~~Designation:D7076–05~~ Designation: D 7076 – 09

Standard Test Method for Measurement of Shives in Retted Flax[1]

This standard is issued under the fixed designation D 7076; 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 test method covers the measurement of shives in retted flax.

~~1.2*Units*—The values stated in either SI units or inch-pounds are to be regarded separately as a standard. The values stated in each system may not be exactly equivalent; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.~~

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *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]

~~D6798Terminology Related to Flax and Linen~~

D 6798 Terminology Relating to Flax and Linen

3. Summary of Test Method

3.1 The sample to be evaluated is to be ground and the resulting mixture placed in the appropriate NIR cell and the spectra taken.

3.2 The data will then be compared to a reference file and the value of shive reported as weight percent.

4. Significance and Use

4.1 Few standards exist to objectively determine flax quality. Shive is the woody core of the stem and has an important effect on quality determination. Shive content will vary depending on the stage of processing and can determine in what products the fiber can be used. Spectroscopic data provide an accurate, precise and rapid determination of the amount of shive in flax fiber.

4.1.1 If there are differences of practical significance between reported test results for two or more laboratories, comparative tests should be performed by those laboratories to determine if there is a statistical bias between them, using competent statistical assistance. As a minimum, test samples that are as homogeneous as possible are drawn from the material from which the disputed test results were obtained, and are randomly assigned in equal numbers to each laboratory. These results from the two laboratories should be compared using a statistical test for unpaired data, a possibility level chosen prior to the testing series. If a bias is found, either its cause must be found and corrected, or future test results for that fiber sample type must be adjusted in consideration of the known bias.

4.2 This test method gives data on shive content of retted flax fiber which can be used as a basis for: *(1)* estimating the net amount of manufacturing fiber obtainable from retted flax fiber; *(2)* along with other measurements, predicting the quality of flax products, particularly their aesthetic properties; *(3)* adjusting processing machinery for maximum efficiency in cleaning; and *(4)* relating shive content to end-product quality and processing efficiency.

5. Apparatus

5.1 *Grinder*—SPEX 8000 mixer mill or equivalent instrument for the initial grinding.

5.2 *NIRSystems Model 6500 Monochrometer* or equivalent instrument—Reference spectra scanned over the range 400 to 2498 nm at 2 nm intervals and stored as log (1/*R*), where *R* is reflectance. Standard 50 mm diameter black minicup with a quartz window is used and equipped with a 15 mm i.d. spacer ring if sample size is limited.

---

[1] This test method is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.17 on Flax and Linen.

~~Current edition approved Jan. 1, 2005. Published February 2005.~~

Current edition approved July 1, 2009. Published August 2009. Originally approved in 2005. Last previous edition approved in 2005 as D7076–05.

[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.

6. Hazards

6.1 When handling or grinding any flax material a breathing mask should be worn.

7. Sampling, Test Specimens, and Test Units

7.1 For acceptable testing, take a lot sample from shipping container as directed in an applicable specification, or as agreed upon between the purchaser and supplier.

7.2 Take measurements at a minimum of five sites within a sample, and three measurements at each site. Means of the three replicates constitute the site reading. For each specimen, report means of the five sites.

7.3 *Sample Handling and Preparation*:

7.3.1 Each specimen to be analyzed should be at least 2 g in weight. Care should be taken not to loose any free shive.

7.3.2 Each 2-g aliquot is to be ground for 3 min in a SPEX 8000 mixer mill. If the grinder cannot hold all 2 g, the aliquots are to be thoroughly mixed after separate grinding.

8. Preparation of Apparatus

8.1 Turn on 6500 and computer and allow enough time for warm up that 11.1 is satisfied.

8.2 Start software.

8.3 Begin scan program running diagnostics checking of signal to noise ratio and wave length accuracy.

8.4 Enter routine scan mode.

9. Calibration and Standardization

9.1 The NIR instrument should be standardized with a calibration set which contains samples with a shive content ranging from 0 to 100 %. This set can be prepared by hand separating fiber and shive, grinding each fraction and preparing blended shive/fiber samples of known composition samples of known weight. A calibration equation will be prepared from these samples through the use of Partial Least Squares (PLS1), Multiple Linear Regression (MLR) or another suitable statistical procedure. These are standard chemometric algorithms which will be part of the instrument software package obtained from the manufacturer.

9.2 To verify or to account for a difference in particle size produced by a second grinder, a second set of standard samples will be run which has been ground using a grinder to provide a uniform particle size. These data will be plotted and a slope/bias correction to the spectral data obtained to account for differences in particle size produced by the grinder.

9.3 Alternatively the calibration file from the USDA instrument can be transferred to the host instrument. This is accomplished by using a set of standardization samples obtained from the manufacturer (Foss in this case) and scanning them on both instruments. A standardization file is built with the standardization routines in the instrument software and applied to the calibration file. This file becomes the calibration for the host instrument and a deterministic model developed as described in 9.1.[3]

10. Conditioning

10.1 Do not precondition the test sample.

10.2 Bring the laboratory sample from prevailing atmosphere to approximate moisture equilibrium with the air of the room in which the test will be preformed by exposing the sample at least 12 h.

11. Procedure

11.1 Perform routine analysis and diagnostics for NIRSystems model 6500 monochrometer.[4]

11.2 Clean quartz window with lens tissue to remove dust and streaks.

11.3 *Packing the Sample Cell*:

11.3.1 Mix the specimen thoroughly.

11.3.2 Using a spatula carefully remove a small amount of the material from the sample bottle and gently place in the cell (5 cm o.d.) or the spacer ring (15 mm i.d.) for specimens less that 2 g, until a small mound covers the ring opening. Do not pack or shake the ground mixture.

11.3.3 Place a white foam board (3 mm thick, previously cut to fit) into the loaded cell.

11.3.4 Label specimen number on the back of the foam board.

11.4 *Scanning the Sample*:

11.4.1 Load scan program appropriate equation file (.eqa).

11.4.2 Scan using the spinning cell attachment with quartz window.

11.4.3 Place the loaded cell in the spinning cell apparatus.

11.4.4 Set instrument to scan 16 reps of internal standard before and after each sample (total sample scan time is about 1 min).

11.4.5 The spectrum of each specimen has reflectance data (log 1/R) for every 2 nm from 400 to 2498 nm (1050 points).

11.4.6 Remove loaded cell from apparatus.

[3] Shenk, J. S., and Westerhaus, M. O., *Crop Sci*., 31, 1991, p. 469.

[4] Available from NIRSystems Inc., Silver Springs, MD, USA.