Designation: D 5054 – 90 (Reapproved 2000)

Standard Test Method for Rubber Chemicals—Diphenyl Guanidine (DPG) and Di-o-tolyl-guanidine (DOTG) Assay[1]

This standard is issued under the fixed designation D 5054; 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 determination of assay of diphenyl guanidine (DPG) and di-o-tolyl-guanidine (DOTG). It is based on a visual titration of DPG or DOTG with hydrochloric acid (HCl).

1.2 The assay is determined as mass percent.

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

D 1193 Specification for Reagent Water[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]

3. Summary of Test Method

3.1 DPG or DOTG is dissolved in methanol and titrated with HCl solution using bromophenol blue indicator.

4. Significance and Use

4.1 DPG and DOTG are used for rubber and latex vulcanization acceleration. The amount of DPG or DOTG may be 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 Alkaline contaminants that are titratable with HCl interfere with the results.

6. Apparatus

6.1 *Erlenmeyer Flask*, 200-cm^3.

6.2 *Graduated Cylinder*, 25-cm^3.

6.3 *Magnetic Stirrer*.

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

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

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.[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.

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 D 1193.

7.3 *Methanol*, analytical reagent.

7.4 *Hydrochloric Acid* (HCl) (0.1 *N*).

7.5 *Bromophenol Blue Indicator Solution* (0.4 g/cm^3—Dissolve 0.4 g bromophenol blue in 1000 cm^3 methanol.

8. Sampling

8.1 Depending upon the purpose 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 1.0 g of the test specimen to the nearest 0.1 mg and carefully transfer it to a 200-cm^3 Erlenmeyer flask.

9.2 Using a graduated cylinder, dissolve the specimen by adding 25 cm^3 of methanol.

9.3 With stirring, add 5 drops of bromophenol blue indicator solution.

9.4 Titrate with 0.1 *N* HCl solution. The color change at the end point is from blue-violet to green.

10. Calculation

10.1 Calculate the percent DPG, *A*, as follows:

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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 April 27, 1990. Published June 1990.

[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," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

$$A = \frac{V \times N \times 0.2112}{W} \times 100 \quad (1)$$

where:
V = volume of 0.1 N HCl used, for titration of the sample, cm^3,
N = normality of the HCl solution,
W = sample used, g, and
0.2112 = millimole mass of DPG.

10.2 Calculate the percent DOTG, *B*, as follows:

$$B = \frac{V \times N \times 0.2393}{W} \times 100 \quad (2)$$

where:
0.2393 = millimole mass of DOTG.

11. Report

11.1 Report the following information:

11.1.1 Proper identification of the sample and

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

12. Precision and Bias

12.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical calculation details.

12.2 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 in this section. The precision parameters should not be used for acceptance or 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.

12.3 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 value, as specified by this test method, of the assay results.

12.4 A DPG sample and a DOTG sample were analyzed in ten laboratories on two different days.

12.5 The results of the precision calculations for repeatability and reproducibility are given in Table 1.

12.6 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value 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.

12.7 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the appropriate value 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.

12.8 Repeatability and reproducibility expressed as a percent 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 percent of the arithmetic mean of the two test results.

12.9 *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.

12.10 The full details and test results of the interlaboratory test program used for this precision section are complete and in the process of being prepared in the proper format for submission as a Research Report to ASTM Headquarters.

13. Keywords

13.1 di-o-tolyl-guanidine; diphenyl guanidine

TABLE 1 DPG/DOTG Assay, %

Material	Average	Within Laboratory[A]			Between Laboratories[A]		
		S_r	*r*	(*r*)	S_R	*R*	(*R*)
DPG	98.20	0.207	0.586	0.597	0.567	1.605	1.64
DOTG	97.16	0.141	0.399	0.411	0.639	1.809	1.86

[A] S_r = repeatability standard deviation.
r = repeatability = 2.83 times the square root of the repeatability variance.
(r) = repeatability (as percent material average).
S_R = reproducibility standard deviation.
R = reproducibility = 2.83 times the square root of the reproducibility variance.
(R) = reproducibility (as percent of material average).