International Standard 6964

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

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

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Foreword

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International Standard ISO 6964 was prepared by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids.*

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

1 Scope and field of application

This International Standard specifies a test method 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 International Standard applies equally to the material for manufacture and to any material taken from a pipe or fitting.

Section one: Test method

2 Principle

Pyrolysis of a specified quantity of mixture at 550 ± 50 °C in a stream of nitrogen for 45 min and calcination at 900 ± 50 °C.

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

NOTE — 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 may lead to an over-estimation of the carbon black content. It the ash yield is more than 1 %, further investigation may be required.

3 Reagents

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.

If necessary, 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 Apparatus

4.1 Silica combustion sample boat, with a sleeve 50 to 60 mm long, calcined to constant mass at a temperature of at least 900 °C, cooled in a desiccator, and weighed to the nearest 0,000 1 g.

4.2 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.

4.3 Muffle furnace.

4.4 Desiccator, capable of holding the sample boat (4.1).

5 Procedure

5.1 Test conditions

Carry out the weighings at standard laboratory temperature (23 ± 2 °C).

5.2 Test portion

Take three test portions as follows.

Weigh, to the nearest 0,000 1 g, approximately 1 g of the material taken from the consignment or from the wall of the pipe or from the fitting, reduced to small fragments.

5.3 Determination

The determination on each test portion shall be carried out as described in 5.3.1 to 5.3.5.

5.3.1 Place the test portion in the sample boat (4.1) and place the boat in the inlet of the combustion tube of the electric tube furnace (4.2) which has been previously heated to 550 ± 50 °C. Fix the nozzle to the tube inlet and then connect it to the outlet of the nitrogen stream after, if necessary, the nitrogen has passed through the purification system; circulate the nitrogen in the apparatus at a rate of 200 cm^3/min for approximately 5 min.

5.3.2 Move the sample boat towards the centre of the furnace, adjust the nitrogen flow rate to 100 cm^3/min and leave to pyrolyse for approximately 45 min.