

Colouring materials in plastics — Determination of colour stability to heat during processing of colouring materials in plastics —

Part 2: Determination by injection moulding

The European Standard EN 12877-2:1999 has the status of a
British Standard

ICS 83.040.30

National foreword

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 5 and a back cover.

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English version

Colouring materials in plastics - Determination of colour stability
to heat during processing of colouring materials in plastics -
Part 2: Determination by injection moulding

Matières colorantes dans les plastiques - Détermination de
la stabilité de la couleur à la chaleur au cours de la mise en
oeuvre des matières colorantes dans les plastiques -
Partie 2: Détermination par moulage-injection

Farbmittel in Kunststoffen - Bestimmung der Beständigkeit
der Farbe gegen Hitze beim Verarbeiten von Farbmitteln in
Kunststoffen - Teil 2: Bestimmung durch Spritzgießen

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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of an identical text or by endorsement, at the latest by April 2000, and conflicting national standards shall be withdrawn at the latest by April 2000.

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. It should be read in conjunction with EN 12877-1.

1 Scope

This part of EN 12877 describes a method for determining the colour stability of colouring materials under defined conditions in plastics by injection moulding. The result of the determination is a relative value, not an absolute one.

The method is applicable for testing colouring materials in thermoplastics that are processible by injection moulding.

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 12877-1:1999	Colouring materials in plastics - Determination of colour stability to heat during processing of colouring materials in plastics - Part 1: General introduction
EN 20105-A02	Textiles - Tests for colour fastness - Part A02: Grey scale for assessing change in colour (ISO 105 - A02:1993)
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 Principle

The colouring material to be tested, where appropriate together with titanium dioxide pigment, is mixed with the uncoloured thermoplastic material (test medium). The mixture is processed, using a suitable dispersing equipment, into a form suitable for injection moulding. The coloured test material obtained is injected into the plate-shaped mould cavity of a screw-injection moulding machine. Commencing at the lowest recommended processing temperature suitable for filling the mould cavity, the test temperature is increased in intervals of 10 °C or 20 °C up to the highest practicable processing temperature, and the dwell time is increased to 5 min.

The colour differences between the moulded plate obtained at the lowest temperature and those prepared at the higher temperatures are determined either colorimetrically or visually. The highest test temperature at which any colour change does not exceed a stipulated degree is taken as a measure of the colour stability of the colouring material under the test conditions (test medium and concentration of the colouring material).

4 Materials

4.1 Test medium

Thermoplastic moulding material, to be agreed between the interested parties.

The coloristic behaviour of the test medium when subjected to heat shall be tested with and without titanium dioxide pigment, using the same procedure. If there are changes, these may be taken into account when expressing test results.

4.2 Titanium dioxide pigment, grade as recommended for use in plastics.

5 Apparatus

5.1 Laboratory mixers

5.1.1 High-speed mixer, for test media in powder form.

5.1.2 Low-speed mixer, for test media in pellet form.

5.2 Suitable dispersing equipment, for example extruder, internal mixer or two-roll mill.

5.3 Suitable grinding equipment, for example pelletizer or chipping machine.

5.4 Screw-injection moulding machine, preferably with hydraulic rotary drive, and non-return valve.

NOTE The machine used should preferably have a screw diameter of 18 mm to 30 mm and an effective screw length of at least 15 times the screw diameter.

5.5 Injection-moulding tool, preferably with bar gate and temperature control, for test specimens as specified in clause 7. The ratio of the volume of the material injection-moulded in a single shot to the maximum swept volume should be in the range from 1 : 3 to 1 : 5.

5.6 Electrical temperature sensor, fitted between the screw and the nozzle of the injection moulding machine. Alternatively, an immersion temperature sensor may be used.

5.7 Spectrophotometer or tristimulus colorimeter, for colour measurements in accordance with ISO 7724-2.

6 Sampling

See EN 12877-1.

7 Test specimens

7.1 Form and dimensions

Plate-shaped test specimens, of thickness at least 2 mm, suitable for colorimetry, shall be used.

7.2 Concentration of colouring material

The colouring material shall be tested in reduced shade (7.2.1) and/or in full shade (7.2.2).

7.2.1 For testing in reduced shade, 1 % of titanium dioxide pigment shall be added to the plastics material, unless otherwise agreed. The concentration of the colouring material in the test medium shall correspond to:

- a) 1/3 standard depth of shade (SD) in accordance with EN 12877-1:1999, annex A, or
- b) 1/25 standard depth of shade (SD) in accordance with EN 12877-1:1999, annex A, or
- c) an agreed ratio of colouring material to titanium dioxide pigment.

NOTE In cases of plastics showing stronger yellowing at higher processing temperatures, higher additions of titanium dioxide pigments can be used.

7.2.2 For testing in full shade, the concentration of the colouring material in the test medium shall correspond to 7.2.1 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.

8 Preparation of the test material

Pre-mix the required quantities of coloured pigment to be tested and titanium dioxide pigment (if applicable) with the test medium, using the high-speed mixer (5.1.1) for test media in powder form, and the low-speed mixer (5.1.2) for test media in pellet form.

Process the mixture, using a suitable dispersing equipment (5.2), into a form suitable for injection moulding. The temperature of the melt during processing shall not be higher than the starting temperature for preparing test specimens for injection moulding.

NOTE 1 The colouring material should be well dispersed in order to ensure reliable results.

NOTE 2 In the case of test media in pellet form, a suitable adhesion-promoting agent (0,1 % to 0,2 %, calculated on the test medium) can be added before mixing to facilitate dispersion and homogenization of the colouring material.

9 Procedure

9.1 Injection moulding tests

9.1.1 Preliminary tests

Carry out preliminary tests to determine a processing schedule for the test medium used. The required cycle in the injection moulding process without back pressure shall be suitable for preparing test specimens at relatively low and relatively high processing temperatures. Measure the test temperature of the medium by using the melt-temperature sensor. Alternatively, use an immersion temperature sensor and measure the temperature of the medium by freely expelling it from the nozzle onto a wooden board. The test temperature shall be a multiple of 10.

Make allowance for test temperatures in intervals of 20 °C within the determined processing schedule. In the case of more temperature-sensitive colouring materials or test media, intervals of 10 °C may be used.

9.1.2 Test procedures

NOTE Method A leads to less machine-dependent differences in thermal stress. A series of plates can be moulded. This allows equilibrium to be achieved and multiple measurements to be carried out, thereby enhancing the reliability of the result.

9.1.2.1 Method A

Process the test material (clause 8) using the normal injection-moulding cycle at the lowest test temperature determined in accordance with 9.1.1. Discard sufficient mouldings until injection-moulded plates consisting of pure test material are obtained. Use these plates as reference specimens. The injection-moulding cycle is then prolonged so that the specified dwell time of 5 min in the barrel is obtained. The resulting plates shall be used as test specimens for this prolonged dwell time.

NOTE The dwell time is given by

$$\text{Cycle time} \times \frac{\text{free barrel volume}}{\text{mould volume}}$$

Then, whilst the moulding machine is in operation on the prolonged cycle, increase the processing temperature until the next higher test temperature is reached. After reaching this temperature discard mouldings until the plates that are produced have received the full thermal stress at this higher temperature. These plates shall be used as test specimens for this processing temperature.

Repeat the same procedure for all further predetermined test temperatures.

9.1.2.2 Method B

Process the test material (clause 8) at the lowest test temperature determined in accordance with 9.1.1. Discard sufficient mouldings until injection-moulded plates consisting of pure test material are obtained. Use these plates as reference specimens. Then, interrupt the cycle for 5 min. Use the second moulding obtained after this period as the test specimen for this prolonged dwell time.

Repeat the same procedure for all further predetermined test temperatures.

9.2 Evaluation of colour difference

Determine the colour difference between the test specimen prepared at the lowest test temperature and those prepared at the higher test temperatures.

NOTE Colouring materials such as C.I. Pigment Red 48, 53 and 57 and dyestuffs can change colour reversibly after moulding and the mouldings should be maintained at room temperature for at least 16 h before assessment.

If colour measurement is specified, proceed in accordance with ISO 7724-2:1984, 4.1.1, and ISO 7724-3. For visual comparison, use the grey scale specified in EN 20105-A02.

10 Expression of results

Express the test results in one of the following ways:

- a) as a graph, giving the colour difference as a function of the test temperature;
- b) as the temperature at which the colour difference on the grey scale does not exceed a defined value. Unless otherwise agreed, values of ΔE_b 3,0 or grey scale 4 may be taken as an indication of heat stability.

NOTE ΔE_b 3,0 and grey scale 4 are not representative of the same degree of colour difference.

Where temperatures are obtained by extrapolation the result shall be rounded to the nearest 10 °C.

11 Test report

The test report shall contain at least the following information:

- a) a reference to this European Standard (EN 12877-2);
- b) all details necessary to identify the colouring material tested;
- c) all details necessary to identify the test medium together with any other additives used, including the titanium dioxide pigment;
- d) all details of preparation of the test specimens, including the thickness of the test specimens;
- e) the type of the apparatus used (mixers, dispersing equipment, injection-moulding machine and tool) and the chosen test conditions (method A or method B), in particular the lowest test temperature used;
- f) the chosen standard depth(s) of shade or full shade and the concentrations of the colouring material and TiO₂ tested;
- g) if colour measurement has been specified: type of spectrophotometer or tristimulus colorimeter as well as standard illuminant and standard observer used;
- h) the result of the test, as indicated in clause 10, and the assessment criteria applied;
- i) any yellowing of the test medium and whether and how this has been taken into account;
- j) any deviation from the test method specified;
- k) the date of the test.

12 Precision

Results obtained may vary according to the injection moulding machine being used and the material being tested.

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