THE DEVELOPMENT OF TECHNIQUES FOR MEASURING
THE HARDNESS OF PETROLEUM WAX

INTRODUCTION

Section II, TAPPI-ASTM Technical Committee on Petroleum Wax is currently investigating various methods for measuring the hardness of softer waxes. At a previous committee meeting (reported in Memo to Files, Section II, dated October 5, 1955) members were invited to choose from a list of promising methods for investigation in their own laboratories. Using the methods chosen, members were to investigate the hardness of certain waxes over a given temperature range and report the results to the committee, which would select the most promising method for future round-robin testing.

We selected and have completed testing with two of the test instruments listed—the Parallel Plate Plastometer and a Modified Needle Penetrometer—used to determine the force necessary to obtain a fixed penetration. We investigated two methods of testing with the Parallel Plate Plastometer. One of these methods proved to be quite time-consuming and little was done with it at this time. Complete data was secured with the other method. It should be pointed out that the calculations involved in securing the viscosity of wax by either of the parallel plate methods were quite long, but have the advantage of giving the hardness of the
material in fundamental units, i.e., poise. Measurement of the force required to obtain a fixed needle penetration proved to be quite straightforward and the repeatability was good.

The results have been reported to the committee chairman, along with photographs of our test apparatus and a drawing of the Penetrometer Adapter. Some interest has been shown in the Penetrometer technique, probably because of its simplicity and similarity to the present needle penetration method for wax at 77°F. As we were the only laboratory reporting on this method, the important aspect of reproducibility between laboratories is not known as it was for several of the other methods investigated in more than one laboratory.

The purpose of this report is to consolidate the various aspects of the investigation of wax hardness in our laboratory and report the status of participation in current wax testing committee activities.

WAXES TO BE TESTED

The three waxes to be tested were supplied by the Shell Oil Company and were designated B-201, B-202, and B-203. Waxes B-201 and B-202 are paraffin waxes, while B-203 is a microcrystalline wax. A letter from Mr. Stan Marple—representing Mr. G. P. Hinds, the Section II chairman, dated October 26, 1955, confirms our working assignment, specifies certain of the test conditions, and gives the following characterization of the waxes as determined in their laboratory.
NEEDLE PENETRATION OF WAXES FOR CO-OPERATIVE TESTING

Penetration, mm/10
(ASTM D-1321-55)

<table>
<thead>
<tr>
<th>Temperature, °F.</th>
<th>95</th>
<th>100</th>
<th>105</th>
<th>110</th>
<th>115</th>
</tr>
</thead>
<tbody>
<tr>
<td>B-201</td>
<td>40</td>
<td>110</td>
<td>150</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>B-202</td>
<td>-</td>
<td>-</td>
<td>31</td>
<td>65</td>
<td>133</td>
</tr>
<tr>
<td>B-203</td>
<td>-</td>
<td>50</td>
<td>72</td>
<td>123</td>
<td>190</td>
</tr>
</tbody>
</table>

In addition, melting points were determined so that the waxes could be poured for sample preparation under standard conditions. The cloud point has been found to give a quick, accurate estimate of the melting point for pure paraffin wax, though less accurate for microcrystalline waxes. The cloud points for these waxes were found to be: B-201, 55°C.; B-202, 60°C.; and B-203, 59°C. Using these temperatures as "expected melting points" for the standard melting point method, the following melting point results were secured:

<table>
<thead>
<tr>
<th>Wax</th>
<th>Melting Point, °C.</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>B-201</td>
<td>55.3</td>
<td>ASTM D 87-42</td>
</tr>
<tr>
<td>B-202</td>
<td>60.0</td>
<td>ASTM D 87-42</td>
</tr>
<tr>
<td>B-203</td>
<td>60.2</td>
<td>ASTM D 127-49</td>
</tr>
</tbody>
</table>

Waxes are to be poured for sample preparation at 30°F. (16.6°C.) above the melting point so that the pouring temperatures for these materials were 71.9, 76.5, and 76.8°C., respectively, for waxes B-201, B-202, and B-203.
METHOD OF SPECIMEN PREPARATION

Specimens were prepared for penetration testing according to the sample preparation and conditioning of sample sections of TAPPI Method T 639 sm-53, plus the changes indicated in a letter from Mr. W. J. Yates dated April 12, 1955. The specimen molds were also as described. The $75 \pm 4^\circ F.$ air-bath temperature was obtained by placing an air-circulating oven (external ports closed) into a $40^\circ F.$ upright refrigerator. The cold refrigerator temperature assured dynamic temperature control with the oven set at $75^\circ F.$ Temperature variation was about $\pm 2^\circ F.$, using a Fenwell regulator and R 19A laboratory relay with a 250-watt incandescent lamp heat source.

Two cylinders, assembled as they would be for filling with wax, are shown at the left of the test apparatus in Figure 1. Note that a sheet of thin aluminum foil is used, which prevents the wax from sticking to the plate. It is stripped off from the wax surface just prior to penetration with the needle. At the right are two poured cylinders ready for insertion to the test bath as shown at the bottom of the tank in Fig. 2. Two such specimens were prepared for each wax at each test temperature.

Reference to Fig. 3 will make the explanation of the parallel plate sample preparation more easily understood. All conditioning time and temperature specifications were according to the above TAPPI Method. The wax was carefully melted in a 6 x 1-inch test tube in a water bath and poured into the receiving cylinder assembled as shown at the right rear of Fig. 3. Three additional bottom plates are placed at different angles to show all sides of the test plates, just in front of the wooden rack.
The shank of the plates is turned downward and inserted into holes provided in the rack and the cylinder slipped over. The plates and cylinders are numbered as pairs which have been properly matched to give a leak-proof fit when assembled.

Just enough wax is poured from a large test tube to give a convex maniscus without overflowing the side of the cylinder. The wax specimen formed is one inch in diameter and about 1/2 inch thick. Wax temperature and mold temperature are carefully controlled during the pouring operation as specified in T639 sm-53.

The molds are left to cool for a minimum of one hour. They are then removed from the air bath and carried to the working area. A small mechanical press was used, if necessary, to force the shank of the plate up even with the bottom of the outside cylinder. The cylinder is then rested on its side, as shown in the foreground of Fig. 3, and a brass plate held against it to hold the specimen in place. A heated knife is used to cut off excess wax, leaving a 1/8-inch thick layer of wax on the test plate surface. Such a completed specimen is shown in front of the test apparatus in Fig. 3. The outside cylinder has been moved over the shank of the plate to avoid damaging the wax specimen on the plate surface. Some experience is necessary to heat the knife blade to just the right temperature. It is heated over a soft Bunsen burner flame. If the knife is too hot, the wax is melted away from the test plate, leaving the slab of wax on the test plate too thin; if the knife is too cool, excessive force is required to cut the wax, frequently breaking the wax away from the test plate. If the knife is
Figure 3.
properly heated, only four or five back and forth motions are required with the knife to cleanly cut off the excess of wax. The specimen and plate are then conditioned in the water bath until tested. Three specimens, ready for immersion in the test bath, can be seen on the left wall of the bath in Fig. 4. Since the test plates are made of steel, they rusted quite badly after long immersion in the water bath. For some of the first runs they were left in the water bath as long as twelve hours before they could be used in the normal sequence of testing. Later-run samples were left in the air bath for longer periods of time (4 to 6 hours) so that water-conditioning time could be maintained as near one hour as possible. This minimized rusting of the test plates. The wax slab tended to protect the critical top surface of the plate from rust damage in either case. Most of the rusting occurred along the cylindrical side and shank of the plates.

A different type of specimen was prepared from the same waxes for what we shall Case 2 parallel plate testing. The accessories used to prepare this type of specimen are shown in the left foreground of Fig. 3. Using the same pouring techniques as before, the wax was poured into a 2-inch long brass sleeve, 3/8-inch inside diameter, fitted with a steel piston. The piston was retracted about 1-3/4 inches prior to the pouring operation. After being allowed to cool in the air bath for a minimum of one hour, the assembly was removed and the piston pushed into the sleeve to extrude the solid wax. The hot knife was again used to cut off "pellets" of wax of the desired length. It was hoped that several 11/32-inch long "pellets" could be secured from a single casting operation, but sections cut from the first inch or more of wax extruded had a small hole in the center
which would make it very difficult to obtain the volume of such sections accurately. They were therefore discarded. The last 11/32-inch long plug (next to the piston) was, however, free of holes and was the portion used in the tests.

The 2-inch diameter plates used for this test are shown at the extreme left in Fig. 3. A reserve-water tube and specific gravity bottle is shown just to the right of the casting sleeve. The sample history from this point includes an hour, or whatever time necessary, in the specific gravity bottle immersed in the test bath, to obtain an accurate volume for the wax pellet, after which it is conditioned along with the two test plates a minimum of 1/2 hour in the water bath before testing.

A previous round robin conducted by the Wax Testing Committee tended to show that water bath conditioning time for similar waxes was not at all critical. Samples conditioned over a range of one to five hours in the water bath gave the same hardness penetration values. We do not believe that air bath conditioning time would be critical either, so that the specimens cast directly on the plate (Case 1) should be directly comparable regardless of the number of hours in either the air or the water bath. Some uncertainty exists, however, in comparing the samples prepared by the wax "pellet" technique because of the repeated heating-cooling sequence required to determine the volume of the sample and condition it to the final testing temperature. The hardness of such samples may not be directly comparable with those prepared for Case 1 testing. Further investigations would be required to clarify this point.
CONTROL OF TEST TEMPERATURE

All test specimens were conditioned in the test bath a minimum of one hour before testing. They were placed on a small 1/2-inch high brass shelf which rests on the bottom shelf of the test bath. Conditioning penetration samples may be seen in Fig. 2 at the bottom of the test bath. Two 7-1/2 by 9-1/2-inch thermopane windows installed in the front and rear panels of the water bath permit clear observation of the specimen and test apparatus below water level. This is essential in accurately placing the needle in penetration testing.

The operation of the constant temperature bath and the details of its construction have been previously described in an instruction manual, "Instructions for the Operation of the Constant Temperature Bath (I.P.C. No. E-2260)", currently filed in the department equipment file. It was necessary to bypass the external circulation pump (Dynapump) used to circulate the bath water proper. The vertical immersion circulation-type pump, also included in the bath, was used to circulate water under the bottom shelf of the bath which covers the cooling coils and heating elements. Using both pumps (room temperature 78°F.), bath temperatures continued to rise over a temperature range from 25°C. to 38°C. and above, without additional heat. Studying the characteristics of each pump separately, the Dynapump was found to be the offender, giving a raise in temperature from 35 to 37°C. over a one-hour period. Under these conditions it would be necessary to use the cooling components of the bath to maintain temperatures of 38°C. or lower. We decided to bypass the Dynapump and use an Eastern rotary pump (I.P.C. No. E-2044) to circulate the bath water, because the precision of temperature control is better using intermittent heat than intermittent cooling.
The thermometer used to indicate water bath temperature was a 0 to 100°C. Wilkins-Anderson thermometer, which has been standardized against a National Bureau of Standards certified thermometer (see Report No. 5). Ice Point and Characteristic thermometer corrections were made as indicated. Emergent Stem corrections were made using a nomograph prepared by the Precision Thermometer and Instrument Company, a copy of which is included as the next page of this report, after checking the accuracy of the nomograph at several temperatures. The thermometer was immersed to the 10°C mark.

A Lab Line Roto-Stat M 61766, mercury-to-platinum thermoregulator was used together with a "Precision" electronic relay control box for precise temperature control. The relay control box was modified by the addition of a switch, plug, socket and electric cord to reverse the control action of this type of thermoregulator for use with the constant temperature bath.

With this equipment, the maximum temperature variation over a temperature range from 95 to 115°C was 0.06°F., well within the ±0.2°F. allowed variation. No temperature variation could be detected with our thermometer between temperatures at the center, side, top, or bottom of the bath if proper emergent stem corrections were made. The rotary-dial setting feature of the Roto-Stat thermoregulator made it easy to make fine adjustments in the regular settings. Only a few minutes was required to set the regulator to a new test temperature and carefully zero it in on the exact temperature required. The ease of setting the regulator saves considerable time compared with the type formerly used (Report No. 11) where it was necessary to split drops of mercury to adjust the mercury capillary weight. The precision with which a given
Glass thermometers are designed for use under a particular condition of immersion, i.e., the depth to which the instrument is placed in the material under investigation. A "total immersion thermometer is calibrated for use under conditions where the instrument is immersed to the point of reading. When a total immersion thermometer is used at an immersion less than to the point of reading, a correction must be made to the observed reading of the thermometer. The nomograph above is used to determine this correction.

For example, a total immersion thermometer range 0 to 100°C. is used at an observed temperature of 80°C. but immersed only to the level of 20°C. on the thermometer stem. The average stem temperature between 20°C. and 80°C. is found by placing the bulb of a second thermometer at the middle of the exposed stem (i.e., at the level of 50°C) and is determined to be 40°C. To find the emergent stem correction, lay a straight edge between 60° on the left-hand scale (i.e., the difference between 80° and 20°) and 40° on the right-hand scale (i.e., the difference between 80° and 40°) and read the correction from the proper center scale (i.e., 0.38°C). The corrected observed reading is 80.38°C.

Corrections determined where observed reading is higher than observed average stem temperature are added to the observed reading. Where the observed reading is lower than observed average stem temperature, corrections are subtracted from the observed reading.

temperature may be obtained or repeated is also improved because of the fine adjustment feature of the regulator. The sensitivity of the new regulator is slightly less, but it is more than adequate for most constant temperature requirements.

TESTING WITH MODIFIED NEEDLE PENETROMETER

Recent attempts by the Committee to measure the hardness of softer waxes, using the ASTM D-5 needle to determine the depth of penetration under a standard load, demonstrate the poor repeatability and even poorer reproducibility of such a method. This is not surprising since the wax specimens themselves have been shown to contain void areas and probably vary in hardness as we go into the interior of the wax specimen.* The short length of the conical section of the needle, 5.3 mm. by actual measurement, compared with the depth of penetration sometimes as great as 35 mm. is another possible reason for the observed variation.

A test which would measure the force required to obtain a depth of penetration not greater than the length of the conical section of the needle should give improved results. In our tests, the hardness of wax at each temperature was reported as the force required to obtain a needle penetration of 40-tenths of a mm. This degree of penetration was selected as a convenient value, giving a safe margin to 53-tenths of a mm. where the taper of the ASTM needle runs out.

The test instrument is a "Precision" Penetrometer No. 1093. It consists of a sturdy base with spirit level for careful leveling in the test bath and an upright rack-and-gear arrangement, with screw and sliding blocks for fine adjustment, for raising and lowering a housing which contains

* Report No. 11 discusses and presents evidence of this effect.
a precision ground shaft, bearings to guide the shaft, and a clutch.
The shaft and needle weigh about 50 grams. Two additional weights are
provided (50 and 100 grams) which slip over the top end of the shaft.
The lower end of the shaft is fitted with a chuck for attachment of the
needle.

A separate plunger is pushed down by hand to contact a collar on
the needle shaft. A precision gear-and-rack arrangement moves the dial
pointer, indicating the distance traveled by the needle shaft from its
zero position. The dial plate is graduated into 380 divisions so that one
revolution corresponds to a 0 to 380 tenths of a mm. range of needle-shaft
travel. When the needle is raised to its top or zero position and the tip
of the needle lowered to that it just touches the surface of the wax specimen
using the coarse and fine adjustment dials, the instrument is ready to
accurately measure the depth of penetration. The depth chosen for this inves-
tigation (4 mm.) corresponds to a dial reading of 40, while the end of the
conical section of the needle will be given by a dial reading of 53.

The instrument is commonly used to indicate the depth of needle
penetration under defined conditions of load, time, and temperature. For
our investigation it was necessary to modify the penetrometer so that the
load could be varied over a wide range to obtain a fixed depth of penetration
with waxes that are widely different in hardness. Since the waxes are expected
to be very soft at higher temperatures, it will be necessary at times to use
loads less than 50 grams, which is the weight of the shaft and needle. Forces
considerably in excess of 50 grams will be required at the lower temperatures.

An adapter was designed and built using a counterweight, string
and pulley system, so that loads could be applied in a continuous range from 0 to 350 grams. The adaptor consists of a piece of 1/8-inch aluminum plate, bent at the top and fitted with two pin-bearing V-pullies. The adaptor is bolted securely to the body of the penetrometer in such a way as not to interfere with normal use of the instrument. The pullies carry a cotton line, attached with a wire clip to the penetrometer shaft at one end and to a small polyethylene bucket at the other end. It is essential that the bucket be of sufficient size to carry 50 grams when filled with water and to weigh as little as possible when empty. This allows a nearly continuous range of weight to be applied to the wax surface by the penetrometer needle. Several new weights, similar to those made for previous testing (Report No. 11) were used to increase the range from 50 to 350 grams. A drawing of the adaptor is included in this report.

Modification of the adaptor, as shown, will probably be required for use with other penetrometers, but we believe that it should be simple to construct a device of this type to counterweigh the shaft of most penetrometers in common use. It is only essential that the adaptor subtract a portion of the needle and shaft weight in as nearly a friction-free manner as possible. In our adaptor this is accomplished by careful centering of the groove of the pulley over the shaft and using pin-bearing type pullies.

Calibration: In order to obtain accurate values for the force applied by the needle to the wax sample, it was necessary to calibrate the instrument. This was done by dropping a second line to the penetrometer shaft through the hole provided in the adaptor and obtaining the weight of the needle and shaft with a laboratory beamed balance (Ohaus, 0.1 g. finest division), being careful that the penetrometer was carefully plumbed below the
ADAPTOR TO BE MOUNTED ON "PRECISION PENETROMETER" NO. 1093
FOR DETERMINING WAX HARDNESS BY MEASURING THE FORCE
REQUIRED TO OBTAIN A CONSTANT NEEDLE PENETRATION
balance. This was repeated, using a series of counterweights—obtained by adding or removing water from the polyethylene bucket—to reduce the effective weight of the needle shaft. A calibration curve was established, over a range from approximately 0 to 50 g., by plotting the counterweight used against the weight determined by the balance. Separate calibration values were obtained for the force required to just allow the shaft to move upward or downward when released by the penetrometer clutch. The forces for "downward" movement represent the actual test condition and were the values used in the final calibration. Higher forces were obtained by adding additional weights to the penetrometer shaft. The following results were obtained:

<table>
<thead>
<tr>
<th>Counterweight, g.</th>
<th>Needle-to-Wax Force, g.</th>
<th>Interpolated from graph assuming weight of poly-ethylene bucket and water balance to just allow shaft to move downward</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of poly-ethylene bucket and water</td>
<td>Weight indicated by balance to just allow shaft to move downward</td>
<td>0</td>
</tr>
<tr>
<td>1.9</td>
<td>46.5</td>
<td>46.3</td>
</tr>
<tr>
<td>5.0</td>
<td>43.1</td>
<td>43.0</td>
</tr>
<tr>
<td>10.0</td>
<td>38.1</td>
<td>37.8</td>
</tr>
<tr>
<td>20.0</td>
<td>27.9</td>
<td>27.3</td>
</tr>
<tr>
<td>46.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
</tbody>
</table>

The weight of the needle and shaft alone is quite difficult to obtain directly, requiring disassembly of the apparatus to remove the shaft. It can be calculated, however, from the data obtained. We see from the table above that a force of 48.1 g. was required to just allow the shaft to move downward. 51.5 g. was required to just allow it to move upward. The
The weight of the needle and shaft should be the average of these two results—49.8 g., which is well within the 50 ± 0.55 g. weight variation allowed by the method. Using this value for the weight of the needle and shaft, the friction between the penetrometer bearings and the shaft is equivalent to 1.8 g. This value should be constant throughout the full range of calibration, if the groove of the pulley is carefully centered over the shaft.

As we add weight to the polyethylene bucket, the friction occurring in the pin bearing-pulley counterweight system becomes a factor. The maximum counterweight which would just allow the shaft to fall was 46.0 g., so that total friction accounts for 3.8 g. The friction of the pin bearing-pulley counterweight system must therefore be equal to 2.0 g., using the maximum counterweight. The right-hand column in the table above gives the weight interpolated from a graph made by drawing a straight line between the weight determined for 0 and 46.0 g. counterweight, respectively. This is equivalent to assuming that the pin bearing-pulley friction will drop in a linear fashion with counterweight, reaching 0 when the counterweight is 0. We see from the table above that the difference between the linear and experimental values is small, reaching a maximum of 0.6 g. for 20.0 g. counterweight.

This gives us a greatly simplified method of calibration and is the calibration used to determine the force applied to the wax sample in this testing program. It is only necessary to obtain the force required to just allow the shaft to move downward, with no counterweight (using a beam balance or other suitable method) and with maximum counter-
weight (determined by successively adding water to the polyethylene bucket and weighing). A straight line is then drawn between these two points and intermediate values interpolated from the line. Such a calibration should compensate for both the friction between the penetrometer bearings and the shaft and the friction occurring in the pin bearing-pulley counterweight system.

Testing: See Fig. 2 for a picture of the apparatus installed in the test bath. Using the penetration values given by the supplier as a guide, the apparatus was adjusted to give the proper needle-to-wax force and a trial penetration made. More or less weight was added to the needle as indicated and another penetration made. This process was continued until at least two penetrations were secured in a range from 30 to 50-tenths of a millimeter, using only one of the conditioned specimens. The results were plotted on graph paper and the weight required for penetration of exactly 40 was then interpolated from the graph. Using this weight, three additional penetrations were made on the second specimen to confirm the test. If the penetrations obtained deviated by more than one from 40 or if the average of three penetrations deviated from 40, the values obtained were plotted with the previous data and a new value interpolated from the graph. If there were no such deviations, the weight required for 40 penetrations was recorded as a measure of the hardness of the wax at a given test temperature.

In many cases it was expedient to calculate the force rather than interpolate it from the graph. The following equation was used:
where:

\[ F_{P40} = \left( \frac{40 - P_L}{P_H - P_L} \times F_H - F_L \right) + F_L \]

\[ F_{P40} = \text{the force required for a penetration of 40 tenths of a millimeter, grams.} \]

\[ P_L = \text{penetration value nearest 40 but less than 40.} \]

\[ P_H = \text{penetration value nearest 40 but more than 40.} \]

\[ F_L = \text{force used to obtain } P_L \]

\[ F_H = \text{force used to obtain } P_H \]

After calculation of the force required, the proper shaft and counterweight were determined from the calibration. Additional weights were added to the Penetrometer if necessary to obtain the required weight. A 10-cc. syringe with needle was found convenient in adjusting the weight of the water added to the polyethylene bucket. The bucket was weighed to the nearest 0.1 gram on the beam balance, then attached to the string and pulley system as before. Evaporation of water from the bucket during the 5-minute period required for conditioning of the needle, caused loss of weight, so that it was necessary to reweigh the bucket between runs.

The duration of all penetrations was 5 seconds. The data obtained is given in Table I.

A plot of temperature vs. needle-to-wax force seems to give a good characterization of the wax as tested. Paraffin B-202 is much harder than B-201 at 95°F, but they appear about the same at 115°F. Microcrystalline wax B-203 softens in a nearly linear manner with temperature.
### TABLE I

Grams Force Required for ASTM D-5 Needle Penetration of 40-tenths of a Millimeter in 5 Seconds

<table>
<thead>
<tr>
<th>Test Temperature, °F</th>
<th>Shell-Wax</th>
<th>B-201</th>
<th>B-202</th>
<th>B-203</th>
</tr>
</thead>
<tbody>
<tr>
<td>95</td>
<td>B-201</td>
<td>128.1</td>
<td>330.1</td>
<td>112.9</td>
</tr>
<tr>
<td>95</td>
<td>B-202</td>
<td>132.2</td>
<td>321.0</td>
<td>110.8</td>
</tr>
<tr>
<td>100</td>
<td>B-203</td>
<td>33.3</td>
<td>257.9</td>
<td>74.7</td>
</tr>
<tr>
<td>105</td>
<td>B-201</td>
<td>25.2</td>
<td>163.3</td>
<td>42.4</td>
</tr>
<tr>
<td>110</td>
<td>B-202</td>
<td>23.8</td>
<td>62.3</td>
<td>19.3</td>
</tr>
<tr>
<td>115</td>
<td>B-203</td>
<td>20.8</td>
<td>24.9</td>
<td>7.4</td>
</tr>
<tr>
<td>115</td>
<td>B-201</td>
<td>20.9</td>
<td>26.9</td>
<td>7.3</td>
</tr>
</tbody>
</table>
Penetration values were obtained at one-day intervals at 95 and 115°F, starting with newly cast specimens. The repeatability appeared good, giving a maximum difference between runs of 7.4% for Wax B-202 tested at 115°F. The average difference was 2.8%.

TESTING WITH PARALLEL PLATE PLASTOMETER

Our prime references are:


Judging from the information given by the above operators, the parallel plate plastometer method should be applicable for the testing of wax under our conditions. Mr. Vaurio states that "the use of the Plastometer is comparatively simple .... (and that wax viscosity) in the neighborhood of 10^5 poises or higher may be measured." This method would have the advantage of giving the hardness of wax in fundamental units, i.e., poises.

The test instrument is used to press a sample of wax placed between two parallel plates. The distance between the plates is recorded as a function of time at constant force and temperature, and viscosity calculated from this data.

A preliminary run was made using Socony Vacuum 125-127 AMP Fully Refined Wax at 115°F, to determine the low-viscosity limit of testing. The viscosity was found to be 4 x 10^5 poises so that the method should be
satisfactory considering the limitations mentioned in Mr. Vaurio’s report.

Theoretical: Dienes’ article includes a rigorous derivation of a formula for the calculation of viscosity from parallel plate data. Starting with the general equation for motion of a Newtonian fluid of viscosity, \( \eta \) we have

\[
\frac{\partial \mathbf{V}}{\partial t} + \rho \mathbf{V} \cdot \nabla \mathbf{V} = -\nabla p + \eta \nabla^2 \mathbf{V} + \frac{1}{3} \nabla (\nabla \cdot \mathbf{V})
\]

where \( \rho \) is the density of the fluid, \( \mathbf{V} \), the velocity vector and \( p \), the fluid pressure. By substitution, integration, and the use of tactic assumptions (with which our test method conforms) Dienes arrives at the general solution,

\[
F = -2 \pi \frac{dh}{dt} \cdot \frac{2h}{h^2} \int_0^R (R^2 - r^2) r dr
\]

where:

- \( F \) = applied force, Dynes
- \( h \) = plate separation
- \( t \) = time
- \( R \) = radius of specimen
- \( r \) = radius to the volume element.

In the evaluation of this integral two cases are considered.

Case 1: The viscous material completely fills the space between the plates. Then \( R = a \) where \( a \) is the radius of the plates. Integration twice gives

\[
\frac{1}{h^2} = \frac{4F}{3\pi \eta a} t + C_1
\]
where \( C_1 \) is the constant integration. \( C_1 \) is of no importance in determining the viscosity, \( \eta \), since \( \eta \) is calculable from the slope of the \( 1/h^2 \) vs. \( t \) curve. Solving for the viscosity gives

\[
\eta = 4F/3 \pi h^4 m
\]

By converting \( F \) (in dynes) to \( w \) (load in pounds) and substituting the radius of the plate (1.27 cm.), the equation becomes

\[
\eta = 7.26 \times 10^4 \times w/m
\]

where

\( \eta \) = viscosity in poises
\( w \) = load in pounds, and
\( m \) = slope-- \( 1/h^2 \) \( (\text{cm}^{-2}) \) vs. \( t \) (seconds).

Case 2: The viscous material does not completely fill the space between the plates. In this case we integrate the general equation from \( r = 0 \) to \( r = R \), giving

\[
F = -\frac{3 \pi V^2}{2 \eta h^5} \frac{dh}{dt}.
\]

Integration with respect to \( t \), gives

\[
\frac{1}{h_4} = \frac{8 \pi F}{3 \eta V^2} + C_2
\]

where \( C_2 \) is the constant of integration.

Using the same apparatus but the 2-inch diameter plate to compress a "pellet" of wax, the working formula is
\[ \eta = 3.72 \times 10^6 \times \frac{w}{mv^2} \]

where

\[ \eta = \text{viscosity in poises} \]
\[ w = \text{load in pounds} \]
\[ m = \text{slope} - \frac{1}{h^4} (\text{cm}^{-4}) \text{ vs. } t \, (\text{seconds}) \text{ and} \]
\[ v = \text{volume of specimen in } \text{cm}^3. \]

It must be pointed out that the linear relations, \( \frac{1}{h^2} \text{ vs. } t \) or \( \frac{1}{h^4} \text{ vs. } t \) for cases 1 and 2, respectively, are a consequence of the assumption of a parabolic law for Newtonian viscous flow. Non-Newtonian viscous flow is characterized by nonlinearity of the rate of shear vs. shear stress curve. This type of nonlinearity would be caused by appreciable "elastic" or "delayed elastic" effects. Because the rates of shear are very low in our testing, such effects should be negligible, or at least constant, after an initial period of nonlinearity.

Other assumptions made in the above derivation include: no wax slippage at the plate, no compression of the wax, and simplification of the mathematical theory by limiting analysis to that portion of the data for which \( R \) is greater than 10 times \( h \). Brittle fracture of the wax would invalidate data because of the first assumption. Erratic viscosity measurement, which would be difficult to interpret, would be obtained if the distance between the plates is not large compared to the radius of the specimen as given by the above restriction.
Description of Test Instrument: The instrument consists of two parallel plates between which the material is pressed, a loading mechanism, and a dial gage micrometer for observing the distance of separation of the plates. (See Fig. 3 for a picture of the test instrument.) The instrument is placed in a constant temperature bath for control of temperatures. (See Fig. 4)

Two types of plates are used for Case 1 testing. The bottom plate, on which the wax specimen is cast, is 1 ±0.001 inch in diameter and 23/32 in. thick. A 1/2-inch diameter, 7/16-inch long shank opposite the flat side of the plate drops into a carefully centered hole in the base of the instrument. The top plate is the same except that it is 3/8-inch thick and the shank is 11/16 inch long. The top plate is held in place by a pin through the shaft of the plastometer and the shank of the plate.

The above plate dimensions are such that plates may be installed or removed from the apparatus without removing the lever arm or dial micrometer. The plates used for the work reported in Report No. 7, Project 1195, were machined to meet the new requirements, allowing us to determine the distance between various combinations of top and bottom plates from the dial reading. Top plates were marked with letters A and B, while the bottom plates were numbered 1 through 9. Nine casting cylinders, for the bottom plates, were made from the cylinders previously used by cutting off one end so that the over-all height was 1-9/32 inches.

The plates used for Case 2 testing were two inches in diameter and approximately 1/4 inch thick. Two plates, similar to the top plates
described above, were stuck to the back surface of the 2-inch diameter plates (using Epon Resin VIII) to give a shank for mounting in the plastometer. The top surface of all plates were carefully lapped on a cast iron lapping plate, using first carborundum grit No. 240 then finishing with grit No. 600. It is essential that the plate surfaces be flat, free of dents or scratches, and perpendicular to the center line of the apparatus.

After setting the dial micrometer to read 0.200 with the A top plate and No. 1 bottom plate, the following dial readings were secured for other combinations. This data was obtained at 78°F. (room temperature). A check in the constant temperature bath at 115°F. gave the same results to the nearest 0.0002 inch. The load was not critical, showing that the leverage system was sufficiently rigid for normal use.

PLASTOMETER DIAL READINGS FOR ZERO PLATE SEPARATION

<table>
<thead>
<tr>
<th>Bottom Plate</th>
<th>A Dial</th>
<th>Negative Variation</th>
<th>B Dial</th>
<th>Negative Variation</th>
<th>2 in. Diam. (one only)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.200</td>
<td>0.0007</td>
<td>0.203</td>
<td>0.0006</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.184</td>
<td>0.0008</td>
<td>0.186</td>
<td>0.0002</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.196</td>
<td>0.0007</td>
<td>0.198</td>
<td>0.0005</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.196</td>
<td>0.0006</td>
<td>0.198</td>
<td>0.0004</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.189</td>
<td>0.0006</td>
<td>0.191</td>
<td>0.0004</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0.191</td>
<td>0.0003</td>
<td>0.193</td>
<td>0.0002</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>0.186</td>
<td>0.0005</td>
<td>0.188</td>
<td>0.0003</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>0.191</td>
<td>0.0004</td>
<td>0.194</td>
<td>0.0006</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>0.202</td>
<td>0.0018</td>
<td>0.203</td>
<td>0.0010</td>
<td></td>
</tr>
</tbody>
</table>

2 in. Diam. (one only) 0.476 0.009
Two figures are given for each plate. The bottom plate was rotated one whole revolution and the maximum dial reading recorded. The difference between this and the minimum dial reading is indicated as the negative variation. High negative variations indicate a lack of smoothness or non-perpendicularity of one or both of the plate surfaces. Bottom plate No. 9 is an example. All of the other plates were within normal tolerances. The positive rather than negative variation is given for the 2-inch diameter plate because rotation tends to give higher dial readings, probably because of nonperpendicularity of the flat surfaces. These plates were marked and used in the minimum dial reading position to avoid the application of shear stresses to the wax specimen.

Load Calibration: The top plate of the plastometer is loaded by placing weights on the weight pan suspended on one end of the weight arm. A precision ground shaft supported vertical to the base of the instrument by two brass bushings transfers the load from the lever arm to the top plate. An adjustable weight is provided at the other end of the weight arm for "zero" adjustment of the applied weight. Because the lever system is rather complicated and load values must be determined to a minimum of three significant figures, it was necessary to calibrate the instrument. First, the bottom plate and dial micrometer were removed. Next, one of the top plates was installed and held in place with the small pin. The "zeroing" weight was then adjusted so that the piston moved upward or downward about the same distance when the lever arm was given a gentle push. The lock nut was then set to keep it in this position.
The apparatus was then supported over a sensitive balance on a small wooden table. (The balance chosen was a 0 to 10-lb. Toledo balance with a 0.01-lb. finest division. Calibrated weights are placed on a pan at the rear of the balance to obtain ranges from 0 to 10 lb. in 1-lb. increments. The maximum reading, 10 lb., is much less load than we will require for testing at low temperatures but accurate calibration in this lower range should allow safe extrapolation to higher loads.) A small shaft was made of 1/4-inch diameter drill rod to transmit the force from the top plate to the balance pan through a hole in the top of the wooden table. Two rubber pads were used to prevent marring of the polished plate surface or the balance pan, and the weight of the pads and shaft taken into account in the final calibration.

The instrument was leveled on the wooden table and the scale reading recorded for a series of applied weights. Gram weights were used for convenience; the load was measured in pounds. Results are given below.

<table>
<thead>
<tr>
<th>Load Applied to Top Plate, lb. (Toledo Balance)</th>
<th>Weight Applied to pan, grams</th>
<th>Piston Move Downward</th>
<th>Piston Move Upward</th>
<th>Average</th>
<th>Average minus weight of calibrating shaft and pads</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>7.709</td>
<td>7.998</td>
<td>7.85</td>
<td>7.72</td>
<td></td>
</tr>
<tr>
<td>400</td>
<td>6.182</td>
<td>6.432</td>
<td>6.31</td>
<td>6.18</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>4.651</td>
<td>4.896</td>
<td>4.77</td>
<td>4.64</td>
<td></td>
</tr>
<tr>
<td>200</td>
<td>3.082</td>
<td>3.366</td>
<td>3.22</td>
<td>3.09</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>1.544</td>
<td>1.811</td>
<td>1.68</td>
<td>1.55</td>
<td></td>
</tr>
<tr>
<td>50</td>
<td>0.773</td>
<td>1.052</td>
<td>0.91</td>
<td>0.78</td>
<td></td>
</tr>
</tbody>
</table>

Note that there was considerable damping of load values. By carefully adjusting the weight on the back pan of the balance it was possible to keep the "null" range within a single scale range, making it easier to calculate
the average scale reading. It is important to deflect the piston the same distance above the scale reading as below to obtain accurate load values for the upward and downward movement of the piston. Plotting the values given in the right-hand column above against the weight added to the pan, we get the expected linear relation. Using the experimental values to calculate a load factor at 200 and 800 grams, we find that the required factors are 6.95 and 7.01, respectively. The average value, 6.98, was used to calculate higher plastometer loads according to the relation:

\[
\text{Total weight added to pan, lb.} \times 6.98 = \text{weight applied to wax specimen, lb.}
\]

In order to understand how well this calibration would be expected to stand up under test conditions, we changed the apparatus in several ways and determined the effect on the calibration. We repeated the calibration with the weight arm out of level by 16°. We did the same with the entire apparatus out of level by 0.4° (an amount easily detected with a spirit level) and also tested with various amounts of the pistons protruding from the brass bushing. None of these changes had any appreciable effect, so that apparently minor deviations from the refined calibration conditions will not affect the load calibration.

The Ames dial gage used to measure plate separation is spring-loaded and applies a force directly to the plastometer shaft. Using a calibration technique similar to the above, the following results were obtained:
These values were added to the calculated load to obtain the total load applied to the wax specimens.

Before testing, the apparatus was immersed in the constant temperature bath and the "zero load" weight adjusted to compensate for the buoyancy of the top plate and the immersed portion of the shaft. The water level was controlled so the instrument was at all times immersed in from 5 to 5-1/2 inches of water.

Method of Use (Case 1): Three castings were made of each wax on three different bottom plates. After cutting and conditioning of the specimen as previously described, the first plate was installed in the test instrument. The instrument is then leveled and adjusted in the water bath as shown in Fig. 4. With the top plate in place, the piston was lowered until the plate surface just contacts the specimen. It is left in this position for 5 minutes to assure that the plate and wax were properly conditioned to temperature. A trial weight is then added to the pan and the rate of piston movement observed from the speed of dial gage deflection.

It is important that the applied load be properly adjusted so that
accurate readings can be obtained from the dial gage. If too high loads are used, the dial gage pointer will move too rapidly to be read accurately. If the applied load is too small, excessively long periods of time are required to obtain sufficient readings. The determination of the proper weight is complicated by the fact that piston speed changes as the plates approach each other. Assuming that we can read the dial accurately if the pointer revolves at a rate of about $1/4$ revolution (25 divisions) in 30 seconds, we can estimate the proper load--using the $1/h^2$ vs. $t$ relationship. If we plot values of $1/h^2$ (cm.$^{-2}$) where $h$ is 0.045 and 0.050 inch against any convenient values for $t$ (seconds) separated from each other by six units, we can connect these points with a straight line and extrapolate the line to the $t$ axis. We then pick off the time required for various increments of plate separation. The following table gives the results obtained:

<table>
<thead>
<tr>
<th>Plate separation range (in.)</th>
<th>Optimum time for extrusion (sec.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.145-0.135</td>
<td>0.5</td>
</tr>
<tr>
<td>0.135-0.125</td>
<td>0.8</td>
</tr>
<tr>
<td>0.125-0.115</td>
<td>1.0</td>
</tr>
<tr>
<td>0.115-0.105</td>
<td>1.2</td>
</tr>
<tr>
<td>0.105-0.095</td>
<td>1.5</td>
</tr>
<tr>
<td>0.095-0.085</td>
<td>2.0</td>
</tr>
<tr>
<td>0.085-0.075</td>
<td>2.5</td>
</tr>
<tr>
<td>0.075-0.065</td>
<td>3.1</td>
</tr>
<tr>
<td>0.065-0.055</td>
<td>6.7</td>
</tr>
<tr>
<td>0.055-0.045</td>
<td>12.0</td>
</tr>
</tbody>
</table>

Note that ten separate ranges are given so that it is usually possible to adjust the weights with a single specimen before reaching the 0.050-inch clearance range.
After the applied load was properly adjusted, the dial was read at 15-second intervals for one minute, at 1-1/2 and 2 minutes and at one-minute intervals thereafter, over a plate separation range of 0.055 to about 0.020 inch.

In some cases it was not possible to adjust the weights quickly enough to be in control at the 0.050-inch plate clearance level. When this happened, the data from such a run was discarded—not used in the final calculation of viscosity. In other cases the wax was too hard to extrude at the required rate under loads as great as 270 pounds—the maximum used with our instrument, so that we had to be satisfied with slower testing speeds. For most of the tests, however, the method worked well.

A stop watch with a dial readable to 0.2 seconds was used to measure elapsed time.

A normal extruded sample is shown on the front wall of the constant temperature bath in Fig. 4.

The remaining two specimens were then tested in exactly the same way, allowing five minutes between tests for reconditioning of the plates and wax specimens after the handling required for installation in the test instrument. The plate containing the specimen was at no time raised above the water surface, so that it was necessary to recondition only to negate the effects of handling. Again the applied weight could be adjusted if desired after observing the completion of the first run. The data from the last two runs (and in some cases the first run) was used to calculate the viscosity.
Testing (Case 2): Case 2 testing was complicated by the necessity for accurately determining the volume of the wax "pellet."

A 25-ml. Pyrex specific gravity bottle was used in this determination. It was first filled with boiled dionized water, then immersed to its neck in the constant temperature bath. After conditioning a minimum of 1/2 hour or until the capillary level was constant, it was removed, wiped dry with a clean, lint-free cloth, and placed on the pan of an analytical balance. Exactly one minute after removal from the bath, a small glass plug was inserted in the capillary opening. (Since test temperatures are above room temperature, the level of the fluid in the capillary falls when it is removed from the bath so that the water does not contact the plug after one minute of cooling at room temperature. This is important because the bottom of the glass plug is open. If water should enter this opening, it would be difficult to dry the plug for the next run.) A similar specific gravity bottle was used as a tare on the other pan of the analytical balance. Exactly five minutes after removal from the bath the bottle was weighed to the nearest 0.0002 gram.

The precautions observed above were found necessary for two reasons. First, the weight falls off rapidly after removal from the bath—evaporation was thought to be the cause. Plugging the capillary opening gave improvement but the bottle still did not come to a constant weight. We decided to use a method of timed intervals to be observed for both the determination of the weight of the bottle containing water only and with the wax pellet
and water. The reproducibility by this method was no better than
0.0025 gram for three runs. Apparently further refinement in technique
will be required to obtain the required precision—about 0.0005 gram.
Perhaps the cover of the specific gravity bottle is not carefully enough
ground to be reproducibly replaced in the bottle. In the present evalua-
tion the results of three runs were averaged to obtain the weight of water
and bottle at a given test temperature. Secondly, it was necessary to use
boiled water to avoid bubble formation in the bottle when it was heated to
test temperature in the constant temperature bath.

Next, the pellet was weighed on the analytical balance (tare
removed) and then placed in the specific gravity bottle (filled with water),
and conditioned to test temperature. A fine piece of wire was found useful
in dislodging bubbles from the wax pellet and the inner surface of the
bottle, and a fine drawn-out bulb pipette was used to adjust the fluid
level in the capillary. Just before removal from the bath, the capillary
level was adjusted by adding an excess of water with the pipette, then
wiping the top of the cover quickly with a piece of ordinary filter paper.
After conditioning for a minimum of one hour, the bottle containing the
wax pellet and water was weighed as before and the volume calculated from
the equation

\[ V = [b - (c-a)] d \]

where

- \( V \) = volume of wax pellet, cm. (no voids within wax pellet
  or adhering air bubbles)
- \( a \) = weight of wax pellet, gram
- \( b \) = weight of bottle and water at temperature \( T \), gram
- \( c \) = weight of bottle, water, and pellet at temperature \( T \), gram
- \( d \) = relative density of water at temperature \( T \).
Relative density values were used since absolute density is the same to three significant figures (\( \bar{d} = d \times 0.999973 \), where \( \bar{d} \) is the absolute density). The density of the wax can be calculated from the formula

\[
d_{\text{wax}} = \frac{a}{V}.
\]

By this method the density of wax B-202 at 95°F. was 0.911—a reasonable value.

After determination of the volume, the wax pellet was placed between the 2-inch diameter parallel plate installed in the instrument in water bath. The instrument (and plates) were conditioned in the test bath at least 1/2 hour prior to the installation of the wax pellet and at least 1/2 hour after the wax pellet was installed. The weight is then applied as before and dial readings recorded vs. elapsed time. Of course it is not possible to use the previous table for adjusting the applied weight. It would be necessary to calculate new values from the \( 1/h^4 \) vs. \( t \) relationship.

In order to satisfy a basic assumption of the derived relationship for viscosity, it was necessary to determine two different conditions in the extrusion process. First, starting with a given pellet of wax, we must know at what plate clearance the radius of the deformed pellet exceeds plate clearance by a factor of 10 or more. Next, we must know at what stage in the compression of the pellet, wax will be extruded from the edge of the test plates. Both are directly related to the volume of the pellet.
Using a trial and error method of calculation, it was determined that the optimum pellet volume was $1 \pm 0.3 \text{ cm}^3$. With our waxes this required a pellet about 11/32-inch long and 3/8-inch diameter. After the volume, $V$, has been determined, the dial reading, $d_{\text{start}}$, at which the radius of the pellet exceeds the plate clearance by a factor of 10 is given by

$$d_{\text{start}} = \left(\frac{V}{51.3}\right) + 0.476.$$ 

Assuming the wax to be incompressible under present testing conditions, the dial reading, $d_{\text{stop}}$, at which wax will extrude from between plates is given by

$$d_{\text{stop}} = 0.125 \times \sqrt[3]{V} + 0.476.$$ 

After the volume was determined, the dial readings calculated from the above equations were used to determine the range of critical readings when running the test and in calculation of viscosity from the data obtained.

**Calculation of Viscosity:** The viscosity by either method (Case 1 or Case 2) was calculated from the applied weight and the slope of the deformation vs. time curve, using the relationship derived in the theoretical section of this report. In addition, the volume was used in Case 2 calculations. Using a slide rule, the values of $1/h^2$ and $1/h^4$ (both in cm.) were calculated and plotted vs. elapsed time on ordinary graph paper. The points should lie in a straight line but often a slight curvature was seen nearer the $h = 0.050$-inch end of the graph. When this occurred the slope was determined at smaller values of plate clearance, usually at about
0.030 inch, although it was necessary to examine each curve separately to select the optimum point for slope determination.

Notwithstanding the theoretical basis for nonlinearity of the deformation vs. time curve (non-Newtonian flow characteristics, bearing friction, etc.), all of the curves contained linear portions fixed by at least three experimental values (usually a 2-minute time interval), making accurate slope determinations possible. This was true of all the waxes tested over a temperature range from 95 to 115°F. Apparently the method is applicable to such materials as wax and instrumental difficulties such as friction between the shaft and the brass bearings, surface finish of the plates, and other factors are not unusually critical.

The slopes obtained varied from 0.0292 to 7.32, indicating that the speed of testing varied within rather wide limits. The average slope was about 2.00. One of the main reasons for this variation was the fact that we were limited by the capacity of the weight arm pan to about 40 pounds applied weight—less than the desired weight for wax B-202 tested at 95°F. Also, using large weights the increments between weights (usually 2 pounds, unless gram weights were used) made it difficult to adjust the weight accurately. The minimum weight used was 200 grams, giving a load of 3.26 lb. to the top plate.

The calculated viscosities for all of the waxes tested are given in Table II. This is the data which was forwarded to the committee chairman for comparison with the other methods being evaluated. Note that
TABLE II

VISCOSITY DETERMINED BY PARALLEL PLATE VISCOMETER, Poises.

<table>
<thead>
<tr>
<th>Test Temperature, °F.</th>
<th>Shell-Wax</th>
<th>Shell-Wax</th>
<th>Shell-Wax</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>B-201</td>
<td>B-202</td>
<td>B-203</td>
</tr>
</tbody>
</table>

**Case 1. Material completely fills space between plates.**

<table>
<thead>
<tr>
<th></th>
<th>B-201</th>
<th>B-202</th>
<th>B-203</th>
</tr>
</thead>
<tbody>
<tr>
<td>95</td>
<td>6.73 x 10^7</td>
<td>7.49 x 10^8</td>
<td>1.62 x 10^6 *</td>
</tr>
<tr>
<td>95</td>
<td>6.02 x 10^7</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>100</td>
<td>5.02 x 10^5</td>
<td>1.98 x 10^8</td>
<td>2.62 x 10^6 **</td>
</tr>
<tr>
<td>105</td>
<td>3.83 x 10^5</td>
<td>1.27 x 10^7</td>
<td>6.38 x 10^5 **</td>
</tr>
<tr>
<td>110</td>
<td>4.23 x 10^5</td>
<td>1.75 x 10^6</td>
<td>2.97 x 10^5 **</td>
</tr>
<tr>
<td>115</td>
<td>4.50 x 10^5</td>
<td>4.01 x 10^5</td>
<td>1.09 x 10^5 **</td>
</tr>
<tr>
<td>115</td>
<td>-</td>
<td>3.45 x 10^5</td>
<td>1.45 x 10^5 **</td>
</tr>
</tbody>
</table>

**Case 2. Compress wax pellet between plates.**

<table>
<thead>
<tr>
<th></th>
<th>B-201</th>
<th>B-202</th>
<th>B-203</th>
</tr>
</thead>
<tbody>
<tr>
<td>95</td>
<td>7.88 x 10^7</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>115</td>
<td>-</td>
<td>2.26 x 10^5</td>
<td>-</td>
</tr>
</tbody>
</table>

* 12 hours in water bath before tested.

** Less than 4 hours in water bath before tested.
only two test runs were made using the Case 2 method. The results for Case 1 are the average of a minimum of two separate runs using two different wax castings and test plates. Several of the waxes were tested on consecutive days, using freshly prepared wax specimens, to determine the repeatability of the method. The Case 1 parallel plate method seems to give a good characterization of the waxes tested over the entire temperature range. The data given for Case 2 tests is too limited for a valid comparison of the two methods of testing. Paraffin B-202 is much harder than B-201 at 95°F., but they appear about the same at 115°F. Note that a reversal occurred in the data between 95 and 100°F. for wax B-203. After reviewing the original notebook entries for this series of tests, the only reason which could be found for the reversal was the difference in conditioning time as given in Table II.

RELATIVE MERITS OF THE TWO METHODS AND CONCLUSIONS

A consideration of the relative merits of two such test methods will have to consider both the simplicity of the tests and the immediate usefulness of the data obtained. Since these methods are being considered as possible means of arriving at purchase specifications—the primary purpose of TAPPI-ASTM tests, it is likely that simplicity of the test method and test apparatus will be of prime importance. The needle penetration method is easier to perform in the laboratory and requires only very simple mathematical treatment to obtain the final results. The parallel plate method requires a greater knowledge of the factors involved in the rheologic study of such materials to perform the test and relatively complex and lengthy mathematical treatment to obtain the final results.
For this reason it may not be acceptable as a routine test method. It should be very useful, however, for research where a more fundamental approach is desired.

Figure 5 is a graphic presentation of the data given in Table I; Fig. 6 is a graph of Case 1 data given in Table II. Penetrations were plotted on a linear scale extending from 0 to 350 grams, while the viscosity data was plotted on a logarithmic scale from $10^5$ to $10^9$ poises—a $10^4$ of 10,000 unit range. This difference in total range must be considered in comparing the repeatability of the two methods at 95 and 115°F. (See Tables I and II)

Both methods gave a surprisingly similar characterization of the waxes tested. Wax B-202 was rated much harder than the other two waxes at 95°F. At 115°F, waxes B-201 and B-202 (both paraffin waxes) are of about equal hardness by either method. The hardness of wax B-202 decreases in a nearly linear fashion with increasing temperature by the penetration method and in a logarithmic relation to temperature by the parallel plate method. The same is roughly true of microcrystalline wax B-203.

It would be interesting to speculate as to the meaning of the nearly constant hardness of wax B-201 (as shown by either method) over a temperature range from 100 to 115°F., after a great decrease in hardness from 95 to 100°F., but we do not have sufficient background information at this time to support such a speculation.
SECTION II,
TAPPI-ASTM COMMITTEE
ON PETROLEUM WAX

FORCE REQUIRED FOR ASTM D-5 NEEDLE PENETRATION
OF 40-TENTHS OF A MILLIMETER IN 5 SECONDS
AT VARIOUS TEMPERATURES

WAX DISTRIBUTOR - SHELL OIL COMPANY

B-201
B-202
B-203

Figure 5.
VISCOSITY AS DETERMINED BY PARALLEL PLATE VISCOMETER AT VARIOUS TEMPERATURES

CASE 1. MATERIAL COMPLETELY FILLS SPACE BETWEEN PLATES

WAX DISTRIBUTOR - SHELL OIL COMPANY

<table>
<thead>
<tr>
<th>Temperature, °F</th>
<th>Viscosity, Poises</th>
</tr>
</thead>
<tbody>
<tr>
<td>95</td>
<td>$10^9$</td>
</tr>
<tr>
<td>100</td>
<td>$10^8$</td>
</tr>
<tr>
<td>105</td>
<td>$10^7$</td>
</tr>
<tr>
<td>110</td>
<td>$10^6$</td>
</tr>
<tr>
<td>115</td>
<td>$10^5$</td>
</tr>
</tbody>
</table>

- B-201
- B-202
- B-203
In summation, both methods gave a remarkably similar characterization of the waxes tested over a temperature range from 95 to 115°F. Because of the high loads encountered in testing the harder waxes by either method, we feel that the present instruments are limited to a maximum hardness of about \(10^9\) poises or 350 grams, respectively. Higher loads may cause bending of the needle in the needle penetration instrument or bending of the lever arm bearing pins or lever arm in the parallel plate instrument. However, both methods can probably be used to test softer waxes. The needle penetration method might be better in this respect as it can be easily calibrated to deliver very small forces to the wax surface. Friction between the bearing and the shaft and in the lever system would be expected to cause difficulty in applying very small loads with the parallel plate instrument, although as little as 50 grams, applied in the calibration, appeared to give satisfactory results.

Mr. Cushman, in a report to the committee on work recently concluded in his laboratory*, gives a penetration value of \(19/10\) of a mm. for wax B-202 tested at 95°F. His tests were conducted using the Standard method for the hardness of paraffin wax which specifies 100-grams load for five seconds. According to this, these methods could probably be used to determine hardness in the range presently covered by the Standard method, i.e., the 0 to 50 tenths of a mm. hardness range. This is probably also true of the parallel plate method of testing. Thus either method might be used to cover the entire range of hardness being considered by the Committee, rather than only the range for softer waxes.

The Case 2 method of parallel plate testing has not been seriously considered as a routine test method because of the necessity, as we presently conceive the test, for accurately determining the volume of each specimen of wax to be tested. It is possible, however, that some of these objections could be overcome, making a more complete evaluation possible. The method offers an interesting test of some of the assumptions used in deriving the viscosity relationship so that a more complete evaluation would be desirable. The data we have obtained (2 determinations) is much too limited to warrant comparison of the Case 1 and Case 2 results obtained.

IMPLICATIONS

We have observed the similarity in test results obtained by two different test methods for three different waxes over a range of temperature. It would be tempting to try to correlate the hardness values obtained by the two methods and, in fact, a brief attempt was made at such a correlation. By dividing the viscosity value obtained by the parallel plate method by the force for 40-needle penetration, we obtained a range of values from $233 \times 10^4$ to $1.29 \times 10^4$. The average of such values, using the first day's test results where repeatability runs were made, was $5 \times 10^4$, but this value can have little meaning considering the wide spread in the values averaged. Also it would be hazardous to generalize such a result because of the limited number of waxes tested. However, such a value might be used to predict the approximate viscosity to the nearest whole poise from the force for needle penetration data,
if such an approximation is required. An attempt to correlate such results by a simple process of division is of course mathematically naive when we consider the complex mechanism by which either of the test methods measure the viscosity of the material. Using the needle penetrometer as an instrument to measure the force to obtain a constant penetration is a simplification of the older method for measuring the depth of penetration at constant loads. But many complications still exist before we can hope to calculate a valid viscosity from such data. Perhaps most important of these is the lack of homogeneity in the wax sample itself. This problem was well demonstrated by the investigation of the depth of penetration using a series of needles and cones (Report No. 11 dated March 1, 1955). It might be possible, using the force for constant-penetration method of testing, to determine the proposed density gradient within the wax sample by testing at different levels of penetration, e.g., 5, 10, 20, and 40 tenths of a mm. Elastic and delayed elastic properties could significantly affect the results of constant depth of penetration testing and would probably be difficult to evaluate by this method.

A careful investigation of wax by the parallel plate viscometer method would have to deal with some of these same problems. First of all, an attempt to compare the two methods should consider that the parallel plate method may utilize an area of wax density different from that of the penetration method. However, the parallel plate method does deform, and thereby test, a much larger proportional volume of wax so that differences in density should be less critical.

In the parallel plate method it is assumed that the extruded
material is incompressible under the conditions of the test. Wax may not satisfy this assumption when subjected to high test loads.

The use of high loads could have an additional effect on the viscosity. Work, defined as force x distance, is proportional to the load used and may produce sufficient heat to alter the viscosity of the sample being tested. To test this possibility we calculated the work, converted to calories, for several of the test runs. Assuming the original thickness of the wax specimen to be 0.125 inches and assuming an average final thickness after extrusion of 0.030 inch, together with values of 0.911 and 0.694 calories per gram for the density and specific heat of wax respectively, we find that

\[ AT = f \times 0.00252 \]

where

\[ AT = \text{calculated temperature change, } ^\circ\text{C.}, \text{ and} \]

\[ f = \text{force applied to wax, lb.} \]

In our evaluation the applied load was the prime variable. This was the result of attempting to test at constant speed, i.e., deformation vs. time, so that the dial could be conveniently and accurately read during the extrusion. Using the above formula, \( \Delta T \) values of 0.68°C. and 0.0082°C. respectively, were obtained for the maximum (270.7 lb.) and minimum (3.26 lb.) loads used in our evaluation. The total time for heat evolution in the above two tests was approximately 16 and 3 minutes, respectively. Thus while the greater amount of heat was evolved over a greater period of time in our testing, this would not necessarily be true of all such testing and it would be important to consider the effects of work on the test sample. The actual temperature change
within the wax sample would, of course, be dependent on many factors including the design, mass, and specific heat of the test plates as they affect the rate of heat transfer, but the magnitude of the $\Delta T$ values obtained by the above calculation should demonstrate the need for caution in using high loads or high rates of testing.

Another problem which would require attention is the effect of "elastic" or "delayed elastic" properties of certain materials. Addition of elastomeric polymers to wax would probably give samples of this type. Theoretically the method is able to determine non-Newtonian flow characteristics by giving nonlinear deformation vs. time curves, but in order to fully evaluate such a phenomena it would probably be necessary to use a wide range of speeds of testing. Our present instrument is probably limited in this respect to relatively small changes in the low rate of testing range. Other instruments such as the Brookfield viscometer accomplished this by varying the speed of the rotating cylinder but are, of course, limited to lower viscosity ranges. The maximum viscosity which can be determined with the Brookfield instrument is 100,000 c.p.s. or $10^3$ poises.

In summation, our present evaluation has not been sufficiently extensive to determine the applicability of the two test methods for general wax evaluation. Both methods appeared promising for the testing of hardness of wax or similar materials over a hardness range from $10^5$ to $10^9$ poises, but it will be necessary to determine the effects of certain other variables to assure general applicability. A wider range of samples should be tested and the effect of higher loads and various speeds of testing determined. In addition, an attempt should be made to calculate
viscosity from constant-needle penetration data*. When the effect of these variables is understood it may be possible to attempt a valid comparison of the viscosity or hardness determined by the two test methods. Understanding the hardness of such materials in terms of mathematical relationship should make it possible to determine the effect of hardness on many of the properties required of wax as a packaging material and should give us a better understanding of the factors related to hardness which limit the usefulness of wax in such applications.

*Page 160, Properties of Asphaltic Bitumen, by J. Pfeiffer, published by the Elsevier Publishing Company, should be a good reference for such a calculation.
WAXING OF CREPE PAPER AND CERTAIN PROPERTIES OF WAXED CREPE PAPER

INTRODUCTION

The purpose of this investigation was to study the problems related to the waxing of high basis weight crepe paper such as might have application in the general packaging field. Our interest in this field is the result of an inquiry from a member mill for suggestions of methods of waxing crepe paper. At the present there is little information on such a converting problem in the literature. This is a report of certain aspects of such a study.

Since there are no specific requirements for the properties of such a converted product, it was necessary to select certain properties for comparison of the various methods of waxing attempted in our laboratory.

Using our laboratory waxer, wax was applied by passing the web of crepe paper under a roll immersed in a tank of molten wax. Excess wax is removed by drawing the web under tension over a series of wire wound rods. A second method consisted of drawing the web of crepe paper across the top of a roll partially immersed in molten wax. This would constitute a normal kiss roll application except that paper-to-roll friction was the only means used to drive the coating roll. Another possibility is squeeze-roll application which consists of passing the web between two pressurized
rubber and/or rubber and steel rolls to remove excess wax. This method was not attempted in this laboratory.

After waxing, the web was cooled by either dipping into a tank of cold water or by allowing it to solidify at room temperature. It was then rewound and set aside for later testing.

Some of the properties of crepe paper considered to be of primary importance in a waxing application are:

1. Behavior on the waxing machine using various methods of applying wax to paper.
2. General appearance of waxed crepe paper
3. Probable range of wax application weight including minimum wax weight.
4. Accurate determination of the amount of wax applied to crepe paper. The difficulty anticipated is the change in basis weight due to stretching on the waxing machine.
5. Accurate determination of the distribution of wax on the crepe base sheet.
6. Loss of crepe caused by waxing of crepe paper due to either the action of the wax on the paper or normal paper tensions on the waxing machine.

All of the properties listed above have been evaluated using one grade of Kraft crepe paper and a single refined paraffin wax. The properties chosen are intended to yield background information concerning the general problems related to the waxing of crepe paper. Specific application would require a more specialized investigation.
EXPERIMENTAL

Materials:

The crepe paper used for this evaluation was obtained from the Mosinee Paper Company on October 2, 1953, and is identified as "Mosinee Kraft Crepe, Basis Weight 40-45, Spec. No. 412-A." It was received in 9 1/2 inch diameter rolls 12 inches wide and was cut into 4 1/2 inch wide rolls on a table saw in the millwright shop for use on the laboratory waxer. The rolls were conditioned a minimum of two months at 73° F., 50% R. H. before waxing.

The physical characteristics of the unwaxed kraft crepe paper, as reported in Code Office Report File No. 55-73219 dated October 15, 1953, are as follows:

<table>
<thead>
<tr>
<th>Property</th>
<th>Max.</th>
<th>Min.</th>
<th>Av.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Basis Weight, lb., 24x36/500</td>
<td>24.3</td>
<td>24.1</td>
<td>24.3</td>
</tr>
<tr>
<td>Caliper, inch</td>
<td>0.0131</td>
<td>0.0127</td>
<td>0.0129</td>
</tr>
<tr>
<td>Bursting strength (Mullen)</td>
<td>26.0</td>
<td>19.0</td>
<td>22.5</td>
</tr>
<tr>
<td>Points</td>
<td></td>
<td></td>
<td>51</td>
</tr>
<tr>
<td>Points per 100 lb.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Elmendorf Tear, g./sheet In</td>
<td>146</td>
<td>118</td>
<td>129</td>
</tr>
<tr>
<td>Across</td>
<td>131</td>
<td>115</td>
<td>127</td>
</tr>
<tr>
<td>Schopper Tensile, lb./inch In</td>
<td>10.2</td>
<td>9.0</td>
<td>9.5</td>
</tr>
<tr>
<td>Across</td>
<td>14.9</td>
<td>11.5</td>
<td>13.4</td>
</tr>
<tr>
<td>Gurley Porosity, sec./100 cc.</td>
<td>3</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Percentage Crepe*</td>
<td>30</td>
<td>23</td>
<td>25</td>
</tr>
<tr>
<td>In</td>
<td>7</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Across</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Tappi Routine Control Method No. 215
The wax selected for this evaluation was Socony Vacuum 125-127 AMP fully refined paraffin wax. No additives were used.

This is the same wax used for the study of polymer addition to wax (Report No. 13) and therefore has been evaluated in this laboratory. Data from that report which applies most directly to the present investigation is given below. Note that these tests characterize the rheologic properties of the wax and are independent of the base sheet used.

**Paraffin Melting Point**

ASTM D 87-42

51.0°C.

**Cloud Point**

Institute Method

53°C.

**Viscosity-Temperature**

(Viscosity determined on descending temperature scale)

<table>
<thead>
<tr>
<th>Temperature, °F.</th>
<th>Viscosity, cps.</th>
</tr>
</thead>
<tbody>
<tr>
<td>250</td>
<td>3.2</td>
</tr>
<tr>
<td>240</td>
<td>3.4</td>
</tr>
<tr>
<td>228</td>
<td>4.1</td>
</tr>
<tr>
<td>222</td>
<td>4.1</td>
</tr>
<tr>
<td>212</td>
<td>4.3</td>
</tr>
<tr>
<td>198</td>
<td>4.1</td>
</tr>
<tr>
<td>188</td>
<td>4.6</td>
</tr>
<tr>
<td>180</td>
<td>4.8</td>
</tr>
<tr>
<td>172</td>
<td>5.8</td>
</tr>
</tbody>
</table>

**Flexibility at 3.9 ± 1.1 °C.**

Percent Elongation

<table>
<thead>
<tr>
<th>Method</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Short Pendulum</td>
<td>0.38</td>
</tr>
<tr>
<td>Long Pendulum</td>
<td>0.44</td>
</tr>
</tbody>
</table>

**Penetration at 77°F.**

ASTM D 1321-54T

15 tenths of a millimeter

Additional test results and a discussion of the methods used are given in the Methods and Results section of Report No. 13.
Waxing of Crepe Paper:

The problems of waxing crepe paper were studied on our laboratory waxer. The waxer is ordinarily used to give a dip-roll application with a wire wound rod metering process. In order to cover a wide range of wax weights it was necessary to decrease the amount of wax by modifying the waxer for the low wax weights by adding two smooth steel bars so that the sheet could be drawn across the top of a roll immersed in molten wax. The roll then serves as a kiss roll to apply wax to the bottom (or reverse surface) of the sheet. The roll is driven by contact with the moving paper web. See Figure 1 for a schematic diagram of the wax application and cooling methods used. Some thought was given to the possibility of trying a squeeze-roll method of coating but no suitable rolls were available which could be easily adapted to the laboratory waxer.

The waxer has provision for quick cooling of the coated web by passing it through a water dip tank, or the machine may be threaded to provide for air cooling of the coated web. Refinements include a variable speed drive (D. C. Thymotrol), a thermostated hot plate for controlled wax temperature and an adjustable constant temperature water bath. The wire wound rods are heated with cartridge-type heaters providing good control of metering rod temperatures. Details of the design of the waxer, calibration of the heaters, calibration of the Thymotrol drive etc., have been reported in Reports numbered 6, 8, 12, and 13. Report No. 13 also contains formulas derived for the calculation of web speed and tension which will be used in this report.

A detailed account of the conditions used to wax crepe paper is
LABORATORY WAXER

Dip Roll - Wire Wound Rod Metering
Water Cooled

Dip Roll - Wire Wound Rod Metering
Air Cooled

Kiss Roll Application
Air Cooled

Kiss Roll Application
Water Cooled

Figure 1
given in Table 1, together with observations of the types of failure noted. Waxed samples are identified by the Research Notebook number, page and position on the page where waxing data was recorded. This method of identification will be used throughout this report.

Web speed was calculated from the formula (Derived for report No. 13)

\[ S = D \times n \times 0.262 \]

Where:

\( S \) = Web speed in feet per minute
\( D \) = Diameter of the rewind roll in inches and
\( n \) = Number of revolutions per minute of rewind roll (obtained from the Tymotrol calibration).

Since the web is attached directly to the rewind roll, web speed as given by the above formula is certainly accurate at a point at or near the rewind roll. (See figure 1) But using a web of crepe paper, which by its very nature is stretchable, the web speed will probably be less than that calculated, in proportion to the amount of stretch, at any given point along the threaded path of the web. Load-elongation studies, to be reported later, indicate that the stretch (defined as "loss of crepe by waxing") was as great as 12% for some of the coating runs. Unwind roll web speed would then be reduced by 12% from the value calculated by the above formula and the speed of an intermediate point, such as the dip or kiss roll, would be reduced in direct proportion to the amount of stretch at that point. However, since we have no way of determining "stretch" directly from the laboratory waxer, web speed calculated on the basis of rewind roll diameter and speed will suffice to characterize the coating conditions attempted in this evaluation.
Closely tied to the problem of stretch on the laboratory waxer is the amount of tension applied at the unwind roll. A small brake is attached to the shaft of the unwind roll which applies controlled tension to the sheet as it is pulled off. The device has been calibrated so that tension may be calculated from the formula* (Derived for Report No. 13)

\[
T \text{ (lb./inch width)} = \frac{(w + 46) 20.8 + 2410}{7.74 \times 10^{-5}} \frac{r}{r}
\]

where, \(T\) is the tension applied to the sheet at the unwind roll, \(w\) is the weight in grams applied to the lever arm and \(r\) is the radius in inches of the unwind roll, for a 4 1/2 inch wide web.

Because this type of brake must be run without lubrication to give reproducible values for the coefficient of friction, wearing of the brake surfaces is a problem. It would be difficult to assess the effect on the tension values given in Table I, of this difficulty but it could be appreciable. Tension data should therefore be examined with these considerations in mind.

Wire wound rods were used to meter off excess wax when coating by the dip-roll method. Several different combinations of metering rods, wound with different wire diameters were used to vary the amount of wax retained by the web. For kiss-roll coating only one wire wound rod was used. Its purpose was to change the direction of web travel so that the coated web could be wound easily after it was air cooled or dipped in a water bath for quick cooling. The rod chosen was wound with 0.02" diameter wire wound in such a manner that the wire-to-wire distance between

*The formula given is slightly different than reported previously due to the difference in the width of the web.
consecutive turns was 0.02 inches.

Metering rod temperatures were obtained with a portable Alnor pyrometer by holding the temperature sensitive head directly on the heated rod as near to the center of the span across the machine as possible. The temperature was controlled at $175\pm 5^\circ F.$, by adjusting the voltage to the cartridge heaters with a variable voltage transformer. Wax bath temperature was maintained at between 180 and $190^\circ F.$, except of samples 1418-41-9 and 1418-41-12 where the wax temperature was $165^\circ F.$ Water temperature was maintained between 39 and $46^\circ F.$, using the portable constant temperature bath to refrigerate and also recirculate the water.

Approximate values for coating weight were obtained using the rapid method previously described in Report No. 13. The results are given in Table I. Using a 3.083 inch square template and razor blade a sample is cut from the web, weighed and the weight in grams multiplied by 100 to give the weight in pounds per ream ($\frac{24\times26}{500}$). Using the same procedure for unwaxed and waxed paper the amount of coating is calculated as the difference between the two weights obtained. Errors inherent in the system include—no control of the humidity of the sample and no allowance for the change in basis weight of the sheet as a result of stretching on the waxing machine. Such determinations are necessary, however, for routine control of the amount of wax applied by the waxer and are given as "Approximate Wax Weight" and "Approximate Wax Pick-Up" in Table I. Later data, based on the results of solvent extraction of the wax from these same samples, will give a more accurate picture of the amount of wax applied.
# TABLE I

**WAXING OF MOSINEE KRAFT CREPE PAPER**  
(Basis Weight 43.2)

<table>
<thead>
<tr>
<th>Coating Conditions</th>
<th>Observations</th>
<th>Approximate Wax Pick-Up</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tension at Unwind Roll</td>
<td>Edge</td>
<td>Wrinkled-Water</td>
</tr>
<tr>
<td>Rod Orientation</td>
<td>Glossy Blocking-Surface</td>
<td>Rewind</td>
</tr>
<tr>
<td>Method (see Figure 1)</td>
<td>Water</td>
<td>Air</td>
</tr>
<tr>
<td>Dip</td>
<td>Kiss</td>
<td>Water Penetration</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Coating Code</th>
<th>Speed*</th>
<th>Metroe**</th>
<th>Edge</th>
<th>Wrinkled-Water</th>
<th>Wax Roll Spotty</th>
<th>Wax Roll Penetration</th>
</tr>
</thead>
<tbody>
<tr>
<td>ge-Position</td>
<td>Ft./Min.</td>
<td>(lb./in. width)</td>
<td>Method (see Figure 1)</td>
<td>Water</td>
<td>Air</td>
<td>Wax Roll Spotty</td>
</tr>
<tr>
<td>40-1</td>
<td>1</td>
<td>1.5</td>
<td>0, 0 &amp; 0</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>40-2</td>
<td>103</td>
<td>0.1</td>
<td>&quot;</td>
<td>x</td>
<td>:x</td>
<td>x</td>
</tr>
<tr>
<td>40-3</td>
<td>2</td>
<td>0.1</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>40-4</td>
<td>118</td>
<td>0.5</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>40-5</td>
<td>127</td>
<td>1.7</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>40-6</td>
<td>77</td>
<td>0.8</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>41-7</td>
<td>83</td>
<td>0.8</td>
<td>34, 0 &amp; 0</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>41-8</td>
<td>79</td>
<td>0.8</td>
<td>34, 28 &amp; 20x</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>41-9</td>
<td>124</td>
<td>0.6</td>
<td>8, 19 &amp; 0</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>41-10</td>
<td>2</td>
<td>0.6</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>41-11</td>
<td>2</td>
<td>0.7</td>
<td>0, 0 &amp; 0</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>41-12</td>
<td>3</td>
<td>0.7</td>
<td>8, 19 &amp; 12</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>42-13</td>
<td>97</td>
<td>0.9</td>
<td>20x</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>42-14</td>
<td>97</td>
<td>0.7</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>42-15</td>
<td>97</td>
<td>0.3</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>42-16</td>
<td>45</td>
<td>0.7</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>42-17</td>
<td>45</td>
<td>0.7</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>43-18</td>
<td>118</td>
<td>0.1</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td>Slight</td>
</tr>
<tr>
<td>43-19</td>
<td>50</td>
<td>0.3</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td>Slight</td>
</tr>
<tr>
<td>43-20</td>
<td>97</td>
<td>0.8</td>
<td>&quot;</td>
<td>x</td>
<td>x</td>
<td>Slight</td>
</tr>
</tbody>
</table>

Note that speeds below 5 feet per minute gave an air cooled specimen even though web is passed thru water bath.  
Metering rods designated in one-thousanth inch increments of tightly wound wire diameter, and listed in order of paper contact.  
Test surface contacts first rod listed by dip coating method.  
Coating applied to end rod contacts reverse surface by kiss roll coating method.  
For rod designated 20x, the x indicates that the wire was not tightly wound.  
Wire-to-wire spacing between consecutive winds was 0.02 inches.  
Wax weight and Wax Pick-Up obtained for control purposes by weighing a specified area of waxed sheet as soon as possible after waxing.  
Such values are approximate because the sheet is not conditioned to standard humidity, and no allowance is made for loss of crepe on the laboratory waxer.
Let us now consider the general characteristics of the coatings prepared by each of the four methods attempted. The columns titled "Observations" in Table I indicate the type of problems encountered in waxing a crepe sheet on the laboratory waxer. Some of these same problems would probably occur in waxing on conventional paper converting equipment. Using a dip roll method of coating, the minimum pick up was approximately 66 pounds per ream or approximately 154% based on the weight of paper. This was obtained at a coating speed of 2 feet per minute using wire wound rods. At faster coating speeds, wax weights were higher even though greater tension and finer wire wound rods were used. The maximum pick up, 240%, was obtained at 83 feet per minute using a No. 34 rod to meter the test surface—coating 1418-41-7. All dip coated samples appeared glossy indicating the presence of wax on the surface of the sheet. Edge blocking in the rewind roll was a problem at higher coating speeds probably because the "Dwell" period in the water bath was not sufficiently long to completely solidify the large amount of wax picked up—in all cases in excess of 65 pounds per ream. (The total time of immersion in the water bath at coating speeds of 1, 50 and 100 feet per minute was approximately 1 minute, 1 second and 0.5 seconds respectively.)

The wax appeared to be well distributed and no difficulty was encountered with water penetration into the sheet.

The dip-coated, air-cooled sample was made at slow speed to allow time for the wax to solidify before rewinding. It gave the same general appearance as the water-cooled samples.
Less wax was applied by the kiss-roll method of coating. The minimum applied was 38 pounds per ream or 89% (based on the weight of paper) applied at a coating speed of 97 feet per minute and 0.9 lbs./inch width of tension. No surface wax was visible on any of the air cooled samples even though wax pickups as great as 119% were obtained, indicating that the wax receptivity of the base sheet is very high. Water-cooled samples gave a slightly glossy appearance indicating the presence of a small amount of surface wax. Apparently some of the wax is solidified on the surface on the sheet by quick chilling. The time for the web to pass from the kiss roll to the water bath surface was 2.4 seconds at a coating speed of 50 feet per minute, speeds in excess of which gave a small amount of surface wax.

Caution must be observed in making these observations that the operator does not mistakenly interpret a dull appearance as indicating the absence of surface wax. Judging from experience with this wax on other base sheets, quick-cooled surface coatings are usually glossy in appearance while slow or air-cooled surface coatings are dull. Therefore, a razor blade was used to scrape the surface of all air-cooled coatings to verify the presence or absence of surface wax. Using this method to complement the microscopic examination, it was observed that dip-roll coatings—probably because of the higher wax weight applied—and quick-chilled kiss-roll coated paper evidenced surface wax. The application of less wax or allowing it more time to become distributed (slow cooling) gave little or no wax on the surface of the sheet. This would probably not be true, however, of coating conditions or coating weights significantly different from those cited.

Kiss-roll coatings which were quick-cooled in the water bath were
spotty in appearance, indicating an uneven distribution of wax. Samples coated in excess of 50 feet per minute were wrinkled by water penetration. Note that the kiss-roll method of coating applies wax to only one side of the sheet. Also the amount of wax is less than applied by the dip coating method so that water penetration is more likely to occur. Apparently at slower coating speeds the wax has sufficient time to distribute itself within the sheet giving a more even distribution and therefore better protection against water absorption. Caution is advisable if we consider wax pickup of less than 100%—perhaps as low as 25 or 30%. Water dip cooling of such coatings would certainly be impractical. Air cooling or cooling the web by passing it over water-cooled rolls would be practical methods of handling the problem. In the lower range of wax pickup, the wax would probably be absorbed and distributed very quickly, minimizing the chance of blocking in the rewind roll, so that it probably not be necessary to cool to as low a temperature.

Wax weights within a range from 89 to 272%, based on the weight of paper, have been obtained. For many applications it would be advisable if not essential to explore the lower wax application range i.e. 5 to 90% wax. Apparently dip-coating methods would not be applicable in this range for this type of crepe sheet because of the high absorbency of the sheet. Of course the amount of wax ultimately retained by the sheet is dependent on the amount of metering. Wire wound rods are apparently not very efficient in removing surface wax from a crepe sheet and probably have almost no ability to remove wax saturated into the sheet. Squeeze rolls would probably be more efficient in removing both surface and internal wax but their use at high roll-to-roll pressure might seriously alter the appearance and characteristics of the crepe sheet. The use of a waxed carrier sheet to apply wax to the crepe base sheet under heat and
mild pressure might be a possible means of obtaining very low wax weights. A coating as low as 3 to 4 pounds per ream can be applied to a base sheet such as a 25# sulfite breadwrapper stock. It might be possible to coat such a sheet, using it to carry wax to the crepe sheet. By melting the wax with a heated roll it should be possible to transfer it to the crepe sheet to give a uniformly distributed low wax load. Such a process would probably not be economically feasible unless a means could be found to use an endless web as the carrier sheet making it a continuous operation.

In our evaluation kiss-roll coating shows promise but it would be necessary to reduce the amount of wax applied. This could probably be accomplished by reducing kiss-roll speed allowing more time for wax to drain from the roll. By present methods, this would imply using slower web speeds since the web drives the roll used to apply wax. This procedure however is not substantiated by our data. Coating 1418-42-14 coated at 97 feet per minute gave a pick up of 107%; coating 1418-42-16 coated at 45 feet per minute and about the same tension, gave a slightly higher pick up--119%. Slippage between the web and roll may have been a factor but does not in the mind of the author explain the apparent discrepancy.

The use of a larger diameter kiss roll should reduce wax weight by our coating method for two reasons. A larger diameter roll would turn proportionately slower at the same web speed applying less wax for the reason previously stated, and since the torque arm of such a roll would be longer, slippage should be reduced. Secondly, the distance between the surface of the wax in the coating tank and the crest of the roll would be greater giving greater opportunity for wax "runoff."
Reducing the kiss roll speed relative to web speed by braking or driving it separately would be another method. Still another is the use of a two roll system where the second roll meters wax from the application roll. These last two methods would probably be the most suitable roll methods for the application of wax in the 5 to 25% wax pick up range. Considering other methods, wax could probably be applied by simply spraying it on to the moving web. This would require equipment to keep the wax in the molten condition during the spraying operation.

PROPERTIES OF LABORATORY-WAXED KRAFT CREPE PAPER

The problems of waxing crepe paper will be better understood if we consider the change in certain physical properties of crepe paper after applying wax. Many different properties could be considered, but a specific application would almost certainly require specialized testing which could not be anticipated in a general evaluation.

Certain properties have been selected which should serve to broaden the background of information available on this type of converting problem.

Wax Pick Up by Solvent Extraction Method:

In order to interpret water vapor permeability and greaseproofness data as well as certain aspects of wax distribution, it will be necessary to accurately determine wax pick up. Ordinary methods for determining coating weight are based on the change in weight of a sample of specified area before and after waxing. Since crepe paper stretches these methods are not applicable because the amount of paper (on a weight basis) bounded by a given area changes with stretch.
The method chosen was the standard method for "Surface and Internal Wax in Waxed Paper" developed and used by the Marathon Corporation of Menasha, Wisconsin. Our copy of this method was received December 12, 1953, and is filed in the TAPPI ASTM Wax Testing Committee, Section VII, files in the Plastics Department. Samples of waxed paper are boiled for five minutes in each of three successive beakers of carbon tetrachloride to remove wax. 600 ml. dye beakers were used rather than the 1000 ml. tall-form beakers suggested. A 250 ml. Erlenmeyer flask, cooled with running cold water, was placed on top of each beaker as a cover and condenser. The solvent was renewed periodically to prevent the build-up of excess concentrations of wax.

The coated paper, conditioned a minimum of 2 weeks at 73°F, 50% R.H., was cut into 2 x 8" strips using an ordinary paper cutter fitted with metal stops fastened to the table surface with double-faced pressure sensitive tape. Using this method, sample dimensions were held to 1/64 inch as measured with a steel rule subdivided into 1/64 inch divisions. After cutting, the two ends of the sample were brought together and held with an ordinary wire staple giving a specimen which could be easily handled for weighing on the analytical balance or transferred from beaker to beaker during the extraction process. After conditioning about one hour, the specimen was weighed and the weight of the staple subtracted to give the actual weight of the sample. Using a second staple, a metal identification tag was attached to the outside of the specimen. The tags were made of brass shim stock, previously boiled in carbon tetrachloride to remove oil. The tags were easily marked with a sharp instrument such as the tip of a file. The sample was then weighed again to obtain the total weight--sample
plus staples and tag.

After extraction of the wax samples were reconditioned to 73°F, 50% R. H. until constant in weight. The difference between this weight and the weight before extraction is the weight of wax removed. The weight of the extracted sheet was calculated from the three weights obtained making allowance for the weight of the staples and metal tag. All weights were obtained to the nearest 0.001 gram. The values obtained by this method are given in Table II.

In order to assure that the Marathon method extracted all or nearly all of the wax, we reextracted several of the samples an additional 3 1/2 hours in a Soxhlet extractor using the same solvent--carbon tetrachloride. This apparatus washes the sample in a renewed bath of solvent about once every 12 minutes throughout the extraction period. The results are given in Table II in terms of percentage of total wax based on the weight of the paper after extraction. Comparing the Marathon and Soxhlet method we see that additional Soxhlet extraction removed only a very small amount of additional wax. If we consider that the additional extraction by the Soxhlet method removed all wax then the Marathon method can be said to remove 99.7% of the wax based on the results of the 6 samples tested. It is certainly true that both methods remove about the same amount of wax. Since the Soxhlet method is a recognized quantitative method of wax determination this comparison verifies that the Marathon method is adequate for our purposes. All samples were extracted by the Marathon method; these results will be the ones used in the interpretation of test results dependent on the accurate determination of wax load.
<table>
<thead>
<tr>
<th>Coating Code</th>
<th>Page-Position</th>
<th>Description</th>
<th>Waxing Tension (lbs./in., width)</th>
<th>Speed (ft./min.)</th>
<th>Marathon Extraction (ave. of 2 runs)</th>
<th>Additional Soxhlet Extraction (one run)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40-1</td>
<td></td>
<td>Dip Coated-Water Cooled</td>
<td>1.5</td>
<td>1</td>
<td>174.5*</td>
<td>174.8</td>
</tr>
<tr>
<td>41-7</td>
<td></td>
<td>&quot;</td>
<td>1.7</td>
<td>77</td>
<td>217.4</td>
<td>216.0</td>
</tr>
<tr>
<td>41-10</td>
<td></td>
<td>&quot;</td>
<td>0.8</td>
<td>83</td>
<td>238.0</td>
<td>238.6</td>
</tr>
<tr>
<td>41-14</td>
<td></td>
<td>&quot;</td>
<td>0.7</td>
<td>2</td>
<td>170.3</td>
<td></td>
</tr>
<tr>
<td>42-19</td>
<td></td>
<td>Kiss Roll Coated-Air Cooled</td>
<td>0.9</td>
<td>97</td>
<td>94.7</td>
<td></td>
</tr>
<tr>
<td>43-19</td>
<td></td>
<td>&quot;</td>
<td>0.8</td>
<td>50</td>
<td>105.6</td>
<td>105.2</td>
</tr>
</tbody>
</table>

* One run rather than two.
As part of this evaluation we cut and weighed several samples of the unwaxed base sheet. The average weight of a 16 square inch sample was 0.726 grams. Multiplying by 59.4, as indicated by the Marathon extraction method, gives a basis weight of 43.2 pounds per ream (24x36/500). This is the value used as basis weight in this investigation.

Wax Distribution—Surface and Internal Wax:

Crepe paper is by its very nature difficult to describe in terms of caliper or thickness and flatness. Before we can describe the distribution of wax applied to a crepe sheet it will be necessary to agree on the terminology used to describe the base sheet itself. By "thickness" we shall mean the average caliper of the paper itself, irrespective of the rugosities produced by the creping process. After a microscopic examination of the base sheet it would seem most expedient to describe the irregularities of the sheet in terms of the ridges (often turned over on top to give the appearance of "hooks") and crevices observed. The hooks occur primarily on one side of the sheet as observed in cross section view, and are often but not always associated with crevices observable on the other side of the sheet. Wax tends to lodge in these hooks and crevices. Instead of roughness we shall speak of "average depth of crepe" in describing the average distance between the two surfaces of the crepe sheet including the extremities of the hooks and ridges (but not the crevices), such distances being measured perpendicular to the face of the sheet. Two of the methods used to remove surface wax utilize firm rubbing with a cloth against a hard surface and in some cases appear to "flatten" the crepe as observed with the microscope. This refers to a decrease in the "average depth of crepe" and is probably the result of forcing down some
of the protruding hooks or otherwise smoothing the irregularities of
the sheet. Stretching the sheet with resultant loss of crepe would have
a similar effect in decreasing the average depth of crepe. This interpretation
should greatly simplify the microscopic examination of waxed crepe given in
Table III.

Total wax pickup has been discussed in the previous section and
the results tabulated for all of the coatings prepared. Three of these
coatings were selected for further study to determine the distribution of
the wax. They were selected to give a range of wax weights and coating
methods. Ideally we wish to know the proportion of wax external to the
sheet structure to that which fills the inter-fiber spaces or permeates
the fibers themselves. We usually refer to the former as surface wax
and the latter as internal wax. Using paper which presents a hard,
smooth surface and does not stretch or otherwise easily deform, surface
and internal wax are quite easily determined to a good degree of accuracy.
Crepe paper, however, presents several unique problems.

The usual technique of scraping the surface with a razor blade
or other sharp edged instrument removes considerable fiber along with
the wax. This was determined by dissolving the wax scraped from a strip
with carbon tetrachloride on a watch glass and observing it under the
microscope. Stretching the strip out by hand, to remove some of the
crape, then scraping, removed about the same amount of wax and about an
equivalent amount of fiber. Microscopic examination of a cross section
of the scraped specimen disclosed that wax was removed from the ridges of
the crape but remained largely undisturbed in the crevices and on the flat
areas of the sheet. The method has the effect of levelling the wax into
### TABLE III

**SURFACE AND INTERNAL WAX BY VARIOUS METHODS**

Mosinee Kraft Crepe--Laboratory Waxed, Basis Weight 43.2

<table>
<thead>
<tr>
<th>Sample Coating Code and Description</th>
<th>Total Wax Based on Wt. of Paper, % (Av. 2 runs)</th>
<th>Surface Wax Removal</th>
<th>Wax Removed Based on Wt. of Paper, % (Av. 5 runs)</th>
<th>Wax Removed Based on Wt. of Wax, % (Av. 5 runs)</th>
<th>Microscopic Examination (Cross Section View X100)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1418-41-7 Dip-coated Water-cooled High wax wt. per coating method</td>
<td>238.0</td>
<td>Heat oven, 58°C. Wipe to remove bead</td>
<td>117.4</td>
<td>65.6</td>
<td>0.3 Wax &quot;layer&quot; (one side) averages about 1/3 thickness of paper Small amt. of wax on both surfaces, primarily in crevices and hooks of crepe sheet. Little or no surface wax</td>
</tr>
<tr>
<td>&quot;</td>
<td>238.0</td>
<td>Flash heat and wipe</td>
<td>160.6</td>
<td>89.7</td>
<td>2.0 &quot; &quot; Wax &quot;layer&quot; average about 1/4 thickness of crepe. Crepe appears slightly flattened. Little or no surface wax</td>
</tr>
<tr>
<td>&quot;</td>
<td>238.0</td>
<td>Flood with C Cl₄ and wipe</td>
<td>100.3</td>
<td>56.0</td>
<td>9.9 &quot; &quot; Wax &quot;layer&quot; average about 1/10 of paper thickness. Crepe appears slightly flattened. Little or no surface wax</td>
</tr>
<tr>
<td>1418-41-11 Dip-coated Water-cooled Low wax wt. per coating method</td>
<td>170.3</td>
<td>Heat oven, 58°C. Wipe to remove bead</td>
<td>44.2</td>
<td>26.0</td>
<td>2.8 Wax &quot;layer&quot; (one side) averages about 1/4 thickness of paper Small amt. of wax on both surfaces, primarily in crevices and hooks of crepe sheet. Little or no surface wax</td>
</tr>
<tr>
<td>&quot;</td>
<td>170.3</td>
<td>Flash heat and wipe</td>
<td>92.8</td>
<td>54.6</td>
<td>1.5 &quot; &quot; Little or no surface wax</td>
</tr>
<tr>
<td>&quot;</td>
<td>170.3</td>
<td>Flood with C Cl₄ and wipe</td>
<td>57.5</td>
<td>33.8</td>
<td>2.9 &quot; &quot; Little or no surface wax; crepe appears flattened.</td>
</tr>
</tbody>
</table>
TABLE III (Continued)
SURFACE AND INTERNAL WAX BY VARIOUS METHODS
Mosinee Kraft Crepe--Laboratory Waxed, Basis Weight 43.2

<table>
<thead>
<tr>
<th>Sample Coating Code and Description</th>
<th>Total Wax Based on Wt. of Paper, % (Av. 2 runs)</th>
<th>Surface Wax Removal</th>
<th>Microscopic Examination (Cross Section View X100)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1418-42-16 Kiss-roll coated, air-cooled Medium wax wt. per coating method</td>
<td>109.3</td>
<td>Heat oven, 58°C. Wipe to remove bead. *</td>
<td>A.D.</td>
</tr>
<tr>
<td>&quot;</td>
<td>109.3</td>
<td>Flash heat and wipe</td>
<td>33.4</td>
</tr>
<tr>
<td>&quot;</td>
<td>109.3</td>
<td>Flood with C Cl4 and wipe. ** (one cycle only)</td>
<td>17.0</td>
</tr>
</tbody>
</table>

* 3 runs instead of 5.
** 4 runs instead of 5.
a flat though discontinuous layer. The stretched and scraped specimen
gave about the same appearance except that the crepe was somewhat flattened
and the wax layer appeared somewhat thinner.

Other methods of wax removal included: hanging the waxed specimen
in a oven controlled at 10 ± 2° F. above the melting point of the wax
and allowing the wax to drain off; flash heating the specimen with a
Bunsen burner flame and wiping immediately with a clean cloth; flooding
the surface of the specimen with carbon tetrachloride and wiping with a
clean cloth; and wiping off wax with a Scott paper hand towel, folded into
a pad, at room temperature. The last method failed because the wax was
too hard to be effectively removed at this temperature. The other three
methods appeared promising and were used to test the three samples selected.

All samples were conditioned at 73° F., 50% R. H. for a minimum
of two months before cutting and weighing. Samples were cut into 2 inch
x 4 inch strips. After treating to remove wax they were reconditioned
until constant in weight to the nearest 0.001 gram (usually 70 hours).

The amount of wax removed by the oven hang method was dependent
on the amount of time the specimen was heated in the oven. Separate
specimens of the same coating were treated for 1, 2, 5, 15, 60 and 120
minutes and the weight loss plotted against time. The change in weight
was large for the first 15 minutes but small thereafter giving a total
change of 2.6% from 1 to 2 hours. One hour was selected as the standard
test time. As the wax melts it runs down the sample and accumulates at
the lower edge before dripping from the strip into the waste pan. After
one hour the oven door was opened and the accumulated bead of wax removed
by quickly wiping with a clean cloth.

Specimens to be flash heated and wiped were first laid flat on a piece of absorbent blotter stock. A bunsen burner flame was played directly on the top surface of the specimen melting some of the wax. The surface was then wiped with a soft clean cotton cloth wiping parallel to the crepe ridges. This was repeated until no more surface wax was visible (as opposed to the glossy appearance of molten wax when melted with the flame). The sheet was then turned over on a new area of absorbent paper and the procedure repeated.

The same general technique was used to remove wax with solvent. The top surface was flooded with carbon tetrachloride using a cloth saturated with the solvent. A second clean, dry cloth was then used to wipe off the solvent. This was repeated until the surface had a dull appearance indicating that surface wax had been removed. Using a new area of absorbent paper the procedure was repeated on the reverse side.

The results obtained by these methods of wax removal are given in Table III in terms of per cent of wax removed based on the weight of completely extracted paper (Marathon extraction method), and also on the initial weight of wax present. Specimens were examined with the microscope to aid in the interpretation of "surface wax" removed by the various methods. Comments are given in the right hand column of Table III. Five specimens of each sample were tested by each method to determine the precision of the methods. Results are given as the average deviation of separate run results from the average. See Table III.

Considering the results as given in Table III, we see that the
most efficient method of removing wax was the Flash Heat and Wipe method. It removed roughly 90, 50 and 30% of the wax present respectively, from the three samples tested. The Oven Heat and the Flood and Wipe methods removed nearly comparable amounts of wax from the first two samples (containing surface wax), but varied considerably on the last sample (with little or no surface wax.) The reproducibility of the Oven Heat method appears good; the Flash Heat and Wipe and the Flood and Wipe methods gave poorer reproducibility. The poorer precision of the last two methods is probably due to the more inexact method of determining the end point of the test.

Since our main objective is to qualitatively determine the amount of surface wax we should focus our attention on the method which most accurately measures this property. The Flash Heat and Wipe method removes all of the surface as shown by microscopic examination. The problem is that it apparently removes internal wax as well. This might be explained by assuming that the capillaries of the cloth used to wipe the specimen are very fine, perhaps even finer than the capillaries formed by the paper fibers, so that wax could be absorbed from the sheet structure. This would be particularly true of "free" molten wax in the interfiber spaces. Wiping pressure may tend to move such wax to the surface of the sheet where it could be easily absorbed by the cloth capillaries. Theoretically, this wiping action could remove significant amounts of internal wax (as we have defined it) unless the wax could be melted in thin successive layers starting with that wax furthest from the sheet. Flash heating should be the best means of accomplishing this.

The theory behind the Flood and Wipe method was that the solvent would dissolve successive layers of wax as it was applied and gently wiped away. Again the cloth absorbs the solvent solution of wax by wicking action. Carbon
tetrachloride was chosen as the solvent because it dissolves wax quite slowly at room temperature. Again the major problem is one of determining the end point. The test removes less wax from comparable samples than the Flash Heat and Wipe method even though the end point is not well defined. This is probably because the solvent does not as seriously disturb the wax in the internal structure of the sheet.

Another problem occurs when we use this method on a sample which microscopically contains little or no surface wax. Technically such a sample should not be tested but this may not always be easy to determine. In a mill for example, it might be difficult to routinely determine the presence of surface wax by microscopic methods. A single Flood and Wipe cycle removed 15.6% of the wax present from such a sample as indicated in Table III.

The Oven Heat method appears to give the best estimate of surface wax for all three samples tested. Microscopic examination showed that the wax had drained well from the flatter areas of the sheet. A small amount of wax remained in the hooks and crevices (probably held by surface tension) but would probably amount to only a small percentage of the total wax removed. The method has the advantage that the end point can be easily controlled by simply timing the interval in the oven. We note that the method gave only negligible weight loss for a sample containing little or no surface wax—a further indication of the appropriateness of the method. A small amount of wax was observed on the surface of the treated specimen which previously contained no surface wax so that apparently extended contact with heat may sweat some of the wax from the interior of the sheet. Such wax did not drip off however and hence was detectable only by microscopic examination.
wax distribution and loss of crepe.

With this background of information on the general problems of determining wax distribution, it should be possible to devise improved methods. One such method occurs to the authors which should be an improvement of the Oven Heat method. We suggest heating the waxed specimen carefully in a circulating air oven set to control temperature at 5 to 10° F. below the melting point of the wax. Since paraffin wax has a sharp melting point it should be possible to soften the wax without melting. The wax could then be rubbed off with a paper towel without disturbing the internal wax. A small amount of wax would probably be left in the hooks and crevices but might be no more serious than presently retained by the Oven Heat method. The method would have the advantage of reducing the possibility of disturbing internal wax. It would probably be necessary to use sleeve ports in the oven opening to maintain adequate control of oven temperatures.

In making microscopic examinations, it was found necessary, because of sample variation, to mount and examine a number of specimens to obtain a representative picture of wax distribution. Strips measuring about 1/4 inch x 1 inch were cut with the long dimension perpendicular to the crepe ridges. They were cut by placing the sample on a glass plate and cutting with a thin razor blade. The blade was cleaned before cutting consecutive sides of the sample to avoid wax build up on the blade which might tend to "plow" up wax giving a false impression of the amount wax present when viewed in cross section. The strip was then supported in a groove in the top of a cork stopper and placed on the stage of a Bausch and Lomb microscope. Transmitted light from a substage lamp was found to be the best means of differentiating surface wax from paper fibers. Samples were first studied under 32 X magnification,
then 100 X for the final examination and description.

In addition to this rather superficial microscopic examination conducted in our laboratory to determine the effectiveness of the wax removal techniques, samples of the same three coatings were submitted to the code office for a more refined microscopic study and photomicrographs.

To prepare sections, a strip of waxed crepe paper (not treated to remove surface wax) approximately 1/2" X 3/4" was placed between two pieces of cellophane and inserted into a slot in a piece of cork. The cork and sample were placed in the microtome object holder and cross sections 25 microns in thickness cut perpendicular to the crepe ridges. A thin film of 2% collodion was applied and allowed to dry to enable sectioning. The section was placed on a glass slide and washed in absolute alcohol to dissolve the collodion and enable the removal of the cellophane and cork. The sections were then washed successively in 95, 70, 50 and 25% alcohol solutions, distilled water and mounted in "C" stain.

A strip of unwaxed crepe base sheet of approximately the same size was prepared by dehydrating in absolute alcohol, embedding in paraffin wax and sectioned with the microtome. After dissolving the collodion and paraffin wax the section was mounted in Canada Balsam for examination.

Photomicrographs of the prepared sections were made at magnifications of 125 and 250X (bright and dark field). Surface photomicrographs were also taken at a magnification of 20X (angle of incidence--80°).

Gloss prints of the photomicrographs obtained are given in Figures 2 through 10. Figures 2 and 3 are of the unwaxed crepe base sheet. Figures 4, 5 and 6 are of waxed crepe containing 238.0% wax and is the same as the
first sample listed in Table III. Figures 7 and 8 are of waxed crepe containing 170.3% wax and is the same as the second sample listed in Table III. Figures 9 and 10 are of waxed crepe containing only 109.3% wax (coated by the kiss roll method) and is the same as the last sample listed in the table. Under each picture of waxed crepe is a description of the sample or a list of observations based on a careful microscopic examination of the prepared sections.

Cross section views show the orientation of the crepe fibers very well and also give a clear picture of the distribution of wax, including surface wax. Surface views are not effective in showing the presence of wax. Comparing the cross section view of unwaxed crepe (Figure 3) with one for waxed crepe (Figure 5) it would appear that the unwaxed crepe specimen was swollen by the specimen preparation method. Perhaps, dissolving the collodion with an alcohol-ether solution distorted the paper structure giving the swollen appearance.
PHOTOMICROGRAPH OF UNWAXED MOSINEE KRAFT CREPE PAPER
(Surface View)

20 x (Angle of incidence---80°)

Figure 2
PHOTOMICROGRAPH OF UNWAXED MOSINEE KRAFT CREPE PAPER

(Cross Section View)

125 X Bright Field

Observations and Remarks--Optical Microscopic Examination (also see figure 2).

Average thickness of paper--Approximately 150 microns
(high 350 microns; low 40 microns)

Surface broken with very abrupt narrow ridges

Figure 3
PHOTOMICROGRAPH OF HEAVILY WAXED MOSINEE KRAFT CREPE PAPER

(Surface View)

20 X (angle of incidence--80°)

Description:
Coating method--Dip coated, metered and water cooled
Wax pick up (based on weight of paper)--238.0%
Wax--Socony Vacuum 125-127 AMP Fully Refined Paraffin
Coating Code--1418-41-7

Figure 4
PHOTOMICROGRAPHS OF HEAVILY WAXED MOSINEE KRAFT CREPE PAPER

(Cross Section View)

Dark Filed 125 X

Bright Field 125 X

Description: Same as Figure 4.
Observations and Remarks—Optical Microscopic Examination (also see Figure 4):
Average thickness of surface wax (one side)—
approximately 1/3 the average thickness of paper
Surface wax on crest of ridges—Very little (less than 5 microns)
Valleys—3/4 to completely filled with wax.
Penetration of wax into paper—complete
Wax observed in the lumen of some fiber cross sections

Figure 5
PHOTOMICROGRAPH OF HEAVILY WAXED MOSINEE KRAFT CREPE PAPER

(Cross Section View)

250 X Dark Field

Description: Same as Figure 5

Figure 6
PHOTOMICROGRAPH OF MODERATELY WAXED MOSINEE KRAFT CREPE PAPER

(Surface View)

20 X (angle of incidence-- 80°)

Description:
Coating method—Dip coated, metered and water cooled
Wax pick up (based on weight of paper)—170.3%
Wax—Sacony Vacuum 125-127 AMP Fully Refined Paraffin
Coating Code—1418-41-11

Figure 7
Description: Same as Figure 7

Observations and Remarks--Optical Microscopic Examination (also see Figure 7):

- Average thickness of surface wax (one side)—approximately 1/4 the average thickness of paper
- Surface wax on crest of ridges—Very little (less than 5 microns)
- Valley—2/3 or more filled with wax
- Penetration of wax into paper—Complete
- Wax observed in the lumen of some fiber cross sections

Figure 8
PHOTOMICROGRAPH OF LIGHTLY WAXED MOSINEE KRAFT CREPE PAPER

(Surface View)

Description:
Coating method--Kiss roll coated, air cooled
Wax pick up (based on weight of paper)--109.3%
Wax--Socony Vacuum 125-127 AMP Fully Refined Paraffin
Coating Code--1418-42-16

20 X (angle of incidence--80°)

Figure 9
PHOTOMICROGRAPHS OF LIGHTLY WAXED MOSINEE KRAFT CREPE PAPER

(Cross Section View)

Dark Field 125 X

Bright Field 125 X

Description: Same as Figure 9
Observations and Remarks--Optical Microscopic Examination (also see Figure 9):
Average thickness of surface wax (one side)--
Approximately 1/10 the average thickness of paper
Valleys (depth and width smaller than 15 microns)--completely filled
Valleys (depth and width larger than 50 microns)--less than 1/2 filled
Penetration of wax into paper--complete
Wax observed in the lumen of some fiber cross sections.

Figure 10
In summation, the best values obtained in our laboratory for the amount of surface wax appeared to be given by the Oven Heat and Drip method. This method rated the amount of surface wax present on three samples of the same order of magnitude as the microscopic examination. Dip coated samples contained an appreciable amount of surface wax. Kiss roll coated samples which were cooled slowly contained no surface wax; water cooling gave a small amount of surface wax.

Only three samples were tested so that caution must be observed in making any generalizations from the results obtained.

**Water Vapor Permeability and Greaseproofness:**

Institute Method 541 was used to determine water-vapor permeability. It consists essentially of exposing the sheet of coated paper to a temperature of 100°F while maintaining a relative humidity of 91% on one side of the sheet and 20% on the other. The permeability is calculated from the weight loss per unit time on the basis of the size of the exposed sample. Results are reported as grams per square meter per 24 hours. The test surface of the coatings prepared for converted product evaluation were exposed to the high humidity.

Tests were run using creased and flat specimens. Institute Method 535 was used for the creasing of paper for the water-vapor permeability test. The crepe was creased to form 2 cm. squares over the entire area of the sample, oriented with adjacent sides perpendicular and parallel respectively to the direction of creping. Each crease is made thru 180°, the final crease being made by gently lowering a weight onto the lightly creased specimen. Creases are made in opposite directions i.e. the second
fold is made about the side opposite to that about which the first fold is made. Continued creasing gives a zig-zag fold and appears like the side of an extended camera bellows.

Because crepe paper would be expected to be anisotropic with respect to crepe direction, two additional sets of specimens were creased and tested. One set was creased in a direction parallel to the crepe ridges only (across machine direction) and the second set creased perpendicular to the crepe direction only (in machine direction). All such creases were made using the normal 2 cm. crease-to-crease spacing.

Waxed crepe samples were also tested in the flat and creased condition for greaseproofness using techniques previously described in Project Report No. 13. Peanut oil was the reagent used. The method is appended to the above mentioned report.

The water vapor permeability and greaseproofness results for creased and flat specimens are given in Table IV. Water vapor permeability results are the average of two duplicate runs; greaseproofness results are the average of five runs. The table also contains total pickup, a brief description of the sample and the code for easy reference to Table I.

Paraffin wax is a good barrier against the transfer of water vapor. One of the major limitations in obtaining good protection with wax treated paper is the difficulty in obtaining and maintaining a continuous film of wax. Obviously, wax in the interior of the sheet cannot form a continuous film because of the presence of paper fibers. Wax, usually defined as surface wax, may also be permeated by paper fibers.
## Table IV

**WATER VAPOR PERMEABILITY AND GREASE RESISTANCE OF WAXED KRAFT CREPE PAPER**

(Basis Weight 43.2 Lbs./Ream 24x36-500)

<table>
<thead>
<tr>
<th>Coating Code</th>
<th>Description</th>
<th>Total Wax based on weight of paper, % Marathon Extraction Method (Average of 2 Runs)</th>
<th>Water Vapor Permeability*, g./24 hr./sq. m. Creased In Flat Normal Machine (Average of 2 Runs)</th>
<th>Greaseproofness** (Peanut Oil) Across Machine Flat Creased (Average of 5 Runs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1418-40-3</td>
<td>Dip Coated-Water Cooled Light wax Weight per Coating Method.</td>
<td>178.3</td>
<td>19.9 39.1 37.4 28.3***</td>
<td>72 hr. 316 sec.</td>
</tr>
<tr>
<td>1418-40-2</td>
<td>Dip Coated-Water Cooled Medium Wax Weight per Coating Method.</td>
<td>236.2</td>
<td>11.0 23.4 20.2 19.6</td>
<td>72 hr. 206 sec.</td>
</tr>
<tr>
<td>1418-41-8</td>
<td>Dip Coated-Water Cooled Heavy Wax Weight per Coating Method.</td>
<td>265.3</td>
<td>5.81 15.9 16.0 9.72</td>
<td>72 hr. 522 sec.</td>
</tr>
<tr>
<td>1418-42-13</td>
<td>Kiss Coated-Air Cooled Light Wax Weight per Coating Method.</td>
<td>94.7</td>
<td>66.6 77.6 85.2 72.2</td>
<td>5 hr. 846 sec.</td>
</tr>
<tr>
<td>1418-42-17</td>
<td>Kiss Coated-Air Cooled Heavy Wax Weight per Coating Method</td>
<td>115.3</td>
<td>29.0 61.2 38.6 58.8</td>
<td>48 hr. 720 sec.</td>
</tr>
<tr>
<td>1418-43-18</td>
<td>Kiss Coated-Water Cooled Light Wax Weight per Coating Method.</td>
<td>96.9</td>
<td>132 131 126 122</td>
<td>24 hr. 330 sec.</td>
</tr>
</tbody>
</table>

* 91% Relative humidity inside cup; 20% outside cup. Test Surface to high humidity.

** Oil applied to test surface. Time to show permeation.

*** One run only.
limiting the amount of protection against water vapor transfer because of the ability of the fibers to wick water thru the wax barrier. Thus the wax film must be continuous in the microscopic as well as macroscopic sense to obtain good protection. Maintenance of a continuous film, once obtained, is dependent to a large degree on the flexibility of the wax. Paraffin wax is quite brittle at room temperature often limiting its use as a coating on ordinary paper or board for general packaging applications. Because of its fragility it must be carefully handled to prevent fracture of the wax film. A common way of accomplishing this is to support the wax film on a stiff material such as heavy board stock. This method works well if the finished product is to be used in the flat form. Another method is to add the wax after the creasing and forming operation, for example the common waxed milk carton. Of course additives are also added to wax to improve its inherent flexibility.

Waxed crepe offers several new possibilities. Such a base sheet should bend in a manner likened to a coil spring, thereby resolving the strains applied to it (particularly in a creasing operation) over a wider area, thus placing less stress on a given area of wax film. The problem of course is to obtain a good surface film on a material of comparative loose structure and very irregular surface (crepe ridges etc.).

Considering the first three samples listed in Table IV, under flat water vapor permeability we see that a high degree of protection can be obtained. 265.3% wax, based on the weight of paper, had a permeability of only 5.8 grams/24 hours/square meter. Considering this and the other two samples, permeability increased linearly with decreasing wax pickup. The fifth result in the table, 29.0 gms. for a kiss roll coated specimen, also appears to follow the linear relationship. The fourth and sixth results are out of line probably
indicating that a total wax pick up in excess of 97% of the weight of the paper is required to obtain adequate surface wax.

Previous study of the same wax on a 25# sulfite base sheet* indicated that about the same amount of protection was obtained using only 41% wax based on the weight of paper. The magnitude of difference, 224.3% or about 106 pounds per ream (24x36/500), indicates that a much greater amount of wax is required to obtain equivalent protection using a crepe base sheet.

Perhaps the most important aspect of the data given in Table IV is the relatively small increase in the permeability of creased specimens compared with flat specimens. For the normal creasing method the permeability increased by an average factor of only 1.9 for the six samples tested. Considering the 1418-41-8 sample, creasing increased permeability by a factor of 2.7. The same wax coated on the sulfite sheet previously mentioned, to give a comparable amount of protection (4.74 gms./24 hrs./sq. m.) increased in permeability by a factor of 19* after creasing by the same method. Illustrating that permeability is closely related to wax flexibility but is also integrally dependant on the characteristics of the base sheet, a single previous result in this laboratory for Cereweld Amber microcrystalline wax coated on 25# sulfite increased in permeability by a factor of 8.1 (26.8 to 218 gms./24 hrs./sq. m.) on creasing by the same method.

The above discussion has considered only the standard creasing method. Considering the special creasing methods we see that creasing in the across machine direction (parallel to the crepe ridges) gave consistently

* Project Report No. 13
lower permeabilities than creasing in the machine direction. Sample 1418-42-17 was the only exception to this trend. Sample 1418-42-13 was the only sample which have a significantly greater permeability by the special creasing method than by the standard method; other samples gave reduced permeability. This would be expected since the total length of crease by the special methods is exactly 1/2 that by the standard method.

Sample 1418-41-8 was our best sample, increasing in permeability by a factor of only 1.6 when creased in the across machine direction while at the same time maintaining a good range of protection (5.81 to 9.72 gms./24 hrs./sq. m.). This was accomplished however only by using a very high wax load. This sample also demonstrated superior greaseproofness. It exceeded the test limit, 72 hours, in the flat condition and gave good comparative protection when creased, 522 seconds.

Considering the last sample of Table IV, some interesting observations can be made. This was the sample which appeared to have surface wax (see Table I) yet permeabilities are significantly higher then for the air cooled sample which did not have surface wax. Apparently water penetration from the waxer cooling bath seriously disturbed the wax coating resulting in the increased permeability of the sheet. Again, cooling with cold roll rather than water dipping seems to be advisable as a means of keeping wax on the surface of the sheet without suffering water penetration effects. Our waxer is not equipped with such a cooling roll at the present time.

The Greaseproofness of flat samples was better for all dip coated samples than for kiss roll coated samples probably because of increased
coating weight giving a surface film of wax. Creased greaseproofness appeared slightly better for kiss roll coated samples than for dip coated samples; the % change from flat to creased condition was significantly less.

**Loss of Crepe:**

As mentioned in previous sections of this report, normal waxing tensions under condition like or similar to our waxing procedure can be expected to remove some of the crepe of Kraft crepe paper. Our equipment requires that appreciable web tension be used, to obtain adequate and reproducible metering by the dip-roll wire-wound rod metering process, or sufficient paper tension to drive the kiss coating roll by the alternate process.

In order to appreciate the seriousness of the problem it will be necessary to know how much crepe was lost under various waxing conditions.

TAPPI Routine Control Method RC—215 is a commonly used method of determining the crepe in paper. It is quite crude, depending on the change in length of hand stretched specimens for the result obtained. Results are expressed to the nearest five percent. Since we are interested in the difference in crepe between waxed and unwaxed or various waxed samples of crepe paper, this method would not be expected to have the necessary precision. No other routine method was available which would fulfill our requirements.

After discussing the problem with Mr. W. A. Wink of our Paper
Evaluation Section we decided to submit representative samples of waxed Kraft crepe and a sample of the unwaxed base sheet for load-elongation study. Loss of crepe will then be determined from the differences in total stretch at maximum applied load. Ten specimens of each sample were tested using the Baldwin Southwark tester. Samples were conditioned a minimum of 2 weeks and tested at 73° F., 50% R.H. The loading rate was 60 pounds per minute. Specimen test size was 6 X 1", with the long dimension in the crepe machine direction.

Figure 11 is a direct tracing of several of the curves obtained. Curves A and B are of crepe paper waxed by the dip-wire wound rod metering method under high and moderately low waxer tensions respectively. Wax weight runs in reverse order as would be expected. Curve C is a tracing obtained of the unwaxed base sheet.

Starting at zero load each of the curves starts to rise. A yield point may be defined as the point of departure from linearity in this region of the curve. The curve then flexes and proceeds to rise at a less rapid but nearly constant rate. Finally, when the maximum tensile strength of the specimen is exceeded the specimen fractures and the curve falls rapidly. Total stretch is defined as maximum elongation measured at maximum load. It is expressed in % units based on the six inch test strip length.

Curves A and B have different yield points but show nearly the same tensile strength. Curve C, for unwaxed crepe, has a lower yield point and tensile strength than either of the waxed specimens.
WAXED (PARAFFIN) KRAFT CREPE - LOAD ELONGATION CURVES

TEST RATE: 60 INCHES PER MINUTE

(BALDWIN SOUTHWARK TESTER)

A. TOTAL WAX 174.5%, WAXING TENSION 1.5 LB./IN.
B. TOTAL WAX 217.4%, WAXING TENSION 0.5 LB./IN.
C. TOTAL WAX 0.0%, WAXING TENSION 0.0 LB./IN.

Figure 11
Considering loss of stretch of waxed specimens, the curve A specimen apparently lost a considerable amount of stretch during the waxing operation. The curve B specimen apparently lost very little. Loss of stretch is calculated by subtracting the average total stretch for a given sample from the total stretch for unwaxed Kraft crepe, both expressed in percentage units.

Table V gives the total stretch and loss of stretch for all samples tested. The amount of stretch at the yield point is also included together with the respective tensile strengths.

As anticipated, loss of crepe is directly related to the tension applied in waxing. The samples tested included three dip-roll coated and three kiss-roll coated samples. At equal applied tension (0.8 lbs./in. width), the kiss-roll coated sample suffered the greatest loss of crepe. This was also true for other values of applied tension. Returning to Figure I (Laboratory Waxer), we see that the use of non-turning steel rods to guide the paper over the coating roll may have increased the actual tension to which the sheet was subjected. If this is true it would explain the high loss of crepe by this method. Also since lower percentages of wax were applied by this method we must be careful to consider the possible effect of wax. Other than a possible effect on the sheet itself, as indicated by slightly higher yield and tensile strength values, the additional wax applied by the dip coating method may tend to lubricate the sheet as it passes over or around various machine components, thereby minimizing the total tension to which the sheet is subjected. Testing specimens drawn through the waxer with no wax and testing specimens of different wax percentages coated at the
same tension would be a great aid in further understanding the characteristics of our laboratory waxer.

In conclusion, considerable loss of crepe can be expected of a sheet such as Mosinee Kraft crepe if it is drawn through the waxing machine at high speeds and tensions. Our first clue of this is the low yield strength of the base sheet itself (See Figure 11). After the sheet is coated and the wax solidified, improved yield strength characteristics would be expected to aid in prevention of further loss of crepe on the waxing machine.

Note that our waxer has only one driven roll—the rewind roll. Controlled tension is applied to the web at the unwind roll but this is not the true tension to which the sheet is subjected. As the sheet is drawn from the unwind roll over various intermediate rolls and rods, particularly the wire wound rods, frictional forces would be expected to accumulate, increasing the total actual web tension, which would probably reach a maximum at a point just before it is reeled in by the rewind roll. The resultant situation must be somewhat analogous to that in an ordinary brake where the applied tension is likened to the force applied to the disk or shoe and resultant paper tension to the frictional output (torque) of the brake. This would explain why relatively small applied tensions, 1.5 lbs./inch width, gave much greater crepe loss than predicted by curve C of Figure 11.

A waxer incorporating additional driven carrier rolls between the unwind and rewind sections would probably minimize crepe loss by reducing the build up of tension at any given point on the machine.
TABLE V

WAXED KRAFT CREPE—LOSS OF CREPE, YIELD AND TENSILE STRENGTH

<table>
<thead>
<tr>
<th>Coating Code (Notebook-Page-Position)</th>
<th>Description</th>
<th>Total Wax, based on weight of paper, %</th>
<th>Marathon Extraction Method (Average of 2 runs)</th>
<th>Waxing Tension (lbs./in)</th>
<th>Yield Strength (lbs./in) (point, %) to yield strength</th>
<th>Maximum tensile strength (lbS./in)</th>
<th>Total Loss of stretch, % on waxing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control (unwaxed)</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>8.9</td>
<td>28.6</td>
</tr>
<tr>
<td>1418-40-1 Dip Coated-Water Cooled</td>
<td></td>
<td>174.5</td>
<td>1.5</td>
<td>6.6</td>
<td>0.6</td>
<td>11.7</td>
<td>15.8</td>
</tr>
<tr>
<td>1418-40-4 &quot; &quot;</td>
<td>217.4</td>
<td>0.5</td>
<td>5.7</td>
<td>0.5</td>
<td>11.9</td>
<td>27.2</td>
<td>1.4</td>
</tr>
<tr>
<td>1418-41-7 &quot; &quot;</td>
<td>238.0</td>
<td>0.8</td>
<td>6.0</td>
<td>0.5</td>
<td>11.2</td>
<td>25.4</td>
<td>3.2</td>
</tr>
<tr>
<td>1418-42-14 Kiss coated-Air cooled</td>
<td>99.9</td>
<td>0.3</td>
<td>4.8</td>
<td>1.0</td>
<td>10.6</td>
<td>26.3</td>
<td>2.3</td>
</tr>
<tr>
<td>1418-42-15 &quot; &quot;</td>
<td>95.0</td>
<td>0.7</td>
<td>4.9</td>
<td>1.0</td>
<td>11.4</td>
<td>23.8</td>
<td>4.8</td>
</tr>
<tr>
<td>1418-43-19 Kiss coated-Water cooled</td>
<td>105.6</td>
<td>0.8</td>
<td>5.0</td>
<td>0.8</td>
<td>10.7</td>
<td>22.2</td>
<td>6.4</td>
</tr>
</tbody>
</table>
Concerning conventional commercial waxers, it would be difficult to predict from our study just how serious the problem of loss of crepe would be. In a given application it would be necessary to determine the amount of crepe loss and the effect of the loss on the particular properties required of the completed product. However it appears that crepe paper may be waxed without a serious loss in crepe.
SUMMARY AND CONCLUSIONS

Using one grade of Kraft crepe paper and a single refined paraffin wax, a study has been made to give background information concerning the general problems related to the waxing of crepe paper. The study has been focused on those properties which were considered to be the most pertinent or revealing aspects of such a converting problem.

Using our laboratory waxer, two methods of coating were attempted. The first was a normal dip coating method utilizing wire-wound rods to meter excess wax from the paper surface. The second was a kiss-roll coating method whereby a stationary revolving roll, partially immersed in wax, is used to apply wax to the moving paper web. Coated webs were then either quick cooled by dipping into a cold water bath or allowed to cool at room temperature. Twenty separate coatings were made utilizing various combinations of the coating and cooling methods as well as various web speeds and tensions to give the maximum range of applied coating weights. Using the dip roll coating method wax weights as high as 265.3%, based on the weight of paper, were produced. The minimum applied was 154.4% using a slow web speed (2 feet per minute) and relatively high waxing tension (0.6 pounds per inch width applied to the unwind roll). The basis weight of the crepe sheet was 43.2 pounds per ream (24X36/500).

All dip roll coated samples contained an appreciable amount of wax on the surface of the sheet whether air or water cooled. Microscopic examination of cross sectioned specimens disclosed that
wax was distributed quite evenly over the ridges, crevices and flat areas of the sheet.

The kiss roll coating method gave a lower range of coating weights i.e. 95.0 to 115.3% on the same basis. Quick cooled specimens had a small amount of surface wax. Air cooled specimens contained no observable surface wax. Water vapor permeability of all kiss roll coated specimens was significantly higher than dip roll coated samples. Water cooling apparently damaged film continuity giving poorer water vapor barrier characteristics.

To obtain the lower range of wax weights metered roll coating would probably be required since the sheet itself absorbs as much as 115% wax. Such methods are the metered kiss-roll, the two-roll coater (one roll meters wax from the applicator roll) or the reverse-roll coater. Non-roll coating methods might include spraying wax on to the moving web or using a lightly waxed carrier sheet to transfer small amounts of wax to the crepe sheet under heat and mild pressure or using a felt pad to apply the wax.

Crepe paper elongates as force is applied in a direction perpendicular to the crepe ridges. Forces, resulting from paper tension, applied to the paper web in a waxing operation can be expected to remove some of the crepe in the sheet. Several waxed specimens were selected for load-elongation testing to determine the amount of crepe loss. As expected, the amount of crepe loss was proportional to the tension applied to the web at the unwind roll. 1.5 pounds per inch width of applied tension gave a crepe loss of 12.8% based on a 28.6% total stretch of a 6 inch long unwaxed crepe specimen. This sample
was dip-roll coated. Kiss roll coating gave proportionally greater crepe loss probably because of higher actual web tensions caused by drawing the sheet over non-turning steel rods to guide it over the coating roll. Such values illustrate the effects possible but the actual tension to which the web is subjected will vary with different waxers. It would be necessary to evaluate any given waxer and determine the effect of crepe loss on the particular properties of interest to properly understand and control crepe loss.

Because of crepe loss normal methods of determining wax weight are not applicable. It was necessary to extract wax by boiling in a solvent (carbon tetrachloride) to obtain accurate values for wax weight. Once it has been established that the extraction method removes essentially all of the wax, the amount of wax present is calculated from the original weight and the weight of extracted paper. Such data was obtained for all of the coatings prepared.

Several methods for determining the amount of wax on the surface of the sheet were tried. These included scraping the surface with a razor blade; wiping with a paper towel; heating in an oven and allowing the wax to drain off; flash heating with a bunsen burner and wiping with a cloth and dissolving wax with cold solvent. Of these, only the oven heat method appeared promising as verified by microscopic examination of the treated specimens. Judging from the data obtained, it will be difficult to obtain accurate values for surface wax using this type of base sheet. Applications requiring precise data of this type will have many problems to consider and may have to rely on a determination which most nearly satisfies the
the particular application i.e. the end-use type test.

Probably the most important property, of those tested, of heavily waxed crepe is the high degree of protection obtainable against water vapor transmission, and in particular the relatively small increase in permeability after severe creasing of the specimen. One of the coatings increased in permeability from 5.31 to only 9.72 grams/24 hours/square meter after severe creasing in the across machine direction. Creasing the same sample in both directions (essentially the same as TAPPI Method T 465 sm-44) gave a permeability of only 15.9 grams/24 hours/square meter. Permeabilities of this order would be of interest in many general packaging applications.

This same trend is observable of kiss-roll coated samples although the range of permeability was much higher. This is believed caused by lower wax weights and the absence of surface wax.

The greaseproofness of flat dip-coated samples was good—exceeding the limits of the test using peanut oil as the test reagent. Greaseproofness of creased samples was also good. One sample, coated by the kiss roll method, withstood the reagent 846 seconds (14.1 minutes). All samples tested withstood the reagent at least 3 minutes after severe creasing.

In conclusion, waxed crepe paper appears promising as a water vapor barrier particularly where serious creasing or other sheet deforming operations are anticipated. Wax weights as high as 200% of the weight of paper may be required however to obtain protection in the range of general interest.
The strength characteristics of waxed crepe paper, as well as its ability to stretch in a nearly linear manner with applied force (between the yield point and fracture point) might find application in a specialized use of this type of converted product. The wax present, its distribution, type and quantity may allow the converter to control certain of the strength characteristics to give a new and interesting material.