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a UgU`!`"XY`8c`c`Yj Ub`Y`VUf]j b]`Ughbcgh]`b`U`\_Y`X]gdYfn]`Y`fb]`]b`VUf]j b]`  
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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

Pigmente und Füllstoffe - Dispergierverfahren und Beurteilung der Dispergierbarkeit in Kunststoffen - Teil 3: Bestimmung der koloristischen Eigenschaften und der Dispergierhärte von Schwarz- und Buntpigmenten in Polyethylen im Walztest

[SIST EN 13900-3:2003](https://standards.itih.ai/catalog/standards/sist/c855e716-7d01-4bf3-ac7c-d41761000000/sist-en-13900-3-2003)

Pigments et matieres de charge - Méthodes de dispersion et évaluation de l'aptitude a 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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**ICS:**

83.040.30	Pomožni materiali in aditivi za polimerne materiale	Auxiliary materials and additives for plastics
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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

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This European Standard was approved by CEN on 12 December 2002.

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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 Management Centre 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 13900-3:2003 (E)****Foreword**

This document (EN 13900-3:2003) 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 August 2003, and conflicting national standards shall be withdrawn at the latest by August 2003.

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

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## 1 Scope

This part of this European Standard 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 this European Standard.

## 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 12877-1, *Colouring materials in plastics - Determination of colour stability to heat during processing of colouring materials in plastics - Part 1: General introduction*. <http://standards.iteh.ai/catalog/standards/sist/c855e716-7d01-4bf3-ac7c-d4c768c4c9e7/sist-en-13900-3-2003>

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 Term and definition

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 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  $DH_{PE}$ .

**EN 13900-3:2003 (E)****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.

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**6 Apparatus**

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**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) 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 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 described in EN 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 1 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  $1800 \text{ min}^{-1}$  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

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##### 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  $140 \text{ }^{\circ}\text{C}$  and  $160 \text{ }^{\circ}\text{C}$  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 should generally be adequate for most two-roll mills. It can if necessary 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 whilst 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.

**EN 13900-3:2003 (E)****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 160 °C to max. 170 °C 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 7724-2:1984, clause 9. These values shall be used to determine the colour strength as specified in EN ISO 787-24:1995, 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<sup>-1</sup>. 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 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 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 the following equation:

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

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 this European Standard (EN 13900-3);
- c) designation of the test specimens and their preparation, including the temperature of milling;