



Standard Test Method for Rubber Compounding Materials: 2-Benzothiazyl Sulfenamide Accelerators—Insolubles¹

This standard is issued under the fixed designation D4934; 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 general procedure for the determination of insoluble impurities of sulfenamides in suitable organic solvents.

1.2 The values stated in SI units are to be regarded as the standard. The values given 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. Referenced Documents

2.1 *ASTM Standards:*²

[D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries](#)

3. Summary of Test Method

3.1 A specimen of sulfenamide is dissolved in a prescribed solvent, stirred, and filtered through a crucible. The insoluble content is calculated from the amount of residue.

4. Significance and Use

4.1 Sulfenamides can degrade in chemical purity and functional performance, usually characterized by a drop in assay, a release of free amine, and an increase in insolubles. This test method may be used as an indication of such degradation.

4.2 Since MBTS (mercaptobenzothiazole disulfide) is a primary degradation product of sulfenamides, the determination of MBTS is a means of assessing possible degradation of

sulfenamides. Insolubles are a means of mercaptobenzothiazyl disulfide (MBTS) content of the sulfenamide; MBTS is a primary degradation product of sulfenamides. Amine salts of mercaptobenzothiazole (MBT) may also be insoluble. However, certain soluble species may also be generated during sulfenamide degradation. Consequently, insolubles are not an absolute measure of purity and can actually decrease with sulfenamide degradation.

5. Apparatus

5.1 *Erlenmeyer Flask*, 300 cm³.

5.2 *Sintered Glass Crucible*, G4.

5.3 *Measuring Cylinder*, 250 cm³.

5.4 *Magnetic Stirrer*.

5.5 *Watch Glass*.

5.6 *Vacuum Flask*.

5.7 *Explosion-proof, Vented Air Circulating Oven*, capable of temperature regulation of $70 \pm 2^\circ\text{C}$.

5.8 *Balance*, analytical, with a sensitivity of ± 0.01 g.

5.9 *Washing Bottle*.

5.10 *Sieve*, 30 Mesh (U.S. Standard), or equivalent (for example, 0.6 mm).

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Methanol*, analytical reagent.

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.11 on Chemical Analysis.

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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 *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.