BRITISH STANDARD

BS EN
12877-4:2000

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

Part 4: Determination by two-roll milling

The European Standard EN 12877-4:1999 has the status of a British Standard

ICS 83.040.30

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 4, an inside back cover and a back cover.

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Colouring materials in plastics - Determination of colour stability to heat during processing of colouring materials in plastics - Part 4: Determination by two-roll milling

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 4: Détermination par calandrage sur bicylindre

Farbmittel in Kunststoffen - Bestimmung der Beständigkeit der Farbe gegen Hitze beim Verarbeiten von Farbmitteln in Kunststoffen - Teil 4: Bestimmung im Dauerwalztest

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

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Foreword

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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 two-roll milling. The result of the determination is a relative value, not an absolute one.

The method is mainly used for testing colouring materials in polyvinyl chloride.

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

A milled sheet is prepared from the plastics material and the colouring material to be tested, ensuring that the colouring material is well dispersed. This sheet is then milled at a specified temperature. At appropriate time intervals samples are taken from which test specimens are prepared by heat pressing. The colour differences between the test specimens obtained for the different milling times are taken as a measure of the colour stability of the colouring material. The colour comparison is performed either colorimetrically or visually.

4 Materials

4.1 Test medium, to be agreed between the interested parties.

When evaluating pigments in polyvinyl chloride, the test medium shall be adequately stabilized to heat. The colour stability of the test medium when subjected to heat shall therefore be tested with and without titanium dioxide pigment, using the same procedure. If there are changes, these shall be taken into account when expressing test results.

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

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5 Apparatus

5.1 Laboratory two roll-mill, with facility for heating. The spacing of the rollers shall be adjustable, and the diameter of the rollers should be between 80 mm and 180 mm (see Note 2). The ratio of the rotational speeds of the two rollers (friction ratio) shall be adjustable, and the rotational speed shall be (20 ± 5) min^{-1}. The ratio bead diameter H_k (bank) to gap width H_s should be $H_k/H_s \geq 20$.

NOTE 1 Chromium-plated rollers should preferably be used.

NOTE 2 It has been found that comparable results on different two-roll mills will be obtained under the following conditions:

- ratio of roller diameters: between 1 : 1 and 1 : 1,5;
- ratio of peripheral speeds: between 1 : 1 and 1 : 1,1.

5.2 Plate press, provided with a heating and cooling system.

5.3 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

The quantity of material shall be sufficient to fill the gap between the rolls and provide the required bead diameter.

Unless otherwise specified or agreed, test specimens of dimensions 50 mm x 50 mm x 1 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**, 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 reduction ratio.

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

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 % (to be preferred for organic pigments and dyestuffs), or 2 % (to be preferred for inorganic pigments) or another agreed appropriate concentration shall be taken.

8 Procedure

Prepare milled sheets on the two-roll mill from the colouring material to be tested and the agreed plastics material. In the case of plasticized polyvinyl chloride use a temperature of (160 ± 5) °C, a gap width of 0,2 mm to 0,4 mm and a total milling time of 8 min including plastification. Cut and fold the sheet repeatedly to achieve adequate dispersion of the colouring material. Take a sample from one of the sheets and prepare a test specimen from it by heat pressing at 170 °C for 1 min, for use as a reference specimen.

Set the roll surface temperatures for PVC_p to between 180 °C and 185 °C and for PVC_u to between 190 °C and 195 °C. Process a sufficient number of sheets so that the recommended minimum bead diameter is achieved (see **5.1**), whilst setting the roller gap to produce a sheet of uniform thickness of about 1 mm. Start timing and 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.

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Take samples after 10 min and 20 min milling time. After a further period of 10 min (total test period 30 min) draw off the mill sheet and take a further sample. Prepare test specimens from each sample by heat pressing at 170 °C for 1 min, using sufficient pressure to achieve a smooth surface.

WARNING - Attention is drawn to the need for efficient fume extraction when testing plasticized polyvinyl chloride on an open mill at elevated temperatures.

Compare the colour of the test specimens with that of the reference specimen either colorimetrically or visually.

NOTE Colouring materials such as C.I. Pigment Red 48, 53 and 57 and deyestuffs can change colour reversible after heat treatment and the specimens 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.

9 Expression of results

Express the test results either as a colour difference in accordance with ISO 7724-3, or as a step of the grey scale, together with the direction of any colour change.

10 Test report

The test report shall contain at least the following information:

a) a reference to this European Standard (EN 12877-4);

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 necessary regarding the preparation of the test specimens and the execution of the test (temperature and test duration);

e) the chosen standard depth(s) of shade or full shade and the concentrations of the colouring material tested;

f) if colour measurement has been specified: type of spectrophotometer or tristimulus colorimeter as well as standard illuminant and standard observer used;

g) conditions of pressing the treated test specimens;

h) the result of the test, as indicated in clause **9**;

i) all visual observations, for example discolouration of the test medium or blooming of the colouring material;

j) any deviation from the test method specified;

k) the date of the test.

11 Precision

Statistical data are not appropriate.

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BSI
389 Chiswick High Road
London
W4 4AL