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Poliolefinske cevi in fitingi - Določanje vsebnosti saj s kalcinacijo in pirolizo - Preskusne metode in osnovna specifikacija

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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23.040.20 Cevi iz polimernih materialov Plastics pipes

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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

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

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.

5.3.3 At the end of this period, return the sample boat to the cold section of the furnace and leave it there for 10 min while maintaining the flow of nitrogen.

5.3.4 Remove the sample boat from the furnace, allow it to cool in the desiccator (4.4) and weigh under the same conditions as prior to the pyrolysis. Record the mass to the nearest 0,000 1 g.

5.3.5 Place the sample boat in the muffle furnace (4.3) at a temperature of 900 ± 50 °C and calcine until all traces of carbon black have disappeared. Allow to cool in the desiccator and weigh.

6 Calculation and expression of results

Calculate the carbon black content, expressed as a percentage by mass, from the formula

$$\frac{m_2 - m_3}{m_1} \times 100$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the sample boat plus the test portion after pyrolysis at 500 °C;

m_3 is the mass, in grams, of the sample boat after calcination at 900 °C, with ash where appropriate.

Calculate the arithmetic mean of the carbon black contents determined on the three test portions and report the result to two significant figures.

NOTE — Where appropriate (see note to clause 2), calculate the ash yield, expressed as a percentage of the original mass, from the formula

$$\frac{m_3 - m}{m_1} \times 100$$

where

m is the mass, in grams, of the sample boat;

m_1 and m_3 have the same meanings as above.

Calculate the arithmetic mean of the ash yields determined on the three test portions and report the result to two significant figures.

7 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) full identification of the sample and the method by which it was taken (from the material delivered, or from the wall of the pipe or from the fitting);

c) the carbon black content, expressed as a percentage by mass, calculated as described in clause 6;

— individual values;

— mean value;

d) the presence or absence of ash after calcination; if the value is greater than 1 % of the original mass of the test portion, report the amount and add a note to the report that the carbon black content may be over-estimated;

e) all operating details not specified in this International Standard, or regarded as optional, as well as any incidents likely to have had an effect on the results;

f) date of the test.

Section two: Basic specification for polyethylene pipes and fittings

8 Carbon black

When determined in accordance with section one of this International Standard, the carbon black content shall be 2,5 ± 0,5 % (m/m).