SLOVENSKI STANDARD
SIST EN 12877-1:2001

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Sredstva za obarvanje v polimernih materialih - Določ evanje barvne stabilnosti na vroč ino med predelovanjem sredstev za obarvanje v polimernih materialih - 1. del: Splošna predstavitev

Colouring materials in plastics - Determination of colour stability to heat during processing of colouring materials in plastics - Part 1: General introduction

Farbmittel in Kunststoffen - Bestimmung der Beständigkeit der Farbe gegen Hitze beim Verarbeiten von Farbmitteln in Kunststoffen - Teil 1: Allgemeine Einleitung

Matieres colorantes dans les plastiques - Détermination de la stabilité de la couleur a la chaleur au cours de la mise en oeuvre des matieres colorantes dans les plastiques - Partie 1: Généralités

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EUROPEAN STANDARD
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This European Standard was approved by CEN on 5 September 1999.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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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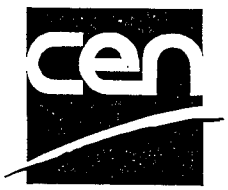

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by April 2000, and conflicting national standards shall be withdrawn at the latest by April 2000.

Annex A (informative) describes a general method for preparing test specimens having standard depth of shade and annex B (informative) comprises tables listing the coefficients required in the calculations.

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, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

This is one of a number of Parts of EN 12877 dealing with methods for determining the colour stability of colouring materials under the influence of the thermal stress encountered during plastics processing. In this context, the common expressions "heat stability" and "heat resistance" are widely used.

Colour changes may occur during processing not only because of thermal degradation of colouring materials and of plastics but also as a result of other changes to them, e.g. solubility in the polymer, changes in dispersion level, recrystallization or crystal modification, and from other complex interactions with the polymer itself or other additives present.

Colour stability during processing is one of the most important performance criteria required by the processor of coloured plastics materials. However, absolute values for colour stability of colouring materials during processing cannot be established.

The test methods described in EN 12877 allow appropriate procedures, conditions and determination criteria to be agreed upon according to the plastics material to be coloured and the processing method. The resulting comparative data is useful in colouring material selection and also in determining their processing stability in the end-use polymer under practical processing conditions.

The term colouring material as used here includes single pigments, dyestuffs and extenders as well as products produced from them such as masterbatches, colour pastes, pigment blends and other pigment preparations designed for use in plastics materials.

1 Scope

This Part of EN 12877 gives a general introduction to the most widely used methods for determination of colour stability during processing of colouring materials in plastics under the influence of heat.

It also specifies those details which are common to the various test methods described in subsequent Parts of EN 12877. The choice of the test method depends on the plastics material to be coloured, the method of processing and the end-use requirements.

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.

EN 12877-2	Colouring materials in plastics - Determination of colour stability to heat during processing of colouring materials in plastics - Part 2: Determination by injection moulding
EN 12877-3	Colouring materials in plastics - Determination of colour stability to heat during processing of colouring materials in plastics - Part 3: Determination by oven test
EN 12877-4	Colouring materials in plastics - Determination of colour stability to heat during processing of colouring materials in plastics - Part 4: Determination by two-roll milling
EN 20105-A02	Textiles - Tests for colour fastness - Part A02: Grey scale for assessing change in colour (ISO 105-A02; 1993)

ISO 842		Raw materials for paints and varnishes - Sampling
ISO 7724-2	1984	Paints and varnishes - Colorimetry - Part 2: Colour measurement
ISO 7724-3		Paints and varnishes - Colorimetry - Part 3: Calculation of colour differences

3 Terms and definitions

For the purposes of this standard the following definitions apply:

colour stability: The resistance to colour change of a standardized test material prepared from the colouring material to be tested and a plastics material, when subjected to heat under defined test conditions.

NOTE 1 For the colour stability under the influence of heat the expressions "heat stability" and "heat resistance" are widely used.

NOTE 2 A change of properties of the test specimens other than colour under the effect of heat is not taken into account in this European Standard.

4 Apparatus

Test apparatus includes a screw injection moulding machine with screw ram (EN 12877-2), a drying oven with forced air ventilation (EN 12877-3) and a laboratory two-roll mill (EN 12877-4).

5 Sampling

Representative samples of the colouring materials to be tested shall be taken as described in ISO 842.

6 Test specimens

The colouring material shall be tested in reduced shade (6.1) and/or in full shade (6.2).

6.1 For testing **in reduced shade**, 1 % of titanium dioxide pigment shall be added to the platics material, unless otherwise specified or agreed. The concentration of the colouring material shall correspond to

a) 1/3 standard depth of shade (SD) (see annex A), or

b) 1/25 standard depth of shade (SD) (see annex A), or

c) an agreed reduction ratio of colouring material to titanium dioxide.

For a test specimen of 1 mm thickness 5 % titanium dioxide pigment are necessary to obtain full hiding.

6.2 For testing **in full shade**, the concentration of the colouring material in the test medium shall correspond to 6.1 a). Alternatively, a concentration of 0,1 % (preferred for organic pigments and dyestuffs), or 2 % (preferred for inorganic pigments) or another agreed appropriate concentration shall be taken.

The preparation of test specimens for the particular test methods for heat stability is described in EN 12877-2, EN 12877-3 and EN 12877-4 respectively.

7 Procedure

7.1 Determination by injection moulding

Test specimens are obtained by injection moulding of the coloured plastics material. A specimen prepared at a low processing temperature at the usual dwell time is used as the reference specimen. Then, the processing temperature is increased in intervals. At the higher processing temperatures, the dwell time is prolonged. For details see EN 12877-2.

The colour properties of the prepared test specimens are assessed in accordance with 7.4.

7.2 Determination by oven test

Test specimens are subjected to specified or agreed test temperatures during specified or agreed test periods. A specimen which is kept at room temperature is used as the reference specimen. For details see EN 12877-3.

The colour properties of the prepared test specimens are assessed in accordance with 7.4.

7.3 Determination by two-roll milling

The coloured test mixture is milled at a specified or agreed roll surface temperature during specified or agreed test periods. After the specified or agreed test periods, test portions are taken from the milled sheet from which specimens suitable for colour measurement are prepared. A specimen obtained from the test mixture after treatment under the usual processing conditions is taken as the reference specimen. For details see EN 12877-4.

The colour properties of the prepared test specimens are assessed in accordance with 7.4.

7.4 Evaluation of colour difference

The resulting colour differences shall be either determined in accordance with ISO 7724-2 and ISO 7724-3, or assessed using the grey scale specified in EN 20105-A02. The colour properties of the uncoloured plastics material, determined by using the same procedure with and without adding titanium dioxide pigment, may be taken into account.

8 Expression of results and test report

See EN 12877-2, EN 12877-3 and EN 12877-4 respectively.

9 Precision

Statistical data can not be established for the precision of the methods in Parts 2 to 4. Comments, where appropriate are given for each method.

Annex A

(informative)

Preparation of test specimens having standard depths of shade

A.1 Scope

The fastness of colourations depends on their depth of shade. It is therefore advantageous to carry out tests for fastness properties of colouring materials and systems containing colouring materials at defined depth of shade. In this annex, the 1/3 and 1/25 standard depths of shade are defined by a formula. In addition, the annex gives information as to how the depth-of-shade characteristics of coloured specimens are to be determined and how the concentration of the colouring material in a specimen has to be changed so that the required standard depth of shade is obtained.

A.2 Definitions

For the purposes of this annex, the following definitions apply.

A.2.1 depth of shade: A measure for the intensity of a colour perception that increases with increasing saturation and, generally, decreases with increasing lightness.

NOTE Colourations having the same depth of shade appear to be prepared by using the same concentrations of colouring materials having the same tinting strength. In this annex, the relationship between depth of shade and saturation and, respectively, lightness,is defined colorimetrically.

A.2.2 standard depths of shade (SD): Depth of shade levels laid down by convention.

NOTE: The depth of shade levels 1/3 and 1/25 have been derived from the depth of shade levels used in the textiles field.

A.3 Standard depths of shade

The depths of shade characteristics $B_{1/3}$ and $B_{1/25}$ for 1/3 and 1/25 SD are given by the following equations (1) and (2) respectively:

$$B_{1/3} = \sqrt{Y} \times (s \times a\,(\phi)_{1/3} - 10) + 29 \quad (1)$$

$$B_{1/25} = \sqrt{Y} \times (s \times a\,(\phi)_{1/25} - 10) + 56 \quad (2)$$

where

s is the measure for the saturation. It is the linear distance between the chromaticity co-ordinates x, y of the specimen and the achromatic point in the chromaticity diagram (basic stimulus E, standard illuminant D65, 10° standard colorimetric observer or respectively, standard illuminant C, 2° standard colorimetric observer), multiplied by 10, i.e.

$$s = 10 \times \sqrt{(x - x_0)^2 + (y - y_0)^2} \quad (3)$$

Table A.1 - Values for x_0 and y_0

Standard illuminant	Standard colorimetric observer	x_0	y_0
D65	10°	0,3138	0,3310
C	2°	0,3101	0,3162

$a(\phi)$ are factors for the evaluation of the saturation measure which depend on both the hue angle ϕ and the standard depth of shade (SD). The values of these factors are given in annex B.

Y is the tristimulus value Y

Standard depth of shade is assumed to be obtained if the depth-of-shade characteristic $B_{1/3}$ or $B_{1/25}$ amounts to 0.

NOTE Standard illuminant D65 and the 10° standard colorimetric observer should preferably be used.

A.4 Calculation of the $a(\phi)$ factors

The factors $a(\phi)$ for 1/3 SD and 1/25 SD that depend on the hue angle ϕ can be calculated within certain ranges of the hue angle by polynomials of third order. This leads to different sets of coefficients for standard illuminant D65/10° standard colorimetric observer and standard illuminant C/2° standard colorimetric observer respectively.

Firstly, the hue angle ϕ (0° to 360°) is calculated from the chromaticity coordinates x,y of the specimen and those of the achromatic point x_0,y_0 for the chosen combination of the standard illuminant and standard colorimetric observer (D65/10° or C/2°), using the following equation (4):

$$\phi = \arctan \frac{y - y_0}{x - x_0} \qquad (4)$$

In the calculation, the following conditions are to be taken into account:

$0° < \phi < 90°$ for $y - y_0 > 0$ and $x - x_0 > 0$

$90° < \phi < 180°$ for $y - y_0 > 0$ and $x - x_0 < 0$

$180° < \phi < 270°$ for $y - y_0 < 0$ and $x - x_0 < 0$

$270° < \phi < 0°$ for $y - y_0 < 0$ and $x - x_0 > 0$

The $a(\phi)$ value is then calculated by a polynom of third order, using the following equation (5):

$$a(\phi) = a(\phi_0) + K_1 \times W + K_2 \times W^2 + K_3 \times W^3 \qquad (5)$$

where

$$W = \frac{\phi - \phi_0}{100} \qquad (5.1)$$

ϕ_0 is nearest lower angle to the hue angle ϕ, the coefficients K1,K2, K3 of which are used in eqation (5). For individual values of the various quantities see annex B.

A.5 Determination of depth-of-shade characteristics of coloured test specimens

Measure the tristimulus values X, Y, Z of the specimens, either for standard illuminant D65/10° standard colorimetric observer or for standard illuminant C/2° standard colorimetric observer, as described in ISO 7724-2:1984, sub-clause 4.1.1 or 4.1.2 respectively.

The choice of measuring conditions depends on the information to be obtained by the measurement. If differences in the surface reflection should not influence the depth of shade (for example in pigment testing), measure in accordance with sub-clause 4.1.1 (including the specular reflection). In this case, subtract from the tristimulus values the following ΔX, ΔY, ΔZ values:

for D65/10°:	$\Delta X = 3{,}8$	$\Delta Y = 4{,}0$	$\Delta Z = 4{,}3$
for C/2°:	$\Delta X = 3{,}9$	$\Delta Y = 4{,}0$	$\Delta Z = 4{,}7$.

If the specular reflection is to be excluded by the measurement, as it corresponds to visual evaluation of the depth of shade, measure in accordance with sub-clause 4.1.2, preferably by using measuring condition 45/0 or 0/45.

NOTE Only in the case of high-gloss flat specimens do both measuring conditions (sub-clause 4.1.1 and 4.1.2) yield the same depths of shade.

Calculate the depth-of-shade characteristics B of the specimens as described in A.3 and A.4 above.

A.6 Calculation of the concentration of colouring material in specimens required for standard depth of shade

A positive (negative) depth-of-shade characteristic B means that the actual concentration of the colouring material in the specimen is too high (low). The approximate concentration required to achieve a value of $B = 0$ (i.e. that which corresponds to standard depth of shade) can be calculated using the following equation (6):

$$c_{req} \approx c \cdot 0{,}9^{B} \qquad (6)$$

where

c is the actual concentration of colouring material in the specimen;

B is the depth-of-shade characteristic B of the specimen.

NOTE: When at least two values for B are plotted against the logarithm of the respective concentrations c as a graph on exponential paper, the required concentration c_{req} for $B = 0$ can be determined by extrapolation or interpolation.