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Size test for paper by ink resistance (Hercules-type method)

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 (1-3) 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.

Approved by the Standard Specific Interest Group for this Test Method



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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 may 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.

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 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.

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.

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 contains 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 from 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.

8.2 *Ceramic calibration tile*. Permanent working standards, e.g. ceramic tiles, to monitor the ability of the equipment to consistently determine a reflectance ratio.

8.3 *Sample holder*. 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.

8.4 *Black disk or block specimen cover*, which rests on the top of the sample holder.

8.5 *Ink dispenser*, 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.

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 may 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.

² Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list, available as part of the CD or printed set of Standards, or on the TAPPI website general Standards page.