Designation: D1991 – 15

Standard Test Method for Rubber Chemicals—2-Mercaptobenzothiazole (MBT)—Assay[1]

This standard is issued under the fixed designation D1991; 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 procedure for the assay of 2-mercaptobenzothiazole (MBT). It is based on a potentiometric titration of MBT with sodium hydroxide.

1.2 The assay is determined as percent by mass.

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

1.4 *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 sample of MBT is dissolved in ethanol. The solution is titrated potentiometrically using a solution of sodium hydroxide as the titrant.

4. Significance and Use

4.1 MBT is commonly used as an accelerator for rubber and latex vulcanization. The purity of MBT may be of importance in predicting performance in rubber compounds and this test method is designed to assess the purity of MBT.

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

5. Apparatus

5.1 *Erlenmeyer Flask,* 250-cm^3.

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

5.3 *Potentiograph.*

5.4 *Glass pH Electrode,* and reference electrode.

5.5 *Graduated Cylinder,* 200-cm^3.

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.[3]

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

6.3 *Aqueous Sodium Hydroxide Solution (40 g NaOH in 1 dm^3 of solution)*—Standardize by accepted analytical techniques to ensure that the maximum error of the normalization factor is not more than 0.001.

6.4 *Ethanol,* denatured with toluene (in the ratio of 100 volumes ethanol to 1 volume toluene).

6.5 *Toluene.*

7. Sampling

7.1 Sampling shall be at the discretion of the analyst to obtain as representative a sample as possible, of the lot to be tested.

8. Procedure

8.1 Weigh (to nearest 0.001 g) about 5 g of the specimen, and transfer into a 250-cm^3 Erlenmeyer flask. Using a graduated cylinder, add 125 cm^3 denatured ethanol (6.4). Titrate the

[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 *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

solution potentiometrically, using a glass electrode system, with NaOH (6.3) as the titrant (A) (see Fig. 1).

9. Calculation

9.1 From the plot of pH versus millilitres of NaOH, as illustrated in Fig. 1, the equivalence point for the potentiometric titration is defined as the inflection point of the curve. This inflection point may be estimated by determination of the midpoint of the tangent slope.

9.1.1 This inflection point is identified as follows:

9.1.1.1 Draw a tangent through the steepest part of the curve. Determine the two points at which the curve departs from this tangent line. The midpoint of the line segment between these two points represents the equivalence point.

9.2 Calculate the MBT content by the following equation:

$$\text{MBT content, \%} = \frac{167.2 \times A \times N \times 100}{W \times 1000} \quad (1)$$

where:

A = volume of sodium hydroxide to the equivalence point, cm^3,
167.2 = molecular mass of MBT,
W = mass g of specimen, and
N = normality of titrant.

10. Report

10.1 Report the following information:

10.1.1 Proper identification of the sample, and

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

11. Precision and Bias

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

11.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory programs as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

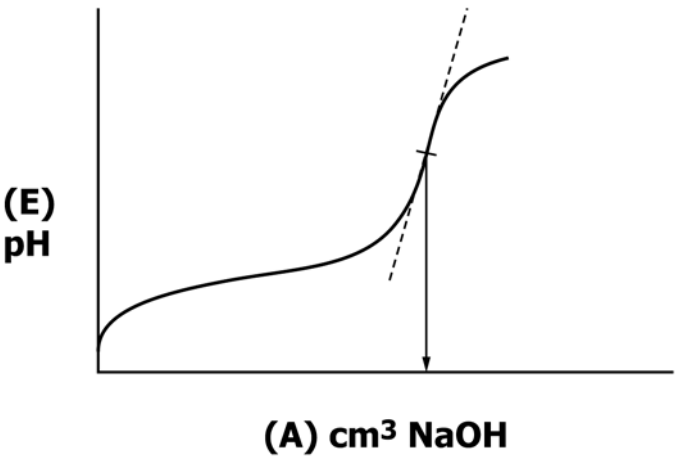

FIG. 1 Potentiometric Titration Using NaOH as the Titrant

11.3 A Type 1 (interlaboratory) precision was evaluated in 1988. Both repeatability and reproducibility are short term. A period of a few days separates replicate test results. A test result is the mean of the assay results.

11.4 An MBT sample was analyzed in ten laboratories on two different days.

11.5 The results of the precision calculations for repeatability and reproducibility are shown in Table 1.

11.6 *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 the tabulated r (for any given level) must be considered as derived from different or nonidentical sample populations.

11.7 *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 (for any given level) must be considered to have come from different or nonidentical sample populations.

11.8 Repeatability and reproducibility expressed as a percentage of the mean level, (r) and (R), have equivalent application statements as above for r and R. For the (r) and (R) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

11.9 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values have not been evaluated for this test method. Bias therefore cannot be determined.

12. Keywords

12.1 accelerators; assay; mercaptobenzothiazole (MBT); thiazole

TABLE 1 MBT Assay[A]

Material	Average %	Within Laboratories			Between Laboratories		
		Sr	*r*	*(r)*	*SR*	*R*	*(R)*
MBT	97.56	0.286	0.810	0.83	1.675	4.74	4.86

[A] Sr = repeatability standard deviation,
r = repeatability—2.83 × the square root of the repeatability variance,
(r) = repeatability (as percentage of material average),
SR = reproducibility standard deviation,
R = reproducibility—2.83 × the square root of the reproducibility variance, and
(R) = reproducibility (as percentage of material average).