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METHOD 1684

Total, Fixed, and Volatile Solids in Water, Solids, and 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 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.

Method 1684

Total, Fixed, and Volatile Solids in Water, Solids, and Biosolids

1.0 Scope and Application

1.1 This method is applicable to the determination of total solids and the fixed and volatile fractions in such solid and semisolid samples as soils, sediments, biosolids (municipal sewage sludge), sludge separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.

1.2 This method is for use in the United States Environmental Protection Agency's (EPA's) data gathering and monitoring programs under the Clean Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation, and Liability Act, and the Safe Drinking Water Act.

1.3 Method detection limits (MDLs) and minimum levels (MLs) have not been formally established for this draft method. These values will be determined during the validation studies.

1.4 This method 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 method are met. Requirements for establishing method equivalency are given in Section 9.1.2.

1.5 Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in Section 9.2.

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 residue from Section 2.1 is cooled, weighed, and dried again at 550°C to drive off volatile solids in the sample.

2.3 The total, fixed, and volatile solids are determined by comparing the mass of the sample before and after each drying step.

3.0 Definitions

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

4.0 Interferences

4.1 Sampling, subsampling, and pipetting multi-phase samples may introduce serious errors. 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, pipette with wide-bore pipettes. 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. 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 in the method. 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 it gains weight while in the desiccator. If the sludge is indeed taking on 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. The residues also lose CO_2 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 both total and volatile 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 drying time and temperature to control losses of volatile inorganic salts if these are a problem.

5.0 Safety

5.1 This method does not address all safety issues associated with its use. The toxicity or carcinogenicity of reagents used in this method have not been fully established. 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 these analyses. Additional information on laboratory safety can be found in References 5-7.

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

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.*

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 Muffle furnace–Capable of maintaining a uniform temperature of 550°C throughout the drying chamber.

6.4 Steam bath for evaporation of liquid samples.

6.5 Desiccator–Moisture concentration in the desiccator should be monitored by an instrumental indicator or with a color-indicator desiccant.

6.6 Drying oven–Thermostatically-controlled, capable of maintaining a uniform temperature of 103°C to 105°C throughout the drying chamber.

6.7 Analytical balance–Capable of weighing to 0.1 mg for samples having a mass up to 200 g.

6.8 Reference weights–2 mg, 1000 mg, and 50g class "S" weights.

6.9 Container handling apparatus–Gloves, tongs, or a suitable holder for moving and handling hot containers after drying.

6.10 Sample handling apparatus–Spatulas, spoonulas, funnels, or other equipment for transfer and manipulation of sample.

6.11 Bottles–Glass or plastic bottles of a suitable size for sample collection.

6.12 Rubber gloves (Optional)

6.13 No. 7 Cork borer (Optional)

6.14 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 can be purchased that contains standard fixed and volatile solids, the laboratory may use that standard. The laboratory may also prepare a spiking solution. One possible recipe is given below for a NaCl-KHP solution.

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 16.10. Begin analysis as soon as possible after collection because of the impracticality of preserving the sample. Refrigerate 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 (40 CFR part 136, appendix B) 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 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 must be established for the analyte, 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 ML. Using the procedures in Section 11, analyze these samples for total, fixed, and volatile solids.

9.2.2.2 Using the results of the four analyses, compute the average percent recovery (x) and the standard deviation (s, Equation 1) of the percent recovery for total, fixed, and volatile 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 1. 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. Sample results are also acceptable for regulatory compliance purposes if

they are associated with a blank that contains less than 1/10 the concentration of the analyte(s) of interest in the associated samples.

9.4 Ongoing Precision and Recovery (OPR).

9.4.1 Prepare an 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 preparation batch (samples of the same matrix 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, fixed, and volatile solids in the OPR sample.

9.4.4 For each analyte, compare the results to the limits for ongoing recovery in Table 1. If all analytes meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, the recovery of an analyte falls outside of the range given, the analytical processes are not being performed properly for that analyte. 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=95% and SR=5%, the accuracy is 85-105%.

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).

9.5.2 The results of duplicate samples analyzed for total, fixed, and volatile solids must be within 10% of the solids determination.

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 ± 10% (i.e. ±0.2 mg) at 2 mg and ± 0.5% (i.e. ±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 mg on the balance. Verify that the balance reads 50.002 ± 10 % (i.e. ± 0.2 mg).

11.0 Procedure

11.1 Total Solids

11.1.1 Preparation of evaporating dishes–If volatile solids are to be measured, ignite clean evaporating dishes and watch glasses at 550°C for 1 hour in a muffle furnace. If only total solids are to be measured, heat dishes and watch glasses at 103°C to 105°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_{dish}").

11.1.2 Preparation of samples.

11.1.2.1 Fluid samples–If the sample contains enough moisture to flow readily, stir to homogenize, place a 25 to 50 g sample aliquot on a 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%. Cover each sample with a watch glass, and weigh to the nearest 0.01 g (record weight as "W_{sample}"). Spread each sample so that it is evenly distributed over the evaporating dish. Evaporate the samples to dryness on a steam bath.

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

11.1.2.2 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 a 25 to 50 g aliquot of the pulverized sample on a 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%. Cover each sample with a watch glass, and weigh (record weight as "W_{sample}"). Spread each sample so that it is evenly distributed over the evaporating dish.

11.1.3 Dry the samples at 103°C to 105°C for 12 hours, minimum, cool to balance temperature in an individual desiccator containing fresh desiccant, and weigh.

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 dessicator until the analyst is ready to weigh them.*

11.1.4 Heat the residue 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 4% or 50 mg, whichever is less. Record the final weight as "W_{total}."

11.2 Fixed and volatile solids.

11.2.1 Transfer the evaporating dishes containing the dried residues (Section 11.1.4) to a cool muffle furnace. Heat the furnace to 550°C and ignite it for 2 hours.

NOTE: *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.*

11.2.2 Cool the residue in a desiccator to balance the temperature. Weigh the residues. Repeat igniting (30 min), cooling, desiccating, and weighing steps until the weight change is less than 4% or 50 mg, whichever is less. Record the final weight as "$W_{volatile}$."

12.0 Data Analysis and Calculations

12.1 Calculate the % solids or the mg solids/kg sludge for total solids (Equation 2), fixed solids, (Equation 3), and volatile solids (Equation 4).

Equation 2

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

or

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

Where:

W_{dish} = *Weight of dish (mg)*
W_{sample} = *Weight of wet sample and dish (mg)*
W_{total} = *Weight of dried residue and dish (mg)*

Equation 3

$$\%\ fixed\ solids = \frac{W_{volatile} - W_{dish}}{W_{total} - W_{dish}} * 100$$

or

$$\frac{mg\ fixed\ solids}{kg\ sludge} = \frac{W_{volatile} - W_{dish}}{W_{total} - W_{dish}} * 1{,}000{,}000$$

Where:

W_{dish} = *Weight of dish (mg)*
W_{total} = *Weight of dried residue and dish (mg)*
$W_{volatile}$ = *Weight of residue and dish after ignition (mg)*

Equation 4

$$\%\ volatile\ solids = \frac{W_{total} - W_{volatile}}{W_{total} - W_{dish}} * 100$$

or

$$\frac{mg\ volatile\ solids}{kg\ sludge} = \frac{W_{total} - W_{volatile}}{W_{total} - W_{dish}} * 1{,}000{,}000$$

Where:

W_{dish} = *Weight of dish (mg)*
W_{total} = *Weight of dried residue and dish (mg)*
$W_{volatile}$ = *Weight of residue and dish after ignition (mg)*

12.2 Sample results should be reported as % solids or mg/kg to three significant figures. Report results below the ML as < ML, or as required by the permitting authority or in the permit. Duplicate determinations must agree within 10% of their average.

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, fixed, and volatile 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 over.

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. Numerous opportunities for pollution prevention exist in laboratory operation. 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 be feasibly 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 N.W., Washington D.C. 20036, (202)872-4477.

15.0 Waste Management

15.1 The Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench

operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult "The Waste Management Manual for Laboratory Personnel", available from the American Chemical Society at the address listed in the Section 14.2.

16.0 References

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16.5 "OSHA Safety and Health Standards, General Industry", (29CFR 1910), Occupational Safety and Health Administration, OSHA 2206, revised January, 1976.

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16.7 "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.

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

16.9 "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL-Ci, Cincinnati, OH 45268, EPA-600/4-79-019, March 1979.

16.10 "Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Biosolid," 1992. EPA/625/R-92/013. Office of Research and Development. USEPA.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 Table 1 - Quality Control Acceptance Criteria for Method 1684.

18.0 Definitions

18.1 Analytical batch–The set of samples analyzed at the same time, to a maximum of 10 samples. Each analytical batch of 10 or fewer samples must be accompanied by a laboratory blank (Section 9.3), an ongoing precision and recovery sample (OPR, Section 9.6), and a set of duplicate samples, resulting in a minimum of five analyses (1 sample, 1 blank, 1 OPR, 2 duplicates) and a maximum of 14 analyses.

18.2 Fixed solids–The residue left in the vessel after a sample is ignited (heated to dryness at 550°C).

18.3 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.

18.4 IPR–See initial precision and recovery.

18.5 Laboratory blank (method blank)–An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment and reagents that are used with samples. The laboratory blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

18.6 Laboratory control sample (LCS)–See Ongoing precision and recovery standard (OPR).

18.7 May–This action, activity, or procedural step is neither required nor prohibited.

18.8 May not–This action, activity, or procedural step is prohibited.

18.9 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.

18.10 Must–This action, activity, or procedural step is required.

18.11 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.

18.12 OPR–See Ongoing precision and recovery standard.

18.13 PAR–See Precision and recovery standard.

18.14 Precision and recovery standard–Secondary standard that is diluted and spiked to form the IPR and OPR.

18.15 Quality control sample (QCS):–A sediment sample containing analytes of interest at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

18.16 Reagent Water– Water that should be free of substances that interfere with analytical methods.

18.17 Sediment sample–A fluvial, sand and/or humic sample matrix exposed to a marine, brackish or fresh water environment. It is limited by this method to that portion which may be passed through a number 10 sieve or a 2 mm mesh sieve.

18.18 Shall–This action, activity or procedural step is required.

18.19 Should–This action, activity, or procedural step is suggested but not required.

18.20 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.

18.21 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.

Table 1 - Quality Control Acceptance Criteria for Method 1684[1]

Analyte	MDL	IPR		OPR
		x	s	x
total solids	3 mg/L	85-110%	10% Rsd	80-110%
fixed solids	7 mg/L	75-110%	20% Rsd	70-110%
volatile solids	7 mg/L	75-110%	30% Rsd	70-110%

[1] Performance criteria are initial estimates. These estimates serve as data quality objectives for the single laboratory validation of this method.