IEC 60811-605 – Physical Tests – Measurement of Carbon Black and/or Mineral Filler in Polyethylene Compounds – Testing Equipment

Description

4 Test method
4.1 General
This part of IEC 60811 shall be used in conjunction with IEC 60811-100. Unless otherwise specified, tests shall be carried out at room temperature.
4.2 Method A – Measurement of carbon black and/or mineral filler content in polyethylene by direct combustion
4.2.1 Sample and test piece preparation
A sample of the outer covering or sheath of sufficient weight shall be taken from one end of the cable. The sample shall be cut in pieces, the dimensions of which shall not exceed 5 mm in any direction.
4.2.2 Test procedure
A combustion boat about 75 mm long shall be heated until it is red hot, allowed to cool in the desiccator for at least 30 min and weighed to the nearest 0,000 1 g. A sample of polyethylene weighing (1,0 ± 0,1) g shall be placed in the boat and the whole weighed to the nearest 0,0001 g. The weight of the boat shall be subtracted to give the weight of the polyethylene to the nearest 0,000 1 g (quantity A).

The boat and the sample shall then be placed in the middle of a hard glass, silica or porcelain combustion tube, with a bore approximately of 30 mm, and (400 ± 50) mm in length. A stopper carrying a thermometer for temperature measurements from 300 °C to 650 °C and a tube for the admission of nitrogen shall then be inserted into one end of the combustion tube so that the end of the thermometer touches the boat. Nitrogen with an oxygen content of less than 0,5 % shall be passed through the combustion tube at (1,7 ± 0,3) l/min and this rate of flow shall be maintained during the subsequent heating. In case of doubt, the oxygen content of the nitrogen shall be limited to 0,01 %.
The combustion tube shall be placed in a furnace and its outlet connected to two cold traps in series, both containing trichlorethylene, the first being cooled with solid carbon dioxide. The outlet tube from the second trap shall lead to a fume hood or the outside atmosphere. Alternatively, it is permissible for the outlet from the combustion tube to lead directly to the outside atmosphere.
The furnace shall then be heated so that the temperature is between 300 °C and 350 °C after about 10 min; about 450 °C after another 10 min and (600 ± 5) °C after a third period of 10 min. This temperature shall then be maintained for 10 min, at the end of which the outlet tube shall be disconnected from the cold traps, if these are used, and the tube containing the boat withdrawn from the furnace and allowed to cool for 5 min, the flow of nitrogen being maintained at the same rate as before.
The boat shall then be removed from the combustion tube through the nitrogen inlet end, allowed to cool in the desiccator for 20 min to 30 min and then re-weighed. The weight of the residue is determined to the nearest 0,000 1 g (quantity B of residue). Subsequently, the boat shall again be introduced into the combustion tube; instead of nitrogen, air or oxygen shall be blown through the tube at an adequate flow rate for a temperature of (600 ±20) °C, and the remaining carbon black shall be

burnt. After it has cooled in the test assembly, the boat shall be removed and weighed again. The mass of the residue is determined to the nearest 0,000 1 g (quantity C of residue).

4.2.3 Expression of results

$$\text{Carbon black content} = \frac{B - C}{A} \times 100\ \%$$

$$\text{Mineral filler content} = \frac{C}{A} \times 100\ \%$$

$$\text{Total filler content} = \frac{B}{A} \times 100\ \%$$

4.3 Method B– Thermogravimetric analysis of the carbon black content in polyolefin compounds

NOTE: This method may be used as an alternative to that in 4.2 when measuring carbon black content of polyethylene. In the event of dispute, the direct combustion method in 4.2 should be used as the reference method.

4.3.1 Principle

Heat a weighed test specimen in a thermogravimetric analyser, starting at 100 °C with 20 K/min up to 950 °C.

NOTE 1 A starting temperature of 100 °C is practical, as the subsequent measurements can be carried out earlier because of the shorter cooling time.

At first, purge the test specimen with dry nitrogen with an oxygen content as described in 4.3.2. When the temperature of 850 °C is reached, switch from dry nitrogen to “synthetic air”. With the switch to air, the combustion of the carbon black that is present will follow.

NOTE 2 Weight loss during the purging stage with nitrogen, up to approximately 800 °C, is due to degradation of the polymer and loss of other minor ingredients.

4.3.2 Reagents

The following reagents shall be used:

– dry nitrogen with an oxygen content of less than 10 mg/kg;

– dry “synthetic air” (a mixture of 80 % nitrogen and 20 % oxygen).

4.3.3 Apparatus

The apparatus comprises:

a) a thermogravimetric analyser;

b) a gas selector;

c) a plotter or other suitable device;

d) an analytical balance.

4.3.4 Procedure

4.3.4.1 Parameters of the apparatus

a) starting temperature 100 °C;

b) heating rate 20 K/min;

c) end temperature 950 °C;

d) weighed test specimen 5 mg to 10 mg;

e) purging gas up to 850 °C dry nitrogen;
f) purging gas from 850 °C to 950 °C dry “synthetic air”.
4.3.4.2 Operation
Operate the apparatus according to the manufacturer’s instructions and the parameters given in 4.3.4.1. Cover the bottom of the crucible with the test specimen, which should consist of a sheet which is as thin as possible. Before the start of the heating period, ensure that an oxygen-free atmosphere is obtained by purging with nitrogen as specified in 4.3.2 for at least 5 min.
4.3.4.3 Evaluation
The share of carbon black in the compound is determined for each single test specimen from the weight change during burning in dry “synthetic air” from 850 °C to 950 °C. The ignition residue at 950 °C is, at the same time, the ash content.

5 Test report
The test report shall be in accordance with that given in IEC 60811-100.

Carbon Black Content Tester According to IEC 60811-605 Method A

- According to IEC 60811-605 Method A
- The test method is based on pyrolytic decomposition of the material in an inert gas ?ow (nitrogen)
- Maximum temp 900C
- Quarts tube
- Rotameter for N2 gas flow adjustment (1,7 ± 0,3) l/min
- Sliding sample carrier
- Alumina sample boat
- Digital temperature and time control
- Glass ware and chemicals according to the standard including trap
- U tube for silica-gel
- Silica-gel pellets
- Pyrogallol and potassium hydroxide powder

Carbon Black Content Test Furnace (CBC Tester)

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Category

1. Equipment for Standards
2. Standards