Designation: D8411 - 21

Standard Test Method for Rubber Compounding Materials—Determination of 2-Mercaptobenzothiazole (MBT) in Rubber¹

This standard is issued under the fixed designation D8411; 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 standard test method covers a procedure for the determination of extractable 2-Mercaptobenzothiazole (MBT) in rubber by high performance liquid chromatography (HPLC) to assess its bioavailability.
- 1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

2.1 ASTM Standards:²

D1193 Specification for Reagent Water

D1991 Test Method for Rubber Chemicals—2-Mercaptobenzothiazole (MBT)—Assay

D5044 Test Method for Rubber Compounding Materials— Free 2-Mercaptobenzothiazole (MBT) in Benzothiazyl Disulfide (MBTS)

3. Summary of Test Method

3.1 This test method allows for the determination of 2-Mercaptobenzothiazole in cured rubber by high performance liquid chromatography apparatus equipped with a diode-array

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

Current edition approved Oct. 1, 2021. Published October 2021. DOI: 10.1520/ D8411-21 detector (HPLC-DAD). The test is conducted after ultrasound extraction of the test sample with a chloroform-methanol mixture and shaking extraction with a simulated acid sweat solution.

4. Significance and Use

- 4.1 MBT is commonly used in rubber compounding as a curing accelerator for rubber and latex products. The determination of MBT in cured rubber is important to assess the risk of a release of this hazardous substance when rubber is in prolonged contact with human skin or mucous membranes.
- 4.2 Neo-formed MBT from MBTS might be created during the process of vulcanization when benzothiazolesulfenamides are used in the compound as accelerators, and therefore, it is part of the total MBT content in the cured rubber.
- 4.3 The purity of MBT as raw material can be evaluated by following Test Method D1991 based on a potentiometric titration of MBT with sodium hydroxide.
- 4.4 MBT is also a major impurity in MBTS. While MBTS is sparingly soluble in any organic solvent, MBT is very soluble. The amount of MBT contained in MBTS as raw material can be determined according to Test Method D5044.
- 4.5 This test method may be used as a quality control tool as well as for research and development work.

5. Apparatus

- 5.1 High performance liquid chromatography apparatus equipped with a diode-array detector (HPLC-DAD).
 - 5.2 Analytical Balance, accuracy ±0.1 mg.
- 5.3 *Ultrasonic Extraction Apparatus*, with a frequency of 40 \pm 5 kHz.
 - 5.4 Shaking Table, with water bath and variable frequency.
- 5.5 *pH Meter,* (digital is recommended), having an accuracy of \pm 0.05 pH.
 - 5.6 Volumetric Flask, 100 cm³.
 - 5.7 Conical flask with stopper, 250 cm³.
 - 5.8 Volumetric Flask, 1 dm³.
 - 5.9 Filter Paper, quantitative.

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.