

WI _____ 060808.07 _____

T _____ 530 _____

DRAFT NO. _____ 2 _____

DATE _____ March 12, 2007 _____

WORKING GROUP

CHAIRMAN _____ Jim Yarborough _____

SUBJECT

CATEGORY _____ Physical Properties _____

RELATED

METHODS _____ See "Additional Information" _____

TAPPI

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.

Size test for paper by ink resistance

(Hercules-type method)

(Revision of T 530 om-02)

(Underscores and strikeouts indicate changes since Draft 1)

1. Introduction

There are many methods for measuring the aqueous resistance of paper and paperboard. The many methods can be placed in categories depending on the way water hold out is measured. One way of classifying tests could be direct versus indirect measurement. Another might be fixed time versus degree of penetration. Ink resistance by the Hercules method is best classified as a direct measurement test for the degree of penetration. Others classify it as a rate of penetration test. There is no one best test for measuring "sizing." Test selection depends on end use and mill control needs. This method is especially suitable for use as a mill control sizing test to accurately detect changes in sizing level.

It offers the sensitivity of the ink float test while providing reproducible results, shorter test times, and automatic end point determination.

2. Scope

2.1 This method (*I-3*) measures the resistance of paper to permeation of an aqueous penetrant and is a useful general purpose test for degree of sizing. It is applicable to most bleached, unbleached, and colored paper or boards which are surface sized and/or internally sized.

2.2 It is not applicable to transparent or translucent papers (e.g., low basis weight papers where ink affects the reflection from the measured surface), colored papers that do not contrast with the green test ink, or papers having as part of their structure an effective water barrier such as polyethylene film. An alternate method should be used for the direct comparison of papers which differ significantly in brightness, color, opacity, basis weight, or filler content, such as may exist among different grades of paper. This limitation does not apply to normal production variations in these properties. It is a good test for comparing different samples of the same grade.

3. Summary

This method employs an aqueous, dark color, dye solution as the penetrant to permit optical detection of the liquid front as it moves through the sheet. The apparatus determines the time required for the reflectance of the sheet surface not in contact with the penetrant to drop to a predetermined percentage of its original reflectance.

4. Significance

4.1 Sizing, as measured by resistance to permeation through or absorption into paper of aqueous liquids, is an important characteristic of many papers. Typical of these are bag, containerboard, butcher's wrap, writing, and some printing grades.

4.2 This method can be used to monitor paper or board production for specific end uses provided acceptable correlation has been established between test values and the paper's end use performance. Due to the nature of the test and the penetrant, it will not necessarily correlate sufficiently to be applicable to all end use requirements. This method measures sizing by rate of penetration. Other methods measure sizing by surface contact, surface penetration, or absorption. Size tests are selected based on the ability to simulate the means of water contact or absorption in end use. This method can also be used to optimize size chemical usage costs.

4.3 This method is closely related to the widely used ink flotation test (Useful Method 481, no longer in print)¹. It has the advantage over the ink flotation test of detecting the endpoint photometrically, thus removing the human judgement factor. It has the disadvantage of being affected by sheet brightness and opacity.

¹ The flotation test uses a 5 cm (2 in.) square sample placed on standard writing ink or other test fluid. The result is reported as the time for a specified degree of color to appear on the upper surface.

4.4 The sensitivity and precision of this method make it attractive for specification purposes provided suitable correlation is established.

5. Applicable documents

Other tests designed to measure the sizing of paper include:

TAPPI T 433, “Water Resistance of Sized Paper and Paperboard (Dry Indicator Method)”

TAPPI T 441, “Water Absorptiveness of Sized Paper and Paperboard (Cobb Test)”

TAPPI T 458, “Surface Wettability of Paper (Angle of Contact Method)”

TAPPI T 491, “Water Immersion Test of Paperboard”

TAPPI T 558, “Surface Wettability and Absorbency of Sheeted Materials Using an Automated Contact Angle Tester”

TAPPI UM 481, “Ink Penetration of Paper” (no longer in print).

TAPPI UM 596, “Water Absorbency of Nonbibulous Paperboard (Water Drop Test)” (No longer published, but is available from TAPPI archives.)

6. Definitions

6.1 For definitions of many terms used in this test method, refer to Terminology ASTM D 1968 or any dictionary of paper industry terms such as TAPPI PRESS “Dictionary of Paper.”

6.2 *Sensitivity*: Sensitivity is the response of the instrument to a standard white ceramic tile. If the bulbs and all glass surfaces of the sizing tester are in good condition, the amount of light reflected by the standard will be high and the meter deflection will be large. Thus, to reduce random error that might be introduced during instrument calibration, be sure that meter deflection is large before proceeding with a sizing test.

6.3 *Linearity*: Overall, linearity reflects the capability of the instrument to accurately and consistently reproduce a given reflectance relationship. Instrument linearity is checked by determining the reflectance of the standard green tile as a percentage of the white tile.

6.4 *Spectral response*: Sensitivity of the device to certain wavelengths of light as measured by an output value.

7. Interferences

There are no known interferences other than colored papers that do not provide an adequate contrast with the green test ink or papers whose structure contain an effective water barrier such as polyethylene film. Based on round robin (see Table 2 in section 19), historical, and empirical data, there seems to be no evidence that calcium carbonate affects the HST test any differently than clay or other fillers. The test appears suitable for use on papers containing

calcium carbonate (4). It is left up to the user to determine the applicability of the test to their own situation, since not all grades of paper have been tested here.

8. Apparatus

8.1 *Single purpose photometer*², meeting the following requirements:

8.1.1 A 45° illumination of the bottom surface of the paper specimen by two halogen incandescent lamps, equidistant from, and 180° apart with respect to the center of the specimen, and powered by a constant voltage source.

8.1.2 A 90° viewing of the specimen by a (measuring) silicon photocell and direct viewing of the lamp filaments by a second (reference) silicon photocell; a means of balancing the two photocell outputs and a means of applying a desired percentage of the output of the reference photocell. The peak spectral response for the silicon photocell is 700 nm.

8.1.3 Effective limitation of heat transfer from the lamps to the specimen by the use of infrared filters and ventilating fans.

8.1.4 Incorporation of an integral timer with manual starting at the beginning of the test and automatic cutoff at the null point.

8.1.5 Permanent working standards, e.g. ceramic tiles, to monitor the ability of the equipment to consistently determine a reflectance ratio.

8.1.6 A holder to position the specimen over the viewing window and to hold ink in contact with the top surface of the specimen during the test. The instrument is constructed with a retaining ring around the viewing window that aligns the holder handle with a line connecting the lamp filaments. The holder has a test area of 16 cm² (2.48 in²) which should give an ink height above the specimen of 6.25 mm (0.25 in.) using the normal 10 mL of ink addition.

8.1.7 A specimen cover, black disc or block, which rests on the top of the sample holder.

8.1.8 A suitable dispenser for the ink that will provide 10 mL of ink repeatedly and quickly to the test specimen at the beginning of the test.

8.2 The following is a list of equipment or supplies used for the method:

Test instrument (covered in section 8.1.1 to 8.1.4)

Ceramic calibration tile (covered in section 8.1.5)

Sample holder (covered in section 8.1.6)

Black disk or block (covered in section 8.1.7)

Ink dispenser (covered in section 8.1.8)

²Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the set of TAPPI Test Methods, or may be available from the TAPPI Quality and Standards Department.

9. Calibration

9.1 Before making measurements, check the calibration of the apparatus using the two ceramic (or porcelain) tiles supplied for this purpose. It is recommended to check the calibration once per day. Calibration values are unique to the set of tiles furnished with each instrument and are not interchangeable between instruments. The tiles can be cleaned with any mild, non-abrasive cleaner and soft cloth.

9.2 Refer to the instrument manual for specific calibration instructions. If the calibration values are outside of recommended tolerances, take the corrective actions indicated in 9.3.

9.3 As the lamp bulbs age, the glass envelopes gradually darken, the light output decreases, and the spectral characteristics change. The effect of these changes due to bulb aging is a gradual decrease in the sensitivity reading and an increase in the linearity reading; therefore, if these values fall outside of the tolerance, consider first whether bulb aging could account for the observed condition. If so, replace the bulbs as the first corrective measure. Failure to obtain acceptable tile readings can also result from improper positioning of the heat filters. If they do not completely intersect the light path from the bulb filaments to the sample area, the optical response will be affected and calibration cannot be achieved. To determine this, carefully view the lamps through the sample opening. If the filaments can be seen without looking through the filters, reposition the filters. Another corrective action is to ensure all surfaces are clean (bulb, filters, glass opening). See the instrument manual for further details on the specific instrument model used.

NOTE 1: Bulb life varies from 3 days to 14 days depending on the instrument. The filament inside of the bulb should be parallel to the inside poles for best life. The bulbs shall be changed anytime the sensitivity or linearity values fall out of specification.

10. Reagents and materials

10.1 *Ink pack.* Ink packs are commercially available ~~have been produced by the manufacturer~~ to eliminate all of the problems associated with make-up and assay of the test solutions. The ink pack consists of two parts: the dye pack and the acid pack. When mixed together in equal volumes, they produce a high-quality, optically correct ink for use with this method.

10.1.1 The dye pack solution is produced to give specific optical properties, not total solids. When mixed accurately, the ink will have an optical density of 0.31 to 0.35 when an aliquot diluted 1000:1 with demineralized water is measured for light absorption at 705 nanometers. In general, dye pack inks suitable for use with this sizing method:

- Must contain a dye that is not substantive to the fiber (i.e., one that does not attach permanently), so that the dye will penetrate the sheet together with the liquid phase.
- Must have precisely controlled composition and chemical properties.
- Should not contain coagulants.
- Should not emit corrosive gases that will attack electrical contacts in the electrical cabinet.
- Should be compatible with the acid or accelerants used in the test solution.

NOTE 2: Most commercial inks do not meet these requirements. However, inks based on naphthol green B dye which also absorb strongly in the 600 - 800 nm spectral range at the peak response of the photocells, do meet these requirements. A consistent-assay (solids and optical performance) naphthol green B dye is no longer commercially available. The supplier of the ink packs has naphthol green B dye custom-manufactured. This dye is supplied in liquid (2.5% solids) form. Optical density and spectral response of all dye packs are quality controlled by the supplier.

10.1.2 Acid packs come in 2% formic acid, (HCOOH), concentration. Formic acid strength is controlled to ± 0.02 percent of target. When combined properly with the dye pack, they form a 1% acid/ink pack. Five (5), 10, 20, and 40 percent acid/ink packs are made by combining customer purchased 10%, 20%, 40%, or 80% formic acid and blending equally with the dye pack. Purchase a grade of acid which specifies the exact percentage concentration, otherwise results will be affected. Store ink solutions in glass or polyethylene containers. Neutral test ink can be prepared by diluting a dye pack aliquot with an equal amount of demineralized water and adding 1 N NaOH solution to pH 7.0 ± 0.1 .

10.1.3 Shelf lives of prepared ink packs at room temperature are:

1% (“#2 Ink”)	6 weeks
5%	4 weeks
10%	2 weeks
20%	1 week
>20%	2 days
Neutral pH ink	1 week

10.2 *Alternative method.* The manufacturer is no longer supplying a dry dye, but for customers still using dry naphthol green B dye, the former method still applies.

10.2.1 *1.0% formic acid ink (#2 ink).* Dissolve 12.5 ± 0.05 g of naphthol green B dye or the corrected weight per the manufacturer’s instructions supplied with the dye. Add formic acid 10.0 ± 0.05 g on a 100% basis. Dilute to 1000 mL and mix thoroughly. Store in glass or polyethylene containers. This ink is stable for six weeks at room temperature after mixing. Discard after six weeks.

10.2.2 *10% formic acid ink.* Dissolve 12.5 ± 0.05 g of naphthol green B dye or the corrected weight per the manufacturer’s instructions supplied with the dye. Add formic acid 100.0 ± 0.05 g on a 100% basis. Dilute to 1000 mL and mix thoroughly. Store in glass or polyethylene containers. This ink is stable for two weeks at room temperature after mixing. Discard after two weeks.

10.2.3 *Higher formic acid inks.* Inks containing 20-60% formic acid can be used for extremely hard-sized and heavy weight paper and board samples. The preparation is the same as outlined for 10% formic acid ink with the suitable adjustment of the amount of acid for the given percent formic acid specification.

10.2.4 *Unacidified (water only) ink.* Some slack sized or lightweight papers may give an endpoint of only a few seconds with the 1% formic acid ink. A neutral ink is then recommended. 12.5 ± 0.05 g of dye is added to distilled water with sufficient NaOH added to adjust the pH to 7.0 ± 0.1 . The maximum shelf life is one week.

10.2.5 *Specialty inks.* If the ink is prepared with a dye other than naphthol green B, the name of the alternate dye should be noted with the results. See Table 1 for example of effects of ink type on HST results.

10.2.6 *Ink analysis.* Determine acidity of an appropriate sample of the ink by potentiometric titration with standard NaOH solution to pH 7. If acidity is outside tolerance limits, it is permissible to adjust it by adding concentrated formic acid or unacidified dye solution. Calculate acidity as follows:

$$\text{Acidity} = (4.6 \times \text{mL of NaOH} \times \text{normality of NaOH}) / \text{mL of ink}$$

$$= \text{g of formic acid} / 100 \text{ mL ink (or aliquot)}$$

Tolerance for 1% formic acid ink: 0.96 - 1.04%

Tolerance for 10% formic acid ink: 9.8 - 10.2%

11. Safety

11.1 **CAUTION:** 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 test method, the user should determine whether any of the chemicals to be used 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.

11.2 Materials used in this method are formic acid and naphthol B green dye. Others may be in use depending on special methods employed. Formic acid is a corrosive liquid and requires caution when handling. Avoid contact of the solution with skin, eyes, and clothing. Avoid breathing its vapor. Use only with adequate ventilation.

12. Sampling and test specimens

12.1 The test units of paper should be selected in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product" and should be conditioned in accordance with TAPPI T 402 "Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products." From each test unit, cut 10 specimens with a minimum dimension of 60 mm (~2.5 in.) in each direction and free of watermarks, creases, and blemishes.

12.2 Identify the top and bottom sides of each specimen and mark accordingly (see TAPPI T 455 "Identification of Wire Side of Paper").

12.3 Identify the machine direction and mark the specimens accordingly. The marking can be conveniently accomplished by cutting rectangular specimens with the long dimension parallel to the machine direction. A convenient specimen size is 70 x 80 mm (2.8 x 3.2 inches).

12.4 Handle specimens by the corners. Do not touch the region that will be in contact with the ink. Finger marks can seriously affect the test and must be avoided in the test area.

13. Conditioning

Condition the test specimens and operate the equipment in a controlled atmosphere in accordance with TAPPI T 402.

14. Procedure

14.1 *General instructions*

14.1.1 Make the test in an atmosphere in accordance with T 402.

14.1.2 Test all specimens with the machine direction parallel to a line connecting the lamp filaments.

14.1.3 Test five specimens with the top side and five with the bottom side to the ink. If specified, the ten tests may be made on the same side. State the % formic acid concentration used for the ink.

14.1.4 Unless otherwise specified, run the test to an 80% reflectance endpoint. If test times are impractically long or short, a higher or lower reflectance endpoint may be used (see 19.2). In all cases, the report must state the endpoint used. The formic acid concentration can also be changed to shorten or lengthen the test as needed.

14.1.5 Maintain the temperature of the test ink supply at $23 \pm 1.0^\circ\text{C}$. Rinse the specimen holder in water at this temperature (and blot dry) to prevent affecting the temperature of the ink in the course of a test. The value of the test can drop 50% for every 7°C rise in ink temperature. Keep the ink away from ventilation slots or other heat sources.

14.2 *Test procedure (Refer to the instrument manual for specific instructions.)*

14.2.1 Turn on the instrument and allow 15 min for warmup.

14.2.2 Set the % reflectance for the desired endpoint. (See Section 19.2 for endpoint considerations.)

14.2.3 Place the paper specimen in the holder (the side that is up will be in contact with the ink), with the machine direction parallel to the handle of the holder. Failure to align the specimen in the machine direction may affect the repeatability of the test results.

14.2.4 Position the holder containing the specimen in the retaining ring on top of the instrument.

14.2.5 Place the cover (black block or disc) over the specimen.

14.2.6 Calibrate the sample to the instrument by setting the initial reflectance to 100%. (Refer to the instrument manual.)

14.2.7 Remove the cover from the holder.

14.2.8 Pour 10 mL of test ink on the specimen and simultaneously start the test timer. The 10 mL of ink is for the specimen holder specified in Section 8.1.6.

14.2.9 Replace the black cover over the specimen. This step is often omitted when judged that the optical density of the ink is sufficient to resist the penetration of outside light. This can be seen by viewing the meter deflection with and without the black cover.

14.2.10 When the % reflectance reaches the setpoint, the test is complete, the timer stops, the signal light goes on, and the buzzer sounds.

14.2.11 Turn off the test timer and silence the buzzer.

14.2.12 Record the test time to the nearest full second. Fractions are not significant.

14.2.13 Remove the holder, discard the ink and specimen, rinse and dry the holder. It is advisable to clean up promptly after completion of the test to avoid specimen rupture and resulting ink spillage.

14.2.14 Repeat 14.2.3 - 14.2.13 for the remaining specimens.

15. Calculations (or interpretations of the observations)

No calculations are necessary.

16. Report

Report the test result to the nearest second as the average test time for the 5 specimens on the top side and the average test time for the 5 specimens on the bottom side, the % reflectance endpoint (e.g. "158 seconds to 80%"), and the % formic acid ink used in the test. If a specialty ink is used, it should be noted. If tests are made on one side of the sheet only, report this fact and the side in contact with the ink.

17. Precision

17.1 The following estimates of precision are based on an interlaboratory study involving 9 materials and 15 laboratories. Repeatability and reproducibility are estimates of the maximum difference (95% confidence) which should be expected when comparing replicate measurements for similar materials under similar test conditions. The samples/statistics are based on the average of 5 tests each on top and bottom, which can increase the variability for some grades, as coated paper or board. The variability is expected to be less when testing only one side. These estimates may not be valid for different materials and testing conditions.

17.1.1 For printing grade papers: (4 grades in the range of 100 - 200 seconds tested with 1% acid/naphthol green ink/ink pack tested to 80% reflectance, including alkaline papers). Material/ink combinations which reached endpoints in less than 20 seconds may have significantly poorer precision.

Repeatability (within a laboratory) = 26%

Reproducibility (between laboratories) = 36%

based on 10 replicates and 15 laboratories

The repeatability and reproducibility values were calculated according to TAPPI T 1200.

17.1.2 See Table 2 for a summary of repeatability and reproducibility for all grades. See the notes below Table 2 regarding bleached board repeatability and reproducibility.

17.2 The precision values reported in the 1989 version of the method were incorrect by the present T 1200 method for reporting precision. In addition, the repeatability value reported in 1989 should have been 20%, not 7% as printed. The new values reported in 17.1.1 are accurate in accordance with T 1200.

17.3 Individuals must determine if these repeatabilities and reproducibilities are satisfactory for their specific purposes.

18. Keywords

Sizing, Sized papers, Paper, Paperboard, Penetration, Ink, Ink absorption, Reflectance.

19. Additional information

19.1 Effective date of issue: to be assigned.

19.2 *Endpoint Selection.* Laboratories or mills testing a wide range of basis weights, or sheets with widely varying levels of sizing, can adjust the reflectance endpoint to complete the test within a reasonable period of time. General recommendations for selecting the endpoint on a given grade of paper are as follows:

1. The reflectance endpoint should be set at a level that will give at least a 30-second test time in order to obtain good reproducibility. Test times of 60 seconds or higher will give maximum precision.
2. Whenever possible, reflectance endpoints in the range of 50 to 80% should be used. Reflectance endpoints of 90% or higher give low meter deflection, and results are affected to a greater extent by paper variations or operator errors. Reflectance endpoints below 40% may give erroneous results. Most papers are almost saturated with ink at this reflectance level. Low reflectance values are sometimes used for papers with low sizing levels if the penetration to the other side is satisfactory.
3. Keeping in mind the above recommendations, a reflectance graph can be run to determine the slope of the time-reflectance curve. From this, select a percent reflectance setting that falls on the straight-line portion of the curve.

19.3 Typical test values on various grades of paper and board are listed in Table 3. Test conditions are usually selected to give a minimum test time of 30 seconds and a maximum test time of about 10 min. High formic acid inks or special inks are required to give reasonable test times on some board grades. For those papers that achieve very low values using this method, the lab should consider modifying the test to increase the results (i.e., lower reflectance, neutral ink). If those modifications are not satisfactory, other test methods should be run that provide values significant enough for control and minimal variation.

19.4 Significant changes in this revision (with no expected effect on the results):

om-96

- Section 17: Updated and corrected precision data.
- Section 7: Suitability of test for use on papers containing calcium carbonate.
- Section 10: Information on Ink Packs.

om-02

- Section 8: Remove circuit diagram.
- Section 14: Simplify procedure and refer user to instrument manual.
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19.5 Factors affecting test times are:

- Ink temperature (see section 14.1.5)
- Calibration (see section 9)
- Broken specimen opening glass
- Ink properties (see section 10 and Table 1)
- Sample preparation (handling, top vs. bottom, etc.)
- Sample conditioning prior to test

Table 1. The effect of ink type on test results of different 65 gsm handsheet systems.

	<i>1% Acid Ink (pH 2)</i>	<i>Neutral Ink (pH 7)</i>	<i>Alkaline Ink (pH 9)</i>	<i>Demineralized Water</i>
0.25% Dispersed Rosin Size, 10% clay	105	151	88	2000+
0.10% AKD Size, 10% PCC	237	394	381	2000+

Table 2. Summary of repeatability and reproducibility statistics for all grades in round robin.

<i>Sample Type</i>	<i>Grand Mean</i>	<i>Repeatability</i>	<i>Reproducibility</i>	<i>Test Conditions</i>
Bleached Bag	7.0	64.2%	70.2%	20% FA Ink
Unbleached Bag	9.1	52.1%	78.2%	20% FA Ink
*20 lb Copy (AKD) White	13.9	33.6%	73.5%	1% FA Ink
Cylinderboard	54.1	34.5%	53.6%	1% FA Ink
*20 lb Copy (ASA) Pink	143.1	27.8%	33.4%	1% FA Ink
60 lb Offset White (acid)	150.9	30.4%	42.2%	1% FA Ink
20 lb Copy White (acid)	156.4	20.9%	38.4%	1% FA Ink
*20 lb Copy (ASA) Blue	184.8	24.8%	30.3%	1% FA Ink
Coated Bleached Board	588.3	35.8%	43.9%	20% FA Ink

* = Contain calcium carbonate filler ~ 15% level.

All "Test Conditions" run at 80% reflectance with the type of ink specified.

FA = Formic Acid; 1% FA = "#2 Ink".

The results for the bleached board sample were the average of 5 top side and 5 bottom side tests. When the data are separated into top side and bottom side the results are:

	Mean	% Repeatability	% Reproducibility
Felt:	652	12.0%	26.2%
Wire:	525	20.8%	41.1%

Table 3. Typical test values for various paper and board grades using the sizing tester.

<i>Paper or Board Sample</i>	<i>Type of Ink Used</i>	<i>Reflectance Endpoint, %</i>	<i>Average Test Time, sec</i>
16-lb bond	1% formic acid ink	80	50
20-lb bond	1% formic acid ink	80	75
20-lb bond pink (alk)	1% formic acid ink	80	143
20-lb bond blue (alk)	1% formic acid ink	80	185
60-lb offset	1% formic acid ink	80	151
40-lb unbleached bag	1% formic acid ink	80	300
18-pt plain chipboard	1% formic acid ink	80	54
150-lb manila file folder	10% formic acid ink	80	250
42-lb unbleached linerboard	10% formic acid ink	80	400
11-pt coated bleached board	10% formic acid ink	85	180
14-pt coated bleached board	40% formic acid ink	70	80
18-pt coated bleached board	40% formic acid ink	70	140
24-pt coated bleached board	40% formic acid ink	70	250
30-pt coated bleached board	40% formic acid ink	70	400

19.6 The values in Table 3 are suggested sample values for selection of an applicable % formic acid ink, and are not target values for a given grade of paper or board. HST values vary approximately to the square of the caliper with everything else held constant.

Literature cited

1. Hammond, J., "The News Penetration Tester," *Paper Trade J.* **103** (21): 37(1936).
2. Instrumentation Study 21A, "The Barss, Knobel, and Young News Penetration Tester," Institute of Paper Chemistry (June 1937).
3. Price, D., Osborn, R. H., and Davis, J. W., "A Liquid Penetration Test for Measuring the Sizing of Paper," *Tappi* **36** (1): 42 (1953).
4. Boone, S., "The Use of T 530 (HST) on Calcium Carbonate Containing Papers," *Tappi* **79** (2): 122 (1996).

Reference

"The Sizing of Paper, 2nd ed," Walter F. Reynolds ed., Chapter 7, TAPPI PRESS (1989).

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

WI _____ 060808.07 _____

T _____ 530 _____

DRAFT NO. _____ 1 _____

DATE _____ March 24, 2006 _____

WORKING GROUP
CHAIRMAN _____ to be assigned _____

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2.2 It is not applicable to transparent or translucent papers (e.g., low basis weight papers where ink affects the reflection from the measured surface), colored papers that do not contrast with the green test ink, or papers having as part of their structure an effective water barrier such as polyethylene film. An alternate method should be used for the direct comparison of papers which differ significantly in brightness, color, opacity, basis weight, or filler content, such as may exist among different grades of paper. This limitation does not apply to normal production variations in these properties. It is a good test for comparing different samples of the same grade.

3. Summary

This method employs an aqueous, dark color, dye solution as the penetrant to permit optical detection of the liquid front as it moves through the sheet. The apparatus determines the time required for the reflectance of the sheet surface not in contact with the penetrant to drop to a predetermined percentage of its original reflectance.

4. Significance

4.1 Sizing, as measured by resistance to permeation through or absorption into paper of aqueous liquids, is an important characteristic of many papers. Typical of these are bag, containerboard, butcher's wrap, writing, and some printing grades.

4.2 This method can be used to monitor paper or board production for specific end uses provided acceptable correlation has been established between test values and the paper's end use performance. Due to the nature of the test and the penetrant, it will not necessarily correlate sufficiently to be applicable to all end use requirements. This method measures sizing by rate of penetration. Other methods measure sizing by surface contact, surface penetration, or absorption. Size tests are selected based on the ability to simulate the means of water contact or absorption in end use. This method can also be used to optimize size chemical usage costs.

4.3 This method is closely related to the widely used ink flotation test (Useful Method 481, no longer in print)¹. It has the advantage over the ink flotation test of detecting the endpoint photometrically, thus removing the human judgement factor. It has the disadvantage of being affected by sheet brightness and opacity.

¹ The flotation test uses a 5 cm (2 in.) square sample placed on standard writing ink or other test fluid. The result is reported as the time for a specified degree of color to appear on the upper surface.

4.4 The sensitivity and precision of this method make it attractive for specification purposes provided suitable correlation is established.

5. Applicable documents

Other tests designed to measure the sizing of paper include:

TAPPI T 433, “Water Resistance of Sized Paper and Paperboard (Dry Indicator Method)”

TAPPI T 441, “Water Absorptiveness of Sized Paper and Paperboard (Cobb Test)”

TAPPI T 458, “Surface Wettability of Paper (Angle of Contact Method)”

TAPPI T 491, “Water Immersion Test of Paperboard”

TAPPI T 558, “Surface Wettability and Absorbency of Sheeted Materials Using an Automated Contact Angle Tester”

TAPPI UM 481, “Ink Penetration of Paper” (no longer in print).

TAPPI UM 596, “Water Absorbency of Nonfibulous Paperboard (Water Drop Test)” (No longer published, but is available from TAPPI archives.)

6. Definitions

6.1 For definitions of many terms used in this test method, refer to Terminology ASTM D 1968 or any dictionary of paper industry terms such as TAPPI PRESS “Dictionary of Paper.”

6.2 *Sensitivity*: Sensitivity is the response of the instrument to a standard white ceramic tile. If the bulbs and all glass surfaces of the sizing tester are in good condition, the amount of light reflected by the standard will be high and the meter deflection will be large. Thus, to reduce random error that might be introduced during instrument calibration, be sure that meter deflection is large before proceeding with a sizing test.

6.3 *Linearity*: Overall, linearity reflects the capability of the instrument to accurately and consistently reproduce a given reflectance relationship. Instrument linearity is checked by determining the reflectance of the standard green tile as a percentage of the white tile.

6.4 *Spectral response*: Sensitivity of the device to certain wavelengths of light as measured by an output value.

7. Interferences

There are no known interferences other than colored papers that do not provide an adequate contrast with the green test ink or papers whose structure contain an effective water barrier such as polyethylene film. Based on round robin (see Table 2 in section 19), historical, and empirical data, there seems to be no evidence that calcium carbonate affects the HST test any differently than clay or other fillers. The test appears suitable for use on papers containing

calcium carbonate (4). It is left up to the user to determine the applicability of the test to their own situation, since not all grades of paper have been tested here.

8. Apparatus

8.1 *Single purpose photometer*², meeting the following requirements:

8.1.1 A 45° illumination of the bottom surface of the paper specimen by two halogen incandescent lamps, equidistant from, and 180° apart with respect to the center of the specimen, and powered by a constant voltage source.

8.1.2 A 90° viewing of the specimen by a (measuring) silicon photocell and direct viewing of the lamp filaments by a second (reference) silicon photocell; a means of balancing the two photocell outputs and a means of applying a desired percentage of the output of the reference photocell. The peak spectral response for the silicon photocell is 700 nm.

8.1.3 Effective limitation of heat transfer from the lamps to the specimen by the use of infrared filters and ventilating fans.

8.1.4 Incorporation of an integral timer with manual starting at the beginning of the test and automatic cutoff at the null point.

8.1.5 Permanent working standards, e.g. ceramic tiles, to monitor the ability of the equipment to consistently determine a reflectance ratio.

8.1.6 A holder to position the specimen over the viewing window and to hold ink in contact with the top surface of the specimen during the test. The instrument is constructed with a retaining ring around the viewing window that aligns the holder handle with a line connecting the lamp filaments. The holder has a test area of 16 cm² (2.48 in²) which should give an ink height above the specimen of 6.25 mm (0.25 in.) using the normal 10 mL of ink addition.

8.1.7 A specimen cover, black disc or block, which rests on the top of the sample holder.

8.1.8 A suitable dispenser for the ink that will provide 10 mL of ink repeatedly and quickly to the test specimen at the beginning of the test.

8.2 The following is a list of equipment or supplies used for the method:

Test instrument (covered in section 8.1.1 to 8.1.4)

Ceramic calibration tile (covered in section 8.1.5)

Sample holder (covered in section 8.1.6)

Black disk or block (covered in section 8.1.7)

Ink dispenser (covered in section 8.1.8)

²Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list in the set of TAPPI Test Methods, or may be available from the TAPPI Quality and Standards Department.

9. Calibration

9.1 Before making measurements, check the calibration of the apparatus using the two ceramic (or porcelain) tiles supplied for this purpose. It is recommended to check the calibration once per day. Calibration values are unique to the set of tiles furnished with each instrument and are not interchangeable between instruments. The tiles can be cleaned with any mild, non-abrasive cleaner and soft cloth.

9.2 Refer to the instrument manual for specific calibration instructions. If the calibration values are outside of recommended tolerances, take the corrective actions indicated in 9.3.

9.3 As the lamp bulbs age, the glass envelopes gradually darken, the light output decreases, and the spectral characteristics change. The effect of these changes due to bulb aging is a gradual decrease in the sensitivity reading and an increase in the linearity reading; therefore, if these values fall outside of the tolerance, consider first whether bulb aging could account for the observed condition. If so, replace the bulbs as the first corrective measure. Failure to obtain acceptable tile readings can also result from improper positioning of the heat filters. If they do not completely intersect the light path from the bulb filaments to the sample area, the optical response will be affected and calibration cannot be achieved. To determine this, carefully view the lamps through the sample opening. If the filaments can be seen without looking through the filters, reposition the filters. Another corrective action is to ensure all surfaces are clean (bulb, filters, glass opening). See the instrument manual for further details on the specific instrument model used.

NOTE 1: Bulb life varies from 3 days to 14 days depending on the instrument. The filament inside of the bulb should be parallel to the inside poles for best life. The bulbs shall be changed anytime the sensitivity or linearity values fall out of specification.

10. Reagents and materials

10.1 *Ink pack.* Ink packs have been produced by the manufacturer to eliminate all of the problems associated with make-up and assay of the test solutions. The ink pack consists of two parts: the dye pack and the acid pack. When mixed together in equal volumes, they produce a high-quality, optically correct ink for use with this method.

10.1.1 The dye pack solution is produced to give specific optical properties, not total solids. When mixed accurately, the ink will have an optical density of 0.31 to 0.35 when an aliquot diluted 1000:1 with demineralized water is measured for light absorption at 705 nanometers. In general, dye pack inks suitable for use with this sizing method:

- Must contain a dye that is not substantive to the fiber (i.e., one that does not attach permanently), so that the dye will penetrate the sheet together with the liquid phase.
- Must have precisely controlled composition and chemical properties.
- Should not contain coagulants.
- Should not emit corrosive gases that will attack electrical contacts in the electrical cabinet.
- Should be compatible with the acid or accelerants used in the test solution.

NOTE 2: Most commercial inks do not meet these requirements. However, inks based on naphthol green B dye which also absorb strongly in the 600 - 800 nm spectral range at the peak response of the photocells, do meet these requirements. A consistent-assay (solids and optical performance) naphthol green B dye is no longer commercially available. The supplier of the ink packs has naphthol green B dye custom-manufactured. This dye is supplied in liquid (2.5% solids) form. Optical density and spectral response of all dye packs are quality controlled by the supplier.

10.1.2 Acid packs come in 2% formic acid, (HCOOH), concentration. Formic acid strength is controlled to ± 0.02 percent of target. When combined properly with the dye pack, they form a 1% acid/ink pack. Five (5), 10, 20, and 40 percent acid/ink packs are made by combining customer purchased 10%, 20%, 40%, or 80% formic acid and blending equally with the dye pack. Purchase a grade of acid which specifies the exact percentage concentration, otherwise results will be affected. Store ink solutions in glass or polyethylene containers. Neutral test ink can be prepared by diluting a dye pack aliquot with an equal amount of demineralized water and adding 1 N NaOH solution to pH 7.0 ± 0.1 .

10.1.3 Shelf lives of prepared ink packs at room temperature are:

1% (“#2 Ink”)	6 weeks
5%	4 weeks
10%	2 weeks
20%	1 week
>20%	2 days
Neutral pH ink	1 week

10.2 *Alternative method.* The manufacturer is no longer supplying a dry dye, but for customers still using dry naphthol green B dye, the former method still applies.

10.2.1 *1.0% formic acid ink (#2 ink).* Dissolve 12.5 ± 0.05 g of naphthol green B dye or the corrected weight per the manufacturer’s instructions supplied with the dye. Add formic acid 10.0 ± 0.05 g on a 100% basis. Dilute to 1000 mL and mix thoroughly. Store in glass or polyethylene containers. This ink is stable for six weeks at room temperature after mixing. Discard after six weeks.

10.2.2 *10% formic acid ink.* Dissolve 12.5 ± 0.05 g of naphthol green B dye or the corrected weight per the manufacturer’s instructions supplied with the dye. Add formic acid 100.0 ± 0.05 g on a 100% basis. Dilute to 1000 mL and mix thoroughly. Store in glass or polyethylene containers. This ink is stable for two weeks at room temperature after mixing. Discard after two weeks.

10.2.3 *Higher formic acid inks.* Inks containing 20-60% formic acid can be used for extremely hard-sized and heavy weight paper and board samples. The preparation is the same as outlined for 10% formic acid ink with the suitable adjustment of the amount of acid for the given percent formic acid specification.

10.2.4 *Unacidified (water only) ink.* Some slack sized or lightweight papers may give an endpoint of only a few seconds with the 1% formic acid ink. A neutral ink is then recommended. 12.5 ± 0.05 g of dye is added to distilled water with sufficient NaOH added to adjust the pH to 7.0 ± 0.1 . The maximum shelf life is one week.

10.2.5 *Specialty inks.* If the ink is prepared with a dye other than naphthol green B, the name of the alternate dye should be noted with the results. See Table 1 for example of effects of ink type on HST results.

10.2.6 *Ink analysis.* Determine acidity of an appropriate sample of the ink by potentiometric titration with standard NaOH solution to pH 7. If acidity is outside tolerance limits, it is permissible to adjust it by adding concentrated formic acid or unacidified dye solution. Calculate acidity as follows:

$$\text{Acidity} = (4.6 \times \text{mL of NaOH} \times \text{normality of NaOH}) / \text{mL of ink}$$

$$= \text{g of formic acid} / 100 \text{ mL ink (or aliquot)}$$

Tolerance for 1% formic acid ink: 0.96 - 1.04%

Tolerance for 10% formic acid ink: 9.8 - 10.2%

11. Safety

11.1 **CAUTION:** 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 test method, the user should determine whether any of the chemicals to be used 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.

11.2 Materials used in this method are formic acid and naphthol B green dye. Others may be in use depending on special methods employed. Formic acid is a corrosive liquid and requires caution when handling. Avoid contact of the solution with skin, eyes, and clothing. Avoid breathing its vapor. Use only with adequate ventilation.

12. Sampling and test specimens

12.1 The test units of paper should be selected in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product" and should be conditioned in accordance with TAPPI T 402 "Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products." From each test unit, cut 10 specimens with a minimum dimension of 60 mm (~2.5 in.) in each direction and free of watermarks, creases, and blemishes.

12.2 Identify the top and bottom sides of each specimen and mark accordingly (see TAPPI T 455 "Identification of Wire Side of Paper").

12.3 Identify the machine direction and mark the specimens accordingly. The marking can be conveniently accomplished by cutting rectangular specimens with the long dimension parallel to the machine direction. A convenient specimen size is 70 x 80 mm (2.8 x 3.2 inches).

12.4 Handle specimens by the corners. Do not touch the region that will be in contact with the ink. Finger marks can seriously affect the test and must be avoided in the test area.

13. Conditioning

Condition the test specimens and operate the equipment in a controlled atmosphere in accordance with TAPPI T 402.

14. Procedure

14.1 *General instructions*

14.1.1 Make the test in an atmosphere in accordance with T 402.

14.1.2 Test all specimens with the machine direction parallel to a line connecting the lamp filaments.

14.1.3 Test five specimens with the top side and five with the bottom side to the ink. If specified, the ten tests may be made on the same side. State the % formic acid concentration used for the ink.

14.1.4 Unless otherwise specified, run the test to an 80% reflectance endpoint. If test times are impractically long or short, a higher or lower reflectance endpoint may be used (see 19.2). In all cases, the report must state the endpoint used. The formic acid concentration can also be changed to shorten or lengthen the test as needed.

14.1.5 Maintain the temperature of the test ink supply at $23 \pm 1.0^\circ\text{C}$. Rinse the specimen holder in water at this temperature (and blot dry) to prevent affecting the temperature of the ink in the course of a test. The value of the test can drop 50% for every 7°C rise in ink temperature. Keep the ink away from ventilation slots or other heat sources.

14.2 *Test procedure (Refer to the instrument manual for specific instructions.)*

14.2.1 Turn on the instrument and allow 15 min for warmup.

14.2.2 Set the % reflectance for the desired endpoint. (See Section 19.2 for endpoint considerations.)

14.2.3 Place the paper specimen in the holder (the side that is up will be in contact with the ink), with the machine direction parallel to the handle of the holder. Failure to align the specimen in the machine direction may affect the repeatability of the test results.

14.2.4 Position the holder containing the specimen in the retaining ring on top of the instrument.

14.2.5 Place the cover (black block or disc) over the specimen.

14.2.6 Calibrate the sample to the instrument by setting the initial reflectance to 100%. (Refer to the instrument manual.)

14.2.7 Remove the cover from the holder.

14.2.8 Pour 10 mL of test ink on the specimen and simultaneously start the test timer. The 10 mL of ink is for the specimen holder specified in Section 8.1.6.

14.2.9 Replace the black cover over the specimen. This step is often omitted when judged that the optical density of the ink is sufficient to resist the penetration of outside light. This can be seen by viewing the meter deflection with and without the black cover.

14.2.10 When the % reflectance reaches the setpoint, the test is complete, the timer stops, the signal light goes on, and the buzzer sounds.

14.2.11 Turn off the test timer and silence the buzzer.

14.2.12 Record the test time to the nearest full second. Fractions are not significant.

14.2.13 Remove the holder, discard the ink and specimen, rinse and dry the holder. It is advisable to clean up promptly after completion of the test to avoid specimen rupture and resulting ink spillage.

14.2.14 Repeat 14.2.3 - 14.2.13 for the remaining specimens.

15. Calculations (or interpretations of the observations)

No calculations are necessary.

16. Report

Report the test result to the nearest second as the average test time for the 5 specimens on the top side and the average test time for the 5 specimens on the bottom side, the % reflectance endpoint (e.g. "158 seconds to 80%"), and the % formic acid ink used in the test. If a specialty ink is used, it should be noted. If tests are made on one side of the sheet only, report this fact and the side in contact with the ink.

17. Precision

17.1 The following estimates of precision are based on an interlaboratory study involving 9 materials and 15 laboratories. Repeatability and reproducibility are estimates of the maximum difference (95% confidence) which should be expected when comparing replicate measurements for similar materials under similar test conditions. The samples/statistics are based on the average of 5 tests each on top and bottom, which can increase the variability for some grades, as coated paper or board. The variability is expected to be less when testing only one side. These estimates may not be valid for different materials and testing conditions.

17.1.1 For printing grade papers: (4 grades in the range of 100 - 200 seconds tested with 1% acid/naphthol green ink/ink pack tested to 80% reflectance, including alkaline papers). Material/ink combinations which reached endpoints in less than 20 seconds may have significantly poorer precision.

Repeatability (within a laboratory) = 26%

Reproducibility (between laboratories) = 36%

based on 10 replicates and 15 laboratories

The repeatability and reproducibility values were calculated according to TAPPI T 1200.

17.1.2 See Table 2 for a summary of repeatability and reproducibility for all grades. See the notes below Table 2 regarding bleached board repeatability and reproducibility.

17.2 The precision values reported in the 1989 version of the method were incorrect by the present T 1200 method for reporting precision. In addition, the repeatability value reported in 1989 should have been 20%, not 7% as printed. The new values reported in 17.1.1 are accurate in accordance with T 1200.

17.3 Individuals must determine if these repeatabilities and reproducibilities are satisfactory for their specific purposes.

18. Keywords

Sizing, Sized papers, Paper, Paperboard, Penetration, Ink, Ink absorption, Reflectance.

19. Additional information

19.1 Effective date of issue: to be assigned.

19.2 *Endpoint Selection.* Laboratories or mills testing a wide range of basis weights, or sheets with widely varying levels of sizing, can adjust the reflectance endpoint to complete the test within a reasonable period of time. General recommendations for selecting the endpoint on a given grade of paper are as follows:

1. The reflectance endpoint should be set at a level that will give at least a 30-second test time in order to obtain good reproducibility. Test times of 60 seconds or higher will give maximum precision.
2. Whenever possible, reflectance endpoints in the range of 50 to 80% should be used. Reflectance endpoints of 90% or higher give low meter deflection, and results are affected to a greater extent by paper variations or operator errors. Reflectance endpoints below 40% may give erroneous results. Most papers are almost saturated with ink at this reflectance level. Low reflectance values are sometimes used for papers with low sizing levels if the penetration to the other side is satisfactory.
3. Keeping in mind the above recommendations, a reflectance graph can be run to determine the slope of the time-reflectance curve. From this, select a percent reflectance setting that falls on the straight-line portion of the curve.

19.3 Typical test values on various grades of paper and board are listed in Table 3. Test conditions are usually selected to give a minimum test time of 30 seconds and a maximum test time of about 10 min. High formic acid inks or special inks are required to give reasonable test times on some board grades. For those papers that achieve very low values using this method, the lab should consider modifying the test to increase the results (i.e., lower reflectance, neutral ink). If those modifications are not satisfactory, other test methods should be run that provide values significant enough for control and minimal variation.

19.4 Significant changes in this revision (with no expected effect on the results):

om-96

- Section 17: Updated and corrected precision data.
- Section 7: Suitability of test for use on papers containing calcium carbonate.
- Section 10: Information on Ink Packs.

om-02

- Section 8: Remove circuit diagram.
- Section 14: Simplify procedure and refer user to instrument manual.
-

19.5 Factors affecting test times are:

- Ink temperature (see section 14.1.5)
- Calibration (see section 9)
- Broken specimen opening glass
- Ink properties (see section 10 and Table 1)
- Sample preparation (handling, top vs. bottom, etc.)
- Sample conditioning prior to test

Table 1. The effect of ink type on test results of different 65 gsm handsheet systems.

	<i>1% Acid Ink (pH 2)</i>	<i>Neutral Ink (pH 7)</i>	<i>Alkaline Ink (pH 9)</i>	<i>Demineralized Water</i>
0.25% Dispersed Rosin Size, 10% clay	105	151	88	2000+
0.10% AKD Size, 10% PCC	237	394	381	2000+

Table 2. Summary of repeatability and reproducibility statistics for all grades in round robin.

Sample Type	Grand Mean	Repeatability	Reproducibility	Test Conditions
Bleached Bag	7.0	64.2%	70.2%	20% FA Ink
Unbleached Bag	9.1	52.1%	78.2%	20% FA Ink
*20 lb Copy (AKD) White	13.9	33.6%	73.5%	1% FA Ink
Cylinderboard	54.1	34.5%	53.6%	1% FA Ink
*20 lb Copy (ASA) Pink	143.1	27.8%	33.4%	1% FA Ink
60 lb Offset White (acid)	150.9	30.4%	42.2%	1% FA Ink
20 lb Copy White (acid)	156.4	20.9%	38.4%	1% FA Ink
*20 lb Copy (ASA) Blue	184.8	24.8%	30.3%	1% FA Ink
Coated Bleached Board	588.3	35.8%	43.9%	20% FA Ink

* = Contain calcium carbonate filler ~ 15% level.

All "Test Conditions" run at 80% reflectance with the type of ink specified.

FA = Formic Acid; 1% FA = "#2 Ink".

The results for the bleached board sample were the average of 5 top side and 5 bottom side tests. When the data are separated into top side and bottom side the results are:

	Mean	% Repeatability	% Reproducibility
Felt:	652	12.0%	26.2%
Wire:	525	20.8%	41.1%

Table 3. Typical test values for various paper and board grades using the sizing tester.

Paper or Board Sample	Type of Ink Used	Reflectance Endpoint, %	Average Test Time, sec
16-lb bond	1% formic acid ink	80	50
20-lb bond	1% formic acid ink	80	75
20-lb bond pink (alk)	1% formic acid ink	80	143
20-lb bond blue (alk)	1% formic acid ink	80	185
60-lb offset	1% formic acid ink	80	151
40-lb unbleached bag	1% formic acid ink	80	300
18-pt plain chipboard	1% formic acid ink	80	54
150-lb manila file folder	10% formic acid ink	80	250
42-lb unbleached linerboard	10% formic acid ink	80	400
11-pt coated bleached board	10% formic acid ink	85	180
14-pt coated bleached board	40% formic acid ink	70	80
18-pt coated bleached board	40% formic acid ink	70	140
24-pt coated bleached board	40% formic acid ink	70	250
30-pt coated bleached board	40% formic acid ink	70	400

19.6 The values in Table 3 are suggested sample values for selection of an applicable % formic acid ink, and are not target values for a given grade of paper or board. HST values vary approximately to the square of the caliper with everything else held constant.

Literature cited

1. Hammond, J., "The News Penetration Tester," *Paper Trade J.* **103** (21): 37(1936).
2. Instrumentation Study 21A, "The Barss, Knobel, and Young News Penetration Tester," Institute of Paper Chemistry (June 1937).
3. Price, D., Osborn, R. H., and Davis, J. W., "A Liquid Penetration Test for Measuring the Sizing of Paper," *Tappi* **36** (1): 42 (1953).
4. Boone, S., "The Use of T 530 (HST) on Calcium Carbonate Containing Papers," *Tappi* **79** (2): 122 (1996).

Reference

"The Sizing of Paper, 2nd ed," Walter F. Reynolds ed., Chapter 7, TAPPI PRESS (1989).

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