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

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

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

Partie 3: Détermination des propriétés colorimétriques et de la facilité de dispersion des pigments noirs et colorés dans le polyéthylène par calandrage sur bicylindre

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

1 Scope

This part of ISO 23900 specifies a method of determining in polyethylene (PE) the colouristic properties of a test pigment relative to a standard, and the ease of dispersion DH_{PE} of pigments from the differences in colour strength on dispersing colouring materials under various conditions.

Method A is appropriate for use with organic powder pigments and carbon black pigments in powder form, many of which are subject to compaction (reagglomeration under pressure), for inorganic pigments in powder form and for pigment preparations in powder or flake form.

Method B is appropriate for testing pigments and pigment preparations in granular form and for inorganic pigments in any form.

The ease of dispersion determined in this way is valid only for the dispersion equipment, dispersion conditions and dispersion medium being used. The use of test conditions differing from those specified may give different results; this applies both to the absolute magnitude and to the relation between values of the ease of dispersion of various pigments. The subscript DH_{PE} is therefore used to designate the value obtained as specified in this part of ISO 23900.

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.

ISO 787-24:1985, *General methods of test for pigments and extenders — Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments — Photometric methods*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 18314-1[1)], *Analytical colorimetry — Part 1: Practical colour measurement*

3 Terms and definitions

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

1) To be published.

3.1
ease of dispersion
DH_{PE}
measure of the rate at which or the degree to which a pigment or extender achieves a given level of dispersion when dispersed in a plastics material

Note 1 to entry: The DH_{PE} is derived from the increase in colour strength achieved by two-roll milling as specified in 8.2, relative to the colour strength achieved as specified in 8.1.

4 Principle

Using a two-roll mill, the pigment under test is dispersed at an appropriate temperature in the polymer. The cooled milled sheet obtained in this way is then subjected to the higher shearing forces resulting from two-roll milling at a narrower gap width. The resulting increase in colour strength (see EN 12877-1) is a measure of the ease of dispersion DH_{PE}.

5 Materials

5.1 Materials for method A

5.1.1 Test medium

Polyethylene, in powder or flake form; of a type and grade to be agreed between the interested parties.

NOTE Where HDPE is used, a phenolic antioxidant can be used to facilitate processing.

5.1.2 Titanium dioxide pigment

Easily dispersing powder grade of a type recommended for use in polyethylene.

5.2 Materials for method B

5.2.1 Test medium

Polyethylene, in granular form; of a type and grade to be agreed between the interested parties.

NOTE Where HDPE is used, a phenolic antioxidant can be used to facilitate processing.

5.2.2 Titanium dioxide pigment

As in 5.1.2 or as a well dispersed polyethylene masterbatch.

6 Apparatus

6.1 Two-roll mill. Equipped with heating facilities and having rollers adjustable for spacing. The roll diameter shall be between 80 mm and 200 mm, and the ratio of the speeds of rotation of the two rollers shall be between 1:1,1 and 1:1,2.

NOTE It has been found that comparable results on different two-roll mills can be obtained under the following conditions

— ratio of roller diameters of the two machines: between 1:1 and 1:1,5;

— ratio of peripheral speeds: between 1:1 and 1:1,1;

— H_k (bank) to H_s (gap width) such as $H_k/H_s \geq 20$.

If smaller roller sets are used (roller diameter e.g. 80 mm), the settings of the thickness of the milled sheet from 0,4 mm to 0,5 mm with the recommended conditions of similarity can lead to difficulties with regard to the requirement for a rolling bank.

6.2 Plate press. Provided with heating facilities and, advantageously, also with cooling facilities.

6.3 Photometer.

7 Sampling

Representative samples of the colouring materials to be tested shall be taken as specified in ISO 15528.

8 Procedure

8.1 Testing of colouristic properties in white reduction

8.1.1 Preparation of the mixtures

8.1.1.1 Preparation of the mixture for method A

In a plastic beaker pre-blend gently 100 parts of polyethylene powder, 1,0 parts titanium dioxide pigment (5,0 parts in the case of carbon black) and where appropriate 0,1 parts antioxidant. Add 0,1 parts of carbon black or organic test pigment or 0,2 parts to 0,5 parts of inorganic test pigment according to type. Mix using a spatula so that no test pigment remains on the beaker walls.

NOTE It can be advantageous to prepare a large quantity of a homogeneous pre-blend of polymer powder, antioxidant and titanium dioxide pigment — for example by mixing for 5 min in a laboratory high speed mixer at 1 800 min^{-1} followed by extrusion and grinding into a suitably fine form — in order to improve reproducibility.

8.1.1.2 Preparation of the mixture for method B

Prepare a mixture of 100 parts of PE granulate (5.2.1) with 1,0 parts titanium dioxide pigment (5,0 parts in the case of carbon black) or an equivalent quantity of titanium dioxide masterbatch (5.2.2) in a plastic beaker or other suitable container.

8.1.2 Two-roll milling

8.1.2.1 Method A

The mixture is added to the rotating mill rolls which have been brought to a defined temperature, previously established as allowing easy handling of the polymer on the mill. Temperatures of between 140 °C and 160 °C have been found suitable for most types of polymer.

With a gap width set at 0,4 mm to 0,5 mm a sheet is formed within approximately 1 min in such a way that the whole of the material forms a continuous sheet on the front roll. Any material falling through the nip shall be returned quickly and carefully from the tray to the rotating mill rolls. The quantity of mixture to be used shall be such that a continuously rotation bead is formed in the nip, once the sheet has been formed.

NOTE A quantity of test mixture based on 100 g polymer is generally adequate for most two-roll mills. It can be increased according to the size of the mill in order to facilitate handling.

The dispersion of the pigment is obtained by cutting and folding of the sheet, once formed, every 30 s while milling with a gap width set at 0,5 mm for a total of 200 rotations of the rolls, counting from the point at which the mixture is added to the mill. According to the diameter of the rolls of the machine being used (see 6.1) the duration of milling shall not be less than 5 min but shall not exceed 10 min.

8.1.2.2 Method B

Place the mixture on the static, previously heated two-roll mill using the gap width and temperatures as described in 8.1.2.1 and allow to pre-heat for 1 min. Start the two-roll mill and prepare a mill sheet in about 1 min. Add the test pigment or pigment preparation slowly and evenly to the rotating sheet across the width of the mill. The dispersion of the pigment then proceeds as in 8.1.2.1.

After each milling operation the rolls shall be cleaned.

8.1.3 Pressing

For photometric measurement it is advantageous to prepare specimens with a high surface gloss and quality.

Such specimens may be obtained by pressing the sheets in a plate press for no longer than 2 min at a temperature between 160 °C and 170 °C maximum, between high gloss chrome steel plates using a spacer frame of 1 mm thickness. The pressed sheets shall be cooled rapidly to room temperature.

8.1.4 Photometric measurement

The colour strength and colouristic properties of the test specimens prepared as specified in 8.1.2 and 8.1.3 shall be measured as specified in ISO 18314-1. These values shall be used to determine the colour strength as specified in ISO 787-24:1985, 8.1 and Clause 9 for the purposes of the calculation of DH_{PE}.

8.2 Testing of ease of dispersion

8.2.1 Preparation of the test samples

The roll gap of the mill is reduced to 0,3 mm and one half of the sheet prepared under 8.1.2 is returned to the rollers maintained at the same temperature as used in 8.1.2 at 25 min^{-1}. Milling is continued for 200 revolutions of the rolls with cutting and folding every 30 s. The sheet is then removed and cooled between metal plates.

This procedure is carried out for each sheet containing the test pigments.

After each milling operation the rolls shall be cleaned.

8.2.2 Pressing and photometric measurement

Pressing and photometric measurement are carried out as specified in 8.1.3 and 8.1.4.

9 Evaluation

The ease of dispersion, DH_{PE}, is expressed as the percentage increase in colour strength following roll milling at 0,3 mm gap width relative to that obtained following milling at 0,5 mm (see 8.1).

It shall be computed from the F values, using Formula (1):

$$DH_{PE} = 100 \times \left(\frac{F_2}{F_1} - 1 \right) \quad (1)$$

where

F_1 is the colour strength value of the test specimen, specified in 8.1;

F_2 is the colour strength value of the test specimen, specified in 8.2.

10 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this part of ISO 23900 (i.e. ISO 23900-3);

c) the designation of the test specimens and their preparation, including the temperature of milling;

d) the description of the titanium dioxide pigment, the antioxidant and the type, grade and form of polymer used;

e) the concentration of the colouring material under test in the polymer, for the respective test specimens;

f) the photometric data obtained and where appropriate ease of dispersion, DH_{PE};

g) the method of colour strength determination;

h) if colour measurement has been specified: the type of photometer as well as the standard illuminant and the standard observer used;

i) any deviation from the test method specified;

j) 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 type and grade of polyethylene 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 compound used in the testing laboratory, and according to the pigment under test.