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Determination of Total Solids in Biomass and Total Dissolved Solids in Liquid Process Samples

Laboratory Analytical Procedure (LAP)

Issue Date: 3/31/2008

A. Sluiter, B. Hames, D. Hyman, C. Payne, R. Ruiz, C. Scarlata, J. Sluiter, D. Templeton, and J. Wolfe

Technical Report
NREL/TP-510-42621
Revised March 2008

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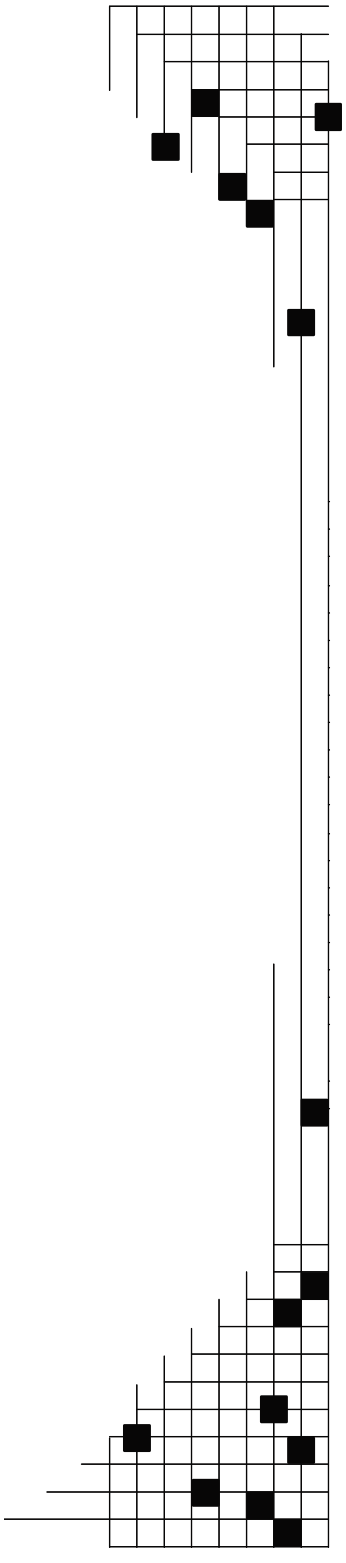

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Procedure Title: Determination of Total Solids in Biomass and Total Dissolved Solids in Liquid Process Samples

Laboratory Analytical Procedure

1. Introduction

1.1 Biomass samples can contain large and varying amounts of moisture, which can change quickly when exposed to air. To be meaningful, the results of chemical analyses of biomass are typically reported on a dry weight basis.

1.2 Portions of this procedure are similar to ASTM E1756-01 and T412 om-02

1.3 The following procedure describes the methods used to determine the amount of solids or moisture present in a solid or slurry biomass sample. It also covers the determination of dissolved solids in a liquor sample. A traditional convection oven drying procedure is covered as well as solids determination using an automatic infrared moisture analyzer. The convection oven method is recommended for liquor samples.

2. Scope

2.1 This procedure is intended to determine the amount of total solids remaining after 105°C drying of a biomass sample.

2.2 All analyses should be performed in accordance with an appropriate laboratory specific Quality Assurance Plan (QAP).

3. Terminology

3.1 *Oven dry weight (ODW)*- the weight of biomass mathematically corrected for the amount of moisture present in the sample at the time of weighing

3.2 *Total solids*- the amount of solids remaining after heating the sample at 105ºC 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

3.3 *Total dissolved solids*- the amount of residue remaining from a 0.2 μm filtered liquor sample after heating the sample at 105ºC to constant weight.

3.4 *Prepared biomass*- biomass prepared according to LAP "Preparation of Samples for Biomass Compositional Analysis".

3.5 *Pretreated biomass*- biomass that has been chemically or thermally altered, possibly changing the structural composition

3.6 *Slurry*- the combined liquid and solid material resulting from biomass pretreatment

3.7 *Liquor*- the liquid fraction of a biomass slurry

4. Significance and Use

4.1 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 an as-received basis to that of an oven dry weight basis.

5. Interferences

5.1 This procedure is not suitable for biomass samples that chemically change upon heating, such as acidic or alkaline biomass samples.

6. Apparatus

6.1 Apparatus required for oven drying method:
6.1.1 Convection drying oven, with temperature control of 105 ± 3°C
6.1.2 Analytical balance, accurate to 0.1 mg
6.1.3 Desiccator containing dessicant
6.2 Apparatus required for moisture analyzer method:
6.2.1 Automated infrared moisture analyzer
6.2.2 Convection drying oven, with temperature control of 105 ± 3°C, optional

7. Reagents and materials

7.1 Reagents
7.1.1 None
7.2 Materials
7.2.1 Aluminum pans, made to fit infrared moisture analyzer if necessary
7.2.2 Glass fiber pads for liquor samples, optional
7.2.3 0.2 μm pore size filters, either large syringe filters with syringes or 50 mm filter units, for liquor samples only

8. ES&H Considerations and Hazards

8.1 Follow all applicable NREL chemical handling procedures.
8.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

9. Sampling, Test Specimens and Test Units

9.1 Test specimens suitable for analysis by this procedure are as-received, air dried, milled, or extractive-free biomass solids and the solid fraction of process samples. Slurry and separated liquor samples are also suitable.
9.2 The test specimen size will be dependent on the type of material and shall be obtained in such a manner as to ensure that it is representative of the entire lot of material being tested.
9.3 This procedure is not suitable for biomass samples that chemically change upon heating, such as acidic or alkaline biomass samples.

10. Procedure

Note: Solid samples usually require 0.5 to 2 grams, slurry samples require 2-5 grams, and liquor samples require 10 ml, per duplicate. Liquor samples should be filtered through a 0.2 μm pore size filter prior to analysis. *Convection oven method is recommended for liquor samples.*

10.1 Convection oven method (use either 10.1 *or* 10.2)

10.1.1 Pre-dry aluminum weighing dishes by placing them in a 105 ± 3ºC drying oven for a minimum of four hours. Cool the dishes in a desiccator. Using gloves or tweezers to handle the dishes, weigh a pre-dried dish to the nearest 0.1 mg. (It may be helpful to place a dry glass fiber pad in the bottom of each pan for liquor samples. Include the weight of the pad with the weight of the pan.) Record this weight.

10.1.2 Thoroughly mix the sample and then weigh out an appropriate amount to the nearest 0.1 mg, into the weighing dish. Liquor samples should be passed through a 0.2 µm filter prior to analysis. Record the weight of the sample plus weighing dish. Analyze each sample in duplicate, at minimum.

10.1.3 Place the sample into a convection oven at 105 ± 3^{o}C for a minimum of four hours. Remove the sample from the oven and allow it to cool to room temperature in a desiccator. Weigh the dish containing the oven-dried sample to the nearest 0.1mg and record this weight.

10.1.4 Place the sample back into a convection oven at 105 ± 3^{o}C and dry to constant weight. Constant weight is defined as ± 0.1% change in the weight percent solids upon one hour of re-heating the sample. Overnight drying is usually required for very wet or liquid samples.

10.2 Automatic infrared moisture analyzer method (use either 10.1 *or* 10.2)

10.2.1 Program the automated moisture analyzer for a standby temperature of 70^{o}C, an analysis temperature of 105^{o}C, and an end point of less than 0.05% solids change in one minute.

10.2.2 Turn on the infrared heating elements and allow them to warm up for approximately 20 minutes. Run the instruments once with an unimportant, disposable sample to bring the heating elements to temperature, if necessary.

10.2.3 Pre-dry aluminum weighing dishes by placing them in a 105 ± 3ºC drying oven for a minimum of four hours or running them through the moisture analyzer once without a sample. If pans are dried in the oven, cool them in a desiccator. Using gloves or tweezers, place an aluminum weighing dish on the balance pan, and tare the balance if necessary.

10.2.4 Quickly transfer desired amount of the thoroughly mixed sample to the weighing dish. Spread the sample evenly over the surface of the weighing dish. Analyze each sample induplicate, at minimum.

10.2.5 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.

10.2.6 Once the sample has been dried to constant weight, as determined by the programmed analysis parameters, the analysis will automatically be terminated. Record the percent solids or percent moisture.

11. Calculations

11.1 Calculate the percent total solids, or percent dissolved solids for a liquor sample, on a 105^{o}C dry weight basis as follows (the automated moisture analyzer will provide the calculated value as part of the instrument printout).

$$\%Total\ Solids = \frac{(Weight_{\ dry\ pan\ plus\ dry\ sample} - Weight_{\ dry\ pan})}{weight_{sample\ as\ received}} \times 100$$

$$\%Dissolved\ Solids = \frac{(Weight_{\ dry\ pan\ plus\ dry\ liquor} - Weight_{\ dry\ pan})}{weight_{liquor\ as\ received}} \times 100$$

11.2 If desired, the percent moisture can also be calculated

$$\%Moisture = 100 - \left(\frac{(Weight_{\ dry\ pan\ plus\ dry\ sample} - Weight_{\ dry\ pan})}{weight_{sample\ as\ received}} \times 100 \right)$$

11.3 To report or calculate the relative percent difference (RPD) between two samples, use the following calculation

$$RPD = \left(\frac{(X_1 - X_2)}{X_{mean}} \right) \times 100$$

Where:

X_1 and X_2 = measured values
X_{mean} = the mean of X_1 and X_2

11.4 To report or calculate the root mean square deviation (RMS deviation) or the standard deviation (st dev) of the samples, use the following calculations.
First find the root mean square (RMS), of the sample using

$$RMS = x_m = mean = \sqrt{\left(\frac{\sum_1^n x}{n} \right)^2}$$

Then find the root mean square deviation, or standard deviation, using

$$RMS deviation = \sigma = stdev = \sqrt{\frac{\sum_1^n (x_i - x_m)^2}{n}}$$

Where:

x_m=the root mean square of all x values in the set
n=number of samples in set
x_i=a measured value from the set

12. Report Format

12.1 Report the result as the percent total solids (or percent moisture), and cite the basis used in the calculations.

12.2 For replicate analyses of the same sample, report the average, standard deviation, and %RPD.

13. Precision and Bias

13.1 An inherent error in any moisture determination involving drying of the sample is that volatile substances other than water may be removed from the sample during drying.

14. Quality Control

14.1 Reported Significant Figures or decimal places: Determined by data quality objectives and laboratory specific Quality Assurance Plan, see LAP "Rounding and Significant Figures".

14.2 Replicates: Run all samples and method verification standards, if applicable, in duplicate, at minimum.

14.3 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.

14.4 Relative percent difference criteria: Each sample must reproduce total solids content $\pm$ 0.5 wt %.

14.5 Method verification standard (MVS): A MVS may be run in duplicate with every batch. Sodium tartrate is a suitable material for use as a MVS, since the moisture content of this material is not greatly affected by its storage conditions. The published moisture loss on drying for sodium tartrate is 15.62% (84.38% total solids).

14.6 Sample size: Determined by sample matrix.

14.7 Sample storage: Samples should be stored in an airtight container. Process samples and high-moisture-content feedstock samples must be refrigerated or frozen until ready for use.

14.8 Standard storage: Not applicable

14.9 Standard preparation: Not applicable

14.10 Definition of a batch: Any number of samples analyzed and recorded together. The maximum size of a batch will be limited by equipment constraints

14.11 Control charts: MVS or a QA/QC material should be control charted to verify reproducibility

14.12 Others: 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.

15. Appendices

15.1 None

16. References

16.1 NREL Laboratory Analytical Procedure, "Determination of Moisture, Total Solids, and Total Dissolved Solids in Biomass Slurry and Liquid Process Samples."

16.2 NREL BAT Team Laboratory Analytical Procedure #001, "Standard Test Method for Determination of Total Solids in Corn Stover."

16.3 TAPPI Method T412 om-02. 2002. "Moisture in Pulp, Paper and Paperboard." Test methods of the Technical Association of the Pulp and Paper Industry 2002-2003.

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