



Designation: ~~D7772~~—~~11~~ D7772 – 15

Standard Test Method for Carbon Black Extractables – Absorbance of Cyclohexane Extract¹

This standard is issued under the fixed designation D7772; 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 method covers the determination of extinction (absorbance) of carbon black cyclohexane extract at 386 nm using a UV spectrophotometer.

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.*

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. Summary of Test Method

3.1 A carbon black sample is gently agitated in cyclohexane at room temperature to remove any extractable material. The mixture is left standing in the dark for 24 h at room temperature. The carbon black is separated from the cyclohexane by filtration, with subsequent determination of the extinction of the filtrate at 386 nm using a 50-mm cuvette.

4. Significance and Use

4.1 This procedure serves as a screening process for carbon blacks used in the manufacturing of products that are to come into contact with food. The cyclohexane extract absorbance at 386 nm must be less than 0.10 for a 50 mm cuvette in order to fulfill various regulatory requirements.

5. Apparatus

5.1 *Spectrophotometer*, 20 nm maximum spectral bandpass, capable of measuring absorbance at 386 nm.

5.2 *Cuvettes*, with an optical light path of 50 mm.

5.3 *Balance*, analytical, sensitivity 0.01 g.

5.4 *Filter Paper*, with a particle retention in liquid less than 2 μm .

5.5 *Glass Filtering Funnels*, 75 mm diameter at top.

5.6 *Volumetric Flask*, 100 cm^3 .

5.7 *Lens Cleaning Tissue*, lint free.

5.8 *Timer*, with 1 s divisions.

5.9 *Oven*, gravity-convection type, capable of temperature regulation of $125 \pm 5^\circ\text{C}$.

5.10 *Desiccator*.

¹ This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.66 on Environment, Health, and Safety.

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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.

5.11 As a good laboratory practice, it could be suggested the use of a Neutral Density Filter with a spectral absorbance calibrated at 386 nm to ensure the spectrophotometer is correctly calibrated.

6. Reagents and Materials

6.1 *Cyclohexane*, analytical reagent grade.

7. Hazards

7.1 This test involves hazardous materials, operations and equipment. This procedure does not attempt to address the safety problems associated with this test. A hazards review must be conducted by all personnel performing the test. It is the responsibility of the user to review all MSDS, manuals and hazards procedures and establish the appropriate safety measures.

8. Calibration, Standardization, and Environmental Conditions

8.1 ~~Standard Laboratory Test Conditions:~~ Conditions:

8.1.1 Unless stated elsewhere, all testing, calibration and standardization should be carried out in the ambient temperature range of $23 \pm 3^\circ\text{C}$.

8.1.2 The date of calibration or testing should also be recorded with any relevant data.

8.1.3 All calibration data should be recorded on a dedicated file.

8.2 *Instrument Set-Up Frequency – Day of Test:*

8.2.1 Turn on the spectrophotometer and allow it to warm up for 30 min.

8.2.2 Set the wavelength to 386 nm and zero the instrument.

8.2.3 Place the Neutral Density Filter inside the sample compartment of the instrument and verify the absorbance value is within the absorbance tolerance. If the filter is found to be outside the limits, verify the filter's absorbance using a more sophisticated UV/Vis Spectrometer. If in this case nothing abnormal is found for the filter, service and calibrate the spectrophotometer.

8.2.4 It is imperative that steps ~~7-2-58.2.5 – 7-2-88.2.8~~ are carried out in a fume hood using disposable nitrile gloves. This kind of gloves are recommended for cyclohexane to prevent skin exposure (cyclohexane can cause skin irritation).

8.2.5 Filter approximately 50 cm³ cyclohexane through the filter paper into a stoppered graduated cylinder.

8.2.6 Transfer a portion of the filtered cyclohexane to a clean cuvette. The cuvette should not be handled on the clear working surface by fingers.

8.2.7 Rinse the cuvette with filtered cyclohexane three times, filling approximately 1/3 full each time. Discard the rinsing cyclohexane into an approved safety can.

8.2.8 Fill the cuvette and wipe the outside surfaces with lint free wiping paper while holding the cuvette in front of a suitable light source to check for smudges. The cyclohexane must be free of any contaminants such as lint particles, which might cause light scattering and influence test results.

8.2.9 Insert the cuvette into the spectrophotometer, adjust it to read the 386 nm wavelength and zero the instrument to achieve 0.000 absorbance at 386 nm.

9. Procedure

9.1 Dry the sample(s) of carbon black for 1 h at $125 \pm 5^\circ\text{C}$ prior to testing.

9.2 Allow the sample to cool to room temperature in a desiccator.

9.3 Weigh 1.00 ± 0.01 g sample and transfer to a 100 cm³ volumetric flask.

9.4 It is imperative that steps ~~8-59.5 – 8-99.9~~ are carried out in a fume hood using disposable nitrile gloves. This kind of gloves are recommended for cyclohexane to prevent skin exposure (cyclohexane can cause skin irritation).

9.5 Add 25 to 30 cm³ of cyclohexane to the sample flask and stopper the flask.

9.6 Shake the mixture gently for a few seconds.

9.7 Fill up to the mark with cyclohexane.

TABLE 1 Precision Parameters for Test Method D7772, Carbon Black Extractables-Absorbance of Cyclohexane Extract (Type 1 Precision)

Material	Number of Labs	Unit Absorbance	Sr	r	(r)	SR	R	(R)
N772	4	0.008	0.005	0.015	203.78	0.007	0.020	269.58
N683 (SRB 8F)	4	0.011	0.004	0.011	101.72	0.009	0.026	249.15
	Average Pooled	0.009	0.005	0.013	146.05	0.008	0.024	259.90