Designation: D1618 – 23

Standard Test Method for Carbon Black Extractables—Transmittance of Toluene Extract[1]

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 (ε) 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:*[2]

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 ±0.01 g.

5.4 *Oven,* gravity-convection type, capable temperature regulation within ±1°C at 125°C and temperature uniformity within ±5°C.

5.5 *Pipet,* automatic, bottle-type, 20 cm^3, with a repeatability of ±0.1.

5.6 *Erlenmeyer Flasks,* 125 cm^3 with ground-glass stopper.

5.7 *Filter Paper,* qualitative grade, 150-mm diameter, pore size of 2 to 3 µm.

5.8 *Glass Filtering Funnels,* 75-mm inside diameter at the top.

5.9 *Beakers,* 50 or 100 cm^3, 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.

[1] 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.

Current edition approved Nov. 1, 2023. Published November 2023. Originally approved in 1965. Last previous edition approved in 2018 as D1618 – 18. DOI: 10.1520/D1618-23.

[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.

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^3 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^3 Erlenmeyer flask with ground-glass stopper.

8.4 Add 20 cm^3 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^3 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

9.1.2 Toluene discoloration value reported to the nearest 0.1 % transmittance.

10. Precision and Bias

10.1 These precision statements have been prepared in accordance with Practice D4483-99. Refer to this practice for terminology and other statistical details.

10.2 It was determined by a D24 task group formed to update the precision statement of this test method that the test precision, in particular the reproducibility, strongly depends on the level of transmittance. Therefore overall average or pooled values might be misleading. This is why the precision table has been subdivided into three separate tables with defined ranges of percentage of transmittance: high (100 to 95 %), medium (94.9 to 85 %) and low (below 85 %).

10.3 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from Tables 1-3.

10.4 A type 1 inter-laboratory precision program was conducted as detailed in Tables 1-3. Both repeatability and reproducibility represent short-term (daily) testing conditions. The testing was performed using two operators in each laboratory performing the test once on each of two days (total of four tests). A test result is the value obtained from a single determination. Acceptable difference values were not measured. The between-operator component of variation is included in the calculated values for r and R.

10.5 The results of the precision calculations for this test are given in Tables 1-3. In each of these tables the materials are arranged in descending "mean level" order. The preferred precision is shown in bold text.

10.6 *Repeatability*—The pooled **absolute** repeatability, r, of this test has been established as 1.55 % transmittance for high transmittance values (100 to 95 %), 2.36 % transmittance for medium transmittance values (94.9 to 85 %) and 2.87 % transmittance for low transmittance values (below 85 % transmittance). Any other value in Tables 1-3 may be used as