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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Carbon Black In Olefin Plastics[1]

This standard is issued under the fixed designation D 1603; 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 determination of the carbon black content in polyethylene, polypropylene, and polybutylene plastics. Its use with acrylic or other polar monomer modifications which might affect the accuracy is not recommended. Determinations of carbon black are made gravimetrically after pyrolysis of the sample under nitrogen. This test method is not applicable to compositions that contain nonvolatile pigments or fillers other than carbon black.

1.1.1 This test method is not applicable to materials containing brominated flame retardant additives at the end.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are given for information only.

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.*

NOTE 1—This test method is similar to ISO 6964-1986(E) in title only. The technical content is significantly different.

2. Referenced Documents

2.1 *ASTM Standards:*

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[2]

2.2 *ISO Standard:*

ISO 6964-1986(E) Polyolefin Pipes and Fittings—Determination of Carbon Black by Calcination and Pyrolysis—Test Method and Basic Specification[3]

3. Significance and Use

3.1 The information provided by this test method is useful for control purposes and is required for calculation of optical absorptivity.

4. Apparatus

4.1 *Electric Furnace*, at least 20 cm (8 in.) long suitable for use with the tubing described in 4.2.

4.2 *Tubing*,, [4] 2.9 cm (⅛ in.) in diameter, approximately twice as long as the furnace described in 4.1.

4.3 *Stoppers*—Two rubber or neoprene stoppers, to fit the tube described in 4.2, unless the tube is fitted with ground joints and mating connectors.

4.4 *Glass Tubing*, 10-mm, of sufficient amount, and matching rubber or plastic tubing for connections.

4.5 *Combustion Boat*, approximately 8 by 1.9 by 1.3 cm (3½ by ¾ by ½ in.). Glazed porcelain, quartz high-silica glass, or platinum is suitable.

NOTE 2—A loose-fitting cover for the combustion boat is optional. If used, it shall be considered a part of the boat and handled and weighed with it.

4.6 *Iron-Constantan Thermocouple*, and a potentiometer or millivoltmeter suitable for determining temperatures in the range 300 to 700 C.

4.7 *Flow Meter*,, [5] suitable for measuring gas flow at rates of 1 to 10 L/min.

4.8 *Traps*, three glass traps with removable ground-glass connected heads and 10-mm diameter inner and connecting tubes.

NOTE 3—Only one trap is required if the entire apparatus train is placed in a fume hood. None is required if in addition, nitrogen of sufficient purity is used and produced by the alternative means provided in Section 5.

4.9 *Drying Tube*—A U-shaped drying tube, having an inside diameter of 20 mm or larger, fitted with ground glass or neoprene stoppers.

4.10 *Glass Wool*.

4.11 *Desiccator*, with desiccant.

4.12 *Bunsen Burner*.

4.13 *Balance*—An analytical balance having a sensitivity of 0.0001 g.

4.14 *Weights*—A set of Class S weights for use with the balance.

5. Reagents and Materials

5.1 *Carbon Dioxide, Solid* (Dry Ice).

[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

In 1993, the scope was revised to clarify limitations related to use of this test method with materials containing brominated additives and to include an ISO equivalency statement. Keywords were also added.

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[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[4] Pyrex, Vycor, or equivalent tubing has been found satisfactory for this purpose.

[5] Precision Bore Rotometer Tube No. 2B-25, available from the Fischer & Porter Co., County Line Road, Warminster, PA 18974 has been found satisfactory.

NOTE 4—The solid carbon dioxide and the trichloroethylene are not required if the entire apparatus train is placed in a fume hood.

5.2 *Desiccant*, such as anhydrous calcium chloride ($CaCl_2$).

5.3 *Nitrogen*, prepurified, having oxygen content below 0.01 %. As a safeguard against accidental leakage, contamination, or inadequate purity, the gas shall be further purified by one of the following procedures:

5.3.1 Passage of the nitrogen through a glass trap inserted ahead of the drying tube (see), filled approximately one third full of potassium hydroxide - pyrogallol solution made to contain 5 g of pyrogallol and 50 g of KOH in 100 mL of water. Technical grade, or better, reagents are satisfactory.

5.3.2 Insertion of a plug, or roll, of clean copper tinsel, foil, or wire 7.5 to 10 cm (3 to 4 in.) long into the combustion tube ahead of the sample (see Fig. 1) so that it is completely within the heated region of the furnace. Take care to prevent channeling of the nitrogen through the plug. The extent of blackening of the copper may be taken as a guide for determining when the plug should be renewed.

5.3.3 Passage of the nitrogen through a combustion tube filled to a length of 15 cm (6 in.) or greater with clean copper tinsel, foil, or wire, and maintained in a furnace at a temperature around 500 C.

5.3.4 The need for the procedures described is eliminated if gas having an oxygen content of less than 0.002 % (20 ppm) is used.

5.4 *Trichloroethylene*, technical grade (Note 4).

6. Procedure

6.1 Assemble the apparatus as shown in Fig. 1. Both cold traps following the combustion tube shall contain trichloroethylene, but only the first need be cooled with solid carbon dioxide. Alternatively, the entire apparatus may be placed in a fume hood and the two traps following the combustion tube omitted. Fill the drying tube with anhydrous $CaCl_2$ or other suitable desiccant. Hold between loose plugs of glass wool.

6.2 Heat a clean combustion boat to red heat in a bunsen flame; then transfer the boat to the desiccator and allow it to cool over fresh desiccant for not less than 30 min.

6.3 Remove the boat from the desiccator and weigh it to nearest 0.0001 g. Immediately place 1.0 ± 0.1 g of the ethylene plastic under test in the boat and quickly weigh to the nearest 0.0001 g.

6.4 Heat the furnace to a constant temperature of 600°C. Adjust the rate of nitrogen flow to 1.7 ± 0.3 L/min. Open the inlet end of the 2.9-cm diameter tube, quickly place the combustion boat with the sample into the tube at the center of the furnace, and adjust the thermocouple so that the weld is touching the boat. Insert copper plug, if this is used (see 5.3.2). Quickly close the furnace and allow heating to proceed for at least 15 min.

NOTE 5—The exact temperature of heating is not critical in the range 500 to 700°C, although a heating time as long as 30 min is desirable at the lower temperature. If desired the sample may be put in the furnace at 300°C or less and the temperature of the furnace then programmed for sample heating to 350°C in 10 min, 450°C in another 10 min, and 500°C after a total of 30 min, finally heating at 500°C for an additional 15 min.

6.5 Move the tube or furnace so that the boat is no longer in the heated zone of the furnace and allow 5 min for cooling, while maintaining the flow of nitrogen. Remove the copper plug, if present, and the boat through the inlet end of the tube and allow it to cool in the desiccator for at least 30 min. Take care that the boat does not become contaminated from any deposits on the walls of the tube. Then quickly reweigh the boat and its contents to the nearest 0.0001 g.

6.6 Make all determinations in duplicate. If carbon black measurements are to be made at values less than 1 %, then the sample boat with the residue of the tube furnace burn shall be placed in a muffle furnace for approximately 10 min to oxidize the carbon black. After heating, cool the sample boat in a desiccator until it is at room temperature. Weigh the boat plus contents to the nearest 0.0001 g (W_o).

6.7 Make all determinations in duplicate.

7. Calculation

7.1 Calculate the carbon black content as follows:

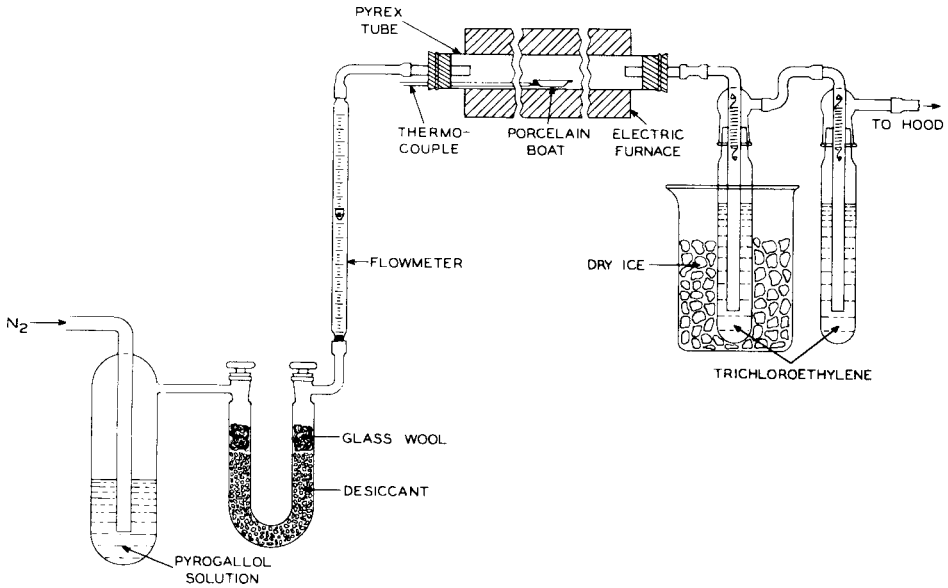

FIG. 1 Assembly of Apparatus

$$\text{Carbon black, \%} = \frac{(W_r - W_o)}{W_s} \times 100 \quad (1)$$

where:
W_r = mass of residue (g) after burning innitrogen,
W_o = mass of residue (g) afterburning in air, and
W_s = mass of sample(g).

8. Report

8.1 Report the following information:

8.1.1 Identity of the sample,

8.1.2 The individual values calculated as described in Section 7, and

8.1.3 The average of the values reported in 8.1.2.

9. Precision and Bias [6]

9.1 The interlaboratory precision, exclusive of sampling error, was determined by an interlaboratory study with seven laboratories to be ±0.156 % (3S) as defined in Practice E 177, over the range 2.5 to 3.0 % carbon black, and ±0.309 % (3S) over the range 35 to 55 % carbon black.

10. Keywords

10.1 carbon black; gravimetric; plastics; polyolefins

[6] Supporting data for this test method is available from ASTM Headquarters. Request RR: D20-1027.