



Designation: D6738 – 01 (Reapproved 2006)

Standard Test Method for Precipitated Silica—Volatile Content¹

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. 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.

Current edition approved Oct. 1, 2006. Published November 2006. Originally approved in 2001. Last previous edition approved in 2001 as D6738 – 01. DOI: 10.1520/D6738-01R06.

 D6738 – 01 (2006)

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).