Designation: D6738 – 15

Standard Test Method for Precipitated Silica—Volatile Content[1]

This standard is issued under the fixed designation D6738; 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 a procedure to determine the volatile content of precipitated hydrated silicas. These volatiles are generally excess water adsorbed onto the surface of the silica. This test method does not determine water of hydration of the silica.

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

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]

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. Significance and Use

3.1 The volatiles content of a precipitated silica may affect the processing properties of a rubber mixture containing silica and the properties of the final product.

4. Apparatus

4.1 *Oven,* gravity-convection type, capable of maintaining 105 ± 5°C.

4.2 *Ground-glass stoppered low-form weighing bottle,* approximately 30 mm in height by 60 mm in diameter.

4.3 *Analytical Balance,* sensitive to 0.1 mg.

4.4 *Desiccator.*

NOTE 1—It is important that the desiccant is very dry, or moisture may be drawn from the desiccant into the sample during cooling.

5. Procedure

5.1 Weigh a weighing bottle with stopper to the nearest 0.1 mg and record its weight (W_1).

5.2 Add approximately 2 g of the silica sample to the weighing bottle and reweigh to the nearest 0.1 mg (W_2).

5.3 Place the weighing bottle with the silica sample into the 105°C oven for 2 h ± 5 min. Place the stopper nearby the weighing bottle.

5.4 Transfer the weighing bottle containing the silica sample and closed with the stopper from the oven into a desiccator and cool for 2 h or until room temperature.

5.5 Remove the weighing bottle containing the silica sample from the desiccator and reweigh to the nearest 0.1 mg (W_3).

NOTE 2—An aluminum pan may be substituted for the weighing bottle in this test method. The results are systematically lower due to moisture pick-up from the atmosphere. The thermal transfer of glass differs from that of aluminum, so cooling time may be shortened using an aluminum pan.

6. Calculation

6.1 The volatile material is given by the following equation:

$$\% \text{ Volatiles} = [(W_2 - W_3)/(W_2 - W_1)] \times 100 \quad (1)$$

where:

W_1 = mass of weighing bottle, g,
W_2 = mass of weighing bottle and silica sample before heating, g, and
W_3 = mass of weighing bottle and silica sample after heating, g.

7. Report

7.1 Report the following information:

7.1.1 Identification of the sample, and

7.1.2 Result reported to the nearest 0.1 %.

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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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.

TABLE 1 Volatile Content (%)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	S_R	r	R
Silica A	5.36	0.06	0.22	0.16	0.62
Silica B	6.45	0.05	0.24	0.15	0.67

[A] The average of the laboratories' calculated averages.

8. Precision and Bias[3]

8.1 The precision of this test method is based on an interlaboratory study of conducted in 2010. Eleven laboratories tested two types of silica samples. Every "test result" represents an individual determination. Each laboratory was instructed to report four replicate test results for each material. Practice E691 was followed for the design and analysis of the data.

8.1.1 *Repeatability Limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "*r*" value for that material; "*r*" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

8.1.1.1 Repeatability limits are listed in Table 1.

8.1.2 *Reproducibility Limit (R)*—Two test results shall be judged not equivalent if they differ by more than the "*R*" value for that material; "*R*" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

8.1.2.1 Reproducibility limits are listed in Table 1.

8.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

8.1.4 Any judgment in accordance with statements 8.1.1 and 8.1.2 would have an approximate 95 % probability of being correct.

8.2 *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.

8.3 The precision statement was determined through statistical examination of 72 results, from nine laboratories, on two different precipitated silica samples.

9. Keywords

9.1 precipitated hydrated silica; silica; volatile content

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1107.