INTERNATIONAL STANDARD

ISO 16532-1

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Paper and board — Determination of grease resistance —

Part 1: Permeability test

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

Partie 1: Essai de perméabilité

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Foreword

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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 16532-1 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*

The following part is under preparation:

— *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 is of particular importance for certain packaging purposes, for example the packaging of food. The packaging should not only provide an effective grease barrier, but should also deter the formation of aesthetically unacceptable grease spots on the packaging surfaces.

Paper and board — Determination of grease resistance —

Part 1: Permeability test

1 Scope

This part of ISO 16532 specifies a method for the determination of the grease resistance of paper and board. The paper or board can be tested creased or uncreased. The test is primarily intended to establish a level of grease resistance by determining the time taken for a simulated “fat material” (palm kernel oil) to penetrate (break-through) the sheet for papers such as food board, and greaseproof and vegetable parchment. It is also applicable to paper and board which have been internally or surface sized with organophobic materials, or made grease resistant by a plastic extrusion coating.

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 4046-4, *Paper, board, pulps and related terms — Vocabulary — Part 4: Paper and board grades and converted products*

3 Terms and definitions

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

3.1
grease resistance
ability of paper or board to resist the formation of surface spots or stains or the permeation of grease through the sheet

3.2
grease permeability
ability of the paper or board to resist penetration of the grease through the sheet

NOTE Grease permeability is described by two characteristics: “break-through time” (actual penetration time) and “show-through time” (visual penetration time).

3.3
break-through time
time elapsed between the application of the test grease, together with a weight, to one side of the test piece and the penetration of grease through to the other side of the test piece

3.4
show-through time
time elapsed between the application of the test grease, together with a weight, to one side of the test piece and the first visual detection of grease stains (or a partial penetration of the grease through the sheet)

NOTE 1 The determination of break-through time is the primary purpose of this part of ISO 16532. However, show-through time may be of interest in special cases, for example, in the study of papers and boards internally or surface sized with organophobic materials, or of papers and boards which have been made grease resistant by plastic extrusion coating. The two tests may be combined, in which case show-through time is determined before break-through time.

NOTE 2 For many grades of paper and board, show-through times and break-through times are nearly identical.

4 Principle

The test pieces are placed on a glass plate, with or without a layer of cellulose wadding, and dyed palm kernel oil is applied, together with a weight, to the upper side of the test pieces. The time elapsed until an indication of partial or actual penetration of the grease through each test piece is noted, as described in the following paragraphs.

For determination of actual penetration (**break-through**) of grease through the test pieces, the end point is indicated by a visual observation of staining of the cellulose wadding in contact with the test pieces.

NOTE In practice, the time needed to penetrate the cellulose wadding (6.1) is included; this is very short and therefore negligible.

For determination of partial penetration (**show-through**) of grease, the end point is determined when the eye can detect spots or stains of grease without the aid of the cellulose wadding.

5 Reagents

5.1 Standard grease consisting of palm kernel oil[1)] or other oil with the following properties:

temperature of liquefaction: 27 ºC to 29 ºC;

dynamic viscosity at 35 ºC: 33,5 mPa·s to 35,0 mPa·s;

refractive index at 40 ºC: 1,44 to 1,45;

dyed with 0,25 % (mass fraction) Sudan red or a similar fat soluble dye.

If the grease is lumpy, it shall be homogenized before use with the aid of a spatula.

Greases other than the standardized material may be used, if they meet the specification outlined above, in which case this fact is to be stated in the test report.

6 Apparatus

6.1 Cellulose wadding, made of bleached chemical pulp (see ISO 4046-4).

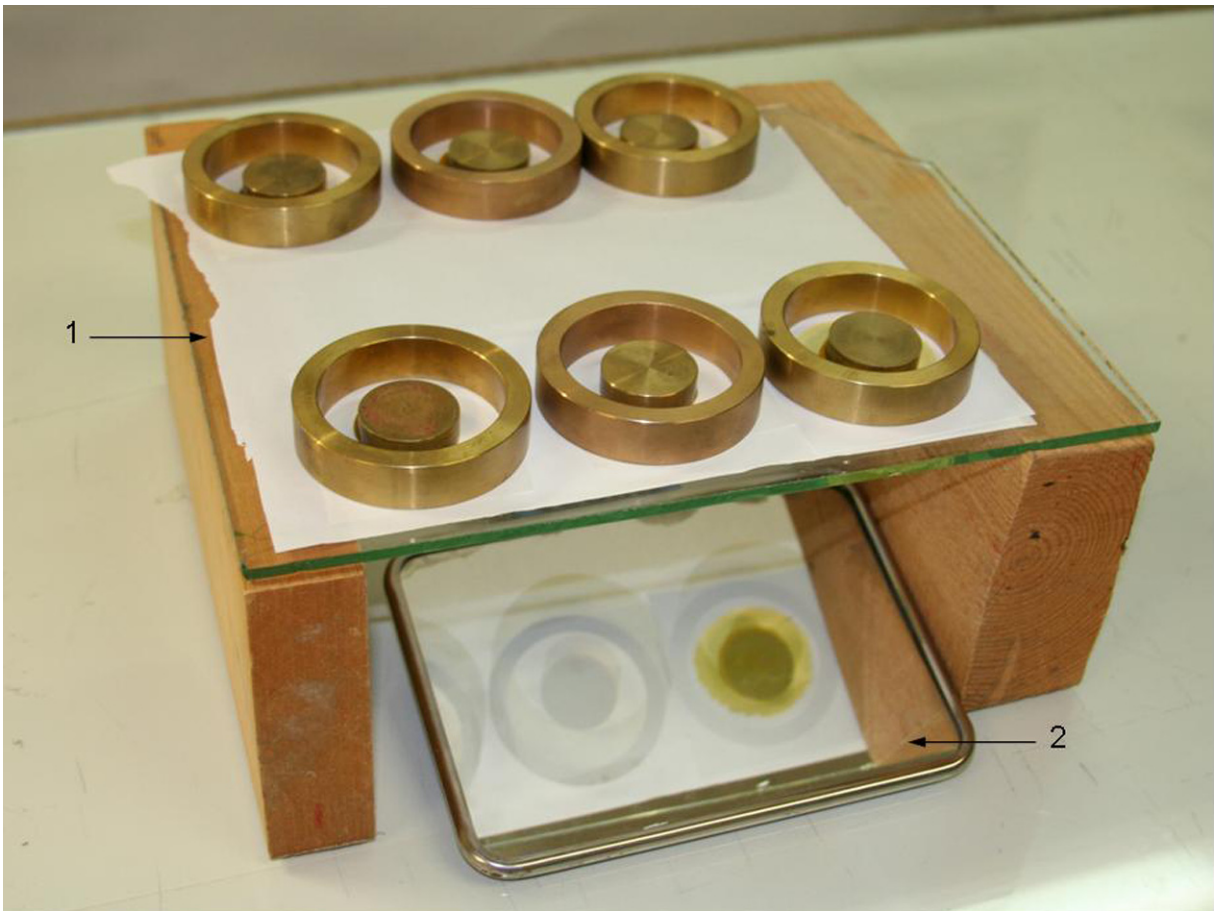
1) Dyed palm kernel oil suitable for this test is commercially available from ISEGA Forschungs- und Untersuchungsgesellschaft mbH, P.O. Box 10565, 63704 Ashaffenburg, Germany (www.isega.de). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the products from ISEGA Forschungs- und Untersuchungsgesellschaft mbH. Equivalent products may be used if they can be shown to lead to the same results.

The penetration time when measured with palm kernel oil shall be less than 15 s.

6.2 Glass plate, not smaller than 220 mm × 350 mm.

The plate shall be supported in such a way that the underside can be viewed in a mirror.

6.3 Mirror, placed beneath the glass plate (6.2) in such a way that the whole of the undersides of the test pieces can be viewed (see Figure 1). Ensure that the mirror has adequate lighting.



Key

1 glass plate

2 mirror

NOTE 1 In the test assembly shown, the horizontal glass plate is supported above bench height so that the mirror can be placed beneath.

NOTE 2 In the example shown, the sample on the right hand side is well past its end point.

Figure 1 — Test assembly for the determination of break-through time

6.4 Creasing apparatus, as described in Annex A.

6.5 Metal template, round or square (approximately 60 mm diameter or 60 mm × 60 mm square), and 2 mm to 3 mm thick, with a hole, of 30 mm diameter. The template is used to apply a controlled volume of grease.

6.6 At least 10 test weights, mass 50 g to 55 g, diameter 30 mm.

6.7 Ten metal rings, mass about 200 g, external diameter 65 mm to 70 mm, internal diameter about 55 mm.

6.8 Stopwatch or **timer.**

7 Sampling

If the test is being made on a lot of paper or board, the sample shall be selected in accordance with 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 the test pieces taken are representative of the paper or board sample.

8 Conditioning

Condition the samples in accordance with ISO 187, at 23 ºC ± 1 ºC and 50 % ± 2 % relative humidity.

NOTE The alternative testing conditions of a temperature of 27 ºC ± 1 ºC and 65 % ± 2 % relative humidity are not applicable to the testing of grease-resistant materials.

9 Preparation of test pieces

From the samples, cut at least 10 test pieces, about 60 mm × 60 mm, with the sides parallel to the machine and cross directions. Mark the directions on the test pieces and the top and wire sides if known. If both sides are to be tested (see 10.1), at least 20 test pieces will be required.

If creasing is required, the procedure described in Annex A shall be followed.

10 Procedure

10.1 General

The test shall be carried out in the same conditions as those used to condition the samples. Carry out at least 10 determinations on the side to be tested. If it is not known which surface is to be in contact with the contents of the package, test both sides. If both sides are to be tested (see 10.1), at least 20 test pieces will be required.

10.2 Determination of show-through time

10.2.1 If show-through time is to be determined, it should preferably be determined separately to break-through time.

10.2.2 Place each test piece with the surface to be in contact with the content of the package upwards, directly on the horizontal glass plate (6.2) as shown in Figure 1.

Place the metal template (6.5) on the test piece. Press it firmly, and completely fill the hole with the standard grease (5.1), bringing it into contact with the test piece. Start the timer (6.8) and draw a straight edge over the top surface of the template, giving the layer of grease a plane upper surface of uniform thickness. Remove the template and centre a metal ring (6.7) on the test piece. Place a weight (6.6) centred on the layer of grease, on each test piece. Note the appearance in the mirror of the first spot or stain and record the time elapsed. If the test pieces are creased, place the template in such a way that the hole is immediately above the intersection of the creases and note which crease has been penetrated. Make observations as in 10.4.

10.2.3 It is possible to combine show-through and break-through tests by noting the first spot or stain as described above. Then **immediately** and **carefully** transfer the test pieces to a thin layer of cellulose wadding (6.1) and continue as in 10.3.3.

10.2.4 If the two tests are combined, care should be taken to ensure that the results for show-through and break-through are recorded independently and correctly.

10.3 Determination of break-through time

10.3.1 If only break-through time is to be measured, place each test piece, with the surface to be in contact with the content of the package upwards, on a thin layer of cellulose wadding (6.1) on the horizontal glass plate (6.2) as shown in Figure 1.

10.3.2 Place the metal template (6.5) on the test piece. Press it firmly, and completely fill the hole with the standard grease (5.1), bringing it into contact with the test piece. Start the timer (6.8) and draw a straight edge over the top surface of the template, giving the layer of grease a plane upper surface of uniform thickness. Remove the template and centre a metal ring (6.7) on the test piece. Place a weight (6.6), centred on the layer of grease, on each test piece. If the test pieces are creased, place the template in such a way that the hole is immediately above the intersection of the creases.

10.3.3 Examine the underside of the test pieces in the mirror and note the time which elapses until the first coloured stains are observed on the cellulose wadding. If the test pieces are creased, note which crease has been penetrated. Make observations as in 10.4.

10.4 Observation intervals

Make observations at least at the following intervals for each side tested:

— every 1 min for the first 10 min;

— every 2 min between 10 min and 30 min;

— every 5 min between 30 min and 60 min;

— every 10 min between 60 min and 150 min;

— every 30 min between 2 h 30 min and 6 h;

— after 24 h (final inspection).

11 Precision

Attempts to establish a precision statement by an interlaboratory trial have not been successful. This was partly due to the subjective nature of the method, and also the method of observation of the results. Recording persistent test values of “greater than” is not conducive to data analysis for repeatability and reproducibility.

12 Expression of results

Calculate the mean and the range for the break-through time and the show-through time if required. If both sides of the test piece were tested, calculate the mean and the range separately for each side.

Express the results as follows:

— below 10 min: to the nearest 1 min;

— 10 min to 30 min: to the nearest 2 min;

— 30 min to 60 min: to the nearest 5 min;

— 60 min to 150 min: to the nearest 10 min;

— 2 h 30 min to 6 h: to the nearest 30 min;

— 6 h to 24 h: express as between 6 h and 24 h;

— after 24 h: express as over 24 h.

13 Test report

The test report shall include the following information:

a) a reference to this part of ISO 16532;

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

c) the date and place of testing;

d) identification of the surface tested;

e) identification of the grease used in the test if other than the prescribed palm kernel oil;

f) creasing, if carried out, and the force if other than 100 N/mm;

g) the break-through time (mean and range) for each side tested;

h) if required, the show-through time (mean and range) for each side tested;

i) any departures from this part of ISO 16532 or any other circumstances that may have affected the results.

Annex A
(normative)

Creasing

A.1 Apparatus

A.1.1 **Creasing bed**, consisting of a flat plate in which a right-angled groove has been cut (Figure A.1) and a bar to fit the groove. The bar shall have a machined, but not cutting, edge (radius of curvature about 0,3 mm).

Other creasing apparatus may be used, provided that the reverse side of the crease is not broken.

A.2 Procedure

A.2.1 Place a test piece on the creasing bed so that the machine direction is parallel with the groove in the bed. Produce a crease by pressing the bar into the groove with a force of 100 N per linear metre of crease for 10 s to 15 s. For practical purposes, this may be achieved by loading the bar with a mass of 1 kg for each linear centimetre to be creased.

Make a second crease at right angles to and intersecting the first. After creasing, examine the reverse side of the test piece to ensure they are unbroken.

Repeat the procedure with all test pieces, with five top side pieces upwards and five wire side pieces upwards.

A.2.2 A force of 100 N/m may not be sufficient to create a distinct crease in very thick boxboard. In this case, additional weights should be applied to the bar and the actual force reported together with the results.

Dimensions in millimetres

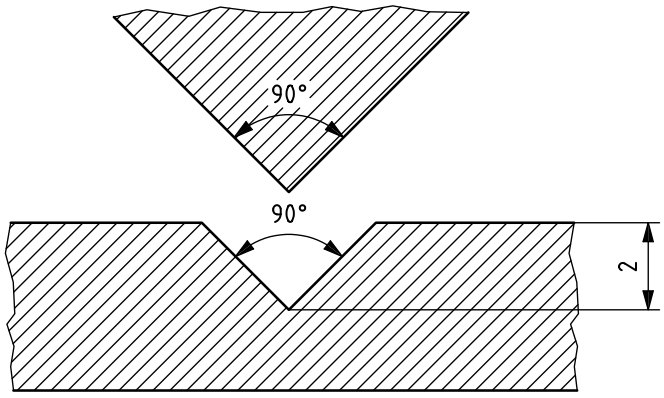


Figure A.1 — Creasing bed

Bibliography

[1] DIN 53116:2003, *Testing of paper — Determination of grease permeability*

ICS 85.060

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