Designation: D7076 – 10 (Reapproved 2021)ε1

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

This standard is issued under the fixed designation D7076; 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.

ε[1] NOTE—Research report information was added to Section 14 editorially in August 2021.

1. Scope

1.1 This test method covers the measurement of shives in retted flax.

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

1.4 *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]

D123 Terminology Relating to Textiles

D6798 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. Terminology

4.1 For all terminology related to Flax, see Terminology D6798.

4.2 For definitions of all other textile terminology, see Terminology D123.

5. Significance and Use

5.1 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.

5.1.1 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.

5.2 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.

6. Apparatus

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

6.2 *NIRSystems Model 6500 Monochrometer* or equivalent instrument—Reference spectra scanned over the range 400 nm 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

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

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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.

quartz window is used and equipped with a 15 mm i.d. spacer ring if sample size is limited.

7. Hazards

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

8. Sampling, Test Specimens, and Test Units

8.1 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.

8.2 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.

8.3 *Sample Handling and Preparation:*

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

8.3.2 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.

9. Preparation of Apparatus

9.1 Turn on 6500 and computer and allow enough time for warm up that 12.1 is satisfied.

9.2 Start software.

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

9.4 Enter routine scan mode.

10. Calibration and Standardization

10.1 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.

10.2 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.

10.3 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 10.1.[3]

11. Conditioning

11.1 Do not precondition the test sample.

11.2 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.

12. Procedure

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

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

12.3 *Packing the Sample Cell:*

12.3.1 Mix the specimen thoroughly.

12.3.2 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.

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

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

12.4 *Scanning the Sample:*

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

12.4.2 Scan using the spinning cell attachment with quartz window.

12.4.3 Place the loaded cell in the spinning cell apparatus.

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

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

12.4.6 Remove loaded cell from apparatus.

12.4.7 Using a thin spatula, remove the foam board and carefully transfer the specimen to the original container.

12.5 Vacuum the cell and spacer to remove dust and clean the quartz window with lens tissue.

12.6 Steps 12.3-12.5 are repeated three times. Shive value will be displayed after each scan.

13. Report

13.1 State the calibration method used.

13.2 Report the following:

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

[4] The sole source of supply of the apparatus known to the committee at this time is NIRSystems Inc., Silver Springs, MD, USA. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.