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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

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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18553 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 International Standard cancels and replaces ISO 11420:1996, *Method for the assessment of the degree of carbon black dispersion in polyolefin pipes, fittings and compounds*, and ISO 13949:1997, *Method for the assessment of the degree of pigment dispersion in polyolefin pipes, fittings and compounds*, which have been combined into a single document.

Annexes A and B form a normative part of this International Standard. Annexes C and D are for information only.

Introduction

Thermoplastic products manufactured for pipeline systems are normally coloured. Typically fine carbon black particles or coloured 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 also, in the case of carbon black, to act as protection of the polymer from degradation by ultra-violet light if the product is stored externally or used for external 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 may also be an indication that anti-oxidants and ultra-violet stabilizers are correctly dispersed, and that the size of agglomerates of 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 arithmetically grading the particle size distribution. It also provides photographs for comparison with microscopic images of samples taken from raw-material compound or products in order to judge subjectively the acceptability of carbon black or pigment dispersion.

A recommended limit of grading of particles/agglomerate size and a list of the photographs from annex B for an acceptable rating of appearance is given in annex D.

It is noted that this method supersedes and merges previously published individual methods for assessment of carbon black and pigment dispersion (see the foreword).

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

1 Scope

This International Standard describes a method with two procedures for the assessment of pigment or carbon black particle and agglomerate size and degree 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 pigmented polyolefin pipes, fittings and compounds with a carbon black content of less than 3 %.

2 Principle

Small samples of raw-material pellet or material removed from the pipe or fitting are heated and compressed between microscope slides. Alternatively, a microtome slice can be taken.

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

3 Apparatus

3.1 General

3.1.1 Microscope, capable of producing suitable magnification, see 4.2 and 5.2, with orthogonal travel, a standard calibrated graticule capable of measuring the particle and agglomerate size, and lighting adequate to avoid optical effects.

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

3.2 For the compression procedure (see 4.1.1)

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

3.2.2 Scalpel, for cutting out specimens.

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

3.3 For the microtome procedure (see 4.1.2)

3.3.1 Microtome, capable of producing a slice of the required thickness (see 4.1.2).

4 Procedure

4.1 Specimen preparation

Two methods of preparation of test specimens are described: a compression procedure and a microtome procedure.

4.1.1 Compression procedure

4.1.1.1 Using a scalpel (3.2.2), cut six specimens, each of mass 0,6 mg ± 0,2 mg for assessing pigment dispersion, or each of mass 0,20 mg ± 0,10 mg for assessing carbon black dispersion, from different parts of the product to be analysed (see notes 1, 2, and 3). Place the six specimens on one or more clean microscope slides (3.1.2), with each specimen approximately equidistant from its neighbour and from adjacent edges of the slide (see note 4). Cover with another (or other) clean microscope slide(s) or cover slip(s) (see note 5).

NOTE 1 It should be noted that difficulty will be encountered with the microscopic examination of specimens which are too thick.

NOTE 2 The specimens are preferably cut along different axes of the product.

NOTE 3 It is recommended that cutting out the specimens take place on a clean surface to minimize the possibility of extraneous contamination.

NOTE 4 Adherence of the specimens may be improved by heating the slide or using a drop of immersion oil or Canada balsam.

NOTE 5 Shims made of metal or another suitable material may be used to ensure that uniform thickness is obtained. For the specimen mass and thickness given, a film at least 4 mm across is obtained (see note 1).

4.1.1.2 If an oven (see 3.2.1) is to be used, clamp the two slides together with spring clips (see 3.2.3). Place the clamped slides in the oven, for instance (see 3.2.1) 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 of at least 60 µm ± 20 µm for assessment of pigment dispersion or to a thickness of 20 µm ± 10 µm for assessment of carbon black dispersion (see note 1 to 4.1.1.1).

Remove the slides from the oven and, when they are cool enough to be handled, remove the clips.

4.1.1.3 Alternatively place the slides on a hotplate or other heating device (see 3.2.1) at a temperature between 150 °C and 210 °C, and apply pressure using a press or a weight sufficient to produce uniform thickness film according to 4.1.1.2.

Cool before removing the slides for the microscopic examination (see 4.2).

4.1.2 Microtome procedure

Cut six specimens from different parts of the product (see note 2 to 4.1.1.1) to produce films of a thickness of 60 µm ± 20 µm for assessment of pigment dispersion or of a thickness of 20 µm ± 10 µm for assessment of carbon black dispersion, and at least 4 mm across in any direction (see note 1 to 4.1.1.1).

Place the six specimens on one or more clean microscope slides (3.1.2), with each specimen approximately equidistant from its neighbour and from adjacent edges of the slide (see note 4 to 4.1.1.1). Cover with another (or other) clean microscope slide(s) or cover slip.

4.2 Microscopic examination

4.2.1 Examination for assessment of degree of dispersion

Examine the particles and agglomerates in each of the six specimens in turn through the microscope (3.1.1) under transmitted light with a recommended magnification of × 100 (see note).

Measure and record the largest dimension of each particle and agglomerate, ignoring those less than 5 µm. Grade according to the size categories given in Table A.1.

NOTE Some pigments may be more visible in polarized light or light of a different intensity. If possible, check that the agglomerates are pigment by varying the light intensity and by using different light sources, for instance transmitted, reflected, or polarized light.

4.2.2 Examination for rating of appearance

If a rating of the appearance is required, examine each specimen in turn through the microscope (3.1.1) under transmitted light with a magnification of at least × 70. Note the appearance of each specimen in comparison with the photomicrographs (see annex B).

5 Expression of results

5.1 Grading of particle and agglomerate size

Using Table A.1, determine the highest particle/agglomerate size grade for each specimen. Calculate the arithmetic mean of the six grades obtained and express the result to a single decimal point, rounded up to the next higher value (see the examples given in annex C).

5.2 Rating of appearance

Note the appearance of each specimen and the overall dominant appearance of the set of specimens.

6 Test report

The test report shall include the following information:

a) all details necessary for complete identification of the material or product tested, including sample type, origin, manufacturer's code number and previous history;

b) a reference to this International Standard;

c) the method of preparation of the film specimens (i.e. compression or microtome) and the thickness of the specimens;

d) the average grading and the individual film gradings of the specimens, in accordance with 5.1;

e) if required, the value of the dominant appearance of the set of specimens and the individual appearance of each film specimen, in accordance with 5.2;

f) details of any deviation from the test method, as well as any incident which may have influenced the results;

g) the date of the test.

Annex A
(normative)

Grading table for particles and agglomerates

(For examples, see annex C)

Table A.1 — Grades based on the largest dimensions of the particles and agglomerates

Grade	Dimensions µm														
	5 to 10	11 to 20	21 to 30	31 to 40	41 to 50	51 to 60	61 to 70	71 to 80	81 to 90	91 to 100	101 to 110	111 to 120	121 to 130	131 to 140	>140
	Maximum number of particles and agglomerates														
0															
0,5	1														
1	3	1													
1,5	6	3	1												
2	12	6	3	1											
2,5		12	6	3	1										
3			12	6	3	1									
3,5				12	6	3	1								
4					12	6	3	1							
4,5						12	6	3	1						
5							12	6	3	1					
5,5								12	6	3	1				
6									12	6	3	1			
6,5										12	6	3	1		
7											12	6	3	1	

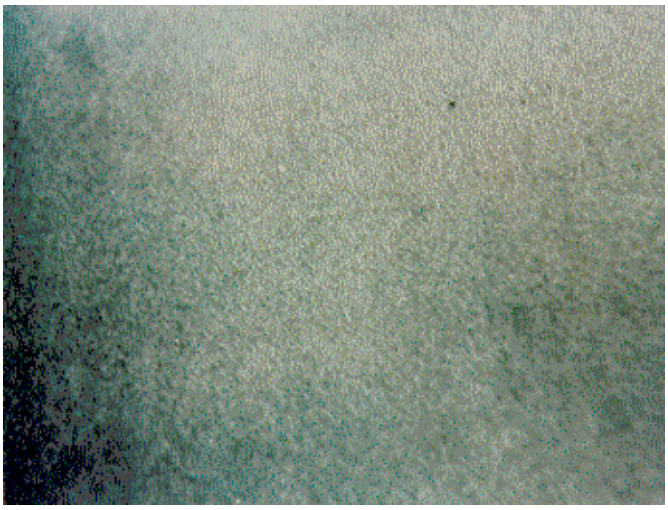
NOTE 1 7 µm corresponds to 0,7 mm under a magnification of × 100 and to 0,49 mm under a magnification of × 70. Similarly, 60 µm corresponds to 6 mm under a magnification of 100.

NOTE 2 All empty upper right cells in the table mean that no particles in the size range are acceptable for the grade in that row.

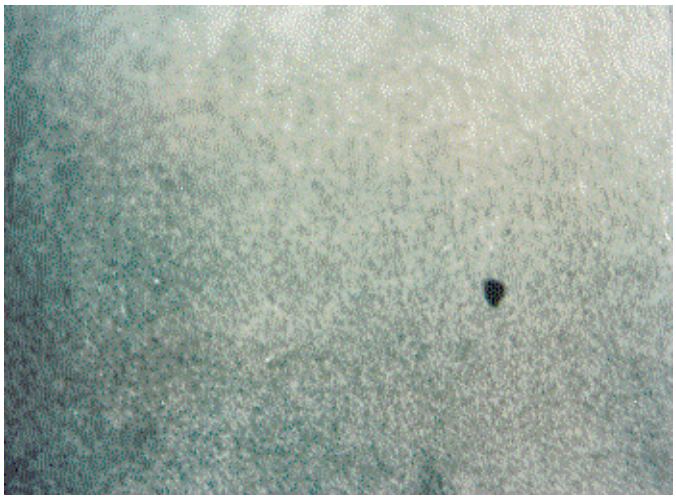
NOTE 3 All empty lower left cells mean no limit to the number of particles in the size range.

Annex B
(normative)

Photomicrographs for evaluation of the appearance of the dispersion



A1



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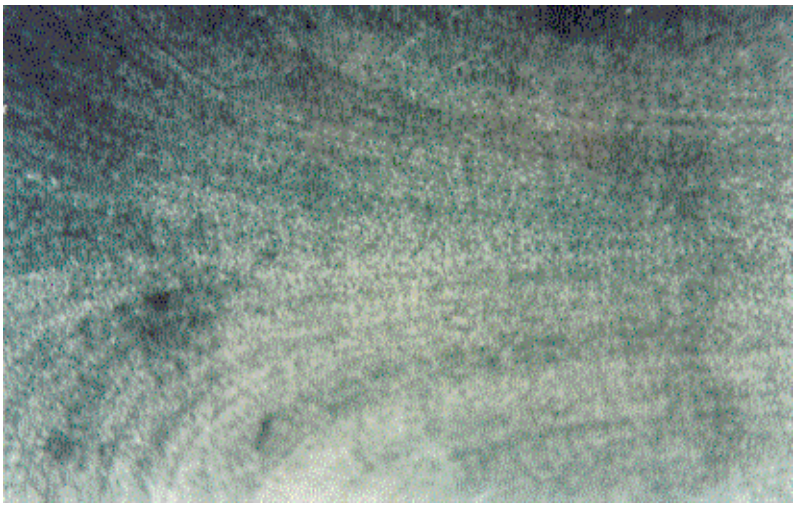

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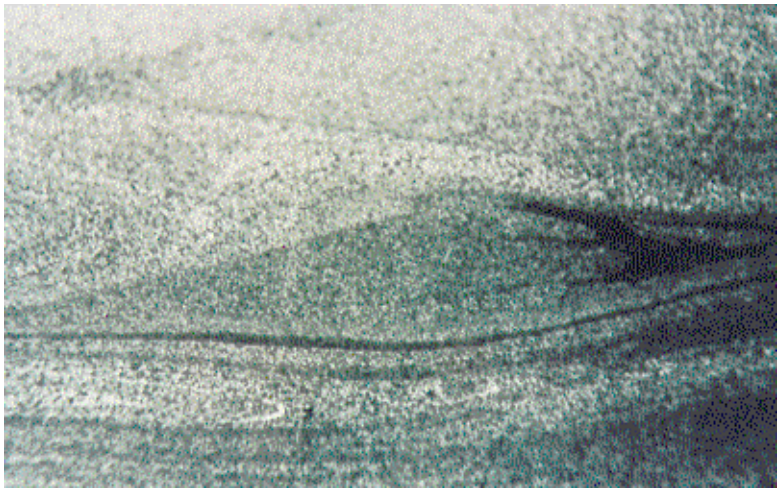

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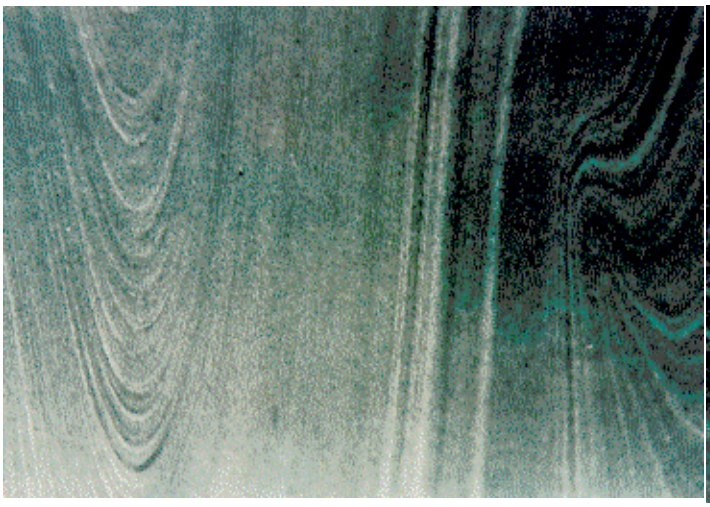
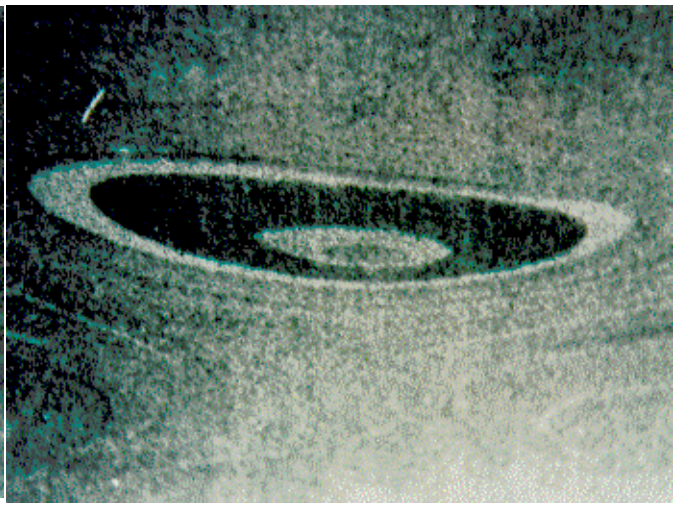
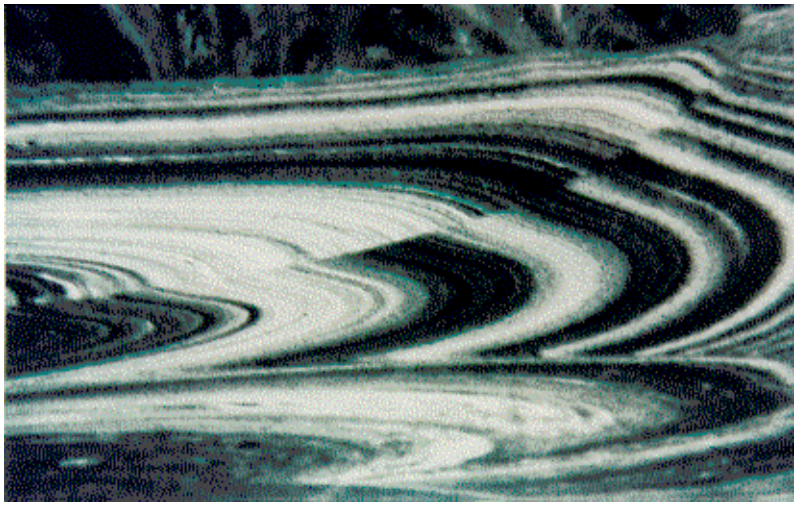
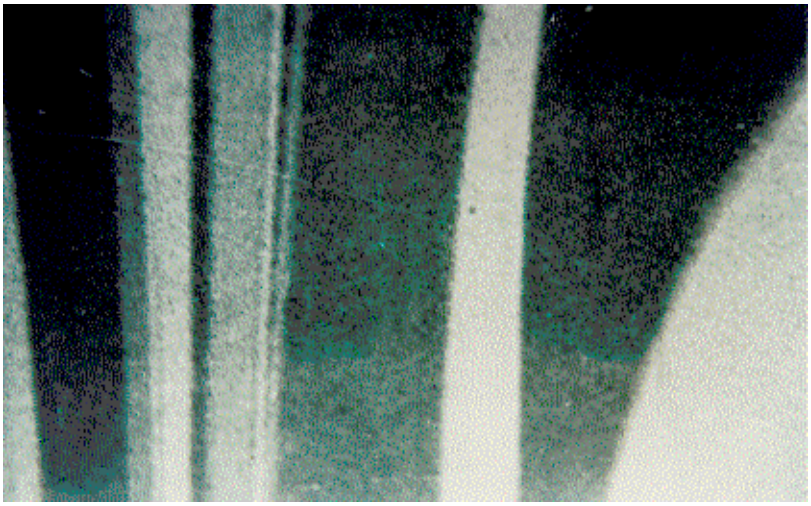




C1 C2



D



E

Annex C
(informative)

Examples of grading of particles and agglomerates

C.1 Example 1

Table C.1 — Number of particles and agglomerates, classified by size, in each of the six specimens, and the resultant grading

Specimen	Dimensions µm				Grade for specimen
	5 to 10	11 to 20	21 to 30	31 to 40	
	Number of particles and agglomerates				
1	3		2	1	2
2	3		5	1	2,5
3		14	2	1	3
4	3		2	2	2,5
5	3		2	4	3
6	3	12	5	7	3,5

Arithmetic mean of the six grades obtained:

(2 + 2,5 + 3 + 2,5 + 3 + 3,5)/6 = 2,75

Result: 2,8 (see 5.1)

C.2 Example 2

Table C.2 — Number of particles and agglomerates, classified by size, in each of the six specimens, and the resultant grading

Specimen	Dimensions µm						Grade for specimen
	5 to 10	11 to 20	21 to 30	31 to 40	41 to 50	51 to 60	
	Number of particles and agglomerates						
1	7	3	9	3		1	3
2	7	3	9	3			3
3	7	3	5	3			2,5
4	19	5		1			2,5
5	19	5			2		3
6						1	3

Arithmetic mean of the six grades obtained:

(3 + 3 + 2,5 + 2,5 + 3 + 3)/6 = 2,833 3

Result: 2,9 (see 5.1)

Annex D
(informative)

Basic specification

The following limits are recommended:

Grading: mean (see 5.1) $\leqslant$ 3.

Appearance rating: not worse than micrograph B in annex B (i.e. only dispersion ratings comparable to photomicrographs A1, A2, A3 and B are acceptable).

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