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

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

INTERNATIONAL ISO

Reference number
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Foreword

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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.

International Standard ISO 11420 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*.

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

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

1 Scope

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

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.

NOTE 1 A similar method may be applied to pigments other than carbon black types and to other polyolefin products such as film and cable coatings, as specified for instance in ISO 13949:—[1], *Method for the assessment of the degree of pigment dispersion in polyolefin pipes, fittings and compounds.*

2 Principle

Small samples of material are removed from the pipe or fitting or raw-material pellet 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. A rating of the appearance of the dispersion is determined by comparison with photomicrographs (see annex B).

3 Apparatus

3.1 For the compression procedure (see 3.3 and 4.1.1).

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

3.1.2 Glass microscope slides: 1 mm thickness is suitable.

3.1.3 Press, weights or spring clips, to maintain pressure.

3.2 For the microtome procedure (see 3.3 and 4.1.2)

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

3.3 For either the compression or the microtome procedure

3.3.1 Microscope, capable of producing a magnification of at least × 100, with orthogonal travel, a standard calibrated graticule capable of measuring the particle and agglomerate size, and lighting adequate to avoid optical effects.

3.3.2 Scalpel, for cutting specimens.

1) To be published.

4 Procedure

4.1 Specimen preparation

4.1.1 Compression procedure

4.1.1.1 Using a scalpel (3.3.2), cut six specimens, each of mass 0,25 mg ± 0,05 mg, from different parts of the product to be analysed (see notes 2, 3 and 4). Place the six specimens on one or more clean microscope slides (3.1.2), with each specimen approximately equidistant from its neighbour and from the edges of the slide (see note 5). Cover with another (or other) clean microscope slide(s) (see note 6).

If an oven (see 3.1.1) is to be used, clamp the two slides together with spring clips (3.1.3).

NOTES

2 The mass, size and thickness of specimens are given as a guide. However, it should be noted that difficulty will be encountered with the microscopic examination of specimens which are too thick.

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

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

5 Adherence of the specimens may be improved by heating the slide.

6 Shims made of metal or another material may be used to ensure that a uniform thickness is obtained. For the specimen mass and thickness given, a film between 3 mm and 5 mm across is obtained (see note 2).

4.1.1.2 Place the clamped slides in an oven (see 3.1.1) controlled at a temperature between 150 °C and 210 °C and leave for at least 10 min until each specimen is pressed out to the required thickness of 25 µm ± 10 µm (see note 2).

Remove the slide from the oven and, when it is cool enough to be handled, remove the clips.

4.1.1.3 Alternatively place the slides on a hotplate or other heating device (see 3.1.1) at a temperature between 150 °C and 210 °C, and apply pressure using a press or a weight sufficient to produce uniform-thickness films.

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

4.1.2 Microtome procedure

Using a scalpel (3.3.2), cut six specimens from different parts of the product (see note 3) to produce films of 25 µm ± 10 µm thickness and between 3 mm and 5 mm across (see note 2).

4.2 Microscopic examination

Examine the particles and agglomerates in each of the six specimens in turn through the microscope (3.3.1) under transmitted light with a magnification of at least × 100.

For each specimen, examine the agglomerates to check that they are really carbon black by varying the light intensity and using transmitted and reflected light if possible. 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.

Compare each specimen with the photomicrographs in annex B, for which the magnification is × 70, for uniformity of appearance, taking into account smears and streaks.

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

Express the appearance as the highest comparative rating with the photomicrographs for each specimen (see annex B). Note the overall dominant rating.

NOTE 7 Although both procedures are applicable, the microtome procedure is preferred for assessment of the rating of appearance.

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) the value of the dominant appearance rating and the individual film ratings of the specimens, 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 agglomerates

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

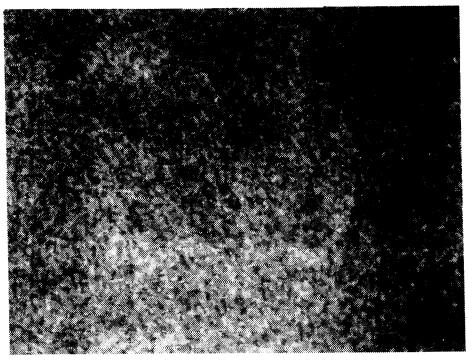
Grade	Dimensions µm														
	5 to 10	11 to 20	21 to 30	31 to 40	41 to 50	51 to 60	61 to 70	71 to 80	81 to 90	91 to 100	101 to 110	111 to 120	121 to 130	131 to 140	141 to 150
	Number of particles and agglomerates														
0	0														
0,5	1	0													
1	≤ 3 + 1		0												
1,5	≤ 6 + ≤ 3 + 1			0											
2	≤ 12 + ≤ 6 + ≤ 3 + 1				0										
2,5	> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0									
3		> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0								
3,5			> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0							
4				> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0						
4,5					> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0					
5						> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0				
5,5							> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0			
6								> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0		
6,5									> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0	
7										> 12 + ≤ 12 + ≤ 6 + ≤ 3 + 1					0

NOTE — 7 µm corresponds to 0,7 mm under a magnification of 100. Similarly 60 µm corresponds to 6 mm under a magnification of 100.

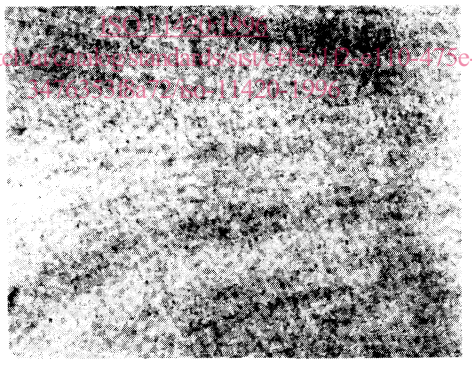
Annex B
(normative)

Photomicrographs for evaluation of the appearance of the dispersion

A 1

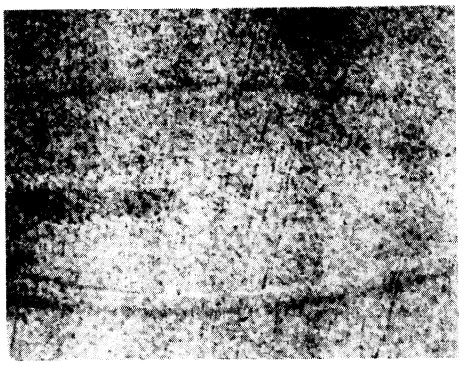


A 2

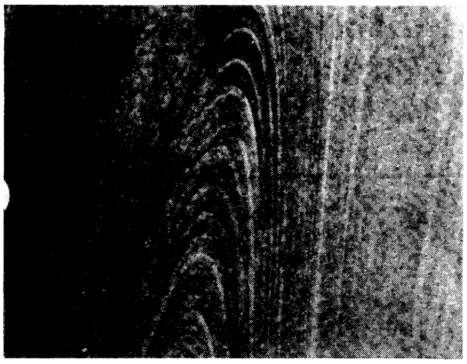


A 3

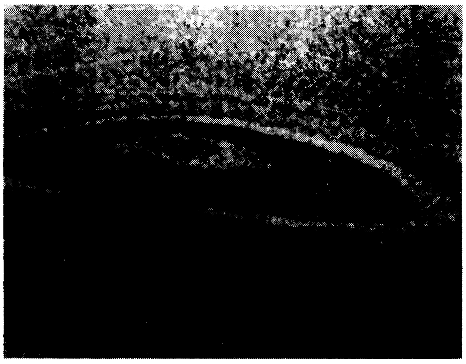
B 1



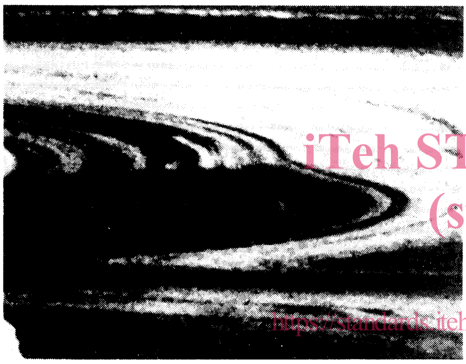
B 2



C 1



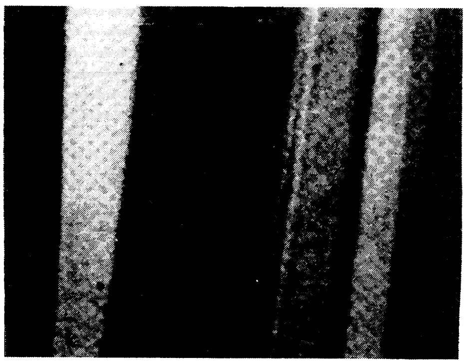
C 2



D 1

D 2

E 1



E 2

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	41 to 50	51 to 60	61 to 70	
	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)