Standard Test Method for Determination of Effect of Moist Heat (50 % Relative Humidity and 90°C) on Properties of Paper and Board[1]

This standard is issued under the fixed designation D 4714; 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.

1. Scope

1.1 This test method covers the procedure for humidified (50 % relative humidity and 90°C) heat treatment of paper or board and the general procedure for testing the heat-treated materials. The purpose is to obtain, by an accelerated test, inferences about the aging qualities of the paper.

1.2 This test method is based on work performed on printing and writing papers but may be used with discretion for other types of papers and boards. This procedure is not intended for use with electrical insulating papers, whose testing is described in Test Methods D 202.

1.3 *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 202 Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation[2]

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Products[2]

D 685 Practice for Conditioning Paper and Paper Products for Testing[3]

D 774 Test Method for Bursting Strength of Paper[3]

D 776 Test Method for Determination of Effect of Dry Heat on Properties of Paper and Board[3]

D 919 Test Method for Copper Number of Paper and Paperboard[3]

D 2176 Test Method for Folding Endurance of Paper by the M.I.T. Tester[3]

2.2 *TAPPI Methods:*

T 212 One percent sodium hydroxide solubility of wood and pulp[4]

T 231 Zero span breaking length of pulp[5]

T 403 Bursting strength of paper[3]

T 414 Internal tearing resistance of paper (Elmendorf-type method)[3]

T 425 Brightness of pulp, paper, and paperboard (Directional reflectance at 457 nm)[3]

T 456 Wet tensile breaking strength of paper and paperboard[3]

T 494 Tensile breaking properties of paper and paperboard (Using constant rate of elongation apparatus)[3]

T 509 Hydrogen ion concentration (pH) of paper extracts—cold extraction method[3]

T 511 Folding endurance of paper (MIT Tester)[3]

3. Summary of Test Method

3.1 Properties of paper or board are compared before and after "accelerated aging" in a humidified atmosphere at an elevated temperature, namely 90°C and 50 % relative humidity.

4. Significance and Use

4.1 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 (*1*) the natural changes that may occur in the material over a period of years and (*2*) the ranking of similar papers with respect to stability.

4.2 Hostile environments that have been used include exposure to heat, to heat and moisture, to visible and ultraviolet radiation, and to sulfur dioxide or ether atmospheric gases.

4.3 Properties compared before and after such exposure may include, but are not limited to, the following:

4.3.1 *Mechanical Properties*—such as folding endurance, bursting strength, tearing resistance, zero-span tensile, and tensile properties (tensile at break, elongation at break and tensile energy absorption),

[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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This test method is related to ISO 5630 – 1986, Paper and Board—Accelerated Aging—Part 3: Moist Heat Treatment, and TAPPI T544 pm-85, Effect of Moist Heat on Properties of Paper and Board.

[2] *Annual Book of ASTM Standards*, Vol 10.01.

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

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

[5] Although there is a TAPPI procedure for zero-span breaking length of pulp (T 231), there is none for paper. Commercial instruments are available for measuring zero-span tensile strength of paper.

4.3.2 *Optical Properties*—such as brightness and opacity, and

4.3.3 *Chemical Properties*—such as pH and alkali solubility.

4.4 For determining the effect of dry oven treatment on paper, see Test Method D 776. It has been determined that the degradation of cellulose is very sensitive to moisture.[6,7] Comparison of accelerated aging with natural aging indicates that some moisture should be present in an accelerated aging atmosphere.[8] Dry accelerated aging of cellulose is much less sensitive and probably does not rank papers in order of stability as accurately as moist accelerated aging. Dry aging is much simpler to use and may be adequate for many purposes, but moist accelerated aging should be used where the greatest correlation with natural aging is needed.

NOTE 1—Earlier editions of this test method have specified 25 % relative humidity at 90°C as this appeared, with very limited data, to correlate better with natural aging. Fifty percent relative humidity at 90°C has been found preferable for three reasons: (*1*) it has become the standard through wide usage, (*2*) paper degrades twice as fast at 50 % relative humidity as at 25 %, and (*3*) humid ovens can maintain 50 % relative humidity at 90°C, but not 25 % at 90°C.

5. Apparatus

5.1 Apparatus for maintaining the temperature at 90°C and the relative humidity at 50 % is required. This may be done through the use of an environmental chamber (humid oven), or by a two-bath system shown in Fig. 1.

5.1.1 Graminski[6,7] has shown that the rate of degradation of folding endurance and of zero span tensile can be approximately doubled by raising the relative humidity at 90°C from 25 to 50 %. Consideration of the required data needed can help guide one in deciding which of the following sets of apparatus is most appropriate for a specific use.

5.2 Environmental chambers are available that can maintain an experimental atmosphere of 90°C and 50 % relative humidity. They have the advantage of convenience, and if their accuracy and precision are carefully monitored, they can be adequate for most purposes, but they cannot control temperature and relative humidity as well as the two-bath system.

5.3 A two-bath system is preferable for research, where it is desirable to control the temperature and relative humidity as closely as possible. The first bath is maintained at 72.8°C and the aging bath at 90°C. The vapor pressure of water at 72.8°C is half the vapor pressure at 90°C, so by saturating air, or other gas, with water vapor at 78.2°C and then passing it through an aging vessel at 90°C, one can easily maintain the relative humidity at 50 % with great accuracy. The temperature of an oil bath can easily be maintained to a precision of 0.1°C.

5.3.1 Purified air from the gas cylinder is passed through tandem fritted glass bubblers, as shown in Fig. 1, through a heated glass or plastic tube (to prevent condensation), to the aging vessel in the second bath. By passing the gas through a coil of glass tubing around the aging vessel, the air attains 90°C before entering the aging chamber.

5.3.2 Approximate dimensions and construction schematic of the humidification vessels and aging vessels are indicated in Fig. 1.

5.3.3 As the vessels are buoyed up by the bath liquid, provision must be made for holding them in place. The preferred approach is to have slots built into the bottom of the aging bath, and then slip the bottoms of the aging vessels into these slots.

5.4 Various test instruments, depending upon the test or tests selected for evaluation, will be required.

6. Sampling

6.1 Select a sample of paper in accordance with Practice D 585 and using any special directions given in the test method(s) used for evaluation.

7. Test Specimens

7.1 Select at random and prepare seven sets of test specimens in accordance with the ASTM test method(s) relevant to the required test(s). Use six of the sets for heat treatment and set one aside as a control.

7.2 Protect the test specimens from strong light.

7.3 Avoid as much as possible handling with bare hands, and avoid undue exposure to the atmosphere of a chemical laboratory.

8. Conditioning

8.1 Precondition the untreated set of test specimens and the aged test specimens, preferably for 24 h, but at least overnight, at 10 to 35 % relative humidity, preferably in circulating air, as described in Practice D 685.

8.2 Transfer the preconditioned specimens to the testing facility maintained at 50 % relative humidity and 23°C as described in Practice D 685. If the transfer requires exposure to non-conditioned atmospheres, the specimens should be enclosed in moisture-resistant envelopes.

[6] Graminski, E. L., Parks, E. J., and Toth, E. E., "The Effects of Temperature and Moisture on the Accelerated Aging of Paper," *ACS Symposium Series No. 95, Durability of Macromolecular Materials*.

[7] Graminski, E. L., Parks, E. J., and Toth, E. E., "The Effects of Temperature and Moisture on the Accelerated Aging of Paper," *NBSIR 78-1443, Report to the National Archives and Records Service*; available from National Technical Information Services (NTIS), Springfield, VA 22151.

[8] Wilson, W. K., and Parks, E. J., "Comparison of Accelerated Aging of Book Papers in 1937 with 36 Years Natural Aging," *Restaurator Vol 4, No. 1*, 1980.

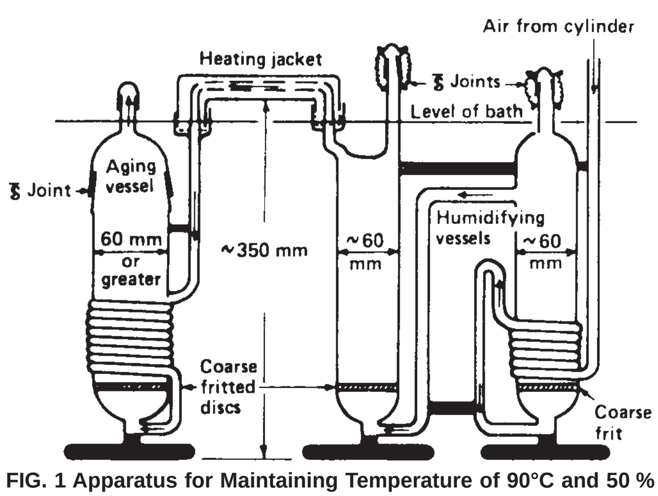

FIG. 1 Apparatus for Maintaining Temperature of 90°C and 50 % Relative Humidity

NOTE 2—Special attention should be given to preconditioning as described in Practice D 685 since the specimens at that time will be considerably out of equilibrium with the test condition.

9. Procedure for Heat Treatment

9.1 *Aging in Environmental Chambers (Humid Ovens)*:

9.1.1 Suspend the sets of test specimens in the test chamber so that the specimens do not touch the walls and are not exposed to direct radiation from the heating coils. Retain one set of test specimens as a control.

9.1.2 Remove one set of test specimens at each of the following times, or as agreed upon: 24 ± 0.25 h, 48 ± 0.5 h, 72 ± 0.75 h, 144 ± 1.5 h, 288 ± 3.0 h, 384 ± 4.0 h, keeping the chamber door open for the shortest possible time.

9.2 *Aging Vessels in Tandem Baths*:

9.2.1 Install the test specimens in aging vessels, placing all samples to be aged for a specific time period in a single vessel. Retain one set of test specimens as a control.

9.2.2 Pass dry air through the aging vessels at a rate of at least 500 mL for at least 15 min.

9.2.3 Switch to air at 50 % relative humidity at a rate of at least 500 mL/min for at least 15 min, and then lower the rate to 50 ± 10 mL/min for the duration of the aging period.

9.2.4 Remove the sets of test specimens at time intervals as described in 9.1.2.

NOTE 3—By agreement between the parties having interest in the data, all or some of the specified times may be used and the data plotted, or the data from only one time obtained and compared with the control.

NOTE 4—The aging vessel or humid oven should contain only one kind of paper at any time in order to prevent the possibility of contamination by distillation or sublimation of paper components or degradation products.

10. Procedure

10.1 Test each set of test specimens as described in the relevant ASTM method or by another appropriate method.

11. Treatment of Data

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

11.1.1 Plot the data, or the log of the data, as a function of time and calculate the slope. The slopes of various papers can then be compared.

11.1.2 Based on the control value as 100 %, calculate the percent retention of the property. Retentions may also be plotted. Retentions of log folding endurance, pH, and air permeability are not applicable.

11.1.3 From a plot of degradation as a function of time, calculate the half-life of the paper.

12. Report

12.1 Include the following particulars in the test report: reference to this test method and reference to the ASTM method, if any, or another method to which the testing procedure conformed.

12.2 Include also in the test report, as specified by the method to which the testing procedure conformed, the following particulars:

12.2.1 Complete identification of the sample.

12.2.2 Date and place of testing.

12.2.3 The time, temperature, and relative humidity of testing.

12.2.4 The mean value and precision of the measured value of the appropriate property of the untreated material.

12.2.5 The mean value and precision of the measured value of the appropriate property of the treated material.

12.2.6 Any other treatment of data agreed upon between the vendor and purchaser.

12.2.7 Any deviations from the relevant ASTM methods or other methods used or any circumstances or influences that might have affected the test results.

13. Precision and Bias

13.1 The precision and bias of the individual test method will be found in the relevant ASTM method.

13.2 The effect of the heat treatment on the precision of the individual test methods is unknown.

14. Keywords

14.1 accelerated aging; aging; moist aging; moist heat; paper; paperboard

APPENDIX

(Nonmandatory Information)

X1. SUMMARY OF WORK ON ACCELERATED AGING

X1.1 Accelerated aging has been carried out at temperatures ranging from below 50°C to more than 150°C, and at relative humidity values from 0 to 100 %. As a rule of thumb, the degradation rate of paper more than doubles for each 10°C rise in temperature. The degradation rate at 50 % relative humidity is about twice that at 25 % relative humidity.

X1.2 Arrhenius discovered an empirical relationship among temperature, reaction rate, and activation energy. If time-degradation plots are developed for several temperatures and relative humidities, the Arrhenius relationship may be used to project beyond the data in hand to predict the number of years a paper will last. This is an interesting academic exercise, but the uncertainty of extrapolation is too great for this approach to be taken very seriously.

X1.3 As the Arrhenius approach requires the development of considerable data to evaluate a few papers, a more practical approach is to select one temperature-relative humidity condition and one aging time. This approach may be used to compare the relative stability of several papers, but it is necessary to have built up a fund of data on the aging of a variety of papers that cover a broad range of stability.

X1.4 Du Plooy[9] studied the degradation of papers at 90, 104, 110, 120 and 135°C, and moisture contents of zero, 0.8, 2.6, 6.7 and 11.9 %. The Arrhenius plots based on these data were straight lines.

X1.5 Graminski,[6] aged paper at 60, 70, 80, and 90°C, and zero, 10, 25, 50, 60, 75, 80, and 90 % relative humidity. Plots of rate of degradation against relative humidity were essentially linear up to 60 % relative humidity, with a slight curvature upwards above 60 %. The rate of degradation at 50 % relative humidity was about double that at 25 %.

X1.6 From data in the literature on the accelerated aging of paper at various temperatures and relative humidities, it appears that the choice of temperature and relative humidity for an accelerated aging procedure is one of convenience. The temperature 90°C and 50 % relative humidity have been selected for the following reasons:

X1.6.1 In order not to upset the usual cellulose-moisture relations, the aging temperature should be below 100°C,

X1.6.2 Below 100°C, the temperature should be as high as conveniently possible in order to speed up the accelerated aging process,

X1.6.3 For convenience, it should be possible to use humid ovens. Humid ovens can be used above *about* 40 % relative humidity, but cannot be used in the 25 % relative humidity range,

X1.6.4 The 90°C–50 % relative humidity condition has been used widely over the past several years.

X1.7 These procedures have been adopted as official standards for the accelerated aging of paper as follows:

Organization and Standard No.	Temperature, °C	R.H.,%
ASTM D 766, TAPPI T 453, ISO 5630/1	105	low
ASTM D 4714, TAPPI T 544[A]	90	50[B]
ISO 5630/3	80	65[C]

[A] Under revision to 50 % R.H.
[B] 90°C and 50 % R.H. is common practice in many U.S. laboratories.
[C] 80°C and 65 % R.H. is common practice in Europe.

[9] Du Plooy, A. B. J., "The Influence of Moisture Content and Temperature on the Aging Rate of Paper," *Appita 34*, No. 4, 1981, p. 287.