Designation: D 6819 – 02ε2

An American National Standard

Standard Test Method for Accelerated Temperature Aging of Printing and Writing Paper by Dry Oven Exposure Apparatus[1]

This standard is issued under the fixed designation D 6819; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—In 6.1, "forced-ventilation oven" was editorially changed to "forced-convection oven" in March 2003.
ε[2] NOTE—Reference to a research report was added in September 2003.

1. Scope

1.1 This test method describes a laboratory procedure for accelerating the aging of printing and writing paper within sealed glass tubes through exposure to elevated temperature within an oven.

1.2 This test method applies to all types of printing and writing paper whether it is plain base paper, has internal additives, is coated, is printed or contains any variants of printing and writing paper found in normal usage.

1.3 This test method specifies the sample preparation and conditions of exposure required to obtain information on the stability of paper to aging with regard to changes in mechanical strength properties brought about by exposure of such paper to elevated temperature.

1.4 This test method provides the means to compare the stability of different papers on a relative basis, but does not attempt to project the exact life expectancy for a given paper. Life expectancy depends in large part on the limits of acceptability beyond which various paper properties are no longer useful, as defined by end-users. For a given paper, those limits will be different for different end-users. This test method does not provide a means to measure or to calculate such life expectancy.

1.5 *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 Products[2]

D 644 Test Method for Moisture Content of Paper and Paperboard by Oven Drying[2]

D 685 Practice for Conditioning Paper and Paper Products for Testing[2]

D 689 Test Method for Internal Tearing Resistance of Paper[2]

D 828 Test Method for Tensile Properties of Paper and Paperboard Using Constant-Rate-of-Elongation Apparatus[2]

D 1968 Terminology Relating to Paper and Paper Products[2]

D 2176 Test Method for Folding Endurance of Paper by the M.I.T. Tester[2]

D 5804 Test Methods for Zero-Span Tensile Strength ("Dry Zero-Span Tensile")[2]

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*.[3]

4. Summary of Test Method

4.1 In this test method conditioned samples of printing and writing paper are placed inside airtight glass tubes to retain moisture and air. They are then aged at a constant elevated temperature in a well-controlled laboratory oven for a fixed test period. The aging of paper due to its interaction with moisture and air is accelerated at the elevated temperature. Mechanical strength properties of the paper are measured before and after the aging process. The extent of retention of these properties provides a measure of the physical strength stability of the paper as it relates to its aging.

5. Significance and Use

5.1 This test method will find use by parties concerned with the relative level of physical strength stability over time of various printing and writing papers.

5.2 The test will provide manufacturers, paper users and other interested parties with rankings of paper stability that

[1] This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Test Methods.

Current edition approved Aug. 10, 2002. Published October 2002.

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

[3] Available from the Technical Association of the Pulp and Paper Industry (TAPPI), P.O. Box 105113, Atlanta, GA 30348, 5th ed., 1996.

identify papers that will be stable and those that will be unstable when aged under normal conditions of use and handling over time.

5.3 The stability rankings may be used for definition of the stability of a given printing and writing paper to aging over time when subjected to reasonable conditions of use and handling. These rankings will not define specific periods of life expectancy, as the limit of mechanical strength property acceptability will be different for various users of a given paper.

6. Apparatus

6.1 Use a laboratory forced-convection oven that maintains a uniform temperature of 100 ± 1.0°C.

6.2 Configure the oven so that conditioned paper, enclosed within airtight glass tubes, can be aged in its interior space.

6.3 Include multiple racks within the oven to allow horizontal orientation of glass tubes inside the oven.

6.4 Provide glass tubes of approximately 36.0-cm^3 internal volume per oven-dry gram of paper to be evaluated. Use screw-on caps that are fitted with O-rings or gaskets that ensure a hermetic seal of the interior contents during the elevated temperature aging. Use materials (glass, cap, O-ring, or gasket) that are thermally stable at or preferably well above the aging temperature of 100°C, and also chemically resistant to acid corrosion. Two sources of tubes of approximately 145-cm^3 internal volume have been tested and found to be satisfactory. They are:

6.4.1 *Kontes Hybridization Tubes*, 35-mm inside diameter by 150-mm long (Kontes No. K736500-3515). These glass tubes have polypropylene screw caps with silicone rubber O-rings.

6.4.2 *Lab-Line Hybridization Tubes*, 38-mm outside diameter by 150-mm long (Lab-Line No. 308-9). These tubes have polypropylene screw caps with TFE-fluorocarbon resin gaskets.

7. Calibration

7.1 Provide an oven in which the internal temperature uniformity can be maintained to within ±1.0°C at 100°C.

7.2 Recalibrate the oven with sufficient frequency to ensure continual maintenance of the required temperature.

8. Conditioning

8.1 Condition the paper samples according to Practice D 685 for a minimum of 24 h both immediately prior to and immediately following the accelerated aging period. Omit the preconditioning step of Practice D 685 following the aging period.

9. Procedure

9.1 At all times throughout this test procedure, handle paper samples only with clean cotton gloves. This means that clean cotton gloves are required for handling of the paper both before and following the aging procedure.

9.2 Select paper samples according to Practice D 585.

9.3 For the sample selected, randomize the sheets of paper.

9.4 From the randomized set of paper sheets, select eight individual sheets (equivalent to 8½ by 11-in. sheets) at random for each test unit. Two test units are required for each sample; one for the testing of the unaged paper, and a second for the testing of the aged paper.

9.5 Thoroughly clean the aging tubes prior to insertion of paper. The required cleaning sequence follows:

9.5.1 Wash thoroughly with non-ionic detergent and tap water.

9.5.2 Rinse the tubes thoroughly with tap water to remove all residual soap.

9.5.3 Rinse three more times with distilled water and allow to drain sufficiently.

9.5.4 Dry the tubes in an oven at about 105°C.

9.5.5 Condition the complete tube assembly (opened) for a minimum of 24 h in a room conditioned at the terms specified in Practice D 685.

9.6 If starting with new, previously unused aging tubes, caps, O-rings or gaskets, bake them at 100 to 105°C for at least 24 h in preparation for their first use, following the completion of the previous cleaning steps.

9.7 After conditioning has been completed, cap the tubes and maintain them in that condition until their use in an aging test.

9.8 For each test unit, cut the paper sheets in the dimensions appropriate for the relevant ASTM strength tests that are to be performed following the aging procedure.

9.9 Place conditioned paper in the tubes for aging, but determine the weight required as the ovendry weight of the paper. To insert the correct ovendry weight, determine the moisture content of the conditioned paper according to Test Method D 644. Subtract the weight of moisture as thus determined from the total conditioned weight of the paper to calculate the correct ovendry weight. Insert an amount of conditioned paper that will provide a ratio of 0.0275 ovendry gram of paper for each cubic centimeter of capped aging tube internal volume.

9.10 For the tubes of 145 cm^3 identified in 6.4, insert an amount of conditioned paper equivalent to 4.0 g ± 0.1 ovendry grams per tube. Four tubes are required to age one test unit with fold strips (40) and tear strips (8 to 16, depending on grammage) divided evenly between the four tubes. For tubes of different volume, adjust the number of strips proportionately to maintain the required 0.0275 ovendry grams of paper per cubic centimeter of internal capped tube volume. Ensure that there are at least 40 fold strips and 8 to 16 tear strips (depending on grammage) in each test unit.

9.11 If the required ovendry weight of paper cannot be acquired by adjustment of specimen dimensions (within the allowable tolerances), cut an additional small portion of paper from the same test unit and add it to the tube to provide the required mass per unit volume for the tube size being utilized.

9.12 While still in the conditioned room, insert the conditioned specimens into the aging tubes. For the tear strength test, roll test strips of the paper (two to four, depending on grammage), one atop the other, for proper sample insertion into the internal periphery of the aging tubes. Ensure that the tear test strips are free of folds or creases at the time they are finally in place in the tube. Insert the fold specimens inside the rolled

tear test strips. Put the caps into place and secure (hand-tight) to provide a complete seal.

9.13 Provide a means to identify the test units being tested by making an identification mark on the exterior of each aging tube.

9.14 Prior to aging, raise the oven temperature to 100°C.

9.15 Insert loaded tubes into the heated oven on their sides and place them so as to be only one layer deep on each oven rack.

9.16 Age the test units continuously for 120 ± 0.5 h.

9.17 Upon removal from the oven, return each tube to the room conditioned in accordance with Practice D 685 and allow them to equilibrate unopened to room temperature for a minimum of 1 h. Note any observable change in paper appearance. Observe these changes through the tube without its having been uncapped. If the color of the aged samples appears different in different tubes, the samples with the lighter color may not be airtight.

9.18 Uncap the tubes upon their return to the ambient temperature of the conditioned room and then allow the specimens to equilibrate for at least 24 h.

9.19 Once the paper has reached equilibrium with room conditions, remove the specimens with tweezers, taking care to ensure that no creases or folds are made in the paper.

9.20 Carefully unroll the tear test strips and place them on a flat, clean, inert surface. Cover them with a sheet of glass or poly(methyl methacrylate) (PMMA). Add weight atop the sheet of glass or poly(methyl methacrylate) (PMMA) in order to provide a total weight of approximately 1 kg applied evenly over the full surface of the tear strips. Leave the weight in place for at least 12 h. The uniform, low-pressure application of force on the surface of the tear test strips is required to reduce the curl in the strips at the time of their testing. At the end of the "flattening" period, carefully remove the weight from the tear test strips.

9.21 Perform tests on the test units in accordance with the relevant ASTM test method. Perform these tests at the same time on unaged and aged (according to the procedure described previously) test units of the same paper sample. As a minimum, the tests to be performed must include:

9.21.1 M.I.T. fold endurance in the machine direction (MD) of the paper, and

9.21.2 Tear strength in the cross direction (CD) of the paper.

10. Calculation and Interpretation of Results

10.1 Calculate the results for any given strength test according to instructions included in the relevant ASTM test method.

10.2 Calculate the % retention of the MIT folding endurance as follows:

$$\text{fold retention, \%} = \frac{\text{fold number (MD) aged sample}}{\text{fold number (MD) unaged sample}} \times 100 \quad (1)$$

10.3 Calculate the % retention of the internal tearing resistance as follows:

$$\text{tear retention, \%} = \frac{\text{tearing force (CD) aged sample}}{\text{tearing force (CD) unaged sample}} \times 100 \quad (2)$$

11. Report

11.1 Report paper stability according to the measured % property retention as in Table 1. For a paper to be classified as stable, all of the paper stability determination results must be classified as stable. That is to say if the paper stability determination results for internal tearing resistance are classified as stable but the results for MIT fold endurance are classified as unstable, the paper shall be judged unstable.

NOTE 1—A higher fold and tear strength retention suggests a more stable paper.

12. Precision and Bias [4]

12.1 Precision and bias requirements are found in the test methods for MIT fold endurance (Test Method D 2176) and Internal Tearing Resistance (Test Method D 689).

13. Keywords

13.1 accelerated aging of paper; life expectancy; physical strength permanence; stability of paper

[4] A research report is available on CD-ROM from ASTM. Request RR:D06–1004.

TABLE 1 Paper Stability Determination

	% Property Retention (after 120-h of aging at 100°C)	
Stability Classification	M.I.T. Fold Endurance (MD)	Internal Tearing Resistance (CD)
Stable	≥50	≥85
Unstable	<50	<85

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL INFORMATION

X1.1 Additional Mechanical Strength Testing

X1.1.1 Alternate mechanical strength tests may be performed at the discretion of the testing authority (note: no paper stability determination values have been determined for these alternate tests). They shall be according to the relevant ASTM or TAPPI test method and may include:

X1.1.1.1 Tensile energy absorption (TEA), and

X1.1.1.2 Zero-span tensile strength.

X1.1.2 Tensile energy absorption testing procedure is found in Test Method D 828. Zero-span tensile strength is found in Test Methods D 5804.

X1.2 Paper Composition

X1.2.1 This method applies to cellulose (that is, wood, cotton, etc.) based fibers. Non-cellulose paper may require research to gain assurance that this method is sound for such papers.

X1.3 Limitations of Temperature Test

X1.3.1 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 thermally induced reactions. In different conditions of natural aging, an infinite range of conditions can be found where one or more of 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.

BIBLIOGRAPHY

(**1**) Forsskåhl, I., *Light Aging Test Method Development: ASTM Research Program into the Effect of Aging on Printing and Writing Papers*, KCL, August 2000.

(**2**) Atalla, R, Bond, J., Hunt, C., and Agarwal, U., *Quantification and Prediction of Aging of Printing and Writing Papers Exposed to Light: ASTM Research Program into the Effect of Aging on Printing and Writing Papers*, USDA Forest Service, Forest Products Laboratory, August 2000.

(**3**) Kaminska, E., Bégin, P., Grattan, D., Woods, D., and Bülow, A., *ASTM/ISR Research Program on the Effects of Aging on Printing and Writing Papers: Accelerated Aging Test Method Development*, Canadian Conservation Institute, January 2001.

(**4**) Shahani, C., Lee, S. B., Hengemihle, F. H., Harrison, G., Song, P., Sierra, M. L., Ryan, C. C., and Weberg, N., *Accelerated Aging of Paper: I. Chemical Analysis of Degradation Products; II. Application of Arrhenius Relationship; III. Proposal for a New Accelerated Aging Test: ASTM Research Program into the Effect of Aging on Printing and Writing Papers*, Preservation Research and Testing Division, Library of Congress, February 2001.

(**5**) Reilly, J. M., Zinn, E., and Adelstein, P., *Atmospheric Pollutant Aging Test Method Development: Report to ASTM*, Image Permanence Institute at Rochester Institute of Technology, June 2000.