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ISO 5630-5

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Paper and board — Accelerated ageing —

Part 5: Exposure to elevated temperature at 100 °C

Papier et carton — Vieillissement accéléré —

Partie 5: Exposition à une température élevée à 100 °C

Reference number
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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

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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 5630-5 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 5630 consists of the following parts, under the general title *Paper and board — Accelerated ageing*:

— *Part 1: Dry heat treatment at 105 °C*

— *Part 3: Moist heat treatment at 80 °C and 65 % relative humidity*

— *Part 4: Dry heat treatment at 120 or 150 °C*

— *Part 5: Exposure to elevated temperature at 100 °C*

— *Part 6: Exposure to atmospheric pollution (nitrogen dioxide)*

Introduction

Exposure of paper or board to a hostile environment, such as some type of radiation, elevated temperature or chemical attack over a period of hours can provide information concerning the natural changes that may occur in the material over a period of years.

The test method described in this part of ISO 5630, for accelerated ageing by exposure of moist paper to an elevated temperature, is based on the method developed by ASTM[6] following an extensive research programme. In this programme, 15 printing and writing papers were tested, representing a wide variety of paper types (acid and alkaline, lignin-containing and lignin-free, and those without an alkaline reserve such as calcium carbonate). This test method proved to give more accurate results than earlier methods regarding the effect of elevated temperature on the mechanical strength and optical stability of such papers. For a full understanding of the stability of the permanence of the paper, a combination of test methods for accelerated ageing should be used.

Paper and board — Accelerated ageing —

Part 5:
Exposure to elevated temperature at 100 °C

1 Scope

This part of ISO 5630 specifies a method for accelerating the ageing of printing and writing papers, through the use of elevated temperature, and assessing its effect on the mechanical properties of fold number and tearing resistance, for the purpose of predicting stability to long-term natural ageing.

It is applicable to all cellulose-based printing and writing papers, including coated papers and filled papers.

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

ISO 1974, *Paper — Determination of tearing resistance (Elmendorf method)*

ISO 5626, *Paper — Determination of folding endurance*

3 Principle

Conditioned paper samples are placed inside airtight glass tubes and aged at an elevated temperature for a certain time. By comparing fold number and tearing resistance before and after the ageing, a measure of the stability of paper strength is obtained.

4 Apparatus

4.1 Laboratory forced-ventilation oven, capable of maintaining a uniform temperature of 100,0 °C ± 1,0 °C, with sufficient racks to allow horizontal orientation of glass tubes.

4.2 Airtight glass tubes, of sufficient internal height to accommodate the test pieces.

The internal volume shall be of a convenient magnitude to meet the mass of test pieces per internal volume of tube specification (see Clause 8). The tubes shall have screw-on caps, fitted with O-rings or gaskets that ensure a hermetic seal of the interior contents during the ageing. The materials (glass, cap and O-ring or gasket) should be thermally stable at, or preferably well above, the ageing temperature of 100 °C, and chemically resistant to acid corrosion.

4.3 Balance, accurate to 0,01 g, for weighing test pieces and, when required, additional portions of sample to ensure that the required mass of sample per unit volume of tube is met (see 9.1).

4.4 Device for cutting additional portions of sample, when required, to meet the required mass of sample per unit volume of tube (see 9.1).

5 Calibration and preparation of tubes for ageing

5.1 Calibration of the oven

Calibrate the oven with sufficient frequency to ensure a temperature of 100,0 °C ± 1,0 °C.

5.2 Preparation of glass tubes for ageing

5.2.1 Used glass tubes

Prior to the insertion of test pieces, thoroughly clean used glass tubes.

The required cleaning sequence is as follows.

a) Wash tubes thoroughly with non-ionic detergent and tap water.

b) Rinse tubes thoroughly with tap water to remove all residual soap.

c) Rinse three more times with distilled water and allow the tubes to drain.

d) Dry the tubes in an oven at about 105 °C.

e) Condition the complete tube assembly (opened) for a minimum of 24 h in the atmosphere used for conditioning the paper samples (Clause 7).

5.2.2 New glass tubes

When starting with new tubes, caps, O-rings or gaskets, dry them at 105 °C for at least 24 h.

Condition the complete tube assembly (opened) for a minimum of 24 h in the atmosphere used for conditioning the paper samples (Clause 7).

5.2.3 Storage of tubes

After conditioning, cap the tubes and maintain them capped until use.

6 Sampling

Always handle the paper samples with clean cotton gloves.

If the average quality of a lot is to be determined, sampling shall be carried out in accordance with ISO 186. If another type of sample is to be tested, make sure that the specimens taken are representative of the sample received.

If the sheets are much larger than A4 in size[1], cut them into sheets approximately A4 in size.

If possible, randomize the sheets. Select enough sheets for testing of aged and unaged paper, including additional material sufficient to meet the required mass of sample per unit volume of tube (9.1) and to allow for invalid tests.

If the sheets are less than A4 in size, select a similar area of paper sufficient for the tests to be performed.

7 Conditioning

Condition the sheets, including those which will not be aged, in accordance with ISO 187 immediately prior to the ageing period.

The sheets which will not be aged shall be kept in the standard atmosphere until their properties are tested.

8 Preparation of test pieces

From the sheets, cut the required number of test pieces of the appropriate dimensions for the tests to be performed on both unaged and aged material (see 9.3). Follow the instructions in ISO 1974 for preparation of test pieces for tearing resistance and the instructions in ISO 5626 for folding endurance (fold number).

NOTE It may be advisable to cut additional material to allow for invalid tests, see ISO 1974 and ISO 5626.

Determine the dry matter content of the conditioned sheets in accordance with ISO 638, and calculate the oven-dry mass.

9 Procedure

9.1 Ageing of test pieces

Calculate the number of airtight glass tubes (4.2) needed such that each tube will contain (0,027 8 ± 0,000 07) g of oven-dry sample per cubic centimetre internal volume of tube. If the calculation does not give an integer number, select the next highest number.

Insert the material to be aged into the glass tubes such that each tube contains approximately the same mass of paper. Pieces which form the tearing resistance test pieces should be rolled together prior to insertion and then inserted in the tubes. The fold-number test pieces should then be inserted inside these.

If the mass of oven dry sample per unit volume of tube is lower than that specified (after selecting the appropriate number of glass tubes), cut additional portions of sample and add to the tubes as required to comply with the specification. As an example, a tube of (145 ± 5) cm^3 internal volume shall contain (4,0 ± 0,1) g (oven-dry mass) per tube. Three tubes of this size may be sufficient for test pieces for fold number and for tearing resistance. The actual number of tubes and the need for additional portions to make up the required grams per cubic centimetre of volume depend on the oven-dry grammage of the sample, the specific dimensions of the test pieces and the number of pieces per test.

Put the caps into place on the tubes and secure (hand-tight) to provide a complete seal. Mark the exterior of the glass tubes for identification of the paper samples inside.

Insert loaded tubes into the heated oven (4.1) and place them one layer deep on each rack. Age the test pieces for (120 ± 0,5) h.

9.2 Removal and equilibration of the aged test pieces

Upon removal from the oven, allow the hot tubes to equilibrate unopened to room temperature for a minimum of 1 h. Note any observable differences in the appearance of samples, without uncapping them.

IMPORTANT — If the samples in some tubes have a lighter colour than the original samples, the tube may not have been airtight. Such samples shall be discarded.

Uncap the tubes in the same standard atmosphere as that used to initially condition the test specimens (Clause 7), and then allow the test pieces to equilibrate for at least 24 h in the tubes. Remove the additional portions of sample, if applicable, and the test pieces with tweezers, taking care to assure that no creases or folds are made in the test pieces.

Carefully unroll the individual pieces comprising the tearing resistance test pieces and place them on a flat, clean, inert surface. Cover the individual pieces with a sheet of glass or acrylic plastic. In order to provide a total weight of approximately 1 kg applied evenly over the full surface of the pieces, add weight at the top of the sheet of glass or acrylic plastic, as required. Leave the weight in place for at least 12 h, then carefully remove any additional weight and the sheet of glass or acrylic plastic from the pieces.

The uniform, low-pressure application of force on the surface of the tearing-resistance test pieces is required to reduce the curl at the time of their testing.

Allow the test pieces to reach equilibrium in the same atmosphere as used for the initial conditioning (Clause 7).

9.3 Testing of aged and unaged papers

Test the tearing resistance of the aged and unaged papers in the cross direction (CD) in accordance with ISO 1974, and fold number in the machine direction (MD) in accordance with ISO 5626.

10 Calculations

Calculate the results in accordance with the instructions in the relevant ISO standard.

Calculate the retention of the fold number, to the nearest percentage, using Equation (1):

$$R_1 = \frac{F_2 \times 100}{F_1} \tag{1}$$

where

R_1 is the retention of fold number, as a percentage;

F_2 is the fold number (MD) of the aged sample;

F_1 is the fold number (MD) of the unaged sample.

Calculate the retention of the tearing resistance, to the nearest percentage, using Equation (2):

$$R_2 = \frac{T_2 \times 100}{T_1} \tag{2}$$

where

R_2 is the retention of the tearing resistance, as a percentage;

T_2 is the tearing resistance (CD) of the aged sample;

T_1 is the tearing resistance (CD) of the unaged sample.

11 Test report

The test report shall include the following information:

a) reference to this part of ISO 5630;

b) reference to the International Standards used for testing the changes in properties due to ageing, if different from those that appear in this part of ISO 5630;

c) all information necessary for complete identification of the sample;

d) the date and place of testing;

e) the temperature and relative humidity of the atmosphere used to condition the samples;

f) the number of tests carried out, if different from this part of ISO 5630;

g) the MD fold numbers and the mean values of the CD tearing resistances of the aged and unaged samples, and the antilogarithms of the standard deviations of the folding endurances and the standard deviations of the CD tearing resistances of the aged and unaged samples;

h) the retentions of the MD fold number and CD tearing resistance, as percentages;

i) any deviations from the relevant International Standard from this part of ISO 5630, or any circumstances or influence which might have affected the test results.

Annex A
(informative)

Interpretation and limitations of test

A.1 Interpretation of test results

A.1.1 General

The test method in this part of ISO 5630 is based on the method developed by ASTM[6] following an extensive research programme (see Introduction). The papers were tested when freshly made and after accelerated ageing. The results give guidance as to which papers will prove to be mechanically stable for long periods of time as opposed to those that will quickly lose stability. Values for fold number retention and internal tearing-resistance retention were discovered to be useful for this test method to gain a reliable estimate of the long-term stability of test papers in natural ageing experience. (For guidance, see Reference [9], p. 44.)

NOTE A high fold number and a high tearing-resistance retention suggest a more stable paper.

A.1.2 Additional mechanical strength testing

Other mechanical strength tests may be performed at the discretion of the testing authority, in accordance with the relevant ISO test method, and may include:

a) tensile energy absorption (TEA), in accordance with ISO 1924-2 or ISO 1924-3;

b) zero-span tensile strength, in accordance with ISO 15361.

NOTE No paper stability determination guidance from the ASTM Paper ageing research programme can be given for these alternative tests.

A.1.3 Determination of stability

For a paper to be classified as stable, all of the paper stability determination results should be classified as stable. That is to say, if the paper stability determination results for internal tearing resistance are classified as stable but the results for fold number retention are classified as unstable, the paper should be judged unstable.

A.2 Limitations of the temperature test

It should be mentioned that natural ageing is variously the result of the action of heat, light and chemicals (e.g. pH), including pollutants from the air that become entrained into the paper. This part of ISO 5630 is intended to characterize only thermally induced reactions. In natural ageing, an infinite range of conditions can be found with a different “mix” of these elements. Therefore, for the greatest understanding of possible future ageing effects, the investigator may wish to accelerate paper ageing separately by elevated temperature, by elevated light flux, and by increased concentration of common pollutant gases. Relevant ISO standard test methods are appropriate means to evaluate these influences.