Designation: D 6789 – 02$^{\epsilon 1}$

An American National Standard

Standard Test Method for Accelerated Light Aging of Printing and Writing Paper by Xenon-Arc Exposure Apparatus[1]

This standard is issued under the fixed designation D 6789; 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—Reference to a research report was added in September 2003.

1. Scope

1.1 This test method describes a laboratory procedure for the exposure of printing and writing paper to xenon-arc light at elevated levels of light flux to permit accelerated aging of that type of 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 optical properties brought about by exposure of such paper to light.

1.3 This test method provides qualitative results regarding paper stability and does not define the life expectancy for a given paper to reach a specified set of optical properties.

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

D 985 Test Method for Brightness of Pulp, Paper, and Paperboard (Directional Reflectance at 457 nm)[2]

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

G 113 Terminology Relating to Natural and Artificial Weathering Tests of Nonmetallic Materials[3]

G 151 Practice for Exposing Nonmetallic Materials in Accelerated Test Devices that Use Laboratory Light Sources[3]

G 155 Practice for Operating Xenon Arc Light Apparatus for Exposure of Nonmetallic Materials[3]

2.2 *TAPPI Test Methods:*

T 254 Cupriethylenediamine Disperse Viscosity of Pulp (Falling Ball Method)[4]

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

T 1206 Precision Statement for Test Methods[4]

3. Terminology

3.1 *Definitions*—Definitions shall be in accordance with Terminology D 1968 or Terminology G 113. For terms used in this specification which are not provided by Terminology D 1968 or Terminology G 113, see the *Dictionary of Paper*.[5]

4. Summary of Test Method

4.1 In this test method, light from a xenon-arc lamp that makes use of filters to simulate natural daylight that has passed through window glass is shone on a paper surface with light flux that is substantially greater than in normal indoor conditions of paper exposure. The light flux is applied in a controlled manner and for a specified period of time. The light flux causes photochemical reactions in the paper that change its reflectance (brightness) and color. By comparing initial and final levels of these parameters against difference criteria, a measure of optical stability is obtained.

5. Significance and Use

5.1 This test method will find use by parties concerned about the relative optical stability 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 light over periods of time.

5.3 The stability rankings may be used for definition of the relative stability of papers to light exposure, but will not define specific periods of life expectancy of a given paper.

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[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.

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

[3] *Annual Book of ASTM Standards*, Vol 14.04.

[4] Available from Technical Association of the Pulp and Paper Industry (TAPPI), P.O. Box 105113, Atlanta, GA 30348; 15 Technology Parkway South, Norcross, GA 30092.

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

6. Apparatus

6.1 Provide a test chamber that utilizes a sealed "long-arc" xenon lamp to illuminate the test samples. The lamp spectrum shall be in accordance with Practice G 155, as per Table 2 of that document.

6.2 Use a glass filtration system in front of the lamp to simulate natural daylight that has passed through window glass. This is to cut off almost all of the very short wavelength light (nominally that which is below 320 nm) as occurs when daylight passes through window glass. Provide the glass filtration system as defined in Practice G 155.

6.3 Provide a cooling system with the instrument such that temperature at the paper surface is maintained at ≥20°C and ≤30°C for all paper types. Air may be used as a cooling medium, but is not mandated so long as relative humidity of about 0.007 kg water/kg of dry air is maintained in the atmosphere above the paper surface and that a supply of oxygen, approximately equivalent to that which is found in standard air, is present at the paper surface. Apart from the oxygen, the remainder of the gas present shall be inert.

6.4 Utilize a test chamber that is designed such that it can be operated so as to ensure that it is free of ozone gas.

7. Calibration

7.1 Control the intensity [irradiance (*E*)] of the xenon arc lamp to 765 ± 75 W/m^2 as measured in the 290 to 800 nm wavelength range.

7.2 Recalibrate the instrument with sufficient frequency to ensure continual preservation of both the light spectrum and the light intensity. For recalibration frequency recommendations, refer to the manufacturer's instructions for the particular instrument in use.

7.3 Arrange the configuration of the test chamber so as to ensure uniformity of light intensity (irradiance) across the paper sample area and in a way that provides ≤10 % deviation from target intensity.

7.4 Check the temperature at the paper surface with sufficient frequency to ensure that it is at ≥20°C and ≤30°C throughout the test. Make these measurements with a properly calibrated optical pyrometer.

8. Conditioning

8.1 Condition all test specimens in the dark prior to and at completion of the light aging exposure in accordance with Practice D 685.

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 Divide the sample equally into two parts. Use one part for exposure in the chamber. Cut a test specimen from this part to the size specified for testing by the test chamber manufacturer. Use the other for optical property tests of the unexposed paper. This is necessary to allow for proper light exposure in the chamber and at the same time to provide enough paper in each part to be cut to the small specimen size required for performance of subsequent standard optical property tests.

9.3 Measure the initial optical properties on both sides of the unexposed paper specimens after conditioning and just prior to insertion in the test chamber. The optical properties to be measured include reflectance (brightness) as found in Test Method D 985 and color according to TAPPI Standard T 524. If test results are different on one side versus the other, report results for each side separately.

9.4 Conduct the test in a temperature and humidity controlled room that is maintained at 23°C and 50 % relative humidity according to Practice D 685.

9.5 Cut test specimens to a size that is the maximum that will fit in the available space provided in the selected test chamber, taking care to ensure that the specimen will be uniformly irradiated over its entire surface.

9.6 Mount the specimens on the appropriate surface of the test chamber with clamps provided with the device. Take care to mount specimens of both sides of the paper for exposure.

9.7 Expose three replicate specimens of each paper to be tested to light from a xenon arc lamp controlled to 765 ± 75 W/m^2 as measured in the 290 to 800 nm wavelength range.

9.8 Expose the specimens for 48 ± 0.5 h. Do not remove the specimens from the chamber during the period of exposure. Remove the specimens from the test chamber at the end of the exposure at the same time of day at which the test was initiated.

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

9.10 Immediately upon removal from the conditioning process, measure the optical properties of the exposed specimens once again, taking care to again measure both sides of the paper sheets. Report any differences that exist between the two sides.

9.11 Measure directional reflectance, *R* (brightness), according to Test Method D 985. Measure yellowness, b^*, according to TAPPI T 524.

10. Calculation and Interpretation of Results

10.1 Calculate the percentage change in reflectance at 457 nm (brightness) according to the following formula:

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

where:
R_i = initial reflectance, and
R_f = final reflectance.

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

$$\text{Change in yellowness, } b^* = \left| b^*_f - b^*_i \right| \quad (2)$$

where:
b^*_f = final yellowness, and
b^*_i = initial yellowness.

10.3 With regard to paper brightness (reflectance at 457 nm) stability, the following classes are specified:

Stable	≤5 % reflectance loss
Moderately Stable	>5 % and ≤20 % reflectance loss
Unstable	>20 % reflectance loss

NOTE 1—Papers in the "Moderately Stable" range may be fully stable for some users. However, if a very high level of optical stability is

required, papers should be selected that meet the "Stable" criteria above.

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

Stable	≤3 points of absolute b^* increase
Moderately Stable	>3 and ≤8 points of absolute b^* increase
Unstable	>8 points of absolute b^* increase

NOTE 2—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.

11. Report

11.1 Report the percent change in reflectance, *R*, (brightness) and the absolute change in yellowness (Δb^*). If there is a difference in these properties between the top and bottom side of the paper, report each separately.

11.2 From the 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 optical properties when exposed to future natural long-term aging experiences in which the paper is exposed to ambient light sources.

11.3 Report the type and manufacturer of the device used for exposure.

11.4 Report the method utilized for cooling the paper surface, the temperature measured at the surface, and the relative humidity of the air above the surface being tested.

11.5 If a much more thorough report of the test is desired, refer to Practice G 151 for a comprehensive list of parameters that may be considered for inclusion in the report.

12. Precision and Bias [6]

12.1 *Precision*—The values of repeatability shown below have been calculated from test results, each of which is the average of three replicate test determinations. The values are based on data obtained at the USDA Forest Products Laboratory in Madison, WI during the research work that led to development of this test method. An array of ten papers was tested. The fiber furnish components of the papers ranged from acid stone groundwood to alkaline cotton fiber. The formation of all papers was somewhat poor and added to the variability of the test results. In general, optically stable papers and those with better formation uniformity have the most repeatable measurements. Those that are poorly formed and are optically unstable have greater variability of measurements and are calculated according to TAPPI T 1206.

12.2 *Repeatability* = 0.39 absolute points of b^* with the range of repeatability for all materials in the study extending from 0.02 to 0.75 absolute points of b^*.

12.3 *Repeatability* = 1.893 percent of *R* (brightness) with the range of repeatability for all materials in the study extending from 0.033 to 4.838 percent of reflectance.

12.4 *Reproducibility* between laboratories awaits a round–robin program to be completed prior to the five-year review of this test method.

12.5 *Bias*—Bias for this procedure cannot be determined because no acceptable standard reference materials are available. Since the measurement of properties is according to standardized test procedures, refer to Test Method D 985 for reflectance (brightness) value bias and to TAPPI T 524 for yellowness information.

13. Keywords

13.1 accelerated light aging; directional reflectance; irradiance; Kubelka-Munk theory; light flux; optical permanence; optical properties; paper stability; photochemical reactions; xenon-arc; yellowness

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

APPENDIX

(Nonmandatory Information)

X1. ADDITIONAL INFORMATION

X1.1 Strength Testing

X1.1.1 Very long-term continuous exposure to natural daylight and to common artificial light has been shown to cause loss of strength in uncoated papers regardless of their fiber composition.

X1.1.2 The most sensitive test by which to measure physical property loss is cellulose degree of polymerization (DP). This method has problems for use in a standard accelerated light aging test. DP can be approximated for lignin-free papers using the well-established CED (cupriethylenediamine) test (TAPPI T 254). However, for lignin-containing papers, a special test that uses a process developed by the Canadian Conservation Institute is required. This procedure calls for partial removal of the lignin with a mild acid chlorite treatment and then uses cadoxen instead of CED. This procedure is required to provide a reliable approximation of loss of DP in lignin-containing papers. The cadoxen method is currently used in only a few laboratories and cannot be considered a standard method.[7]

X1.2 Additional Useful Information

X1.2.1 Post Color Number (PC) change may be useful to track. PC is calculated from Kubelka-Munk theory according to the following equations:

$$k/s = (1 - R_\infty)^2 / 2R_\infty \quad \text{(X1.1)}$$

and

$$PC = 100[(k/s)_{final} - (k/s)_{initial}] \quad \text{(X1.2)}$$

where:

k = absorption coefficient,
s = scattering coefficient, and
R_∞ = reflectance at 457 nm for an "infinitely" thick pad of the material.

NOTE X1.1—The pad must be thick enough so that light does not reach the back surface.

X1.2.2 For calculation of PC Number, the reflectance values at 457 nm are most often used. PC calculations based on reflectance measurements in the reflectance spectrum from 250 to 750 nm wavelength are also valid.

X1.2.3 The absorption coefficient (k) is a linear parameter with respect to chromophore concentration, whereas the reflectance (brightness) is not. To calculate the values of k and s, the basis weight (W) of the paper and the results of two reflectance measurements are used. The measurements are the reflectance of a sheet of paper over a black (zero reflectance) backing of zero reflectance (R_o) and the reflectance of an "infinitely" thick pad of paper sheets (R_∞). If the reflectance measurements are made in the wavelength region 250 to 750 nm, informative absorption coefficient spectra (*k-spectra*) can be obtained which more closely characterize chromophores causing yellowing.

NOTE X1.2—In the calculations of PC and k, R can be measured at 457 nm for an "infinitely" thick pad (R_∞) or alternatively for one sheet backed with a white background (R_w). In both of these calculations, R_o is obtained from the reflectance of one sheet of paper over a black backing. Either calculation is an approximation and involves an element of error because photochemical yellowing is a surface phenomenon and the irradiated sheet is not homogeneously yellowed, which is a requirement for applying the Kubelka-Munk theory. The most accurate way of measurement would be to irradiate thin sheets (10 to 20 g/m^2) and to calculate the absorption coefficient, k, according to Kubelka-Munk theory. The absorption coefficient is a linear parameter with respect to chromophore concentration.

X1.3 Correlation Between Natural and Accelerated Photoaging

X1.3.1 Within the framework of an extensive ASTM-sponsored light aging research program, solar simulator-based (xenon) accelerated photoaging studies were able to reliably rank the relative stabilities of lignin-containing and, for the most part, lignin-free papers in a manner that paralleled photostability in natural environments.[8,9] It is believed therefore that this protocol provides a reliable basis for accelerated assessments of the relative stability of papers when exposed to electromagnetic radiation in the near ultraviolet and visible wavelength regions.

NOTE X1.3—Lignin-containing papers showed a significant loss of brightness after 2.5 years of natural aging in the ASTM research program. In addition, all lignin-free papers showed some loss in brightness after this period of natural aging. Acid kraft paper showed the greatest loss in brightness of the lignin-free papers. With continued exposure, it is likely that additional lignin-free papers will be found to be unstable.

X1.3.2 It should be mentioned that natural aging is variously the result of the action of light, heat, chemicals and pH, including pollutants from the air that become entrained into the paper. This protocol is intended to characterize only photo-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 light flux, by elevated temperature, and by increased concentration of common pollutant gases.

X1.4 Classes of Stability

X1.4.1 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 definitions of the optical stability of paper can be made.

[7] Kaminska, E., "Determination of Degree of Polymerization of Cellulose in Ligneous Papers," *Symposium Proceedings*, Material Research Society, Vol 462, 1997, pp. 45-51.

[8] Atalla, R., Bond, J., Hunt, C., and Agarwal, U., *Quantification and Prediction for 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.

[9] Forsskåhl, I., *Light Aging Test Method Development: ASTM Research Program into the Effect of Aging on Printing and Writing Papers*, KCL, June 2000.

ADDITIONAL REFERENCES

(**1**) 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.

(**2**) 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.

(**3**) 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.