



Standard Test Method for Mercaptobenzothiazole Sulfenamide Assay by Reduction/ Titration¹

This standard is issued under the fixed designation D4936; 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 assay on mercaptobenzothiazole (MBT) sulfenamides. It is based on a titration of the basic amines liberated upon reduction of the sulfenamides with hydrogen sulfide gas (H_2S)^{2,3} or 2-mercaptobenzothiazole.

1.2 Any free amine (HNR_2) or weak acid salts of the corresponding amine ($\text{HX}\cdot\text{HNR}_2$) are titrated prior to reduction. This titer is used to calculate percent basic impurity (as free amine) in the sample.

1.3 With the indicated modifications, this test method is applicable to all MBT sulfenamides, that is, *N*-cyclohexyl-2-benzothiazolesulfenamide (CBS), *N,N*-diisopropyl-2-benzothiazyl sulfenamide (DIBS), 2 (morpholinothio) benzo-thiazole (MBS), *N,N*-dicyclohexyl-2-benzothiazyl sulfenamide (DCBS), *N*-*tert*-butyl-benzothiazole-sulfenamide (TBBS), and 4-morpholinyl-2-benzothiazyl disulfide (MBSS).

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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.* For specific hazard statements, see Section 9.

2. Referenced Documents

2.1 ASTM Standards:⁴

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 Goodyear Paper, Industrial and Engineering Chemistry Production Research and Development, Vol 2, 1963, p. 16.

³ *Elastomerics*, August 1981, pp. 34–44.

⁴ 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.

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3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *IpOH/Tol solvent*, *n*—titration solvent containing five volumes isopropanol and three volumes toluene.

3.1.2 “*lot*” *sample*, *n*—a production sample representative of a standard production unit.

3.1.3 *potassium hydrogen phthalate acidimetric standard*, *n*—Fisher P-243.⁵

3.1.4 *primary titrant*, *n*—0.25 to 0.30 *N* aqueous hydrochloric acid (HCl).

3.1.5 *reducing solution*, *n*—IpOH/Tol solvent saturated with hydrogen sulfide gas (H_2S) at 25°C (about 1.1 g H_2S /100 mL solvent).

3.1.6 *test unit*, *n*—the actual material used in the analysis. It must be representative of the “lot” sample.

3.2 Abbreviations:

3.2.1 THAM—tris (hydroxymethyl) aminomethane alkali-metric standard (Fisher T-395).³

4. Summary of Test Method

4.1 *Procedure A*—For CBS, TBBS, MBSS, MBS-90, and MBS, a weighed specimen is dissolved in the appropriate solvent, the “free amine” blank is titrated with standard acid, and the sulfenamide is reduced with H_2S . That is,



where:

Bt = the 2-benzothiazole radical.

BtSH = 2-mercaptobenzothiazole.

HNR_2 = free amine.

⁵ The sole source of supply of the apparatus known to the committee at this time is Fisher Scientific Co., 711 Forbes Ave., Pittsburgh, PA 15219. 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.