INTERNATIONAL STANDARD

ISO 5630-3

Second edition
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Paper and board — Accelerated ageing —

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

Papier et carton — Vieillissement accéléré —

Partie 3: Traitement à la chaleur humide à 80 °C et 65 % d'humidité relative

Reference number
ISO 5630-3:1996(E)

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.

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.

International Standard ISO 5630-3 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*, Subcommittee SC 2, *Test methods and quality specifications for paper and board.*

This second edition cancels and replaces the first edition (ISO 5630-3:1986), which has been technically revised.

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 °C or 150 °C*

Annexes A and B of this part of ISO 5630 are for information only.

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Introduction

Exposure of paper or board to a hostile environment, such as some types of radiation, elevated temperature, or chemical attack over a period of hours, may provide information concerning the natural changes that may occur in the material over a period of years.[1, 2]

Hostile environments that have been used include dry heat, heat and moisture, visible and ultraviolet radiation, and sulfur dioxide gas.

Properties compared before and after such exposure include mechanical, chemical and optical properties.

It has been determined that the degradation of cellulose is very sensitive to moisture.[3, 4] The rate of degradation increases about 25 % when the relative humidity increases from 60 % to 70 %. In order to be representative of natural conditions in many countries where conditions of high humidity, and perhaps high temperature, are common, it is desirable that in an accelerated ageing atmosphere paper should have the same moisture content as in a natural ageing atmosphere.[5, 6] For this reason, after studying the ageing of many papers under different conditions of temperature and relative humidity, 80 °C and 65 %relative humidity have been selected[5, 6] for tests of accelerated ageing.

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Paper and board — Accelerated ageing —

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

1 Scope

This part of ISO 5630 specifies a procedure for moist heat treatment of paper or board and the general approach for testing properties of the heat-treated materials. This method is based on work on printing and writing papers, but may be used with discretion for other types of paper and board.

The procedure is not recommended for papers such as resin-impregnated or varnish-treated papers, which increase in physical strength on heating.

The procedure is not applicable to certain electrical insulating papers, for which different conditions apply (see ISO 5630-4).

This part of ISO 5630 does not specify the tests to be made on the paper or board. It is left to the interested parties to determine which tests are appropriate for the type of paper or board being evaluated.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 5630. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 5630 are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 186:1994, *Paper and board — Sampling to determine average quality.*

ISO 187:1990, *Paper, board and pulps — Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples.*

3 Principle

Test specimens of paper or board are heated for a specified time at 80 °C and 65 % relative humidity. Agreed properties of the test specimens are compared before and after this moist heat treatment.

4 Apparatus

4.1 Ageing vessels, capable of being maintained at a temperature of (80 ± 0,5) °C and (65 ± 2) % relative humidity.

The temperature and relative humidity may be maintained either by the use of climatized cabinets and automatic control of temperature and humidity or by the use of constant-temperature baths (see annex A).

NOTE 1 Graminski *et al.*[3, 4] have shown that the rate of degradation of folding endurance and of zero-span tensile strength can be increased by changing the relative humidity at elevated temperatures. Therefore, for maximum precision, the temperature should be closely controlled, to within 0,5 °C or better, in order to hold the relative humidity variation to within 2 %.

4.2 Test equipment, relevant to the property tests agreed by the interested parties, complying with the relevant International Standard test method, if any, or with another appropriate standard test method.

4.3 Desiccator, or other preconditioner, maintained at 10 % to 35 % relative humidity.

5 Sampling

When possible, sampling shall be carried out in accordance with ISO 186.

6 Preparation of test specimens

Select and prepare five sets of test specimens in accordance with the relevant International Standard, if any, or another standard method relevant to the required test.

Protect the test specimens from light.

Avoid handling the specimens with bare hands and avoid their undue exposure to the atmosphere of a chemical laboratory.

NOTE 2 It is convenient to cut the test specimens oversize and then cut them to their correct size after ageing has been completed.

7 Heat treatment

Treatment shall be carried out in the dark. Without bending or folding, suspend four of the five sets of test specimens (clause 6) in ageing vessels (described in 4.1) in the oil bath, or in a climatized cabinet. Circulate air at the rate of (50 ± 25) ml/min at (80 ± 0,5) °C and (65 ± 2) % relative humidity through each of the ageing vessels immersed in the constant-temperature bath.

Remove a set of test specimens after (24 ± 0,25) h, (48 ± 0,5) h, (72 ± 0,75) h and (144 ± 1,5) h exposure to heat treatment.

NOTES

3 By agreement between vendor and purchaser, all of these specified exposure times may be used and the data plotted, or the data from only one exposure time taken and compared with the control.

4 The ageing vessel or cabinet should not contain more than one type of paper at any time, in order to prevent possible contamination by distillation or sublimation of products.

5 A suitable rack, made for example of stainless-steel wire, may be used to suspend the test specimens in the ageing vessels. By this means, two tiers of test specimens may be suspended in ageing vessels of the size suggested in annex A.

While this treatment is being carried out, keep a fifth set of untreated test specimens in the dark.

8 Preconditioning and conditioning

8.1 On completion of the moist heat treatment in accordance with clause 7, precondition the treated and untreated sets of test specimens as prescribed in ISO 187, and store in a desiccator (4.3) until tested.

8.2 On completion of the preconditioning treatment (8.1), transfer both the treated and untreated sets of test specimens to an atmosphere in accordance with ISO 187 and condition for at least 4 h, and preferably overnight.

9 Property testing

Test each of the specimens for the properties previously determined to be appropriate for the type of paper or board being evaluated (see clause 1). Use the relevant International Standard, if any, or any other appropriate standard method.

10 Expression of results

The following are some of the ways that the data may be presented.

a) Record the means and standard deviations of the test data for the aged and unaged test specimens.

b) Where the units of measurement allow, calculate the retention of the property, expressed as a percentage of the unaged value.

Retentions may be plotted.

NOTE 6 When the fold test is used as a measure of the resistance to ageing, it is recommended that the percentage retention be calculated from the number of double folds recorded before and after ageing and not the folding endurance (logarithm to the base 10 of the fold number).

c) A statistical test for significance of change in property due to accelerated ageing may be made.

11 Test report

The test report shall include the following information:

a) reference to this part of ISO 5630, i.e. ISO 5360-1:1996;

b) reference to the International Standard, if any, or any other standard test method used to determine the properties of the material;

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

d) the time, temperature and relative humidity of the ageing treatment;

e) the mean value and standard deviation of the test results of the relevant property determinations of the untreated test specimens;

f) the mean value and standard deviation of the test results of the relevant property determinations of the treated test specimens;

g) any other treatment of data agreed upon between vendor and purchaser;

h) any deviation from the relevant International Standards or other standards used, or any circumstances or influences which might have affected the test results;

i) date and place of testing.

Annex A
(informative)

Apparatus for moist heat treatment

This apparatus allows a high degree of control of temperature and relative humidity and is therefore the preferred system.

Two oil baths are required. An immersion heater in each bath, controlled by a relay box and thermoregulator, is used to maintain the temperature within ± 0,1 °C of the required value.

NOTE 7 The required relative humidity is deemed to have been achieved if the temperature can be shown to have been maintained to within ± 0,1 °C.

The oil should be continuously circulated in each bath by means of an immersion-type pump to ensure uniform temperature.

In the first bath, which is maintained at 69,7 °C (the vapour pressure of water at 69,7 °C is 65 % of the vapour pressure at 80 °C), air is saturated with water vapour in tandem fritted glass bubblers. This air is passed through a heated (to prevent condensation) glass or stable plastic tube to an ageing vessel in the second bath maintained at 80 °C. The air should be passed through a coil of glass tubing in the bath held at 80 °C, preferably wrapped around the ageing vessel, in order to ensure the air has reached 80 °C before entering the ageing vessel.

Humidifying vessels of height 250 mm and diameter 60 mm have been found to be satisfactory.

Ageing vessels of height 300 mm and diameter 60 mm with a standard 60/50 taper joint have been found suitable, but vessels of any convenient size may be used.

The system is shown schematically in figure A.1. Figure A.2 shows a typical apparatus.

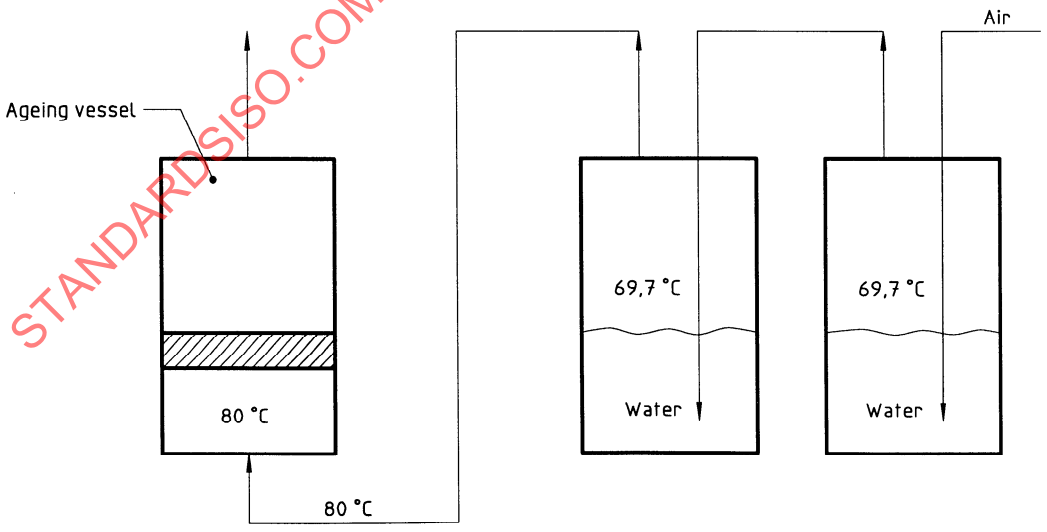

Figure A.1 — Schematic diagram of moist-heat-treatment apparatus

Dimensions (approximate) in millimetres

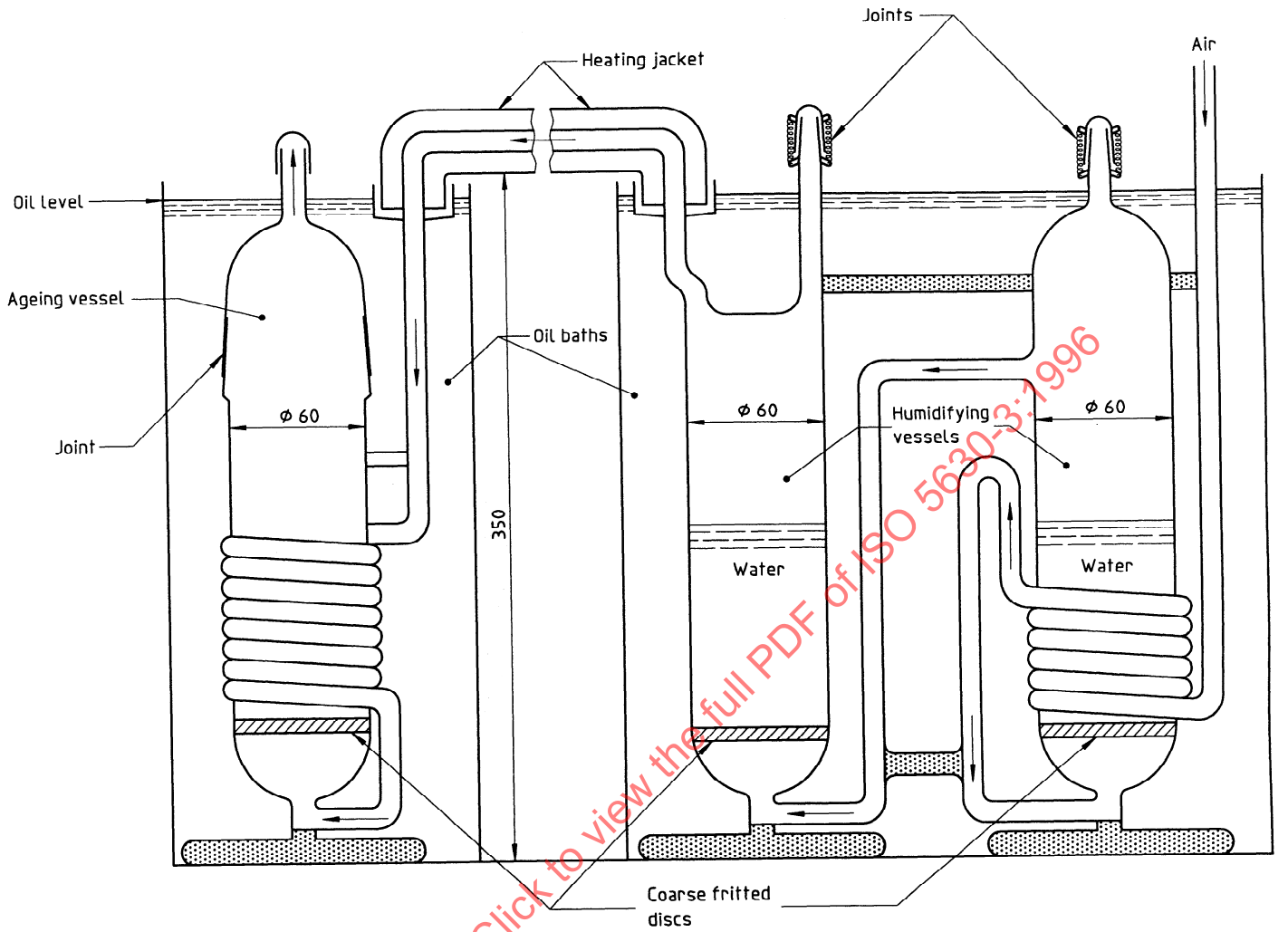

Figure A.2 — Typical apparatus for moist heat treatment