INTERNATIONAL STANDARD

ISO 12625-3

Second edition
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Tissue paper and tissue products —

Part 3: Determination of thickness, bulking thickness and apparent bulk density and bulk

Papier tissue et produits tissue —

Partie 3: Détermination de l'épaisseur, de l'épaisseur moyenne d'une feuille en liasse et de la masse volumique moyenne et de la main

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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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 12625-3 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 cooperation between ISO and CEN (Vienna Agreement).

This second. edition cancels and replaces the first edition (ISO 12625-3:2005), which has been technically revised.

With regard to ISO 12625-3:2005, the following minor changes have been made:

a) title changed; "bulk" added;

b) the accuracy data in Annex A were adjusted to the existing equipment capabilities and calibration procedures;,

c) preparation of test pieces more precisely described, 7.1 and 7.4 reformulated;

d) editorial updating.

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) illuminant;*

— *Part 16 Determination of optical properties — Opacity (paper backing) — Diffuse reflectance method*

Introduction

Thickness is an important property of tissue paper and tissue products.

In the tissue industry, thickness-related parameters, such as the roll diameter of rolled products (kitchen towel) or the stack height of folded products (paper towels) are often measured. However, the fact that not only end-use tissue products, but also the base tissue paper from which these products are made, is the subject of trade between companies and countries, means that there is a genuine need for a consistent measure of thickness that can be applied to tissue products at any stage of their manufacture.

The thickness of tissue paper and tissue products is known to be dependent on the pressure applied to the material at the time of measurement. Several different loading pressures, pressure-foot diameters and loading speeds have been used within the tissue industry. This part of ISO 12625 has been prepared by harmonizing those standards applicable to tissue and tissue products currently in use. It specifies a single loading pressure, foot diameter and loading speed to be used for all thickness measurements of tissue and tissue products.

It is expressly stated that the detection of impurities and contraries in tissue paper and tissue products should be applied according to ISO 15755.

For the determination of moisture content in tissue paper and tissue products, ISO 287 or ISO 638 should be applied.

Tissue paper and tissue products —

Part 3:
Determination of thickness, bulking thickness and apparent bulk density and bulk

1 Scope

This part of ISO 12625 specifies a test method for the determination of thickness and bulking thickness and the calculation of apparent bulk density and bulk of tissue papers and tissue products under a pressure of 2,0 kPa.

NOTE This part of ISO 12625 has been developed to provide a consistent test method for the determination of thickness and density of tissue paper and tissue products. Corresponding test methods for paper and board in general are covered in ISO 534.

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 12625-1, *Tissue paper and tissue products — Part 1: General guidance on terms*

ISO 12625-6, *Tissue paper and tissue products — Part 6: Determination of grammage*

3 Terms and definitions

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

3.1
single-ply thickness
distance between the two principal surfaces of a single ply of tissue paper measured under the applied static load specified in this part of ISO 12625

Note 1 to entry: A ‘ply’ of tissue is an unlaminated tissue, like that made directly from a tissue machine.

3.2
single sheet thickness
distance between the two principal surfaces of a single sheet of tissue product measured under the applied static load specified in this part of ISO 12625

Note 1 to entry: A ‘sheet’ of tissue is a laminated or unlaminated tissue like that present in the finished tissue product.

3.3
bulking thickness
thickness of a single sheet of tissue paper or tissue product, calculated from the thickness of several superimposed sheets, measured under the applied static load specified in this part of ISO 12625

3.4
apparent bulk density
mass per unit volume of tissue paper or tissue product, calculated from its grammage and bulking thickness

Note 1 to entry: The apparent bulk density is expressed in grams per cubic centimetre.

3.5
bulk
apparent specific bulk volume
inverse of density

Note 1 to entry: The bulk is expressed in cubic centimetres per gram.

3.6
grammage
mass of a unit area of tissue paper or tissue product as determined by the procedure in ISO 12625-6

Note 1 to entry: to entry The grammage is expressed in grams per square metre (g/m^2).

4 Principle

Measurement of the thickness of a test piece of tissue paper sampled during the manufacturing process or of a tissue product supplied as a finished article. The measurement being made as the distance between a fixed reference plate on which the sample rests and a parallel pressure-foot that exerts a specified load on the area under test.

5 Apparatus

5.1 Precision dead-weight micrometer

This has two parallel, horizontal faces, flat to within 0,003 mm, between which the test piece is placed. The lower face shall be fixed and the upper face (pressure-foot) moveable in a direction perpendicular to the plane of the fixed face.

The upper, circular, pressure-foot shall have a diameter of (35,7 ± 0,1) mm giving a nominal area of 10,0 cm^2 and shall be parallel to the lower face within limits defined in Annex B.3.

The lower face shall be constructed to support the test piece such that the test piece lies flat while under test. In practice, the lower face should have minimum dimensions 20 % larger than the diameter of the pressure-foot. The pressure between the two faces shall be (2,0 ± 0,1) kPa (see Annex C).

The measuring speed shall be (2,0 ± 0,2) mm/s.

The instrument read-out/scale shall be graduated in increments of 0,001 mm.

The opening between the pressure-foot and the lower face is set by agreement between the instrument supplier and the customer. For most thickness measurements, instruments with an opening of 10 mm or 12 mm will normally be suitable. When only single sheet or single-ply measurement is required, an opening of 2 mm to 3 mm is sufficient.

5.2 Gauge blocks

These are used for calibrating the micrometer, corresponding to approximately 10 %, 30 %, 50 %, 70 % and 90 % of the full-scale reading of the micrometer. The thickness of each gauge at 23 °C shall be known to an accuracy of 0,001 mm or better.

5.3 Balance and attachments

A suitable balance and attachments or calibrated load cell capable of measuring up to 300 g with an accuracy of 0,01 g to be used for calibration of the pressure-foot load.

6 Conditioning

Condition the samples according to ISO 187. The sample shall remain in the standard atmosphere throughout the testing.

7 Preparation of test pieces

7.1 General

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.

Each test piece shall be free from perforations and faults not normally inherent in the tissue.

Test piece dimensions are not critical, but they shall allow measurements to be performed with a minimum spacing of 50 mm between each measurement area and 10 mm to the edges of the test piece.

Large specimens may be cut to a reasonable size. When cutting, the test piece shall not be subjected to pressure that could alter the thickness.

7.2 Single-ply thickness

Prepare 10 test pieces sampled either directly from the tissue machine, or, in case of single-ply finished product, during or after the converting process. Single-ply thickness shall not be performed on separated plies of a multi-ply product.

Do not attempt to separate plies that are bonded with adhesive or significant pressure.

Plies from different positions in a multi-ply product shall not be assumed to be the same.

7.3 Single-ply sheet thickness

Prepare 10 test pieces of single or multi-ply product sampled during or after the converting process.

7.4 Bulking thickness

Multi-ply sheets shall not be separated into individual plies. Stacks shall normally contain 12 plies. In case of products for which the number of plies do not divide 12, a number of plies as close as possible of 12 has to be considered. Where the stack height using 12 plies is too large for the maximum opening of the instrument in use, a lower number of plies may be used, but this should not be less than eight. In all cases, report the number of sheets and the number of plies per sheet used.

Prepare a sufficient number of stacks to be able to perform 10 separate measurements. All stacks shall comprise the same number of superimposed sheets, all oriented in the same way.

8 Procedure

Place the micrometer on a horizontal vibration-free surface within the conditioned atmosphere defined in ISO 187 and allow it to 'warm-up' according to the manufacturer's instructions.

The working faces of the micrometer shall be clean.

Check the zero setting of the micrometer and adjust it if necessary.

Verify the calibration of the micrometer as defined in B.1.

Raise the pressure-foot and insert the test pieces between the lower face and the pressure-foot. Allow the pressure-foot to move down onto the test pieces at the controlled speed (5.1). After 5 s, record the thickness to the nearest 0,001 mm.

Repeat the measurement on the remaining test pieces until at least 10 measurements have been recorded. Between successive readings, make sure that the working faces are free from dust.

If the apparent bulk density or the bulk of the sample is to be calculated, determine the grammage of the sample by the test method described in ISO 12625-6.

9 Calculation

9.1 Single-ply thickness

Calculate the mean and standard deviation of the 10 (or more) readings and report the single ply thickness t_p to the nearest 0,01 mm and standard deviation to three significant figures.

9.2 Single sheet thickness

Calculate the mean and standard deviation of the 10 (or more) readings and report the single sheet thickness t_s to the nearest 0,01 mm and standard deviation to three significant figures.

9.3 Bulking thickness

Calculate the mean and standard deviation of the 10 (or more) readings. Divide both by the number of sheets (not plies) in each pack and report the result as the bulking thickness t_b to the nearest 0,01 mm and standard deviation to three significant figures.

9.4 Apparent bulk density

Calculate the apparent bulk density, x, in grams per cubic centimetre according to Equation (1):

$$x = \frac{g}{t_b \times 1000} \tag{1}$$

where

g is the grammage, in grams per square metre (g/m^2);

t_b is the bulking thickness, in millimetres (mm).

Report the apparent bulk density to two significant figures.

9.5 Bulk (apparent specific bulk volume)

Calculate the bulk, y, in cubic centimetre per gram according to Equation (2):

$$y = \frac{t_b \times 1000}{g} \tag{2}$$

where

t_b is the bulking thickness, in millimetres (mm);

g is the grammage, in grams per square metre (g/m^2).

Report the bulk to three significant figures.

10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 12625;

b) the date and place of testing;

c) all details necessary to identify the material, including a statement of the number of plies as received and the location of individual plies if single-ply thickness testing has been requested for a multi-ply product; if bulking thickness testing has been requested from a multi-ply product, identify the sample (sheet) and state the number of individual plies in each sheet and the number of sheets in each pack;

d) conditioning atmosphere used according to ISO 187;

e) the number of measurements made to obtain each reported mean value;

f) any departure from this part of ISO 12625 or any other circumstances that may have affected the results.

Annex A
(informative)

Precision

In 2011, 11 laboratories from nine European countries, tested two samples. In total, 10 test pieces for single ply thickness and five test pieces for bulking thickness were tested.

The data has been obtained from CEPI-CTS, the Comparative Testing Service of the Confederation of European Paper Industries. The data presented in Tables A.1, A.2, A.3 and A.4.

The calculations were made according to ISO/TR 24498[5] and TAPPI T 1200 sp-07.[6]

The repeatability standard deviation reported in Table A.1 and A.3 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.[7]

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 the between-laboratory standard deviation. The reproducibility standard deviation includes both the between-laboratories standard deviation and the standard deviation within a laboratory, viz.:

$$s^2_{\text{repeatability}} = s^2_{\text{within lab}} \quad \text{but} \quad s^2_{\text{reproducibility}} = s^2_{\text{within lab}} + s^2_{\text{between lab}}$$

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.

Table A.1 — Single sheet thickness — Estimation of the repeatability

Sample	Number of laboratories	Mean single sheet thickness mm	Repeatability standard deviation s_r mm	Coefficient of variation $C_{V,r}$ %	Repeatability limit r mm
Sample 1	10[a]	0,11	0,002 30	2,13	0,006 38
Sample 2	11	0,30	0,005 40	1,79	0,015 0

[a] One outlier.

Table A.2 — Single sheet thickness — Estimation of reproducibility

Sample	Number of laboratories	Mean single sheet thickness mm	Reproducibility standard deviation s_R mm	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mm
Sample 1	10[a]	0,11	0,004 70	4,35	0,013 0
Sample 2	11	0,30	0,014 4	4,78	0,040 0
[a] One outlier.					

Table A.3 — Bulking thickness — Estimation of repeatability

Sample	Number of laboratories	Mean bulking thickness mm	Repeatability standard deviation s_r mm	Coefficient of variation $C_{V,r}$ %	Repeatability limit r mm
Sample 1	10	0,09	0,001 42	1,55	0,003 94
Sample 2	8[a]	0,28	0,001 70	0,612	0,004 71
[a] Two outliers.					

Table A.4 — Bulking thickness — Estimation of reproducibility

Sample	Number of laboratories	Mean bulking thickness mm	Reproducibility standard deviation s_R mm	Coefficient of variation $C_{V,R}$ %	Reproducibility limit R mm
Sample 1	10	0,09	0,002 41	2,63	0,006 69
Sample 2	8[a]	0,28	0,003 89	1,40	0,010 8
[a] Two outliers.					

Annex B
(normative)

Measurement conditions

B.1 Calibration

Calibration shall be checked as frequently as is necessary to ensure continued accuracy. For instruments in frequent use, a daily check is advised for accuracy using a single thickness gauge. Foot parallelism and foot pressure shall be checked in accordance to the supplier's calibration instructions, based on the frequency of usage. Formal confirmation of accuracy, foot parallelism, evenness of the faces and foot pressure is described in B.2, B.3 and Annex C, respectively.

The gauge block used should reflect the thickness of the products normally being tested.

Check the micrometer at the temperature as in which the tests are performed, according to ISO 187.

B.2 Accuracy

Clean the pressure faces by drawing a clean sheet of A4 copy paper (or equivalent) through the loaded faces. Set the instrument at zero. Insert a gauge block between the anvil and pressure-foot, lower the pressure foot and perform the measurement. Use only one gauge block at a time. Check the thickness readings at approximately 10 %, 30 %, 50 %, 70 % and 90 % of full-scale reading.

The maximum deviation from the true thickness shall be less than 0,003 mm or ± 0,5 % of the reading, whichever is the greater.

Repeated zero settings or repeated checks with gauges shall not differ by more than 0,003 mm or ± 0,5 % of the reading, whichever is the greater.

B.3 Parallelism of the faces

Measure the parallelism of the faces using the following procedure:

a) Take one of the gauge blocks (5.2), open the gap between the pressure faces and insert the gauge block between the pressure face about 2 mm from the edge at one side of the pressure-foot. Allow the pressure faces to close on the gauge block and note the micrometer reading.

b) Open the gap between the pressure faces and insert the same gauge between the pressure faces about 2 mm from the edge on the opposite of the pressure-foot. Allow the pressure faces to close on the gauge and again note the micrometer reading.

c) Calculate the difference d_1 between the readings noted in a) and b).

d) Repeat the procedure described in a) and b) with the same thickness gauge block at positions between the pressure foot about 2 mm from the edge of the pressure faces, and on a diameter perpendicular to that passing through the points referred to in a) and b).

e) Calculate the difference d_2 between the readings noted in d).

f) Repeat the procedure described above using, in turn, each of the remaining gauges.

g) For each thickness gauge block at which micrometer readings are taken, calculate the error of parallelism using Equation (B.1):

$$x = 0{,}5\sqrt{d_1^2 + d_2^2} \tag{B.1}$$

where

x is the parallelism deviation;

d_1 is the difference between the readings noted in a) and b);

d_2 is the difference between the readings noted in d).

The parallelism is satisfactory if, at each particular thickness, the parallelism error x is no more than 0,006 mm.

B.4 Evenness of the faces

Check that both pressure faces are even by the following procedure:

— Carefully clean the faces of the micrometer (see B.2).

— Raise the pressure-foot slightly and view the gap against a bright light.

— Observe the gap from two perpendicular directions in plane.

The gap shall appear perfectly uniform from both directions.

Annex C
(informative)

Foot pressure

Any suitable method that determines to the specified accuracy, the force required to just prevent the pressure foot from descending, can be used. The force required should be (2,0 ± 0,1) N.

The following are examples using a balance or calibrated load cell.

a) Attach a fine wire to the pressure-foot where it projects through the top of the micrometer and measure the force with a calibrated spring balance or beam balance with one pan removed. Allow for the weight of the removed pan and added wire.

b) Support the pressure-foot with a stirrup made of a metal plate with a hole larger than the pressure-foot and bottomed with a metal disk having a thickness about equal to the average thickness of the test pieces. Suspend the stirrup from one arm of a beam balance with the pan removed and measure the force. Allow for the difference in weight between the removed pan and the stirrup.

c) As a) or b) but using a calibrated load cell suspended from a rigid mounting.

Bibliography

[1] ISO 287, *Paper and board — Determination of moisture content of a lot — Oven-drying method*

[2] ISO 534, *Paper and board - Determination of thickness, density and specific volume*

[3] ISO 638, *Paper, board and pulps — Determination of dry matter content — Oven-drying method*

[4] ISO 15755, *Paper and board — Estimation of contraries*

[5] ISO/TR 24498, *Paper, board and pulps — Estimation of uncertainty for test methods*

[6] TAPPI Test method T 1200 sp-07, *Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility*

[7] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

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