Designation: D7772 – 15

Standard Test Method for Carbon Black Extractables – Absorbance of Cyclohexane Extract[1]

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 ± 5°C.

5.10 *Desiccator.*

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:*

[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.

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	0.009						
	Pooled		0.005	**0.013**	146.05	0.008	**0.024**	259.90

8.1.1 Unless stated elsewhere, all testing, calibration and standardization should be carried out in the ambient temperature range of 23 ± 3°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 8.2.5 – 8.2.8 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 cm^3 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 ⅓ 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 ± 5°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 cm^3 volumetric flask.

9.4 It is imperative that steps 9.5 – 9.9 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 cm^3 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.

9.8 Close the flask with a stopper, gently shake the mixture again for a few seconds and place it in dark for 24 h at room temperature.

9.9 After 24 h, filter the mixture through the filter paper and collect the filtrate in a graduated cylinder. As soon as the filtration is complete, stopper the graduated cylinder until ready to test.

9.10 Using the same cuvette as described in 8.2.6, rinse and fill the cuvette with sample filtrate.

9.10.1 The same cuvette must be used for the blank and all samples, as differences in the optical density of different cuvettes will cause errors.

9.11 Insert the cuvette into the spectrophotometer and record the absorbance reading obtained at 386 nm.

10. Report

10.1 Report the following information:

10.1.1 Proper identification of the carbon black sample.

10.1.2 Optical light path of the cuvette used: 50 mm.

10.1.3 Cyclohexane extinction to the nearest 0.01 absorbance measurement.

11. Precision and Bias[3]

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

11.2 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.

11.3 A type 1 inter-laboratory precision program was conducted in 2014 as detailed in Table 1. Both repeatability and reproducibility represent short term (daily) testing conditions.

[3] A research report is forthcoming.

The testing was performed using two operators in each laboratory performing the test once on each of two days (total of four tests). Acceptable difference values were not measured. The between operator component of variation is included in the calculated values for r, (r), R, and (R).

11.4 The results of the precision calculations for this test method are given in Table 1. The materials are arranged in ascending "mean level" order. The preferred precision estimates are absolute (see bolded values in Table 1).

11.5 *Repeatability*—The pooled repeatability, r, for cyclohexane extinction has been established as 0.013 absorbance units (see Table 1). The best estimate of the test precision is given by the pooled value and this should be used unless there is a good reason to use a different value. Any other value in Table 1 may be used as an estimate of repeatability, if justified, such as when testing the same material as, or a material similar to, those in the table. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability value on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate action taken.

NOTE 1—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, etc., which generated the two test results.

11.6 *Reproducibility*—The pooled reproducibility, R, for cyclohexane extinction has been established as 0.024 absorbance units (see Table 1). The best estimate of the test precision is given by the pooled value and this should be used unless there is a good reason to use a different value. Any other value in Table 1 may be used as an estimate of reproducibility, if justified, such as when testing the same material as, or a material similar to, those in the tables. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility value on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

11.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference materials do not exist for this test method so it is not possible to establish reference (true) test property values to be used in determining bias. Bias, therefore, cannot be determined for this test method.

12. Keywords

12.1 absorbance; carbon black; cyclohexane extinction; UV spectrophotometer

RELATED MATERIAL

German Federal Communication: 23. Mitteilung zur Untersuchung von Kunststoffen, Reinheitsprüfung von Rußen BGesundhBl. 18, 268 (1972).

German Federal Communication: 82. Mitteilung d. BGA BGesundhBl. 15, 268 (1972).

German Federal Communication: 178. Mitteilung der BGA BGesundhBl. 31, 363 (1988).