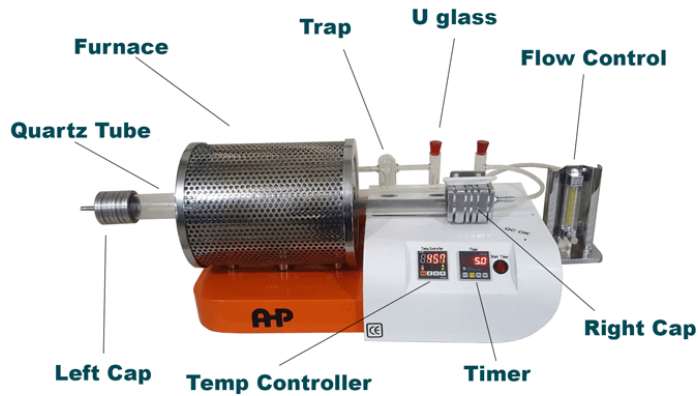


ASTM D 1603 Carbon Black Content in Olefin Plastics / Testing Equipment

Description



Right Cap on Sliding Mechanism Moves Right and Left Along with Quartz Tube

5 Apparatus

- 5.1 Electric Furnace, at least 20 cm (7.9 in.) long suitable for use with the tubing described in 5.2.
 - 5.2 High Temperature Glass Combustion Tube, 4 of appropriate diameter and approximately twice as long as the furnace described in 5.1.
 - 5.3 Stoppers—Two rubber or neoprene stoppers, to fit the tube described in 5.2, unless the tube is fitted with ground joints and mating connectors.
 - 5.4 Glass Tubing, approximately 10 mm (0.39 in.) in diameter, of sufficient amount, and matching rubber or plastic tubing for connections.
 - 5.5 Combustion Boat, approximately 8 by 1.9 by 1.3 cm (3.15 by 0.75 by 0.51 in.). Glazed porcelain, quartz high-silica glass, or platinum is suitable.
 - 5.6 Iron-Constantan Thermocouple, and a potentiometer or millivoltmeter suitable for determining temperatures in the range 300 to 700°C (572 to 1292°F).
 - 5.7 Flow Meter, suitable for measuring gas flow at rates of 1 to 10 L/min.
 - 5.8 Traps, three glass traps with removable ground-glass connected heads and 10-mm (0.39-in.) 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 6.
- 5.9 Drying Tube—A U-shaped drying tube, having an inside diameter of 20 mm (0.79 in.) or larger, fitted with ground glass or neoprene stoppers.
 - 5.10 Glass Wool.
 - 5.11 Desiccator, with desiccant.
 - 5.12 Bunsen Burner or Muffle Furnace—Electric resistance-heated or microwave-heated furnace capable of heating the combustion boat to red heat.

NOTE 4—When an electric furnace is used, position it in a wellventilated hood. When a microwave furnace is used, position it within or adjacent to a hood and the exhaust tube vented into the hood to prevent the breathing of byproducts of any combustion. An air flow rate of 2.8 m³/min through the microwave oven is recommended.

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

6 Reagents and Materials

6.1 Carbon Dioxide, Solid (Dry Ice).

NOTE 5—The solid carbon dioxide and the trichloroethylene are not required if the entire apparatus train is placed in a fume hood. 6.2 Desiccant, such as anhydrous calcium chloride (CaCl₂).

6.3 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:

6.3.1 Passage of the nitrogen through a glass trap inserted ahead of the drying tube (see Fig. 1), 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.

6.3.2 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.

6.3.3 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 (932°F).

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

6.4 Trichloroethylene, technical grade (Note 5).

7 Sampling and Test Specs

7.1 The test specimens can be in a variety of forms which fit in the combustion boat but must satisfy the requirements of 8.3. Soiled articles must be washed and printed articles are wiped clean with a suitable solvent.

8 Procedure

NOTE 6—The procedure below assumes that the combustion tube can be easily removed from the furnace. If this is not the case, alternate methods of inserting and removing sample boats are acceptable as long as the temperature, purge time, and flow rate requirements are met.

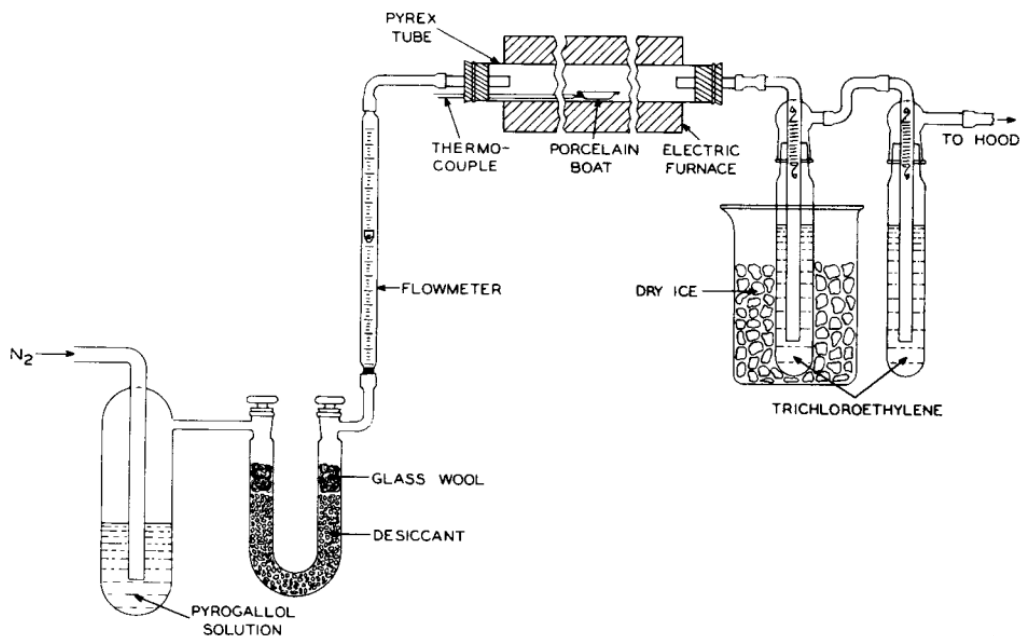


FIG. 1 Assembly of Apparatus

8.1 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.

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

8.3 Remove the boat from the desiccator and weigh it to nearest 0.0001 g (w_1). Immediately place 1.0 $\pm$ 0.1 g of the ethylene plastic under test in the boat and quickly weigh to the nearest 0.0001 g (w_2).

8.4 Heat the furnace to a constant temperature of 600°C (1112°F).

8.5 With the combustion tube removed from the furnace, adjust the rate of nitrogen flow through the tube to 1.7 $\pm$ 0.3 L/min. Open the inlet end of the combustion tube, quickly place the combustion boat with the sample into the tube positioned so the boat will be at the proper temperature when in the furnace. Close the inlet to the tube and allow the nitrogen to flow for a minimum of 5 min to purge oxygen from the system prior to placing the tube in the furnace. If the furnace is controlled by an external thermocouple, adjust the thermocouple so that the weld is in proximity to the boat. Insert the copper plug, if this is used (see 6.3.2). Quickly place the tube into the furnace, close the furnace, and allow heating to proceed for at least 15 min.

NOTE 7—The exact temperature of heating is not critical in the range 500 to 700°C (932 to 1292°F), 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 (572°F) or less and the temperature of the furnace then programmed for sample heating to 350°C (662°F) in 10 min, 450°C (842°F) in another 10 min, and 500°C (932°F) after a total of 30 min, finally heating at 500°C (932°F) for an additional 15 min.

NOTE 8—If the combustion tube and furnace are of adequate size, several sample boats may be tested simultaneously if they are positioned in the furnace within the area which meets the temperature requirements.

8.6 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 (w_3).

8.7 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. This is to correct for the presence of residual inorganic matter in the sample. 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_4).

8.8 Two determinations are made for each sample.

9 Calculation

9.1 Calculate the carbon black content as follows:

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

where:

$W_r = w_3 - w_1 = \text{mass of residue (g) after burning in nitrogen,}$

$W_o = w_4 - w_1 = \text{mass of residue (g) afterburning in air, and}$

$W_s = w_2 - w_1 = \text{mass of sample (g).}$

[Carbon Black Content Test Furnace \(CBC Tester\)](#)

[Key Tech Notes about Carbon Content Testing as per ISO 6964](#)

Category

1. Equipment for Standards
2. Standards