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Technical Association of Pulp and Paper Industry
Suggested Method T 652

Standard Test Method for Blocking and Picking Points of Petroleum Wax[1]

This standard is issued under the fixed designation D1465; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the blocking point and picking point of petroleum wax.

1.2 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are provided for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)

D938 Test Method for Congealing Point of Petroleum Waxes, Including Petrolatum

D2423 Test Method for Surface Wax on Waxed Paper or Paperboard

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *wax blocking point*—the lowest temperature at which film disruption occurs across 50 % of the waxed paper surface when the test strips are separated.

3.1.2 *wax picking point*—the temperature at which the first film disruption occurs on the waxed paper when test strips are separated.

4. Summary of Test Method

4.1 Paper test specimens are coated with the wax sample, folded with the waxed surfaces together, and placed on a blocking plate. The plate is heated at one end and cooled at the other end to impose a measured temperature gradient along its length. After a conditioning period on the plate, the specimens are removed, unfolded, and examined. The points of initial film disruption (picking point) and where 50 % of the specimen width is disrupted (50 % blocking point) are noted. The temperatures of corresponding points on the blocking plate are reported as the picking point and blocking point or blocking range.

5. Significance and Use

5.1 Blocking of waxed paper, because of the relatively low temperatures at which it may occur, can be a major problem to the paper-coating industry. For example, when the waxed surfaces in a roll of waxed paper stick together, upon separation the surface films are marred and the glossy finish is destroyed. The wax picking point and the wax blocking point indicate an approximate temperature range at or above which waxed surfaces in contact with each other are likely to cause surface film injury.

6. Apparatus

6.1 *Wax Coating Device*—Any manual- or machine-driven waxer that gives uniform coatings of the desired weights to a strip of paper not less than 51 mm (2 in.) wide.

6.2 *Trimming Board,* or other device for cutting paper strips.

6.3 *Paper Scales,* sensitive to 0.002 g, or suitable balance for measuring basis weights of unwaxed and waxed paper.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricantsand is the direct responsibility of Subcommittee D02.10.0A on Physical/Chemical Properties. This test method was prepared jointly by the Technical Association of the Pulp and Paper Industry and the American Society for Testing and Materials.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

6.4 *Blocking Plate Assembly,* consisting of a metal plate 305 mm (12 in.) wide, 13 mm to 51 mm (½ in. to 2 in.) thick, and 559 mm to 914 mm (22 in. to 36 in.) long. It is heated at one end and cooled at the other end to provide a 457 mm to 762 mm (18 in. to 30 in.) test section having an average temperature gradient of 0.8 °C to 2.2 °C (1.5 °F to 4.0 °F)/in. The temperature gradient across the width of the plate from the longitudinal axis to 25 mm (1 in.) from the edge must not exceed 0.3 °C (0.5 °F). The temperature at any point should not vary more than 0.3 °C (0.5 °F). Details of two acceptable blocking plates are given in Fig. 1 and Fig. 2.

6.4.1 *Eight Foam Rubber Strips,* 25 mm to 38 mm (1 in. to 1½ in.) wide by 6 mm to 13 mm (¼ in. to ½ in.) thick, extending the length of the test section on the blocking plate.

6.4.2 *Eight Sets of Steel Weights,* 25 mm by 25 mm (1 in. by 1 in.) in cross section made from stock having a density range from 7800 kg/m^3 to 8000 kg/m^3 (0.28 lb/$in.^3$ to 0.29 lb/$in.^3$). The weights may be in single lengths or in sections to give sets equaling the length of the test section of the blocking plate.

6.5 *Temperature Recorder,* or indicator for measuring the temperature gradient of the blocking plate. The instrument and thermocouples when calibrated shall consistently be accurate to ±0.3 °C (0.5 °F).

6.6 *Temperature Measuring Devices:*

6.6.1 *Thermometers*—For water bath and wax bath on the waxing device, use two ASTM Tag Closed Tester Thermometers having a range from –5 °C to 110 °C (20 °F to 230 °F), graduated in 0.5 °C (1 °F) subdivisions, and conforming to Thermometer 9C or 9F as prescribed in Specification E1.

6.6.2 Alternatively, bath temperatures may be monitored with a calibrated thermometer containing a liquid other than mercury or an electronic temperature measuring device of equivalent precision and accuracy to the thermometers described in 6.6.1.

7. Test Paper

7.1 *Paper*—Cereal glassine of basis weight 46 g/m^2 to 51 g/m^2 (28.5 lb/3000 ft^2 to 31.5 lb/3000 ft^2).

8. Sampling

8.1 Select a portion of the wax that is representative of the whole sample. The amount required will depend on the size of the waxing device used.

9. Standardization of Apparatus

9.1 Turn the blocking plate on with all sponge rubber strips and metal blocks in position. Allow sufficient time (3 h minimum) for the plate to reach an equilibrium temperature condition.

9.2 Calibrate a 30-gauge "test" thermocouple as follows: Heat a beaker of water to about 43.3 °C (110 °F) and measure its temperature with a temperature measuring device which is accurate to 0.3 °C (0.5 °F). Insert the "test" thermocouple in the water and measure the temperature with a manual potentiometer which is accurate to at least ±0.5 °C (±1 °F). A portable precision type potentiometer or a type *K* potentiometer, both of which are accurate to at least 0.5 °F (0.3 °C) in this temperature range, is preferred.

9.3 Lay the calibrated "test" thermocouple across the width of the plate with its junction directly over one of the imbedded thermocouples. Be sure that one of the sponge rubber strips covers the junction completely. Replace the weights on the rubber strips. After 3 min to 4 min, note the temperature readings on the hand potentiometer and the corresponding point on the recorder. If the reading of the test thermocouple plus its calibration factor is more than 0.5 °C (1 °F) from that of the recorder reading, check the accuracy of the recorder separately. If the recorder is satisfactory, the trouble may result

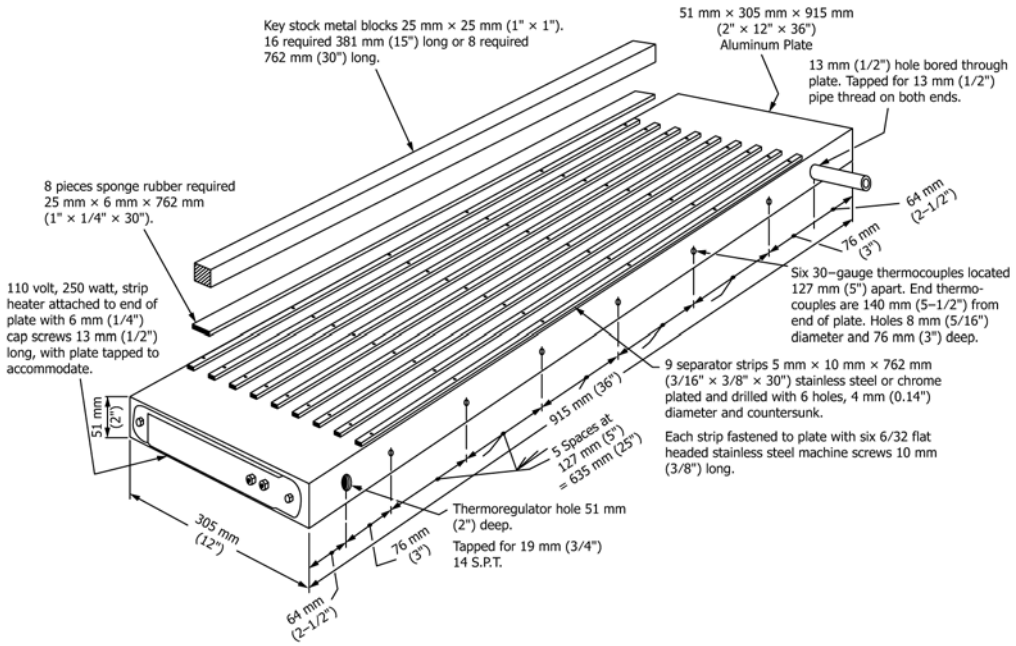

FIG. 1 Type A Blocking Plate

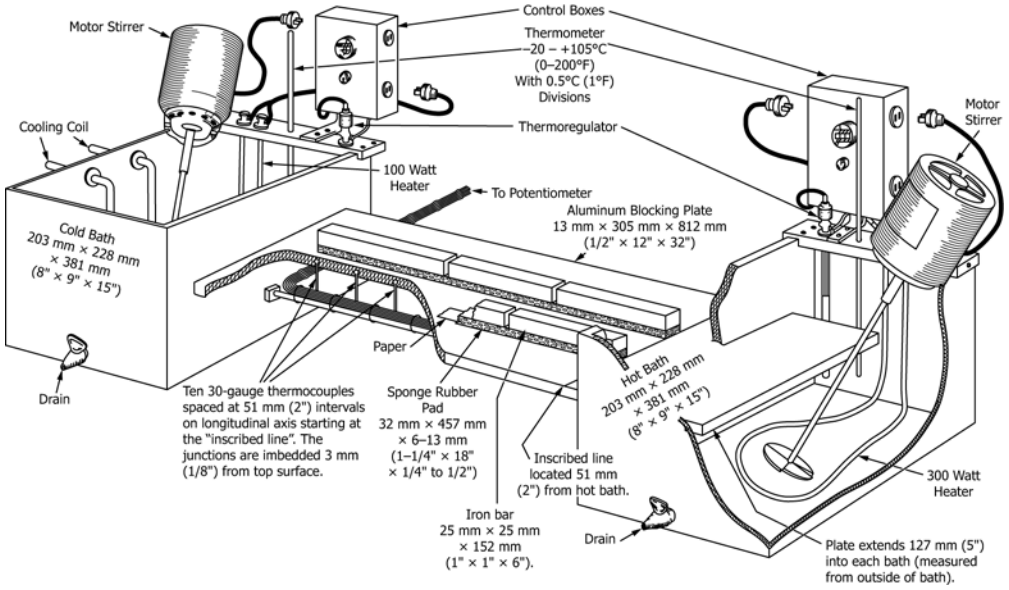

FIG. 2 Type B Blocking Plate

from the thermocouple being damaged or not in solid contact with the plate. Replace this thermocouple, taking care to tamp it into place so that its junction is held firmly against the plate metal. Check the new thermocouple and each of the other imbedded thermocouples in a like manner. Note the calibration factor of each.

9.4 For best operation, install the blocking plate in a constant temperature room. Check the plate calibration periodically, after installing new thermocouples, or if the plate temperatures vary more than 0.5 °C (1 °F) from the normal conditions.

10. Preparation of Waxed Paper Test Specimens

10.1 Heat the wax sample to at least 22 °C (40 °F) above its expected melting or congealing point according to Test Method D87 or Test Method D938, using an air or water bath so as not to overheat portions of the sample. After cleaning the waxing device, fill the wax bath with the sample to be tested. Hold the wax bath and the doctor rods or squeeze rolls at a temperature at least 22 °C (40 °F) above the expected melting or congealing point of the wax. For machines not having positive temperature control (heated electrically or by hot water) of the doctor rods or squeeze rolls, use infrared lamps or precontact the rolls with hot wax to heat them to about the wax bath temperature.

10.2 Use paper conditioned at least a week at 23 °C ± 2.0 °C (73 °F ± 3.5 °F) and 50 % ± 5 % relative humidity. Load the wax coating device with the paper passing through the wax bath. Coat the paper with 6.5 g/m^2 to 10 g/m^2 (4 lb/3000 ft^2 to 6 lb/3000 ft^2) of wax on one side and 3.2 g/m^2 to 10 g/m^2 (2 lb/3000 ft^2 to 6 lb/3000 ft^2) of wax on the other.

10.3 Cool the wax coated paper to room temperature in ambient air.

NOTE 1—As an alternative procedure, the wax coated paper may be cooled by passage through a water bath. There is evidence, however, the water quenching can cause variation in picking and blocking point values obtained on some waxes.

10.4 Measure the coating weight using Test Method D2423, with the exception of substituting the blocking point strips in Test Method D1465 for the 100 mm by 100 mm square of waxed paper in Test Method D2423. If the weights are other than 6.5 g/m^2 to 10 g/m^2 (4 lb/ream to 6 lb/ream) of wax on the test surface and 3.2 g/m^2 to 10 g/m^2 (2 lb/ream to 6 lb/ream) of wax on the back side, prepare another waxed paper specimen.

10.5 If the coating weights are satisfactory, cut two test specimens from the waxed test paper, each specimen 25 mm (1 in.) wide and twice the length of the test section of the blocking plate.

10.6 Condition the waxed specimens for at least 24 h at 23 °C ± 2.0 °C (73 °F ± 3.5 °F) and 50 % ± 5 % relative humidity.

11. Procedure

11.1 The blocking plate may be left on continuously. If starting with a cold plate, allow at least 3 h for the plate to heat up and reach an equilibrium condition with the pads and weights in place. Check the temperature recorder to be sure the plate is at the desired temperature.

11.2 The blocking plates have room for seven or eight rows of specimens across the plate. Fold the specimens to the length of the test section with the surfaces face to face. Cut single unwaxed glassine paper strips (or glassine, if desired) the same size as the folded, waxed test specimen. Insert the folded waxed specimen between the two unwaxed strips and place it longitudinally on the blocking plate with the folded end on the "start" line at the hot end. Place a second waxed specimen and an unwaxed strip in a similar manner on top of the first specimen. Not more than two of these test specimens should be placed in each testing space. Carefully smooth out the wrinkles

in the specimens. Place any other specimens to be tested in other rows on the blocking bar in the same manner. If the blocking plate is not filled to capacity with specimens, insert sponge rubber pads and weights in the empty spaces.

11.3 After 17 h exposure on the blocking plate, remove the specimens together with the unwaxed spacer strips. Cool them for a minimum of 5 min. Carefully peel off the unwaxed strips and pull the specimens apart starting at *cold* end at a rate of about 152 mm (6 in.)/s.

11.4 Examine the test surfaces closely and mark the picking point and the 50 % blocking point. The picking point is the first of a series of spots of actual film disruption nearest the "cold" end of the strip. The 50 % blocking point is the first point nearest the cold end where 50 % of the width of the strip shows film disruption. Fine, isolated lines across the strip are not rated as the 50 % blocking point. Loss of gloss or spotting without film disruption is not considered as blocking or picking.

NOTE 2—Several techniques have been used for viewing the test strips. These include viewing against reflected light, dusting the strips with graphite, or placing an opaque object behind the test strip.

NOTE 3—Loss of gloss (blooming) can sometimes be distinguished from film disruption by lightly wiping the surface of the test paper with a cotton gauze pad.

12. Calculation and Report

12.1 Plot the temperature gradient curve for the instrument from the temperature recorder readings. Aside from a slight rise in temperature when the specimens are first placed on the plate, the temperature of any single point should not vary more than 0.3 °C (0.5 °F).

12.2 Measure the distance from the fold to the two blocking point marks for each specimen with the wax sample being tested. Note the blocking temperatures on the temperature gradient curve corresponding to these distances.

12.3 Report the average results to the nearest 0.5 °C (1 °F). A wax with a 41 °C (106 °F) initial film disruption and a 50 % blocking point of 43 °C (110 °F) would be reported as:

12.3.1 *Picking Point*—41 °C (106 °F).

12.3.2 *Blocking Point*—43 °C (110 °F).

13. Precision and Bias

13.1 *Precision*—Results should not differ from the mean by more than the following amounts:

13.1.1 *Picking Point:*

13.1.1.1 *Repeatability* (one operator and apparatus), 2.8 °C (5 °F).

13.1.1.2 *Reproducibility* (different operators and apparatus), 3.6 °C (6.5 °F).

13.1.2 *Blocking Point:*

13.1.2.1 *Repeatability* (one operator and apparatus), 1.7 °C (3 °F).

13.1.2.2 *Reproducibility* (different operators and apparatus), 3.3 °C (6 °F).

NOTE 4—The precision as stated in Section 13 was obtained by a cooperative program among seven laboratories using two standard reference waxes which have sharp blocking points. Where a sharp blocking point cannot be obtained because of the composition of the wax sample being tested, poorer precision in test results will be encountered.

13.2 *Bias*—The procedure in this test method has no bias because the measurement of blocking and picking points can be defined only in terms of a test method.

14. Keywords

14.1 blocking point; picking point; waxed coating; waxed paper