USE OF A STRAIN GAGE FOR WAX SEALING STRENGTH TESTS

The sealing strength of waxed paper presents a testing problem because of the low loads involved. It has been found that the sealing strength values range around 6 to 10 grams per inch of seal width. The commercial testers available at The Institute of Paper Chemistry are all too insensitive to give accurate measurements at low loads. This report describes a strain gage transducer which has been found valuable in extending the testing range of an ordinary tensile tester and making it possible to obtain a record of the test.

The principle of the strain gage is based upon the fact that the resistance of a wire changes as it is stretched. The wire is made a part of a Wheatstone bridge circuit in which the change in resistance is a function of the force or load applied to the strain gage or its mount.

Our first attempt was to prepare a bonded type strain gage. This involved cementing an SR-4 type A5-1 strain gage to the inside and outside surface of a clock spring steel ring about 6 cm. in diameter and 1.8 cm. wide. The electrical output of this device was adequate when coupled to a special amplifier built by the Physics Department and used in conjunction with a 5 m.a. Angus-Esterline recorder. This equipment was not available for continued use, however. Attempts to use this strain gage with the
Brown Electronik recording potentiometer were unsatisfactory due to the lower gain available in its amplifier.

It was decided that instead of going to the expense of a preamplifier or another model of a Brown Electronik a possible answer might be the use of an unbonded strain gage.

A Statham transducer, model G1-4250, was fitted with a suitable support so that it could be mounted in the Amsler Universal Tester. This model has a force range of +4 ounces, a displacement of +0.0015 inches, a nominal bridge resistance of 250 ohms, rated input of 8 volts, full scale open circuit output of 18 millivolts and a natural frequency of 430 cps.

The bridge voltage was obtained with either a 6-volt storage battery or a simple power supply for direct current. The power supply consisted of a Tri-volt (6, 8 and 10 volts) Jefferson Electric Company bell ringing transformer, a Sarkes-Tarzian selenium rectifier, model AH100, and a suitable electrolytic condenser and resistance. This was housed in an 8 by 8 by 8-inch aluminum box together with the bridge balance and calibration controls. The circuit used is shown in Figure 1.

The bridge was balanced with a General Radio 20,000 ohm potentiometer. This control should probably be replaced by a multi-turn type of potentiometer, such as the Helipot, for better accuracy and ease of manipulation. A 30-0-30 microammeter was used to indicate bridge balance.
The calibration was accomplished by dead weight loading and controlling the bridge output with two cascaded potentiometers to get a direct reading on the Brown Electronik Recording Potentiometer. The bridge was first balanced with the calibration controls in the most sensitive position.

A double-pole double-throw switch was used to switch from the bridge adjustment to the recording circuit. A polarity switch was incorporated to simplify change-over when setting up the equipment.

The test specimens for wax sealing strength are usually cut to a 3-inch width. No. 3B.D. Automatic Pencil Sharpener Co. paper clips with 2-3/4 inch wide jaws were used to support the sealing strength specimens. It was found desirable to support these clamps at the center instead of by the handle in order to provide a straight-in-line force with the motion of the transducer armature.

The sealing strength tests of waxed paper were run at a jaw separation rate of 5 inches per minute. The control valve of the Amsler tester appeared to give this rate with the small ram in operation when set at 4.5.

The output of the transducer was more than ample for the use intended. Other models of the Statham transducer may be obtained with lower or higher force ranges.

fr mab
$R_1 = 18,000 \text{ ohms } 1 \text{ watt}$

$R_2 = 25,000 \text{ ohms}$

$R_3 = 22,000 \text{ ohms}$

$R_4 = 70,000 \text{ ohms}$

$R_5 = 500 \text{ ohms}$

$C_1 = 25 \mu F 250 \text{ V.D.C.}$

$T = \text{Tri Volt Jefferson Electric Co., Bell ringing transformer}$

$P = 6 \text{ V. Pilot Light}$

$S_1 = \text{Switch S.P.S.T.}$

$S_2 = \text{Switch DPDT reversing}$

$S_3 = \text{Switch DPDT}$

**Fig. 1**

Wax Sealing Strength Tester

Strain Gage Circuit
INTRODUCTION

This report will indicate the status of current participation in ASTM-TAPPI Wax Testing Committee, Section II, for evaluation of penetration of wax. The phase of the program under evaluation in this laboratory concerns depth of penetration as a function of the angle included by two sides of the penetrating instrument. Temperature was carefully controlled and special precautions taken to assure that the needle was carefully cleaned and conditioned between separate runs.

The current investigation is an independent study assumed to investigate the homogeneity of penetrated wax sample as a function of the density of the wax casting based on the hypothesis that the hardness of a wax varies as we go into the interior of the wax specimens prepared according to the proposed method. See the minutes of the last meeting transmitted with a letter from W. R. Turner to committee members, dated December 9, 1954, which lists "items brought out in discussion as points requiring attention" concerning the proposed method.

Note that, except for special attention to conditioning and cleaning of the needle, we have departed from the suggested line of attack. We feel
that attention should be directed to the more fundamental problem of first analyzing the character of the wax specimen, instead of trying to perfect a specific means of testing something which may not be uniform.

METHOD - EQUIPMENT

The method transmitted by a letter from G. P. Hinds, Jr. to Section II committee members, dated August 20, 1954, was used, except as noted below with reference to specific sections of the method concerned.

Method is headed T-639 m-54 and is the same as ASTM Method D-1321054-T.

Apparatus, Paragraph 3. Four different needles were used:

- ASTM D-5 needle
- S.O.D. needle
- 20° cone
- 30° cone.

The angles included by two sides of these needles were measured by a technique described in the Preliminary Operations section of this report. They were found to give effective angles of penetration in a range from 10 to 30°. The ASTM D-5 and Standard Oil Development (S.O.D.) needles have similar angles, except that the effective length of the S.O.D. needle is much longer.

Apparatus, Paragraph 4. Because of the greater diameter of the 20° and 30° cones, larger test specimen containers were used. They consist of a brass cylinder open at both ends having 2-inch inside diameter, 1\(\frac{1}{2}\)-inch height, and 5/32-inch wall thickness. A 3/16-inch deep shoulder, 3/32-inch thick, in the center of the inside wall retains the wax casting.
Note 3, last portion. A 12 x 12 x 24-inch long aquarium type constant temperature bath was used for conditioning and testing of the wax specimens. A 4½-inch high platform resting on the floor of the bath covers an auxiliary steam coil and serves as a support for the penetrometer. The top of the platform was not rigid enough to give a firm levelling base for the penetrometer, so it was covered with a 11½-inch square sheet of double strength glass. This gave the necessary support and the instrument could be properly levelled. Temperature control equipment consisted of a sensitive mercury-to-platinum thermoregulator, "Precision" scientific relay, three 250-watt knife blade immersion heaters. Two Variacs were used to proportion voltage to the heaters for control of "lag" and "overshoot". A small bilge pump mounted on the 4½-inch shelf for proper immersion gave the circulation rate required. Temperature was controlled with the use of this equipment to within 0.10°F. of the specified testing temperatures.

Apparatus, Paragraph 8. Thermometers: A National Bureau of Standards certified Wesco thermometer No. 34 06 149 was used to measure the test bath temperature. It has a 0 to 105°C. range and 0.1°C. finest division.

Preparation of Test Specimen.

The melted wax was poured into molds which were preconditioned for a minimum of 15 minutes in an air circulating oven at 73°F. ± 1.5°F. (Oven placed in 40° refrigerator to obtain controlled temperature of 73°F., which is below normal room temperature.) Poured molds were conditioned for
one hour at the same temperature, then transferred immediately to the water bath for final conditioning. Because the heat capacity of the units adjacent to or in contact with the wax may affect the character of the wax casting because of the differential cooling rates, the casting orientation is hereby defined. Two casting molds are equipositioned on a 3 x 6-inch double strength glass plate suspended at the ends by two 3/4-inch wooden blocks resting on the oven floor. Three inch square pieces of 0.003-inch caliper aluminum foil was inserted between the plate and the glass. After the specified conditioning time, the molds were filled with wax.

**Procedure, Paragraph 1.**

The test specimens were conditioned and tested in a water bath controlled to 100 ± 0.1°F and 115°F ± 0.1°F.

**Note 5. Disregard.**

**Procedure, Paragraph 2.**

100 grams total penetration force (including needle) was used for all needles.

**Procedure, Last Paragraph.**

A wax casting was tested, using first the ASTM D-5 needle, then the 30° cone. The ASTM D-5 needle (smaller diameter) penetrations were positioned according to the proposed method. For the larger angled needle, penetrations were at least 3/8 inch from the side of the container and
between the previous penetrations. Since four penetrations were made, the plane of penetration was thus rotated by $45^\circ$ for the second run of penetrations. The same was done with the S.O.D. and $20^\circ$ penetration needles.

The needle was dried with a paper towel, being careful not to damage the tip; cleaning with a cloth saturated with filtered trichloroethylene, again dried with a clean, dry cloth, and returned to the water bath for 5 minutes to allow reconditioning of the needle prior to the next penetration.

The molds were cleaned between pourings by warming over a flame and placing in a covered dish of toluene. It was then wiped dry with a clean cloth and placed in a low-boiling solvent as an aid to complete drying of the mold. Heptane was the solvent selected. Molds were again wiped with a clean cloth and preconditioned at the air bath pouring temperature previously noted.

Two identical castings of Moore and Munger wax were prepared, using aluminum foil to prevent them from sticking to the glass, and also to provide a good reflective surface. This foil was stripped from one immediately before placing in the water bath. The foil was left in place on the second mold until 5 minutes prior to testing (the needle conditioning interval) and the character of both surfaces recorded.

The first impression was that the newly stripped casting prevented a more reflective surface, but this conclusion was largely due to the occurrence of numerous air bubbles on the surface of the casting which was
conditioned with the foil covering. One hour later both specimens had the same general appearance, i.e., both penetration surfaces were covered with bubbles. Because the lack of bubbles was of considerable advantage in positioning the needle, the castings were stripped just prior to testing, allowing the 5-minute period desirable to assure conditioning of the surface.

Due to the rather long period of testing (about 1½ hours) as a consequence of the time required for conditioning of a needle between each run, the occurrence of bubbles on the surface again became a problem. When necessary, these bubbles were removed by lightly brushing the test surface with a small piece of cloth without removing the casting from the water bath. The casting was then reconditioned for 5 minutes before testing. By the above procedure a good reflective surface was maintained at all times using Moore and Munger 100 Wax.

It is possible to explain the phenomenon of bubble formation without respect to the time at which the foil is removed. If we assume that the foil gives a good reflective surface to the wax casting, the occurrence of bubbles can then be explained by the position of the casting while conditioning and a reasonable course of action prescribed. After the foil is removed, a casting is usually conditioned for the remainder of the time with the test surface upward. Prior to stripping of the foil, it is generally conditioned with the test surface down. It is plausible to assume that air driven from the wax at higher temperatures (due to air either physically or chemically entrapped into the wax during the manufacture, melting, or pouring procedure) will rise in the wax until it reaches the
surface if the consistency of the wax is low enough to allow passage of such air bubbles. Thus, the bubbles will appear largely on the upper surface of the casting as it is conditioned if the surface is not covered with foil. Thus, a casting conditioned with its test surface down, the normal procedure if the casting is stripped just prior to testing, would be expected to be free of air bubbles.

Regardless of the nature of bubble formation, the aluminum foil technique of casting appears to give a smoother, more reflective surface than the glycerin technique previously used. Note that bubble formation has never been a problem with Shell Wax P-417-410.

PREPARATORY OPERATIONS AND CALCULATIONS

A. Penetration needles and cones - Calculation of angles.

1. Method: The diameter of the tip of the penetrating instrument was measured with the use of a Spencer microscope fitted with an eyepiece micrometer. Care was taken to avoid parallax error by focusing sharply on the needle tip held firmly to a glass slide with scotch tape, so that the bevel of the needle was retained parallel to the slide surface.

A vernier caliper was used to measure the length and diameter of the top of the needles and cones.

2. Calibration factor for eyepiece micrometer:

Objective 10X

1 unit = 7.58 microns
1 micron = 0.001 mm.
3. Measurement of Four Needles and Cones used:

ASTM D-5 Needle

\[ 20 \times 7.58 \times 0.001 = 0.152 \text{ mm}. \]

\[ 1.0 \times 2 = 0.5 \]

\[ 0.152 \times 2 = 0.076 \]

\[ 0.50 - 0.08 = 0.42 \text{ mm}. \]

\[ \log 0.42 = 9.6232 - 10 \]

\[ \log 5.3 = 0.7243 \]

\[ \log \tan = 8.8989 - 10 \]

\[ \frac{\pi}{2} \approx 4.52^\circ \]

\[ 2 \times \frac{\pi}{2} = 9.06^\circ \]

\[ = 9.1^\circ \]
Calculation of error in calculated angle - \(2\alpha\)

\[
\begin{align*}
\log 0.42 &= 9.6232 - 10 \\
\log 5.4 &= 0.7324 \\
\log \tan \alpha' &= 8.8908 - 10 \\
\alpha' &= 4.45^\circ \\
2\alpha' &= 8.9^\circ \\
9.06 - 8.90 &= 0.16^\circ \\
\text{Therefore: } 2\alpha &= 9.1 \pm 0.2^\circ
\end{align*}
\]

Similar calculations for the angles of the three remaining needles and cones give:

\[
\begin{align*}
\text{S.O.D.} & \quad 2\Theta = 8.9 \pm 0.04^\circ \\
20^\circ \text{ cone} & \quad 2\chi = 19.8 \pm 0.1^\circ \\
30^\circ \text{ cone} & \quad 2\upsilon = 42.1 \pm 0.7^\circ \\
& \quad 2\phi = 29.8 \pm 0.1^\circ
\end{align*}
\]

The \(42.1^\circ\) cone at the tip of the \(30^\circ\) cone was discounted and the \(29.8 \pm 1.0^\circ\) angle used in the following interpretations.

B. Calculated Weight to Load Needle for 100 Gram Total.

The needles were weighed to the nearest 0.1 gram, using the Torsion Balance and standard analytical weights. The ASTM D-5 needle and penetration assembly were weighed by rigidly supporting the penetrator over the pan of an ordinary beam balance counterbalanced with a 50 gram weight and releasing the clutch used to retain the assembly. This method is liable to error caused by friction of the assembly to the pan bearing surface, but should serve to check the instrument manufacturer's specification for total assembly and needle weight—50.0 grams. 50 \(\pm\) 1 gram was about the best value obtained by our weighing method. The figure 50.0 was used for the calculations which follow in tabular form:
Nominal Needle Designation

<table>
<thead>
<tr>
<th></th>
<th>30°</th>
<th>20°</th>
<th>S.O.D.</th>
<th>D-5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Actual weight of needles, g.</td>
<td>14.3</td>
<td>8.1</td>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>Needle D-5 assembly weight - 50.0 g.</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Assembly weight</td>
<td>47.5 g.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Weight of base of constructed &quot;weight&quot;, g.</td>
<td>31.4</td>
<td>31.55</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Weight added with solder, g.</td>
<td>6.8</td>
<td>12.85</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Total weight of constructed &quot;weight&quot;, g.</td>
<td>38.2</td>
<td>44.40</td>
<td>50.0</td>
<td>50.0</td>
</tr>
<tr>
<td>Total weight of assembly, needle and &quot;weight&quot;, g.</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
<td>100.0</td>
</tr>
</tbody>
</table>

The penetrometer is equipped with a 50.0 g. weight. 38.2 and 44.4 gram weights were prepared by winding wire solder around a flanged steel nut using the Torsion Balance to measure the weights secured. The built-up weights were marked with corresponding needle or cone size.

C. Thermometer and Corrections.

A Wesco thermometer, No. 34 06 149, 0 to 105°C. with 0.1°C. divisions -- certified in 1944 by the National Bureau of Standards, was used. This thermometer is calibrated for total immersion, but was immersed to the +10°C. position. Emergent stem corrections were made by classic formulae to give corrected temperatures.
Testing at 100°F. (37.78°C.)

Ice point and characteristic thermometer correction: -0.10°C.
Emergent stem correction: (Mean temperature 25-26°C.,
immersed to +10°C.) +0.05°C.
Total correction: -0.05°C.
Nominal temperature: 37.73°C.

Testing at 115°F. (46.11°C.)

Ice point and characteristic thermometer correction: -0.09°C.
Emergent stem correction: (Mean temperature 25-26°C.,
immersed to +10°C.) +0.12°C.
Emergent stem correction: (Mean temperature 27°C.,
same immersion): +0.11°C.
Emergent stem correction: (Mean temperature 28°C.,
same immersion): +0.10°C.
Observed mean temperature of mercury column: 27°C.
Total correction: +0.02°C.
Nominal temperature: 46.13°C.

D. Voltages Used--Temperature Control of Bath.

Three 250 watt knife blade immersion heaters were used, either
one or two of them could be used intermittently, being switched on and off
by a mercury-to-platinum thermoregulator and a "Precision" scientific relay.
Two Variacs were used to proportion the voltage to the intermittent and
auxiliary heaters for control of "lag" and "overshoot". The voltages used
under present test conditions and the maximum observed temperature variation
for the two runs made are given below.

Testing at 100°F.

90 volts continuously -- one 250 watt heater
105 volts intermittently -- two 250 watt heaters
Maximum observed temperature variation (two runs): ± 0.02°F.
Testing at 115°F.

55-65 volts continuously - two 250 watt heaters

100-110 volts intermittently - one 250 watt heater

Maximum observed temperature variation (2 runs): ±0.04°F.

E. Master Sample Preparation and Pouring Temperature.

All wax on hand of each type, i.e., Moore and Munger 100, and Shell P417-410, were melted together in a stainless steel beaker, heated to 225°F., stirred well and cast into a master cast for the current round robin testing program. Sources are as given below:

**Moore and Munger 100 Wax**

1/2 pound new wax - Department File 3E55
Approximately 1/2 pound previously used wax - Round Robin, September, 1954. Note Book 1349, pages 9 - 27

**Shell P-417-410**

1 pound new wax - Department files 41-445 and 164-218

Pouring temperatures were the same as previously reported, page 5, Report No. 9, Project 1685.

PROCEDURE

See Method Section of this report.

In addition to testing with needles previously described, a
brief series of tests were conducted using Shore Instrument Company
Durometer Testers "A" and "D", according to a suggestion by Mr. Hinds at
the September, 1954 committee meeting to the effect that the Durometer
Model A-2 gave reasonably consistent differentiation between waxes of
varying hardness in the Shell Oil Company laboratories (See Memorandum
from Mr. Vaurio to Files dated October 13, 1954.)

To implement our testing program, we tested the same wax castings
prepared for penetrometer penetrations. The sample was raised from the
water bath surface and tested immediately by quickly pressing the Durometer
on the flat wax surface. The highest reading observed as soon after contact
of the surface as possible was recorded. (It was observed that the needle
falls off quickly after the maximum value is reached, reaching the 0 value
in a very short period of time for both waxes tested at 115°F.). Accumulated
wax on the bottom of the Durometer was wiped off with dichloroethylene
saturated cloth. The tester was placed on the table top (73° ± 2°F.)
between separate runs.

Four tests were made by each of two operators to test the between-
operator variation within the technique described. The data secured is
reported in the Experimental Results section of this report.

EXPERIMENTAL RESULTS

See Table I, which is a copy of the data prepared for distribution
to committee members.

The relation of angle included by the two sides of the needle or
TABLE I
HARDNESS OF WAX AT ELEVATED TEMPERATURES

Effect of the angle of the penetrator on the extent of penetration

MOORE AND MUNGER 100 WAX

<table>
<thead>
<tr>
<th>Needle</th>
<th>Rate of Penetration</th>
<th>Average Penetration</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM D-5</td>
<td>65 61 65 61</td>
<td>63</td>
</tr>
<tr>
<td>S. O. D.</td>
<td>56 54 54 54</td>
<td>55</td>
</tr>
<tr>
<td>20° Cone</td>
<td>33 34 34 34</td>
<td>34</td>
</tr>
<tr>
<td>30° Cone</td>
<td>27 26 26 27</td>
<td>27</td>
</tr>
<tr>
<td>Shore &quot;A&quot;</td>
<td>32 28 35 33</td>
<td>32</td>
</tr>
<tr>
<td>Operator 1</td>
<td>32 28 35 33</td>
<td>32</td>
</tr>
<tr>
<td>Operator 2</td>
<td>35 40 38 45</td>
<td>40</td>
</tr>
<tr>
<td>Shore &quot;D&quot;</td>
<td>3 4 5 5</td>
<td>4</td>
</tr>
<tr>
<td>Operator 1</td>
<td>3 4 5 5</td>
<td>4</td>
</tr>
<tr>
<td>Operator 2</td>
<td>5 9 6 8</td>
<td>7</td>
</tr>
</tbody>
</table>

Test Temperature: 100.00 ± 0.06°F.

SHELL P - 417 - 410 WAX

<table>
<thead>
<tr>
<th>Needle</th>
<th>Rate of Penetration</th>
<th>Average Penetration</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM D-5</td>
<td>40 39 40 40</td>
<td>40</td>
</tr>
<tr>
<td>S. O. D.</td>
<td>38 39 38 39</td>
<td>39</td>
</tr>
<tr>
<td>20° Cone</td>
<td>23 23 23 24</td>
<td>23</td>
</tr>
<tr>
<td>30° Cone</td>
<td>17 17 18 17</td>
<td>17</td>
</tr>
<tr>
<td>Shore &quot;A&quot;</td>
<td>35 35 40 38</td>
<td>37</td>
</tr>
<tr>
<td>Operator 1</td>
<td>35 35 40 38</td>
<td>37</td>
</tr>
<tr>
<td>Operator 2</td>
<td>50 45 50 48</td>
<td>48</td>
</tr>
<tr>
<td>Shore &quot;D&quot;</td>
<td>4 4 6 5</td>
<td>5</td>
</tr>
<tr>
<td>Operator 1</td>
<td>4 4 6 5</td>
<td>5</td>
</tr>
<tr>
<td>Operator 2</td>
<td>5 8 5 6</td>
<td>6</td>
</tr>
</tbody>
</table>

Test Temperature: 115.00 ± 0.08°F.

<table>
<thead>
<tr>
<th>Needle</th>
<th>Rate of Penetration</th>
<th>Average Penetration</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM D-5</td>
<td>126 114 124 115</td>
<td>120</td>
</tr>
<tr>
<td>S. O. D.</td>
<td>105 105 104 105</td>
<td>105</td>
</tr>
<tr>
<td>20° Cone</td>
<td>71 71 72 73</td>
<td>72</td>
</tr>
<tr>
<td>30° Cone</td>
<td>57 57 57 57</td>
<td>57</td>
</tr>
<tr>
<td>Shore &quot;A&quot;</td>
<td>18 14 16 15</td>
<td>16</td>
</tr>
<tr>
<td>Operator 1</td>
<td>18 14 16 15</td>
<td>16</td>
</tr>
<tr>
<td>Operator 2</td>
<td>20 19 18 20</td>
<td>19</td>
</tr>
<tr>
<td>Shore &quot;D&quot;</td>
<td>1 0 0 1</td>
<td>1</td>
</tr>
<tr>
<td>Operator 1</td>
<td>1 0 0 1</td>
<td>1</td>
</tr>
<tr>
<td>Operator 2</td>
<td>0 1 1 0</td>
<td>1</td>
</tr>
</tbody>
</table>

Test Temperature: 115.00 ± 0.06°F.

Calculated angle included by two sides of needle or cone:

<table>
<thead>
<tr>
<th>Needle</th>
<th>Calculated Angle</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM D-5</td>
<td>9.1 ± 0.2°</td>
</tr>
<tr>
<td>S. O. D.</td>
<td>8.9 ± 0.04°</td>
</tr>
</tbody>
</table>
Figure 1.

Angle vs. Penetration

KEY:
- ASTM D-5 Needle
- S.O.D. Needle
- 20° Cone
- 30° Cone

Shell P-417-410 Wax
Test Temperature - 115.00°F ± 0.08°F

Moore & Munger Wax
Test Temperature - 115.00°F ± 0.08°F

Moore & Munger Wax
Test Temperature - 100.00°F ± 0.10°F

Shell P-417-410 Wax
Test Temperature - 100.00°F ± 0.10°F

Penetration depth (1/10 millimeter)

Angle included by two sides of needle or cone (degrees)
cone versus the depth of penetration are plotted in Figure 1; for each wax and test temperature. See Figure 1.

SUMMARY AND CONCLUSIONS

We have completed a study of the testing of hardness of wax at elevated temperatures, using a Precision penetrometer and varying the angle of the needle or penetration cone. Also included is an evaluation of two Shore Durometer hardness testers.

The cones used were approximately one inch long and had shafts to fit the penetrometer. The weight of each of the cones was greater than the regular D-5 needle. Special weights were used to maintain a constant weight of 100 grams (including the weight of the needle or cone) for each penetrator. The tips of the cones were ground flat as in the case of the ASTM D-5 needle. The actual angles of the penetrators were calculated from the physical dimensions measured with a micrometer and a microscope fitted with a measuring square. Two-inch molds were used to permit the use of larger cones.

The data we have compiled, although limited in scope, tend to bring out certain relationships which may aid in the development of a suitable test for the hardness of wax at elevated temperatures. There is good reason to believe that the hardness of wax varies as we go into the interior of wax specimens prepared according to the proposed method. The sharp deviation of the curves in Figure 1 and the photographic examination of the nature of the inside of wax specimens previously reported, would
tend to substantiate this belief. The greater the angle of the cone, the more consistent is the relationship between depth of penetration and the angle of the cone. The ASTM D-5 needle shows the greatest deviation, probably due to the short length of the conical section.

It would appear that the use of a 30° cone with the Precision penetrometer would probably lead to more consistent hardness values for wax tested at elevated temperatures. The 30° cone is the only penetrometer that did now show a reversal in the order of hardness of the two waxes in going from 100°F. to 115°F. The two waxes used in this study are the same as those used in the last round robin tests.

Results obtained with the Shore hardness testers are shown in Table I. The waxes at the higher temperatures are so soft that the maximum reading obtained with the Shore Hardness testers falls off too rapidly to allow different operators to get consistent results.

The conclusions above substantiate our previous thoughts that attention should be turned to the more fundamental problem of first analyzing the wax specimens instead of trying to grope for a specific means for testing something that is not uniform. First of all, we must define clearly what we are seeking. Are we interested in the average hardness of the wax mass, or are we not more concerned with the property of the wax as related to its end use? Aside from the use of wax in candles, etc., there are few applications where a great thickness of wax is required. Therefore, would not a hardness test of the wax near the surface of the specimen be of importance? Or, on the other hand, would it not be important to prepare
specimens which would be more uniform throughout in order to minimize variations which have been shown to exist inside the specimens prepared according to the present method? If a 30° cone is able to give more consistent results than a 10° needle, what might be expected of a 180° cone or flat surface?

Answers to some of the above questions would give a sound basis on which to proceed in developing a test for the hardness of wax at elevated temperatures.

In addition to the more general conclusions noted above, it should be stated that we feel that the method of conditioning and testing directly in a large volume constant temperature bath, while greatly simplifying the required equipment, also serves to give better over-all temperature control than pumping of water from the main to testing bath as described in the proposed method.

It was also observed that careful cleaning of the penetrator surfaces was facilitated by removing the needle from the test bath and cleaning with a solvent between consecutive penetrations.
ROUND-ROBIN BLOCKING POINT DETERMINATION

INTRODUCTION

The following is a report of the various improvements that have been made on a laboratory waxer and blocking point apparatus prior to participating in the current round-robin testing program for the determination of the blocking point of paraffin wax. It will include a report of work in ascertaining the repeatability of the proposed method within this laboratory and, finally, a report of final data reported to the committee chairman.

LABORATORY WAXER IMPROVEMENTS

A D.C. Thymotrol Drive Unit has been installed as a controlled speed drive unit on the laboratory waxer. The drive is a BC-74 full-wave rectifier type, 1/2 h.p. at full speed d.c. motor. Current is drawn from two electronic rectifier tubes guaranteed for 3000 hr. by the General Electric Company. Torque is constant and is listed at 24.2 oz.-ft. maximum. Horsepower varies directly with speed. The standard speed range is 1725-86 r.p.m. for a 20:1 speed ratio. Speed is adjusted by turning a 2-watt, 10,000-ohm potentiometer with 100 markings on a 320° dial.

Eighty-six feet per minute, the standard minimum operating speed of the drive, was considered too fast for waxing operations requiring low speed. A 0.75:1 V-belt ratio was used to drive the input
shaft of a 9.2:1 Boston gear reducer. The rewind shaft is driven by the output shaft of the gear reducer by a 2.7:1 V-belt and sheave arrangement. The pulley sizes are thus motor, 4 inches; input shaft of reducer, 3 inches; output shaft of reducer, 2-1/2 inches; and rewind roll, 6-3/4 inches. The ratio of the speed of the Thymotrol shaft to rewind roll is thus 18.6:1. The r.p.m. delivered to the rewind roll was obtained with a one-second sweep electric timer and a commercial Productimeter (one division per revolution) throughout the full range. Distinct dial settings gave reproducible speed in r.p.m. within 1 r.p.m. (the finest division of the Productimeter) throughout the entire range.

R.P.M. CALIBRATION--THYMOTROL

<table>
<thead>
<tr>
<th>Dial Setting</th>
<th>R.P.M. Rewind Shaft</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.2</td>
</tr>
<tr>
<td>10</td>
<td>9.5</td>
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<tr>
<td>20</td>
<td>19.5</td>
</tr>
<tr>
<td>30</td>
<td>30.0</td>
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<tr>
<td>40</td>
<td>38.5</td>
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<tr>
<td>50</td>
<td>45.5</td>
</tr>
<tr>
<td>60</td>
<td>53.0</td>
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<td>70</td>
<td>59.5</td>
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<tr>
<td>80</td>
<td>67.5</td>
</tr>
<tr>
<td>90</td>
<td>76.0</td>
</tr>
<tr>
<td>100</td>
<td>82.0</td>
</tr>
</tbody>
</table>

These data were graphed. The speed required (r.p.m.) to maintain constant web speed (feet per minute) throughout the range of rewind roll build-up diameters was calculated on the basis of standard formulas for the circumference and web delivery rate to the rewind roll. The necessary dial settings were then interpolated from the above mentioned graph. Actual web speeds were specified in the test method on the basis of the
interval of time, 0.5 sec., required for the web to travel from the
last doctor rod to water bath surface, a distance of 9 inches. The
calculated speed is thus 90 feet per minute. The adjustments mentioned
were made in intervals of 1/2-inch roll diameter increase, during a
continuous waxing operation. The maximum error calculated at the
highest web speed, 90 feet per minute, at minimum roll size (3-1/2 inch
diameter) was 9%. This error diminished to about 6% for normal waxing
procedures and could be further diminished by adjusting the speed at
smaller intervals of roll diameter.

CONSTANT SPEED CALIBRATION—THYMOTROL

Calculated Speed: 90 Ft. Per Minute

<table>
<thead>
<tr>
<th>Roll Diameter (inches)</th>
<th>Dial Setting to Obtain 90 Ft. Per Minute</th>
<th>Nominal Roll Size (inches)*</th>
<th>Calculated r.p.m.</th>
</tr>
</thead>
<tbody>
<tr>
<td>3-1/2</td>
<td>--</td>
<td>3-1/2</td>
<td>98.3</td>
</tr>
<tr>
<td>4</td>
<td>--</td>
<td>3-3/4</td>
<td>86.0</td>
</tr>
<tr>
<td>4-1/2</td>
<td>92</td>
<td>4</td>
<td>76.4</td>
</tr>
<tr>
<td>5</td>
<td>82</td>
<td>4-1/4</td>
<td>68.8</td>
</tr>
<tr>
<td>5-1/2</td>
<td>73</td>
<td>4-1/2</td>
<td>62.5</td>
</tr>
<tr>
<td>6</td>
<td>66</td>
<td>4-3/4</td>
<td>57.3</td>
</tr>
<tr>
<td>6-1/2</td>
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<td>5</td>
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</tr>
<tr>
<td>7</td>
<td>55</td>
<td>5-1/4</td>
<td>49.1</td>
</tr>
<tr>
<td>7-1/2</td>
<td>51</td>
<td>5-1/2</td>
<td>45.9</td>
</tr>
<tr>
<td>8</td>
<td>47</td>
<td>5-3/4</td>
<td>43.0</td>
</tr>
<tr>
<td>8-1/2</td>
<td>43</td>
<td>6</td>
<td>40.5</td>
</tr>
<tr>
<td>9</td>
<td>40</td>
<td>6-1/4</td>
<td>38.2</td>
</tr>
<tr>
<td>9-1/2</td>
<td>37</td>
<td>6-1/2</td>
<td>36.2</td>
</tr>
<tr>
<td>10</td>
<td>35</td>
<td>6-3/4</td>
<td>34.4</td>
</tr>
</tbody>
</table>

*The size of the roll procured by sighting along one side of the rewind
roll using a rule so oriented as to give a 3-1/2 reading for a 3-1/2 inch
roll diameter.

Thymotrol has proven to be a very satisfactory drive for the
waxing operations attempted thus far in this laboratory.
A refrigerated bath was temporarily assembled to furnish water to the water-dip tank. The components (compressor, coil, etc.) will later be moved to a more permanent insulated cabinet.

The cooling components consisted of a 1/4-h.p. freon compressor fitted with an expansion valve and pressure bulb and 50 feet of copper cooling coil. The temperature control is accomplished with a Lanco pressure differential switch.

Water is pumped from the refrigerated bath to the water-dip tank by a 1/8-h.p. motor driven Eastern rotary pump, Model DB. The water flows through the baffled water tank, out the low-head overflow and into a pail at floor level, from which it is returned to the refrigerated bath by a small bilge pump. The flow rates were balanced using a simple hose clamp on the line between the Eastern pump and the water-dip tank. Flow rate was adjusted so that the bilge pump was just able to return the overflow volume to the tank. Two and one-half gallons per minute (limited by the capacity of the bilge pump) flow rate should be considered maximum with present equipment. Coating a web with about 20 lb. per ream at maximum speed caused the water temperature to rise approximately 1.5°F., which is well within the variation allowed by the method. The low-head overflow in the dip tank will remove a maximum of about 5 gallons per minute (3 ft. external head), so that this capacity could be utilized by a better system of returning the fluid to the refrigerated bath.
BLOCKING PLATE IMPROVEMENTS AND CALIBRATION

BLOCKING PLATE RELAY SYSTEM: All of the components of the electronic equipment necessary to heat, control and record the temperature of the blocking plate were moved into a 15 by 9 by 7-inch metal utility cabinet. A schematic drawing of the electronic circuit follows.

115 V. J
I

A - Multitap Transformer
B - 600-Watt Cartridge Heater
C - Cutler Hammer 51B Relay
115 Volt, N/C, SPDT.
D - Sensative Fenwell Thermostat, No. 17300.
E - Pilot Lamp
F - Operational Pen - Pot.
G - "General" Thermostats
Model A - 200
H - R 19A Laboratory Relay
I - Brown Recording Potentiometer.
J - Six Position Sweep Switch
"Alnor" One R.P.H.

Schematic Drawing—Electrical Components

Note that electronic recording potentiometer, heater, etc., can be plugged directly into the utility cabinet allowing a highly compact system. The single-pole, double-throw, Cutler-Hammer relay was installed to provide the cartridge heater of the blocking plate with either of two voltages selected from the multitap transformer by means of two rotary
power switches. The voltages selected are dependent upon the span of the gradient required with consideration to minimizing the voltage difference. This method allows superior control of "lag" and "overshoot" as compared with the limited selection of voltages allowed by a single-throw relay—zero and selected. Fifty-five and 95 volts, respectively, to the high and low sides of the relay would seem to be optimum condition for the gradient established for the round-robin testing program.

Twenty-four hundred milliliters of water per minute were passed through the blocking plate to provide the proper low-end temperature. This condition gives a 3.9°F. gradient per inch, with a 139°F. maximum temperature. Relay action as activated by the Fenwell thermoswitch was 40% on, 60% off, in one complete cycle for 2-minute interval. The temperature variation at the 8 cm. position as measured by the recording potentiometer was 0.4°C. A 0.8°F. per inch gradient, 134°F. maximum, established for the narrow blocking point range required 0 and 55 volts, respectively, and no water. Relay action as activated by the Fenwell thermoswitch was 35% on, 65% off, in one complete cycle per 4-minute interval.

A standard laboratory relay and two general thermostats have been installed as a safety device. The thermostats mounted along the edge of the plate on the cold and hot end will discontinue the power source to the multitap transformer if the temperature should rise appreciably at the lower end of the plate during the test period, as during failure of the well water used to cool the lower temperature portion of the plate.
An attempt was made to utilize a single-pole, double-throw, Silent Power Relay (American Instrument Company) as the switch between the low and high sides of the multitap transformer. It consists of two single-pole, single-throw mercury switches rotated by the magnetic field of the soft iron coil. The $8^\circ-10^\circ$ angle of rotation secured was not enough to give a definite break of one switch before the other switch made contact. Also, there was some tendency for the mercury to splash within the switch making this adjustment even more difficult. The use of the unit was discontinued in favor of the Cutler-Hammer relay because its operation was considered safer for our purposes.

IMPROVED COOLING METHODS: Previous experience with the blocking plate has served to demonstrate that an appreciable gradient exists in a transverse direction across the blocking plate at points at equal distance from the hot end of the plate. As a means of correcting this difficulty, the water piping arrangement to the plate was changed so that the water is brought in at the center of the base of the plate. A spiral aluminum ribbon was installed in the water passage (a hole drilled the full width of the plate and tapped for 3/8-inch pipe fittings) to give optimum fluid contact with the surfaces of the passage. The water thus passes from the center of the plate to either edge and is exhausted through either of two needle valves to a drain located at floor level. It was found necessary to install a Fulflo commercial water filter in the inlet piping to maintain flow control with the needle valve over the 17-hour test period. Debris in the well water is
sufficient to fill one of the cotton twine filters in a 24-hour period, after which the filter is replaced as routine procedure. The continuous flow rate, as low as 1200 ± 120 ml. per minute, was maintained over the 24-hour operating period from each side of the blocking plate.

It is necessary that the cooling water be maintained at a minimum rate of flow with consideration to the effect on temperature stability and transverse gradient in order to keep the average temperature gradient of the plate within the 1.5 to 4.0°F. per inch requirement of the proposed method. Higher rates of flow cool the lower end of the plate to a lower temperature, and since the high temperature portion of the plate must be maintained above the expected blocking point of materials tested, the average gradient might then exceed the limit of the proposed method.

ALUMINUM SEPARATOR STRIPS: In connection with another project, the possibility of wax contamination from one blocking specimen to another between the sheets of glycine appeared to be a problem. We decided to fit our blocking plate with separator strips similar to those illustrated in Figure 1 of the proposed method. Seven 3/16-inch thick, 1/2-inch wide extruded aluminum strips, 24 inches long, were glued to the surface of the blocking plate using Shell Oil Company's Epon adhesive No. 8. They were oriented so that six 1-1/4 inch wide blocking areas were formed leaving the outside of the last separator strips 1/2 inch from the edge of the plate. One-inch strips of conditioned (one week at 73°F., 50% relative humidity) sulfite bread wrapper paper were cut utilizing the
slitting attachment of the laboratory waxer for use in separating individual waxed specimens on the blocking plate.

Not only has the possibility of cross contamination been virtually eliminated, but the blocked strips appear less wrinkled than by the previous method. As anticipated, however, the rubber pads are more quickly contaminated with wax requiring more frequent replacement.

CALIBRATION FOR ROUND-ROBIN TESTING: Standardization of apparatus, paragraphs 7b and c of "Proposed TAPPI Method for Blocking Point of Paraffin Wax," transmitted with a letter to committee members dated November 10, 1954, suggests a method of calibration for a blocking point apparatus and recorder. A "test" couple is immersed in a beaker of water together with a calibrated thermometer. The water is carefully heated to 110°F. The mv. of the test couple is measured with a Type-K potentiometer, and the deviation from the classic value noted as a correction factor. The test couple is then placed directly over the couples in normal position along the full gradient of the plate, covered with the sponge pads and weighted bars, aged, and the test couple mv. equivalent (Type-K potentiometer) compared with the recorded couple. If this difference is more than 1°F., less the correction factors of the test couples, certain procedures are prescribed.

The suggested method was used in the final calibration of the blocking plate. Experimental results follow:
CALIBRATION OF PEENED 30-GAUGE COPPER CONSTANTAN THERMOCOUPLE

Primary Reference: NBS Certified Thermometer, Wesco 39 06 149, -5 to 105°C. in 0.1°C.

Secondary Reference: Calibrated Wilkens-Anderson Thermometer, -5 to 104°C. in 0.1°C.

<table>
<thead>
<tr>
<th>Secondary Reference, Temperature of Water (°C.)</th>
<th>Total Correction to Thermometer (°C.)</th>
<th>Corrected Temperature (°C.)</th>
<th>Mv. Type K Potentiometer (°C.)</th>
<th>Correction to Thermocouple (°C.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>43.26</td>
<td>+0.02</td>
<td>43.28</td>
<td>1.734</td>
<td>43.00</td>
</tr>
</tbody>
</table>

CALIBRATION OF BLOCKING PLATE—RECORDER

<table>
<thead>
<tr>
<th>Distance Hot End (cm.)</th>
<th>Corrected Test Couple (°C.)</th>
<th>Recorded Couple (°C.)</th>
<th>Correction (°C.)</th>
<th>Correction (°F.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>58.6</td>
<td>58.9</td>
<td>-0.3</td>
<td>-0.6</td>
</tr>
<tr>
<td>53</td>
<td>21.3</td>
<td>21.0</td>
<td>+0.3</td>
<td>+0.6</td>
</tr>
</tbody>
</table>

The blocking plate and recorder were thus considered calibrated for the current round-robin blocking point determination.

PROCEDURE

No difficulty was encountered in waxing test specimens for the current round-robin.

Special attention was given to the possibility of cross contamination of waxes in the waxer during a 4-day program of coating two different waxes. The second of the two waxes coated on a particular day
was left in the wax tank and became the first wax coated on the next regular day of waxing. This minimized the number of cleanups required thus minimizing the possibility of contamination. The waxer, including all parts that contact wax, was cleaned by wiping with a clean cloth while hot, wiping with a cloth saturated with toluene, and finally wiped again with a clean dry cloth. Toluene was poured into the pin bearings of the wax-dip roll to assure that all wax was removed. The hot Mayer rods and holders were immersed in toluene and then wiped dry to assure clean surfaces.

TESTING: In addition to testing the blocking strips with a rather wide gradient ($3.8^\circ F$ per inch of blocking plate length), we decided to make a brief study of the effect on the character and precision of selection, of the picking and blocking point secured, by varying the average temperature gradient between roughly the limits specified in the method. To accomplish this, we heated the blocking point apparatus in the normal manner but allowed the other end of the blocking plate to stabilize in a room controlled at $73^\circ F$. This gave an average temperature gradient of per $0.8^\circ F$ inch throughout the effective blocking area.

EXPERIMENTAL RESULTS

The results reported to the committee chairman follow: See Tables I and II.
TABLE I.

RESULTS OF TAPPI-ASTM ROUND ROBIN BLOCKING TESTS

Test conditions are those established at October 1954 meeting and outlined in Mr. H. F. Hitchcox's letter of January 4, 1955.

(Six specimens prepared and tested in each run.
Speed of waxing: 90 ft. per minute.
Specimens were conditioned 24-30 hours at 73°F., 50% R.H.)

<table>
<thead>
<tr>
<th>Run</th>
<th>Picking, °F</th>
<th>Blocking, °F</th>
<th>Date of Test</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sinclair PM-362</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>103</td>
<td>105</td>
<td>2-2-55</td>
</tr>
<tr>
<td>2</td>
<td>103</td>
<td>105</td>
<td>2-3-55</td>
</tr>
<tr>
<td>3</td>
<td>102</td>
<td>104</td>
<td>2-8-55</td>
</tr>
<tr>
<td>4</td>
<td>103</td>
<td>105</td>
<td>2-11-55</td>
</tr>
<tr>
<td></td>
<td>Esso W-4748</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>121</td>
<td>123</td>
<td>2-2-55</td>
</tr>
<tr>
<td>2</td>
<td>122</td>
<td>124</td>
<td>2-3-55</td>
</tr>
<tr>
<td>3</td>
<td>122</td>
<td>125</td>
<td>2-8-55</td>
</tr>
<tr>
<td>4</td>
<td>121</td>
<td>124</td>
<td>2-11-55</td>
</tr>
</tbody>
</table>
TABLE II.

EFFECT OF TEMPERATURE GRADIENT ON BLOCKING POINTS

<table>
<thead>
<tr>
<th>Run</th>
<th>Picking, F₀</th>
<th>Average Deviation Between Strips</th>
<th>Blocking, F₀</th>
<th>Average Deviation Between Strips</th>
<th>Date of Test</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>121.2</td>
<td>0.57</td>
<td>123.3</td>
<td>0.20</td>
<td>2-2-55</td>
</tr>
<tr>
<td>2</td>
<td>122.0</td>
<td>0.35</td>
<td>123.7</td>
<td>0.27</td>
<td>2-3-55</td>
</tr>
<tr>
<td>3</td>
<td>121.6</td>
<td>0.17</td>
<td>124.6</td>
<td>0.17</td>
<td>2-8-55</td>
</tr>
<tr>
<td>4</td>
<td>121.3</td>
<td>0.28</td>
<td>123.5</td>
<td>0.10</td>
<td>2-11-55</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td>121.5</td>
<td>123.8</td>
<td>0.19</td>
<td></td>
</tr>
</tbody>
</table>

WAX: Esso W-4748  Blocking Plate Gradient 3.8 F. degrees per inch

WAX: Esso W-4748  Blocking Plate Gradient 0.8 F. degrees per inch

1   | 123.0       | 0.23                             | 124.6       | 0.13                             | 2-9-55       |
SUMMARY AND CONCLUSION

We have completed the blocking point determinations according to the procedure outlined by Mr. H. F. Hitchcox in his letters of November 10, 1954 and January 4, 1955.

Blocking points were determined on four separate days, following the specified conditioning time, for Esso W-4748 and Sinclair PM-362. See Table I.

In addition to the above testing, we decided to make a brief study of the effect of varying the blocking plate temperature gradient on the picking and blocking points observed. The blocking points observed at a gradient of 3.8°F. per inch are compared in Table II with blocking points observed at a lower gradient of 0.8°F. per inch (achieved by heating one end of our "Marathon type" blocking point apparatus and allowing the other end of the blocking plate to stabilize in a room controlled at 73°F.).

In addition to the expected results of increased distance between picking and blocking points, we noticed that the areas of film disruption appeared more diffuse, causing greater difficulty in selecting the picking and 50% blocking points. However, the precision with which we could select the picking and blocking points proved to be about the same for either gradient condition. The lower gradient gave slightly higher picking and blocking points; however, the number of specimens tested was too small to base any statistical significance on this difference. The use of the 3.8°F. gradient gave definitely sharper picking and blocking points for the wax tested.