
Tissue paper and tissue products —
Part 7:
Determination of optical properties —
Measurement of brightness and colour
with D65/10° (outdoor daylight)

Papier tissue et produits tissue —

*Partie 7: Détermination des propriétés optiques — Mesurage du
degré de blancheur et de la couleur avec l'illuminant D65/10°
(lumière du jour extérieure)*



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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).

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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

ISO 12625-7 was prepared by European Committee for Standardization (CEN) Technical Committee CEN/TC 172 *Pulp, paper and board*, in collaboration with Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 2, *Test methods and quality specifications for paper and board*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 12625-7:2007), which has been technically revised.

ISO 12625 consists of the following parts, under the general title *Tissue paper and tissue products*:

- Part 1: General guidance on terms;
- Part 3: Determination of thickness, bulking thickness, apparent bulk density and bulk;
- Part 4: Determination of tensile strength, stretch at break and tensile energy absorption;
- Part 5: Determination of wet tensile strength;
- Part 6: Determination of grammage;
- Part 7: Determination of optical properties — Measurement of brightness and colour with D65/10° (outdoor daylight);
- Part 8: Water-absorption time and water-absorption capacity; basket-immersion test method;
- Part 9: Determination of ball burst strength;
- Part 11: Determination of wet ball burst strength;
- Part 12: Determination of tensile strength of perforated lines — Calculation of perforation efficiency;
- Part 15 Determination of optical properties — Measurement of brightness and colour with C/2° (indoor daylight)
- Part 16 Determination of optical properties — Opacity (paper backing) — Diffuse reflectance method

Introduction

Brightness and colour measurement may be performed under various illumination and observation conditions. This part of ISO 12625 deals with D65/10° conditions, which refer to an outdoor daylight.

C/2° conditions (indoor daylight) are considered in ISO 12625-15. Although both international standards deal with brightness and colour, results obtained are usually different and do not correlate.

Optical measurement are affected by the geometry of the instruments used and by the texture of the material. The design of the instrument to be used according to this part of ISO 12625, and the routine to be adopted for its calibration, are specified in ISO 2469 and ISO 11475.

The optical properties are related to the visual appearance of the material. Therefore, although optical properties are intrinsic properties of tissue paper, they are not functional properties.

Brightness shall not be confused with the optical property called CIE-whiteness that is based on reflectance data obtained over the full visible spectral range (VIS) in contrast to the measurement of brightness which is limited to the blue region of VIS.

Due to the importance for some countries three different test methods for the determination of optical properties were developed:

- Part 7: *Determination of optical properties — Measurement of brightness and colour with D65/10° (outdoor daylight);*
- Part 15: *Determination of optical properties — Measurement of brightness and colour with C/2° (indoor) daylight;*
- Part 16: *Determination of optical properties — Opacity (paper backing) — Diffuse reflectance method;*

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Tissue paper and tissue products —

Part 7:

Determination of optical properties — Measurement of brightness and colour with D65/10° (outdoor daylight)

1 Scope

This part of ISO 12625 specifies testing procedures for the instrumental determination of brightness and colour of tissue paper and tissue products viewed under outdoor daylight conditions. It also gives specific instructions for the preparation of test pieces (single-ply, multi-ply products) and for the optical measurements of products, where special precautions may be necessary.

NOTE The properties called C/2° brightness and colour are measured with an instrument adjusted to a much lower UV content than that specified in this part of ISO 12625. The measurements of C/2° brightness and colour are described in ISO 12625-15.

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 186, *Paper and board — Sampling to determine average quality*

ISO 187, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples*

ISO 2469:2007, *Paper, board and pulps — Measurement of diffuse radiance factor*

ISO 5631-2:2008, *Paper and board — Determination of colour by diffuse reflectance — Part 2: Outdoor daylight conditions (D65/10 degrees)*

ISO 11475, *Paper and board — Determination of CIE whiteness, D65/10 degrees (outdoor daylight)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 reflectance factor

R

ratio of the radiation reflected by a body to that reflected by the perfect diffuser under the same conditions of illumination and detection

Note 1 to entry: The reflectance factor is expressed as a percentage.

Note 2 to entry: The reflectance factor is influenced by the backing if the body is translucent.

3.2 diffuse (radiance) reflectance factor

R

ratio of the reflection from a body to that from the perfect reflecting diffuser under the same conditions of diffuse illumination and normal detection

Note 1 to entry: The ratio is often expressed as a percentage.

Note 2 to entry: Adapted from ISO 2469:2007, definition 3.5.

3.3 intrinsic reflectance factor

R_{∞}

diffuse reflectance factor of a layer or pad of material thick enough to be opaque, i.e. such that increasing the thickness of the pad by doubling the number of sheets results in no change in the measured reflectance factor

[SOURCE: ISO 2469:2007, definition 3.6]

3.4 D65 brightness

intrinsic reflectance factor measured with a reflectometer having the characteristics described in ISO 2469, equipped with a filter or corresponding function having an effective wavelength of 457 nm (and a half bandwidth of 44 nm), and adjusted so that the UV content of the irradiation incident upon the test piece corresponds to that of the CIE standard illuminant D65

Note 1 to entry: The filter function is described more fully by the weighing function factors given in ISO 2470-2[2].

3.5 tristimulus values

X_{10}, Y_{10}, Z_{10}

amounts of the three reference colour stimuli, in a given chromatic system, required to match the stimulus considered

Note 1 to entry: In ISO 5631-2 the CIE standard illuminant D65 and the CIE 1964 (10°) standard observer are used to define the trichromatic system.

Note 2 to entry: The subscript 10 is applied to conform to the CIE convention that tristimulus units have the subscript 10 when the CIE 1964 (10°) standard observer is used.

[SOURCE: ISO 5631-2:2008, definition 3.5]

3.6 colour (D65/10°)

L^* , a^* and b^* values of the sample according to the CIELAB 1976 system, corresponding to the CIE standard illuminant D65, described in ISO 11664-2 and the CIE 1964 supplementary standard colorimetric observer, described in ISO 11664-1, [2] determined by measurement under the conditions specified in ISO 5631-2

Note 1 to entry: The quantity L^* is a measure of the lightness of the test piece, where $L^* = 0$ corresponds to black and $L^* = 100$ is defined by the perfect reflecting diffuser. Visually, the quantities a^* and b^* represent respectively the red-green and yellow-blue axes in colour space, such that

- $+a^*$ is a measure of the degree of redness of the test piece;
- $-a^*$ is a measure of the degree of greenness of the test piece;
- $+b^*$ is a measure of the degree of yellowness of the test piece;
- $-b^*$ is a measure of the degree of blueness of the test piece;
- if both a^* and b^* are equal to zero, the test piece is grey.

4 Principle

A test piece is illuminated diffusely in a standardized instrument and the light reflected normal to the surface is either allowed to pass through a defined optical filter and then measured by a photodetector or measured by an array of photosensitive diodes, where each diode responds to a different effective wavelength. The brightness is then determined directly from the output from the photodetector or by calculation from the photosensitive diode outputs using the appropriate weighting function and colour coordinates are calculated for D65/10° conditions.

5 Apparatus

5.1 Reflectometer or spectrophotometer, having the geometric, spectral and photometric characteristics described in ISO 2469 and calibrated in accordance with the provisions of ISO 2469, and equipped for the measurement of blue reflectance factor

5.1.1 In the case of a filter reflectometer, the radiation falling upon the test piece shall have a UV content corresponding to that of the CIE standard illuminant D65, adjusted or verified by the help of the fluorescent reference standard (5.2.2).

5.1.2 In the case of an abridged spectrophotometer, the instrument shall have an adjustable filter with a cut-off wavelength of 395 nm or some other system for adjustment and control, and this filter shall be adjusted or the system shall be calibrated with the help of the fluorescence reference standard (5.2.2), so that the UV content of the illumination falling upon the sample corresponds to that of the CIE standard illuminant D65.

5.2 Reference standard for calibration of the instrument

5.2.1 Non-fluorescent reference standard for photometric calibration, issued by an ISO/TC 6 authorized laboratory in accordance with the provisions of ISO 2469.

5.2.2 Fluorescent reference standard for use in adjusting the UV content of the radiation incident upon the sample, having a CIE whiteness (D65/10°) unit assigned by an ISO/TC 6 authorized laboratory as prescribed in ISO 11475:2004, Annex B.

NOTE 1 Greater precision in the D65 brightness measurement would be attained if a fluorescent reference standard having an assigned D65 brightness unit were used. It is, however, important for the industry to have only one UV-filter adjustment for all measurements under CIE illuminant D65 conditions. For this reason, a reference standard having an assigned CIE whiteness (D65/10°) values as prescribed in ISO 11475 is preferred.

NOTE 2 To be used sufficiently frequently to ensure satisfactory calibration and UV adjustment.

5.3 Working standards

5.3.1 Two plates of flat opal glass, ceramic or other suitable non-fluorescent material, cleaned and calibrated as described in ISO 2469.

NOTE In some instruments, the function of the primary working standard may be taken over by a built-in internal standard.

5.3.2 Stable plastic or other tablet, incorporating a fluorescent whitening agent.

5.3.3 Black cavity, having a reflectance factor which does not differ from its nominal value by more than 0,2 %, at all wavelengths. The black cavity should be stored upside down in a dust-free environment or with a protective cover.

NOTE The condition of the black cavity can be checked by reference to the instrument maker.

6 Calibration

6.1 Using the values assigned to the non-fluorescent reference standard (5.2.1), calibrate the instrument according to the instrument maker's instruction with the UV-cut-off filters removed from the radiation beams. The setting of the UV-adjustment filter is not important at this stage.

6.2 Using the appropriate measurement procedure, measure the radiance factors of the fluorescent reference standard (5.2.2); calculate the whiteness values and compare the value obtained with that assigned to the fluorescent reference standard.

A measured whiteness unit higher than the assigned unit indicates that the relative UV-content is too high and vice versa.

6.3 Using the UV-adjustment filter or other adjustment device, adjust the UV-content of the illumination until measurement gives the correct whiteness value.

6.4 Repeat the calibration as described in 6.1 using the non-fluorescent reference standard (5.2.1) with the UV-adjustment filter in the position which gave the correct whiteness value. Repeat the measurement of the whiteness of the fluorescent reference standard (5.2.2) as described in 6.2. If the whiteness unit obtained does not agree with the assigned value, adjust the position of the UV-adjustment filter until measurement gives the correct whiteness value as described in 6.3.

6.5 Repeat 6.4 until the correct value for the whiteness of the fluorescent reference standard is obtained with the instrument correctly calibrated to the non-fluorescent reference standard. The UV-content is now correctly adjusted with respect to whiteness to a relative UV-content equivalent to the D65 illuminant. Record the setting of the UV-adjustment.

NOTE 1 This setting is equivalent to the D65 illuminant and CIE 1964 (10°) observer with respect to whiteness. Variations in the green/red tint value may still arise and it cannot be assumed that the tristimulus values and other parameters will also be exactly those applicable to the D65 illuminant.

NOTE 2 In some instruments, the procedure indicated in 6.2 to 6.5 is performed automatically.

6.6 Assign reference values to working standards.

Perform D65/10° brightness and CIE- L^* , a^* and b^* measurements on the non-fluorescent material (5.3.1). Assign these reference values to the non-fluorescent material as working standard.

Perform D65/10° brightness and CIE- L^* , a^* and b^* measurements on the fluorescent material (5.3.2). Assign these reference values to the fluorescent material as working standard.

This working standard may only be used in the specific instrument in which its value was assigned and shall only be used to monitor changes in the lamps. A new value shall be assigned with a fluorescent reference standard of level 3 (5.2.2), if the lamps are changed or the used working standards show significant deviations.

NOTE Instead of using L^* , a^* and b^* values R_x , R_y , R_z can be used as assigned reference values.

7 Sampling

If the tests are being made to evaluate a lot, the sample shall be selected in accordance with ISO 186. If the tests are being made on another type of sample, make sure, the specimens taken are representative of the sample received.

When sampling finished roll products, eliminate at least the first six layers and the last six layers because of the possible presence of adhesive or mechanical damage.

Mark the samples for identification, and make sure that the two sides of the paper or of the product can be distinguished.

8 Conditioning

Condition the samples according to ISO 187, and keep them in the standard atmosphere throughout the test. Preconditioning with elevated temperatures should not be applied since it might change the optical properties.

9 Preparation of test pieces

Cut test pieces of at least 50 mm x 50 mm or 50 mm in diameter, which are free from dirt, perforation and any defect. Assemble sufficient test pieces in a pad with their top sides uppermost; the number of test pieces should be such that doubling the number does not alter the reflectance factor.

Protect the pad by placing a protecting sheet on both the top and bottom of the pad. Avoid contamination and unnecessary exposure to light or heat.

If the pads are very voluminous and bulky, steps shall be taken to expel the air. The pads should be carefully compressed between the protecting sheets.

Mark the pad in one corner to identify the sample and the marked side.

10 Procedure

10.1 General

Remove the protecting sheets from the pad of test pieces and measure the optical properties on the top side and if required on the reverse side of the test pieces, as described in the relevant subclause below.

Steps should be taken, without damaging the material, to ensure that the pad is pressed against the measuring opening under sufficient pressure to give a compact pad which does not intrude into the measurement sphere.

10.2 Measurement of D65 brightness

The UV-content of the illumination shall be adjusted to correspond to the D65 illuminant, as described in [Clause 6](#).

Remove the protecting sheets from the pad of the test pieces and measure the D65 brightness (reflectance factor at an effective wavelength of 457 nm) of the marked side of the test-piece pad. Read and record the value to the nearest 0,05 % reflectance factor or better. Move the uppermost test piece to the bottom of the pad and determine the reflectance factor for the next test piece, and similarly for the following test pieces, until a total of at least 10 readings have been made.

If required, turn the test pad upside down and repeat the procedure on the other side. Calculate the D65 brightness as indicated in [11.1](#).

NOTE 1 If, in the case of fluorescent samples, measurements are made with 420 nm cut-off filters placed in the light beams, it is possible to determine the D65 brightness without the contribution of the fluorescent whitening agent, but this is not in the scope of this part of ISO 12625.

NOTE 2 In the case of non-fluorescent samples, the D65 brightness and the ISO C/2° brightness are identical.

10.3 Measurement of colour (D65/10°)

The UV-content in the illumination is adjusted to match the CIE standard illuminant D65, as described in [Clause 6](#).

If a filter instrument is used, check that the correct filters are inserted in the light beams.

Remove the protecting sheets from the pad of test pieces and determine the X_{10}, Y_{10} and Z_{10} tristimulus values for the marked side of the top test piece. Record the results to the nearest 0,1 value, move the uppermost test piece to the bottom of the pad and repeat the measurement for the next; proceed in this way until at least 10 readings have been made. If required, turn the test pad upside down and repeat the procedure on the other side. Calculate the CIELAB colour coordinates for each test piece. Calculate the colour coordinates as indicated in 11.2.

11 Calculation

11.1 D65 brightness

Calculate the mean brightness value as the D65-brightness of the sample in percent to the nearest 0,1 % and its standard deviation.

In case of measurement on both sides calculate and report the mean brightness value for both sides separately.

11.2 Colour (D65/10°)

11.2.1 Single value

Calculate the single colour values as CIE- L^* , a^* and b^* coordinates of the sample from the tristimulus values X , Y , Z , by means of the following equations:

$$L^* = 116 \left(\frac{Y}{Y_n} \right)^{\frac{1}{3}} - 16 \quad (1)$$

$$a^* = 500 \left[\left(\frac{X}{Y_n} \right)^{\frac{1}{3}} - \left(\frac{Y}{Y_n} \right)^{\frac{1}{3}} \right] \quad (2)$$

$$b^* = 200 \left[\left(\frac{Y}{Y_n} \right)^{\frac{1}{3}} - \left(\frac{Z}{Z_n} \right)^{\frac{1}{3}} \right] \quad (3)$$

where X_n , Y_n , Z_n are the tristimulus values of the perfect reflecting diffuser under D65 conditions. These are given as the "white point" values in ISO 5631-1:2009, Annex A.

Alternative equations shall, however, be used if any of the ratios X/X_n , Y/Y_n , $Z/Z_n \leq (24/116)^3$ are satisfied as follows:

- If $(X/X_n) \leq (24/116)^3$, replace the term $(X/X_n)^{1/3}$ in Formula (2) by the expression $(841/108)(X/X_n) + 16/116$.
- If $(Y/Y_n) \leq (24/116)^3$, replace the term $(Y/Y_n)^{1/3}$ in Formula (1), (2) and (3) by the expression $(841/108)(Y/Y_n) + 16/116$.
- If $(Z/Z_n) \leq (24/116)^3$, replace the term $(Z/Z_n)^{1/3}$ in Formula (2) by the expression $(841/108)(Z/Z_n) + 16/116$.

NOTE 1 The term $(25/116)^3$ is approximately equal to 0,008 856.

NOTE 2 The term $(841/108)^3$ is approximately equal to 7,787.

NOTE 3 Formula (1) transforms to $L^* = 903,3 (Y/Y_n)$ when $(Y/Y_n) \leq (24/116)^3$.

11.2.2 Mean value

Calculate the mean colour values as CIE- L^* , a^* and b^* values of the sample to the nearest 0,1 value. In case of measurements on both sides calculate and report the mean colour value for both sides separately.

11.2.3 Dispersion of the results

Since the three-dimensional statistical calculations are extremely complicated, the following simple procedure for assessing the dispersion is recommended.

Calculate, for each test piece, the deviation ΔE_{ab}^* from the mean according to Formula (4):

$$\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4)$$

where ΔL^* , Δa^* and Δb^* are the differences between the L^* , a^* and b^* values of the test piece and the corresponding mean values $\langle L^* \rangle$, $\langle a^* \rangle$ and $\langle b^* \rangle$.

Calculate the mean $\langle \Delta E_{ab}^* \rangle$ value. This is known as the Mean Colour Difference from the Mean (MCDM) value and defines the dispersion in terms of a sphere of radius $\langle \Delta E_{ab}^* \rangle$ about the mean point in CIELAB space.

NOTE This calculation uses the expression for the colour differences between two samples which can be calculated in these coordinates as:

$$\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (5)$$

where ΔL^* , Δa^* and Δb^* are the differences between the values of the two samples.

12 Test report

The test report shall include the following information:

- a reference to this part of ISO 12625, i.e. ISO 12625-7:2014;
- date and place of testing;
- conditioning atmosphere used;
- all details necessary to identify the material;
- the type of instrument used and a reference to the Authorized Laboratory providing ISO reference standards of level 3 for calibration of the instrument;
- the mean and standard deviation D65 brightness and/or the D65 colour for each side and if required, the mean of the two sides;
- any departure from this part of ISO 12625 or any other circumstance that may have affected the results.

Annex A (informative)

Precision

A.1 General

In July 2012, an international round-robin was performed by nine different laboratories according to this part of ISO 12625.

For brightness measurement, six samples of white and near white base papers and converted products were considered. For colour measurements, three additional coloured products were also considered.

For each series of sample, 10 measurements have been performed on the referenced side.

A.2 Brightness

For Brightness, the calculations have been made according to ISO/TR 24498,^[4] and TAPPI T 1200, sp-07.^[5]

The repeatability standard deviation reported in [Table A.1](#) is the "pooled" repeatability standard deviation that is, the standard deviation is calculated as the root-mean-square of the standard deviations of the participating laboratories. This differs from the conventional definition of repeatability in ISO 5725-1.^[6]

The repeatability and reproducibility limits reported are estimates of the maximum difference which should be expected in 19 of 20 instances, when comparing two test results for material similar to those described under similar test conditions. These estimates may not be valid for different materials or different test conditions.

Repeatability and reproducibility limits are calculated by multiplying the repeatability and reproducibility standard deviations by 2,77.

NOTE $2,77 = 1,96 \sqrt{2}$, provided that the test results have a normal distribution and that the standard deviation s is based on a large number of tests.