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Tissue paper and tissue products —

Part 7: Determination of optical properties

Papier tissue et produits tissués —

Partie 7: Détermination des propriétés optiques

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Contents

Page

Foreword ... iv
Introduction ... v
1 **Scope** ... 1
2 **Normative references** ... 1
3 **Terms and definitions** ... 2
4 **Apparatus** ... 3
5 **Sampling and conditioning** ... 3
6 **Preparation of test pieces** ... 4
7 **Procedure** ... 4
8 **Calculation** ... 6
9 **Test report** ... 7
10 **Precision** ... 8
Annex A (informative) **Test methods available for the determination of optical properties** ... 9
Bibliography ... 10

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 12625-7 was prepared by the 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 co-operation 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 and apparent bulk density*

— *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*

— *Part 8: Water-absorption time and water-absorption capacity, basket-immersion test method*

— *Part 9: Determination of ball burst strength*

Introduction

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.

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. It is recommended that agreement with respect to the properties to be measured is reached by the parties concerned, from case to case. If it is desired to achieve a match with products based on other materials, for example, between table napkins and candles, visual comparison may be essential.

Tissue paper and tissue products —

Part 7:
Determination of optical properties

1 Scope

This part of ISO 12625 specifies test methods for the instrumental determination of optical properties of tissue paper and tissue products. The various test methods that are available have been, or are being, developed as International Standards. They are listed and explained in Annex A.

This part of ISO 12625 also gives recommendations regarding relevant optical properties to be measured for different grades of tissue paper and tissue products and gives specific instructions for the preparation of test pieces (single-ply, multi-ply products) and for the optical measurements of creped products and embossed products, where special precautions may be necessary if the test-piece surfaces are uneven and if the materials are bulky, so that air is entrapped between the sheets.

2 Normative references

The following referenced documents are indispensable for the application of this document. 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 reflectance factor*

ISO 2470:1999, *Paper and board — Measurement of diffuse blue reflectance factor (ISO brightness)*

ISO 2471, *Paper and board — Determination of opacity (paper backing) — Diffuse reflectance method*

ISO 5631, *Paper and board — Determination of colour (C/2°) — Diffuse reflectance method*

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

ISO 11476, *Paper and board — Determination of CIE whiteness, C/2° (indoor illumination conditions)*

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 reflecting diffuser under the same conditions of illumination and detection

NOTE 1 The reflectance factor is expressed as a percentage.

NOTE 2 The reflectance factor is influenced by the backing if the body is translucent.

3.2
diffuse reflectance factor
R
ratio of the reflection from a body to that reflected by the perfect reflecting diffuser under the same conditions of diffuse illumination and normal detection

NOTE The ratio is often expressed as a percentage.

3.3
intrinsic reflectance factor
R_∞
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 leads to no change in the measured reflectance factor

NOTE Adapted from ISO 2469:1994.

3.4
luminous reflectance factor
R_y
reflectance factor defined with reference to the CIE illuminant C, described in CIE Publication 15.2[1] and the CIE 1931 colour-matching function $\bar{y}$ (λ) (described in ISO/CIE 10527[2]), and corresponding to the attribute of visual perception of the reflecting surface

NOTE Adapted from ISO 2471:1998.

3.5
single-sheet luminous reflectance factor
R_0
luminous reflectance factor of a single sheet of paper with a black cavity as backing

[ISO 2471:1998]

3.6
diffuse blue reflectance factor
ISO brightness
R_{457}
intrinsic reflectance factor measured with a reflectometer having the characteristics described in ISO 2469, Annex A, equipped with a filter or corresponding function having an effective wavelength of 457 nm and a width of 44 nm, described more fully by the weighting function factors given in Annex A and Table A.1 (in ISO 2470:1999), and adjusted so that the UV-content of the illumination incident upon the test piece corresponds to that of the CIE illuminant C

NOTE Adapted from ISO 2470:1999.

3.7
D65 brightness
intrinsic reflectance factor measured at an effective wavelength of 457 nm under the conditions specified in ISO 2470 when the UV-content of the illumination has been adjusted as specified in ISO 11475 to conform to the D65 illuminant (referred to in ISO 2470)

3.8
CIE-whiteness (C/2°)
W
measure of whiteness derived from the CIE tristimulus values corresponding to the CIE standard illuminant C, described in CIE Publication 15.2[1], and the CIE 1931 standard colorimetric observer, described in ISO/CIE 10527[2], determined under the conditions specified in ISO 11476

3.9
CIE whiteness (D65/10°)
W_{10}
measure of whiteness derived from the CIE tristimulus values corresponding to the CIE standard illuminant D65, described in ISO 10526[3] and the CIE 1964 supplementary standard colorimetric observer, described in ISO/CIE 10527[2], determined under the conditions specified in ISO 11475 and expressed as whiteness units

3.10
colour (C/2°)
L^*, a^* and b^* values of the sample according to the CIELAB 1976 system, corresponding to the CIE illuminant C, described in CIE Publication 15.2[1] and the CIE 1931 standard colorimetric observer, described in ISO/CIE 10527[2], determined by measurement under the conditions specified in ISO 5631

3.11
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 10526[3] and the CIE 1964 supplementary standard colorimetric observer, described in ISO/CIE 10527[2], determined by measurement under the conditions analogous to those specified in ISO 5631

3.12
opacity (paper backing)
ratio, expressed as a percentage, of the single-sheet luminous reflectance factor, R_0, to the intrinsic luminous reflectance factor, R_∞, of the same sample

[ISO 2471:1998]

4 Apparatus

4.1 Reflectometer, either a filter colorimeter or an abridged spectrophotometer, as specified in and calibrated according to ISO 2469.

Use the apparatus as specified in the relevant International Standard.

5 Sampling and conditioning

Unless otherwise agreed between the parties concerned, take samples in accordance with ISO 186 and condition them in accordance with ISO 187. Mark the samples for identification, and make sure that the two sides of the paper or of the product can be distinguished.

If testing is to be carried out on a sample as received, ensure that the test pieces taken are representative of this sample.

6 Preparation of test pieces

Before preparing the test pieces, it may be necessary to consider whether measurements are to be made on a material or on a product. A single test piece may be a multi-ply item, even in the case of a material if it is produced as, for example, a two-ply sheet.

Avoiding watermarks, dirt and obvious defects of the sample, cut rectangular test pieces of about 75 mm × 150 mm. Assemble not less than 20 pieces with the top side upwards in a pad using, if necessary, a number greater than 20 to ensure that the pad is opaque (see 3.3).

Protect the pad by placing a protecting sheet on both the top and bottom. 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.

7 Procedure

7.1 General

Remove the protecting sheets from the pad of test pieces and measure the optical properties on the top side and 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.

7.2 Measurement of brightness and whiteness (of white or near-white materials)

Four different test methods are available for measuring brightness and whiteness under both C-illuminant and D65-illuminant conditions. Brightness is measured only in the blue region of the spectrum, whereas whiteness is calculated from the result of measurements made over the full visual spectral range. The CIE standard illuminant D65 has a significantly higher UV-content than the CIE illuminant C, and it is therefore more sensitive to the presence of fluorescent whitening agents in the paper. The D65 illuminant is considered to relate to outdoor conditions, whereas the C illuminant represents indoor daylight. The choice of which method to use should be agreed upon between the parties, in relation to the intended end-use situation.

7.2.1 ISO brightness (diffuse blue reflectance factor)

Using the procedure described in ISO 2470, which specifies that the UV-content of the illumination shall be adjusted to correspond to the C illuminant, measure the ISO 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 not less than ten readings has been made.

If required, turn the test pad upside down and repeat the procedure on the other side. Calculate the ISO brightness as indicated in 8.1.

7.2.2 D65 brightness

This method is being developed within ISO/TC 6, the essential difference from ISO 2470 being that the UV-content of the illumination shall be adjusted to correspond to the D65 illuminant as described in ISO 11475.

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 not less than ten readings has 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 8.2.

7.2.3 CIE whiteness (C/2°)

Operate and calibrate the apparatus as described in ISO 11476, with the UV-adjustment filter adjusted so that the UV-content of the illumination matches the CIE illuminant C, as described in ISO 2470.

Without touching the test area, use the procedure appropriate to the instrument and determine the CIE whiteness of the marked side of the test-piece pad. Read and record the value to the nearest whiteness unit and also record the tint to the first decimal place. Move the uppermost test piece to the bottom of the pad and determine the whiteness value for the next test piece, and similarly for the following test pieces, until a total of not less than ten readings has been made.

If required, turn the test pad upside down and repeat the procedure on the other side. Calculate the whiteness and tint values as indicated in 8.3.

If an assessment of the fluorescence component of the whiteness is required, place the specified UV cut-off filter in the light beam and measure the intrinsic radiance factor of each test piece without UV-excitation. Calculate the whiteness without UV-excitation as indicated in 8.3. Calculate the fluorescent component of the CIE whiteness as the difference between the two whiteness values measured with and without UV-excitation.

7.2.4 CIE whiteness (D65/10°)

Operate and calibrate the apparatus as described in ISO 11475, with the UV-adjustment filter adjusted as described in ISO 11475 so that the UV-content of the illumination matches the CIE standard illuminant D65.

Without touching the test area, use the procedure appropriate to the instrument and determine the CIE whiteness of the marked side of the test-piece pad. Read and record the value to the nearest whiteness unit and also record the tint to the first decimal place. Move the uppermost test piece to the bottom of the pad and determine the whiteness value for the next test piece, and similarly for the following test pieces, until a total of not less than ten readings has been made.

If required, turn the test pad upside down and repeat the procedure on the other side. Calculate the whiteness and tint values as indicated in 8.4.

If an assessment of the fluorescence component of the whiteness is required, place the specified UV cut-off filter in the light beam and measure the intrinsic radiance factor of each test piece without UV-excitation. Calculate the whiteness without UV-excitation as indicated in 8.4. Calculate the fluorescent component of the CIE whiteness as the difference between the two whiteness values measured with and without UV-excitation.

7.3 Measurement of colour

Two methods are available for the measurement of colour, one for CIE illuminant C conditions and one for CIE standard illuminant D65 conditions. The D65 illuminant is considered to relate to outdoor conditions, whereas the C illuminant represents indoor daylight. The choice of which method to use should be agreed upon between the parties in relation to the intended end-use situation. If the colour coordinates are presented as a complement to the whiteness values, the choice of colour standard shall match the choice of whiteness standard.

7.3.1 Colour (C/2°)

Use the procedure described in ISO 5631. If there is a possibility that fluorescent agents may be present, the UV-content of the illumination shall be adjusted to match the CIE illuminant C, as described in ISO 2470.

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 either measure the R_x, R_y and R_z values or read and record the reflectance factor values and determine the X, Y and Z tristimulus values for the marked side of the top test piece. Record the results to the nearest 0,05 unit or better, 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 ten readings have been made. Invert the pad and repeat the procedure for the other side. Calculate the CIELAB colour coordinates for each test piece. Calculate the colour coordinates as indicated in 8.5.

7.3.2 Colour (D65/10°)

This method is being developed within ISO/TC 6. The essential difference from the determination in ISO 5631 is that the UV-content in the illumination is adjusted to match the CIE standard illuminant D65, as described in ISO 11475.

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 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 ten readings have been made. Invert the pad and repeat the procedure for the other side. Calculate the CIELAB colour coordinates for each test piece. Calculate the colour coordinates as indicated in 8.6.

7.4 Measurement of opacity

In the case of opacity, it is usually the product that is to be measured, i.e. a single test piece may be a multi-ply sheet. Do not attempt to separate multi-ply products into single plies.

In the case of a filter instrument, check that the correct filters are inserted in the light beams, as described in ISO 2471.

Remove the protecting sheets from the pad of test pieces. Place one test piece over the black cavity, with its marked side facing the sample aperture, and read the reflectance factor to the nearest 0,05 % reflectance factor unit or better; record the value as the luminous reflectance factor $R_{y,0}$. Repeat this for the same side of at least four more test pieces.

Beginning with the same sheet on the top of the pad, place the test-piece pad against the sample aperture and measure its intrinsic luminous reflectance factor $R_{y,\infty}$ to the nearest 0,05 % reflectance factor unit. Move the top sheet to the bottom of the pad and make a new measurement. Repeat this for a total of at least five measurements.

This text is not intended to imply that the two measurements shall necessarily be made in this order, nor that the measurements of R_∞ and R_0 shall be made alternately. The five measurements of R_0 may be made before or after the five measurements of R_∞, if such a procedure is preferred, or the measurements may be made alternately.

Invert the pad and repeat the procedure for the other side.

Calculate the opacity as indicated in 8.7.

8 Calculation

8.1 ISO brightness

Calculate the mean value for each side separately, if required. Otherwise, calculate the overall mean. Report the ISO brightness to the nearest 0,1 percentage unit.

8.2 D65 brightness

Calculate the mean value for each side separately, if required. Otherwise, calculate the overall mean. Report the D65 brightness to the nearest 0,1 percentage unit.

8.3 CIE whiteness (C/2°)

Calculate the mean CIE whiteness (C/2°) and the mean tint value for each side separately, if required. Otherwise, calculate the overall mean. Report the CIE whiteness (C/2°) to the nearest integer and the tint value with one decimal. Calculate and report the fluorescence component as the whiteness difference to the nearest integer.

8.4 CIE whiteness (D65/10°)

Calculate the mean CIE whiteness (D65/10°) and the mean tint value for each side separately, if required. Otherwise, calculate the overall mean. Report the CIE whiteness (D65/10°) to the nearest integer and the tint value with one decimal. Calculate and report the fluorescence component as the whiteness difference to the nearest integer.

8.5 Colour (C/2°)

Calculate the L^*, a^* and b^* values for each test piece separately. Calculate the mean L^*, a^* and b^* values. Report to the nearest 0,1 percentage unit.

8.6 Colour (D65/10°)

Calculate the L^*, a^* and b^* values for each test piece separately. Calculate the mean L^*, a^* and b^* values. Report to the nearest 0,1 percentage unit.

8.7 Opacity

Calculate the opacity, O, as a percentage, using the expression:

$$O = \frac{100 \times R_{y,0}}{R_{y,\infty}} \tag{1}$$

where

$R_{y,0}$ is the luminous reflectance factor of a single sheet over a black background;

$R_{y,\infty}$ is the luminous reflectance factor of an opaque pad.

Calculate the mean opacity for each side separately. If the values for the two sides differ by more than 0,2 %, the sides shall be identified and the results reported separately. If the difference is equal to or less than 0,2 %, the overall average shall be reported. Report the opacity to three significant figures.

9 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) if conditioning is agreed, kind of condition and conditioning atmosphere;

d) all details necessary to identify the material, including a statement of the number of plies as received and, if necessary, the location of individual plies;

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

f) a precise identification of the properties measured, including the illuminant (C or D65), and a reference to the relevant International Standard;

g) the mean of each side, if required, or the overall mean, expressed as specified in Clause 8 (and the standard deviations, if this is to be included);

h) any departure from the standard procedure or any other circumstances that may have affected the results.

10 Precision

In an interlaboratory study, including seven different tissue samples, twelve laboratories measured optical properties according to this part of ISO 12625. Only results (mean value and standard deviation between laboratories means) from laboratories (total 6 laboratories) using instruments and calibration procedure which conform with the requirements given in ISO 2469 are given in Table 1. Both samples containing and not containing fluorescent whitening agents, and samples with and without colour, are included. ΔW shows the contribution to the whiteness from the fluorescent whitening agent.

Table 1

Sample	A		B		M		N		O		H		J	
	not embossed		embossed		kitchen roll, embossed		base paper		base paper		red, 3-ply		blue, 3-ply	
	mean	s	mean	s	mean	s	mean	s	mean	s	mean	s	mean	s
C/2°														
R_x	86,1	0,2	85,5	0,3	88,9	0,4	93,5	0,3	85,0	0,2	27,9	0,6	6,3	0,1
R_y	84,3	0,2	87,0	0,3	87,4	0,4	92,9	0,3	84,5	0,2	14,2	0,7	7,9	0,3
R_z	75,8	0,5	77,0	0,6	77,1	0,3	91,8	0,2	86,9	0,2	6,6	0,7	17,8	0,4
L^*	93,6	—	94,7	—	94,9	0,1	97,2	0,1	93,6	0,1	44,5	—	33,8	—
a^*	− 0,9	—	− 5,9	—	− 1,5	0,1	0,6	0,1	1,8	0,1	48,2	—	6,3	—
b^*	7,4	—	7,6	—	7,8	0,1	0,7	0,1	− 1,8	0,2	23,5	—	− 26,7	—
ΔE[a]	0,17	—	0,34	—	0,16	0,08	0,17	0,06	0,20	0,10	3,08	—	1,58	—
opacity	43,2	1,3	74,2	0,3	—	—	—	—	—	—	—	—	—	—
D65/10°														
white-ness	54,7	0,5	52,3	1,2	50,6	0,7	90,0	0,2	92,4	0,6	—	—	—	—
ΔW_{10}	0		0		− 0,9		15,1		29,6		—		—	

[a] ΔE is the mean deviation from the mean CIELAB values.

NOTE The instruments used and the calibration procedure conform with the requirements of ISO 2469.