Designation: D 776 – 92 (Reapproved 2001)

An American National Standard

Standard Test Method for Determination of Effect of Dry Heat on Properties of Paper and Board[1]

This standard is issued under the fixed designation D 776; 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 specifies the procedure for dry 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 problems, 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[3]

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

D 828 Test Method for Tensile Breaking Properties of Paper and Paperboard Using Constant-Rate-of-Elongation Apparatus[3]

D 829 Test Methods for Wet Tensile Breaking Strength of Paper and Paper Products[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]

D 4714 Test Method for Determination of Effect of Moist Heat (50 % Relative Humidity and 90°C) on Properties of Paper and Board[3]

E 145 Test Method for Gravity–Convection and Forced-Ventilation Ovens[4]

2.2 *TAPPI Methods:*[5,6]

T 212 One Percent Sodium Hydroxide Solubility of Wood and Pulp

T 423 Folding Endurance of Paper (Schopper Type Tester)

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

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

3. Summary of Test Method

3.1 Properties of paper or board are compared before and after "accelerated aging" in a dry atmosphere at an elevated temperature.

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 the natural changes that may occur in the material over a period of years.

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 gas.

4.3 Properties compared before and after such exposure include mechanical properties, such as folding endurance and tearing resistance, optical properties such as brightness and

[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 TAPPI T 453 pm-85, Effect of Dry Heat on Properties of Paper and Board, and ISO 5630/1-1982, Paper and Board—Accelerated Aging—Part 1: Dry Heat Treatment.

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

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

[4] *Annual Book of ASTM Standards*, Vol 14.02.

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

[6] Other Methods—Zero-Span Tensile Strength: Although there is a TAPPI procedure for zero-span breaking length of pulp (T 231 Zero-Span Breaking Length of Pulp), there is none for paper. Commercial instruments are available for measuring the zero-span tensile strength of paper.

opacity, and chemical properties such as pH and alkali resistance. For additional information, see Test Methods D 828, D 829, D 919, D 2176, TAPPI Methods T 509, T 212, T 423, and T 425.

4.4 For determining the effect of moist heat treatment on paper see Test Method D 4714. It has been determined that the degradation of cellulose is very sensitive to moisture.[7,8] Comparison of accelerated aging with natural aging indicates that some moisture should be present in an accelerated aging atmosphere.[9] 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.

5. Apparatus

5.1 *Oven*, that will maintain a uniform temperature of 105± 2°C where the specimens are located with means of shielding them from direct radiation from the heating element, and otherwise meeting the requirements for Type II forced-ventilation ovens of Grade A as specified in Specification E 145.

5.2 *Test Instruments*, depending on the test, or tests, selected for evaluation.

6. Sampling

6.1 Select a sample of paper in accordance with Methods 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 five sets of test specimens in accordance with the ASTM test method(s) relevant to the required test(s). Use four of the sets for heat treatment and set one as 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. Preparation of Test Specimens

8.1 Suspend the specimens to be heated in the oven so that the air of the oven has free access to all surfaces of each strip, and so that the strips do not touch any part of the oven. Heat at 105 ± 2°C. Keep the oven closed except when removing test specimens and ensure that no other material is in the oven during the heat treatment.

8.2 Remove a set of test specimens at 24 ± 0.25 h, 48 ± 0.5 h, 72 ± 0.7 h, and 144 ± 1.5 h. Longer aging times may be used, if necessary, but additional sets of test specimens will be needed.

NOTE 1—By agreement between vendor and purchaser, all of these specified times may be used and the data plotted, or the data from only one time taken and compared with the control.

NOTE 2—The oven should not contain more than one type of paper at any time, in order to prevent the possibility of contamination by distillation or sublimation of products.

9. Conditioning

9.1 At least 2 h, and preferably overnight, before completion of the heat treatment, place the untreated set of test specimens in a desiccator or other preconditioner maintained at less than 35 % relative humidity at 23°C.

9.2 On completion of the heat treatment, transfer both the treated and untreated sets of test specimens to the same conditioning enclosure (50 ± 2 % relative humidity, 23 ± 1°C) as described in Method D 685.

NOTE 3—Special attention should be given to reconditioning as described in Method D 685 since the specimens at that time will be considerably out of equilibrium with the test condition.

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 vendor and purchaser.

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[7] 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.*

[8] 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.

[9] Wilson, W. K., and Parks, E. J., "Comparison of Accelerated Aging of Book Papers in 1937 with 36 Years Natural Aging," *Restaurator 4:1* (1980).

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

13. Precision and Bias

13.1 The precision and bias of the individual test methods listed in Section 2 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.

13.3 The dry heat aging procedure in Test Method D 776 for measuring the effect of dry heat on the properties of paper and board has no bias value because the effect of dry heat is defined only in terms of the specific conditions described in this test method.

14. Keywords

14.1 accelerated aging; aging qualities; dry heat; paperboard