



## Standard Test Method for Carbon Black In Olefin Plastics<sup>1</sup>

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 ( $\epsilon$ ) 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<sup>2</sup>

#### 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<sup>3</sup>

### 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.

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>3</sup> Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

### 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*,<sup>4</sup> 2.9 cm ( $\frac{1}{8}$  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 ( $3\frac{1}{2}$  by  $\frac{3}{4}$  by  $\frac{1}{2}$  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*,<sup>5</sup> 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).

<sup>4</sup> Pyrex, Vycor, or equivalent tubing has been found satisfactory for this purpose.

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