
Pigmenti in polnila - Metode dispergiranja in ocenjevanje disperzibilnosti v polimernih materialih - 4. del: Ugotavljanje barvnih lastnosti in dispergiranja belih pigmentov v polietilenu z valjanjem z dvema valjčkoma

Pigments and extenders - Methods of dispersion and assessment of dispersibility in plastics - Part 4: Determination of colouristic properties and ease of dispersion of white pigments in polyethylene by two-roll milling

Pigmente und Füllstoffe - Dispergierverfahren und Beurteilung der Dispergierbarkeit in Kunststoffen - Teil 4: Bestimmung der koloristischen Eigenschaften und der Dispergierhärte von Weißpigmenten in Polyethylen im Walztest

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Pigments et matières de charge - Méthodes de dispersion et évaluation de l'aptitude à la dispersion dans les plastiques - Partie 4: Détermination des propriétés colorimétriques et de la facilité de dispersion des pigments blancs dans le polyéthylène par calandrage sur bicylindre

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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EN 13900-4:2004 (E)**Foreword**

This document (EN 13900-4:2004) has been prepared by Technical Committee CEN/TC 298 "Pigments and extenders", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2004, and conflicting national standards shall be withdrawn at the latest by October 2004.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This Part of EN 13900 specifies a method of determining the colouristic properties of a test pigment in polyethylene (PE) relative to a standard, and the ease of dispersion DH_{PE} of pigments from the differences in tinting strength of dispersing colouring materials under various conditions.

The method is appropriate for use with white pigments.

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 can 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 EN 13900.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 787—24:1995, *General method of tests for pigments and extenders — Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments — Photometric methods (ISO 787-24:1985)*.

EN ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling (ISO 15528:2000)*.

ISO 7724—2:1984, *Paints and varnishes — Colorimetry — Part 2: Colour measurement*.

3 Terms and definitions

For the purposes of this European Standard, the following term and definition applies.

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.

The DH_{PE} is derived from the increase in tinting strength achieved by two-roll milling as specified in 8.2, relative to the tinting 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 tinting strength is a measure of the ease of dispersion DH_{PE} .

EN 13900-4:2004 (E)**5 Materials****5.1 Polyethylene**

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

NOTE Where HD-PE is used, a phenolic antioxidant can be used as a slip agent to facilitate processing.

5.2 Carbon black polyethylene masterbatch

based on an easily dispersing type of carbon black as recommended for use in polyethylene.

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 will 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) should be $H_k/H_s \geq 20$.

If smaller roller sets are used (roller diameter e.g. 80 mm), the settings of the thickness of the milled sheet to 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, preferably, also with cooling facilities.

6.3 Photometer**7 Sampling**

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

8 Procedure**8.1 Testing of colouristic properties in Polyethylene in admixture with carbon black****8.1.1 Preparation of the mixture**

In a plastic beaker pre-blend gently 100 parts of polyethylene and 0,05 parts carbon black in prepared form (5.2). Add 5 parts of titanium dioxide test pigment. Mix using a spatula so that no test pigment remains on the beaker walls.

NOTE The masterbatch should be diluted in the test polymer to facilitate incorporation and handling.

A quantity of test mixture based on 100 g polymer will generally be adequate for most two-roll mills. It may if necessary be increased or decreased according to the size of the mill in order to facilitate handling.

8.1.2 Preparation of the test sample

The mixture is added to the rotating rolls of the two-roll mill previously heated to a defined temperature established as being appropriate for the polymer being used. The ratio of the speeds of rotation of the two rollers shall be between 1 : 1,1 and 1 : 1,2.

NOTE A temperature between 140 °C and 160 °C has been found suitable for most types of polymer.

A sheet is formed within 1 min in such a way that the whole of the material forms a continuous sheet on the front roll. The dispersion of the pigment is obtained by cutting and folding of the sheet every 30 s whilst milling for a further 7 min at 25 min⁻¹ with the gap width maintained at 0,5 mm. After the full 8 min milling the sheet is removed and allowed to cool to room temperature, unless photometric measurement is carried out directly on the rotating sheet.

After each milling operation the rolls shall be cleaned.

8.1.3 Pressing

Sufficiently large pieces are cut out of the sheets prepared from the standard and the test sample and pressed in a spacing frame between polished chromed press plates so as to ensure a surface suitable for photometric measurement.

NOTE Photometric measurement can also be carried out directly on the rotating sheet.

8.1.4 Photometric measurement

The tinting strength of the test specimens prepared as specified in 8.1.2 and 8.1.3 shall be measured as specified in ISO 7724-2:1984, clause 9. These values shall be used to determine the tinting strength as specified in EN ISO 787-24:1995, 8.1 and clause 9 for the purposes of the calculation.

8.2 Testing of ease of dispersion

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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 7 min with cutting and folding every 30 s. Then the sheet is removed and cooled between metal plates, unless photometric measurement is carried out directly on the rotating sheet.

This procedure is carried out for each sheet containing the test pigments, the rolls being cleaned after each milling operation.

8.2.2 Pressing and photometric measurement

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

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9 Evaluation

The ease of dispersion $[DH_{PE}]$ is expressed as the percentage increase in tinting 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 the following equation:

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

where:

F_1 is the tinting strength of the test specimen specified in 8.1;

F_2 is the tinting strength 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 EN 13900 (EN 13900-4);
- c) designation of the test specimens and their preparation, including the temperature of milling;
- d) type and concentration of the carbon black pigment in the masterbatch;
- e) type, grade and form of the test polymer used;
- f) photometric data obtained, how obtained and the ease of dispersion $[DH_{PE}]$;
- g) method of tinting strength determination;
- h) any deviation from the test method specified;
- i) date of the test.

11 Precision

This standard defines the principles of the method and the procedures to be used, but allows variation as regards the dimensions of the machinery used 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.