WI 020808.09

T New (WI3004)

DRAFT NO. 2

DATE May 13, 2003

**TAPPI**

WORKING GROUP CHAIRMAN RB Arnold

SUBJECT CATEGORY Physical Properties

RELATED METHODS See "Additional Information"

**CAUTION:**
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

# Accelerated temperature aging of printing and writing paper by dry oven exposure apparatus (Proposed new Provisional Method)

**(Lines in text or margins indicate changes since last draft)**

## 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 The 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 method does not provide a means to measure or to calculate such life expectancy.

## 2. Summary

2.1 In this 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.

## 3. Significance

3.1 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.

3.2 The test will provide manufacturers, paper users and other interested parties with rankings of paper stability that identify papers that will be stable and those that will be unstable when aged under normal conditions of use and handling over time.

3.3 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.

## 4. Applicable standards

4.1 4.2 TAPPI *Standards*

- T 231 Zero-Span Breaking Strength of Pulp (Dry Zero-Span Tensile)
- T 400 Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product
- T 402 Standard Conditioning and Testing Atmosphere for Paper, Board, Pulp Handsheets and Related Products
- T 412 Moisture in Pulp, Paper and Paperboard
- T 414 Internal Tearing Resistance of Paper (Elmendorf-Type Method)

- T 494 Tensile Breaking Properties of Paper and Paperboard (Using Constant Rate of Elongation Apparatus)
- T 511 Folding Endurance of Paper (MIT Tester)

## 5. Apparatus

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

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

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

5.4 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:

Kontes hybridization tubes, 35-mm I.D. x 150-mm long (Kontes No. K736500-3515). These glass tubes have polypropylene screw caps with silicone rubber O-rings.

Lab-Line hybridization tubes, 38-mm o.d. x 150-mm long (Lab-Line No. 308-9). These tubes have polypropylene screw caps with Teflon fluorocarbon resin gaskets.

Other tubes of similar high quality laboratory glass can be found from other manufacturers.

## 6. Calibration

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

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

## 7. Conditioning

7.1 Condition the paper samples according to TAPPI T 402 for a minimum of 24-hours both immediately prior to and immediately following the accelerated aging period. Omit the preconditioning step of ASTM D685 following the aging period.

## 8. Procedure

8.1 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.

8.2 Select paper samples according to TAPPI T 400.

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

8.4 From the randomized set of paper sheets, select eight individual sheets (equivalent to 8½ x 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.

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

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

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

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

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

8.5.5 Condition the complete tube assembly (opened) for a minimum of 24-hours in a room conditioned at the terms specified in TAPPI T 402.

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

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

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

8.9 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 TAPPI T 412. 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 grams of paper for each cubic centimeter of capped aging tube internal volume.

8.10 For the tubes of 145 $cm^3$ identified above in Section 5.4, insert an amount of conditioned paper equivalent to 4.0 grams ±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.

8.11 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.

8.12 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.

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

8.14 Prior to aging, raise the oven temperature to 100°C ± 1°C.

8.15 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.

8.16 Age the test units continuously for 120 ± 0.5-hours.

8.17 Upon removal from the oven, return each tube to the room conditioned per TAPPI T 402 and allow them to equilibrate unopened to room temperature for a minimum of 1-hour. 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.**

8.18 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-hours.

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

8.20 Carefully unroll the tear test strips and place them on a flat, clean, inert surface. Cover them with a sheet of glass or Plexiglas®. Add weight atop the sheet of glass or Plexiglas® 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-hours. 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.

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

8.21.1 M.I.T. fold endurance in the machine direction (MD) of the paper

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

## 9. Calculation and interpretation of results

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

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

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

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

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

## 10. Report

10.1 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.

**TABLE 1 Paper Stability Determination**

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

**A higher fold and tear strength retention suggests a more stable paper.**

## 11. Precision

11.1. Precision data are found in the TAPPI test methods for MIT fold endurance (TAPPI T 511) and Internal Tearing Resistance (TAPPI T 414).

## 12. Keywords

Paper, Accelerated tests, Aging tests, life tests, physical properties, durability, stability, strength tests.

## 13. Additional information

13.1 Effective date of issue: to be assigned

13.2 *Additional Mechanical Strength Testing*

13.2.1 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:

13.2.2 Tensile energy absorption (TEA)

13.2.3 Zero-span tensile strength

13.2.4 Tensile energy absorption testing procedure is found in TAPPI T 494. Zero-span tensile strength is found in TAPPI T 231.

13.3 Paper Composition

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

13.4 Limitations of Temperature Test

13.4.1 It should be mentioned that natural aging is variously the result of the action of heat, light, and chemicals (e.g. 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.

## Literature cited


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.; 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.; 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.; 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.; Adelstein, P.; Atmospheric Pollutant Aging Test Method Development: Report to *ASTM*; Image Permanence Institute at Rochester Institute of Technology; June 2000

*Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Director of Quality and Standards.* ■