



Designation: **D1618 – 99 (Reapproved 2011) D1618 – 17**

## Standard Test Method for Carbon Black Extractables—Transmittance of Toluene Extract<sup>1</sup>

This standard is issued under the fixed designation D1618; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the measurement of the degree of toluene discoloration by carbon black extractables and is useful in controlling the reaction processes for production of carbon black. This test method may not be applicable to carbon blacks with high extractables.

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

1.4 *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:*<sup>2</sup>

[D1799 Practice for Carbon Black—Sampling Packaged Shipments](#)

[D1900 Practice for Carbon Black—Sampling Bulk Shipments](#)

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

### 3. Terminology

3.1 *Definitions:*

3.1.1 *carbon black toluene discoloration, n*— the transmittance, at 425 nm, of the filtrate obtained from the toluene extract of carbon black, compared to that of pure toluene.

### 4. Significance and Use

4.1 The toluene discoloration value provides an estimate of toluene-soluble discoloring residues present on the carbon black.

### 5. Apparatus and Reagent

5.1 *Spectrophotometer*, with tungsten filament lamp, 20-nm maximum spectral bandpass, capable of measuring percent transmittance at a 425-nm wavelength. The instrument is to be operated in accordance with the manufacturer's directions for optimum performance.

5.2 *Cuvets*, rectangular, with an optical light path of 10 mm.

5.3 *Balance*, analytical, with a sensitivity of  $\pm 0.01$  g.

5.4 *Oven*, gravity-convection type, capable temperature regulation within  $\pm 1^\circ\text{C}$  at  $125^\circ\text{C}$  and temperature uniformity within  $\pm 5^\circ\text{C}$ .

5.5 *Pipet*, automatic, bottle-type, 20 cm<sup>3</sup>, with a repeatability of  $\pm 0.1$ .

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee [D24](#) on Carbon Black and is the direct responsibility of Subcommittee [D24.31](#) on Non-Carbon Black Components of Carbon Black.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- 5.6 *Erlenmeyer Flasks*, 125 cm<sup>3</sup> with ground-glass stopper.
- 5.7 *Filter Paper*, qualitative grade, medium retention, medium-fast filter speed, 150-mm diameter.
- 5.8 *Glass Filtering Funnels*, 75-mm inside diameter at the top.
- 5.9 *Beakers*, 50 or 100 cm<sup>3</sup>, with pouring lips.
- 5.10 *Wiping Paper*, lint-free.
- 5.11 *Cotton Swabs*.
- 5.12 *Fume Hood*, fully enclosed on three sides, with spark-proof fan and motor.
- 5.13 *Toluene*, analytical reagent grade.

## 6. Sampling

- 6.1 Samples shall be taken in accordance with Practice [D1799](#) or Practice [D1900](#).

## 7. Standardization of Apparatus

- 7.1 Turn on the spectrophotometer and allow it to warm for the length of time specified by the manufacturer.
- 7.2 Check the zero reading of the spectrophotometer in accordance with the manufacturer's instructions, and adjust if necessary.
- 7.3 Place the funnel with the filter paper into an Erlenmeyer flask. Filter approximately 30 cm<sup>3</sup> of toluene into the flask and stopper the flask.
- 7.4 Pour a portion of the toluene into the beaker with the pouring lip for simplifying the transfer of the toluene to the cuvet.
- 7.5 Rinse the cuvet with the filtered toluene three times, filling approximately one third full each time. Discard this rinsing toluene into an approved safety container.

NOTE 1—The cuvet must be handled on the ground-glass sides only. Do not touch the smooth, clear sides with the fingers.

- 7.6 Fill the cuvet and wipe the outside surfaces with the lint-free wiping paper while holding the cuvet in front of a suitable light source. The toluene must be free of any contaminants, such as lint particles, which might cause light scattering, thus influencing the test results. If necessary, rewipe the outside until perfectly clean, or clean the inside surfaces with a cotton swab. Repeat [7.5](#) if the inside surfaces are cleaned.

- 7.7 . Insert the cuvet into the spectrophotometer and adjust it to read 100 % transmittance at the 425-nm wavelength.

## 8. Procedure

- 8.1 Dry an adequate sample of carbon black at 125 ± 1°C for 60, +5, –0 min, using a gravity-convection oven.

NOTE 2—An infrared lamp must not be used for drying samples, as it will vaporize some of the extractable materials.

- 8.2 Allow the sample to cool to room temperature in a desiccator.
- 8.3 Weigh 2.00 ± 0.01 g of the carbon black and transfer it to a 125-cm<sup>3</sup> Erlenmeyer flask with ground-glass stopper.
- 8.4 Add 20 cm<sup>3</sup> of toluene to the sample flask and stopper the flask.

NOTE 3—If necessary, larger quantities of carbon black and toluene may be used, but the quantities must remain in this ratio of 1 g/10 cm<sup>3</sup> of toluene.

- 8.5 Without delay, shake the mixture vigorously either by hand or machine for 60, +5, –0 s.
- 8.6 Immediately pour as much of the mixture as possible into the glass funnel with filter paper, which has previously been prepared and inserted into an Erlenmeyer flask.

8.7 The filtrate must be free of visible carbon black particles, otherwise the sample preparation shall be repeated ([8.3](#)). If the problem persists, use filter paper with smaller pore size for this particular sample.

- 8.8 As soon as filtration is complete, stopper the flask until ready to test.
- 8.9 Check standardization of the spectrophotometer at 425 nm in accordance with Section [7](#).
- 8.10 Pour a portion of filtrate out of the stoppered flask into a beaker with a pouring lip.
- 8.11 Using a cuvet matched to the one in [7.5](#), or the same cuvet as used in [7.5](#), rinse and fill the cuvet in the same manner as in [7.5](#) and [7.6](#).
- 8.12 Insert the cuvet into the spectrophotometer and record the percent transmittance obtained at 425 nm to the nearest 0.1 %.

## 9. Report

- 9.1 Report the following information:
  - 9.1.1 Proper identification of the sample, and