately insert the oxygen-sensing probe into the BOD bottle. Displace enough sample with the probe to fill flared top of bottle and isolate its contents from the atmosphere. Be sure that no large bubbles are present. Activate the probe stirring mechanism or magnetic stirrer.

- 11.5.1.3** After the meter reading has stabilized, record initial DO reading and start timing device. Record DO data at 15-second intervals for the first two minutes of the analysis, then every 60 seconds. Record data over a 15-minute period or until DO is no longer decreasing at a steady rate, whichever occurs first. When DO is no longer decreasing at a steady rate, this could indicate that DO has become rate-limiting.
- 11.5.1.4** The oxygen probe may not be accurate below 1 mg DO/L. Any data that are recorded at or below 1 mg/L should not be used to calculate SOUR.
- 11.5.1.5** When multiple analyses are performed, calibration of the oxygen probe must be checked before each analysis against a sample of reagent water of known DO concentration.

#### **11.5.2** Manometer or respirometer

- 11.5.2.1** Follow manufacturer's instructions for instrument startup, sample volume, and sample container requirements.
- 11.5.2.2** After meter reading has stabilized, record initial reading, and start timing device. Record data at 15-second intervals for the first two minutes of the analysis, then every 60 seconds. Record data over a 15-minute period or until DO is no longer decreasing at a steady rate, whichever occurs first. When DO is no longer decreasing at a steady rate, this could indicate that DO has become rate-limiting.
- 11.5.2.3** Refer to manufacturer's instructions for lower limiting DO value.
- 11.5.2.4** When multiple analyses are performed, instrument calibration must be checked before each analysis or as recommended by the manufacturer. For self-calibrating instruments, refer to the manufacturer's instructions.

## **12.0 Data Analysis and Calculations**

- 12.1** Oxygen probe— Plot observed readings (DO, mg/L) versus time (minutes). Determine the slope of the linear portion of the curve. The absolute value of the slope is the oxygen consumption rate in milligrams per liter per minute (mg/L · min). A minimum of five data points in the linear portion of the DO versus time curve is required to calculate the oxygen uptake rate.
- 12.2** Manometer or respirometer—Refer to manufacturer's instructions for calculating the oxygen consumption rate.
- 12.3** Calculate the specific oxygen consumption rate (SOUR) for the sample, in milligrams per gram per hour (Equation 2).

## Equation 2

$$SOUR_T = \frac{OUR}{TS} \times \frac{60 \text{ min}}{h}$$

Where:

$SOUR_T$  = specific oxygen uptake rate in the sample, (mg/g)/h

$OUR$  = oxygen uptake rate in the sample, (mg/L)/min

$TS$  = total solids in the sample, (g/L)

$T$  = temperature of the sample during digestion and analysis, °C

- 12.4** The SOUR for the sample will reflect the temperature in the digester from which the sample was drawn. This value is corrected to 20°C (Equation 3), if the sample temperature is not 20°C. This correction ( $\Theta$ ) is applicable only if the sample temperature was between 10°C and 30°C and the sample was maintained at that temperature from the time of collection until the end of analysis (Section 4.4).

## Equation 3

$$SOUR_{20} = SOUR_T \times \Theta^{(20 - T)}$$

Where:

$SOUR_{20}$  = specific oxygen uptake rate at 20°C, (mg/g)/h

$SOUR_T$  = specific oxygen uptake rate in the sample, (mg/g)/h

$T$  = temperature of the sample during digestion and analysis, °C

$\Theta$  = 1.05 above 20°C

1.07 below 20°C

## 13.0 Method Performance

- 13.1** Method performance (quality control acceptance criteria) for method blanks and duplicate samples will be determined during the laboratory validation of this method.

## 14.0 Pollution Prevention

- 14.1** Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. The Environmental Protection Agency has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot feasibly be reduced at the source, the Agency recommends recycling as the next best option.
- 14.2** For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington D.C. 20036, (202) 872-4477.

## 15.0 Waste Management

- 15.1** The laboratory is responsible for complying with all Federal, State, and local regulations governing waste management, particularly hazardous waste identification rules and land disposal restrictions, and for protecting the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. An overview of requirements can be found in *Environmental Management Guide for Small Laboratories* (EPA 233-B-98-001).

## 16.0 References

- 16.1** Code of Federal Regulations 40, Ch. 1, Part 136, Table 1B, Part 141 and 141.23.
- 16.2** "Standard Methods for the Examination of Water and Wastewater," 18th ed., American Public Health Association, 1015 15th Street NW, Washington, DC 20005. 1-35: Section 1090 (Safety); 2-77: Section 2710 B (Oxygen Consumption Rate); 4-102: Section 4500 OG (Membrane Electrode Method). 1992.
- 16.3** U.S. Environmental Protection Agency, 1992. Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge. Publ 625/R-92/013. Office of Research and Development, Washington, DC.
- 16.4** U.S. Environmental Protection Agency, 1998. Method 1691: Municipal Biosolids Sampling Guidance. Draft, September 1998. Office of Water, Washington, DC.
- 16.5** U.S. Environmental Protection Agency, 1998. Method 1684: Total, Fixed, and Volatile Solids in Water, Solids and Biosolids. Draft, October 1998. Office of Water, Washington, DC.
- 16.6** "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3<sup>rd</sup> Edition, 1979.

## 17.0 Tables, Diagrams, Flowcharts, and Validation Data

Tables containing method requirements for QA/QC will be added after the validation study has been performed.

## 18.0 Definitions

- 18.1** Analytical batch—A set of up to 10 samples analyzed during the same eight-hour shift. Each analytical batch of 10 or fewer samples must be accompanied by a laboratory blank (Section 9.3), and a duplicate sample (Section 9.4), resulting in a minimum of three analyses (1 sample, 1 blank, and 1 duplicate sample) and a maximum of 12 analyses.
- 18.2** Biosolids—The treated residuals from wastewater treatment that can be used beneficially.
- 18.3** Dissolved oxygen (DO)—The quantity of oxygen present in solution.
- 18.4** Fixed solids—The residue left in the vessel after a sample is ignited (heated to dryness at 550°C).

- 18.5** Laboratory blank (method blank)—A reference matrix sample consisting of 5 g of sterile sand (Section 7.2) in 500 mL of sterile reagent water (Section 7.1) that is treated exactly as an environmental sample including exposure to all glassware, equipment and reagents that are used with environmental samples. The laboratory blank is used to determine if interferences are present in the laboratory environment, the reagents, or the apparatus.
- 18.6** May—This action, activity, or procedural step is neither required nor prohibited.
- 18.7** May not—This action, activity, or procedural step is prohibited.
- 18.8** Must—This action, activity, or procedural step is required.
- 18.9** Oxygen consumption rate (oxygen uptake rate, OUR)—The rate of oxygen usage by microorganisms in a biological system.
- 18.10** Rate-limiting—The level of dissolved oxygen is rate-limiting when it is the factor that controls the rate of oxygen uptake by the microorganisms in a sample.
- 18.11** Sewage sludge—solid, semi-solid, or liquid residue generated during the treatment process of domestic sewage in a treatment works.
- 18.12** Shall—This action, activity or procedural step is required.
- 18.13** Should—This action, activity, or procedural step is suggested but not required.
- 18.14** Specific oxygen uptake rate (SOUR)—The mass of oxygen consumed per unit time per unit mass of total solids in sewage sludge.
- 18.15** Total solids—The residue remaining after evaporation of liquid from and subsequent drying of a sample of sewage sludge in an oven at 103°C to 105°C.
- 18.16** Volatile solids—The weight loss after a sample is ignited (heated to dryness at 550°C). Determinations of fixed and volatile solids do not distinguish precisely between inorganic and organic matter because the loss on ignition is not confined to organic matter. It includes losses due to decomposition or volatilization of some mineral salts.

## Appendix A: Total Solids in Solids and Biosolids

### 1.0 Scope and Application

- 1.1 This procedure is applicable to the determination of total solids in such solid and semisolid samples as soils, sediments, sludges separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.
- 1.2 This procedure is taken from EPA Method 1684: *Total, Fixed, and Volatile Solids in Water, Solids and Biosolids*.
- 1.3 Method detection limits (MDLs) and minimum levels (MLs) have not been formally established for this draft procedure. These values will be determined during the validation of Method 1684.
- 1.4 This procedure is performance based. The laboratory is permitted to omit any step or modify any procedure (e.g., to overcome interferences, to lower the cost of measurement), provided that all performance requirements in this procedure are met. Requirements for establishing equivalency are given in Section 9.1.2 of Method 1683.
- 1.5 Each laboratory that uses this procedure must demonstrate the ability to generate acceptable results using the procedure in Section 9.2 of this appendix.

### 2.0 Summary of Method

- 2.1 Sample aliquots of 25-50 g are dried at 103°C to 105°C to drive off water in the sample.
- 2.2 The mass of total solids in the sample is determined by comparing the mass of the sample before and after each drying step.

### 3.0 Definitions

- 3.1 Total Solids—The residue left in the vessel after evaporation of liquid from a sample and subsequent drying in an oven at 103°C to 105°C.
- 3.2 Initial precision and recovery (IPR)—Four aliquots of the diluted PAR analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed the first time this method is used and any time the method or instrumentation is modified.
- 3.3 Method detection limit (MDL)—The lowest level at which an analyte can be detected with 99 % confidence that the analyte concentration is greater than zero.
- 3.4 Ongoing precision and recovery standard (OPR, also called a laboratory control sample)—A laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and accuracy.
- 3.5 Additional definitions are given in Section 18.0 of Method 1683.

## 4.0 Interferences

- 4.1** Sampling, subsampling, and pipeting multi-phase samples may introduce serious errors (Reference 16.1). Make and keep such samples homogeneous during transfer. Use special handling to ensure sample integrity when subsampling. Mix small samples with a magnetic stirrer. If visible suspended solids are present, pipet with wide-bore pipets. If part of a sample adheres to the sample container, intensive homogenization is required to ensure accurate results. When dried, some samples form a crust that prevents evaporation; special handling such as extended drying times are required to deal with this. Avoid using a magnetic stirrer with samples containing magnetic particles.
- 4.2** The temperature and time of residue drying has an important bearing on results (Reference 16.1). Problems such as weight losses due to volatilization of organic matter, and evolution of gases from heat-induced chemical decomposition, weight gains due to oxidation, and confounding factors like mechanical occlusion of water and water of crystallization depend on temperature and time of heating. It is therefore essential that samples be dried at a uniform temperature, and for no longer than specified. Each sample requires close attention to desiccation after drying. Minimize the time the desiccator is open because moist air may enter and be absorbed by the samples. Some samples may be stronger desiccants than those used in the desiccator and may take on water. If uptake of water by a sample is suspected, the operator should weigh the sample to see if gains weight while in the desiccator. If the sample is indeed taking up water, then a vacuum desiccator should be used.
- 4.3** Residues dried at 103°C to 105°C may retain some bound water as water of crystallization or as water occluded in the interstices of crystals. They lose CO<sub>2</sub> in the conversion of bicarbonate to carbonate. The residues usually lose only slight amounts of organic matter by volatilization at this temperature. Because removal of occluded water is marginal at this temperature, attainment of constant weight may be very slow.
- 4.4** Results for residues high in oil or grease may be questionable because of the difficulty of drying to constant weight in a reasonable time.
- 4.5** The determination of total solids is subject to negative error due to loss of ammonium carbonate and volatile organic matter during the drying step at 103°C to 105°C. Carefully observe specified ignition time and temperature to control losses of volatile inorganic salts if these are a problem.

## 5.0 Safety

- 5.1** Refer to Section 5.0 of Method 1683 for safety precautions.

## 6.0 Equipment and Supplies

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**NOTE:** *Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

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- 6.1** Evaporating Dishes—Dishes of 100-mL capacity. The dishes may be made of porcelain (90-mm diameter), platinum, or high-silica glass.

- 6.2 Watch glass—Capable of covering the evaporating dishes (Section 6.1).
- 6.3 Steam bath—For evaporation of liquid samples.
- 6.4 Desiccator—Moisture concentration in the desiccator should be monitored by an instrumental indicator or with a color-indicator desiccant.
- 6.5 Drying oven—Thermostatically-controlled, capable of maintaining a uniform temperature of 103°C to 105°C throughout the drying chamber.
- 6.6 Analytical balance—Capable of weighing to 0.1 mg for samples having a mass up to 200 g.
- 6.7 Reference weights—2 mg, 1000 mg, and 50 mg class “S” weights.
- 6.8 Container handling apparatus—Gloves, tongs, or a suitable holder for moving and handling hot containers after drying.
- 6.9 Bottles—Glass or plastic bottles of a suitable size for sample collection.
- 6.10 Rubber gloves.
- 6.11 No. 7 Cork borer (Optional).
- 6.12 Dessicant (Optional).

## 7.0 Reagents and Standards

- 7.1 Reagent water—Deionized, distilled, or otherwise purified water.
- 7.2 Quality Control Spiking Solution—If a commercially available standard that contains total solids can be purchased, the laboratory may use that standard. The laboratory may also prepare a spiking solution. One possible recipe is given below for a sodium chloride-potassium hydrogen phthalate standard (NaCl-KHP).
  - 7.2.1 Dissolve 0.10 g sodium chloride (NaCl) in 500 mL reagent water. Mix to dissolve.
  - 7.2.2 Add 0.10 g potassium hydrogen phthalate (KHP) to the NaCl solution (Section 7.2.1) and mix. If the KHP does not dissolve readily, warm the solution while mixing. Dilute to 1 L with reagent water. Store at 4°C. Assuming 100% volatility of the acid phthalate ion, this solution contains 200 mg/L total solids, 81.0 mg/L volatile solids, and 119 mg/L fixed solids.

## 8.0 Sample Collection, Preservation, and Storage

- 8.1 Use resistant-glass or plastic bottles to collect sample for solids analysis, provided that the material in suspension does not adhere to container walls. Sampling should be done in accordance with Reference 16.2. Begin analysis as soon as possible after collection because of the impracticality of preserving the sample. Refrigerate the sample at 4°C up to the time of analysis to minimize microbiological decomposition of solids. Preferably do not hold samples more than 24 hours.

Under no circumstances should the sample be held more than seven days. Bring samples to room temperature before analysis.

## 9.0 Quality Control

**9.1** Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the ongoing analysis of laboratory reagent blanks, precision and recovery standards, and matrix-spiked samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data thus generated. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

**9.1.1** The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.

**9.1.2** In recognition of advances that are occurring in analytical technology, the analyst is permitted certain options to improve separations or lower the costs of measurements, provided that all performance specifications are met. If an analytical technique other than the techniques specified in this method is used, that technique must have a specificity equal to or better than the specificity of the techniques in this method for total, fixed, and volatile solids in the sample of interest. Specificity is defined as producing results equivalent to the results produced by this method for laboratory-prepared solutions (Section 7.2) that meet all of the QC criteria stated in this method.

**9.1.2.1** Each time a modification is made to this method, the analyst is required to repeat the Initial Precision and Recovery (IPR) test in Section 9.2.2 to demonstrate that the modification produces results equivalent to or better than results produced by this method. If the detection limit of the method will be affected by the modification, the analyst must demonstrate that the MDL is less than or equal to the MDL in this method or one-third the regulatory compliance level, whichever is higher. The tests required for this equivalency demonstration are given in Section 9.2.

**9.1.2.2** The laboratory is required to maintain records of modifications made to this method. These records include the following, at a minimum:

**9.1.2.2.1** The names, titles, addresses, and telephone numbers of the analyst(s) who performed the analyses and modification, and of the quality control officer who witnessed and will verify the analyses and modification.

**9.1.2.2.2** A listing of pollutant(s) measured (total, fixed, and/or volatile solids).

**9.1.2.2.3** A narrative stating reason(s) for the modification.

**9.1.2.2.4** Results from all quality control (QC) tests comparing the modified method to this method, including:

- (a) Initial precision and recovery (Section 9.2.2).
- (b) Analysis of blanks (Section 9.3).
- (c) Accuracy assessment (Section 9.5).
- (d) Ongoing precision and recovery (Section 9.4).

**9.1.2.2.5** Data that will allow an independent reviewer to validate each determination by tracing the instrument output (weight, absorbance, or other signal) to the final result. These data are to include:

- (a) Sample numbers and other identifiers.
- (b) Sample preparation dates.
- (c) Analysis dates and times.
- (d) Analysis sequence/run chronology.
- (e) Sample weights.
- (f) Make and model of analytical balance and weights traceable to NIST.
- (g) Copies of logbooks, printer tapes, and other recordings of raw data.
- (h) Data system outputs, and other data to link the raw data to the results reported.

**9.1.3** Analyses of laboratory blanks are required to demonstrate freedom from contamination. The procedure and criteria for blank analyses are described in Section 9.3.

**9.1.4** Analyses of ongoing precision and recovery (OPR) samples are required to demonstrate that the sample preparation and analysis are in control. The procedure and criteria for OPR samples are described in Section 9.4.

**9.2** Initial demonstration of laboratory capability - The initial demonstration of laboratory capability is used to characterize laboratory performance and method detection limits.

**9.2.1** Method detection limit (MDL) - The method detection limit should be established for total solids using the QC spiking solution (Section 7.2). To determine MDL values, take seven replicate aliquots of the diluted QC spiking solution and process each aliquot through each step of the analytical method. Perform all calculations and report the concentration values in the appropriate units. MDLs should be determined every year or whenever a modification to the method or analytical system is made that will affect the method detection limit.

**9.2.2** Initial Precision and Recovery (IPR) - To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

**9.2.2.1** Prepare four samples by diluting the QC spiking solution (Section 7.2) to 1-5 times the MDL. Using the procedures in Section 11, analyze these samples for total solids.

**9.2.2.2** Using the results of the four analyses, compute the average percent recovery ( $\bar{x}$ ) and the standard deviation ( $s$ , Equation 1) of the percent recovery for total solids.

Equation 1

$$s = \sqrt{\frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n-1}}$$

Where:

$n$  = number of samples

$x$  = % recovery in each sample

$s$  = standard deviation

**9.2.2.3** Compare  $s$  and  $x$  with the corresponding limits for initial precision and recovery in Table 2 (to be determined in validation study). If  $s$  and  $x$  meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however,  $s$  exceeds the precision limit or  $x$  falls outside the range for recovery, system performance is unacceptable. In this event, correct the problem, and repeat the test.

### 9.3 Laboratory blanks

**9.3.1** Prepare and analyze a laboratory blank initially (i.e. with the tests in Section 9.2) and with each analytical batch. The blank must be subjected to the same procedural steps as a sample, and will consist of approximately 25 g of reagent water.

**9.3.2** If material is detected in the blank at a concentration greater than the MDL (Section 1.3), analysis of samples must be halted until the source of contamination is eliminated and a new blank shows no evidence of contamination. All samples must be associated with an uncontaminated laboratory blank before the results may be reported for regulatory compliance purposes.

### 9.4 Ongoing Precision and Recovery

**9.4.1** Prepare an ongoing precision and recovery (OPR) solution identical to the IPR solution described in Section 9.2.2.1.

**9.4.2** An aliquot of the OPR solution must be analyzed with each sample batch (samples started through the sample preparation process (Section 11) on the same 12-hour shift, to a maximum of 10 samples).

**9.4.3** Compute the percent recovery of total solids in the OPR sample.

**9.4.4** Compare the results to the limits for ongoing recovery in Table 2 (to be determined in validation study). If the results meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, the recovery of total solids falls outside of the range given, the analytical processes are not being performed properly. Correct the problem, reprepare the sample batch, and repeat the OPR test. All samples must be associated with an OPR analysis that passes acceptance criteria before the sample results can be reported for regulatory compliance purposes.

**9.4.5** Add results that pass the specifications in Section 9.4.4 to IPR and previous OPR data. Update QC charts to form a graphic representation of continued laboratory performance. Develop a statement of laboratory accuracy for each analyte by calculating the average percent recovery (R) and the standard deviation of percent recovery (SR). Express the accuracy as a recovery interval from R-2SR to R+2SR. For example, if R=05% and SR=5%, the accuracy is 85-115%.

**9.5** Duplicate analyses

**9.5.1** Ten percent of samples must be analyzed in duplicate. The duplicate analyses must be performed within the same sample batch (samples whose analysis is started within the same 12-hour period, to a maximum of 20 samples).

**9.5.2** The total solids of the duplicate samples must be within 10%.

## **10.0 Calibration and Standardization**

**10.1** Calibrate the analytical balance at 2 mg and 1000 mg using class "S" weights.

**10.2** Calibration shall be within  $\pm 10\%$  (i.e.  $\pm 0.2$  mg) at 2 mg and  $\pm 0.5\%$  (i.e.  $\pm 5$  mg) at 1000 mg. If values are not within these limits, recalibrate the balance.

**10.3** Place a 50 g weight and a 2 g weight on the balance. Verify that the balance reads  $50.002 \pm 10\%$  (i.e.,  $\pm 0.2$  mg).

## **11.0 Procedure**

**11.1** Preparation of evaporating dishes—Heat dishes and watch glasses at  $103^{\circ}\text{C}$  to  $105^{\circ}\text{C}$  for 1 hour in an oven. Cool and store the dried equipment in a desiccator. Weigh each dish and watch glass prior to use (record combined weight as “W<sub>dish</sub>”).

**11.2** Preparation of samples

**11.2.1** Fluid samples—If the sample contains enough moisture to flow readily, stir to homogenize, place a 25 to 50 g sample aliquot on the prepared evaporating dish. If the sample is to be analyzed in duplicate, the mass of the two aliquots may not differ by more than 10%. Spread each sample so that it is evenly distributed over the evaporating dish. Evaporate the samples to dryness on a steam bath. Cover each sample with a watch glass, and weigh (record weight as “W<sub>sample</sub>”).

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**NOTE:** *Weigh wet samples quickly because wet samples tend to lose weight by evaporation. Samples should be weighed immediately after aliquots are prepared.*

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**11.2.2** Solid samples—If the sample consists of discrete pieces of solid material (dewatered sludges, 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 a 25 to 50 g sample aliquot of the pulverized sample on the prepared evaporating dish. If the sample is to be analyzed in duplicate, the mass of the two aliquots may not differ by more than 10%.

Spread each sample so that it is evenly distributed over the evaporating dish. Cover each sample with a watch glass, and weigh (record weight as “ $W_{\text{sample}}$ ”).

- 11.3** Dry the samples at 103°C to 105°C for a minimum of 12 hours, cool to balance temperature in an individual desiccator containing fresh desiccant, and weigh. Heat the residue again for 1 hour, cool it to balance temperature in a desiccator, and weigh. Repeat this heating, cooling, desiccating, and weighing procedure until the weight change is less than 5% or 50 mg, whichever is less. Record the final weight as “ $W_{\text{total}}$ ”

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**NOTE:** *It is imperative that dried samples be weighed quickly since residues often are very hygroscopic and rapidly absorb moisture from the air. Samples must remain in the desiccator until the analyst is ready to weigh them.*

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## 12.0 Data Analysis and Calculations

- 12.1** Calculate the % solids or the mg solids/kg sludges for total solids (Equation 2).

Equation 2

$$\% \text{ total solids} = \frac{W_{\text{total}} - W_{\text{dish}}}{W_{\text{sample}} - W_{\text{dish}}} \times 100$$

or

$$\frac{\text{mg total solids}}{\text{kg sludge}} = \frac{W_{\text{total}} - W_{\text{dish}}}{W_{\text{sample}} - W_{\text{dish}}} \times 1,000,000$$

Where:

$W_{\text{sample}}$  = Weight of wet sample and dish (mg)

$W_{\text{total}}$  = Weight of dried residue and dish (mg)

$W_{\text{dish}}$  = Weight of dish (mg)

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- 12.2** Sample results should be reported as % solids or mg/kg to three significant figures. Report results below the ML as < the ML, or as required by the permitting authority or in the permit.

## 13.0 Method Performance

- 13.1** Method performance (MDL and quality control acceptance criteria) will be determined during the multi-lab validation of this method.
- 13.2** Total solids duplicate determinations must agree within 10% to be reported for permitting purposes. If duplicate samples do not meet this criteria, the problem must be discovered and the sample must be run again.

## 14.0 Pollution Prevention

- 14.1** Pollution prevention details are given in Section 14 of Method 1683.

## **15.0 Waste Management**

**15.1** Waste management details are given in Section 15 of Method 1683.

## **16.0 References**

**16.1** "Standard Methods for the Examination of Water and Wastewater," 18th ed. and later revisions, American Public Health Association, 1015 15th Street NW, Washington, DC 20005. 1-35: Section 1090 (Safety), 1992.

**16.2** U.S. Environmental Protection Agency, 1992. Control of Pathogens and Vector Attraction in Sewage Sludges. Publ 625/R-92/013. Office of Research and Development, Washington, DC.

## **17.0 Tables, Diagrams, Flowcharts, and Validation Data**

**17.1** Tables containing method requirements for QA/QC will be added after the validation study has been performed.