International Standard

ISO 18553

Method for the assessment of the degree of pigment or carbon black dispersion in polyolefin pipes, fittings and compounds

Méthode d'estimation de la dispersion du pigment et du noir de carbone dans les tubes, raccords et compositions à base de polyoléfines

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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 document 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 18553:2002), which has been technically revised. It also incorporates the Amendment ISO 18553:2002/Amd. 1:2007.

The main changes are as follows:

— the microtome method for preparing test pieces has been made the primary method, but the compression method remains an option;

— for testing of materials, samples can now be taken from pellet, moulded sheet or melt index extrudate, using test pieces cut by microtome;

— more prescriptive descriptions of preparing samples and test pieces from pipe and fittings have been given;

— the magnification to be used to assess the size of particles and agglomerates is no longer specified (a magnification appropriate to allow identification of the different size ranges is to be used);

— for the rating of appearance, a more precise evaluation has been given.

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.

Introduction

Thermoplastic products manufactured for pipeline systems are normally pigmented. Typically, fine carbon black particles or pigments are used. These are normally incorporated into the raw material prior to either extrusion of pipe or injection moulding of pipe fittings. The purpose of colouring is to allow identification of the pipeline in service and, in the case of carbon black, to act as protection of the polymer from degradation by ultra-violet light if the product is stored outdoor or used for outdoor service. It is important that the carbon black or pigment particles are correctly dispersed in the polymer, and hence the final product, to ensure that the physical, mechanical and surface protection properties are maintained. Correct dispersion can also be an indication that antioxidants and ultra-violet stabilizers are correctly dispersed, and that the size of agglomerates or particles is not excessive.

This method provides procedures for assessing the degree of dispersion by physical measurement of the size of the dispersed particles and arithmetic grading of the particle size distribution. It also provides photographs for comparison with microscopic images of samples taken from raw-material compounds or products in order to judge subjectively the acceptability of carbon black or pigment dispersion.

A recommended limit of particles/agglomerate size grading and an acceptable rating of appearance is given in Annex D.

Method for the assessment of the degree of pigment or carbon black dispersion in polyolefin pipes, fittings and compounds

1 Scope

This document describes a method with two procedures for the assessment of carbon black or pigment particle and agglomerate size, and the rating of dispersion in polyolefin pipes, fittings and compounds.

The method is applicable to polyolefin pipes and fittings, as well as raw material in pellet form, with the choice of procedure to be determined by the referring specification.

The method is applicable to carbon black or pigmented polyolefin pipes, fittings and compounds with a carbon black content of less than 3 % by weight, and pigment content of less than 5 % by weight.

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 293, *Plastics — Compression moulding of test specimens of thermoplastic materials*

ISO 1133-1, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

Small samples of raw-material pellet, melt index extrudate, compression moulded sheet, or material removed from the pipe or fitting are taken for assessment. Microtome slices are cut from the samples for examination. Alternatively, samples are heated and compressed between microscope slides for examination.

The test pieces produced are examined microscopically and the sizes of particles and agglomerates are measured, recorded and graded by comparison with a tabulated grading system (see Table A.1).

A particle/agglomerate size grading is determined from an average of the gradings determined for six test pieces.

If required, a rating of the appearance of the dispersion is determined by comparison with photomicrographs (see Annex B).

5 Apparatus

5.1 General

5.1.1 Microscope, capable of producing suitable magnification (see 6.2.1 and 6.2.2) with orthogonal travel, a standard calibrated graticule capable of measuring the particle and agglomerate size, and lighting adequate to avoid optical effects. Alternatively, microscopes with a digital camera system may be employed with software to facilitate measurement, and calibrated with a stage micrometer.

5.1.2 Glass microscope slides, 1 mm thickness is suitable, with a thin cover slip.

5.2 For the microtome procedure (see 6.1.2)

5.2.1 Microtome, capable of producing a slice of the required thickness (see 6.1.2).

5.3 For the compression procedure (see 6.1.3)

5.3.1 Oven or hotplate or other type of heating device, capable of operating at a controlled temperature between 150 °C and 210 °C.

5.3.2 Scalpel, for cutting out specimens.

5.3.3 Press, **weights** or **spring clips**, to maintain pressure.

6 Procedure

6.1 Test piece preparation

6.1.1 Two methods of preparation of test pieces are described for raw materials: a microtome procedure and alternatively a compression procedure. The microtome procedure is the preferred procedure and in case of a dispute it shall apply.

For the preparation of test pieces from products (pipes, fittings), the microtome procedure shall be used. Cutting of the samples from which the test pieces are taken shall take place on a clean surface, to minimize the possibility of extraneous contamination.

NOTE The compression procedure will not necessarily give the same result as using the microtome procedure because of the movement of material during compression, and the potential spreading of agglomerates. An example illustrating such a spreading of agglomerates is given in Annex E.

6.1.2 Microtome procedure

6.1.2.1 Testing samples of raw material

The following three procedures are applicable, but in case of dispute, measurement on pellets shall apply.

a) Test pieces are taken by slicing a sample of pellet. A nominal thickness of (20 ± 5) µm for black materials and (40 ± 10) µm for pigmented materials shall be produced. Six slices from six pellets shall be taken to produce six test pieces with an area of a minimum of 12,5 mm^2 each for preparation for examination in accordance with 6.2. However, in the case of pellet of <4 mm diameter, test pieces shall be combined to meet the minimum required area of 12,5 mm^2. A maximum of two slices can be taken from one pellet, but the slices shall be at least 100 µm apart for black materials, and at least 200 µm for pigmented materials.

   NOTE 1 A slice from a pellet of cylindrical shape and 4 mm diameter provides an area of 12,5 mm^2.

b) Alternatively, a sheet of 4 mm thickness shall be compression moulded in accordance with ISO 293, using sufficient pellets to minimize flow of material. Using the microtome, slices of the material of the specified thickness and distance apart [see 6.1.2.1 a)] shall be taken from the cross-section of the sheet to produce six test pieces of a minimum of 12,5 mm^2 each for examination.

c) Alternatively, following a melt flow rate (MFR) test carried out in accordance with ISO 1133-1, samples shall be taken from the remaining material in the cylinder. After completing the melt flow rate test, the die shall be removed and the rest of the material shall be pushed out of the cylinder to produce a suitable sample for test piece preparation. Using the microtome, slices of the material of the specified thickness and distance apart [see 6.1.2.1 a)] shall be taken to produce six test pieces of a minimum of 12,5 mm^2 each for examination.

NOTE 2 MFR defines time-temperature profile and is carried out to assess the raw material. The production of a sample with the MFR plastometer is an alternative, well-defined method to produce a sliceable sample, which is produced with a low flow influence on material. However, using this method can give a worse result, i.e. a higher grading and worse appearance rating.

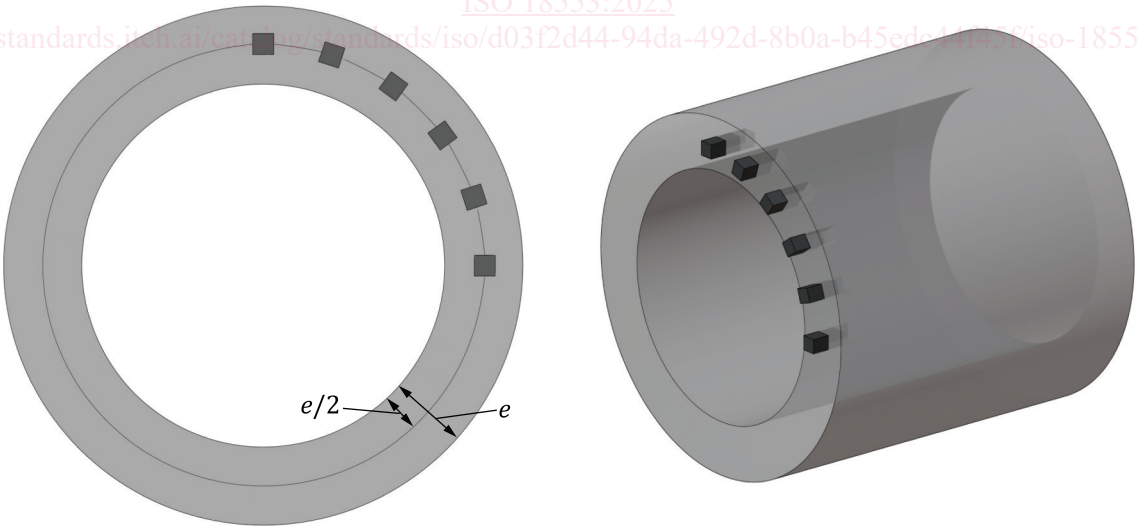
6.1.2.2 Testing of samples of injection moulded fitting products

Cut six through-wall samples perpendicular to the fitting axis of a sufficient size to enable test pieces of the required area to be cut from different parts of the product, taken from the mid wall or full wall depending on the thickness. Microtome six test pieces of an area of a minimum of 12,5 mm^2 each and of thickness (20 ± 5) µm for black samples, and of thickness (40 ± 10) µm for pigmented samples.

6.1.2.3 Testing of samples of pipe

For pipe of ≥63 mm diameter, six evenly spaced samples of an area of a minimum of 12,5 mm^2 each are to be taken from the mid-wall around a 90° quadrant cut from the pipe, see Figure 1. For <63 mm diameter pipes, six samples of an area of a minimum of 12,5 mm^2 each shall be taken from around the circumference, see Figure 2. In case of insufficient area, remaining samples shall be obtained from an axial cross-section at least three times the nominal diameter away from the original position.

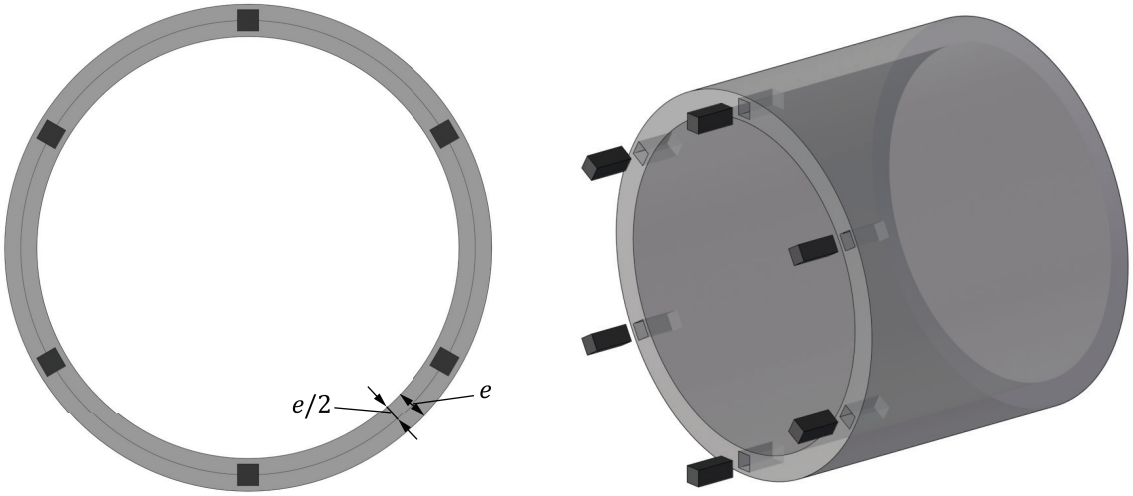
From each sample, microtome a test piece of a minimum of 12,5 mm^2 area perpendicular to the pipe axis to produce slices of (20 ± 5) µm from black samples or (40 ± 10) µm from pigmented samples.



Key

e pipe wall thickness

Figure 1 — Sampling from ≥63 mm diameter pipe



Key

e pipe wall thickness

Figure 2 — Sampling from <63 mm diameter pipe

6.1.2.4 Preparation for examination

Place the six test pieces on one or more clean microscope slides (5.1.2), with each test piece approximately equidistant from its neighbour and from adjacent edges of the slide (see Note). Cover with another clean microscope slide(s) or cover slip.

NOTE Adherence of the test piece can be improved by heating the slide or using a drop of immersion oil or Canada balsam diluted in *p*-xylene, which can improve optical quality. The slices can be compressed with an outside load to release volatiles and to flatten the slices on the glass slide.

6.1.3 Compression procedure for testing raw materials

6.1.3.1 Using a scalpel (5.3.2), cut six samples, each of mass (0,40 ± 0,10) mg for assessing pigment dispersion, or each of mass (0,20 ± 0,05) mg for assessing carbon black dispersion, from six pellets of the raw material. Place the six specimens on one or more clean microscope slides (5.1.2), with each specimen approximately equidistant from its neighbour and from adjacent edges of the slide. Cover with another clean microscope slide(s) or cover slip(s).

NOTE Shims made of metal, or another suitable material can be used to ensure that uniform thickness is obtained. For the test piece mass and thickness given, a film at least 4 mm across is obtained.

6.1.3.2 If an oven (see 5.3.1) is to be used, clamp the two slides together with spring clips (see 5.3.3). Place the clamped slides in the oven, maintained at a temperature between 150 °C and 210 °C and leave for at least 10 min until each specimen is pressed out to a film of thickness (40 ± 10) µm for assessment of pigment dispersion or to a thickness of (20 ± 5) µm for assessment of carbon black dispersion. Remove the slides from the oven, and when they are cool enough to be handled, remove the clips.

6.1.3.3 Alternatively, place the slides on a hotplate or other heating device (see 5.3.1) at a temperature between 150 °C and 210 °C and apply pressure using a press or a weight sufficient to produce a uniform thickness film. Cool before moving the slides for the microscopic examination (see 6.2).