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

Part 5: Determination by filter pressure value test

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

Partie 5: Détermination de la valeur de pression du filtre lors d'un essai

Reference number
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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 5:
Determination by filter pressure value test

1 Scope

This part of ISO 23900 specifies a method of assessing the degree of dispersion of a colorant in a thermoplastic polymer.

The method is suitable for testing colorants in the form of colour concentrates in all polymers used for extrusion and melt-spinning processes.

The filter pressure value (FPV) 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 method of preparing the colour concentrate is not specified in this part of ISO 23900. The results obtained for individual colorants are therefore comparable only when the same method of preparation for colour concentrates is used.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 10088-1, *Stainless steels — Part 1: List of stainless steels*

3 Terms and definitions

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

3.1
filter pressure value
FPV
pressure difference between the start pressure and the maximum pressure generated by extrusion in front of a screen pack related to the amount of colorant tested

4 Principle

The test mixture, consisting of a colour concentrate and a basic test polymer, is passed through an extruder fitted with melt pump and screen pack with breaker plate. In front of the screen pack is a melt pressure transducer. The pressure difference between the start pressure and the maximum pressure is used to calculate the filter pressure value (FPV).

5 Material

5.1 Colour concentrate

Homogeneous preparation of a colorant in an appropriate thermoplastic polymer.

5.2 Basic test polymer

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

NOTE The development work on this part of ISO 23900 was carried out in polypropylene (PP).

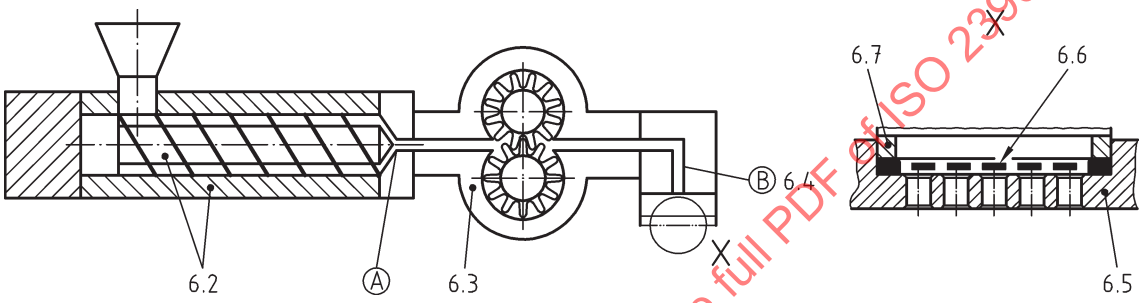
5.3 Test mixture

A homogeneous mixture of a colour concentrate (5.1) and the basic test polymer (5.2) as specified in Clause 7.

6 Apparatus

6.1 General

Figure 1 illustrates the principle construction of the apparatus (see 6.2 to 6.7).



Key

A melt pressure transducer in front of the melt pump

B melt pressure transducer in front of the filter

Figure 1 — Apparatus

6.2 Extruder

A single screw extruder with non-grooved barrel and a screw without dispersing elements shall be used. A screw with a diameter between 19 mm and 30 mm and with a length of 20 L/D to 30 L/D (length/diameter) is recommended. It is necessary to have a melt pressure transducer in front of the melt pump (A) to measure the pressure of the melt. An electronic controller with screw speed/pressure feedback loop is necessary in order to maintain this pressure constant, preferably at a level between 30 bar and 60 bar, to ensure that the melt pump is completely filled and to ensure optimum homogeneity of the melt.

6.3 Melt pump

The melt pump, preferably a metering pump, shall provide a constant throughput of 50 cm^3/min to 60 cm^3/min.

6.4 Melt pressure transducer

The pressure range shall be preferably between 0 bar and 100 bar for mixture 1 (7.2) and between 0 bar and 350 bar for mixture 2 (7.3). The accuracy of the melt pressure transducer (B) shall be within ± 1 % with a repeatability of less than ± 0,1 %.

The resolution of the pressure measurement should be 0,1 bar.

6.5 Breaker plate

A breaker plate, as shown in Figure A.1, shall be used to support the screen-pack and defines its free area.

6.6 Filter

6.6.1 General

The filter media is that part of the system which influences the differential pressure used as the basic data for determining the results of the test.

The differential pressure increase is dependent on the retention characteristics of the filter media.

In order to have comparable results it is important that the filter media is defined in detail and assembled exactly to specification.

Screen-packs are used as filter media. The screen-pack is assembled from ultrasonically cleaned filter discs, having a filter diameter of 33,8 mm (±0,1 mm), in a multi-layer construction, preferably held together in an aluminium frame. All screens shall be made from a suitable material appropriate to the polymer used, e.g. stainless steel in accordance with EN 10088-1, Type 1.4404. Any change in specification (e.g. weaving pattern, surface condition, number of apertures per unit length or aperture width) can lead to a different test result.

6.6.2 Screen-pack 1

Two-layer construction, where the first layer is a reverse plain dutch weave 615/108 warp/weft per 25,4 mm with a wire diameter of 0,042 mm/0,14 mm and the second layer (support mesh) is a square mesh plain weave 0,63 mm aperture width with a wire diameter of 0,40 mm calendered (for further details see ISO 9044).

6.6.3 Screen-pack 2

Two-layer construction, where the first layer is a reverse plain dutch weave 615/132 warp/weft per 25,4 mm with a wire diameter of 0,042 mm/0,13 mm and the second layer (support mesh) is a square mesh plain weave 0,63 mm aperture width with a wire diameter of 0,40 mm calendered (for further details see ISO 9044).

6.6.4 Screen-pack 3

Three-layer construction, where the first layer is a twilled dutch weave 165/1400 warp/weft per 25,4 mm with a wire diameter of 0,071 mm/0,040 mm and the second layer (support mesh) is a square mesh plain weave 0,25 mm aperture width with a wire diameter of 0,16 mm and the third layer (support mesh) is a square mesh plain weave 0,63 mm aperture width with a wire diameter of 0,40 mm calendered (for further details see ISO 9044).

It is recommended to request confirmation from the supplier that the above specifications are used for the screen-pack. Especially the number of apertures per unit length and the wire diameters of the individual layers are extremely critical for the result of the filter pressure value test.

NOTE The use of further finer screen-packs than described in 6.6.2 may be agreed between the interested parties.

6.7 Sealing ring

The sealing ring or aluminium frame of the filter disc should have a diameter of 33,8 mm ± 0,1 mm and an inside diameter of 28 mm ± 0,1 mm.

If the screen-pack has no aluminium frame a sealing ring is to be used.

7 Preparation of test mixtures

7.1 General

The colour concentrate (5.1) and the basic test polymer (5.2) are mixed together, for example in a glass or plastics container, to provide the homogeneous test mixture.

Mixture 1 (7.2) is recommended for colour pigments and mixture 2 (7.3) is recommended for white and carbon black pigments.

NOTE 1 The use of other mixtures may be agreed between the interested parties.

NOTE 2 Colorant quantities below 5,0 g will lead to insufficient accuracy.

7.2 Mixture 1

A test mixture of 200 g (100 %), including 5,0 g colorant (2,5 %) is used.

NOTE If the colour concentrate contains 40 % colorant the quantities are: 12,5 g colour concentrate and 187,5 g basic test polymer.

7.3 Mixture 2

A test mixture of 1 000 g (100 %), including 80,0 g colorant (8 %) is used.

NOTE If the colour concentrate contains 40 % colorant the quantities are: 200 g colour concentrate and 800 g basic test polymer.

8 Procedure

8.1 Pre-conditioning

The complete apparatus (Clause 6) should be pre-heated to the processing temperature appropriate for the basic test polymer.

The equipment should be cleaned or adequately purged with the basic test polymer (5.2) before each test is started.

8.2 Determination

Mount a new screen-pack (6.6.2 to 6.6.4) in front of the breaker plate (6.5) and the measuring equipment in such a way that the melt flows through the finer screen first and through the breaker plate last. A sealing ring (6.7) shall prevent leakage of the mixture around the edge of the screen-pack.

Allow sufficient time for the screen-pack and the breaker plate to reach the temperature of the equipment. This time depends on the equipment being used. The basic test polymer (5.2) is then plasticized in the extruder and passed through the screen-pack with a defined melt volume throughput until the melt temperature and pressure remain constant. The machine conditions should guarantee a constant melt temperature, with temperature deviations of less than ± 2°C.

Measure the start pressure p_s developed by the basic test polymer directly in front of the screen-pack. The start pressure p_s should be constant. When the hopper is empty and the extruder screw is just visible, add the test mixture (5.3).

NOTE A pressure drop can occur because of different rheological properties of basic test polymer and test mixture.

After feeding of the test mixture is completed, 100 g basic test polymer are added just as the extruder screw becomes visible again.

The test is finished as soon as the extruder screw once again becomes visible. Use the recorded data to evaluate the maximum pressure p_{max} and to calculate the filter pressure value.

Remove the screen-pack while still hot and purge the apparatus thoroughly with basic test polymer for the next test.

9 Evaluation

The filter pressure value, FPV, defined as the increase of pressure per gram colorant, is calculated by using Formula (1):

$$FPV = \frac{(p_{max} - p_s)}{m_c} \quad (1)$$

where

FPV is the filter pressure value, in bar per gram (bar/g);

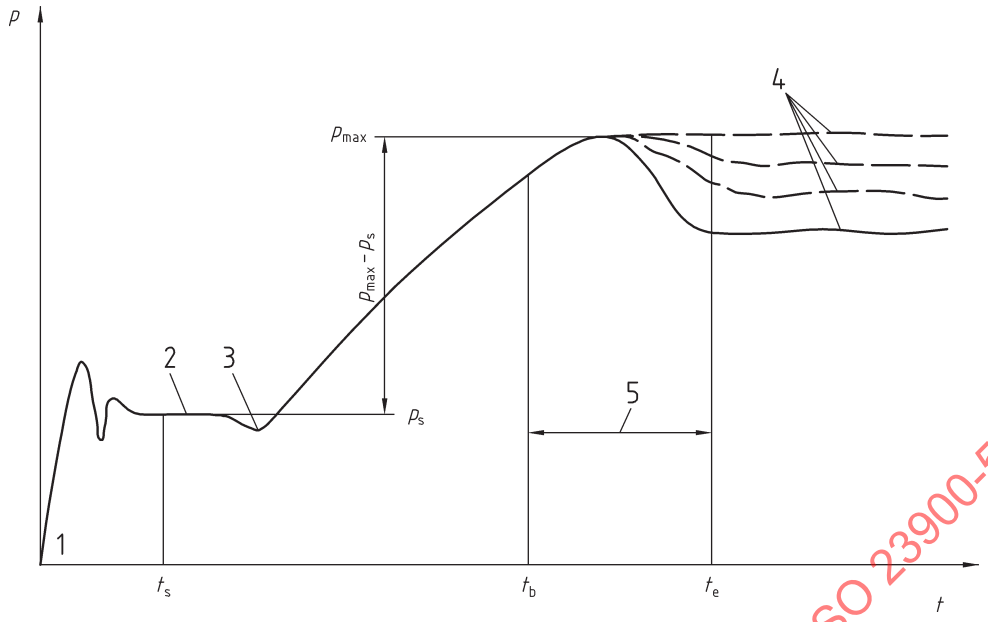
p_s is the start pressure, in bar;

p_{max} is the maximum pressure, in bar;

m_c is the colorant quantity used in the test, in grams.

It is recommended to express the filter pressure value accurate to one decimal place.

Figure 2 illustrates an example of a typical pressure curve.



Key

1	start period
2	start pressure p_s
3	possible pressure drop because of rheological properties
4	different pressure development depending on the test mixture
5	purging with 100 g of basic test polymer
p	pressure
p_s	start pressure
p_{max}	maximum pressure
t	time
t_s	measuring of p_s and filling of the hopper with basic test mixture
t_b	feeding of the test mixture is completed
t_e	end of monitoring of pressure and determination of the maximum pressure p_{max}

Figure 2 — Typical pressure curve

10 Test report

The test report shall contain at least the following information:

a) a reference to this part of ISO 23900 (i.e. ISO 23900-5);

b) all necessary details to identify the colour concentrate tested;

c) all necessary details to identify the basic test polymer;

d) the description of the test mixture (Clause 7);

e) all necessary details about the test procedure and the test conditions (e.g. test equipment, type of screen-pack, melt temperature, melt volume throughput);

f) the result of the test, as indicated in Clause 9;

g) any deviation from the test method specified;

h) the date of the test.

11 Precision

This part of ISO 23900 defines the principles of the method and the procedures to be used, but allows variation as regards the dimensions of the machinery and the composition of the test mixture used. Precision data thus cannot be established for the method itself, precision should be determined by repeatability and reproducibility studies according to the equipment and test mixture used in the testing laboratory, and according to the colorant under test.

Annex A
(normative)

Breaker plate

Dimensions in millimetres

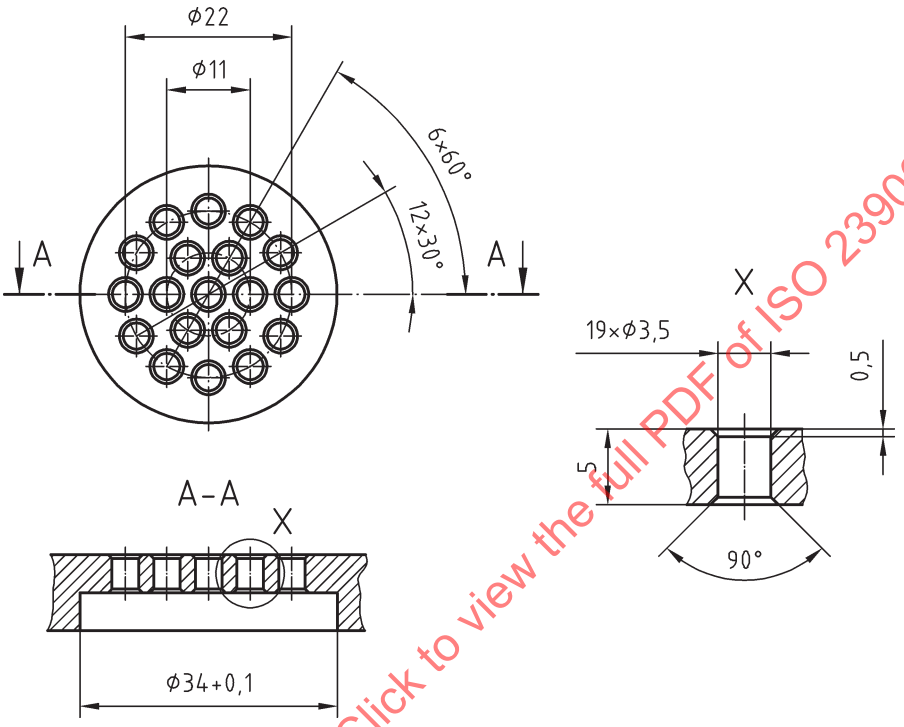


Figure A.1 — Breaker plate