



*Chemical Analysis and Testing Task
Laboratory Analytical
Procedure*

LAP-001

Procedure Title: Standard Method for Determination
of Total Solids in Biomass

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Laboratory Analytical Procedure #001

1. Introduction

- 1.1 Biomass samples are hygroscopic materials and can contain large and varying amounts of moisture. To be meaningful, the results of chemical analyses of biomass are typically reported on a dry weight basis. The following procedure describes the method used to determine the amount of solids (or moisture) present in a solid biomass sample.
- 1.2 This procedure has been adopted by ASTM as an ASTM Standard Test Method.

2. Scope

- 2.1 This method is intended to determine the amount of total solids remaining after 105°C drying of a solid sample.
- 2.2 All analyses shall be performed according to the guidelines established in the Ethanol Project Quality Assurance Plan (QAP).

3. References

- 3.1 NREL CAT Task Laboratory Analytical Procedure #012, "Standard Test Method for Moisture, Total Solids, and Total Dissolved Solids in Biomass Slurry and Liquid Process Samples."
- 3.2 TAPPI Method T210 om-58. 1991. "Weighing, Sampling and Testing Pulp for Moisture." Technical Association of the Pulp and Paper Industry Standard Methods.
- 3.3 Vinzant, T.B., L. Ponfick, N.J. Nagle, C.I. Ehrman, J.B. Reynolds, and M.E. Himmel. 1994. "SSF Comparison of Selected Woods From Southern Sawmills." Appl. Biochem. Biotechnol. 45/46:611-626.

4. Significance and Use

- 4.1 The total solids content of a biomass sample is the amount of solids remaining after all volatile matter has been removed by heating the sample at 105°C to constant weight. Conversely, the moisture content is a measure of the amount of water (and other components volatilized at 105°C) present in such a sample.

- 4.2 The results of the chemical analyses of biomass samples are typically reported on a 105°C dry weight basis. The total solids content of a sample is used to convert the analytical results obtained on another basis to that of a dry weight basis.

5. Apparatus

- 5.1 Analytical balance, sensitive to 0.1 mg.
- 5.2 Automated infrared moisture analyzer (such as Denver Instrument Company IR-100 or equivalent) or a convection (drying) oven, with temperature control of $105 \pm 3^{\circ}\text{C}$.
- 5.3 Desiccator.
- 5.4 Aluminum foil weighing dishes.

6. ES&H Considerations and Hazards

- 6.1 Follow all applicable NREL Laboratory Specific Hygiene Plan guidelines.

7. Sampling, Test Specimens and Test Units

- 7.1 Test specimens suitable for analysis by this procedure are "as received", air-dried, milled, or extractive-free biomass solids and the solid fraction of samples generated during the pretreatment, fractionation, or fermentation of biomass. If the total solids (or moisture) content of the whole slurry or liquid fraction of these process samples are to be determined, the CAT Task Laboratory Analytical Procedure #012, "Standard Test Method for Moisture, Total Solids, and Total Dissolved Solids in Biomass Slurry and Liquid Process Samples", must be used instead.
- 7.2 The test specimen shall consist of approximately 2 to 10 g of sample obtained in such a manner as to ensure that it is representative of the entire lot of material being tested.
- 7.3 Materials containing a large amount of free sugars or proteins will caramelize or brown under direct infrared heating elements. Total solids in these materials should be done by the Convection Oven Procedure.
- 7.4 This procedure is not suitable for biomass samples that visibly change on heating, such as unwashed acid-pretreated biomass still containing free acid.

8. Convection Oven Procedure

- 8.1 This method involves drying a sample at $105^{\circ}\text{C} \pm 3^{\circ}\text{C}$ in a convection oven. Each sample must be run in replicate (duplicates, at minimum).
- 8.2 Accurately weigh a predried aluminum foil weighing dish to the nearest 0.1 mg. Record this weight.
- 8.3 Thoroughly mix the sample and then weigh out 1 to 5 grams, to the nearest 0.1 mg, into the weighing dish. Record the weight of the sample plus weighing dish.
- 8.4 Place the sample into a convection oven at $105^{\circ}\text{C} \pm 3^{\circ}\text{C}$ and dry to constant weight ($\pm 0.1\%$ change in the amount of moisture present upon one hour of reheating). Typically overnight drying is required for very wet samples.
- 8.5 Remove the sample from the oven and place in a desiccator; cool to room temperature.
- 8.6 Weigh the dish containing the oven-dried sample to the nearest 0.1 mg and record this weight.

9. Infrared Moisture Analyzer Procedure

- 9.1 This method employs an automated infrared moisture analyzer. Each sample should be run in replicate (duplicates, at minimum).
- 9.2 Program the automated moisture analyzer for a standby temperature of 95°C , an analysis temperature of 105°C , and a pre-determined end of analysis criteria of a rate of weight change that does not exceed 0.05% in one minute.
- 9.3 Turn on the infrared heating elements. Once the analysis temperature of 105°C has been reached, allow the instrument to equilibrate at that temperature for 30 minutes.
- 9.4 Place an aluminum foil weighing dish on the balance pan. For wetter samples, it may be useful to place a quartz pad in the weighing dish to help disperse the moisture. Shut the hood of the instrument and wait five minutes to insure that the dish and pad are completely dry before taring the balance.
- 9.5 Quickly transfer 1 to 3 grams of the thoroughly mixed sample to the weighing dish. Spread the sample as evenly as possible over the surface of the weighing dish.

- 9.6 As soon as the instrument balance stabilizes, shut the hood of the instrument and proceed with the analysis, following the instructions in the instrument operation manual.
- 9.7 Once the sample has been dried to constant weight, as determined by the programmed analysis parameters, the analysis will be automatically terminated by the instrument.

10. Calculations

- 10.1 Calculate the percent total solids on a 105°C dry weight basis as follows (the automated moisture analyzer will provide the calculated value as part of the instrument printout):

$$\% \text{ Total solids} = \frac{\text{weight dried sample plus dish} - \text{weight dish}}{\text{weight sample as received}} \times 100$$

- 10.2 If desired, the percent moisture can also be calculated:

$$\% \text{ Moisture} = \left[1 - \frac{(\text{weight dried sample plus dish} - \text{weight dish})}{\text{weight sample as received}} \right] \times 100$$

11. Report

- 11.1 Report the result as percent total solids (or percent moisture) to two decimal places, and cite the basis used in the calculations.
- 11.2 For replicate analyses of the same sample, report the average, standard deviation, and %RPD.

12. Precision and Bias

- 12.1 Convection Oven Procedure: Data obtained by replicate testing of hybrid poplar in one laboratory gave a standard deviation in moisture content of 0.19% and a CV% of 0.20%. Replicate testing of sodium tartrate gave a standard deviation in total solids of 0.21% and a CV% of 0.23%.

- 12.2 Infrared Moisture Analyzer Procedure: Data obtained by replicate testing of hybrid poplar in one laboratory gave a standard deviation in moisture content of 0.20% and a CV% of 0.21%. Replicate testing of sodium tartrate gave a standard deviation in total solids of 0.58% and a CV of 0.60%.
- 12.3 An inherent error in any moisture determination involving drying of the sample is that volatile substances other than water are removed from the sample during drying.

13. Quality Control

- 13.1 *Reported significant figures:* Report the percent total solids (or percent moisture) to two decimal places.
- 13.2 *Replicates:* At minimum, all samples and the method verification standard are to be analyzed in duplicate.
- 13.3 *Blank:* This gravimetric analysis utilizes a balance blank with every batch of samples, consisting of a weighing dish passed through all steps of the procedure. The difference in weight must be less than the equivalent of a 0.5% error.
- 13.4 *Relative percent difference criteria:* For the infrared drying method the maximum %RPD for duplicate analysis of a sample is 4.0%. For the convection oven method the maximum %RPD is 1.1%. If the stated %RPD is exceeded, the sample must be rerun.
- 13.5 *Method verification standard:* A method verification standard must be run in duplicate with every batch. Sodium tartrate is a suitable material for use as a method verification standard, since the moisture content of this material is not greatly affected by its storage conditions. The published "loss on drying" for sodium tartrate is 15.62% (84.38% total solids).
- 13.6 *Calibration verification standard:* Not applicable.
- 13.7 *Sample size:* A minimum of two grams of sample are required for duplicate analyses. If there is insufficient sample, the result will be flagged and the lack of precision will be noted.
- 13.8 *Sample storage:* Samples shall be stored in an airtight container. Process samples and high-moisture-content feedstock samples must be refrigerated. Every effort shall be made to ensure that a representative aliquot is taken for analysis.
- 13.9 *Standard storage:* Not applicable.

- 13.10 *Standard preparation:* Not applicable.
- 13.11 *Definition of a batch:* Any number of samples which are analyzed and recorded together. The maximum size of a batch would be limited by the equipment constraints. A batch cannot be larger than what is practical with the equipment.
- 13.12 *Control charts:* The result of each replicate analysis of the method verification standard is recorded along with the average, %RPD, and a laboratory book/page reference. The average value obtained for each analysis of the method verification standards is to be control charted.
- 13.13 *Other:* Biomass can rapidly gain or lose moisture when in contact with air. During the weighing steps, minimize the amount of time the sample is exposed to the air.