Designation: D 269 – 97 (Reapproved 2002)

Standard Test Method for Insoluble Matter in Rosin and Rosin Derivatives[1]

This standard is issued under the fixed designation D 269; 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 the determination of the amount of insoluble matter in rosin and rosin derivatives as described in Terminology D 804.

1.2 *This standard does not purport to address all of the safety problems, 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:*

D 804 Terminology Relating to Naval Stores Including Tall Oil and Related Products[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

3. Significance and Use

3.1 Rosin, particularly gum and wood rosin, occasionally contains small amounts of contamination such as sand, dirt or bark. Rosin derivatives occasionally contain traces of insoluble material as a result of the raw materials used in their production or they may be generated during the production process. In all instances the presence of such insoluble material should be minimal. This test method describes a rapid and reliable procedure for determining the amount of such insoluble matter. It is based on the knowledge that rosin and most of its derivatives are soluble in numerous organic solvents whereas most common contaminants are not. It is especially useful for internal quality control rather than sales specifications.

4. Apparatus

4.1 *Beaker*, 800 mL.

4.2 *Magnetic Stirring Hot Plate with Polytetrafluoroethylene (PTFE) Stirbar*, or hot plate with manual stirring rod.

4.3 *Precut Stainless Steel Circular Screen*, 325 mesh with 0.0014-in. wire diameter. (44-µm openings) as described in Specification E 11.

4.4 *Two-Piece Filter Apparatus*, appropriate to hold the stainless steel screens without leaking.

4.5 *Analytical Balance*, capable of weighing 0.0001 g.

4.6 *Laboratory Tweezers*.

4.7 *Forced Draft Oven*.

5. Reagents

5.1 *Clean Toluene, Hexane, Mineral Spirits*, or other suitable solvent for the specific material to be checked in, as agreed upon between the customer and the supplier.

6. Procedure

6.1 Rinse the pre-cut screen thoroughly with the solvent to clean it before use.

6.1.1 Dry the clean screen at 105 to 110°C for 30 min, cool in a desiccator, and weigh.

6.1.2 Record the weight of the dry screen to the nearest 0.0001 g.

6.1.3 Place the screen in the filter apparatus and secure it to prevent leakage.

NOTE 1—Always use tweezers when handling the pre-cut screen.

6.2 Weigh 100 ±0.1 g of freshly powdered material to be tested into an 800-mL beaker. Add 150 mL of solvent. Place a PTFE-coated magnetic stir bar into the beaker, and place the beaker on a hot plate. Heat and stir the material until it is completely dissolved. Do not boil the solvent.

6.3 Immediately pour the solution through the screen. Rinse the beaker and filter apparatus three times with additional hot solvent.

6.4 Disassemble the filter apparatus, remove the screen, and place it in a forced draft oven, contaminated side up. Dry the screen to constant weight at 105 to 110°C. (1 h is usually sufficient), cool in a desiccator, and weigh. Record the weight of the dry contaminated screen to the nearest 0.0001 g.

7. Calculation

$$\text{Percent insolubles} = \frac{\text{weight contaminated screen} - \text{weight clean screen.}}{\text{weight of sample}} \times 100$$

8. Report

8.1 Report the percent insoluble matter to the nearest 0.01 %.

[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint, Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.34 on Naval Stores.

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

9. Precision and Bias

9.1 It is not practical to measure the precision and bias of the procedure in this test method because this test method is primarily used for internal quality control purposes rather than for customer specification purposes.

10. Keywords

10.1 insoluble; rosin; rosin derivatives