**Designation:** ~~D7076 – 10~~ <u>D7076 – 10 (Reapproved 2015)<sup>ε1</sup></u>

# 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.

ε1 NOTE—The terminology section was added editorially in April 2015.

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

## 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 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.

## 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 11.112.1 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

**TABLE 1 Summary of Precision and Bias Analysis for Measurement of Shive Content (%) in Retted Flax**

| Sample | Average | Repeatability Standard Deviation $s_r$ | Reproducibility Standard Deviation $\bar{x}$ $s_R$ | Repeatability Limit r | Reproducibility Limit R |
|---|---|---|---|---|---|
| 1 | 0.15 | 0.5618 | 1.3999 | 1.5731 | 3.9198 |
| 2 | 10.88 | 0.7949 | 1.4153 | 2.2257 | 3.9630 |
| 3 | 20.14 | 0.6897 | 1.1416 | 1.9312 | 3.1965 |
| 4 | 31.13 | 0.2448 | 1.9018 | 0.6854 | 5.3251 |
| 5 | 40.73 | 0.6555 | 2.0239 | 1.8353 | 5.6668 |
| 6 | 51.37 | 0.5044 | 2.4763 | 1.4123 | 6.9336 |
| 7 | 60.31 | 0.4011 | 1.3269 | 1.1232 | 3.7153 |
| 8 | 69.99 | 0.3623 | 2.7942 | 1.0146 | 7.8236 |
| 9 | 79.77 | 0.5672 | 2.8398 | 1.5883 | 7.9514 |
| 10 | 89.41 | 1.3213 | 4.0920 | 3.6996 | 11.4576 |
| 11 | 98.57 | 0.5887 | 3.4107 | 1.6484 | 9.5499 |
| 12 | 19.21 | 0.3332 | 0.8894 | 0.9329 | 2.4903 |
| 13 | 27.15 | 0.5076 | 0.7963 | 1.4212 | 2.2295 |
| 14 | 35.36 | 0.6395 | 0.9469 | 1.7906 | 2.6513 |
| 15 | 51.78 | 0.4892 | 0.8152 | 1.3697 | 2.2825 |
| 16 | 66.07 | 0.5855 | 1.9470 | 1.6394 | 5.4517 |
| 17 | 34.90 | 0.2710 | 0.8698 | 0.7587 | 2.4355 |
| 18 | 23.06 | 0.6322 | 1.5068 | 1.7702 | 4.2191 |
| 19 | 38.02 | 1.7109 | 8.6843 | 4.7906 | 24.3159 |
| 20 | 18.53 | 1.9017 | 8.1436 | 5.3248 | 22.8022 |
| 21 | 49.23 | 2.0655 | 10.6839 | 5.7834 | 29.9150 |
| 22 | 9.26 | 2.8163 | 11.3705 | 7.8856 | 31.8375 |
| 23 | 2.24 | 2.2125 | 12.5514 | 6.1951 | 35.1440 |
| 24 | 13.32 | 4.5512 | 9.2063 | 12.7434 | 25.7777 |
| 25 | 7.31 | 2.4626 | 4.1993 | 6.8954 | 11.7580 |
| 26 | 3.77 | 1.4387 | 4.1103 | 4.0283 | 11.5089 |
| 27 | 45.09 | 3.6865 | 7.8500 | 10.3221 | 21.9801 |
| 28 | 39.36 | 4.8082 | 9.0834 | 13.4629 | 25.4335 |