



Designation: D6739 – 20

Standard Test Method for Silica—pH Value¹

This standard is issued under the fixed designation D6739; 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 is used to measure the pH of a 5 % mass per volume (m/V) silica/water suspension or mechanical dispersion and is indicative of the relative acidity or alkalinity of the silica.

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

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

3. Significance and Use

3.1 The pH level of silica, as measured by this test method, is known to affect the vulcanization of some rubber compounds. Refer to Test Method **E70** for a fuller understanding of pH and a more detailed procedure for making pH measurements.

¹ This test method is under the jurisdiction of ASTM Committee **D11** on Rubber and Rubber-like Materials 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.

4. Apparatus

4.1 *pH Meter*, (digital is recommended), having an accuracy of ± 0.05 pH and equipped with a combination electrode.

4.2 *Mortar and Pestle*.

4.3 *Beaker*, glass, 150 cm³ with watch glass.

4.4 *Magnetic Stirring Bar and Stirrer*.

5. Reagents

5.1 *Degassed, Neutral Water*. Boil a container of either distilled or deionized water for 10 min, cover, and allow to cool to room temperature, or purge the water with nitrogen gas for 20 min using a fritted bubbler.

5.2 *Buffer Solutions*, pH of 4.00, 7.00, and 10.00.

6. Procedure

6.1 Pulverize pelleted or granulated silica to a fine powder, using a mortar and pestle.

6.2 Weigh 5 g of silica to the nearest 0.1 g into a 150 cm³ glass beaker.

6.3 Add 100 cm³ of degassed, neutral water.

6.4 Cover the glass beaker with a watch glass and stir the mixture for 5 min at room temperature.

6.5 Standardize the pH meter with two buffer solutions that bracket the expected pH value of the silica. Rinse the electrode with distilled water and wipe clean after each test.

6.6 Place the electrode in the silica/water mixture, continuing to stir with the magnetic stirrer. When a constant pH is obtained, record the pH to the nearest 0.05 pH unit.

6.7 Rinse the electrode with distilled water. Store electrode in accordance with manufacturer's instructions when not in use.

7. Report

7.1 Report the following information:

7.1.1 Identification of the sample, and

7.1.2 Result obtained, reported to the nearest 0.1 unit.

TABLE 1 Precision Parameters for ASTM D6739, Silica—pH Value, (Type 1 Precision)

Material	Period	Units	pH unit	Sr	r	(r)	SR	R	(R)
		Number of Laboratories (M/V) ^A	Mean Level						
Silica A	2010	10(1/1)	6.63	0.011	0.031	0.47	0.052	0.15	2.22
Silica B	2010	10(1/3)	6.75	0.002	0.006	0.09	0.056	0.16	2.35

^A Total number of outliers removed for Mean and Variability.

8. Precision and Bias³

8.1 These precision statements have been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical details.

8.2 A type 1 inter-laboratory precision program was conducted in 2010 using two different types of silica material. Data was received from eleven laboratories. In each laboratory four single tests were performed on each of the two materials on the same day (total of four replicates). A test result is the value obtained from a single determination. Both repeatability and reproducibility represent short-term (daily) testing conditions. Acceptable difference values were not measured.

8.3 In the initial outlier evaluation at the 95 % confidence level, one laboratory was an outlier for mean and variation for both materials. This laboratory's values were so different from those of the other 10 laboratories that they were distorting the outlier evaluations. Therefore, this laboratory's data was removed from the data set and the analysis repeated using the data from the remaining 10 laboratories.

8.4 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from Table 1.

8.5 The results of the precision calculations for this test method are given in Table 1. The evaluation at a 95 % confidence level identified 1 mean outlier for each material, 1 variability outlier for Silica A, and 2 variability outliers for Silica B. The subsequent evaluation at a 98 % confidence level identified no additional mean outliers for either material, no

additional variability outliers for Silica A, and 1 additional variability outlier for Silica B. This information is summarized in the "Number of Laboratories" column of Table 1. With just two materials, the preferred precision cannot be determined.

8.6 *Repeatability*—The **absolute** repeatability limits, *r*, of this test method are as shown in Table 1, for each of the materials as listed in the table. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate action taken.

NOTE 1—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, etc., which generated the two test results.

8.7 *Reproducibility*—The **absolute** reproducibility limits, *R*, of this test method are as shown in Table 1, for each of the materials as listed in the table. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

8.8 *Bias*—Bias is the difference between a test value and a reference value. However, for this test method, bias has not been determined.

9. Keywords

9.1 pH; silica

³ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1108.