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Paper and board — Determination of grease resistance —

Part 3: Turpentine test for voids in glassine and greaseproof papers

Papier et carton — Détermination de l'imperméabilité aux graisses —

Partie 3: Essai à la térébenthine pour papiers glassine et papiers ingraissables

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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 16532-3 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 2, *Test methods and quality specifications for paper and board*.

ISO 16532 consists of the following parts, under the general title *Paper and board — Determination of grease resistance*:

— *Part 1: Permeability test*

— *Part 2: Surface repellency test*

— *Part 3: Turpentine test for voids in glassine and greaseproof papers*

Introduction

The resistance of paper and board to penetration by fats, greases and oils in paper and board is of particular importance for certain purposes, for example the packaging of food. The packaging should not only provide an effective grease barrier, but should also prevent the formation of aesthetically unacceptable grease spots on the packaging surfaces.

Paper and board — Determination of grease resistance —

Part 3:
Turpentine test for voids in glassine and greaseproof papers

1 Scope

This part of ISO 16532 specifies a method for the determination of the grease resistance of paper and board. It provides an accelerated comparison of the relative rates at which oils or greases, such as those commonly found in foodstuffs, can be expected to penetrate voids in papers such as greaseproof or glassine, where the grease or oil resistance is provided by mechanical means only. It is not applicable to grades of paper or board that are given grease or oil resistance by means of a coating or internal treatment for which ISO 16532-1 or ISO 16532-2 apply.

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 the 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
grease resistance
ability of the paper or board to resist the formation of surface spots or stains, or the permeation of grease through the sheet

3.2
voids
places in the paper where the arrangement of the fibres is such that oil or grease can penetrate the sheet

4 Principle

The test piece is placed on a sheet of coated paper on a flat surface and a small mound of sand is placed on the test piece. The sand is then saturated with coloured turpentine. The time taken for a stain to appear on the coated paper beneath the sand is noted.

5 Reagents

5.1 Pure gum spirits of turpentine, with a density of 860 kg/m^3 to 875 kg/m^3 at 20 °C.

5.2 Anhydrous calcium chloride.

5.3 Oil-soluble dye (for example Sudan red).

5.4 Sand, Ottawa cement-testing sand, screened to pass an 800 µm screen but retained on a 630 µm screen.

NOTE Ottawa cement-testing sand is available commercially from some laboratory suppliers.

5.5 Test solution: to 100 ml of turpentine (5.1), add 5 g of anhydrous calcium chloride (5.2) and 1,0 g of oil-soluble dye (5.3) and store in a stoppered bottle (6.7). Shake well, and let the solution stand for at least 10 h, shaking occasionally. Then filter through a dry filter paper at a temperature of 23 °C ± 1 °C, and store in the stoppered bottle (6.7). Ensure that the bottle is airtight.

6 Apparatus

6.1 Tube of any rigid material, 25 mm internal diameter and at least 25 mm in height, the ends of which have been smoothed.

6.2 Automatic pipette, of appropriate maximum volume, calibrated to deliver 1,1 ml of test solution.

6.3 Printing paper, flat, white, coated and calendered sheet or sheets of a convenient size to support at least 10 test pieces.

A grammage of 100 g/m^2 to 120 g/m^2 is recommended (see ISO 536).

6.4 Stopwatch or **timer**, accuracy ± 0,5 s.

6.5 Watch glass, diameter 76 mm.

6.6 Scoop, of capacity 5 g. Verify the capacity of the scoop by weighing 2 charges of sand (5.4) on an analytical balance (6.8): each charge should weigh (5,0 ± 0,1) g. The design of the scoop should preferably facilitate the pouring of the sand (5.4) into the tube (6.1).

6.7 Stoppered glass bottle, of capacity 100 ml.

6.8 Analytical balance, of minimum capacity 10 g and with a scale interval of 0,01 g; accuracy class ordinary (IIII)[1)].

7 Sampling

If the test is being made on a lot of paper or board, the sample shall be selected in accordance to ISO 186. If the test is being made on another type of sample, report the source of the sample and, if possible, the sampling procedure used. Make sure that the test pieces taken are representative of the paper or board sample.

1) Accuracy classes for non-automatic weighing instruments are described in OIML R 76-1[5].

8 Conditioning

Condition the sample in accordance with ISO 187. The alternative conditions specified in ISO 187 shall not be used, as it has been determined that the temperature has a strong influence on the test results. Thus only 23 °C ± 1 °C shall be used. Place the bottle of test solution (5.5) in the conditioned atmosphere and allow its temperature to come into equilibrium.

9 Preparation of test pieces

Prepare from the conditioned sample, in the same conditioned atmosphere (see Clause 8), ten test pieces of 100 mm × 100 mm, identifying, if possible, five pieces as the top side and five pieces as the wire side. Mark the first side of each test piece as side 1.

10 Procedure

10.1 Place a coated paper sheet or sheets (6.3), on a smooth, flat horizontal surface. Then, alternatively place the first marked side (top side if known or side 1 if unknown) of a test piece, followed by the other side, such that no test piece extends beyond the edge of the coated paper.

10.2 Rest one end of the tube (6.1) squarely on a test piece and with the scoop (6.6) put 5,0 g ± 0,1 g of the sand (5.4) in the tube. Remove the tube immediately after addition of the sand by carefully lifting it vertically.

NOTE The purpose of the tube is solely to ensure that a uniform sand pile is applied to the test piece.

10.3 Add 1,1 ml ± 0,05 ml of the test solution (5.5) to the sand pile, using an automatic pipette (6.2). Start the timing device (6.4) as the last drop of test solution leaves the pipette.

10.4 At specific intervals (see Clause 11), gently slide the test piece to a new unexposed position of the coated paper and examine the previously exposed position for signs of staining. The first sign of staining, which indicates that the test solution (5.5) has penetrated the test piece, is the end-point of the test.

10.5 Record the time elapsed from the addition of the test solution (5.5) to the sand until the first sign of staining of the coated paper. If staining has not occurred after 30 min (1 800 s), terminate the test.

If it takes longer than 120 s for the test solution (5.5) to penetrate the test piece, cover the sand pile with the watch glass (6.5).

10.6 Repeat 10.2 to 10.4 for the remaining test pieces.

NOTE If the first pair of tests show that the end-point times exceed 900 s, the test may be expedited by placing the remaining 8 test pieces on the coated paper, adding sand (5.4) and then test solution (5.5), followed by starting the timer (6.4) as in 10.3 at 10 s intervals.

11 Observation intervals

Make observations at least at the following intervals:

— every 15 s between 0 and 1 min;

— every 1 min between 1 min and 5 min;

— every 5 min between 5 min and 30 min.

If the end-point times for the first two test pieces exceed 15 min, make observations every 5 min, starting at 15 min.

12 Expression of results

Calculate the mean penetration time, in seconds, for all test pieces to two significant figures. If the results from the two sides can be distinguished, either as top and wire sides or by a clear difference in the values, then calculate separately the means of the two sides. Also note the maximum and minimum values.

If, in the calculation of any mean values, one or more results is greater than 1 800 s (30 min), include these values as 1 800 and report the mean as $> X$ (where $>$ indicates greater than). For maximum values greater than 1 800 s, record as $>$ 1 800.

EXAMPLE Assuming that it is not possible to identify the top and wire sides, record as follows:

	Side 1	Other side
	1 800	600
	1 700	550
	1 600	500
	1 700	600
	1 800	500
Mean	> 1 700	550
Minimum	1 600	500
Maximum	> 1 800	600

13 Test report

The test report shall include the following information:

a) a reference to this part of ISO 16532 (i.e. ISO 16532-3:2010);

b) all information for the complete identification of the sample;

c) the date and place of testing;

d) when possible, identification of the side or sides tested;

e) for each sample the mean, and maximum and minimum values, and if applicable, individual results for the two sides;

f) any departure from this part of ISO 16532 or any other circumstances that might have affected the results.

Annex A
(informative)

Precision

A.1 General

In 2008, five paper samples of varying greaseproof levels were subjected to an international round-robin in which 10 laboratories participated. Five tests were carried out on both the top side and wire side of each sample. The results were statistically analysed for repeatability and reproducibility and are summarized in Tables A.1 and A.2.

For samples 3 to 5, no significant difference between results for the two sides were found, therefore the repeatability and reproducibility values are mean values of both sides.

The calculations have been made according to ISO/TR 24498[2] and TAPPI T 1200[4].

The repeatability standard deviation indicated in Table A.1 is the “pooled” repeatability standard deviation, that is, the standard deviation 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[1].

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 $2{,}77 = 1{,}96\sqrt{2}$, 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 Repeatability

Table A.1 — Estimation of the repeatability of the test method

Sample	Mean value s	Standard deviation s_r s	Coefficient of variation CV %	Repeatability limit r s
1, wire side[a]	1 403	177	12,6	490
2	> 1 800	Not calculated[b]	Not calculated[b]	Not calculated[b]
3	82,8	35,4	43,0	98,2
4	1 141	197	14,0	549
5	22,4	4,6	20,7	12,8

[a] Almost all results for the top side were > 1 800 s.

[b] Repeatability values have not been calculated, since all results were > 1 800 s.

A.3 Reproducibility

Table A.2 — Estimation of the reproducibility of the test method

Sample	Mean value s	Standard deviation s_R s	Coefficient of variation CV %	Reproducibility limit R s
1, wire side[a]	1 403	580	41,3	1 609
2	> 1 800	Not calculated[b]	Not calculated[b]	Not calculated[b]
3	82,8	51,8	62,5	143
4	1 141	466	33,0	1 295
5	22,4	12,9	58,0	36,0

[a] Almost all results for the top side were > 1 800 s.

[b] Reproducibility values have not been calculated, since all results were > 1 800 s.

Bibliography

[1] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[2] ISO/TR 24498:2006, *Paper, board and pulps — Estimation of uncertainty for test methods*

[3] TAPPI Test Method T 454 om-06, *Turpentine test for voids in glassine and greaseproof papers*

[4] TAPPI Test Method T 1200 sp-07, *Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility*

[5] International Recommendation OIML R 76-1, *Non-automatic weighing instruments — Part 1: Metrological and technical requirements — Tests*

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