



Standard Test Method for P-Phenylenediamine Antidegradants—Purity by High Performance Liquid Chromatography¹

This standard is issued under the fixed designation D 5666; 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 Practice D 4676 by high performance liquid chromatography using ultraviolet detection and external standard calculations.

1.2 Expertise in high performance liquid chromatography (HPLC) is necessary to the successful application of this test method.

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.*

2. Referenced Documents

2.1 ASTM Standards:

D 3853 Terminology Relating to Rubber and Rubber Latexes—Abbreviations for Chemicals Used in Compounding²

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

D 4676 Classification for Rubber Compounding Materials—Antidegradants²

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*—The following abbreviations are in accordance with Terminology D 3853 and ISO 6472:

3.2.1 *77PD*—N,N' bis-(1,4-dimethylpentyl)-*p*-phenylenediamine.

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

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

3.2.4 *PPD*—*p*-phenylenediamine.

3.2.5 *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 or laboratory data system with the amount of analyte being determined by external calibration.

5. Significance and Use

5.1 This test method is designed to determine the purity of *p*-phenylenediamine antidegradants.

5.2 Since the results of this test method are based on an integrated peak area as determined by HPLC, it is assumed that all analytes of interest are resolved from interfering peaks.

6. Interferences

6.1 Components co-eluting with the analyte of interest will cause erroneous results; thus it is required that the system be capable of providing a minimum of 10 000 theoretical plates.

7. Apparatus

7.1 *Liquid Chromatograph*, consisting of the following:

7.1.1 Precision chromatographic pump,

7.1.2 Variable wavelength UV detector,

7.1.3 A method for thermostating the column at $35 \pm 1^\circ\text{C}$, for example, a column oven or water jacket,

7.1.4 A fixed injector made of either a 20 mm³ (μL) rheodyne loop or an automatic sampler.

7.2 *HPLC Columns*, consisting of:

7.2.1 A precolumn packed with C18 grafted silica with particle size of 35 to 40 μm (100 to 150 mm), and

7.2.2 A column of 10- to 15-cm length packed with C18

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 Feb. 15, 1995. Published April 1995.

² *Annual Book of ASTM Standards*, Vol 09.01.

³ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.