# Percent Solids Determination

References: Method **2540G,** Standard Methods For the Examination of Water and Wastewater, APHA 18th edition, 1992.

## 1. Scope and Application

**Matrices:** This method is applicable to samples such as soils, sediments, sludges separated from water and wastewater treatment processes, sludge cakes, and any tissue.

**Definitions:** Refer to Alpha Analytical Quality Manual.

Percent solids are the total amount of solid material remaining in the weighing tin, after sample drying at a defined temperature, expressed as a percent. This measurement is used in the calculation of analytical results, both organic and inorganic, to express these results on a "dry" weight basis.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts and in the interpretation of data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

## 2. Summary of Method

A well-mixed aliquot of solid sample is placed into an aluminum weighing tin. The aliquot dried to a constant weight, generally overnight, in an oven at 103°C - 105°C. The aliquot is then re-weighed, and calculations are applied (Section 11) to determine the percent of solid material.

### 2.1 Method Modifications from Reference

None.

## 3. Reporting Limits

Method Detection Limit (MDL) studies for the determination of quantitation limits, do not apply to this method or SOP.

# 4. Interferences

**4.1** The temperature at which the sample is dried has an important bearing on the results because weight losses due to volatilization of organic matter, water crystallization, gases from heat induced chemical decomposition, as well as weight gains due to oxidation, are dependant on temperature and the time of heating. Oven temperature and time must be recorded at the time the samples are placed into the oven and at the time they are removed from the oven.

**4.2** It is important that all weighing, pre- and post-sample drying, be performed quickly and at ambient temperatures. Sample cooling must take place in a dessicator, to avoid loss or gain of weight with temperature fluctuations and the re-introduction of moisture to the samples. Tongs must be used when handling weighing tins, as oil residue from hands may contribute to the sample weights. Small differences in weights can significantly affect the final calculation.

**4.3** All weights must be recorded on the Top loading balance to the nearest 0.01g or on the Analytical balance to 0.0001g. The balance to be used depends upon the amount of sample being dried. If less than 1g is to be dried, smaller weighing tins, and the Analytical balance, must be used.

**4.4** Decanting Standing Water from Sediments Samples

The practice of decanting standing water from sediments and soil samples is **only performed upon Client Request.** The Project Manager must be consulted prior to initiating the following protocol.

- Samples will be inspected upon arrival by the Sample Custodian for standing water.
- If the samples do not have standing water, or have 0.0 - 0.25 inches, or 0.0 – 1.0 centimeters of standing water, this will be considered to be pore water that has settled out of the sample during shipping. This water will be mixed back into the sample prior to the removal of any aliquot for percent solids determination.
- If the sample has more than 0.0 - 0.25 inches, or 0.0 – 1.0 centimeters of standing water, the Project Manager will be contacted by the Sample Custodian to inspect the samples. The Project Manager will give the instruction to decant the water, or to mix the water into the sample. The Project Manager must make this decision either through prior knowledge of the site and the client requirements, or must contact the client for instructions.
- If instructed to decant the water, all samples will be decanted at this time by the Sample Custodian or designee. An “X” will be marked on the top of the sample jar in indelible ink and a notation that the standing water has been decanted from the sample will made in the Percent Solids logbook. The Project Manager will also note this in the project narrative to the client.
- If instructed to mix the standing water back into the sample, this will be done at the time the aliquot for percent solids is removed, and no further notations are needed.
- At no time are any analysts, other than the Sample Custodian or designee, to decant standing water from samples. If samples are removed from refrigerators or freezers and the analyst notes that there is standing water on the sample, this is to be mixed back into the sample, as this water is to be assumed to be due to further settling of the pore water.

# 5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

**5.1** The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in the centrally located file storage, in the common area outside of the Information Technology (IT) offices.

**5.2** Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting devise and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

**5.3** The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to chemicals must be reduced to the lowest possible level by whatever means available. All standards and reagents shall be prepared in a hood while using the proper PPE.

**5.4** Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is two times greater than the normal working volume. Concentrated solvents, acids or bases present a moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Department Manager immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Department Manager immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

**5.5** Tongs must always be used when removing hot aluminum weighing tins from the oven and transferring them to the dessicator to avoid burning the skin.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

Samples are typically collected in 4oz or 8oz glass jars.

## 6.2 Sample Preservation

No preservatives are used. See Section 6.4.

## 6.3 Sample Shipping

No special sample shipping requirements. Typical shipping procedures may be found in the Sample Receipt and Login SOP (SOP/01-01).

## 6.4 Sample Handling

Samples are stored at 4°C ± 2°C. Sediment and tissue samples are stored at -20°C ± 10°C.

Refer to the Sample Receipt and Login SOP (SOP/01-01) and the Sample Custody SOP (SOP/01-02).

# 7. Equipment and Supplies

**7.1 Analytical balance:** Capable of weighing to the nearest 0.0001g, and Top loading balance capable of weighing to the nearest 0.01g.

The balances used for each weighing (both Top loading and Analytical) for this method are checked daily before use with Class S-1 weights, in the weight range of use, by a designated analyst, or appointed alternate. These daily checks are documented in the "Balance Calibration" logbook. All balances are serviced annually by an outside service technician.

**7.2 Aluminum weighing tins:** "Standard" size from Fisher Scientific (No.: 08-732), or equivalent. "Small" size for weighing less than 1g of material from Perkin-Elmer (No.: N241-1362), or equivalent.

**7.3 Drying oven:** Set at 103°C – 105°C, vented and/or placed under a fume hood if possible.

**7.4 Clean stainless steel spatulas:** One for each sample to be weighed.

**7.5 Dessicator:** With a desiccant containing a color indicator for moisture content.

**7.6 Indelible ink Sharpie**

**7.7 Tongs and tweezers**

# 8. Reagents and Standards

No standards or reagents are required for this method.

# 9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

## 9.1 Blank(s)

Not applicable to this method.

## 9.2 Laboratory Control Sample (LCS)

Not applicable to this method.

## 9.3 Initial Calibration Verification (ICV)

Not applicable to this method.

## 9.4 Continuing Calibration Verification (CCV)

Not applicable to this method.

## 9.5 Matrix Spike

Not applicable to this method.

## 9.6 Laboratory Duplicate

Duplicate analyses are performed 1 per 20 samples, or at least 1 per batch, whichever is more frequent. The RPD between the duplicates must be less than 10%. If the RPD between the duplicates exceeds 10% the analyst shall check the calculations for errors. If no calculation errors are found, repeat the analysis for the batch.

## 9.7 Method-specific Quality Control Samples

None.

## 9.8 Method Sequence

- Samples 1-20
- Sample Duplicate

# 10. Procedure

## 10.1 Equipment Set-up

Samples are obtained from the Sample Custodian. Note removal of the samples in the Internal Tracking COC in the Sample Management office. If samples are removed from the freezer, note the date of removal from, and return to, the freezer, in the freezer logbook for hold time tracking purposes.

**10.1.1** If a sample has been selected by the client for the Duplicate analysis, this sample must be weighed in duplicate. Otherwise, select the sample that appears to have the greatest total sample mass for the duplicate.

**10.1.2** If samples are those that have been selected for re-extraction, the % Solids are **not** reset. The original % Solids data is used throughout the sample set.

## 10.2 Initial Calibration

Not applicable to this method.

## 10.3 Equipment Operation and Sample Processing

### 10.3.1 Sample aliquots:

**NOTE:** If there is ever any question about the amount of sample that can be taken for the percent solid determination, see the Department Supervisor for guidance.

#### 10.3.1.1 Soils, Sediments, Sludges and Tissue (homogenized tissue samples that yield greater than 20g of total weight):

- Mix the sample well. See Section 4.4 regarding Client requests for standing water and sediment samples.
- Use the Top loading balance and the *standard* size aluminum weighing tin.
- Label the tins with the dish number for each sample to be weighed using an indelible ink Sharpie.
- Tare the balance to read zero.
- Place the tin on the balance and allow the balance to stabilize. Record the weight of the empty tin in the “mass of dish” column “A” in the Percent Solids logbook to the nearest 0.01g.
- Aliquot approximately 5-10g of sample into the tin.
- Allow the balance to stabilize and record the weight of the sample plus the tin to the nearest 0.01g, in the “mass of dish & wet sample” column “B” in the Percent Solids logbook.

#### 10.3.1.2 Tissue (homogenized tissue samples that yield between 5-20g of total weight) and other low weight solid samples:

- Mix the sample well. See Section 4.4 regarding Client requests for standing water and sediment samples. If weighing a homogenized tissue, use caution to preserve as much tissue sample as possible for future analyses.
- Use the Top loading balance and the *standard* size aluminum weighing tin.
- Label the tins with the dish number for each sample to be weighed using an indelible ink Sharpie.
- Tare the balance to read zero.
- Place the tin on the balance and allow the balance to stabilize. Record the weight of the empty tin in the “mass of dish” column “A” in the Percent Solids logbook to the nearest 0.01g.
- Aliquot approximately 1-2g of sample into the tin.

- Allow the balance to stabilize and record the weight of the sample plus the tin to the nearest 0.01g, in the “mass of dish & wet sample” column “B” in the Percent Solids logbook.

**10.3.1.3 Tissue (homogenized tissue samples that yield less than 5g of total weight):**

- Carefully mix the homogenate, if possible, using caution to preserve the tissue sample for future analyses.
- Use the Analytical balance and either the *standard* or *small* size aluminum weighing tins.
- Label the tins with the dish number for each sample to be weighed using an indelible ink Sharpie.
- Tare the balance to read zero.
- Place the tin on the balance and allow the balance to stabilize. Record the weight of the empty tin in the “mass of dish” column “A” in the Percent Solids logbook to the nearest 0.0001g.
- Aliquot approximately 200-500mg of sample into the tin.
- Allow the balance to stabilize and record the weight of the sample plus the tin to the nearest 0.0001g, in the “mass of dish & wet sample” column “B” in the Percent Solids logbook.

**10.3.2** Place the tins in the drying oven overnight, until a constant weight is achieved (see Section 10.3.5, 10.3.6). Record the oven temperature when samples are put into the oven.

**10.3.3** Using tongs or tweezers, quickly and carefully, remove the samples from the oven and place them into the dessicator to cool to room temperature. The samples must stay in the dessicator for at least 20 minutes.

**10.3.4** Tare the balance to zero.

**10.3.5** Remove the samples one at a time from the dessicator and place the dried sample, in the tin, on the balance and allow the balance to stabilize. Record the weight to the appropriate decimal place in the “mass of dish & dry sample” column “C” of the Percent Solids logbook. All post-drying weighing must be performed as quickly as possible, preferably within 2 minutes, of removing the samples from the dessicator, to prevent any atmospheric moisture from returning to the dried samples.

**10.3.6** If a constant weight is not achieved, the sample must be placed back into the oven for approximately one hour. Repeat the cooling and desiccating cycle after this hour. This cycle should be repeated until the weight change is less than 4% or 50mg of the previous weight, whichever is less.

## 10.4 Continuing Calibration

Not applicable to this method.

### 10.5 Preventive Maintenance

Ovens are calibrated on an annual basis by an instrument service company. Certificates are kept on file.

Analytical balances are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file. The calibration of the balances is verified on a daily basis and records are kept in a Logbook.

## 11. Data Evaluation, Calculations and Reporting

### 11.1 Calculations

**11.1.1** Go to the Excel spreadsheet labeled "Percent" on the server.

**11.1.2** Transcribe the logbook entries into the correct columns on the spreadsheet template. Save this template as the date the samples were initially weighted (*i.e.,* 081302.xls). The spreadsheet will calculate the % Solid and the RPD for the duplicate analysis, using the following formulas:

$$\textbf{\% Solid} = \frac{\text{Mass of dish \& dry sample "C"} - \text{Mass of dish "A"}}{\text{Mass of dish \& wet sample "B"} - \text{Mass of dish "A"}} \times 100$$

$$\textbf{RPD} = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = result of sample
R2 = result of the duplicate sample

If percent moisture is requested, as opposed to percent solid, apply the following formula:

$$\textbf{\% Moisture} = 100 - \text{\% Solid}$$

**11.1.3** Go to the LIMS and "batch" and "associate" all samples in this analytical batch.

**11.1.4** Enter the results obtained from the Excel spreadsheet.

**11.1.5** Forward the hand recorded raw data from the Percent Solid logbook, the Excel spreadsheet and the LIMS batch printout to the Department Manager for secondary review.

**11.1.6** Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, G-016. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

**11.1.7** Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry in that log. Such logbooks shall be archived so as to remain available for at

least 10 years. All laboratory notebooks must follow the specifications in the Logbook Useage Work Instructions (WI 108-01), and all record keeping and document control practices.

### 11.2 Data Evaluation and Reporting

All results are reportable without qualification if the QC criteria defined in Section 9 are met. If any QC parameters are not met, all associated samples must be evaluated for re-analysis. See Sections 9 for discussion, including corrective action, for any QC outlier.

## 12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to Section 11.2.

## 13. Method Performance

### 13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

MDL/LOD/LOQ Studies are not applicable to this method.

### 13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

IDC/DOC are not applicable to this method.

#### 13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

#### 13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

## 14. Pollution Prevention and Waste Management

The Alpha *Hazardous Waste and Sample Disposal* SOP (G-006), must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

All solid waste generated during this analysis must be stored in satellite containers in the preparation laboratory labeled “Soil/Solid Waste”.

Once the solid waste satellite containers are full, they must be emptied into a 55-gallon drum marked “Soil/Solid Waste”.

# 15. Referenced Documents

Sample Receipt and Login SOP (SOP/01-01)

Sample Custody SOP (SOP/01-02)

Hazardous Waste and Sample Disposal SOP (G-006)

Logbook Useage Work Instructions (WI 108-01)

# 16. Attachments

None.