



Designation: D 6738 – 01

Standard Test Method for Precipitated Silica—Volatile Content¹

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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. Significance and Use

2.1 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.

3. Apparatus

3.1 *Oven*, gravity-convection type, capable of maintaining $105 \pm 5^\circ\text{C}$.

3.2 *Aluminum Pans*, oil free.

3.3 *Analytical Balance*, sensitive to 0.1 mg.

3.4 *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.

4. Procedure

4.1 Weigh an aluminum pan to the nearest 0.1 mg and record its weight (W_1).

4.2 Add approximately 2 g of the silica sample to the pan and reweigh to the nearest 0.1 mg (W_2).

4.3 Place the pan with the silica sample into the 105°C oven for $2\text{ h} \pm 5\text{ min}$.

4.4 Transfer the pan containing the silica sample from the oven into a desiccator and cool for 1 h or until room temperature.

4.5 Remove the pan containing the silica sample from the desiccator and reweigh to the nearest 0.1 mg (W_3).

NOTE 2—A ground-glass stoppered low-form weighing bottle (approximately 30 mm in height by 60 mm in diameter) may be substituted for the aluminum weighing dish in this test method. The sample then is dried in the oven and cooled in the desiccator with the stopper removed, but final weighing would be done with the stopper in the bottle to minimize moisture pick-up from the atmosphere. The thermal transfer of glass differs from that of aluminum, so cooling time may be lengthened using a glass container.

5. Calculation

5.1 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 aluminum pan, g,

W_2 = mass of aluminum pan and silica sample before heating, g, and

W_3 = mass of aluminum pan and silica sample after heating, g.

6. Report

6.1 Report the following information:

6.1.1 Identification of the sample, and

6.1.2 Result reported to the nearest 0.1 %.

7. Precision and Bias

7.1 Precision and bias studies have not been conducted.

8. Keywords

8.1 precipitated hydrated silica; silica; volatile content

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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