INTERNATIONAL STANDARD

ISO 534

Fourth edition 2011-11-15

Paper and board — Determination of thickness, density and specific volume

Papier et carton — Détermination de l'épaisseur, de la masse volumique et du volume spécifique





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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 534 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 2, *Test methods and quality specifications for paper and board.*

This fourth edition cancels and replaces the third edition (ISO 534:2005), which has been technically revised.

The essential changes in this International Standard are the reduction of the field of application, because now it is clearly stated that the measurement of bulking thickness is not intended for board, and the deletion of the alternative pressure [(50 \pm 5) kPa] for the thickness measurement. In some countries, particularly in North America, the 50 kPa pressure is still widely used and different results will be obtained depending on which pressure is used. At the same time, new precision data have been inserted.

Paper and board — Determination of thickness, density and specific volume

1 Scope

This International Standard specifies two methods for measuring the thickness of paper and board:

- a) the measurement of a single sheet of paper or board as a single sheet thickness;
- b) the measurement of a pack of sheets of paper as a bulking thickness.

This International Standard also specifies calculation methods

- for the apparent sheet density and for the apparent bulk density, and
- for the apparent specific sheet volume and for the apparent specific bulk volume

from the thickness determinations.

This International Standard is not applicable to corrugated fibreboard. In addition, the measurement of bulking thickness, method b) above, is not suitable for board¹⁾.

NOTE The two methods generally lead to different results. These methods are not applicable to tissue paper and tissue products. For tissue paper and tissue products, ISO 12625-3 should be used.

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 536, Paper and board — Determination of grammage

3 Terms and definitions

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

3.1

single sheet thickness

distance between one surface of a paper or board and the other, measured under an applied static load, using this test method

3.2

bulking thickness

thickness of a single sheet of paper, calculated from the thickness of several superimposed sheets in a pack, and measured under an applied static load, using this test method

¹⁾ For the definition of "board", see ISO 4046-3:2002, definition 3.16.

ISO 534:2011(E)

3.3

apparent sheet density

mass per unit volume, expressed in grams per cubic centimetre, and calculated from the single sheet thickness (3.1)

NOTE This term is normally applicable to paper or board.

3.4

apparent bulk density

mass per unit volume, expressed in grams per cubic centimetre, and calculated from the bulking thickness (3.2)

NOTE This term is normally applicable to paper.

3.5

apparent specific sheet volume

volume per unit mass, expressed in cubic centimetres per gram, and calculated from the single sheet thickness (3.1)

NOTE This term is normally applicable to paper or board.

3.6

apparent specific bulk volume

volume per unit mass, expressed in cubic centimetres per gram, and calculated from the bulking thickness (3.2)

NOTE This term is normally applicable to paper.

Principle 4

- Measurement of the single sheet thickness (3.1) or of the bulking thickness (3.2), according to the test requirements, by means of a precision micrometer.
- Calculation of the apparent sheet density (3.3) or apparent bulk density (3.4) of the paper or board, 4.2 from a knowledge of its grammage and thickness.
- 4.3 Calculation of the apparent specific sheet volume (3.5) or apparent specific bulk volume (3.6) of the paper or board, from a knowledge of its grammage and thickness.

Apparatus 5

Dead-weight micrometer, incorporating two plane, parallel, circular pressure faces, between which the paper or board is placed for measurement.

The pressure exerted between the pressure faces during the thickness measurement shall be (100 ± 10) kPa.

The two pressure faces shall form an integral part of the micrometer, such that one face is fixed (the anvil) and the other is movable in a direction perpendicular to the plane of the fixed face.

One face shall be $(16,0 \pm 0,5)$ mm in diameter and the second face shall be of such a size that it is in contact with the whole area of the other face when the micrometer reads zero. Thus, a circular region of a test piece, nominally 200 mm² in area, is subjected during the thickness measurement to the pressure exerted by the faces.

The performance requirements of the micrometer shall be such that, when calibrated according to the method given in Annex A, the micrometer complies with the required pressure of (100 ± 10) kPa and the performance requirements shown in Table 1 (see also 9.1).

Table 1 — Micrometer-performance requirements

Micrometer characteristics	Maximum permitted value a			
Indication error	$\pm 2,5~\mu m$ or $\pm 0,5~\%$ of the reading			
Error of parallelism between pressure faces	5 μm or 1 %			
Repeatability of measurement (as standard deviation)	1,2 μm or 0,5 %			
^a The maximum permitted value of a micrometer characteristic is the greater of the two values.				

5.2 Thickness gauges, corresponding to approximately 10 %, 30 %, 50 %, 70 % and 90 % of the full-scale reading of the micrometer. The thickness of each gauge shall be known to an accuracy of $0.3 \mu m$.

6 Sampling

If the tests are made to evaluate a lot, select the sample in accordance with ISO 186. If the tests are made on another type of sample, make sure that the test pieces taken are representative of the sample received.

7 Conditioning

Condition the sample in accordance with ISO 187.

8 Preparation of test pieces

8.1 General

Prepare the test pieces in the same standard atmospheric conditions as those used to condition the sample. Avoid areas with folds, creases, cracks or other defects which could influence the results.

8.2 Single sheet thickness

Cut not more than two test pieces from each specimen taken at random from the sample available, with minimum dimensions 60 mm \times 60 mm. Ensure that the test piece dimensions are not so large that the micrometer reading is affected by the test piece mass that overhangs the lower pressure face while a measurement is being made. When measuring board, do not use test pieces with dimensions exceeding 100 mm \times 100 mm. These test piece dimensions are usually satisfactory for making measurements on paper.

Prepare at least 20 test pieces.

8.3 Bulking thickness

Cut sheets at random from the sample available, preferably having dimensions 200 mm \times 250 mm, the 200 mm dimension being in the machine direction (see Figure 1). If this is not possible, prepare smaller sheets of at least 150 mm \times 150 mm.

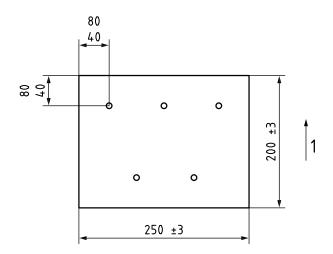
Make up a pack of ten sheets to form the test piece; each sheet shall be oriented in the same direction and with the same side up and the same dimensions. Each sheet shall be independent of the rest. For example, it is not permitted to fold one sheet and insert it folded into the test piece to form two or more sheets. The number of sheets in a test piece shall normally be ten.

Prepare at least four test pieces, and make sure that the number of sheets and their sizes in each test piece is the same.

In special circumstances, such as for thick or very thin sheets or when agreed between the parties concerned, a smaller or larger number of sheets, or a smaller or larger sheet, may be used.

The number of sheets used and their size shall be reported.

Dimensions in millimetres



Key

machine direction (MD)

Figure 1 — Positions of measurements on a test piece for bulking thickness

Procedure

9.1 General

Prior to the use of the micrometer (5.1) or when calibrating it, ensure that the anvil, pressure foot and thickness gauges (5.2) are clean.

Particularly in the case of the anvil and pressure foot, small pieces of fibre can collect on these surfaces, causing erroneous high values.

When thickness gauges (5.2) are used in calibration, they should be gently wiped with alcohol using a nonlinting absorbent material.

NOTE 2 The above requirements do not apply to 9.3.3.

Verification and calibration of micrometer

At appropriate time intervals, calibrate the micrometer in the conditioning atmosphere used for the measurement of thickness, and verify its performance using the method given in Annex A.

For micrometers used frequently, the indication error and repeatability of measurement should be determined daily. The pressure exerted by the pressure faces and their error of parallelism should be determined at monthly intervals.

9.3 **Determinations**

9.3.1 Determination of single sheet thickness

Carry out the test in the standard atmospheric conditions in which the samples were conditioned.

Place the micrometer on a horizontal vibration-free surface and place the test piece between the open pressure faces of the micrometer at a position at least 20 mm from any edge of the test piece. Permit the test piece to be held by the pressure face, by very carefully allowing the movable pressure face to move steadily and slowly, at a velocity less than 3 mm/s, towards the anvil so that any punching effect is avoided.

Record the micrometer reading at the end of a dwell time of 1 s to 2 s. Avoid imposing any manual stress on the test piece or micrometer while a reading is being made. Make only one measurement on the test piece.

Repeat the above procedure for the remaining test pieces.

9.3.2 Determination of bulking thickness

Carry out the test in the standard atmospheric conditions in which the samples were conditioned.

Place the micrometer on a horizontal vibration-free surface and place the test piece between the open pressure faces of the micrometer in one of the positions shown in Figure 1. Permit the test piece to be held by the pressure face, by very carefully allowing the movable pressure face to move steadily and slowly, at a velocity less than 3 mm/s, towards the anvil so that any punching effect is avoided.

Record the micrometer reading at the end of a dwell time of 1 s to 2 s. Avoid imposing any manual stress on the test piece or micrometer while a reading is being made.

Repeat the measurement for each of the other four positions shown in Figure 1, situated between 40 mm and 80 mm from the edges of the test piece and distributed along the two edges which are in the cross-direction of the paper.

Repeat the above procedure for the remaining test pieces.

9.3.3 Determination of grammage

If the apparent density, or the apparent specific volume, of the paper or board is to be calculated, determine the grammage of representative material taken from the sample, by the method specified in ISO 536.

10 Calculation and expression of results

10.1 Single sheet thickness

- **10.1.1** Calculate the mean value of not less than 20 readings made in accordance with 9.3.1, and express the result, in micrometres, to three significant figures.
- **10.1.2** Record the maximum and minimum values of the single sheet thickness.
- **10.1.3** Calculate the standard deviation of the single sheet thickness.
- 10.1.4 Calculate the confidence interval for the mean at the 95 % confidence level.

10.2 Bulking thickness

- **10.2.1** Calculate the mean value of not less than 20 readings made in accordance with 9.3.2, corresponding to not less than five measurements for each of the four test pieces. Divide it by the number of sheets comprising each test piece to obtain the bulking thickness of a single sheet of paper. Express the result, in micrometres, to three significant figures.
- **10.2.2** Record the maximum and minimum values of the bulking thickness.
- **10.2.3** Calculate the standard deviation of the bulking thickness.
- **10.2.4** Calculate the confidence interval for the mean at the 95 % confidence level.

10.3 Apparent density

10.3.1 Apparent sheet density

Calculate the mean apparent sheet density, d_s , in grams per cubic centimetre, according to Equation (1):

$$d_{s} = \frac{g}{\delta_{s}} \tag{1}$$

where

- g is the grammage, in grams per square metre, of the paper;
- δ_{s} is the mean single sheet thickness, in micrometres, of the paper.

Report the result to two decimal places.

10.3.2 Apparent bulk density

Calculate the apparent bulk density, d_b , in grams per cubic centimetre, according to Equation (2):

$$d_{\mathsf{b}} = \frac{g}{\delta_{\mathsf{b}}} \tag{2}$$

where

- g is the grammage of the paper, in grams per square metre;
- δ_b is the mean bulking thickness, in micrometres, of the paper.

Report the result to two decimal places.

NOTE The apparent bulk density of a paper, calculated from the bulking thickness, is not necessarily the same as the apparent density of the same paper, calculated from the single sheet thickness, determined using the same apparatus.

10.4 Apparent specific volume

10.4.1 Apparent specific sheet volume

Calculate the apparent specific sheet volume, v_s , in cubic centimetres per gram, according to Equation (3):

$$v_{s} = \frac{\delta_{s}}{\sigma} \tag{3}$$

where

- $\delta_{\rm s}$ is the mean single sheet thickness, in micrometres, of the paper;
- g is the grammage, in grams per square metre, of the paper.

Report the result to two decimal places.

10.4.2 Apparent specific bulk volume

Calculate the apparent specific bulk volume, v_b , in cubic centimetres per gram, according to Equation (4):

$$v_{\mathsf{b}} = \frac{\delta_{\mathsf{b}}}{g} \tag{4}$$

where

- δ_b is the mean bulking thickness, in micrometres;
- g is the grammage, in grams per square metre, of the paper.

Report the result to two decimal places.

NOTE The specific bulk volume of a paper, calculated from the bulking thickness, is not necessarily the same as the specific volume of the same paper, calculated from the single sheet thickness, determined using the same apparatus.

11 Test report

The test report shall include the following information:

- a) a reference to this International Standard, i.e. ISO 534:2011;
- b) precise identification of the sample;
- c) the conditioning atmosphere used;
- d) if measured, the mean bulking thickness, in micrometres to three significant figures, the maximum and minimum values, the standard deviation and the confidence interval for the mean at the 95 % confidence level;
- e) if measured, the mean single sheet thickness, in micrometres to three significant figures, the maximum and minimum values, the standard deviation and the confidence interval for the mean at the 95 % confidence level;
- f) if required, the apparent sheet density or the apparent bulk density, in grams per cubic centimetre, to two decimal places;
- g) if required, the apparent specific sheet volume or the apparent specific bulk volume, in cubic centimetres per gram, to two decimal places;
- h) the number of test pieces used for the test;
- i) in the case of bulking thickness, the number of sheets and their sizes used for each test piece;
- j) the number of readings taken;
- k) if determined, the grammage of the sample, according to 9.3.3;
- I) any departure from this International Standard, together with any circumstances that may have influenced the results.

Annex A

(normative)

Verification of micrometer performance and calibration

A.1 General

Verify the performance of the micrometer in the conditioned atmosphere in which thickness measurements are to be made, using the following tests in the order given.

If the micrometer performance is not within the tolerance appropriate to a particular measurement (see 5.1), make the necessary correction and recommence the series of tests.

A.2 Pressure exerted between pressure faces

Use any suitable means of verifying the accuracy and uniformity of the pressure exerted between the pressure faces.

A.3 Indication error and repeatability of measurement

- With the pressure faces in contact with one another, set the micrometer reading to zero. Do not reset the zero reading during the following procedure.
- Open the gap between the pressure faces, allow it to close again (see 9.2) so that the pressure faces make contact with one another, and note the micrometer reading. Repeat this procedure at least five times.
- Take one of the thickness gauges specified in 5.2, open the gap between the pressure faces, interpose the gauge, allow the faces to close upon the gauge (see 9.2) and note the micrometer reading. Avoid direct handling of the thickness gauges when cleaning or positioning them. Repeat this procedure at least five times.
- A.3.4 Repeat the procedure described in A.3.3, using, in turn, each of the remaining thickness gauges.
- NOTE The thickness gauges are used singly, not in combination.
- Repeat the procedure described in A.3.2. A.3.5
- A.3.6 For each gauge thickness at which micrometer readings are taken, calculate:
- the repeatability of measurement, that is, the standard deviation of the five (or more) readings taken, and
- the indication error, i.e. the difference between the mean of the five (or more) readings taken and the b) gauge thickness.

A.4 Parallelism of pressure faces

- **A.4.1** Take one of the thickness gauges specified in 5.2, open the gap between the pressure faces and interpose the gauge, as near as possible to one edge of the faces. Allow the pressure faces to close upon the gauge (see 9.2) and note the micrometer reading.
- **A.4.1.1** Open the gap between the pressure faces and interpose the thickness gauge as near as possible to the edge of the faces diametrically opposite the edge used in A.4.1. Allow the pressure faces to close upon the gauge (see 9.2) and again note the micrometer reading.
- **A.4.2** Repeat the procedure described in A.4.1, using positions as near as possible to the edge of the pressure faces and on a diameter perpendicular to that passing through the points referred to in A.4.1.
- **A.4.3** Repeat the procedure described in A.4.1, A.4.1.1 and A.4.2, using, in turn, each of the remaining thickness gauges.

NOTE The thickness gauges are used singly, not in combination.

A.4.4 For each gauge thickness at which micrometer readings are taken, calculate the error of parallelism, E, according to Equation (A.1):

$$E = 0.5\sqrt{d_1^2 + d_2^2} \tag{A.1}$$

where

- d₁ is the difference between the readings corresponding to opposite ends of a diameter of the pressure faces;
- d_2 is the difference between the readings corresponding to opposite ends of a diameter of the pressure faces perpendicular to that used to obtain d_1 .

Annex B

(informative)

Precision

B.1 General

When the second edition of this International Standard was developed, the precision of this test method was considered and studies of the repeatability and reproducibility of the two methods of measuring thickness were carried out. These data from ISO 534:1988 are presented in B.2.

In this fourth edition, new precision data from 2010 for single sheet thickness have been added. The data for single sheet thickness have been obtained from CEPI-CTS, the Comparative Testing Service of the Confederation of European Paper Industries. The data from CEPI-CTS are presented in B.3.

B.2 Precision data from previous edition

B.2.1 Single sheet thickness

Repeatability B.2.1.1

Under routine laboratory conditions, repeatability varies from 0,8 µm to 2,2 µm with a mean value of 1,3 µm, or from 1,1 % to 2,6 % with a mean value of 2,0 %.

The difference between two single test values found on identical test material, by one operator using the same micrometer within a short time interval, will exceed the repeatability on average not more than once in 20 instances, in the normal and correct operation of the method.

The values quoted above compare with a value of about 1,5 µm, calculated according to ISO 5725-2 [2] from the specified micrometer-performance requirements. The difference arises from the inherent variability of paper.

B.2.1.2 Reproducibility

Under routine laboratory conditions, reproducibility varies from 4,2 µm to 8,6 µm with a mean value of 5,9 µm, or from 4,7 % to 10,9 % with a mean value of 7,9 %.

The difference between two single and independent results found by two operators, working in different laboratories on identical test material, will exceed the reproducibility on average not more than once in 20 instances, in the normal and correct operation of the method.

The values quoted above compare with a value of about 3,2 µm, calculated according to ISO 5725-2 [2] from the specified micrometer-performance requirements. The difference arises not only from the inherent variability of paper but also from environmental and operator differences.

B.2.2 Bulking thickness

B.2.2.1 Repeatability

Under routine laboratory conditions, repeatability varies from 0,1 µm to 0,5 µm with a mean value of 0,31 µm, or from 0,1 % to 0,9 % with a mean value of 0,5 %.

The difference between two single test values found on identical test material, by one operator using the same micrometer within a short time interval, will exceed the repeatability on average not more than once in 20 instances, in the normal and correct operation of the method.

The values quoted above compare with a value of about 0,3 %, calculated according to ISO 5725-2 [2] from the specified micrometer-performance requirements. The difference arises from the inherent variability of paper.

B.2.2.2 Reproducibility

Under routine laboratory conditions, reproducibility varies from 1,7 μ m to 3,4 μ m with a mean value of 2,7 μ m, or from 2,4 % to 6,2 % with a mean value of 3,7 %.

The difference between two single and independent results found by two operators, working in different laboratories on identical test material, will exceed the reproducibility on average not more than once in 20 instances, in the normal and correct operation of the method.

The values quoted above compare with a value of about 0,65 % calculated according to ISO 5725-2 [2] from the specified micrometer-performance requirements. The difference arises not only from the inherent variability of paper but also from environmental and operator differences.

B.2.3 Precision of density and specific volume values

The precision of the density and specific volume values can be assessed from the precision of the thickness and grammage measurements, but the true precision cannot be determined since the calculation is based on mean values and not on data for the individual test pieces.

B.3 Data from CEPI-CTS

In 2010, 18 laboratories, from 14 European countries, tested four samples according to ISO 534. The data for single sheet thickness have been obtained from CEPI-CTS, the Comparative Testing Service of the Confederation of European Paper Industries. The data are presented in Tables B.1 and B.2.

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

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

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

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

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

$$s_{\text{repeatability}}^2 = s_{\text{within lab}}^2$$
 but $s_{\text{reproducibility}}^2 = s_{\text{within lab}}^2 + s_{\text{between lab}}^2$

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.

Sample	Number of laboratories	Mean single sheet thickness µm	Repeatability standard deviation	Coefficient of variation $C_{V,r}$ %	Repeatability limit r µm
			μm		
Sample level 1 ^a	18	62,5	1,72	2,75	4,77
Sample level 2 a	18	76,1	1,64	2,16	4,55
Sample level 3 a	18	211	1,6	0,8	4,4
Sample level 4 a	18	592	4,9	0,8	13,6
a Levels 1, 2, 3 and 4 a	are according to the c	lassification by the Con	federation of Europea	an Paper Industries (C	CEPI).

Table B.2 — Estimation of the reproducibility

Sample	Number of laboratories	Mean single sheet thickness	Reproducibility standard deviation	Coefficient of variation $C_{V,R}$	Reproducibility limit R
		μm	s _R µm	%	μm
Sample level 1 a	18	62,5	1,62	2,59	4,49
Sample level 2 a	18	76,1	1,40	1,84	3,88
Sample level 3 a	18	211	2,0	0,9	5,5
Sample level 4 a	18	592	3,3	0,6	9,15

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- [4] ISO/TR 24498:2006, Paper, board and pulps Estimation of uncertainty for test methods
- [5] TAPPI Test method T 1200 sp-07, Interlaboratory evaluation of test methods to determine TAPPI repeatability and reproducibility
- [6] ISO 5725-1:1994, Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions

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