Designation: D6739 – 11 (Reapproved 2015)

Standard Test Method for Silica—pH Value[1]

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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 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.

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 cm^3 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 cm^3 glass beaker.

6.3 Add 100 cm^3 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.

8. Precision and Bias[3]

8.1 The precision of this test method is based on an interlaboratory study conducted in 2010. Eleven laboratories

[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.

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

TABLE 1 pH

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	S_R	r	R
Silica A	6.61	0.01	0.07	0.04	0.21
Silica B	6.74	0.01	0.08	0.01	0.22

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

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 80 results, from ten laboratories, on two different precipitated silica samples.

9. Keywords

9.1 pH; silica