Designation: D 5095 – 91 (Reapproved 2002)

Standard Test Method for Determination of the Nonvolatile Content in Silanes, Siloxanes and Silane-Siloxane Blends Used in Masonry Water Repellent Treatments[1]

This standard is issued under the fixed designation D 5095; 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 describes a procedure for the determination of the nonvolatile content (N_s) of silanes, siloxanes, and blended silane-siloxane materials used in masonry water repellent treatments and is applicable to both solvent- and waterborne materials.

1.2 *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 health and safety practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3980 Practice for Interlaboratory Testing of Paint and Related Materials[3]

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[4]

3. Summary of Test Method

3.1 A designated quantity of test material is weighed into a preweighed aluminum dish containing the catalyst solution, mixed, allowed to stand for 60 min at room temperature, and then heated in an oven at 110 ±5°C for 60 min. The nonvolatile content of the test material is calculated by subtracting the solids of the catalyst solution from the total solids by weight of the test solution.

4. Significance and Use

4.1 This test method is used to determine the nonvolatile content of silanes, siloxanes, and silane-siloxane blended materials used in masonry water-repellent treatments. It can be used for the purpose of calculating the volatile organic compound (VOC) content of these materials under specified test conditions.

5. Apparatus

5.1 *Aluminum Dishes*, 58-mm diameter by 18-mm high with a smooth (planar) bottom surface. Precondition the dishes for 30 min in an oven at 110 ±5°C and store in a desiccator prior to use.

5.2 *Forced Draft Oven*, Type IIA or Type IIB as specified by Specification E 145.

5.3 *Syringes*, 1-mL and 5-mL.

5.4 *Analytical Balance*, capable of weighing to 0.1 mg.

6. Reagents

6.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type IV of Specification D 1193.

6.2 *p-Toluenesulfonic Acid*, monohydrate.[5]

6.3 *Alcohol*, technical grade ethanol or isopropanol.

7. Hazards

7.1 In addition to other precautions, provide adequate ventilation, consistent with accepted laboratory practice, to limit the accumulation of solvent vapors.

8. Procedure

8.1 *Catalyst Solution*—Prepare a catalyst solution containing a mixture of 0.5 % *p*-Toluenesulfonic acid in either ethanol or isopropanol. Thoroughly mix the solution. Prepare sufficient catalyst solution to perform all tests.

8.1.1 The nonvolatile content of the test material can be calculated only if the same catalyst solution is used throughout the test. Each time a new batch of catalyst solution is used, its nonvolatile content must be determined.

8.2 Determine the nonvolatile matter, in triplicate, of the catalyst solution as follows:

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.47 on Masonry Treatments.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] Discontinued; see *1998 Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 14.04.

[5] The sole source of supply of the solution of *p*–Toluenesulfonic acid known to the committee at this time is King Industries, Science Road, Norwalk, CT 06852. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

8.2.1 Weigh an aluminum dish to 0.1 mg. Record the weight as W_1.

8.2.2 Using a 5-mL syringe, weigh 3 ±1.0 g, to 0.1 mg, by difference, of the catalyst solution into the preweighed aluminum dish. Record the weight of catalyst solution as W_3.

8.2.3 Heat the aluminum dish containing the catalyst solution in a forced draft oven for 60 min at 110 ±5°C.

8.2.4 Remove the dish from the oven and immediately place in a desiccator. Seal the desiccator and allow the dish to cool to ambient temperature.

8.2.5 Reweigh the dish to 0.1 mg. Record the weight as W_2.

8.2.6 Calculate the nonvolatile matter of the catalyst solution, N_c, in accordance with 9.1.

8.3 Determine the percent nonvolatile content, in triplicate, of the test specimen as follows:

8.3.1 Thoroughly mix the test materials before use.

8.3.2 Weigh an aluminum dish to 0.1 mg. Record the weight as W_4.

8.3.3 Using a 5-mL syringe, weigh 3 ±1.0 g, to 0.1 mg, by difference, of the catalyst solution into the preweighed aluminum dish. Record the weight as W_6.

8.3.4 Using a 1-mL syringe, weigh 1.0 ±0.1 g, to 0.1 mg, by difference, of the test specimen into the weighing dish containing the catalyst solution. Record the weight of the test specimen as S.

8.3.4.1 Weighings must be done quickly to limit weight loss due to volatilization. If there is insufficient moisture present when testing solvent-borne silane materials, it is advisable to add up to 0.3 g of reagent grade water to the dish containing the mixture of catalyst and test solutions.

8.3.5 Gently swirl the dish to mix the materials. Allow the materials to stand at room temperature for 60 min.

8.3.6 Heat the dish containing the mixture of catalyst and test solutions in a forced-draft oven for 60 min at 110 ±5°C.

8.3.7 Remove the dish from the oven and immediately place in a desiccator. Seal the desiccator and allow the dish to cool to ambient temperature.

8.3.8 Reweigh the aluminum dish to 0.1 mg and record the weight as W_5.

8.3.9 Calculate the nonvolatile content, N_s, of the test specimen in accordance with 9.2.

9. Calculation

9.1 Calculate the nonvolatile matter, N_c, in the catalyst solution as follows:

$$N_c = (W_2 - W_1) / W_3 \quad (1)$$

where:

N_c = nonvolatile matter of catalyst solution expressed as a decimal fraction,
W_1 = weight of aluminum dish, g,
W_2 = weight of dish plus catalyst solution after heating, g, and
W_3 = weight of catalyst solution before heating, g.

9.2 Calculate the nonvolatile content, N_s, in the test specimen as follows:

$$N_s = 100 \frac{[(W_5 - W_4) - (W_6)(N_c)]}{S} \quad (2)$$

where:

N_s = nonvolatile content of test specimen, percent,
W_4 = weight of the aluminum dish, g,
W_5 = weight of aluminum dish plus the catalyzed test material after heating, g,
W_6 = weight of catalyst solution used in test specimen before heating, g,
N_c = nonvolatile matter of catalyst solution, decimal fraction, (average of two determinations), and
S = weight of test specimen before heating, g.

10. Report

10.1 Report the following information:

10.1.1 The average values obtained for the nonvolatile content of the catalyst solution, N_c.

10.1.2 The average values obtained for the percent nonvolatile content of the test specimen, N_s.

11. Precision and Bias

11.1 The precision estimated for this test method is based on an interlaboratory study in which one operator in each of five laboratories tested in triplicate on two different days four water repellent materials containing between 14 to 65 % nonvolatiles. The results were analyzed statistically in accordance with Practice D 3980. The intralaboratory standard deviation was found to be 0.283 % absolute with 17 df and the interlaboratory coefficient of variation 7.02 % relative with 16 df. Based on the standard deviation and coefficient of variation, the following criteria should be used for judging at the 95 % confidence level, the acceptability of results.

11.1.1 *Repeatability*—Two results, each the mean of triplicates, obtained by the same operator should be considered suspect if they differ by more than 0.84 % absolute.

11.1.2 *Reproducibility*—Two results, each the mean of triplicates, obtained by operators in different laboratories should be considered suspect if they differ by more than 7.02 % relative.

11.2 *Bias*—Bias has not been established for this test method.

12. Keywords

12.1 masonry water repellents; nonvolatile matter content; silanes; siloxanes