BS EN ISO 12625-4:2016



BSI Standards Publication

Tissue paper and tissue products

Part 4: Determination of tensile strength, stretch at maximum force and tensile energy absorption (ISO 12625-4:2016)



National foreword

This British Standard is the UK implementation of EN ISO 12625-4:2016. It supersedes BS EN ISO 12625-4:2005 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PAI/11, Methods of test for paper, board and pulps.

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Tissue paper and tissue products - Part 4: Determination of tensile strength, stretch at maximum force and tensile energy absorption (ISO 12625-4:2016)

Papier tissue et produits tissue - Partie 4: Détermination de la résistance à la rupture par traction, de l'allongement à la force maximale et de l'absorption d'énergie à la rupture par traction (ISO 12625-4:2016)

Tissue-Papier und Tissue-Produkte - Teil 4: Bestimmung der breitenbezogenen Bruchkraft, der Dehnung bei maximaler Kraft und des Arbeitsaufnahmevermögens (ISO 12625-4:2016)

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European foreword

This document (EN ISO 12625-4:2016) has been prepared by Technical Committee ISO/TC 6 "Paper, board and pulps" in collaboration with Technical Committee CEN/TC 172 "Pulp, paper and board" the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by June 2017, and conflicting national standards shall be withdrawn at the latest by June 2017.

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The text of ISO 12625-4:2016 has been approved by CEN as EN ISO 12625-4:2016 without any modification.

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Foreword

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This document was prepared by the European Committee Standardization (CEN) Technical Committee CEN/TC 172, *Pulp, paper and board*, in collaboration with ISO Technical Committee 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 12625-4:2005), which has been technically revised with the following changes:

- a) "stretch at break" was replaced by "stretch at maximum force";
- b) in Clause 7, a more detailed description of the preparation of the test pieces was included;
- c) the number of test pieces required has been clarified with more detailed information;
- d) in <u>Clause 8</u>, the procedure for the measurement was clarified;
- e) in <u>Clause 10</u>, additional information is to be included in the test report;
- f) this document has been editorially updated.

A list of all parts in the ISO 12625 series can be found on the ISO website.

Tissue paper and tissue products —

Part 4:

Determination of tensile strength, stretch at maximum force and tensile energy absorption

1 Scope

This document specifies a test method for the determination of the tensile strength, stretch at maximum force and tensile energy absorption of tissue paper and tissue products. It uses a tensile-testing apparatus operating with a constant rate of elongation.

It also specifies the method of calculating the tensile index and the tensile energy absorption index.

In cases where impurities and contraries have to be determined, ISO 15755^[6] applies for these detections in tissue paper and tissue products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1924-2, Paper and board — Determination of tensile properties — Part 2: Constant rate of elongation method (20 mm/min)

ISO 7500-1, Metallic materials — Calibration and verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Calibration and verification of the force-measuring system

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 12625-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1

tensile strength

maximum tensile force per unit width that a test piece will withstand before breaking in a tensile test

3.2

tensile index

tensile strength (3.1) divided by grammage

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3.3

stretch at maximum force

ratio of the elongation of a test piece, over its initial length, at the moment when the maximum tensile force is reached during a tensile test

Note 1 to entry: Stretch at maximum force is expressed as a percentage of the initial length.

3.4

tensile energy absorption

TEA

amount of energy absorbed per unit surface area of a test piece while being stretched, until the onset of break (the moment of maximum tensile force) in a tensile test

[SOURCE: ISO 12625-1:2011, 4.59]

3.5

tensile energy absorption index

tensile energy absorption (3.5) divided by grammage

[SOURCE: ISO 1924-2:2008, 3.7]

4 Principle

A test piece of tissue paper or tissue product, of given dimensions, is stretched to break at a constant rate of elongation using a tensile-testing apparatus that measures and records the tensile force as a function of the elongation of the test piece.

From the recorded data, the tensile strength, the corresponding stretch at maximum force and the tensile energy absorption can be calculated.

The test can be carried out by a vertical or a horizontal tensile strength tester. Precision data are available in Annex A.

5 Apparatus

5.1 Tensile-testing apparatus

5.1.1 General

The tensile-testing apparatus shall be in accordance with ISO 1924-2. It is capable of stretching a test piece of tissue paper or tissue product of given dimensions, at a constant rate of elongation of (50 ± 2) mm/min, and recording the tensile force as a function of elongation on a strip chart recorder or any equivalent device.

The elongation shall be recorded to an accuracy of ± 0.1 mm. The measurement of the elongation shall start at a tension of (5 \pm 1) N/m.

The force-measuring system shall measure loads with an accuracy of $\pm 1~\%$ of the reading or $\pm 0.1~$ N, whichever is greater, and shall be calibrated and verified to conform to the requirements of ISO 7500-1.

5.1.2 Means for measuring the area of the force-elongation curve

The tensile-testing apparatus shall provide a means for measuring the area between the force-elongation curve and the elongation axis to an accuracy of ± 2 % of the true value as determined by numerical integration of the raw data.

NOTE Most modern tensile testers are equipped with an electronic or electro-mechanical integrator that can be used for this purpose. The area can also be determined from a graphical output of the data on chart paper using a planimeter.

5.1.3 Clamps

The tensile-testing apparatus shall have two clamps of at least 50 mm in width. Each clamp shall be designed to grip the test piece firmly along a straight line across the full width of the test piece, without causing any damage, and shall have means for adjusting the clamping force.

The clamps should preferably grip the test piece between a cylindrical and a flat surface, with the plane of the test piece tangential to the cylindrical surface. Other types of clamps may be used if it can be ensured that the test piece does not slip or suffer any damage during the test.

During the test, the clamping lines shall be parallel to each other within an angle of 1°. The clamping lines shall be perpendicular to the direction of the applied tensile force and to the longest dimension of the test piece to the same level of accuracy.

The distance between the clamping lines (i.e. the test span length) shall be adjusted to (100 ± 1) mm, except that a test span length of (50 ± 1) mm shall be used for finished paper products of which one or both of the dimensions is insufficient to provide a test piece of the length required in 7.2.

NOTE Toilet tissue sheets having one or both dimensions of about 98 mm are an example of such a material.

5.2 Cutting device

The cutting device shall be capable of repeatedly cutting test pieces $(50,0 \pm 0,5)$ mm wide, with undamaged, straight, smooth and parallel edges.

6 Conditioning

Condition the samples according to ISO 187 and keep them in the standard atmosphere throughout the test.

Conditioning shall be done prior to the preparation of test pieces.

7 Preparation of test pieces

7.1 General

- **7.1.1** 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 that the specimens taken are representative of the sample. Each test piece shall be free from perforations and faults not normally inherent in the tissue.
- **7.1.2** For converted tissue products, testing shall be done on the product as received, regardless of the number of plies which are supplied as a product unit. Generally, a single finished product sheet is suitable for use as a test piece.
- **7.1.3** Tissue that has not been converted into a finished product shall be tested as a single ply unless otherwise agreed between the parties concerned.

7.2 Dimensions

Each test piece shall be (50.0 ± 0.5) mm in width and at least 150 mm in length. With the exception of tissue paper or tissue products with an embossed pattern over all or part of the surface, the test pieces shall be free of creases, kinks, wrinkles, folds or other thickness variations.

For finished products with dimensions or perforations which make cutting of a test piece of at least 150 mm impossible, cut the longest piece possible. In such cases, a test span length of (50 ± 1) mm

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for the tensile tester clamps (5.1.3) shall be used. This deviation from the normal procedure shall be recorded in the test report.

7.3 Number of test pieces

Cut at least 10 test pieces in the machine direction and 10 test pieces in the cross direction for the selected specimens, making a total of at least 20 test pieces from each sample of tissue paper or tissue product.

8 Procedure

- **8.1** Ensure that the tensile-testing apparatus is calibrated and check the zero position of the recording device. Check that the clamps are aligned to meet the requirements in 5.1.3.
- **8.2** The elongation rate between the clamps shall be kept constant at (50 ± 2) mm/min (5.1).
- **8.3** Place the test piece in the clamps so that any observable slack is eliminated but the test piece is not placed under any significant strain (5.1). The test piece shall not be clamped with a tension greater than 5 N/m.
- **8.4** Do not touch the test area of the test piece between the clamps with the fingers. Align the test piece as parallel as possible to the pulling direction, tightly clamp the test piece and initiate the test.
- 8.5 Test the test pieces from each sample. Record all readings, except for test pieces that break within 5 mm from the clamping line, until 10 valid results are available for each direction. If more than 20 % of the test pieces cut from a particular specimen break within 5 mm of the clamping line, reject all the readings obtained for that specimen. Inspect the apparatus for conformity with the specifications and take the appropriate remedial measures.

9 Calculation

9.1 General

Calculate and report the results separately for the machine direction and for the cross direction from Formula (1) to Formula (6).

9.2 Tensile strength

Calculate the mean maximum tensile force (\overline{F}) in newtons from all the single values (F) available representing acceptable test results. Calculate the mean tensile strength (\overline{S}) from Formula (1):

$$\overline{S} = \frac{\overline{F}}{w_x} \times 10^3 \tag{1}$$

where

- \overline{S} is the tensile strength, expressed in newtons per metre (N/m);
- \overline{F} is the maximum tensile force in newtons (N);
- w_x is the initial width, in millimetres, of the test piece (50 mm).

Report the tensile strength in newtons per metre to three significant figures.

9.3 Tensile index

Calculate the tensile index, *I*, from Formula (2):

$$I = \frac{\overline{S}}{g} \tag{2}$$

where

- I is the tensile index, expressed in newton metres per gram (Nm/g);
- \overline{S} is the tensile strength in newtons per metre (N/m);
- g is the grammage, in grams per square metre (g/m²), determined according to ISO 12625-6.

Report the tensile index in newton metres per gram to three significant figures.

9.4 Stretch at maximum force

Calculate the mean elongation at break corresponding to the stretch of the test piece at the moment when the maximum force is reached. Calculate the stretch at maximum force, *A*, from Formula (3):

$$A = \frac{\varepsilon}{l} \times 100 \tag{3}$$

where

- *A* is the mean stretch at maximum force in percentage (%);
- ε is the mean elongation at maximum force in millimetres (mm);
- l is the length of the test piece between the clamps, before elongation (see 5.1.3), in millimetres (mm).

Report the result to the first decimal place.

9.5 Tensile energy absorption

Determine the area under the force-elongation curve up to the point of maximum tensile force and calculate the tensile energy absorption, *Z*, from Formula (4):

$$Z = \frac{E}{w \times l} \times 1000 \tag{4}$$

and the mean tensile energy absorption, \overline{Z} , from Formula (5):

$$\overline{Z} = \frac{\sum Z}{n} \tag{5}$$

where

- Z is the tensile energy absorption by a single test piece during the test until maximum force, in joules per square metre (J/m^2);
- \overline{Z} is the mean tensile energy absorption in joules per square metre (J/m²);
- E is the work, in millijoules, equivalent to the area under the force/elongation curve (m]);

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- *w* is the initial width, in millimetres, of the test piece (50 mm);
- *l* is the initial test length, in millimetres (standard 100 mm);
- *n* is the number of single values of the tensile energy absorption.

Report the mean tensile energy absorption in joules per square metre (I/m^2) to the first decimal place.

9.6 Tensile energy absorption index

Calculate the tensile energy absorption index, I_Z , using Formula (6):

$$I_Z = \frac{\overline{Z}}{q} \tag{6}$$

where

- I_Z is the tensile energy absorption index, in joules per gram (J/g);
- \overline{Z} is the mean tensile energy absorption, in joules per square metre (J/m²);
- g is the grammage, in grams per square metre (g/m²), determined in accordance with ISO 12625-6.

Report the tensile energy absorption index, in joules per gram, to the first decimal place.

10 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 12625-4;
- b) the date and place of testing;
- c) the description and identification of the sample (for example, product category and dimensions under test);
- d) the number of single values used to calculate the tensile strength, elongation at maximum force and tensile energy absorption;
- e) the mean tensile strength in machine and cross direction in newtons per metre to three significant figures;
- f) the standard deviation or coefficient of variation to two significant figures;
- g) the mean stretch at maximum force, in percentage, in the machine and cross direction, rounded to the first decimal place, standard deviation or coefficient of variation;
- h) the tensile energy absorption in machine and cross direction, in joules per square metre, rounded to the first decimal;
- i) any deviation from this document and any other circumstances that may have affected the test results;
- j) the conditioning atmosphere used.

Annex A (informative)

Precision

A.1 General

The data have been obtained from CEPI-CTS, the Comparative Testing Service of the Confederation of European Paper Industries. The results are from a test round performed in 2012 by qualified laboratories. Two samples were tested according to this document. The data for tensile strength are presented in A.2. The data for stretch at maximum force are presented in A.3.

The repeatability and reproducibility results from the test are reported in Table A.1 to Table A.4.

Precision data for tensile energy absorption is not available at the moment.

The calculations were made according to ISO/TR 24498[7] and TAPPI T 1200 sp-07[8].

The repeatability standard deviation reported in <u>Table A.1</u> and <u>Table A.3</u> is the "pooled" repeatability standard deviation, which means that 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[3].

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 1 The repeatability standard deviation and the within-laboratory standard deviation are identical. However, the reproducibility standard deviation is not the same as between-laboratories standard deviation. The reproducibility standard deviation includes both the between-laboratories standard deviation and the standard deviation within a laboratory, viz.:

$$s_{repeatability}^2 = s_{within \, lab}^2$$

but

$$s_{reproducibility}^2 = s_{within \, lab}^2 + s_{between \, lab}^2$$

NOTE 2 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.

A.2 Tensile strength

Table A.1 — Repeatability results of an interlaboratory test by qualified laboratories (tensile strength)

Sample	Number of laboratories	Mean tensile strength	Repeatability standard deviation	Coefficient of variation	Repeatability limit
			s_r	$C_{V,r}$	r
		N/m	N/m	%	N/m
A	11	121	9,0	7,4	25
В	10	643	27	4,2	74

Table A.2 — Reproducibility results of an interlaboratory test by qualified laboratories (tensile strength)

Sample	Number of laboratories	Mean tensile strength	Reproducibility standard deviation	Coefficient of variation	Reproducibility limit
			s_R	$C_{V,R}$	R
		N/m	N/m	%	N/m
A	11	121	11	9,1	30
В	10	643	30	4,7	84

A.3 Stretch at maximum force

Table A.3 — Repeatability results of an interlaboratory test by qualified laboratories (stretch at maximum force)

Sample	Number of laboratories	Mean stretch at maximum force	Repeatability standard deviation	Coefficient of variation	Repeatability limit
			s_r	$C_{V,r}$	r
		%	%	%	%
A	10	16,8	1,7	9,9	4,6
В	10	15,5	0,76	4,9	2,1

Table A.4 — Reproducibility results of an interlaboratory test by qualified laboratories (stretch at maximum force)

Sample	Number of laboratories	Mean stretch at maximum force	Reproducibility standard deviation	Coefficient of variation	Reproducibility limit
			s_R	$C_{V,R}$	R
		%	%	%	%
A	10	16,8	1,9	11	5,1
В	10	15,5	1,3	8,1	3,5

Bibliography

- [1] ISO 287, Paper and board Determination of moisture content of a lot Oven-drying method
- [2] ISO 638, Paper, board and pulps Determination of dry matter content Oven-drying method
- [3] ISO 5725-1, Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions
- [4] ISO 12625-1, Tissue paper and tissue products Part 1: General guidance on terms
- [5] ISO 12625-6, Tissue paper and tissue products Part 6: Determination of grammage
- [6] ISO 15755, Paper and board Estimation of contraries
- [7] ISO/TR 24498, Paper, board and pulps Estimation of uncertainty for test methods
- [8] TAPPI T 1200 sp-07, Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility





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