



Standard Test Method for Accelerated Pollutant Aging of Printing and Writing Paper by Pollution Chamber Exposure Apparatus¹

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^{e1} NOTE—Reference to a research report was added September 2003.

1. Scope

1.1 This test method describes a laboratory procedure for the exposure of printing and writing paper to common atmospheric pollutant gas at elevated levels of concentration to permit accelerated aging of such paper.

1.2 This test method specifies the sample preparation and conditions of exposure required to obtain information on the relative stability of paper with regard to change in mechanical strength and optical properties brought about by exposure of such paper to common atmospheric pollutant gas.

1.3 This test method provides qualitative results regarding paper stability and does not define the exact life expectancy for a given paper to reach a specified set of strength or optical properties. The limit(s) of acceptability for a specified set of properties must be defined by each end user and will determine the life expectancy of the paper to be tested.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard and Related Products²

D 685 Practice for Conditioning Paper and Paper Products for Testing²

D 1968 Terminology Relating to Paper and Paper Products²

D 2176 Test Method for Folding Endurance of Paper by the M.I.T. Tester²

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process³

2.2 TAPPI Test Method:

T 524 Color of Paper and Paperboard (45°/0° Geometry)⁴

3. Terminology

3.1 *Definitions*—Definitions shall be in accordance with Terminology D 1968. For terms used in this specification which are not provided by Terminology D 1968, see the *Dictionary of Paper*.⁵

4. Summary of Test Method

4.1 In this test method, a specially designed pollution chamber is used to expose sheets of paper that are separately hung in the chamber. Exposure of the paper is to an elevated concentration of nitrogen dioxide gas. The gas 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 in its optical properties. By comparing initial and final levels of these parameters against specified difference criteria, a measure of the stability of paper strength and optical properties is obtained.

5. Significance and Use

5.1 This test method will find use by parties concerned about the influence of common atmospheric pollutant gases on the permanence of the physical strength and optical properties of various printing and writing papers.

5.2 The test will provide manufacturers, paper users and other interested parties with quantified rankings of paper stability that identify papers that are stable, moderately stable and unstable when exposed to common atmospheric pollutant gases over periods of time.

5.3 The stability rankings may be used for definition of the stability of paper to pollutant gas exposure, but will not define specific periods of life expectancy, as the limits of acceptable

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² *Annual Book of ASTM Standards*, Vol 15.09.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from the Technical Association of the Pulp and Paper Industry, P.O. Box 105113, Atlanta, GA 30348.

⁵ Available from TAPPI, 5th ed., 1996.

physical strength and optical properties will be different for various users of a given paper.

6. Apparatus

6.1 Use a test chamber that is designed to provide a very uniform flow of pollutant gas (diluted in standard commercial dry air) over all surfaces of individually suspended sheets of paper. Utilize materials for construction of the components of the chamber exposed to the gas stream that are highly resistant to the corrosive nature of the pollutant gas.

6.2 Provide a control system that ensures very precise flow of the pollutant gas into the chamber. The gas concentration in the pollution chamber shall be monitored and recorded with appropriate equipment and instruments.

6.3 Provide a separate and independent system to measure and control relative humidity in the test chamber by addition of steam.

6.4 Include an exhaust system in the design of the pollution apparatus that permits pollutant gas flow (volume/h) to be continuously removed. The amount to be removed each hour shall be equal to approximately 5 % of the volume in the chamber. This is to ensure that there is no buildup in the chamber of products of degradation emitted from the paper during the period of exposure.

6.5 Utilize a test chamber that will ensure uniform and separated positioning of individual sheets of paper within the chamber. Its design shall be such that all paper surfaces receive a flow of gas that has uniform velocity and concentration over the surface of all sheets.

6.6 Provide safety systems in the workspace surrounding the test chamber to ensure that any gas that may escape from the system will be thoroughly and quickly removed from the workspace in a manner that is safe and environmentally sound.

7. Sampling and Test Specimens

7.1 Select a paper sample according to Practice D 585.

7.2 For the set of test specimens selected, randomize the sheets of paper according to accepted statistical procedure.

8. Calibration

8.1 Provide a system for delivery of gas to the test chamber such that a flow of gas containing 50 ± 2 ppm of nitrogen dioxide in dry air will be continuously delivered to the chamber.

8.2 Recalibrate the instrument with sufficient frequency to ensure continual delivery of the required gas flow.

9. Conditioning

9.1 Condition all test specimens in the dark prior to and following the pollutant-aging test according to Practice D 685.

10. Procedure

10.1 Measure the initial M.I.T. folding endurance and yellowness (b^*) of the paper after conditioning of the paper. Conduct this test just prior to insertion into the test chamber of specimens from the same lot as were initially tested. Measure M.I.T. folding endurance according to Test Method D 2176. Measure yellowness on the top-side of the paper according to the b^* value of the CIE system according to TAPPI T 524.

10.2 Because papers of different basis weight will have more or less initial folding endurance, vary the tension on the specimen so that a target range between 400 and 700 double folds will be obtained from each initial specimen prior to aging. While this range is desirable, specific papers may have initial double fold strengths either above or below this range. An effort should be made to cause the paper to fail with less than 1000 double folds, but even this may not be possible for the strongest papers and is not mandatory. To adjust initial double fold strength may require changing the weight on the plunger of the fold endurance instrument. As specified in Test Method D 2176, use only weights between 500 and 1500 g. Provide the same tension load to each specimen after pollution aging as was applied to that specimen before aging.

10.3 Select a set of specimens for aging that is sufficiently large to ensure 95 % confidence that the test results represent the population of paper being surveyed and that shall be in accordance with Practice E 122.

10.4 Suspend specimens from the four corners of the test chamber and thread them through rods provided for their suspension.

10.5 Expose all specimens to 50 ppm of nitrogen dioxide in dry air as controlled in the gas input stream for 120 ± 0.5 h. Do not insert additional specimens or remove specimens from the chamber during the period of exposure.

10.6 Maintain the internal space of the test chamber at 23°C and 50 % relative humidity during the conduct of the procedure using control methods as specified in Practice D 685.

10.7 Immediately upon removal from the test chamber, condition the paper specimens in the dark for 24 h according to Practice D 685.

10.8 Immediately upon removal from the conditioning process, once again measure the fold endurance and yellowness of the specimens.

10.9 Measure resistance to fold strength loss (M.I.T. fold retention) and resistance to yellowing (Δb^*) according to Test Method D 2176 and TAPPI T 524 respectively.

11. Calculation and Interpretation of Results

11.1 Calculate the percentage change in fold endurance according to the following formula:

$$\% \text{ Change} = \frac{F_i - F_f}{F_i} \times 100 \quad (1)$$

where:

F_i = initial fold endurance, and

F_f = final fold endurance.

11.2 Calculate the absolute change in yellowness according to the following formula:

$$\text{Change in yellowness, } \Delta b^* = |b_f^* - b_i^*| \quad (2)$$

where:

b_f^* = final yellowness, and

b_i^* = initial yellowness.

11.3 With regard to loss of fold endurance, the following classes are specified:

11.3.1 High strength stability: ≥ 50 % M.I.T. Fold Endurance retention.

11.3.2 Low strength stability: <50 % M.I.T. Fold Endurance retention.

11.4 With regard to change of yellowness, the following classes are specified:

11.4.1 High optical stability: ≤ 0.5 points of absolute b^* increase.

11.4.2 Moderate optical stability: $>0.5 \leq 5.0$ points of absolute b^* increase.

11.4.3 Optically unstable: >5.0 points of absolute b^* increase.

NOTE 1—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, even though the changes in optical properties may position it in the “unstable” category.

12. Report

12.1 Report the percent change in fold endurance and the percent change in yellowness (Δb^*).

12.2 From the percent change values and the classes of stability defined in Section 10, report whether a tested specimen is judged likely to be stable, moderately stable, or unstable in terms of its strength and optical properties when exposed to future natural long-term aging experiences in which common atmospheric pollutant gases are present.

13. Precision and Bias ⁶

13.1 Precision and bias requirements are found in the test methods for M.I.T. fold endurance (Test Method D 2176) and yellowness (TAPPI T 524) measurement.

14. Keywords

14.1 accelerated aging of paper; atmospheric pollutant gas; life expectancy; optical permanence; oxides of nitrogen gas; physical strength permanence; stability of paper

⁶ A research report is available on CD-ROM from ASTM. Request RR:D06-1004.

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL INFORMATION

X1.1 Classes of Stability

X1.1.1 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) must be defined by end-users. It is only with such information in hand that accurate definition of the strength and optical life expectancy of paper can be made.

X1.2 Limitations of Pollutant Test

X1.2.1 It should be mentioned that natural aging is variously the result of the action of heat, light, and chemicals (for

example, pH), including pollutants from the air that become entrained into the paper. This protocol is intended to characterize only pollutant-induced reactions. In different conditions of natural aging, an infinite range of conditions can be found where these elements are differently “mixed.” Therefore, for the greatest understanding of possible future aging effects, the investigator may wish to accelerate paper aging separately by elevated temperature, by elevated light flux, and by increased concentration of common pollutant gases.

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