



Standard Test Method for Rubber Chemicals—Free 2-Mercaptobenzothiazole (MBT) in Benzothiazyl Disulfide (MBTS)¹

This standard is issued under the fixed designation D 5044; 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 the procedure for estimation of the acidic impurities in benzothiazyl disulfide (MBTS).

1.2 The values stated in SI units are to be regarded as the 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:*

D 1193 Specification for Reagent Water²

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries³

3. Summary of Test Method

3.1 A sample of MBTS is dissolved in solvent. After addition of an acetate buffer, water, and starch solution, the solution is titrated with iodine.

3.2 MBTS is sparingly soluble in any organic solvent. MBT, major impurity in MBTS, is very soluble. Stirring therefore will dissolve MBT, MBTS could stay undissolved.

4. Significance and Use

4.1 2-Mercaptobenzothiazole (MBT) is usually the major impurity in MBTS. Free MBT may be determined by this test method.

4.2 MBT and MBTS are used for rubber and latex vulcanization acceleration. The amount of MBT in MBTS may be of importance in predicting performance in rubber compounds and for raw material purchase and control.

4.3 This test method may be used as a quality control tool and for research and development work.

5. Apparatus

5.1 *Erlenmeyer Flask*, 300-cm³.

5.2 *Graduated Cylinders*, 10-cm³, 50-cm³, 250-cm³.

5.3 *Buret*, 10-cm³.

5.4 *Analytical Balance*, having a sensitivity of ± 0.1 mg.

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 shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Types I, II, or III of Specification D 1193.

6.3 *Isopropanol*, analytical reagent.

6.4 *Toluene*, analytical reagent.

6.5 *Acetic Acid*, 100 % analytical reagent.

6.6 *Sodium Acetate*, analytical reagent.

6.7 *Water*, distilled.

6.8 *Iodine Solution* (0.1 N).

6.9 *Starch Indicator Solution*—Slurry 2 g of soluble starch with 10 cm³ of water and dilute with 90 cm³ of boiling water.

6.10 *Sodium Acetate Solution*—Dissolve 60 g of sodium acetate in water to make 600 cm³ of solution.

6.11 *Acetate-Buffer*—Add 100 cm³ acetic acid (see 6.5) to 600 cm³ sodium acetate solution (6.9).

6.12 *Solvent*—Mix 5 volumes Isopropanol with 3 volumes toluene.

7. Sampling

7.1 Sampling shall be at the discretion of the analyst to

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. Current edition approved June 10, 2002. Published July 2002. Originally published as D 5044-97. Last previous edition D 5044-97.

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

³ *Annual Book of ASTM Standards*, Vol 09.01.

⁴ *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.