



Standard Test Method for Rubber Chemicals—Diphenyl Guanidine (DPG) and Di-o-tolyl-guanidine (DOTG) Assay¹

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 Water²

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries³

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-cm³.

6.2 *Graduated Cylinder*, 25-cm³.

6.3 *Magnetic Stirrer*.

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

6.5 *Buret*, 50-cm³ 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/cm³—Dissolve 0.4 g bromophenol blue in 1000 cm³ 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-cm³ Erlenmeyer flask.

9.2 Using a graduated cylinder, dissolve the specimen by adding 25 cm³ 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:

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 *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."