Standard Test Method for
Carbon Black—Solvent Extractables[1]

1. Scope

1.1 This test method covers the procedure for measuring carbon black solvent extractables.

1.1.1 This test method determines the total material extracted from carbon black by toluene under specified conditions. The procedure is also applicable to other solvents such as cyclohexane, etc.

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.* For a specific precautionary statement, see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3051 Practice for Carbon Black—Stating the Precision of ASTM Test Methods (Withdrawn 1990)[3]

D3396 Practice for Carbon Black— Measuring the Precision of ASTM Test Methods (Withdrawn 1990)[3]

3. Summary of Test Method

3.1 A specimen of carbon black is extracted for 48 h (or other specified time) in a Soxhlet extractor, the solvent removed by controlled temperature evaporation, and the extracted residue determined gravimetrically. If the carbon black extractables are to be subjected to additional analysis, see Section 7.

4. Significance and Use

4.1 The carbon black extract test procedure provides a uniform and precise method for the gravimetric determination of organic-solvent extractables on carbon black. Accuracy and precision are acceptable for most specification or regulatory purposes, or both. However, carbon black with very low extract (less than 0.02 %) may require a more rigorous extraction procedure. The actual chemical composition of the extract can vary significantly with the type and origin of the carbon black.

5. Apparatus

5.1 *Soxhlet Extraction Apparatus,* with interchangeable ground joints, 40 mm I.D. extraction tube, and 250-cm^3 flask.[4]

5.2 *Extraction Thimble,* glass, 35-mm diameter by 90-mm height, with coarse porosity fritted disc.[5] When filled with 50 cm^3 of toluene, drainage time for the first 40 cm^3 should not exceed 95 s. Discard thimbles taking longer.

5.3 *Heater,* for extraction flask, with adjustable heat input. A hot plate extraction heater or hemispherical heating mantle is preferred.

5.4 *Filter Funnel,* glass fritted disc type, with medium porosity fritted disc.

5.5 *Filter Flask,* 125 or 250 cm^3.

5.6 *Water Bath,* or other suitable warming means for evaporating solvent from extract at a controlled temperature of 40 to 50°C.

5.7 *Container,* suitable for final solvent removal and weighing of residue. A lightweight aluminum dish 63 by 17.5 mm, a TFE-fluorocarbon cup or a 50-cm^3 glass beaker is suggested.[6]

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[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.

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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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Fisher Scientific No. 9-556B available from Fisher Scientific Co., 203 Fisher Ave., Pittsburgh, PA 15219, has been found satisfactory. The use of Kontas K-586100 (glass jacketed) available from Kontas Glass Co., 8000 Spruce St., Vineland, NJ 08360, simplifies insulating the apparatus and reduces the possibility of breakage.

[5] Fisher Scientific 9-653B available from Fisher Scientific Co., has been found satisfactory.

[6] Fisher Scientific No. 8-732-5C (aluminum dish) available from Fisher Scientific Co., and Ventron No. 2034 (teflon cup) available from Ventron Instrument Corp., Cahn Division, 16707 S. Carmenita Rd., Cerritos, CA 90701, have been found to be satisfactory.

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

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

5.10 *Desiccator.*

5.11 *Analytical Balance,* with a sensitivity of ±0.1 mg.

6. Reagents and Materials

6.1 *Toluene,* having a residue after evaporation less than 10 ppm (preferably less than 2 ppm).[7]

6.2 *Nitrogen.*

6.3 *Glass wool,* extracted with toluene, air-dried, followed by drying in a convection oven at 125°C for 1 h. Store in a closed container until ready to use.

6.4 *Aluminum Foil.*

7. Precautions

7.1 For safety reasons, conduct the extraction and solvent evaporation in an approved fume hood. Carbon blacks may contain polynuclear aromatic compounds, some of which are carcinogens. These compounds, when present, are so strongly bound to the carbon black that they are biologically inactive. However, they may be removed by extraction with toluene or other appropriate solvents. Avoid skin contact with solvent extracts from such carbon blacks.

7.2 For most pelleted blacks, the depth of 60 mm in the thimble should provide a sample of 15 to 25 g. Unpelleted blacks should be predensified sufficiently that this depth will require a sample of at least 10 g.

7.3 Do not use joint lubricant on the ground joints. To minimize vapor loss by leakage, the joints may be sealed with TFE-fluorocarbon inserts or wrapped with TFE-fluorocarbon laboratory tape.

7.4 New thimbles should be washed before use by suction with hot HCl and then rinsed with distilled or deionized water to remove loose particles of foreign matter such as dust. Thimbles are conveniently cleaned by back washing under suction with appropriate media (water, acetone, etc.). If the fritted disc becomes fouled with carbon black, it may be necessary to burn it out by slowly heating the thimble from room temperature to 500°C in a muffle furnace. One hour is usually sufficient. The use of chromic acid cleaning solution is not recommended. The permeability of the clean thimble should be checked periodically. Discard thimbles with drainage times exceeding the 95 s specified under 5.2.

7.5 To prevent contamination, it is recommended that all glassware used in this procedure be reserved for the extraction of carbon black. If this is not feasible, the glassware must be extracted before use in accordance with 7.6.

7.6 New glassware, including thimbles, must be extracted, repeatedly if necessary, with toluene or other solvent until the blank is acceptable (preferably less than 0.3 mg/150 cm^3 solvent).

7.7 Two or three glass beads may be added to the extraction flask to promote smooth boiling. Do not use porous boiling chips.

7.8 An extraction must be discarded if there is any visible carbon black in the extraction flask or if the thimble does not drain properly and overflows at any time during the extraction.

7.9 The nitrogen should be delivered through a glass or metal tip. Do not allow plastic or rubber to contact the solvent.

7.10 To protect from dust, a loose cover over the evaporation vessel is recommended.

7.11 Solvent evaporation may be conducted in a vacuum evaporator, if available.

7.12 Any further analysis of the extracted material typically requires that it be in solution. Complete re-solution of the dry extract may be difficult or impossible. Therefore, if further analysis is to be performed, an additional sample of the carbon black should be extracted and solvent removal carried only to the desired reduced volume. It is recommended that exposure to strong light, especially sunlight, be avoided during extraction and subsequent handling, and that the extract be blanketed with inert gas (nitrogen) if retained for any length of time.

7.13 It has been found that there may be significant variation in the amount of residue found in different bottles of toluene bearing the same lot number. Consequently, blanks should be run on each bottle.

8. Procedure

8.1 Dry an adequate specimen of the carbon black for 1 h in the gravity-convection oven set at 125°C in an open container of suitable dimensions so that the depth of the black is no more than 10 mm. Cool and store the sample in a desiccator.

8.2 Weigh, into a clean, dry extraction thimble, sufficient dried carbon black to fill the thimble to a depth of approximately 60 mm. Record the weight of black to 0.1 g. Place a plug of glass wool above the black. Insert the thimble into the extraction tube, place 150 cm^3 of toluene in the extraction flask, and assemble the apparatus (see 7.1 – 7.6).

8.3 Extract continuously for 48 h (or other time if specified), heating so that the cycle time between siphonings is 4 to 5 min (see 7.7). Wrap aluminum foil loosely around the extraction tube and upper part of the flask to reduce heat loss. Terminate the extraction with the extraction cup nearly full to reduce the quantity of solvent to be subsequently evaporated from the residue in the extraction flask (See 7.8).

8.4 Filter the contents of the extraction flask into a clean filter flask using moderate suction, rinsing the extraction flask and filter funnel with several small portions of toluene.

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[7] Distilled-in-glass toluene available from Burdick and Jackson, 1953 T.S. Harvey, Muskegan, MI 49442 and Photrex grade available from J.T. Bauer Chemical Co., 223 Red School Ln., Phillipsburg, NJ 08865 are preferred products. Analytical reagent grade available from Mallinckrodt, Inc., 675-T McDonnell Blvd., St. Louis, MO 63134 has generally been found acceptable.

8.5 Evaporate the contents of the filter flask at 40 to 50°C until approximately 10 cm^3 remains. Direct a stream of dry nitrogen onto the surface of the solution to hasten evaporation (see 7.9 – 7.11).

8.6 Transfer the remaining solution to the previously dried and weighed (to 0.1 mg) final container, rinsing the flask with several small portions of toluene. Continue evaporation to essentially dryness (see 7.12).

8.7 To complete solvent removal to constant residue mass, place the container in a vacuum oven at 40°C. Setting the container on the floor of the oven should be avoided; a non-conductive support or equivalent should be used. Slowly reduce the oven pressure to approximately 3.3 kPa and thereafter hold at that level. After a suitable time (usually 45 min to 1 h), cool the container and contents to room temperature in a desiccator and weigh to 0.1 mg. Return to the oven for 15 min, cool, and reweigh. Repeat as necessary until the mass is constant. Subtract the original mass to obtain the mass of residue.

8.8 Make duplicate blank determinations on each bottle of toluene placed in use. Follow this procedure exactly but omit the carbon black (see 7.13).

9. Calculation

9.1 Calculate the mass percent extractables to the nearest 0.01 % as follows:

$$\text{Extractables, mass \%} = \frac{R - B}{W} \times 100 \qquad (1)$$

where:

R = mass of residue, g,
B = mass of residue from blank, g, and
W = mass of carbon black sample, g.

10. Report

10.1 Report the following information:

10.1.1 Proper identification of the sample,

10.1.2 The solvent used,

10.1.3 The extraction time, and

10.1.4 The result obtained, reported to the nearest 0.01 %.

11. Precision and Bias

11.1 These precision data are based on single tests of three samples by five laboratories on two days. The range of the samples studied was from about 0.06 % to about 1.02 %. Precision is expressed in absolute terms.

NOTE 1—The precision is based on limited data in that only five laboratories participated in the interlaboratory study.

11.2 In test method statistical terminology, bias is the difference between an average test value and the reference or true test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by this test method. Bias, therefore, cannot be determined.

11.3 *Repeatability* (*Single-Operator*)—The repeatability has been estimated to be ±0.06 percentage points. Two test results are significantly different if their difference exceeds 0.09 percentage points.

11.4 *Reproducibility* (*Multilaboratory*)—The reproducibility has been estimated to be ±0.09 percentage points. Two test results are significantly different if their difference exceeds 0.13 percentage points.

NOTE 2—This precision statement was developed following the guidelines presented in Practices D3051 and D3396.

12. Keywords

12.1 carbon black; cyclohexane; extract; silica gel adsorbent aromatic/polar fractions; solvent extractables; toluene