Designation: D4714 – 96 (Reapproved 2007)

An American National Standard

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 D4714; 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 D202.

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:*[2]

D202 Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation

D585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product

D685 Practice for Conditioning Paper and Paper Products for Testing

D774/D774M Test Method for Bursting Strength of Paper[3]

D776 Test Method for Determination of Effect of Dry Heat on Properties of Paper and Board[3]

D919 Test Method for Copper Number of Paper and Paperboard[3]

D2176 Test Method for Folding Endurance of Paper by the M.I.T. Tester

2.2 *TAPPI Methods:*[4]

T 212 One Percent Sodium Hydroxide Solubility of Wood and Pulp

T 231 Zero Span Breaking Length of Pulp

T 403 Bursting Strength of Paper

T 414 Internal Tearing Resistance of Paper (Elmendorf-type Method)

T 425 Brightness of Pulp, Paper, and Paperboard (Directional Reflectance at 457 nm)

T 456 Wet Tensile Breaking Strength of Paper and Paperboard

T 494 Tensile Breaking Properties of Paper and Paperboard (Using Constant Rate of Elongation Apparatus)

T 509 Hydrogen Ion Concentration (pH) of Paper Extracts—Cold Extraction Method

T 511 Folding Endurance of Paper (MIT Tester)

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:

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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 Standard Documents Relating to Paper and Paper Products.

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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] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

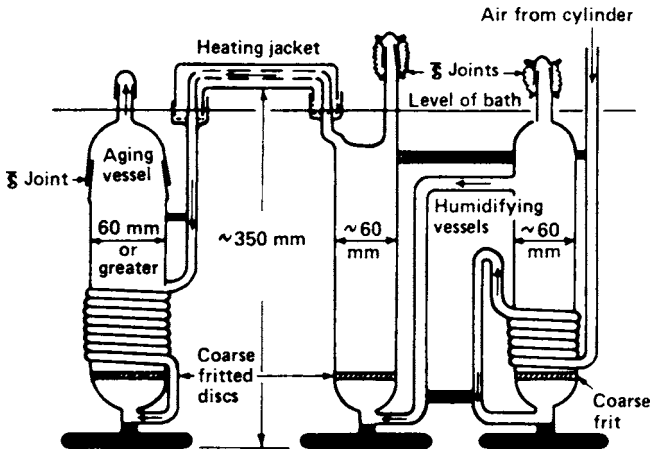

FIG. 1 Apparatus for Maintaining Temperature of 90°C and 50 % Relative Humidity

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),

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 D776. It has been determined that the degradation of cellulose is very sensitive to moisture.[5,6] Comparison of accelerated aging with natural aging indicates that some moisture should be present in an accelerated aging atmosphere.[7] 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[5, 6] 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 D585 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 D685.

[5] 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* .

[6] 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.

[7] 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.