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Polyolefin pipes and fittings — Determination of carbon black content by calcination and pyrolysis — Test method

Tubes et raccords en polyoléfines — Détermination de la teneur en noir de carbone par calcination et pyrolyse — Méthode d'essai

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, Subcommittee SC 5, *General properties of pipes, fittings and valves of plastic materials and their accessories — Test methods and basic specifications*.

This second edition cancels and replaces the first edition (ISO 6964:1986), which has been technically revised. The main changes compared with the last edition are the following:

— Conventional and microwave muffle furnace test methods, and a thermogravimetric analyzer (TGA) test method have been added.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Polyolefin pipes and fittings — Determination of carbon black content by calcination and pyrolysis — Test method

1 Scope

This document specifies test methods for the determination of the carbon black content of polyolefin compositions used in particular for the manufacture of pipes and fittings, and provides a basic specification for polyethylene pipes and fittings.

This document applies equally to the material for manufacture and to any material taken from a pipe or fitting.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11358-1, *Plastics — Thermogravimetry (TG) of polymers — Part 1: General principles*

3 Principle

It is possible to determine the carbon black content of polyolefin compositions by one of the following three methods:

a) Pyrolysis of the sample at (550 ± 50) °C in a stream of nitrogen for 45 min followed by calcination at (900 ± 25) °C, by using an electrical tube furnace (Method A).

b) Pyrolysis of the sample in a quartz crystal crucible with lid, by using a muffle furnace. According to the type of muffle furnace used there are two different procedures:

   1) Conventional muffle furnace (Method B1): pyrolysis from (325 ± 25) °C to (550 ± 25) °C at 15 °C/min and at (550 ± 25) °C for (10 ± 0,5) min followed by calcination at (900 ± 25) °C.

   2) Microwave muffle furnace (Method B2): pyrolysis at (520 ± 25) °C for (10 ± 0,5) min followed by calcination at (900 ± 25) °C.

c) Pyrolysis of the sample at a constant rate in a thermogravimetric analyzer (TGA) under inert atmosphere at 800 °C followed by calcination under oxidizing atmosphere at 900 °C (Method C).

NOTE 1 Carbon black is decomposed from 500 °C in air or oxygen. Therefore, the loss observed between 500 °C and 700 °C in air or oxygen corresponds to the overall decomposition of the carbon black.

NOTE 2 If the composition contains, in addition to the carbon black, additives likely to decompose at 900 °C, for example ingredients such as calcium carbonate, the calculation can lead to an over-estimation of the carbon black content. If the ash yield is more than 1 %, further investigation can be required.

Calculate the carbon black content from the difference in mass before and after calcination and pyrolysis.

4 Method A: Electrical tube furnace

4.1 Reagents

4.1.1 Dry nitrogen, having an oxygen content less than 20 ppm, under pressure in a steel cylinder provided with a pressure-reducing valve and flow meter.

NOTE If required, the nitrogen can be purified by bubbling the gas through a pyrogallol solution or by passing it over heated copper tinsel, foil, wire or turnings or by passing it through a gas purifier prior to passing into the furnace.

4.2 Apparatus

4.2.1 Silica combustion sample boat, with a sleeve of 50 mm to 60 mm long.

4.2.2 Electric tube furnace, fitted with a device to allow the sample boat to be inserted and withdrawn. The tube is fitted with nozzles to admit the nitrogen and to evacuate the fumes. A diaphragm closed by means of a glass-wool bung placed behind the entry nozzle ensures that the nitrogen stream is distributed uniformly.

The minimum length of the electric tube furnace should be ≥ 3 times the length of the sample boat, and the minimum length of the quartz tube should be ≥ 7 times the length of the sample boat.

4.2.3 Desiccator, capable of holding the silica sample boat (4.2.1).

4.2.4 Balance, with an accuracy of ± 0,1 mg.

4.2.5 Timer, with an accuracy of ± 1 s.

4.3 Procedure

4.3.1 Test conditions

Determination of mass shall be carried out in a room at standard temperature (23 ± 2) °C.

4.3.2 Sampling

Test specimen may be in the form of pellet or finished product. In the last case, the specimen shall be reduced to small fragments.

4.3.3 Conditioning

The test sample shall be conditioned for 24 h at (23 ± 2) °C before preparation.

4.3.4 Silica sample boat preparation for the test

Take the silica sample boat which shall be clean and weighed, and proceed as follows:

Place the silica sample boat in the electric tube furnace and adjust the temperature to (900 ± 25) °C. Once this temperature is reached, leave to calcination for approximately 1 h. Then, dry the silica sample boat in the desiccator at room temperature and weigh. Place again the silica sample boat into the desiccator for 30 min and weigh again. This operation shall be repeated until constant mass, i.e. until two consecutive weighing do not differ by more than 0,5 mg. This weighing is recorded as m.