WI 120808.18

T 453

DRAFT NO. 2

DATE April 28, 2013

WORKING GROUP CHAIRMAN Jim Abbott

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.

Effect of dry heat on properties of paper and board (Revision of T 453 sp-08)

(underscores and strikeouts indicate changes from Draft 1)

1. Scope

1.1 This practice ~~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 aging test, inferences regarding the aging qualities of the paper (*1*, *2*).

1.2 The practice ~~method~~ is based on work that has been done with printing and writing papers, but it may be used with discretion on other types of papers and boards.

2. Summary

Selected test properties of paper or board are compared before and after accelerated aging in a forced ventilation oven at 105°C.

3. Significance

3.1 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 or days, 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 long term stability.

3.2 Hostile environments that have been used include exposure to heat, to heat and moisture, to visible and ultraviolet radiation, and to sulfur dioxide, nitrogen dioxide, and ozone gas.

3.3 Properties that may be compared before and after exposure include, but are not limited to, mechanical properties, such as burst, tensile properties, folding endurance and tearing resistance; optical properties, such as brightness, yellowness and opacity; and chemical properties, such as pH and alkali solubility.

3.4 Research suggests that, except for color changes, the results of heating under the prescribed conditions roughly correlate with those of natural aging (*3*, *4*, *5, 6, 7*).

3.5 It has been determined that the degradation rate of cellulose is very sensitive to the moisture content of the sample (*8, 9, 10*).

3.6 TAPPI T 544 "Effect of Moist Heat on Properties of Paper and Board" is available for estimating the effect on paper of aging at 90°C and 50% relative humidity.

3.7 Dry aging of paper is much less sensitive than moist aging, and may not rank papers in order of stability as well as moist aging, but it is much simpler to use. Caution must be exercised in applying either method to a wide variety of paper grades.

4. Apparatus

4.1 *Oven*, forced ventilation, that will maintain a uniform temperature of 105° ± 2°C in the region of the specimens, with means of shielding them from direct radiation from the heating elements (ASTM E 145, Type II, Grade A). Do not place the oven in an area of high humidity as high exterior humidity can affect the results.

5. Test methods

5.1 TAPPI Test Methods: one or more of the following methods may be used for estimating the effect of dry heat treatment on papers.

5.1.1 TAPPI T 212 "One Percent Sodium Hydroxide Solubility of Wood and Pulp."

5.1.2 TAPPI T 400 "Sampling and Accepting A Single Lot of Paper, Paperboard, Containerboard, or Related Product."

5.1.3 TAPPI T 402 "Standard Conditioning and Testing Atmosphere for Paper, Board, Pulp Handsheets, and Related Products."

5.1.4 TAPPI T 550 "Determination of Equilibrium Moisture in Paper and Paperboard for Chemical Analysis."

5.1.5 TAPPI T 414 "Internal Tearing Resistance of Paper (Elmendorf-Type Method)."

5.1.6 TAPPI T 423 "Folding Endurance of Paper (Schopper-Type Tester)."

5.1.7 TAPPI T 430 "Copper Number of Pulp, Paper, and Paperboard."

5.1.8 TAPPI T 452 "Brightness of Pulp, Paper, and Paperboard (Directional Reflectance at 457 nm)."

5.1.9 TAPPI T 456 "Wet Tensile Breaking Strength of Paper and Paperboard ("Wet Tensile Strength")."

5.1.10 TAPPI T 494 "Tensile Properties of Paper and Paperboard (Using Constant Rate of Elongation Apparatus)."

5.1.11 TAPPI T 509 "Hydrogen Ion Concentration (pH) of Paper Extracts (Cold Extraction Method)."

5.1.12 TAPPI T 511 "Folding Endurance of Paper (MIT Tester)."

5.1.13 TAPPI T 553 "Alkalinity of Paper as Calcium Carbonate (Alkaline Reserve of Paper)."

5.1.14 TAPPI T 524 "Color of Paper and Paperboard (45°/0° Geometry)."

5.1.15 TAPPI T 1200 "Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility."

5.2 Other methods.

5.2. 1 *Zero-span tensile strength*: Although there is a TAPPI procedure for zero-span breaking length of pulp (TAPPI 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.

5.3 Some general guidelines.

5.3.1 Always determine pH ~~pH always must be determined~~ for all aging periods.

5.3.2 Tensile energy absorption is especially valuable, for it is an index of the capacity of a paper to hold up during use.

6. Sampling

To determine conformance to product specifications, select a sample of paper according to TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product." Otherwise, obtain a sample appropriate to the reason for testing. Use any special directions given in the specific test method used for evaluation.

7. Test specimens

7.1 Select at random and prepare seven sets, or whatever number is agreed upon, of test specimens in accordance with TAPPI test methods relevant to the required tests.

7.2 Protect the specimens as much as possible from exposure to light.

7.3 Avoid (as much as possible) handling the specimens with the fingers, and avoid undue exposure to the atmosphere of a chemical laboratory.

8. Procedure for heat treatment

8.1 Suspend the sets of test specimens in the oven so that the specimens do not touch the walls of the oven, or are exposed to radiation from the heating coils. Retain one set ~~Make sure one set is retained~~ untreated as a control.

8.2 Remove one set of test specimens at each of the following times (in hours) or as agreed upon: 24 ± 0.25, 48 ± 0.5, 72 ± 0.75, 144 ± 1.5, 288 ± 3, and 384 ± 4.0. Keep the oven door open the shortest time possible when removing specimens.

8.3 Place only one kind of paper in the oven at any time in order to prevent the possibility of contamination by distillation or sublimination of paper components.

NOTE 1: By agreement between vendor and purchaser, 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 2:** The oven should contain only one kind of paper at any time in order to prevent the possibility of contamination by distillation or sublimination of paper components.~~

9. Conditioning for subsequent testing

9.1 Precondition the untreated set of test specimens at least overnight at 23ºC at 10 to 35% relative humidity, preferably in circulating air (TAPPI T 402).

9.2 Transfer the preconditioned specimens and the aged specimens to the testing facility maintained at 23°C and 50% relative humidity (TAPPI T 402). If the transfer requires exposure to non-conditioned atmospheres, the specimens should be enclosed in moisture-resistant envelopes. Condition for 24 hours before testing.

9.3 Recondition as described in TAPPI T 402.

~~**NOTE 3:** Special attention should be given to preconditioning as described in TAPPI T 402.~~

10. Procedure for testing

Test each set of test pieces as described in the relevant TAPPI, or 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.

11.1.3 Based on plots of the degradation of selected properties as a function of time, a half-life of the paper can be calculated. Half-life is that point at which properties have been reduced to half of their original value when the paper was freshly made.

11.4 A test for statistical significance of change in properties due to accelerated aging should be made.

12. Report

12.1 Include the following particulars in the test report: reference to this TAPPI method and reference to the TAPPI 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.

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

13. Precision

13.1 This practice ~~test method~~ does not have a ~~single~~ precision value.

13.2 The precision of the individual test methods will be found in the relevant TAPPI method. Use these individual test method precision statements for relative comparison only, as the effect of heat treatment on precision is unknown.

14. Statistical significance

14.1 When comparing the sample means from test results of two test specimens (in this case, unaged, A, and aged paper, B), it is assumed that the variability in performance of each is unknown but, for first approximation, can be assumed to be about the same (*11*). After the arithmetic means (X_A and X_B) and the standard deviations of test results (s_A and s_B) are calculated, a pooled value s_p for the standard deviation is calculated:

$$s_p = \sqrt{\frac{(n_A - 1)s_A^2 + (n_B - 1)s_B^2}{n_A + n_B - 2}}$$

where n_A = number of test specimens of unaged papers, and n_B = number of test specimens of aged papers.

14.2 Then a value for u, the test criterion for detecting a difference at 95% confidence, may be calculated:

$$u = t\, s_p \sqrt{\frac{n_A + n_B}{n_A n_B}}$$

14.3 The value of t is obtained from a statistical table of t distribution and depends on the number of observations and the 95% confidence level.

14.4 If the difference between X_A and X_B is larger than u, one can conclude, with 95% confidence, that there is a true difference between the performances of A and B.

15. Additional information

15.1 Effective date of issue: to be assigned.

15.2 This method was revised and reclassified as a Standard Practice in 2003. In 2013, several editorial changes were made, and the references to several ASTM standards that are no longer active were deleted. ~~A new ASTM method that uses elevated temperatures to accelerate the aging of printing and writing papers is based on a comprehensive study of paper aging recently completed by ASTM. It is ASTM D6819 "Standard Test Method for Accelerated Temperature Aging of Printing and Writing Paper by Dry Oven Exposure Apparatus." (*10, 11*)~~

~~15.3 This method is practically identical in technical content to ASTM D 776 "Standard Test Method for Determination of Dry Heat on Properties of Paper and Board."~~

15.3 ~~15.4~~ The following procedures have been adopted as official standards for the accelerated aging of paper:

15.5 The aging condition 90°C and 50% relative humidity is widely used.

	Temperature, °C	*R.H., %*
~~ASTM D 776,~~ TAPPI T 453, and ISO 5630-1	105	Low
~~ASTM D 4714,~~ TAPPI T 544	90	50
ISO 5630-3 ~~5630/3~~	80	65

16. Keywords

Aging, Accelerated tests, Paper properties, Heat, Heat treatment, Stability, Paper, Paperboard

Literature cited

1. Rasch, R. H., "Accelerated Aging Test for Paper," *J. Res Natl. Bur. Standards* **7**: 466 (1931).

2. Rasch, R. H., and Stone, C. D., "Estimating Stability of Paper by Heating," *Paper Trade J.* **95** (4): 28 (1932).

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5. Van Royen, A. H. H., "Comparison of the Accelerated Aging of Pulps with Their Normal Aging at Room Temperature," *Papierwereld* **12** (9): 219-225 (1958).

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7. Shahani, C., Lee, S.B., Heggemille, F.H., Harrison, G., Song, P., Sierra, M. L., Ryan, O.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 Effects of Aging on Printing and Writing Papers," Preservation Research and Testing Division, Library of Congress, February 2001.

8. 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*, R. K. Eby, Editor.

9. 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.

10. Du Plooy, A. B. J., "The Influence of Moisture Content and Temperature on Aging of Paper," Appita **34**, No. 4, 287, (1981).

11. Natrella, Mary Gibbons, NBS Handbook No. 91, Experimental Statistics, 1966.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■

WI 120808.18

T 453

DRAFT NO. 1

DATE November 27, 2012

WORKING GROUP CHAIRMAN to be determined

SUBJECT CATEGORY Physical Properties

RELATED METHODS See "Additional Information"

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Effect of dry heat on properties of paper and board (Five-year review of T 453 sp-08)

1. Scope

1.1 This 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 aging test, inferences regarding the aging qualities of the paper (*1*, *2*).

1.2 The method is based on work that has been done with printing and writing papers, but it may be used with discretion on other types of papers and boards.

2. Summary

Selected test properties of paper or board are compared before and after accelerated aging in a forced ventilation oven at 105°C.

3. Significance

3.1 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 or days, 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 long term stability.

3.2 Hostile environments that have been used include exposure to heat, to heat and moisture, to visible and ultraviolet radiation, and to sulfur dioxide, nitrogen dioxide, and ozone gas.

3.3 Properties that may be compared before and after exposure include, but are not limited to, mechanical properties, such as burst, tensile properties, folding endurance and tearing resistance; optical properties, such as brightness, yellowness and opacity; and chemical properties, such as pH and alkali solubility.

3.4 Research suggests that, except for color changes, the results of heating under the prescribed conditions roughly correlate with those of natural aging (*3*, *4*, *5, 6, 7*).

3.5 It has been determined that the degradation rate of cellulose is very sensitive to the moisture content of the sample (*8, 9, 10*).

3.6 TAPPI T 544 "Effect of Moist Heat on Properties of Paper and Board" is available for estimating the effect on paper of aging at 90°C and 50% relative humidity.

3.7 Dry aging of paper is much less sensitive than moist aging, and may not rank papers in order of stability as well as moist aging, but it is much simpler to use. Caution must be exercised in applying either method to a wide variety of paper grades.

4. Apparatus

4.1 *Oven*, forced ventilation, that will maintain a uniform temperature of 105° ± 2°C in the region of the specimens, with means of shielding them from direct radiation from the heating elements (ASTM E 145, Type II, Grade A). Do not place the oven in an area of high humidity as high exterior humidity can affect the results.

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5.1.14 TAPPI T 524 “Color of Paper and Paperboard (45°/0° Geometry).”

5.1.15 TAPPI T 1200 “Interlaboratory Evaluation of Test Methods to Determine TAPPI Repeatability and Reproducibility.”

5.2 Other methods.

5.2. 1 *Zero-span tensile strength*: Although there is a TAPPI procedure for zero-span breaking length of pulp (TAPPI 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.

5.3 Some general guidelines.

5.3.1 pH always must be determined for all aging periods.

5.3.2 Tensile energy absorption is especially valuable, for it is an index of the capacity of a paper to hold up during use.

6. Sampling

To determine conformance to product specifications, select a sample of paper according to TAPPI T 400 “Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product.” Otherwise, obtain a sample appropriate to the reason for testing. Use any special directions given in the specific test method used for evaluation.

7. Test specimens

7.1 Select at random and prepare seven sets, or whatever number is agreed upon, of test specimens in accordance with TAPPI test methods relevant to the required tests.

7.2 Protect the specimens as much as possible from exposure to light.

7.3 Avoid (as much as possible) handling the specimens with the fingers, and avoid undue exposure to the atmosphere of a chemical laboratory.

8. Procedure for heat treatment

8.1 Suspend the sets of test specimens in the oven so that the specimens do not touch the walls of the oven, or are exposed to radiation from the heating coils. Make sure one set is retained untreated as a control.

8.2 Remove one set of test specimens at each of the following times (in hours) or as agreed upon: 24 ± 0.25, 48 ± 0.5, 72 ± 0.75, 144 ± 1.5, 288 ± 3, and 384 ± 4.0. Keep the oven door open the shortest time possible when removing specimens.

NOTE 1: By agreement between vendor and purchaser, 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 2: The oven should contain only one kind of paper at any time in order to prevent the possibility of contamination by distillation or sublimination of paper components.

9. Conditioning for subsequent testing

9.1 Precondition the untreated set of test specimens at least overnight at 23ºC at 10 to 35% relative humidity, preferably in circulating air (TAPPI T 402).

9.2 Transfer the preconditioned specimens and the aged specimens to the testing facility maintained at 23°C and 50% relative humidity (TAPPI T 402). If the transfer requires exposure to non-conditioned atmospheres, the specimens should be enclosed in moisture-resistant envelopes. Condition for 24 hours before testing.

NOTE 3: Special attention should be given to preconditioning as described in TAPPI T 402.

10. Procedure for testing

Test each set of test pieces as described in the relevant TAPPI, or 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.

11.1.3 Based on plots of the degradation of selected properties as a function of time, a half-life of the paper can be calculated. Half-life is that point at which properties have been reduced to half of their original value when the paper was freshly made.

11.4 A test for statistical significance of change in properties due to accelerated aging should be made.

12. Report

12.1 Include the following particulars in the test report: reference to this TAPPI method and reference to the TAPPI 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.

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

13. Precision

13.1 This test method does not have a single precision value.

13.2 The precision of the individual test methods will be found in the relevant TAPPI method. Use these individual test method precision statements for relative comparison only, as the effect of heat treatment on precision is unknown.

14. Statistical significance

14.1 When comparing the sample means from test results of two test specimens (in this case, unaged, A, and aged paper, B), it is assumed that the variability in performance of each is unknown but, for first approximation, can be assumed to be about the same (*11*). After the arithmetic means (X_A and X_B) and the standard deviations of test results

$$s_p = \sqrt{\frac{(n_A - 1)s_A^2 + (n_B - 1)s_B^2}{n_A + n_B - 2}}$$

(s_A and s_B) are calculated, a pooled value s_p for the standard deviation is calculated:

where n_A = number of test specimens of unaged papers, and n_B = number of test specimens of aged papers.

14.2 Then a value for u, the test criterion for detecting a difference at 95% confidence, may be calculated:

$$u = t\, s_p \sqrt{\frac{n_A + n_B}{n_A n_B}}$$

14.3 The value of t is obtained from a statistical table of t distribution and depends on the number of observations and the 95% confidence level.

14.4 If the difference between X_A and X_B is larger than u, one can conclude, with 95% confidence, that there is a true difference between the performances of A and B.

15. Additional information

15.1 Effective date of issue: to be assigned.

15.2 This method was revised and reclassified as a Standard Practice in 2003. A new ASTM method that uses elevated temperatures to accelerate the aging of printing and writing papers is based on a comprehensive study of paper aging recently completed by ASTM. It is ASTM D6819 "Standard Test Method for Accelerated Temperature Aging of Printing and Writing Paper by Dry Oven Exposure Apparatus." (*10, 11*)

15.3 This method is practically identical in technical content to ASTM D 776 "Standard Test Method for Determination of Dry Heat on Properties of Paper and Board."

15.4 The following procedures have been adopted as official standards for the accelerated aging of paper:

	Temperature, °C	*R.H., %*
ASTM D 776, TAPPI T 453, and ISO 5630	105	Low
ASTM D 4714, TAPPI T 544	90	50
ISO 5630/3	80	65

15.5 The aging condition 90°C and 50% relative humidity is widely used.

16. Keywords

Aging, Accelerated tests, Paper properties, Heat, Heat treatment, Stability, Paper, Paperboard

Literature cited

1. Rasch, R. H., "Accelerated Aging Test for Paper," *J. Res Natl. Bur. Standards* **7**: 466 (1931).

2. Rasch, R. H., and Stone, C. D., "Estimating Stability of Paper by Heating," *Paper Trade J.* **95** (4): 28 (1932).

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7. Shahani, C., Lee, S.B., Heggemille, F.H., Harrison, G., Song, P., Sierra, M. L., Ryan, O.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 Effects of Aging on Printing and Writing Papers," Preservation Research and Testing Division, Library of Congress, February 2001.

8. 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*, R. K. Eby, Editor.

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10. Du Plooy, A. B. J., "The Influence of Moisture Content and Temperature on Aging of Paper," Appita **34**, No. 4, 287, (1981).

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Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■