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ISO 5630-6

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Paper and board — Accelerated ageing —

Part 6: Exposure to atmospheric pollution (nitrogen dioxide)

Papier et carton — Vieillissement accéléré —

Partie 6: Exposition à la pollution atmosphérique (dioxyde d'azote)

Reference number
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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 5630-6 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)*

NOTE Part 2: *Moist heat treatment at 90 °C and 25 % relative humidity* was withdrawn in 1992.

Introduction

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

This test method for accelerated ageing by exposure of paper to an elevated concentration of nitrogen dioxide is based on the method developed by ASTM [1] following an extensive research program. In this program, 15 printing and writing papers were tested, representing a wide variety of paper types (acid and alkaline, lignin-containing and lignin-free, and those with and without an alkaline reserve such as calcium carbonate). This method proved to be the one most relevant to judge the effects of atmospheric pollutant gases on the long-term mechanical strength and optical stability of such papers. To get a full understanding of the stability of the paper to long-term natural ageing effects, a combination of test methods for accelerated ageing is used.

Paper and board — Accelerated ageing —

Part 6:
Exposure to atmospheric pollution (nitrogen dioxide)

1 Scope

This part of ISO 5630 specifies a method for accelerating the ageing of printing and writing papers through exposure to nitrogen dioxide gas and assessing the effect on mechanical and optical properties for the purpose of predicting stability to long-term natural ageing that occurs due to exposure to polluted air. 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 5626, *Paper — Determination of folding endurance*

ISO 5631-1, *Paper and board — Determination of colour by diffuse reflectance — Part 1: Indoor daylight conditions (C/2°)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
pollutant gas
nitrogen dioxide gas

3.2
polluted air
mixture of dry air and a volume fraction of 0,005 % ± 0,000 2 % of nitrogen dioxide

4 Principle

Sheets of paper are aged at an accelerated rate by exposing them to polluted air comprising an elevated concentration of nitrogen dioxide gas. This air is circulated uniformly around the external surfaces of the paper in a controlled manner and for a specified period of time. The gas reacts chemically with the ingredients of the paper and causes changes in its physical strength and optical properties. By comparing the fold number and the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate before and after ageing, a measure of the stability of paper strength and optical properties is obtained.

5 Reagents

5.1 Dry air, of high quality, purified, for control of the gas concentration in the ageing chamber.

5.2 Nitrogen dioxide, of high quality, pure, obtained from an industrial supplier.

WARNING — Nitrogen dioxide is a highly toxic gas at the concentrations specified in this test method. Follow the manufacturer's safety data sheets for the safety of personnel performing this test.

6 Apparatus

6.1 Gas delivery system, capable of delivering the required mixture (see 3.2) of nitrogen dioxide gas (5.2) and dry air (5.1) to the ageing chamber at the required rate.

Recalibrate the nitrogen dioxide concentration and flow-monitoring instruments with sufficient frequency to ensure continual delivery of the required gas flow.

6.2 Ageing chamber, of a material resistant to corrosive gases, having a volume sufficient to contain the number of individual paper sheets required for testing (10.2) without them touching each other or the chamber walls, with length, depth and width roughly equal, having no windows and allowing a uniform flow of the polluted air to all surfaces of the paper sheets.

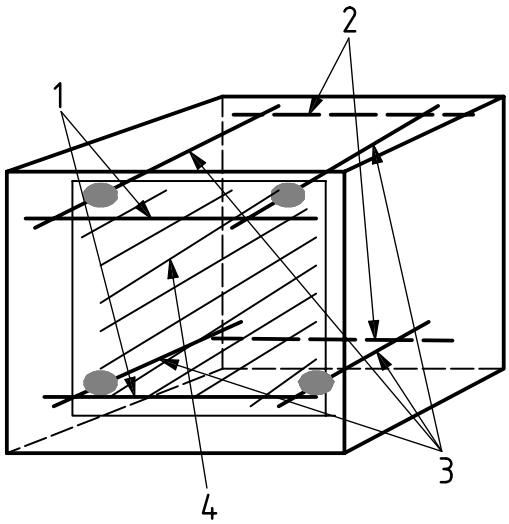
NOTE In the ASTM study, a chamber volume of 0,6 m^3 was found to be adequate for exposing sheets approximately 210 mm × 300 mm (see Reference [1] in the Bibliography).

6.3 Four fixed horizontal hangers and four removable support bars, of a size which fully utilizes the ageing chamber (6.2) whilst preventing the paper from touching the chamber walls, and designed such that the removable bars can be easily moved in and out of the chamber. The support bars are located such that they prevent wrinkling of the paper sheets but do not impose a load on them. Figure 1 shows a paper sheet with holes in all four corners, supported by the removable bars.

6.4 Gas control system, that ensures precise flow of the polluted air (3.2) into the ageing chamber. The design of the apparatus shall be such that the gas concentration entering or already in the ageing chamber can be monitored and recorded with appropriate equipment and instruments.

NOTE A method for monitoring and recording the gas concentration is described in *Paper aging — ASTM's Paper Aging Research Report Program* [1].

6.5 System for controlling relative humidity and temperature, that measures and controls the relative humidity and temperature of the polluted air entering or already in the ageing chamber.



Key

1 front horizontal hangers
2 rear horizontal hangers
3 support bars (removable)
4 paper sheet

Figure 1 — Paper sheet mounted in the ageing chamber

6.6 Exhaust system for polluted air, that permits the gas to be continuously removed. The design should be such that the amount removed each hour is equal to approximately 5 % of the volume in the chamber. This is to ensure that there is no build-up in the chamber of products of degradation emitted from the paper during the period of exposure.

NOTE Safety systems are required in the workspace surrounding the ageing chamber to ensure that any gas that might escape from the system will be thoroughly and quickly removed from the workspace in a manner that is safe and environmentally sound.

7 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. Select sufficient sheets of adequate size for testing the fold number and colour of aged and unaged specimens (10.2).

Randomize the specimens.

8 Conditioning

Immediately prior to the ageing period, condition the specimens in the dark, including those which will not be aged, in accordance with ISO 187.

The specimens that will not be aged shall be kept in the dark in the same standard atmosphere until their properties are tested.

9 Preparation of test specimens for ageing

Carry out the preparation of the test specimens in the same atmosphere as that used for conditioning (Clause 8).

The test specimens shall be of a size suitable for hanging in the ageing chamber (6.2) without touching the chamber walls. If the specimens are too large, reduce them to a suitable size.

Punch holes in all four corners of the test specimens which will be subjected to accelerated ageing, such that when mounted on the support bars no load is imposed on them and they are kept free of wrinkles.

10 Procedure

10.1 Ageing

Insert the support bars through the test specimens and mount the support bars and test specimens on the horizontal hangers in the ageing chamber (6.2). Suspend the test specimens such that no two specimens touch each other and none touches the ageing chamber walls.

Expose the test specimens for 120 h $\pm$ 0,5 h to a flow of polluted air containing 0,005 % $\pm$ 0,000 2 % by volume of nitrogen dioxide (5.2) in dry air (5.1), brought to the same relative humidity and temperature as that used for conditioning (Clause 8). The flow rate shall be such that the volume of polluted air passing through the ageing chamber per hour is equal to approximately 5 % of the volume of the chamber.

Do not insert additional test specimens into the ageing chamber or remove test specimens from it during the period of exposure.

The ageing chamber shall be kept in the same standard atmosphere as used for conditioning (Clause 8) for the whole exposure period.

NOTE During the first hour, approximately, the gas concentration in the ageing chamber will be reaching equilibrium.

10.2 Testing of aged and unaged test specimens

Immediately after ageing, condition the aged test specimens in the dark in the same atmosphere as used for the initial conditioning (Clause 8), according to ISO 187, but omitting preconditioning.

From the aged and unaged test specimens, cut test pieces for testing the fold number and colour. Determine the fold number in the machine direction (MD) in accordance with ISO 5626 and the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate in accordance with ISO 5631-1, of both the aged and unaged test specimens.

11 Calculation

Calculate the MD fold numbers and the mean values of the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate according to the instructions in the relevant International Standard.

Calculate the retention of the fold number, to the nearest percent, as follows:

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

where

R is the fold-number retention, as a percentage;

F_1 is the fold number (MD) before ageing;

F_2 is the fold number (MD) after ageing.

Calculate the change in the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate, to three significant figures, as follows:

$$(\Delta b^*) = \left| b^*_2 - b^*_1 \right| \quad (2)$$

where

(Δb^*) is the change in the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate;

b^*_1 is the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate before ageing;

b^*_2 is the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate after ageing.

12 Test report

The test report shall include the following information:

a) a reference to this part of ISO 5630;

b) a 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 and age the samples;

f) the number of tests carried out, if different from that in the relevant International Standard;

g) the fold numbers and the mean values of the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate of the aged and unaged samples the antilogarithms of the standard deviations of the folding endurance and the standard deviations of the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate of the aged and unaged samples;

h) the fold-number retention and the change in the b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate;

i) any deviations from the International Standards used, and any circumstances and influence which might have affected the test results.

Annex A
(informative)

Interpretation and limitations of ageing tests

A.1 Interpretation of test results

A.1.1 The test method in this part of ISO 5630 is based on the method developed by ASTM [1] following an extensive research program. In this program, 15 papers were tested, representing a wide variety of paper types (acid and alkaline, lignin-containing and lignin-free, and those with and those without an alkaline reserve such as calcium carbonate). The papers were tested when freshly made and after accelerated ageing. The results give guidance about which papers will prove to be stable for long periods of time, as opposed to those that will quickly lose stability. Values for fold-number retention and increase in b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate were discovered that may be utilized by users of this test method to gain a reliable estimate of the long-term stability of test papers in natural ageing experience. (For guidance, refer to pages 23 to 28 of the section titled *Atmospheric Pollutant Aging Test Method Development* as found on the compact disk *Paper aging — ASTM's Paper Aging Research Report Program* [1].)

NOTE A higher fold retention and a lower b^* CIE 1976 ($L^*a^*b^*$) colour space coordinate increase suggest a more stable paper.

A.1.2 It is very important to note that what is stable paper for one user may be unstable for another. Therefore, the limits of acceptability (the points at which a paper is no longer useful for its intended purpose) are to be defined by end-users. It is only with such information in hand that an approximate definition of the long-term stability of the strength and optical properties of a specific paper can be made.

NOTE If all that is desired is legibility of a printed text, paper can become significantly yellowed and still meet the requirements of the end-user.

A.2 Limitations of pollution test

It should be mentioned that natural ageing is 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 protocol is intended to characterize only pollution-induced reactions. In different conditions of 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.

Annex B
(informative)

ASTM permission statement

This part of ISO 5630 is based on ASTM D6833-02e1 (Standard Test Method for Accelerated Pollutant Aging of Printing and Writing Paper by Pollution Chamber Exposure Apparatus), Copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428, USA. Reprinted by permission of ASTM International.

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