Designation: D7047 – 15$^{\varepsilon 1}$

Standard Test Method for Swell Volume of Plantago Insularis (Ovata, Psyllium)[1]

This standard is issued under the fixed designation D7047; 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—Editorially corrected referencing in 11.1 in August 2015.

1. Scope

1.1 Quantitative test method to determine the swell volume of plantago insularis (Ovata, Psyllium).

1.2 The purpose of this test method is to provide a means of evaluating the swell volume, millilitres per gram, of psyllium hydrophilic mucilloid.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are provided for information only.

1.4 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.4.1 For purposes of comparing, a measured or calculated value(s) with specified limits, the measured or calculated value(s) shall be rounded to the nearest decimal or significant digits in the specified limits.

1.4.2 The procedures used to specify how data are collected/recorded or calculated, in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analysis methods for engineering design.

1.5 *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]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1193 Specification for Reagent Water

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D6026 Practice for Using Significant Digits in Geotechnical Data

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 For common definitions of technical terms in this standard, refer to Terminology D653.

4. Summary of Test Method

4.1 Psyllium substrate is saturated with simulated intestinal fluid and the swell volume recorded after 24 h.

5. Significance and Use

5.1 The meaning of the test is related to the manufacturing and end use of the material, to determine characteristics of products.

5.2 The volume of swell reflects the amount of hydrophilic mucilloid present in psyllium.

5.3 A manufacturer of raw psyllium will base the grade of psyllium produced on multiple properties of which swell volume is one.

[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.25 on Erosion and Sediment Control Technology.

Current edition approved June 1, 2015. Published July 2015. Originally approved in 2004. Last previous edition approved in 2012 as D7047–12. DOI: 10.1520/D7047-15.

[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.

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5.4 The higher the grade of psyllium the higher the swell volume, thus a greater percent of mucilloid present. For the erosion control industry, the higher the swell volume of the psyllium the greater it's bonding strength and relative performance.

5.5 Erosion control contractors and those writing erosion control specifications will use this test method to evaluate the grade of psyllium being used as a hydraulically applied erosion control product. The swell volume will help determine the application rate of psyllium needed to meet the erosion control performance criteria.

6. Apparatus

6.1 *Electronic Balance, as defined in Guide D4753,* with a minimum capacity of 100 g and minimum readability of 0.001 g.

6.2 *Graduated 250 mL Cylinder,* with stopper.

6.3 *1000 mL Flask.*

6.4 *60 Minute Timer,* graduated in one minute intervals.

6.5 *pH Meter,* calibrated to manufacturer's specifications.

7. Reagents

7.1 Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.3 Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type IV.

7.3 Monobasic potassium phosphate (KH_2PO_4).

7.4 Sodium hydroxide (NaOH).

8. Sampling

8.1 For a commercially available pre-blended and quality controlled product, separate 30 g of psyllium sample from an undamaged bag, one sample per blended lot.

8.2 For unblended commercially prepackaged material, separate a 30 g sample from an undamaged bag by taking one third from the top of the bag, one third from the middle of the bag, and one third from the bottom of the bag

9. Procedure

9.1 *Preparation of Simulated Intestinal Fluid TS (Test Solution):*

9.1.1 Add 900 mL of reagent water to a 1000-mL graduated flask.

9.1.2 Add 6.8 g of monobasic potassium phosphate.

9.1.3 Add 0.896 g of sodium hydroxide.

9.1.4 Adjust the pH to 7.5 ± 0.1 with a 0.5 M premixed sodium hydroxide solution.

9.1.5 Adjust the volume with reagent water.

9.2 *Swell Volume:*

9.2.1 Transfer 125 mL of simulated intestinal fluid TS to a 250-mL graduated cylinder.

9.2.2 Add 1.75 g of psyllium substrate, stopper and shake until a uniform suspension is formed. Measure and record the amount of psyllium substrate added to the nearest 0.01 g.

9.2.3 Dilute the fluid to 250-mL with simulated intestinal fluid.

9.2.4 Set the timer for 30 minutes, stopper and shake the graduated cylinder for 1 minute every 30 minutes for 8 hours.

9.2.5 Allow the gel to settle for 16 additional hours (24 hours total) at room temperature.

9.2.6 Measure and record the volume of gel to the nearest 1 mL.

10. Calculations

10.1 Calculate the swell volume as follows:

$$\text{Sell Volume}(\text{mL/g}) = V/G \quad (1)$$

where:

V = millilitres of gel, and
G = grams of psyllium.

11. Report: Test Data Sheet(s)/Form(s)

11.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.4.

11.2 Record as a minimum the following general information (data):

11.2.1 Sample/specimen identifying information, such as Product Name, Production Lot Number and Quantity, Date of Production and any other product identifying information.

11.3 Record as a minimum the following test specimen data:

11.3.1 The amount of psyillium substrate, 1.75 g.

11.3.2 The volume of the gel to the nearest 1 mL.

11.3.3 Record the swell volume, to the nearest one millilitre per gram (mL/g).

11.3.4 Laboratory performing the test, lab technician performing the test, and date of testing.

12. Precision and Bias[3]

12.1 The precision of this test method is based on an intralaboratory study of ASTM WK34944 – New Standard Test Method for Swell Volume of Psyllium, conducted in 2012. A single laboratory participated in this study, testing four different materials over time. Every "test result" represents an individual determination. The laboratory reported three replicate test results for each material. Except for the use of only one laboratory, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. D18-1021.

12.1.1 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D18-1021. Contact ASTM Customer Service at service@astm.org.

constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.1.1.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

12.1.1.2 Repeatability limits are listed in Table 1.

12.1.2 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

12.1.2.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

TABLE 1 Swell Volume (mL/g)

	Average	Repeatability Standard Deviation	Repeatability Limit
	$\bar{x}$	S_r	r
Sample 1	23.0	0.0	0.0
Sample 2	56.3	0.6	1.6
Sample 3	15.0	0.0	0.0
Sample 4	23.0	0.0	0.0

12.1.2.2 Reproducibility limits cannot be calculated from a single laboratory's results.

12.1.3 The terms repeatability limit and reproducibility limit are used as specified in Practice E177.

12.1.4 Any judgment in accordance with statement 9.1.1 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results essentially guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit as a general guide, and the associated probability of 95 % as only a rough indicator of what can be expected.

12.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

12.3 The precision statement was determined through statistical examination of 12 results, from a single laboratory, on four materials described as:

Sample 1: Moderate Swell Psyllium
Sample 2: High Swell Psyllium
Sample 3: Low Swell Psyllium
Sample 4: Moderate Swell Psyllium

13. Keywords

13.1 erosion control; insularis; mucilloid; ovata; plantago; psyllium; swell

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