



Designation: D5666 – 95 (Reapproved 2023)

Standard Test Method for Rubber Chemical Antidegradants—Purity of *p*-Phenylenediamine (PPD) Antidegradants by High Performance Liquid Chromatography¹

This standard is issued under the fixed designation D5666; 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 purity of Type I, II, and III *p*-phenylenediamine (PPD) antidegradants as described in Classification [D4676](#) by high performance liquid chromatography (HPLC) using ultraviolet detection and external standard calculations.

1.2 Expertise in HPLC is necessary to the successful application of this test method.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

[D3853 Terminology Relating to Rubber and Rubber Latices—Abbreviations for Chemicals Used in Compounding](#)

[D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries](#)

¹ This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials 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.

[D4676 Classification for Rubber Compounding Materials—Antidegradants](#)

[2.2 ISO Standards:](#)³

[ISO 5725 Precision of Test Methods](#)

[ISO 6472 Rubber Compounding Ingredients—Abbreviations](#)

3. Terminology

3.1 Definitions:

3.1.1 *external standard calculation*—a method of calculating the percent composition by measuring the area of the analyte peak, multiplying by a response factor, and dividing by the sample concentration. All components are assumed to be resolved from the component of interest.

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

3.1.3 *specimen*—the actual material used in the analysis, also known as the test portion. It must be representative of the lot sample.

3.2 Abbreviations:

3.2.1 The following abbreviations are in accordance with Terminology [D3853](#) and ISO 6472:

3.2.2 *77PD*—N,N' bis-(1,4-dimethylpentyl)-*p*-phenylenediamine.

3.2.3 *DTPD*—N,N'-ditolyl-*p*-phenylenediamine.

3.2.4 *IPPD*—N-isopropyl-N'-phenyl- *p*-phenylenediamine.

3.2.5 *PPD*—*p*-phenylenediamine.

3.2.6 *6PPD*—N-(1,3 dimethylbutyl)-N'-phenyl-*p*-phenylenediamine.

4. Summary of Test Method

4.1 A specimen is dissolved in acetonitrile and a fixed loop volume is analyzed by isocratic HPLC using a thermostated C18 reversed phase column and an ultraviolet (UV) detector. Peak areas are determined using a chromatographic integrator

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.