Designation: D5051 – 06 (Reapproved 2012)

Standard Test Method for Rubber Compounding Materials—Benzothiazyl Disulfide (MBTS)—Assay[1]

This standard is issued under the fixed designation D5051; 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 of benzothiazyl disulfide (MBTS). It is based on a titration of free iodine liberated upon reduction of MBTS, with potassium iodide (KI) in acid medium.

1.2 The assay is determined as mass percent.

1.3 Free 2-mercaptobenzothiazole (MBT) content is not determined.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Summary of Test Method

3.1 In an acid medium, MBTS is reduced with KI to MBT and free iodine. The free iodine is titrated with standard sodium thiosulfate solution.

3.2 MBTS is sparingly soluble in any organic solvent; while MBT, formed during the reaction of MBTS with KI is very soluble. Therefore after reagents are added as indicated in the procedure, continue to stir till all MBTS has reacted.

4. Significance and Use

4.1 The test method is designed to assess the purity of MBTS, which is used for rubber and latex vulcanization acceleration. The amount of MBTS is of importance in predicting performance in rubber compounds and for raw material purchase and control.

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

5. Interferences

5.1 KI-reducible contaminants interfere with the results.

6. Apparatus

6.1 *Erlenmeyer Flask,* 300 cm^3.

6.2 *Graduated Cylinder,* 5 cm^3.

6.3 *Magnetic Stirrer,* with hot plate.

6.4 *Buret,* 50-cm^3 capacity.

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

7. Reagents and Materials

7.1 *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.[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.

7.2 *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 D1193.

7.3 *Acetic Acid,* 100 %, analytical reagent.

7.4 *Isopropanol,* analytical reagent.

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[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 May 1, 2012. Published July 2012. Originally approved in 1990. Last previous edition approved in 2006 as D5051 – 06. DOI: 10.1520/D5051-06R12.

[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.

7.5 *Toluene,* analytical reagent.

7.6 *Potassium Iodide* (KI), analytical reagent.

7.7 *Sodium Thiosulfate* ($Na_2S_2O_3$) (0.1 *N*).

7.8 *Starch Indicator Solution*—Slurry 2 g of soluble starch with 10 cm^3 of water and dilute with 90 cm^3 of boiling water.

7.9 *Hydrochloric Acid* (HCl) (2 *N*).

7.10 *Solvent:* Mix 5 volumes isopropanol with 3 volumes toluene.

8. Sampling

8.1 Depending upon the purposes of the testing, sampling shall be at the discretion of the analyst to obtain as representative a sample as possible of the lot to be tested.

9. Procedure

9.1 Accurately weigh about 0.5 g of the test specimen to the nearest 0.1 mg and carefully transfer it to a 300-cm^3 Erlenmeyer flask.

9.2 Add 50 cm^3 of solvent to the specimen, which may not dissolve completely.

9.3 With stirring, add the reagents in the following sequence: 50 cm^3 of distilled water, about 6 g of KI, 25 cm^3 of 2 *N* HCl, and 25 cm^3 of acetic acid (see 3.2). Sample will now be dissolved.

9.4 Immediately titrate (see Note 1) the liberated iodine with 0.1 *N* $Na_2S_2O_3$ solution. Toward the end of the titration, add 5 cm^3 of starch indicator solution. The end point proceeds from blue-violet to yellow to colorless (milky).

NOTE 1—The final titration must be carried out as soon as acetic acid is added. If allowed to stand, a reduction of free iodine will occur, giving a lower MBTS assay.

9.5 Obtain a blank titration by proceeding from 9.2 to 9.4.

10. Calculation

10.1 Calculate the percent MBTS as follows:

$$Percent\ MBTS = \frac{(A - B) \times (N \times 0.33248)}{2\ W} \times 100 \qquad (1)$$

where:

A = volume of $Na_2S_2O_3$ (see 7.7) required for titration of the sample, cm^3,
B = volume of $Na_2S_2O_3$ required for titration of the blank, cm^3,
N = normality of the $Na_2S_2O_3$ solution,
W = mass of the test specimen in g, and
0.33248 = millimole mass of MBTS.

TABLE 1 Precision for MBTS Assay[A]

	Mean	Within-Laboratory[B]			Between Laboratory		
Material	Value (a)	Sr	r	(r)	SR	R	(R)
IRM-MBTS	96.79	0.134	0.380	0.39	0.397	1.12	1.16

[A] Precision results for 5 laboratories, mean value in percent assay for MBTS.
[B] Sr = repeatability standard deviation
r = repeatability, in measurement units
(r) = repeatability (relative) in percent
SR = reproducibility standard deviation
R = reproducibility, in measurement units
(R) = reproducibility (relative) in percent

11. Report

11.1 Report the following information:

11.1.1 Proper identification of the sample and

11.1.2 Results obtained from two individual determinations and their average, reported to the nearest 0.1 %.

12. Precision and Bias[4]

12.1 This precision and bias section has been prepared in accordance with Practice D4483. Please refer to this practice for terminology and other statistical calculation details.

12.2 The precision results in this precision and bias section given an estimate of the precision of this test method with the materials (rubbers, etc.) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance or rejection testing or any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific testing protocols of the test method.

12.3 A Type 1 interlaboratory test program (ITP) was conducted in 1997 on a sample of IRM-MBTS. Six laboratories participated in the ITP conducting duplicate tests on each of 2 successive test days. A test result for the assay is the value obtained from one analysis operation. The database generated by this ITP was divided into two parts; Part 1 used the first of the duplicates on each day and Part 2 used the second of the duplicates. A complete statistical analysis according to D4483 was conducted for each part. The analysis results of each part where then combined (averaged) for the final values as given in this section. Thus the precision results pertain to between day single determinations for the assay values for MBTS.

12.4 The D4483 analysis revealed that one of the laboratories had excessive within-laboratory variation (high k-value) and the results from this laboratory were deleted. The precision is therefore based on a five laboratory ITP.

12.5 *Repeatability*—The repeatability r, of this test method has been established as the value tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated r (for any given level) must be considered as derived from different or non-identical sample populations.

12.6 *Reproducibility*—The reproducibility R, of this test method has been established as the value tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated R must be considered to have come from different or non-identical sample populations.

12.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias cannot therefore be determined.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1083.

13. Keywords

13.1 mercaptobenzothiazole; mercaptobenzothiazole disulfide