# International Standard ISO 5630/3

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

# 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° et 65 % d'humidité relative*

**First edition — 1986-12-15**



**UDC 676.3/.7 : 620.193.918.2** **Ref. No. ISO 5630/3-1986 (E)**

**Descriptors :** paper, paperboards, tests, aging tests (materials), heat treatment, humidity.



Price based on 5 pages

## 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 5630/3 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps.*



Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.





Printed in Switzerland

# Paper and board — Accelerated ageing — Part 3 : Moist heat treatment at 80 °C and 65 % relative humidity

## 0 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 exposure to dry heat, to heat and moisture, to visible and ultraviolet radiation, and to 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 temperature and relative humidity conditions, 80 °C and 65 % relative humidity has been selected.[5,6]

ISO 5630 consists of the following parts:

Part 1: Dry heat treatment.

Part 2: Moist heat treatment at 90 °C and 25 % relative humidity.

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

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

## 1 Scope and field of application

This part of ISO 5630 specifies a procedure for high moisture heat treatment of paper or board and the general procedure for testing 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 contain any statement about 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 References

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

ISO 187, *Paper and board — Conditioning of samples.*



## 3 Principle

Heating test pieces of paper or board for a specified time at 80 °C and 65 % relative humidity. Comparison of properties of the sample before and after this heat treatment.

## 4 Apparatus

**4.1 Apparatus** for maintaining an atmosphere at a temperature of 80 ± 0,5 °C and (65 ± 1) % relative humidity.

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

NOTE — Graminski *et al.*[3,4] have shown that the rate of degradation of folding endurance and of zero span tensile strength can be varied by changing the relative humidity at elevated temperatures. Therefore, the temperature must be controlled to within 0,5 °C (preferably 0,1 °C) in order to hold the relative humidity variation within 1 %.

**4.2 Test equipment,** appropriate to the 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 pieces

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

Protect the test pieces from strong light.

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

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

## 7 Procedure for heat treatment

Heat treatment shall be carried out in the dark. Without bending or folding, suspend four of the five sets of test pieces (clause 6) in ageing vessels (described in the annex) in the oil bath, or in a climatized cabinet. Pass air at the rate of 50 ± 25 ml/min at 80 ± 0,5 °C and (65 ± 1) % relative humidity through the ageing vessels in the constant-temperature bath.

Remove a set of test pieces after 24 ± 0,25 h, 48 ± 0,5 h, 72 ± 0,75 h and 144 ± 1,5 h.

NOTES

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

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

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

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

## 8 Preconditioning and conditioning

**8.1** On completion of the heat treatment, transfer both the treated and untreated sets of test pieces to a desiccator or other preconditioner maintained at 10 % to 35 % relative humidity and 23 °C. Leave for at least 2 h, and preferably overnight.

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

## 9 Procedure for testing

Test each of the test pieces 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

Record the means and standard deviations of the test data for the aged and unaged test pieces.

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

a) where the units of measurement allow, calculate the retention, expressed as a percentage, of the property, based on the untreated value as 100 %;

NOTE — 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).

Retentions may also be plotted.

b) a statistical test for significance of change in properties due to accelerated ageing should be made.



## 11 Test report



The test report shall include the following information :

a) reference to this part of ISO 5630;

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

The test report shall also include, as specified by the standard method to which the testing procedure conformed, the following information :

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

d) date and place of testing;

e) the time, temperature and relative humidity of testing;

f) the mean value and standard deviation of the test determinations of the appropriate property of the untreated test pieces;

g) the mean value and standard deviation of the test determinations of the appropriate property of the treated test pieces;

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

i) any deviations from the relevant International Standards or other standards used, or any circumstances or influences which might have affected the test results.

## Bibliography

[1] CARDWELL, R.D. Ageing of paper, Doctoral thesis, N.Y. State. College of Forestry, Syracuse, N.Y., 1973.

[2] LUNER, P. Paper permanence, *Tappi* **52** 1969 : 796-805.

[3] GRAMINSKI, E.L., PARKS, E.J. and TOTH, E.E. The effects of temperature and moisture on the accelerated ageing of paper. *ACS Symposium Series* No. 95, *Durability of Macromolecular Materials,* R.K. Eby (Ed.), 1979.

[4] GRAMINSKI, E.L., PARKS, E.J. and TOTH, E.E. The effects of temperature and moisture on the accelerated ageing of paper. NBSIR 78-1443, Report to the National Archives and Records Service. Available from : Springfield, VA, National Technical Information Service (NTIS).

[5] BANSA, H. and HOFER, H.H. Die Aussagekraft einer kunstlichen Alterung von Papier für Prognosen über seine Benutzbarkeit. *Restaurator* **6** (1,2) 1984 : 21-60.

[6] BANSA, H. and HOFER, H.H. Die Beschreibung der Benutzbarkeitsqualität gealterter Papiere in Bibliotheken und Archiven. *Das Papier* **34** (8) 1980 : 348-355.

# Annex

## Oil-baths

This procedure is capable of great precision with respect to temperature and relative humidity.

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.

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.

Humidifier 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 1. Figure 2 shows a typical apparatus.



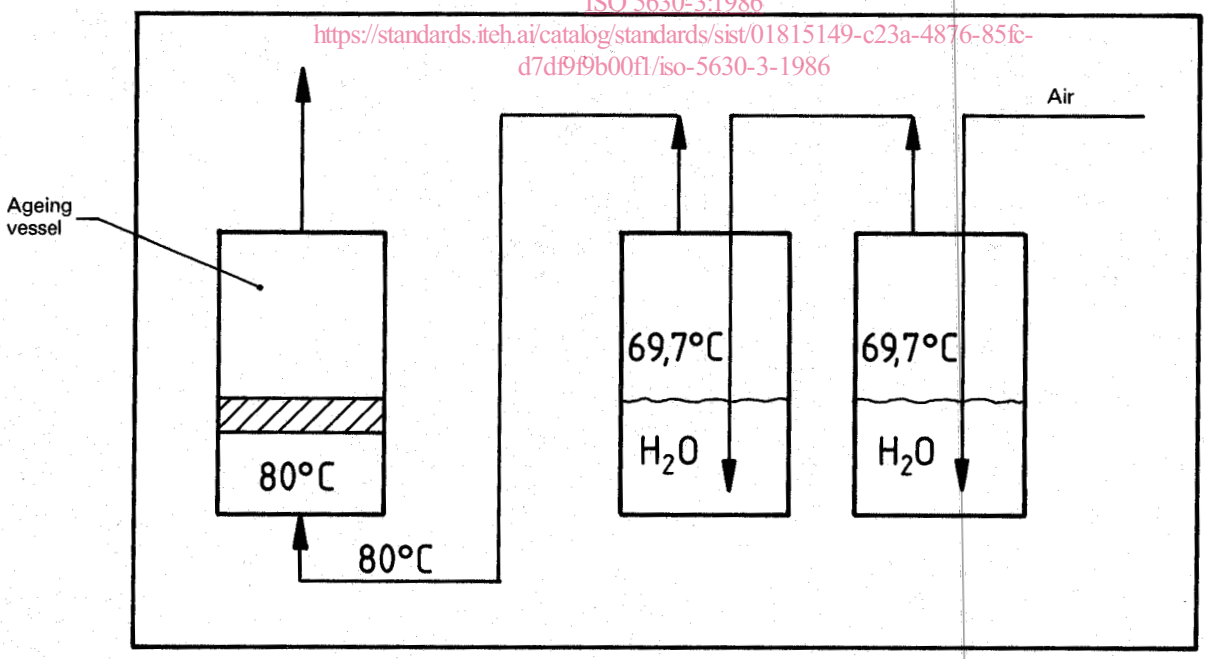


**Figure 1 — Schematic diagram of apparatus**

Dimensions (approximate) in millimetres

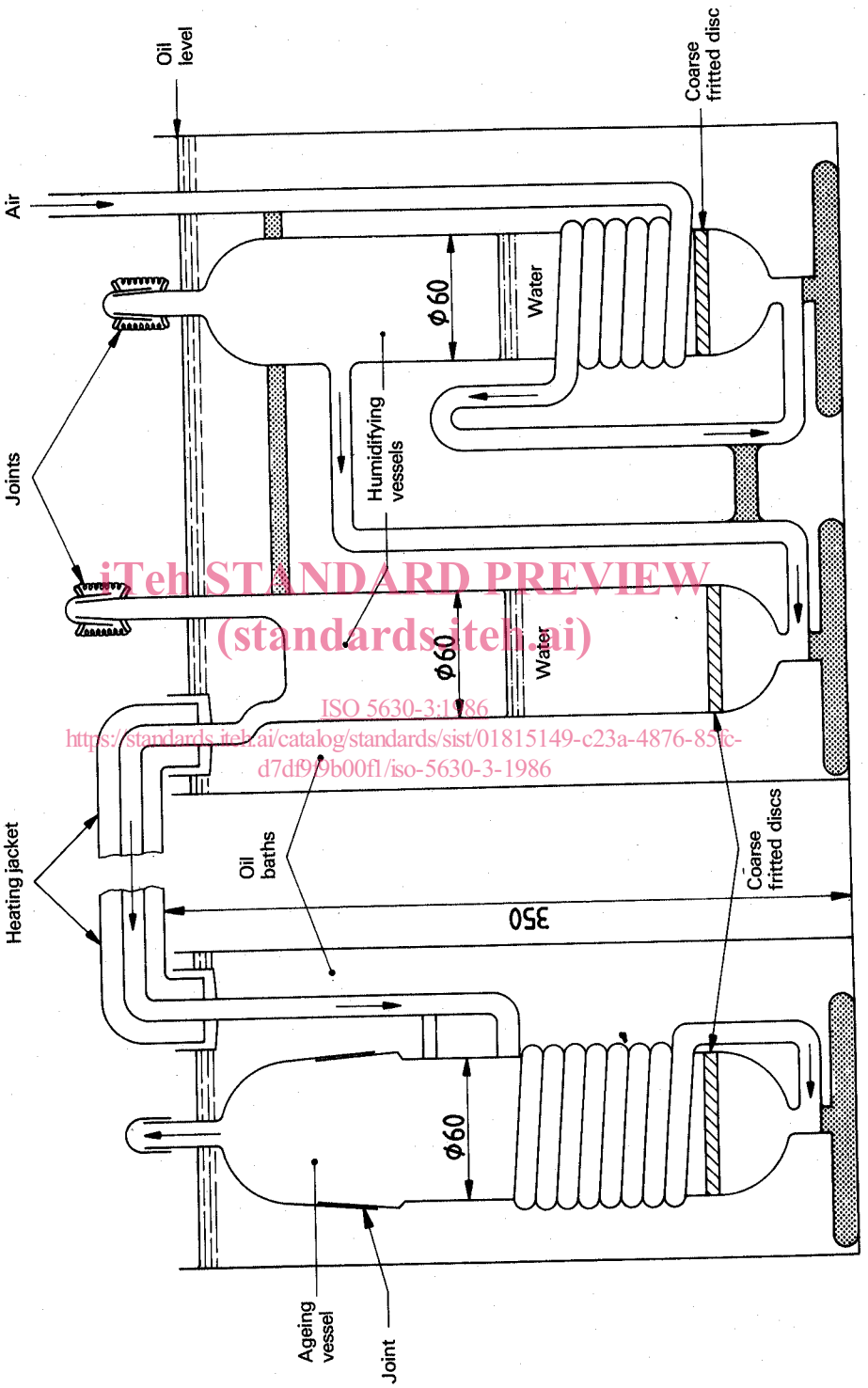




Figure 2 — Typical apparatus