
International Standard



5630/2

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Paper and board — Accelerated ageing — Part 2 : Moist heat treatment at 90 °C and 25 % relative humidity

Papier et carton — Vieillissement accéléré — Partie 2 : Traitement à la chaleur humide à 90 °C et 25 % d'humidité relative

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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.

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 5630/2 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.

Paper and board — Accelerated ageing — Part 2 : Moist heat treatment at 90 °C and 25 % 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] Comparison of accelerated ageing with natural ageing indicates that some moisture should be present in an accelerated ageing atmosphere.^[5,6] Dry accelerated ageing of cellulose is much less sensitive and probably does not rank papers in order of stability as accurately as moist accelerated ageing. It is much simpler to use and may be adequate for many purposes, but moist accelerated ageing should be used where the greatest correlation with natural ageing is needed.

ISO 5630 at present consists of the following parts :

Part 1 : Dry heat treatment

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

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

A method of accelerating the ageing of paper and board at 80 °C and 65 % relative humidity will form the subject of ISO 5630/3.

1 Scope and field of application

This part of ISO 5630 specifies a procedure for humidified 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.

NOTE — ISO 5630/4 is primarily intended for high purity papers such as are used for insulation in electrical equipment.

2 References

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

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

3 Principle

Comparison of the properties of paper or board before and after "accelerated ageing" in a humidified atmosphere at an elevated temperature.

4 Apparatus

4.1 Apparatus, for maintaining an atmosphere at a temperature of $90 \pm 0,2$ °C and $25 \pm 0,5$ % relative humidity.

A suitable apparatus is described in the annex.

A humidity oven may be used if the tolerances for temperature and relative humidity can be met.

NOTE — Graminski *et al.*^[3,4] have shown that the rate of degradation of folding endurance and of zero span tensile strength can be approximately doubled by raising the relative humidity at 90 °C from 20 % to 30 %. Therefore, the temperature must be controlled to less than 0,5 °C (preferably 0,1 °C) in order to hold the relative humidity variations within 1 %.

4.2 Test equipment, complying with the relevant International Standard test method, if any, or with another appropriate standard test method.

Some important test methods that may be used to check the degradation of paper during ageing include pH, brightness, folding endurance and tearing resistance.

Other tests that are very useful for this purpose are hot alkali solubility (1 %), copper number, alkali resistance, zero-span tensile strength, wet strength as a percentage of dry strength, moisture regain, tensile energy absorption, and wet tensile energy absorption.

5 Sampling

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 any other standard method used to determine the properties of the material.

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. Suspend four of the five sets of test pieces (clause 6) in the humidity oven, if used, or the ageing vessels in the oil-bath in such a way that the test pieces do not contact each other or the sides of the ageing vessel. Pass air at the rate of 50 ± 25 ml/min at $90 \pm 0,2$ °C and $25 \pm 0,5$ % relative humidity through the ageing vessels in the constant temperature bath. The rate of flow of air is not critical.

Remove a set of test pieces at $24 \pm 0,25$; $48 \pm 0,5$; $72 \pm 0,75$ and $144 \pm 1,5$ 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 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 from 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.

8 Conditioning

8.1 Before completion of the heat treatment, place the untreated set of test pieces in a desiccator or other preconditioner maintained at 10 % to 35 % relative humidity at 23 °C, and leave for at least 2 h, and preferably overnight.

8.2 On completion of the heat treatment, transfer both the treated and untreated sets of test pieces to the same conditioned enclosure in accordance with ISO 187.

9 Procedure for testing

Test each set of test pieces as described in the relevant International Standard, if any, or by another appropriate standard method.

10 Expression of results

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

- a) plot the data, or the logarithms of the data, as a function of time and calculate the slope — the slopes for various papers can then be compared;
- b) 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.

- c) a test for statistical 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, for each period of treatment;
- h) any other treatment of data agreed upon between vendor and purchaser;
- j) any deviations from the relevant International Standards or other standards used, or any circumstances or influences which might have affected the test results.

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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 $\pm 0,1$ °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 57,3 °C (the vapour pressure of water at 57,3 °C is one-fourth of the vapour pressure at 90 °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 90 °C. The air should be passed through a coil of glass tubing in the bath held at 90 °C, preferably wrapped around the ageing vessel, in order to ensure the air has reached 90 °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.

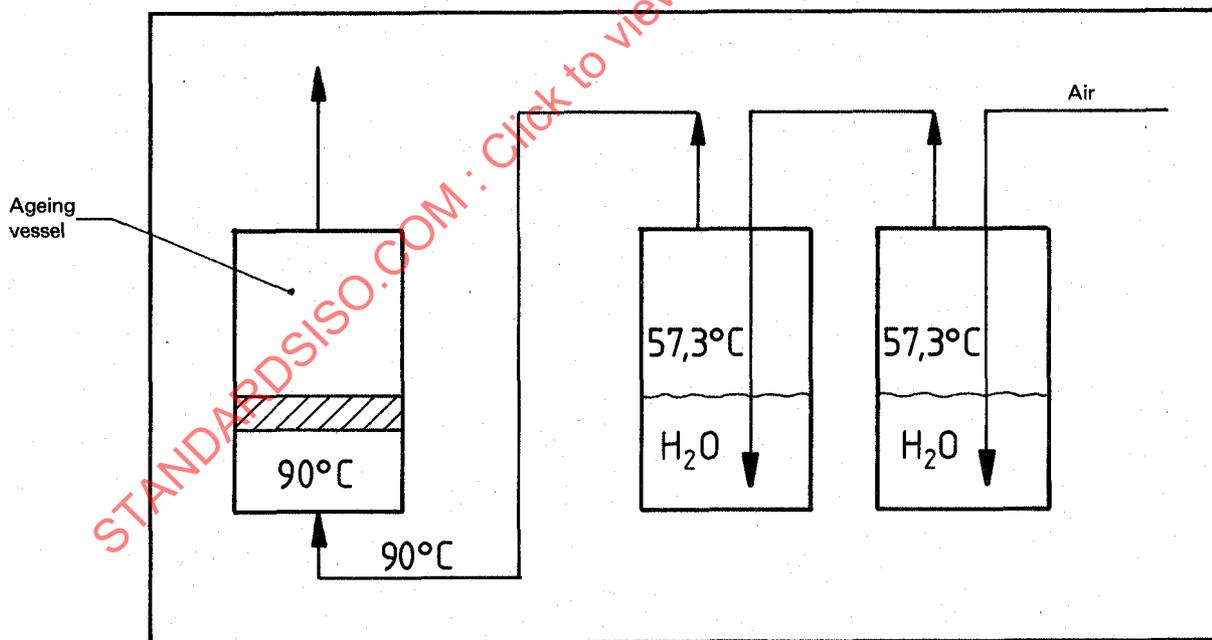


Figure 1 — Schematic diagram of apparatus