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**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**

*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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## 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](http://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](http://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 4:

# Determination of colouristic properties and ease of dispersion of white pigments in polyethylene by two-roll milling

## 1 Scope

This part of ISO 23900 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 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<sup>1)</sup>, *Analytical colorimetry — Part 1: Practical colour measurement*

## 3 Terms and definitions

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

### 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 tinting strength achieved by two-roll milling as specified in 8.2, relative to the tinting strength achieved as specified in 8.1.

1) To be published.

## 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  $DH_{PE}$ .

## 5 Materials

### 5.1 Polyethylene

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

NOTE Where HDPE 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 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 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 specified in 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.

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

A quantity of test mixture based on 100 g polymer is generally adequate for most two-roll mills. It may 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 while milling for a further 7 min at 25 min<sup>-1</sup> 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 18314-1. These values shall be used to determine the tinting strength as specified in ISO 787-24:1985, 8.1 and Clause 9 for the purposes of the calculation.

## 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 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 specified in [8.1.3](#) and [8.1.4](#).

## 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 Formula (1):

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

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 ISO 23900 (i.e. ISO 23900-4);
- c) the designation of the test specimens and their preparation, including the temperature of milling;
- d) the type and concentration of the carbon black pigment in the master batch;
- e) the type, grade and form of the test polymer used;
- f) the photometric data obtained, how obtained and the ease of dispersion,  $DH_{PE}$ ;
- g) the method of tinting strength determination;
- h) any deviation from the test method specified;
- i) 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 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.