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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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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.