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

Part 2: Determination of colouristic properties and ease of dispersion in plasticized polyvinyl chloride by two-roll milling

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

Partie 2: Détermination des propriétés colorimétriques et de la facilité de dispersion dans le polychlorure de vinyle plastifié par calandrage sur bicylindre

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 2: Determination of colouristic properties and ease of dispersion in plasticized polyvinyl chloride by two-roll milling

1 Scope

This part of this ISO 23900 specifies a method of determining the colouristic properties of a test pigment relative to a standard, and the ease of dispersion $DH_{PVC\text{-}P}$ of pigments from the differences in colour strength on dispersing colouring materials under various conditions in plasticized polyvinyl chloride (PVC-P) compounds.

The method is appropriate for use with organic and inorganic black and colour pigments and for pigment preparations.

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 $DH_{PVC\text{-}P}$ is therefore used to designate the value obtained as specified in this part of ISO 23900.

The principle of this part of ISO 23900 may also be used for routine quality control purposes by reference to the photometric data generated from the sheets milled at 130 °C. For quality control purposes, the ratio of pigment to TiO_2 may be agreed between the interested parties. Ratios of 1:10 for organic pigments and 0,2 to 0,5:1 for inorganic pigments are suggested as convenient and widely used standard ratios.

Annex A is informative and gives a description of a suitable basic compound.

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*

EN 12877-1, *Colouring materials in plastics — Determination of colour stability to heat during processing of colouring materials in plastics — Part 1: General introduction*

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_{PVC-P}
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_{PVC-P} 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 (160 ± 5) °C in the basic compound. The milled sheet obtained in this way is then subjected to the higher shearing forces resulting from two-roll milling at (130 ± 5) °C. The resulting increase in colour strength is a measure of the ease of dispersion DH_{PVC-P}.

5 Material

A suitable and recommended basic compound is described in Annex A. An alternative compound may be agreed between the interested parties and is to be mentioned in the test report.

The quantity of the pigment used shall be such that a depth of shade (as specified in EN 12877-1) of approximately 1/25 SD is achieved.

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, with cooling facilities as well.

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 Milling at (160 ± 5) °C

The mixture consisting of the pigment under test and the basic compound shall be processed to produce a milled sheet. A test specimen having dimensions of 1 mm by at least 50 mm × 50 mm shall be produced from a portion of the milled sheet.

NOTE In this method, increased shear is obtained by milling the reference sheet at a lower temperature of 130 °C, as a result of which the wetting-out properties of the PVC-P compound are also changed. The routine use of this method thus requires either that the temperature of a single mill is increased and decreased, which is time consuming, or that two mills operating simultaneously at different temperatures are employed. An alternative principle by which the milling temperature is maintained, and increased shear is generated by reducing the gap as in EN 12877–4, is being investigated for comparability and as a potentially more efficient method.

8.1.1 Premixing of the test materials

The prescribed quantities of colouring material and basic PVC-P compound required for the test specimens are mixed together in a suitable container, for example using a mixing shaker, for 5 min.

In the case of colouring materials in paste form, it is recommended to mix the components in a polyethylene or polypropylene beaker by hand using a mixing rod (not of glass) until the mixture appears to be homogeneous.

8.1.2 Two-roll milling

The pre-mixed material is added to the rotating mill rolls which have been brought to a temperature of (160 ± 5) °C. Any material falling through the nip shall be returned quickly and carefully from the tray to the moving 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. Set the gap so that the sheet has a uniform thickness of 0,4 mm to 0,5 mm thickness across its width.

Mill the material, limiting the width of the sheet by frequent cutting to one half and by reversal and lateral rolling, to prevent the material from running up onto the roll guides and to achieve adequate dispersion of the colouring material. Alternatively it may be removed repeatedly and returned without delay to the rolls to ensure thorough mixing. In this case, the number of such repetitions shall be defined as part of the method and recorded in the test report.

Mixing is carried out for a total of 200 rotations of the rolls. 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.

The sheet is then removed from the rolls. To facilitate this step the roll gap and, if necessary, roll speed and friction may be adjusted.

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 165 °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.

8.2 Milling at (130 ± 5) °C

The remainder of the milled sheet prepared as specified in 8.1 shall be used for this purpose. The roller gap shall be set, and maintained unchanged during roll milling, such that a milled sheet having a thickness of 0,4 mm to 0,5 mm is produced. The roller temperatures shall be maintained at 130 °C ± 5 °C.

The milled sheet shall first be passed through the roller gap unfolded, then folded once and passed without delay through the gap again. This procedure (i.e. folded once) shall be repeated 10 times. A test specimen having dimensions of 1 mm by at least 50 mm × 50 mm shall be prepared by pressing as specified in 8.1.3.

9 Photometric measurement

The colour strength of the test specimens prepared as specified in 8.1 and 8.2 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 $DH_{PVC\text{-}P}$.

10 Evaluation

10.1 Evaluation of colouristic properties in a white reduction

For quality control purposes the colouristic properties and colour differences of the test specimens relative to a standard shall be measured as specified in Clause 9.

NOTE The colouristic properties in a full-shade system can be carried out in a similar manner but without addition of TiO_2.

10.2 Evaluation of the ease of dispersion

The ease of dispersion, $DH_{PVC\text{-}P}$, is the percentage increase in colour strength following roll milling at 130 °C. It shall be computed from the F values, using Formula (1):

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

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.

11 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 23900-2);

c) the designation of the test specimens and their preparation;

d) the description of the basic compound;

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

f) the photometric data obtained and where appropriate ease of dispersion [$DH_{PVC\text{-}P}$];

g) the method of colour strength determination;

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

i) any deviation from the test method specified;

j) the date of the test.

12 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 PVC compound 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.

Annex A
(informative)

Description of the basic compound

A.1 PVC-P compound suitable for determining ease of dispersion by two-roll milling

a) 65,00 parts vinyl chloride polymer, suspension PVC

b) 33,50 parts plasticizer — di-iso-decly phthalate (DIDP)

c) 1,50 parts epoxidized soybean oil

d) 1,30 parts liquid barium zinc stabilizer

e) 0,20 parts lubricant — stearic acid

f) 5,34 parts[2)] titanium dioxide pigment

A.2 Specifications

a) Suspension PVC

K value 70 ± 1

b) Plasticizer — di-iso-decyl phthalate

Of a quality recommended for use in PVC compounds.

c) Epoxidized soybean oil

Of a quality recommended for use in PVC compounds.

d) Liquid barium zinc stabilizer

Having a barium content of 10,2 to 12,2 and a zinc content of 1,95 to 2,35.

NOTE Alternatively barium zinc stabilizer systems resulting in equivalent heat stability can be used.

e) Stearic acid

Of a quality recommended for use in PVC compounds.

f) Titanium dioxide pigment

An easily dispersing powder grade recommended for plastics should be used.

Composition: rutile type with organic and inorganic surface treatment and with minimum 93 % TiO_2.

A.3 Preparation of PVC-P compound

The PVC polymer, stabilizer and lubricant are pre-mixed in a high speed mixer until the mixture reaches a temperature of 70 °C. Titanium dioxide pigment is then added and mixed for about 2 min (there is a danger of greying through metal abrasion if longer mixing times are used). Subsequently the pre-mixed quantities of plasticizer and epoxidized soybean oil are added as a thin stream as mixing continues. The

2) Equivalent to 5 parts TiO_2 in 100 parts of finished compound.

mixture reaches a temperature of about 100 °C during this process, following which the homogeneous compound is cooled with agitation to room temperature.

PVC-P compounds should be stored in a cool place in a closed container for not longer than 2 years under exclusion of light.

A.4 Assessing the basic compound

It is necessary to assess the basic compound, since the titanium dioxide pigment which it contains can also be further dispersed by the roll milling specified in 8.2. This will result in an increase in the opacity or hiding powder of the basic mixture, leading to a change in the F value of the test specimen prepared by the roll milling specified in 8.2. In determining the ease of dispersion, DH_{PVC-P}, the change in the opacity or hiding power of the basic mixture can be disregarded if it is equivalent to less than 3 %. Otherwise, it is necessary to correct the test results.

In order to assess the basic compound, the procedure specified in this part of ISO 23900 should be carried out as stated in Clause 8, and a dyestuff soluble in the basic mixture should be used in place of the pigment under test. It is recommended that a concentration of 0,05 % C.I. Solvent Violet 13 or Solvent Violet 36 be added to the basic compound. This leads to a colouration of approximately 1/25 standard depth of shade. The subsequent procedure is as specified in 8.1 and 8.2. The milled sheets obtained should be used to determine the values F_1 and F_2 as specified in Clause 10. These values are used to compute the factor C:

$$C = \frac{F_1^*}{F_2^*} \tag{A.1}$$

If $C > 1{,}03$, then Formula (1) (see Clause 10) should be replaced by Formula (A.2):

$$DH_{PVC-P} = 100\frac{(C \cdot F_2 - 1)}{F_1} \tag{A.2}$$