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

This standard is issued under the fixed designation E1757; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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), cellulosic feedstocks pretreated to improve suitability for ~~fermentation~~ fermentation, cereal grains, cereal grain fermentation mash, cereal grain fermentation beer, and fermentation residues into a uniform material suitable for compositional analysis. This practice is intended for samples that need to be dried prior to 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~~ values given in parentheses ~~are for information only.~~after SI units are provided for information only and are not considered standard.

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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

2.2 *Other Documents:*

AOAC 935.29 Moisture in Malt - Gravimetric Method (AOAC 2005)[3]

NFTA 2.2.2.5 Dry Matter by Oven Drying for 3 hr at 105 °C[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

3.1.2 *beer, n—*the mash after it has undergone fermentation and has been deemed complete by virtue of the carbohydrates being converted into renewable fuel by enzymes and fermenting organisms.

3.1.3 *mash, n—*a mixture or slurry of water (including recycled water streams) and ground grain.

3.1.4 *prepared ~~biomass~~—biomass, n—*biomass that has been prepared according to this practice.

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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.

[3] Available from AOAC International, 2275 Research Blvd., Suite 300, Rockville, MD 20850-3250, http://www.aoac.org.

[4] Available from National Forage Testing Association (NFTA), PO Box 1470, Stuart, FL 34995, https://www.foragetesting.org/.

3.1.5 *predominantly cellulosic, adj*—feedstock that has an average adjusted cellulosic content of 75 %, measured on a dry mass basis; furthermore, this "adjusted cellulosic content" is the percent of organic (non-ash) material that is cellulose, hemicellulose, or lignin.

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~~ 0.6 cm (2 by 2 by ¼ ~~in.)~~ 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~~ 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~~45 °C (Test Method B) or by lyophilization (Test Method C).

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

4.4 ~~This practice~~ Preparation Method A 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~~−80 mesh fraction and it should be analyzed independently from the ~~-20/+80~~−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~~−80 mesh fraction may cause problems in filtering operations and should be handled appropriately.

4.5 *Preparation Method D*—Method suitable for cereal grains, cereal grain fermentation mash, cereal grain fermentation beer, and cereal grain fermentation residues that are generally stable.

4.6 *Preparation Method E*—Method suitable for cereal grains, cereal grain fermentation mash, cereal grain fermentation beer, and cereal grain fermentation residues that are biologically or enzymatically active.

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 in. to ½ in.) with at least ~~twenty-four~~24 riffle openings across the 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,*~~*Sample Mills,* for grinding samples.

5.5.1 Sample grinders must be capable of grinding samples to a sufficiently fine powder.

5.5.1.1 *Discussion*—Many analytical test methods have specific particle size requirements. For example, when analyzing for starch, a particle size of less than 500 µm on average is typically required.

5.5.2 For materials that are predominantly cellulosic and fermentation residues from predominantly cellulosic materials that have been prepared in accordance with Methods A, B, or C, a knife cutting style mill is recommended.

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

5.5.3 For cereal grains, cereal grain fermentation mash, cereal grain fermentation beer, and cereal grain fermentation residues that have been prepared in accordance with Methods D or E, an impact style mill is recommended.

5.6 *Drying Oven,* ~~45 ± 3° (Test Method B only).~~capable of maintaining temperatures between 45 and 55 °C (Test Methods B and D).

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~~−50 °C (Test ~~Method~~Methods C ~~only).~~and E).

6. ~~Preparation: Test~~Procedure: Preparation 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~~−20/+80 mesh batches. If necessary, combine all of ~~-80~~−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~~−20 °C until needed.

7. Report: Preparation Method A

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

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

where:

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

$$fraction_{80},\,\% = 100\,\% - fraction_{20/80}\,\% \tag{2}$$

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~~3 °C for a minimum of 3 h. Remove the container, place the container in a ~~dessicator~~desiccator, 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.~~3 °C. Allow the material to dry for 36 to ~~48 h.~~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.