



Standard Test Method for Rubber Compounding Materials—Determination of the Basic Nitrogen Content in Rubber Antioxidant: Polymerized TMQ¹

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1. Scope

1.1 This test method covers the determination of the nitrogen content of polymerized 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ). It is based on a potentiometric titration of an acetone solution of TMQ with perchloric acid in acetic acid.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Section 9.

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. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *lot sample*—a production sample representative of a standard production unit, normally referred to as “the sample.”

4. Summary of Test Method

4.1 A sample of 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ) is dissolved in acetone and the nitrogen in TMQ is determined by a potentiometric titration with perchloric acid in acetic acid.

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.

5. Significance and Use

5.1 This test method is designed to determine the nitrogen content of oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ) antioxidant. TMQ is used for heat protection of vulcanized rubber.

5.2 This test method is suitable for assessing product specifications since nitrogen content is related to product performance, that is, antioxidant efficiency and heat protection. For comparison of product quality at different production facilities sufficient interlaboratory accuracy and precision are required.

6. Interferences

6.1 Based on past experience, one significant source of error in this test method is the titration end point assessment. Problems can be avoided by closely following the procedure.

6.2 Theoretically, any material containing basic nitrogen, capable of forming HCl-salt, will be measured by this test method. Extensive high performance liquid chromatograph (HPLC) analyses of the product indicates that the most significant interfering impurities are products formed by side reactions between aniline and acetone, such as those given in Fig. 1.

6.3 Other possible interfering impurities may include the anilino end groups attached to TMQ oligomers, such as those given in Fig. 2.

7. Apparatus

7.1 *Standard Laboratory Glassware and Equipment.*

7.2 *Potentiometer.*

7.3 *Reference Electrode*, platinum electrode in an electrolytic glass cell with a frit of porosity 4, filled with a saturated solution of lithium perchlorate in acetic acid (see Fig. 3).

7.4 *Glass Electrode.*

7.5 *Weighing Pipet*, 5 cm³.

7.6 *Buret*, 25 cm³, Class A, graduated in 0.1 cm³ increments.

7.7 *Buret*, 50 cm³, Class A, graduated in 0.1 cm³ increments.