INTERNATIONAL STANDARD

ISO 16532-2

First edition
2007-09-15

Paper and board — Determination of grease resistance —

Part 2: Surface repellency test

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

Partie 2: Essai de résistance au mouillage de surface

Reference number
ISO 16532-2:2007(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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 16532-2 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: Grease permeability method*

— *Part 2: Surface repellency test*

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

Introduction

The resistance to the penetration of fats, greases and oils by paper and board 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 2: Surface repellency test

WARNING — The method specified in this part of ISO 16532 involves the use of some hazardous chemicals, which are highly inflammable and are reported to be health hazards. Care shall be taken to ensure that the relevant safety precautions are observed.

1 Scope

This part of ISO 16532 specifies a method for the determination of the grease resistance of paper and board. Primarily, it is intended to establish an approximate level of grease resistance, by testing the degree of surface repellency and/or antiwicking characteristics of paper and board, internally or surface sized with organophobic materials such as fluorocarbons. Some packaging paper and board products, designed for containing pet foods, food for human consumption and other materials, are produced in this way, and any surface stains, due to grease wetting or penetrating the paper or board surface, are aesthetically unacceptable.

This test is not intended to determine the permeability of grease through the paper or board, for which ISO 16532-1 applies.

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*

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
surface repellency
ability of the surface of the paper or board to resist any wetting reaction to applied grease materials

3.3
Kit rating
highest number for a series of test solutions that can be placed on the surface of the paper or board without causing a wetting interaction by darkening the paper or board surface

NOTE A higher number implies a better grease resistance for the sample tested.

4 Principle

A range of numbered solutions referred to as Kit ratings is prepared, consisting of a mixture of castor oil and two solvents. Differing degrees of dilution of the oil with the solvents provides varying degrees of "aggressiveness", and therefore different Kit ratings for the solution to be applied. A drop of one such solution is applied to the paper or board surface and the end-point is determined by establishing which of the solutions is the first to cause a wetting interaction by darkening the paper or board surface.

5 Reagents

5.1 **Castor oil**, analytical grade.

5.2 ***n*-Heptane**, analytical grade.

5.3 **Toluene**, analytical grade.

5.4 **Kit test solutions**, prepared from the above reagents using the ratio of mixtures shown in Table A.1 in Annex A.

6 Apparatus

6.1 **Absorbent tissue or cotton swatch** (examples, cotton cloth or filter paper), to wipe away Kit test solutions at the end of the test.

6.2 **Balance**, capable of weighing 2 000 g to within 0,1 g.

6.3 **Graduated cylinders**, made of glass, of capacity 100 ml and 500 ml; for preparing Kit test solutions.

6.4 **Stock bottles**, made of glass, with foil-lined caps, glass stoppers, or polythene-lined caps to prevent evaporation losses; for prepared Kit test solutions.

6.5 **Stopwatch or timer**.

6.6 **Test bottles**, with glass stoppers and glass eye-droppers; for use during testing.

6.7 **Testing area**, located in a laboratory with a conditioned atmosphere (see 10.1). The testing area should be a clean, dark bench top with bright, uniform lighting. If the bench top does not have a dark surface, each test piece will need to be backed up with a darker material before testing.

7 Sampling

If the test is being carried out on a lot or consignment 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 that the test pieces taken are representative of the paper or board sample.

8 Conditioning of samples

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

9 Preparation of test pieces

From each sample, cut 10 test pieces with a dimension of at least 50 mm × 150 mm. Label or identify, in some manner, each side of the test specimen.

Use care when handling the test pieces. The use of plastic gloves to protect the sample is recommended. Do not test in an area that has fingerprints, oil from a previous test, or any other defects in the test piece which may affect the results.

10 Procedure

10.1 The test shall be carried out in a conditioned room, having a temperature of 23 ºC ± 1 ºC and 50 % ± 2 % relative humidity. Carry out at least 5 determinations on the side to be tested. If the side to be in contact with the fatty product is known, test this side. If not, test both sides.

10.2 Place each test piece on a clean, flat surface, with the test side uppermost, in the testing area (6.7).

10.3 Select one of the test pieces and a Kit test solution (5.4) of intermediate number contained in one of the test bottles (6.6). From a height of about 10 mm, gently release a drop of the test solution from the test bottle onto the surface of the test piece, and start the timer (6.5). Ensure that the glass eye-dropper of the test bottle does not touch the surface of the test piece.

10.4 After 15 s quickly remove the excess test solution with a clean tissue or cotton swatch (6.1) and **immediately examine** the surface to which the test solution was applied. The end-point of the test is denoted by a darkening of any part of the paper or board surface where the test solution was applied.

NOTE 1 If the surface where the testing solution was applied is **not** examined immediately, volatile components of the test solution will be lost and the testing spot may regain its original light-reflectance value and the end-point of the test will not have been reached.

NOTE 2 Darkening of the paper or board surface is a result of wetting of the test piece by the test solution.

NOTE 3 Detection of the end-point may be difficult in paper or board which has been highly refined, or in sheets which have a low thickness and opacity.

10.5 If the end-point is attained on this first test, select an untested area on the same test piece and repeat the procedure in 10.3 and 10.4 using a lower test solution number. Continue this procedure until the highest numbered test solution that does not exhibit an end-point (as in 10.4) is identified. The number of this test solution is the Kit rating for the test piece. Repeat the above procedure for the remaining four test pieces and record their Kit ratings.

NOTE Results from the first test can be used as an indication of which test solution to use for the first drop on the remaining four test pieces.

11 Expression of results

Calculate the mean Kit rating to the nearest even Kit rating number and note the maximum and minimum values of the five test pieces, for each sample of paper or board.

12 Precision

Twelve samples of paper, representing Kit ratings of 1 to 12, were subjected to an international round-robin exercise by 5 laboratories. The results were statistically analysed for repeatability and reproducibility and these values are recorded in Table 1.

Table 1 — Estimates of test method precision

Expected Kit ratings	Repeatability r	Repeatability ratio %	Reproducibility R	Reproducibility ratio %
1 to 5	1,0	10,0	0,8	30,0
6 to 9	0,9	10,5	3,8	45,0
10 to 12	0,6	6,0	1,5	14,0

The results show good repeatability within laboratories and a good reproducibility between laboratories for high Kit ratings. A lower reproducibility between laboratories is evident, particularly for mid-range Kit ratings, which reflects the subjective nature of the test.

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) date and place of testing;

d) conditioning atmosphere used;

e) identification of the side or sides tested;

f) the mean Kit rating rounded to the nearest even Kit rating number for each sample;

g) the number of test results obtained to determine the mean for each sample;

h) the maximum and minimum test results for each sample;

i) any departure from this part of ISO 16532 and any circumstances that may have affected the results.

Annex A
(normative)

Mixtures of reagents for preparing Kit test solutions

Table A.1 — Mixtures of reagents for preparing Kit test solutions[1]

Kit rating Solution number	**Castor oil** g	**Toluene** ml	***n*-Heptane** ml
1	960,0	0	0
2	864,0	50	50
3	768,0	100	100
4	672,0	150	150
5	576,0	200	200
6	480,0	250	250
7	384,0	300	300
8	288,0	350	350
9	192,0	400	400
10	96,0	450	450
11	0	500	500
12	0	450	550

For certain applications, the range of Kit ratings may be extended beyond 12 by continuing to adjust the proportion of toluene and *n*-heptane. If this is done, the report shall clearly state the ratio of toluene to *n*-heptane used to arrive at the extended Kit rating.

Do not measure the reagents by addition, since there will be a loss of volume by mixing.

NOTE Weight measurements are used for castor oil (5.1) as its high viscosity makes volume measurements less accurate. The density of castor oil is between 0,957 g/ml and 0,961 g/ml at 25 °C, and a value of 0,960 g/ml is used for the purposes of calculation. Any increase in the density of castor oil by its use at 23 °C is insignificant.

Store the solutions in labelled stock bottles (6.4). As required, fill each test bottle (6.6) with the appropriate Kit number solution from the stock bottles.

The stock and test bottles should be tightly closed when not in use, to prevent loss of solvent from evaporation (and a consequent change in composition and properties of the solution). It is recommended that solutions in the stock bottles should be renewed every two months.

The solutions in the test bottles should be changed regularly depending on usage. For frequent daily usage, solutions should be replaced weekly with fresh solution from the stock bottles; for less frequent usage, a monthly change is adequate.

Bibliography

[1] TAPPI test method T 559 cm-02, *Grease resistance test for paper and paperboard*, Tappi Press, Atlanta, GA, USA

[2] LANGE, J., PELLETIER, C. and WYSER, Y. Novel method for testing the grease resistance of pet food packaging. *Packaging Technology Science,* 2002, **15**, pp. 65-74

ICS 85.060

Price based on 6 pages