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Tissue paper and tissue products —

Part 15: Determination of optical properties — Measurement of brightness and colour with C/2° (indoor daylight) illuminant

Papier tissue et produits tissue —

Partie 15: Détermination des propriétés optiques — Mesurage du degré de blancheur et de la couleur avec l'illuminant C/2° (lumière du jour à l'intérieur)

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Foreword

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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).

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 12625-15 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 for quality specifications for paper and board*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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 C/2° conditions, which refer to an indoor daylight.

D65/10° conditions (outdoor daylight) are considered in ISO 12625-7. Although both standards deal with brightness and colour, results obtained are usually different and do not correlate.

Optical measurements 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 2470-1.

The optical properties are related to the visual appearance of the material in a specified illumination condition. Although optical properties are intrinsic properties of tissue paper, they are not functional properties.

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

As preferences for the properties to be specified can vary by country/market, 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 — Measurement of opacity (paper backing) — Diffuse reflectance method*.

Tissue paper and tissue products —

Part 15:
Determination of optical properties — Measurement of brightness and colour with C/2° (indoor daylight) illuminant

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 in indoor 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 D65 brightness and colour are measured with an instrument adjusted to a much higher UV content than that specified in this part of ISO 12625. The measurements of D65 brightness and colour are described in ISO 12625-7.

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, *Paper, board and pulps — Measurement of diffuse radiance factor*

ISO 2470-1:2009, *Paper, board and pulps — Measurement of diffuse blue reflectance factor — Part 1: Indoor daylight conditions (ISO brightness)*

ISO 5631-1:2009, *Paper and board — Determination of colour by diffuse reflectance — Part 1: Indoor daylight conditions (C/2°)*

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 reflectance radiance 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.

[SOURCE: ISO 2469:2007]

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]

3.4
C/2° 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 illuminant C

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

3.5
tristimulus values
X, Y, Z
amount of the three reference colour stimuli, in a given chromatic system, required to match the stimulus considered

Note 1 to entry: In this part of 12625 as in ISO 5631-1, the CIE standard illuminant C and the CIE 1931 (2°) standard observer are used to define the trichromatic system.

Note 2 to entry: No subscript is applied to conform to the CIE convention that tristimulus values have no subscript when the CIE 1931 (2°) standard observer is used [the subscript 10 is applied for tristimulus values that are obtained using the CIE 1964 (10°) standard observer].

[SOURCE: ISO 5631-1:2009]

3.6
C/2° colour
CIELAB colour space, L^*, a^*, and b^* coordinates of the sample according to the CIELAB 1976 system, corresponding to the CIE standard illuminant C, described in ISO 11664-2 and the CIE 1964 supplementary standard colorimetric 2° observer, described in ISO 11664-1, determined by the measurements obtained under the conditions specified in ISO 5631-1

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, and

— $-b^*$ 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 standard 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 C/2° conditions.

5 Apparatus

5.1 Reflectometer or spectrophotometer

The apparatus have the geometric, spectral, and photometric characteristics described in ISO 2469 and calibrated in accordance with the provisions of ISO 2469 and ISO 2470-1, 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 C, adjusted or verified by using 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 C.

5.2 Reference standard for calibration of the instrument

5.2.1 Non-fluorescent reference standard, for photometric calibration, issued by an 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 C/2° brightness value assigned by an authorized laboratory as prescribed in ISO 2470-1:2009, Annex B.

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 standard, incorporating a fluorescent whitening agent.

5.3.3 Black cavity, having a reflectance factor which does not differ from its nominal values 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 instruments 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 C/2° brightness value of the fluorescent reference standard (5.2.2) and compare the value with the assigned to the fluorescent reference standard.

A measured C/2° brightness value higher than that 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 C/2° brightness value.

6.4 Repeat 6.1, 6.2, and 6.3 until the correct value for the C/2° brightness 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 a relative UV-content equivalent to the CIE illuminant C. Record the setting of the UV-adjustment.

NOTE In some instruments, the procedure indicated in 6.2 to 6.4 is performed automatically.

6.5 Assign reference values to working standards.

Perform C/2° 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 C/2° 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 deviations more than 1 brightness unit.

NOTE Instead of using L^*, a^*, b* values, R_x, R_y, R_z may also 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 shall not be applied since it might change the optical properties.

9 Preparation of test pieces

Cut test pieces of at least 50 mm × 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. Measure the optical properties on the top side and, if required, the reverse side as well following the procedure 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 C/2° brightness

The UV-content of the illumination shall be adjusted to correspond to the CIE illuminant C.

Measure the C/2° 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 C/2° brightness as indicated in 11.1.

NOTE 1 In the case of fluorescent samples, if measurements are made with 420 nm cut-off filters placed in the light beams, it is possible to determine the C/2° brightness without the contribution of the fluorescent whitening agent, but this is outside of the scope of this International Standard.

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

10.3 Measurement of (C/2°) colour

The UV-content in the illumination shall be adjusted to correspond to the CIE illuminant C.

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

Determine the *X*, *Y*, and *Z* tristimulus values for the marked side of the top test piece (or CIELAB values, if the instrument is designed to report directly in this colour space). Record the results to the nearest 0,1 unit, 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 C/2° colour coordinates as indicated in 11.2.

11 Calculation

11.1 C/2° brightness

Calculate the mean brightness value as the C/2° brightness of the sample in percent to the nearest 0,1 % and its standard deviation.

When both sides (top and reverse side) are measured, calculate and report the mean brightness value for the two sides separately.

11.2 C/2° Colour

11.2.1 Single values

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 formulae:

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

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

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

where X_n, Y_n, Z_n are the tristimulus values of the perfect reflecting diffuser under C/2° conditions. These are given as the "white point" values in ISO 5631-1:2009, Annex A.

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

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

b) 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 $(841/108)\,(Y/Y_n) + 16/116$.

c) If $(Z/Z_n) \leq (24/116)^3$, replace the term $(Z/Z_n)^{1/3}$ in Formula (3) by the expression $(841/108)\,(Z/Z_n) + 16/116$.

NOTE 1 The term $(24/116)^3$ is approximately equal to 0,008 856.

NOTE 2 The term (841/108) 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 C/2° colour values as CIE-L^*, a^*, and b^* coordinates of the sample to three significant figures.

When both sides (top and reverse side) are measured, calculate and report the mean CIE-L^*, a^*, and b^* values for two 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 the mean values $< L^* >$, $< a^* >$, and $< b^* >$ of the L^*, a^*, and b^* values.

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 $< L^* >$, $< a^* >$, and $< b^* >$.

Calculate the mean $< \Delta E_{ab}^* >$ value. This is known as the mean C/2° colour difference from the mean (MCDM) value and defines the dispersion in terms of a sphere radius $< \Delta E_{ab}^* >$ about the mean point in CIELAB space.

NOTE This calculation uses the expression for the C/2° colour differences between two samples which may 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) a reference to this part of ISO 12625;

b) date and place of testing;

c) conditioning atmosphere used;

d) all details necessary to identify the material;

e) the type of instrument used and a reference to the authorized laboratory providing ISO reference standards of level 3 for calibration of the instrument;

f) the mean and standard deviation C/2° brightness and/or the C/2° colour values and if required, the mean of the two sides (top and reverse side);

g) 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 interlaboratory test was performed on six samples by nine different laboratories according to this part of ISO 12625. White and near white base papers and converted products were considered for all the tested properties, but for colour measurements, three coloured products were also assessed.

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

The calculations were made according to ISO/TR 24498[4] and TAPPI T 1200.[5]

The repeatability standard deviation reported in Table A.1 is the "pooled" repeatability standard deviation that is the standard deviation that 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 (Table A.2) 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.

NOTE Repeatability and reproducibility limits are calculated by multiplying the repeatability and reproducibility standard deviations by 2,77, where $R = 1,96\sqrt{2} \cdot s$.

A.2 Brightness

Table A.1 — Estimation of repeatability

Samples	Number of laboratories	Mean brightness – UV C %	Repeatability standard deviation s_r %	Coefficient of variation $C_{V,r}$ %	Repeatability limit r %
toilet-4-ply white	9	82,2	0,2	0,2	0,4
toilet-3-ply white	7[b]	76,6	0,1	0,2	0,4
toilet-3-ply white	8[a]	57,2	0,2	0,3	0,5
hanky-4-ply white	8[a]	87,0	0,1	0,1	0,2
towel-2-ply white	8[a]	81,1	0,2	0,3	0,7
base sheet 1 ply	7[b]	84,6	0,1	0,41	0,3

[a] one outlier

[b] two outliers