

# TABULATION

STANDARD-SPECIFIC INTEREST GROUP BALLOT  
CLASSICAL METHOD



DATE: 09/04/2019

Please return report to:

TAPPI  
Standards Department  
15 Technology Parkway South  
Peachtree Corners, GA 30092

TO: N/A

DUE DATE: N/A

RE: T 544, WI 190808.01, Draft 1

FAX: +1-770-446-6947  
standards@tappi.org

The results of the Ten-Year Review SSIG ballot of the subject document are as follows:

Ballots returned = 8 (73%)

RECONFIRM as CLASSICAL METHOD 7

WITHDRAW 0

ABSTENTION 1

REVISE and UPGRADE to OFFICIAL METHOD or PROVISIONAL METHOD 0

Classical Methods which receive comments or votes for withdrawal or revision require at least a second ballot to the SSIG after WGC determines direction.

No comments received.

Copies of comments from SSIG ballot are attached.

Please review and return this form with recommendations and dispositions of comments along with your **MARKED UP COPY** for preparation of the next ballot. **DO NOT RE-TYPE.**

RECOMMENDATIONS AND DISPOSITIONS OF COMMENTS:  
(MUST be in BLACK or RED ink for reproduction)

No changes to Draft 1, Standard ready for Fall SARG. Classical Methods do not go to ANSI.

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Continuation on attached sheet(s)-PLEASE DO NOT WRITE ON REVERSE

Your completion of this form constitutes the working group chairman's report. Please send copies of your resolution of comments to those SSIG members who made comments. This form and attachments should be returned to the Standards Department.

WORKING GROUP CHAIRMAN \_\_\_\_\_

Signature

\_\_\_\_\_ Date

**T 544 - Draft 1, due Mon, Mar 11, 2019**

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***Voting Summary by Option***

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<b><i>Option</i></b>	<b><i># Votes</i></b>
Reconfirm as CM	7
Revise and upgrade to OM or PM	0
Withdraw (comment required)	0
Abstention	1
<b>TOTAL VOTES RECEIVED</b>	<b>8</b>

***Voting Statistics***

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<b>Total Members of SSIG</b>	11
<b>Percent return</b>	73%
<b>Votes received</b>	8
<b>Total votes minus abstentions</b>	7
<b>Percent affirmative</b>	100%

***Voting Details***

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<b><i>Voter Name</i></b>	<b><i>Interest Category</i></b>	<b><i>Vote</i></b>
Chawla, Rohit	Supplier - equipment	Reconfirm as Classical Method
Coyle, William	Converter	Reconfirm as Classical Method
Crawshaw, Dennis	Producer	Reconfirm as Classical Method
Guay, Donald	Producer	Reconfirm as Classical Method
Perng, Yuan-Shing	Educator	Reconfirm as Classical Method
Rantanen, Walter	Supplier - service/general	Reconfirm as Classical Method
Witkowski, Terry	General Interest	Reconfirm as Classical Method
Gedrich, Sarah	General Interest	Abstention
Neuman, David	Producer	Did not vote
Swinehart, Dirk	General Interest	Did not vote
Thurman, R. Gary	General Interest	Did not vote

**T 544 - Draft 1, due Mon, Mar 11, 2019**

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***Voter Comments Collected During Ballot Voting***

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**No comments were made on this ballot**

**T 544 - Draft 1, due Mon, Mar 11, 2019**

***Voting Summary by Voter Interest Category***

<i>Interest Category</i>	<i>Reconfirm as CM</i>	<i>Revise and upgrade to OM or PM</i>	<i>Withdraw (comment required)</i>	<i>Abstention</i>	<i>Not Returned</i>			<i>Total</i>
						<i>Qt.</i>	<i>%</i>	
Converter	1	0	0	0	0	1	9.1%	
Educator	1	0	0	0	0	1	9.1%	
General Interest	1	0	0	1	1	4	36.4%	
Producer	2	0	0	0	0	3	27.3%	
Supplier - equipment	1	0	0	0	0	1	9.1%	
Supplier - service/general	1	0	0	0	0	1	9.1%	
Total	7	0	0	1	1	11	100.0%	

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WI \_\_\_\_\_ 190808.01 \_\_\_\_\_

T \_\_\_\_\_ 544 \_\_\_\_\_

DRAFT NO. \_\_\_\_\_ 01 \_\_\_\_\_

DATE \_\_\_\_\_ January 14, 2019 \_\_\_\_\_

WORKING GROUP  
CHAIRMAN \_\_\_\_\_ To be determined \_\_\_\_\_

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.

## **Aging of paper and board with moist heat (Ten-year review of Classical Method T 544 cm-08)**

### **1. Scope**

1.1 This method specifies the procedure for humidified heat treatment of paper or board and the general procedure for testing the heat-treated materials. The purpose is to obtain inferences regarding the long-term aging qualities of the paper.

1.2 The method is based on work done on printing and writing papers but may be used with discretion for other types of papers and boards.

### **2. Summary**

Properties of paper or board are compared before and after "accelerated aging" in a humidified atmosphere at an

elevated temperature (50% RH, 90°C).

**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 usage, 2) paper degrades twice as fast at 50% relative humidity as at 25%, and 3) humid ovens can maintain 50% relative humidity, but not 25%, at 90°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, 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.

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 compared before and after such 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 TAPPI T 453 "Effect of Dry Heat on Properties of Paper" is available for evaluating the effect of dry oven treatment on paper. It has been determined that the degradation rate of cellulose is very sensitive to the amount of moisture in the sample (1,2). Comparison of accelerated aging with natural aging indicates that some moisture should be present in an accelerated aging atmosphere (3). 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. While moist aging is more predictive than dry aging, caution must be exercised in applying either method to a wide range of paper grades and end uses.

### 4. Apparatus

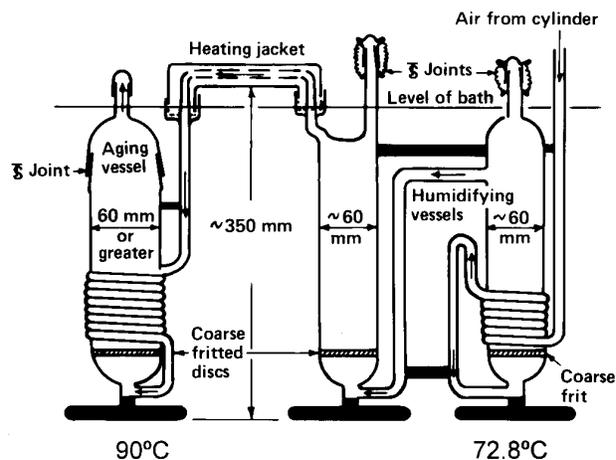
4.1 *A system for maintaining the temperature at 90°C and the relative humidity at 50%* is required. This may be done through the use of an environmental chamber, or by a two-bath system as shown in Fig. 1.

4.2 *Environmental chambers* are available that can maintain an experimental atmosphere at 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.

4.3 *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 of the vapor pressure of water at 90°C, so by saturating air (or other gas) with water vapor at 72.8°C, and 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 should be maintained to  $\pm 0.1^\circ\text{C}$ .

4.3.1 Purified air from a gas cylinder is passed through tandem fritted glass bubblers, as shown in Fig. 1, through a heated (to prevent condensation) glass or plastic tube, 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.

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



**Fig. 1** Apparatus for maintaining temperature of 90°C and 50% RH.

4.3.3 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 bath, and slip the bottom of the vessel into the slots.

4.4 *Test instruments*, depending on the test, or tests, selected for evaluation.

## 5. Test methods

5.1 TAPPI Test Methods: one or more of the following methods may be used for estimating the effect of moist 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 Pulp, 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 Strength of Pulp (Dry Zero-Span Tensile)”], 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 must always 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 test specimens as much as possible from exposure to light.
- 7.3 Avoid (as much as possible) handling test specimens with the fingers, and avoid undue exposure to the atmosphere of a chemical laboratory.

## 8. Procedure for heat treatment

- 8.1 *Aging vessels in tandem baths*

8.1.1 Install the test specimens in aging vessels adjusted for internal temperature to be 90°C - one time period in one aging vessel. Retain one set of test specimens as a control.

8.1.2 Pass dry air through the aging vessels at a rate of about 500-mL per minute for about ten minutes.

8.1.3 Switch to air at 50% relative humidity at a rate of about 500 mL per minute for about ten minutes, and then lower the rate to  $50 \pm 10$  mL per minute for the duration of the aging period.

8.1.4 Remove one set of test specimens at each of the following times (in hours) or as agreed upon:  $24 \pm 0.25$ ,  $48 \pm 0.5$ ,  $72 \pm 0.75$ ,  $144 \pm 1.5$ ,  $288 \pm 3$ , and  $384 \pm 4.0$ .

8.2 *Environmental chambers (temperature and humidity controlled ovens)*

8.2.1 Suspend the sets of test specimens in the test chamber pre-adjusted to an internal RH of 50% at 90°C, so that the specimens do not touch the walls of the oven, or are exposed to radiation from the heating coils.

8.2.2 Remove the sets of test specimens as outlined in 8.1.4, keeping the chamber door open the shortest time possible.

**NOTE 2:** 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 3:** The reaction 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.

## **9. Conditioning for subsequent testing**

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

9.2 Transfer the preconditioned 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.

**NOTE 4:** Special attention should be given to preconditioning as described in TAPPI T 402, as the specimens at that time will be considerably out of equilibrium with the standard test atmosphere.

## **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 to a condition of property failure can be calculated. Half-life is defined here as that point at which a given property has been diminished to one-half of its value when the paper was freshly made. Life expectancy to a given limit of acceptability for various properties will be different for users that specify differing limits of acceptability. Such life expectancy will be approximate at best.

11.1.4 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 (4). 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  $X_A - X_B$  is larger than  $u$ , one can conclude, with 95% confidence, that there is a true difference between the performances of A and B.

## 15. Keywords

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

## 16. Additional information

16.1 Effective date of issue: To be assigned.

16.2 This method was revised and reclassified as a Standard Practice in 2003, and was reclassified as a Classical Method in 2008. A new ASTM method that uses elevated temperatures to accelerate the aging of printing and writing papers is based on a study of paper aging recently completed by ASTM. It is ASTM D 6819 "Standard Test Method for Accelerated Temperature Aging of Printing and Writing Papers by Dry Oven Exposure Apparatus" (7, 8).

16.3 This method now is practically identical to ASTM D 4714 "Standard Test Method for Determination of Moist Heat (50% Relative Humidity and 90°C) on Properties of Paper and Board." This method was originally for 25% relative humidity.

16.4 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	90	50
ISO 5630/3	80	65
ASTM D 6819	100	sealed glass tube

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

## 17. Summary of research on accelerated aging

17.1 Accelerated aging has been carried out at temperatures 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%.

17.2 Arrhenius (5) 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 may last, but only if very long studies are conducted at temperatures significantly below 60°C. This is an interesting academic exercise, and the treatment of the data can yield some useful information. However, the uncertainty of extrapolation is too great for this approach to be taken very seriously.

17.3 As the Arrhenius approach requires the development of considerable data to evaluate a few papers, a more practical (but not as useful) approach is to select one temperature-relative humidity condition, and one aging time (although multiple aging times are much better). This approach may be used to compare the relative stability of several papers. Regardless of the approach, it is necessary to build up a fund of data on several papers covering a wide range of stability.

17.4 Du Plooy (6) 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.

17.5 Graminski (1, 2) 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%.

17.6 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, within limits, is one of convenience. 90°C and 50% relative humidity have been selected for the following reasons.

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

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

17.6.3 For convenience, it should be possible to use environmental chambers (humid ovens). These can be used above *about* 40% relative humidity.

17.6.4 The 90°C -50% relative humidity condition has been widely used.

17.7 Differential thermal analysis (DTA) and thermogravimetric analysis (TGA) (9, 10, 11) have been used to

study the stability of paper. Although the approach appeared to be very promising, not enough data were obtained to completely validate the technique.

## References

1. 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.
2. 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.
3. 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).
4. Natrella, Mary Gibbons, NBS Handbook No. 91, Experimental Statistics, 1966.
5. Arrhenius, S., Uber die Reaktionsgeschwindigkeit bei der Inversion von Rohrzucker durch Sauren, *Z. Physik. Chemic* **4**, 226, (1889).
6. Du Plooy, A. B. J., "The Influence of Moisture Content and Temperature on the Aging of Paper," *Appita* **34** (4): 287 (1981).
7. Kaminska, E., Bégin, P., Grattan, D., and 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.
8. Shahani, C., Lee, S. B., Hengemihle, 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 Method: ASTM Research Program into the Effect of Aging on Printing and Writing Papers," Preservation Research and Testing Division, Library of Congress, February, 2001.
9. Herbert, R. L., Tryon, M. and Wilson, W. K., "Differential Thermal Analysis of Some Papers and Carbohydrate Materials," *Tappi* **52**: 1183 (1969).
10. Parks, E. J., "Thermal Analysis of Modified Cellulose," *Tappi* **54**: 537 (1971).
11. Parks, E. J. and Hebert, R. L., "Thermal Analysis of Ion Exchange Reaction Products of Wood Pulps with Calcium and Aluminum Cations," *Tappi* **55**: 1510 (1972).

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