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Standard Practice for
Preparation of Biomass for Compositional Analysis[1]

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1. Scope

1.1 This practice covers a reproducible way to convert hardwoods, softwoods, herbaceous materials (such as switchgrass and sericea), agricultural residues (such as corn stover, wheat straw, and bagasse), wastepaper (such as office waste, boxboard, and newsprint), feedstocks pretreated to improve suitability for fermentation and fermentation residues into a uniform material suitable for compositional analysis.

1.2 Milling and sieving actions both produce large amounts of dust. This dust can be a nuisance hazard and irritant. Use appropriate respiratory protection as needed. If excessive amounts of dust are allowed to become airborne a potential explosion hazard is possible. Provide appropriate dust control measures as needed.

1.3 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are for information only.

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *ambient conditions*—a temperature of 20 to 30°C (68 to 85°F), less than 50 % relative humidity.

3.1.2 *prepared biomass*—biomass that has been prepared according to this practice.

4. Significance and Use

4.1 *Preparation Method A*—Method suitable for the preparation of large quantities (>20 g) of field collected samples into a form appropriate for compositional analysis. Woody samples must first be available as chips of a nominal 5 by 5 by 0.6 cm (2 by 2 by ¼ in.) or less and twigs not exceeding 0.6 cm (¼ in.) diameter. Herbaceous materials may be processed as whole straw. It is recommended that wastepaper should be shredded into pieces less then 1 cm (½ in.) wide. Furthermore, it is recommended that twigs, straw and wastepaper should not exceed 61 cm (24 in.) in length to facilitate handling.

4.2 *Preparation Methods B and C*—Test methods are suitable for very moist feedstocks, samples that would not be stable during prolonged exposure to ambient conditions, or for drying materials when room conditions deviate from the ambient conditions described in 3.1.1. These test methods are also suitable for handling small samples of biomass (<20 g). The drying step is done in a convection oven at 45°C (Test Method B) or by lyophilization (Test Method C).

4.3 This practice is not intended for materials that will already pass through a 20 mesh sieve or cannot be dried by the described methods to a total solids content of greater then 85 %, based on an oven dried weight.

4.4 This practice will separate the milled material into two fractions, a −20/+80 mesh fraction and a −80 mesh fraction.

4.4.1 Extraneous inorganic materials will accumulate in the -80 mesh fraction and it should be analyzed independently from the -20/+80 mesh fraction. Weighted results from the two fractions can then be combined to obtain results for materials on an "as received" basis.

NOTE 1—During analysis, the very fine consistency of the -80 mesh fraction may cause problems in filtering operations and should be handled appropriately.

5. Apparatus

5.1 *Balance,* sensitive to 0.1 g.

5.2 *Riffle Sampler with Pans*—A manual sample divider that splits the milled biomass into a number of alternate elements. Riffle divisions should be in the range from 6.4 mm to 12.7 mm (¼ to ½ in.) with at least twenty-four riffle openings across the

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[1] This practice is under the jurisdiction of ASTM Committee E48 on Bioenergy and Industrial Chemicals from Biomass and is the direct responsibility of Subcommittee E48.05 on Biomass Conversion.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

top. The feed chute and riffles should have a slope of at least 60°. Three pans are needed, one to pour the sample into the riffler, and two to collect the two subsamples.

5.3 *Sieve Set,* No. 20 (850 µm), No. 80 (180 µm) stackable sieves with lid and bottom pan. Sieves and bottom pan should be 8.9 cm (3½ in.) in height. Sieves conform to Specification E11.

5.4 *Sieve Shaker,* provides motion in both horizontal and vertical axes.

5.5 *Knife Mill,* for grinding samples.

NOTE 2—A Wiley Mill, size No. 4 with a 2-mm screen, is suitable for samples >20 g, and the intermediate model, with 1-mm screen, is suitable for samples <20 g that will not be sieved.

5.6 *Drying Oven,* 45± 3° (Test Method B only).

5.7 *Freeze-Drier*—System with vacuum chamber and pump capable of maintaining a pressure of <1 torr and a cold finger in the chamber capable of maintaining a temperature of −50°C (Test Method C only).

6. Preparation: Test Method A

6.1 This test method is suitable for larger quantities (>20 g) of biomass as described in 4.1.

6.2 The raw biomass material should be spread out on a suitable surface to air dry prior to any milling. Do not pile the material deeper then 15 cm. Turn the material at least daily to ensure even drying and minimize molding of material that may contain significant amounts of moisture. The material is considered dried when the change in weight is less than 1 % in 24 h.

6.3 The air-dried material is fed into the knife-mill and is milled to pass through a 2 mm screen in the bottom of the mill. Milled wastepaper does not need to be sieved and can be used directly for composition analysis. Milled biomass materials should be sieved as follows.

6.4 The sieving is set up by stacking the sieves in the following order, starting at the bottom: start with the bottom pan, next stack the 80 mesh sieve, followed by the 20 mesh sieve. Milled material is placed no more then 7 cm deep in the 20 mesh sieve.

6.5 Place the cover on the sieve stack and secure the stack in the sieve shaker.

6.6 The sieves need to be shaken for 15 ± 1 min. At the end of the time period remove the sieves. The fraction retained on the 20 mesh sieve (+20 mesh fraction) should be reprocessed beginning at step 6.3. The fraction retained on the 80 mesh sieve (−20/+80 mesh fraction) should be retained for compositional analysis. The material in the bottom pan is the fines (−80 mesh). Retain this material for ash analysis.

6.7 Repeat 6.3 – 6.6 until all of the milled material will pass through the 20 mesh sieve. If necessary, combine all of the -20/+80 mesh batches. If necessary, combine all of -80 mesh batches. Weigh the combined −20/+80 mesh fraction and the combined fines to the nearest 0.1 g. Record the −20/+80 mesh fraction weight as $Wt_{20/80}$ and the fines fraction weight as Wt_{80}.

6.8 If multiple sieved samples were combined they must be uniformly blended back together into a single sample. Pour the −20/+80 mesh fraction into the riffle sampler, and then recombine the two subdivided samples in the bottom pans back together. Repeat this division and recombination an additional three times. To correctly use the riffle sampler, the sample must be poured evenly onto all the riffle openings at the same time. A pan, as wide as the riffle opening, should be used. Pour the sample evenly off the entire side of the pan and not from the end or the corner, nor from a container such as a jar.

6.9 If the total sample needs to be subdivided into smaller samples, use the riffler at this time to divide the main sample.

6.10 If the prepared sample is not analyzed immediately after sieving and riffling, the sample should be stored in an air-tight container or sealable polyethylene bag and kept at −20°C until needed.

7. Report: Preparation Method A

7.1 Calculate the percent of each fraction in the original, whole biomass:

$$fraction_{20/80},\ \% = (Wt_{20/80} \times 100\,\%)/(Wt_{20/80} + Wt_{80}) \qquad (1)$$

where:

$Wt_{20/80}$ = weight of −20/+80 mesh fraction, g, and
Wt_{80} = weight of fines fraction, g.

$$fraction_{80},\ \% = 100\,\% - fraction_{20/80}\,\% \qquad (2)$$

7.2 The mass fraction is used to weigh analytical results when the two fractions differ in composition, but the results are to be reported on the original, whole biomass.

8. Procedure: Preparation Method B

8.1 This test method is suitable for very wet biomass that is at risk for mold growth during drying, for wet pretreated biomass or fermentation residues (sludges) that might degrade if allowed to stand for prolonged periods, or for drying biomass when the prevailing conditions do not meet the ambient conditions defined in 3.1.1.

8.2 Dry a suitable container to hold the biomass at 45 ± 3°C for a minimum of 3 h. Remove the container, place the container in a dessicator and allow to cool to room temperature. Weigh the container to the nearest 0.1 g and record this weight as W_t.

8.3 Place the biomass material into the dried container. Do not pile the material deeper than 1 cm. Weigh the container and biomass to the nearest 0.1 g and record this weight as W_i.

8.4 Place the container into a drying oven maintaining the temperature at 45 ± 3°C. Allow the material to dry for 36 to 48 h.

8.5 Remove the container and biomass from the drying oven, place in a desiccator and allow to cool to room temperature. Weigh the container and biomass to the nearest 0.1 g and record this weight as W_f.

8.6 For small quantities (<20 g) containing material that would not pass through a 20 mesh screen, reduce the particle size of the solid residue by knife-milling the entire sample through an intermediate size knife-mill with a 1 mm screen.

8.7 For larger quantities (>20 g) containing material that would not pass through a 20 mesh screen, reduce the particle size for the solids by knife-milling the entire sample and sieve according to 6.3 – 6.7.

8.8 Return the milled sample to the drying oven, maintaining the temperature at 45 ± 3°. Keep the sample in the drying oven at 45 ± 3°C for at least 4 h . Allow the samples to cool to room temperature in a desiccator. Weigh each sample to the nearest 0.1 mg and record this mass. After weighing, return the samples to the drying oven at 45°C for 1 h, cool again in the desiccator, and weigh again. Repeat this step until the mass of the samples varies by less than 1% from the previous weighing.

9. Report: Preparation Method B

9.1 Calculate the percent of the total solids obtained by drying at 45°C:

$$\%T_{45} = (W_f - W_t)/(W_i - W_t) \times 100\% \tag{3}$$

where:

$\%T_{45}$ = percent total solids in sample dried at 45°C,
W_t = tare weight of dried container,
W_i = initial weight of container and sample, and
W_f = final weight of container and sample.

9.2 For larger milled and sieved samples calculate the percent of each fraction in the biomass dried at 45°C according to the equations in 7.1.

9.3 The mass fraction is used to weigh analytical results when the two fractions differ in composition, but the results are to be reported on the original, whole biomass.

10. Procedure: Preparation Method C

10.1 This test method is suitable for very wet biomass that is at risk for mold growth during drying, for wet pretreated biomass or fermentation residues (sludges) that might degrade if allowed to stand for prolonged periods, or for drying biomass when the prevailing conditions do not meet the ambient conditions defined in 3.1.1. This test method is also suitable to handle materials that are heat sensitive and would degrade if subjected to the drying oven in Test Method B.

10.2 Weigh a suitable freeze-drier container to the nearest 0.1 g and record this weight as W_t.

10.3 Place the biomass material into the container. For solid samples do not fill the container more than half full. For flowable materials, that is, fermentation residues, fill the container only with enough material to give a uniform coating of 0.5 cm on the walls of the container when the sample is frozen. Weigh the container and biomass to the nearest 0.1 g and record this weight as W_i.

10.4 Place the container into a dry ice/acetone slush and slowly turn the container (10 r/min) to freeze the material into a uniform layer on the walls of the container.

10.5 Immediately place the container on the freeze-drier and allow the material to dry until all visible traces of ice and frost are gone from the sample. Typically this takes 12 h for small (<20 g) samples, and can extend to more than 96 h for large samples (~250 g).

10.6 Remove the container and biomass from the freeze-drier and allow it to warm to room temperature. Weigh the container and biomass to the nearest 0.1 g and record this weight as W_f.

10.7 For small quantities (<20 g) containing material that would not pass through a 20 mesh screen, reduce the particle size of the solid residue by knife-milling the entire sample through an intermediate size knife-mill with a 1 mm screen.

10.8 For larger quantities (>20 g) containing material that would not pass through a 20 mesh screen, reduce the particle size for the solids by knife-milling the entire sample and sieve according to 6.3 – 6.7.

10.9 Use this prepared sample immediately for compositional analysis.

11. Report: Preparation Method C

11.1 Calculate the percent of the total solids obtained by freeze-drying:

$$\%\,T_{fd} = (W_f - W_t)/(W_i - W_t) \times 100\% \tag{4}$$

where:

$\%\,T_{fd}$ = percent total solids of a freeze-dried sample,
W_t = tare weight of freeze-drier container,
W_i = initial weight of container and sample, and
W_f = final weight of container and sample.

11.2 For larger milled and sieved samples calculate the percent of each fraction in the freeze-dried biomass according to the equations in 7.1.

11.3 The mass fraction is used to weigh analytical results when the two fractions differ in composition, but the results are to be reported on the original, whole biomass.

12. Precision and Bias

12.1 *Repeatability*—Duplicate determinations on splits of the gross sample, by the same operator, using the same sieves, shall duplicate the percent mass fractions within 2 % absolute.

12.2 *Reproducibility*—No information is available on duplicate determinations on splits of the gross sample by different operators.

12.3 *Bias*—Since there is not an appropriate standard reference material, no statement about bias can be made.

13. Keywords

13.1 biomass; fermentation residue; hardwood; herbaceous; softwood; wastepaper

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