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Pigments and extenders — Methods of dispersion and assessment of dispersibility in plastics —

Part 6: Determination by film test

Pigments et matières de charge — Méthodes de dispersion et évaluation de l'aptitude à la dispersion dans les plastiques —

Partie 6: Détermination par essai de film

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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.

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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 256, *Pigments, dyestuffs and extenders*.

ISO 23900 consists of the following parts, under the general title *Pigments and extenders — Methods of dispersion and assessment of dispersibility in plastics*:

— *Part 1: General introduction*

— *Part 2: Determination of colouristic properties and ease of dispersion in plasticized polyvinyl chloride by two-roll milling*

— *Part 3: Determination of colouristic properties and ease of dispersion of black and colour pigments in polyethylene by two-roll milling*

— *Part 4: Determination of colouristic properties and ease of dispersion of white pigments in polyethylene by two-roll milling*

— *Part 5: Determination by filter pressure value test*

— *Part 6: Determination by film test*

Pigments and extenders — Methods of dispersion and assessment of dispersibility in plastics —

Part 6: Determination by film test

1 Scope

This part of ISO 23900 specifies a method assessing the degree of dispersion of colorants[1)] and/or extenders in a thermoplastic polymer.

The method is suitable for testing colorants and/or extenders in the form of concentrates or compounds in all polymers used for extrusion processes.

NOTE Defects such as gels, black specks, holes in the test film are not in the scope of this part of ISO 23900.

The film test result determined according to this method is valid only for the equipment, conditions and test polymer being used. The use of test conditions differing from those specified might give different results. The preparation methods of concentrates or compounds are not specified in this part of ISO 23900. The results obtained for individual colorants and/or extenders are therefore comparable only when the same conditions of preparation for concentrates or compounds and a comparable detection system are used.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
speck
defect caused by agglomerates, aggregates and primary particles of the colorant and/or extender, impurities of basic test polymer

2.2
primary particle of the colorant
smallest single unit detectable by physical methods

Note 1 to entry: Suitable physical methods are, for example, optical and electron microscopy.

2.3
aggregate
particle comprising strongly bonded or fused particles where the resulting external surface area may be significantly smaller than the sum of calculated surface areas of the individual components

Note 1 to entry: The forces holding an aggregate together are strong forces, for example, covalent bonds, or those resulting from sintering or complex physical entanglement.

Note 2 to entry: Aggregates are also termed secondary particles and the original source particles are termed primary particles.

[SOURCE: ISO/TS 27687:2008, 3.3]

1) For the definition of colorant see ISO 4618:2014, 2.60 colouring material.

2.4
agglomerate
collection of weekly bound particles or aggregates or mixtures of the two where the resulting external surface area is similar to the sum of the surface areas of the individual components

Note 1 to entry: The forces holding an agglomerate together are weak forces, for example van der Waals forces, or simple physical entanglement.

Note 2 to entry: Agglomerates are also termed secondary particles and the original source particles are termed primary particles.

[SOURCE: ISO/TS 27687:2008, 3.2]

2.5
total speck size range
specified upper and lower limits of particle sizes

2.6
speck size class
one or more categories defined by a minimum and maximum value of speck sizes within the total speck size range

2.7
inspected film area
film area from the beginning to the end of the measurement

Note 1 to entry: The unit is square metres.

2.8
speck area ratio
total speck area divided by inspected film area

Note 1 to entry: The unit is mm^2/m^2.

2.9
transmission illumination
illumination whereby light source and optical sensor are arranged on opposite sides of the film

2.10
pixel
smallest image-forming element to which a grey level is assigned

[SOURCE: ISO 21227-1:2003, 3.4.5]

2.11
grey level
shade of grey assigned to a pixel

Note 1 to entry: The shades are usually positive integer values taken from the grey scale.

[SOURCE: ISO 21227-1:2003, 3.4.7]

2.12
grey scale
series of grey levels between white and black

EXAMPLE The 8-bit grey scale has 2^8 (=256) grey levels. Grey level 0 corresponds to black, grey level 255 (the 256^{th} level) to white.

[SOURCE: ISO 21227-1:2003, 3.4.8]

2.13
resolution
number of pixels per unit length on the surface of an object

Note 1 to entry: If the resolution in the X- and Y-direction is different, both values have to be reported.

[SOURCE: ISO 21227-1:2003, 3.4.6]

2.14
brightness
average grey level of a specified part of the image

[SOURCE: ISO 21227-1:2003, 3.5.3]

2.15
contrast
difference between the grey levels of two specified parts of the image

[SOURCE: ISO 21227-1:2003, 3.5.4]

2.16
calibration film
film with specified amount of defects which has already been assessed

Note 1 to entry: It is used to check the reproducibility and repeatability of the parameter settings.

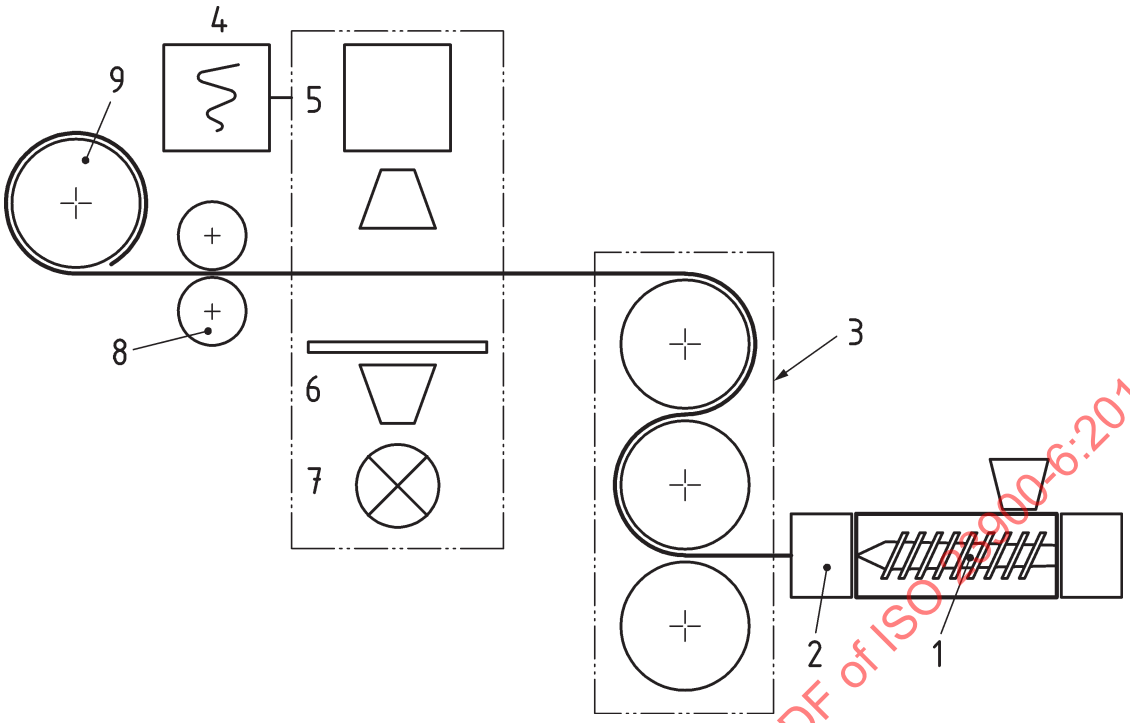
3 Principle

The test mixture, consisting of a colour concentrate and a basic test polymer or the compound, is passed through an extruder fitted with a blown film unit or a cast film unit followed by appropriate downstream equipment.

After extrusion, the transmission illumination enables the detection of specks within the film by means of a camera inspection system.

The resulting variations in contrast or brightness, caused by the presence of particles (primary particles, aggregates, agglomerates), are assessed using appropriate software.

Figure 1 illustrates the principle construction of the apparatus.



Key

1	extruder	6	diffuser, optional
2	die	7	light source
3	take off unit[a]	8	speed determination[b]
4	data processing	9	winder
5	camera system		

a Take off units with two rollers are also possible.

b Different kinds of speed controller systems are possible.

Figure 1 — Scheme for cast film extrusion

4 Materials

4.1 Concentrate

Homogeneous preparation of colorants and/or extenders in an appropriate thermoplastic polymer.

4.2 Basic test polymer

Thermoplastic polymer of a type and grade to be agreed between the interested parties, preferably of a film grade.

NOTE The development work on this part of ISO 23900 was carried out in low density polyethylene (LDPE).

4.3 Test mixture

A homogeneous mixture of a concentrate (4.1) and the basic test polymer (4.2) or a compound as specified in Clause 6.

5 Apparatus

5.1 Extruder, without dispersing elements. No filter should be used in the extrusion line.

For laboratory testing a single screw extruder with a diameter between 19 mm and 30 mm and with a length of 20 L/D (length/diameter) to 30 L/D (length/diameter) is recommended.

5.2 Blown film unit or **cast film unit**.

5.3 Camera detection system.

5.4 Diffuser, if necessary.

NOTE The usage of a diffuser can increase the reproducibility of results when examining highly transparent films.

6 Preparation of test mixtures

6.1 General

The concentrate (4.1) and the basic test polymer (4.2) are mixed together, for example in a glass or plastic container, to provide the test mixture (4.3). Alternatively, a compound can be used.

The viscosities of concentrate and test polymer should be compatible in order to get a homogeneous distribution.

The test polymer and the processing should not influence the detection of specks.

6.2 Test mixture

The test mixture depends on the type of colorant and/or extender and polymer. It should therefore be agreed between the interested parties prior to testing.

If the produced film made from the test mixture is not homogeneous, a compound shall be produced prior to film extrusion.

EXAMPLE 1 Organic colorant or carbon black: a total amount of 1000 g (100 %) test mixture contains 975 g polymer, 25 g colour concentrate with 40 % colorant content (contains 10 g organic colorant or carbon black = 1 % in the test mixture).

EXAMPLE 2 Inorganic colorant: a total amount of 1 000 g (100 %) contains 950 g polymer, 50 g colour concentrate with 40 % colorant content (contains 20 g inorganic colorant = 2 % in the test mixture).

7 Procedure

7.1 Calibration

The equipment shall be calibrated in order to guarantee the correct measurement of size and numbers of black dots on the calibration film.

The whole system should be verified with a calibration film with printed or plotted black dots under operating conditions. Three different sizes of certified printed or plotted black dots should be used for calibration of the system within the range of interest, e.g. values near the expected maximum, average and minimum particle sizes to be measured with the system. Additionally, the calibration film shall include a printed black dot with a defined diameter of 25 % to 50 % of the inspection width. The uniformity of the field of inspection shall be checked.

7.2 Size ranges and resolution

The theoretical limit for resolution of objects by size using image analysis is 1 pixel. Defects should be assessed speck by speck with the minimum number of 4 pixel.

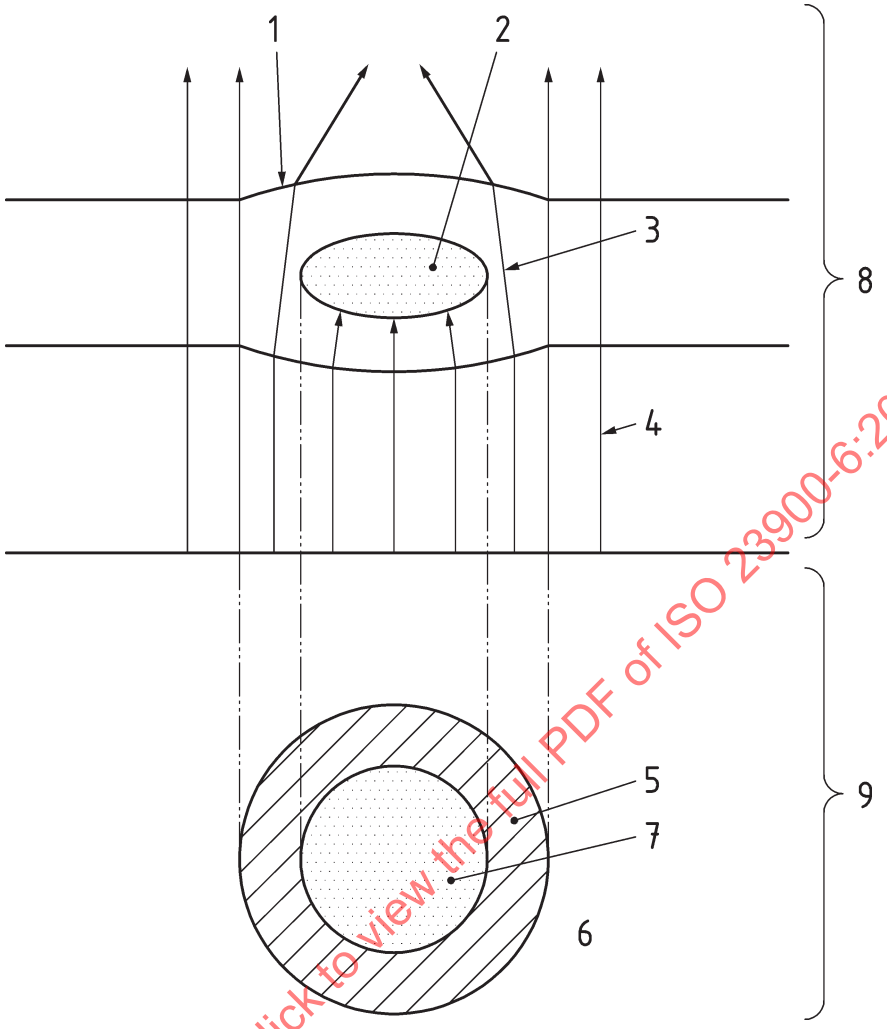
EXAMPLE By the use of an optical resolution of 19 µm/pixel, the theoretically minimum of equivalent circular diameter is 42 µm based on an evaluation of minimum 4 pixel.

It is necessary to specify speck size classes. This should be done by the interested parties.

NOTE 1 The maximum of the highest class is unlimited.

For quantitative analysis, pixels should be converted to length and area units prior to any reporting.

NOTE 2 The determination of the real size of specks is not feasible in any case as their embedding in the film leads to optical imperfections. The speck may cause a film deformation that will induce an image distortion caused by lenticular light refraction (see Figure 2).



Key

1 deformation of the film surface by embedded speck
2 speck
3 refraction of light by the film surface
4 lights beam of a parallel light source
5 area with light refraction in the border speck area
6 area with brightness level
7 speck shadowing
8 cross section of a film
9 topographic view of a film

Figure 2 — Image distortion caused by lenticular light refraction of a speck

7.3 Pre-conditioning

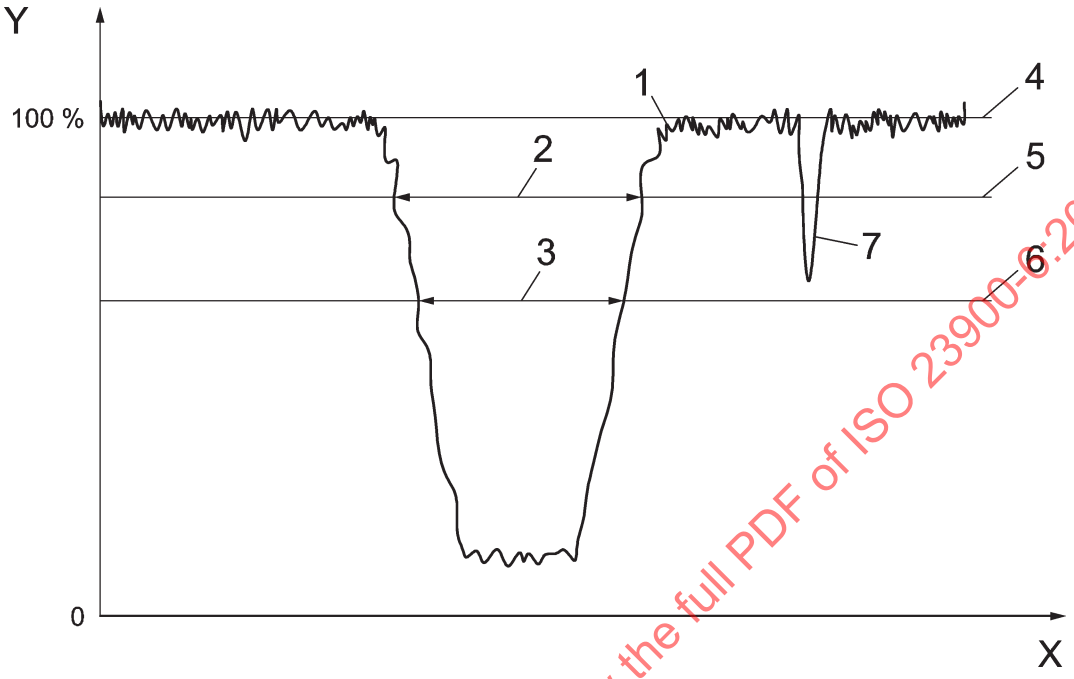
The extruder (5.1), the blown film or the cast film unit (5.2) should be pre-heated to the processing temperature for the basic test polymer.

The equipment should be cleaned or adequately purged with the basic test polymer (4.2) before each test is started.

The sensitivity of the detection system shall be adjusted. The average signal should be between 60 % and 85 % of the grey scale to avoid over-modulation of the light sensor.

The speck detection level shall be adapted to ensure an appropriate distinction between noise and speck signal.

NOTE The difference between brightness and speck detection level is influencing the probability that a defect is detected or not. If the difference between the brightness and speck detection level is too large, only a limited amount of specks will be detected (see Figure 3).



Key

X	pixel no.	4	brightness
Y	grey level in %	5	speck detection level A
1	signal	6	speck detection level B
2	speck width A	7	speck not detected with level B
3	speck width B		

Figure 3 — Influence of the adjustment of the speck detection level

7.4 Test procedure

7.4.1 Production of the test polymer film

The extruder line shall run with basic test polymer until the camera system detects a quality of the film equivalent to the typical level of the basic test polymer.

For a folded blown film, it is recommended to cut the tube and to examine only one layer to avoid overlapping of specks.

7.4.2 Production of the test mixture film

When the hopper is empty and the extruder screw is just visible, add the test mixture (4.3).

When the colour of the film is uniform, the camera detection system shall be activated.

The test is finished as soon as a pre-defined area (0,5 m^2 minimum) has been inspected. Use the recorded data to evaluate the film defects.

After the test, purge the extruder thoroughly with basic test polymer for the next test.

