BRITISH STANDARD

BS EN
1244:1998

# Adhesives — Determination of the colour and/or colour changes of adhesive coats under the influence of light

The European Standard EN 1244:1998 has the status of a British Standard

ICS 83.180

# National foreword

This British Standard is the English language version of EN 1244:1998.

The UK participation in its preparation was entrusted to Technical Committee STI/52, Adhesives, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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**Summary of pages**

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 6, an inside back cover and a back cover.

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EUROPEAN STANDARD
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# EN 1244

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Descriptors: Adhesives, coatings, tests, colorimetry, artificial light tests, determination, colour fastness, specimen preparation

English version

# Adhesives — Determination of the colour and/or colour changes of adhesive coats under the influence of light

Adhésifs — Détermination de la couleur et/ou des changements de couleur des revêtements adhésifs sous l'influence de la lumière

Klebstoffe — Bestimmung der Farbe und/oder der Farbänderung von Klebaufstrichen unter Lichteinwirkung

This European Standard was approved by CEN on 24 January 1998.

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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**CEN**

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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



Ref. No. EN 1244:1998 E

## Foreword

This European Standard has been prepared by Technical Committee CEN/TC 193, Adhesives, the secretariat of which is held by AENOR.

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 August 1998, and conflicting national standards shall be withdrawn at the latest by August 1998.

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.

## Contents

## 1 Scope

This European Standard is applicable to solvent-based, dispersion, hot-melt and reactive adhesives, their basic constituents, and related products the coating of which are coloured and/or subject to a change of colour by light. It describes a method of measuring the colour of an adhesive coat and the magnitude of a change of colour under the influence of light.

A change of colour of an adhesive coat can be very undesirable in practice, in particular if goods made of white or bright-coloured materials such as paper, textiles or leather are adhesively bonded and material surfaces are contaminated by coated adhesive.

## 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 923, *Adhesives — Terms and definitions.*

EN 1066, *Adhesives — Sampling.*

EN 1067, *Adhesives — Examination and preparation of samples for testing.*

CIE Publication No 15.2 (1986), *Colorimetry.*

ISO 554:1976, *Standard atmospheres for conditioning and/or testing — Specifications.*

ISO 2602:1980, *Statistical interpretation of test results — Estimation of the mean — Confidence interval.*

ISO 105-B02:1988, *Textiles — Tests for colour fastness — Part B02: Colour fastness to artificial light: Xenon arc fading lamp test.*

## 3 Definitions

For the purposes of this standard, the definitions in accordance with EN 923, CIE Publication No 15.2, and the following definitions apply:

**3.1**

**adhesive coat**

adhesive layer applied to an adherend

**3.2**

**adhesive film**

adhesive coat separated from the substrate after setting

**3.3**

**colour**

sensation perceived by the human eye

## 4 Principle

The colour of an adhesive coat is measured by colorimeter, converting its readings according to the CIE recommendations (CIE publication No 15.2) to CIELAB values.

The change of colour of an adhesive coat is determined by exposing an adhesive coat for a specified time under specified conditions to artificial light that is very similar in spectral composition to natural daylight but greater in intensity, and then measuring the colour of the coat before and after the exposure to light, and calculating the CIELAB colour differences.

## 5 Safety

Persons using this standard shall be familiar with normal laboratory practice.

This standard does not purport to address all the safety problems, if any, associated with its use.

It is the responsibility of the user to establish safety and health practices and to ensure compliance with any European and national regulatory conditions.

## 6 Apparatus

**6.1** *Cardboard*, about 250 g/m$^2$ in mass in relation to the surface, with a smooth surface and a very high degree of whiteness as carrier for adhesive coats and film.

The cardboard shall be free from hydroxybutyl toluene (BHT) or other ingredients liable to cause discoloration by migration to avoid interferences or false results.

**6.2** *Glass sheets*, with a completely even surface (plate glass) for the preparation of adhesive film.

**6.3** *Coating device*, allowing the spreading of the adhesive to be tested on cardboard (see **6.1**) or glass sheet (see **6.2**) producing on a flat surface a coating of 100 mm $\times$ 30 mm and a uniform thickness of $(0{,}1 \pm 0{,}02)$ mm after drying, cooling and setting, which shall be measured by any suitable methods.

**6.4** *Colorimeter*, for measuring the colour of test specimens both unexposed and exposed to light (see also **8.2**) and converting the readings to the CIE colour space values $L^*$, $a^*$ and $b^*$ and calculating the CIELAB colour differences $\Delta L^*$, $\Delta a^*$, $\Delta b^*$ and $\Delta E^*_{ab}$ automatically according to the CIE recommendations (CIE publication No 15.2). This colorimeter shall also be suitable for the determination of the yellowness index YI.

**6.5** *Light exposure apparatus*, containing the following elements.

**6.5.1** *Xenon arc lamp*, of correlated colour temperatures ranging from 5 000 °K to 6 500 °K to conform with ISO 105:B02 and a rating in the range 1,5 kW (as suggested in ISO 105:B02), dependent on the type of equipment and cooling system.

**6.5.2** *Light filter*, placed between the xenon arc lamp and specimen holders so that the ultra-violet spectrum is steadily reduced.

The transmission of the filter shall be at least 90 % between 380 nm and 750 nm, falling to 0 % between 310 nm and 320 nm.

**6.5.3** *Heat filter*. The spectrum of the xenon arc contains an appreciable amount of infra-red radiation.

For minimizing this radiation an efficient glass or water filter system is necessary.

**6.5.4** *Test chamber*, of a light source is surrounded by 10 or more holders made from an inert material for test specimens (see **7.1**), size 130 mm × 45 mm, arranged in an equal distance on a carousel in such a manner that the intensity of radiation on the plane of the specimen reaches about 1 000 W/m$^2$. The intensity of radiation shall not vary more than 10 % from the mean value calculated on all samples. The rotation rate of the carousel shall be from 1 min$^{-1}$ to 5 min$^{-1}$.

The irradiation of the test specimens shall be selectable between turning sample operations (periodic light-dark cycles) or stationary sample operations (permanent exposure to light).

A humidified stream of air shall pass through the test chamber over the surfaces of the specimens.

The relative humidity of the test chamber shall be controlled by a contact hygrometer.

**6.5.5** *Cover mask*. For a partial exposure of the test specimens to light cover masks of stainless steel are required, opening after specified times an unexposed section of the surface of the specimens, of a minimum size of 30 mm × 15 mm, for exposure.

The masks shall tightly cover the unexposed part of the specimen so that a sharp borderline is produced between the unexposed and the exposed parts.

In order to avoid adhesion of the adhesive coat to the test specimen it is useful to coat the surface of the cover mask in direct contact with the specimen with an inert release agent (e.g. a polyfluorohydrocarbon).

**6.5.6** *Black panel temperature sensor*, for measuring the temperature of the surface of the test specimens when tested.

**6.5.7** *Radiation measuring device*, allowing measurement of the intensity (W/m$^2$) and/or the dosage of radiation (J/m$^2$) on the level of the test specimens.

NOTE For assessing the dosage of radiation the Blue Wool Reference Standard specified in ISO 105-B02 should be used.

## 7 Test specimens

### 7.1 Preparation of the test specimens

Take a sample of the adhesive to be tested in accordance with EN 1066 and examine and prepare it in accordance with EN 1067.

Pour a test portion of this sample on a sheet of cardboard (see **6.1**) and spread the adhesive using a coating device (see **6.3**) to give coats of size (100 mm × 30 mm).

During coating use a generous quantity of adhesive of thickness (0,1 ± 0,02) mm after complete evaporation of all volatile adhesive constituent (e.g. water, solvents, etc.).

The dry coat shall be of uniform thickness, have uniform surface structure and shall not contain any bubbles.

To fulfil these requirements, the adhesive to be tested shall be applied on cardboard (see **6.1**) or on a glass sheet (see **6.2**), if necessary after dilution with a suitable solvent, in several coats (always after drying the preceding coat).

If the cardboard (see **6.1**) is deformed or damaged by the application of the adhesive, prepare an adhesive film by coating the adhesive on a glass sheet (see **6.2**) and attach the film in full contact to the cardboard (see **6.1**) forming a test specimen.

A suitable release agent shall be used in a required amount to facilitate or to allow separation of the adhesive film from the glass sheet.

Care shall be taken when attaching the adhesive film to the cardboard to ensure that the surface covered with the release agent is brought into contact with the cardboard.

### 7.2 Number of test specimens

Unless otherwise indicated prepare and use, for each test, five test specimens.

NOTE Some adhesive coats can also change their colour during storage in the dark. In such a case a reference test specimen should be stored in the dark to determine the colour change during the test period in accordance with this standard and this should be recorded in the test report.

## 8 Procedure

### 8.1 Storage of the test specimens

Store all specimens until completely set in the dark for at least 10 days in a standard atmosphere of 23 °C/50 % RH in accordance with ISO 554.

### 8.2 Colour measurement of unexposed specimens

Standardize the colorimeter using a white reference standard (standard illuminant D65, standard observer 10°, geometry of measurement d/8°) in accordance with the colorimeter manufacturer instructions.

Determine the CIE L$^*$, a$^*$ and b$^*$ values of the test specimens with the colorimeter, in accordance with CIE publication No 15.2.

Note the values in the test report.

**8.3 Exposure of the test specimens to light**

Mount the test specimens partly covered by the mask (see **6.5.5**) and the holders in such a manner that none of them varies its position in the exposure apparatus (see **6.5**) with respect to the light source during exposure.

Operate the machine with all holders regardless of the number of test specimens inserted.

Position a temperature sensor (see **6.5.6**) and a radiation measuring device (see **6.5.7**) in such a manner that they receive the same radiation as the surface of the test specimens.

During exposure turn all test specimens with the holders periodically by 180° from light to dark and from dark to light position.

Control the power of the machine during exposure permanently. To avoid a change in surface structure ensure that the surface temperature of the test specimens does not exceed 45 °C.

Record the surface temperature measured in the test report.

Do not interrupt the exposure of the test pieces except for short time maintenance work required by the machine (e.g. change of a xenon burner or a filter, or cleaning of a filter, etc.) or for the movement of the cover mask of the specimens (see **8.4.2** and **8.4.3**).

**8.4 Time of the exposure to light**

The general composition of adhesive manufactured from materials of extremely different light stability does not permit the same time or the same stages of exposure to light to be specified for all adhesive coats.

The time or time stages of exposure have to be adjusted to the light stability of the test specimens and to the requirements of the adhesive in service.

Preliminary tests shall be carried out if required.

Practical experience demands for the following tests.

**8.4.1** ***Light stability test with a specified time of exposure***

The test specimen shall not show any change of colour or any change beyond a specified limit.

**8.4.2** ***Discoloration test without a specified time of exposure***

The change of colour of one test specimen or different test specimens is evaluated during the course of time.

**8.4.3** ***Comparative test to a standard without a specified time of exposure***

The change of colour of one test specimen or different test specimens is evaluated during the course of time in comparison to a specified standard.

During testing according to **8.4.2** and **8.4.3** uncover parts of the test specimens depending on the change of colour observed after selected periods of time by shifting the cover mask section by section.

Continue the exposure to light as long as precise evaluation is possible.

For a comparative test to a standard (see **8.4.3**) the use of a radiation measuring device (see **6.5.7**) is not required.

**8.5 Colour measurement of exposed specimens**

This test shall be carried out as described in **8.2**.

Before measurement store, if necessary, all test specimens in the dark at standard atmosphere 23 °C/50 % RH in accordance with ISO 554.

If the colour of the test specimens could change with time, measure immediately or store at −20° in a black plastic bag.

The black plastic bag shall be free of butyl hydroxytoluene (BHT) or related species, a cause of discoloration by migration, to avoid interference or false results.

## 9 Expression of results

**9.1 Determination of colour**

Express the results by indicating the CIE colour scale values $L^*$, $a^*$ and $b^*$ of each tested specimen according to the CIE recommendations (CIE publication No 15.2).

All values shall represent the arithmetic mean in accordance with ISO 2602 of at least five readings.

Record all values in the test report.

**9.2 Determination of colour change**

Express the results by indicating the CIE colour scale values $L^*$, $a^*$ and $b^*$ of each tested specimen both unexposed and exposed to light and the CIE colour differences $\Delta L^*$, $\Delta a^*$, $\Delta b^*$ and $\Delta E^*_{ab}$ of these values calculated by a colorimeter (see **6.4**) in accordance with the CIE recommendations (CIE publication No 15.2). All values shall represent the arithmetic mean according to ISO 2602 of at least five readings.

Record all values in the test report.

NOTE The yellowing of near-white or near-colourless specimens determined by the colorimeter (see **6.4**) is sometimes expressed by the yellowness index (YI) characterizing the magnitude of yellowness as a deviation in chroma from whiteness or water whiteness in the dominant wavelength (ranging from 570 nm to 580 nm).

## 10 Test report

The test report shall include:

a) a reference to this European Standard;

b) complete identification of the adhesive tested, in particular the name, designation, manufacturer, type and date of manufacture, supply, number of batch, etc.;

c) detailed description of the preparation of the test specimens, preparation of the adhesive coat or film, coating device used, coat or film thickness, number of coats and, if required, the dilution of the adhesive, complete identification of the cardboard by indicating manufacturer, whiteness, unit weight and surface structure;

d) the rotation rate of the carousel used in the test chamber;

e) a description of the colorimeter used (type, manufacturer, calibration, etc.);

f) type and conditions of the colour measurement (type of illumination and observer, geometry of measurement). If the test specimens are exposed to light for the determination of a colour change, description of type, manufacturer and details of the light exposure machine (type of burner, filter system, etc.) and the conditions of the exposure (time, stages, and intensity of irradiation, surface temperature of the specimens, etc.);

g) test results in accordance with clause **9**;

h) any modification to the procedure described, and any incident which may have affected the results;

i) the date of the test.

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