PROJECT REPORT FORM

PROJECT NO. 1685
COOPERATOR Institute
REPORT NO. 1
DATE February 27,1953
NOTE BOOK 758
PAGE 60 TO 69
SIGNED William C. Krueger
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CONSTRUCTION OF TEMPERATURE GRADIENT BAR

INTRODUCTION

In order to measure the blocking properties of wax for use in packaging a temperature gradient plate was designed and constructed. This report is to serve as a record of the details and materials of construction.

MATERIALS AND DESIGN

The plate itself is an aluminum plate 1-1/2 inches thick. A diagram of the plate and the placement of holes is shown in Figure 1. The heater is a Chromalox Cartridge heater No. C-509A, 600 watts, 3/4-inch diameter, with type A leads. The heater is approximately the same length as the width of the plate. The temperature of the heater is controlled by using a variable transformer (Powerstat). The water is piped to the cold end with 3/8-inch aluminum piping. The rate of flow was controlled by a constant head device. Well water at a temperature of 50°F. was used at a rate of approximately 1500 ml. per minute.

The plate itself is placed on a sheet of 1/4-inch thick Transite. The leads from the heater are brought into a single end condulet Type "E" which is attached to the plate on the end of the heater.

The bars used for weighting the sample are made from 1-inch square bar stock cold rolled steel, which was chrome plated.

The bars are 24-inches long.

The cushioning material used was 1/4-inch sponge rubber cut the same size as the bars. The rubber has a hardness of a Durameter-A reading of 26.

CALIBRATION OF GRADIENT BAR

The gradient bar was calibrated by determining the temperature at known points along the length and width of the bar. A student's type Leeds and Northrup potentiometer was used to determine the temperature as sensed by copper-constantan thermocouples with the hot ends under the rubber pressure pad and the cold ends at 0°C. The standard cell which was available was not reliable and the thermocouples were calibrated against an A.S.T.M. thermometer (64 cm. long and reading from -10 to +104 degrees Centigrade with 0.1 degree divisions). According to Dr. George Mueller of the Marathon Corporation their gradient bar temperatures are determined to the nearest 0.5°F. as recorded by a Brown Electronic Recording Potentiometer (-30 to +230°F). Blocking points, according to Dr. Mueller are significant to $\pm$2°F. due to the subjectivity introduced in picking the blocking point. Hence it is felt that our procedure for calibrating the gradient bar is more than adequate for the use intended.

The results of the calibration are shown in Table I. Some difficulty was noted with contact potentials while tapping the homemade key furnished by the physics group. A piece of Teflon sheet placed over the key helped minimize this effect. The thermocouple switch used was made up of a regular radio type 10 position wafer switch. A heavier switch with better contacts would be desirable for routine tests. A voltage regulator ahead of the variable voltage transformer would be good insurance against variation due to

any line voltage fluctuations.

TABLE I

CALIBRATION OF GRADIENT BAR

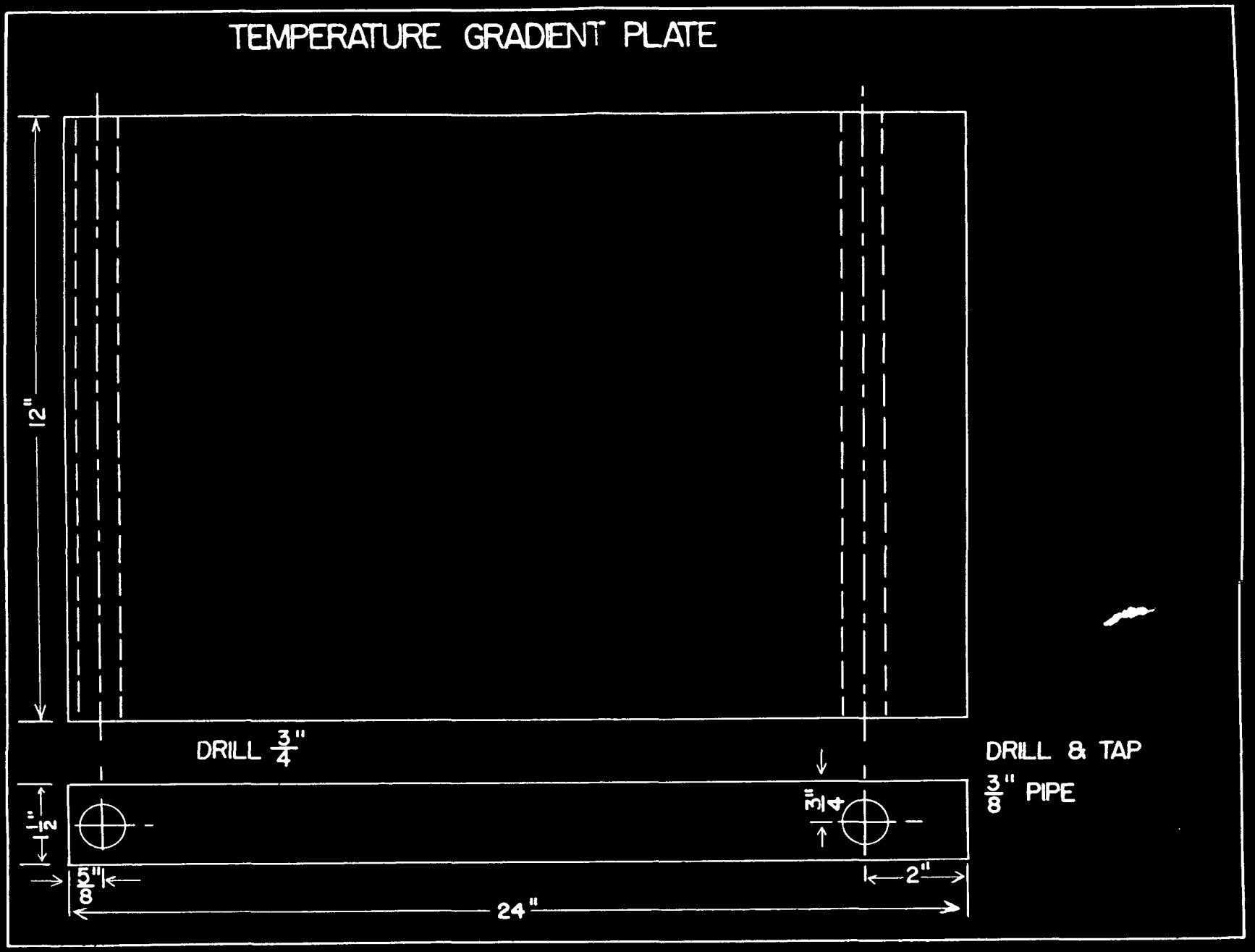
Distance from hot end. cm.	Temperature under bar, °C. Front	Middle	Back	
60	--	20.0	20.7	(0.7)
55	22.9	23.2	23.3	0.4
50	23.3	26.5	27.0	3.7
45	29.8	29.8	31.0	1.2
40	34.2	34.3	34.1	0.2
35	37.7	37.6	38.2	0.6
30	41.8	41.7	41.6	0.2
25	48.7	47.7	46.0	2.7
20	49.7	51.7	50.3	2.0
15	57.0	57.2	56.0	1.2
10	61.3	61.6	57.9	3.7
5	66.7	66.1	64.3	2.4

Note: Temperature of water: Incoming 10.0°C. Outgoing 10.3°C.

Flow rate: 1656 ml./minute

Variable voltage transformer: 60 volts

wck/rm


TEMPERATURE GRADIENT PLATE
12"
DRILL 3/4"
DRILL & TAP
3/8" PIPE
3/4"
1 1/2"
5/8"
2"
24"

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SEALING STRENGTH OF WAX USING A STRAIN-GAGE ATTACHMENT ON THE SCHOPPER-RIEGLER PAPER TESTER

This report covers the development of a strain-gage technique for measuring the sealing strength of wax paper. This development gives us equipment capable of measuring the small forces encountered in tests of this type. Our previous work with the Baldwin-Southwark tester in the Container laboratory and other tensile equipment available at the Institute indicated that the forces being measured are so small that none of the conventional equipment was entirely satisfactory.

The Physics department has recently designed and built a strain-gage amplifier which it was believed could be used for this purpose in conjunction with a suitable strain-measuring head designed and built by Mr. Hardacker and attached to the Schopper-Riegler tensile tester. The specimen had been sealed with a modified Palo-Meyer technique.

Procedure

The specimens tested consisted of wax paper, either commercial materials or experimental materials left over from other tests. Specimen width varied from 1-1/2 to 3 inches. The rate of testing was 5 inches per minute jaw-separation. The strain-gages were applied in a push-pull arrangement by cementing one S.R.-4 type A5-1 strain-gage on the inside of a spring

steel ring and another on the outside. The ring was approximately 3/4 inch wide and 2-1/4 inches in diameter. The spring was of approximately 15 mils thickness; it was a piece of clock spring. This spring-steel ring was fitted with brass clamps to take the place of the top set of jaws of the Schopper-Riegler paper tester. The regular clamp was then suspended from the bottom of the spring. In order to handle the wider sample width two spring paper clamps were used in the regular Schopper-Riegler jaws. The action of the spring clamps was strengthened by using two springs instead of one.

Since this is our first use of this piece of equipment the following operational directions are given for the strain-gage amplifier:

(1) Turn the electric switch to "off".

(2) Set meter potentiometer at zero.

(3) Connect all cables.

(4) Turn on power switch; allow amplifier to warm up over night if possible.

(5) Turn selector switch to zero and X-10 position.

(6) Adjust phase and the resistance balance control (helipot) to give maximum shadow angle with sharp edges on the electric eye.

(7) Switch to zero at one position and refine values.

(8) Switch to operate position desired (X-1 for paraffin wax seal-strength.).

(9) Turn meter potentiometer to maximum.

(10) Turn resistance balance control to zero the meter needle.

(11) Hang calibration weight from upper jaws, adjust extreme right control to adjust meter reading to correspond with calibration weight.

(12) Remove weight and adjust balance control to zero the needle of meter

(13) Replace the weight and adjust right-hand controls again. Repeat 12 and 13 as necessary.

(14) Proceed with testing. Use the high speed on the Angus-Esterline recording milliammeter.

Special note: A special clamp is used to hold the bottom clamp of the strain-gage ring whenever loading the tester or when adding calibration weight. It was found that the amplifier selector switch should not be turned to the "off" position during the periods of test as this turns the bridge current off and the strain-gages are slow (due to thermal effects) in recovering equilibrium. The force required to delaminate the various wax paper specimens was recorded on the Angus-Esterline recorder. There was some vibration noted in the Institute Schopper-Riegler tester and the drive motor was replaced in order to minimize this vibration. There is a slight friction of the pen on the Angus-Esterline recorder which must be taken into account while calibrating the instrument. The curves were quite irregular as might be expected. Table I gives the results as estimated from the curves. The strain-gage amplifier was readjusted after each test using a 50-gram weight for calibration. The chart speed was approximately 1.4 inches per minute.

TABLE I

SEALING STRENGTH OF WAX PAPER

Wax Paper	Sealing temperature °F.	Width of Specimen in.	Sealing Strength gram/in.		
			Av.	Min.	Max.
Brown wax paper	210	3	9.0	8.0	10.0
White wax paper No. 52-72345 Side A to side A	170	2	6.5	5.5	8.0
White wax paper 52-72345 Side A to side A	190	2	6.0	4.9	7.2
White wax paper 52-72345 Side B to side B	170	2	6.0	5.7	7.0
White wax paper 52-72345 Side B to side B	190	2	6.5	5.8	8.0
White wax paper 52-72345 Side B to side B	200	2	5.0	4.3	6.3
White wax paper 52-72301 Side A to side A	210	2	14.0	10.0	23.0
No. 52-72301 Side B to side B	210	2	12.0	10.0	12.8
Tastee Breadwrap	210	1.5	31.5	29.0	41.0
Wax-Seal A self-sealing wax paper with rubber latex on one side (Munising Paper Co.)	Room Temp.	3	4.5	3.5	6.2

Conclusions

This equipment appears to work fairly satisfactorily for measuring the sealing strength of wax paper. A good spread in the various wax papers was noted and by checking the calibration after each trial a reasonable degree of accuracy can be expected. The technique is superior to the Palo-Meyer wax seal-strength tester which we saw at Marathon and which cost approximately $3000 and which has some undesirable mechanical features.

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SCUFF TESTS

The Marathon Corporation has designed and built an instrument for evaluating a tendency for wax to scuff. It may be well understood that a wax that has a tendency to scuff or rub off would not be as desirable as a wax that does not scuff. One can imagine the annoyance that might be caused by a white film of wax deposited on a new suit by the wrapper on a loaf of bread. Very little consideration to this quality of wax has been given prior to the work done by section VIII of the TAPPI-ASTM joint committee for the study of wax. Dr. George Mueller of the Marathon Corporation is chairman of this committee and has submitted representative types of waxed paper for evaluation on the Marathon scuff tester which the Institute has recently purchased.

The following description is given of the waxed papers submitted by Dr. Mueller. (1) ES-116—a very high melting highly paraffinic wax, (2) P-4634--a wax-polyethylene blend, (3) A standard 140 to 142°F. melting point Standard Oil of Indiana paraffin, (4) TAPPI A--a Continential Oil Company paraffin wax.

PROCEDURE

All the tests were carried out under TAPPI standard conditions of humidity and temperature (50% relative humidity and 73°F).

Five 9-inch by 1-inch strips were cut from the center of the 2-inch wide rolls of wax paper. These strips were folded in half, making a double strip 1 inch by 4-1/2 inches so that the surface to be scuffed is on the outside. The specimens were weighed to the nearest 0.0001 gram on an analytical balance. The gripper jaws were moved to the forward position. The folded end of the test specimen was fastened in the gripper jaws.

The scuffing blocks were properly cleaned and the specimen placed between them. A 2000 gram weight was placed on top of the upper scuffing block. The screw crank was turned at approximately 120 rpm. until the specimen was free. The specimen was reweighed and the loss in weight noted.

The scuffing blocks were cleaned with a brass wire brush, then with a clean cloth saturated with carbon tetrachloride solvent after each test.

RESULTS

The results are given in Table I, which shows the original weight of the sample, the weight after scuffing, and the weight of wax scuffed off. There were three separate trials of 5 specimens each made with the low-scuff waxes and two trials made with the high-scuff waxes.

Figure 1 illustrates the scuffing produced by placing a piece of black velvet paper (flock-coated paper) under the top scuffing block

and 2000 gram weight so that the wax is picked up by the black paper. This gave a visual estimation of scuffing which appeared to agree quite well with the results of the Marathon scuff tester.

CONCLUSIONS

The Marathon scuff tester appears to differentiate waxes as far as the amount of wax which can be removed when scraped under standard conditions. This tester is being evaluated by a round robin test program sponsored by TAPPI-ASTM Technical Committee on Wax. Our results will be compared with those from other laboratories.

TABLE I

WAX SCUFF TESTS

(Marathon Wax Scuff Tester)

Wax: ES-116

	Sample	Original Wt., Grams	Wt., After Scuffing, Grams	Wt., of Wax Scuffed Off, Grams
Trial I	1.	0.3564	0.3549	.0015
	2.	0.3593	0.3577	.0016
	3.	0.3500	0.3486	.0014
	4.	0.3548	0.3535	.0013
	5.	0.3535	0.3524	.0011
	Average			.0014
Trial II	1.	0.3545	0.3525	.0020
	2.	0.3568	0.3547	.0021
	3.	0.3585	0.3566	.0019
	4.	0.3571	0.3558	.0013
	5.	0.3549	0.3535	.0014
	Average			.0017
Trial III	1.	0.3543	0.3533	.0010
	2.	0.3526	0.3516	.0010
	3.	0.3550	0.3539	.0011
	4.	0.3516	0.3500	.0016
	5.	0.3509	0.3493	.0016
	Average			.0013

TABLE I (Continued)

Wax: Standard 140/142 Paraffin

	Sample	Original Wt., Grams	Wt., After Scuffing, Grams	Wt., of Wax Scuffed off, Grams
Trial I	1.	0.3425	0.3390	.0035
	2.	0.3428	0.3397	.0031
	3.	0.3473	0.3446	.0027
	4.	0.3451	0.3429	.0022
	5.	0.3484	0.3457	.0027
	Average			.0028
Trial II	1.	0.3456	0.3427	.0029
	2.	0.3490	0.3464	.0026
	3.	0.3491	0.3463	.0029
	4.	0.3496	0.3470	.0026
	5.	0.3426	0.3399	.0027
	Average			.0027

TABLE I (Continued)

Wax: P-4634

	Sample	Original Wt., Grams	Wt. After Scuffing, Grams	Wt. of Wax Scuffed Off, Grams
Trial I	1.	0.3392	0.3374	.0018
	2.	0.3397	0.3381	.0016
	3.	0.3417	0.3401	.0016
	4.	0.3380	0.3367	.0013
	5.	0.3370	0.3355	.0015
	Average			.0016
Trial II	1.	0.3432	0.3418	.0014
	2.	0.3429	0.3414	.0015
	3.	0.3421	0.3409	.0012
	4.	0.3419	0.3403	.0016
	5.	0.3400	0.3386	.0014
	Average			.0014
Trial III	1.	0.3454	0.3437	.0017
	2.	0.3414	0.3396	.0018
	3.	0.3388	0.3371	.0017
	4.	0.3445	0.3432	.0013
	5.	0.3415	0.3399	.0016
	Average			.0016

TABLE I (Continued)

Wax: TAPPI A

	Sample	Original Wt., Grams	Wt., After Scuffing, Grams	Wt., of Wax Scuffed Off, Grams
Trial I	1.	0.3582	0.3522	.0060
	2.	0.3586	0.3533	.0053
	3.	0.3543	0.3491	.0052
	4.	0.3566	0.3510	.0056
	5.	0.3539	0.3483	.0056
	Average			.0055
	1.	0.3597	0.3540	.0057
	2.	0.3604	0.3552	.0052
	3.	0.3588	0.3536	.0052
	4.	0.3593	0.3543	.0050
	5.	0.3547	0.3489	.0058
	Average			.0054

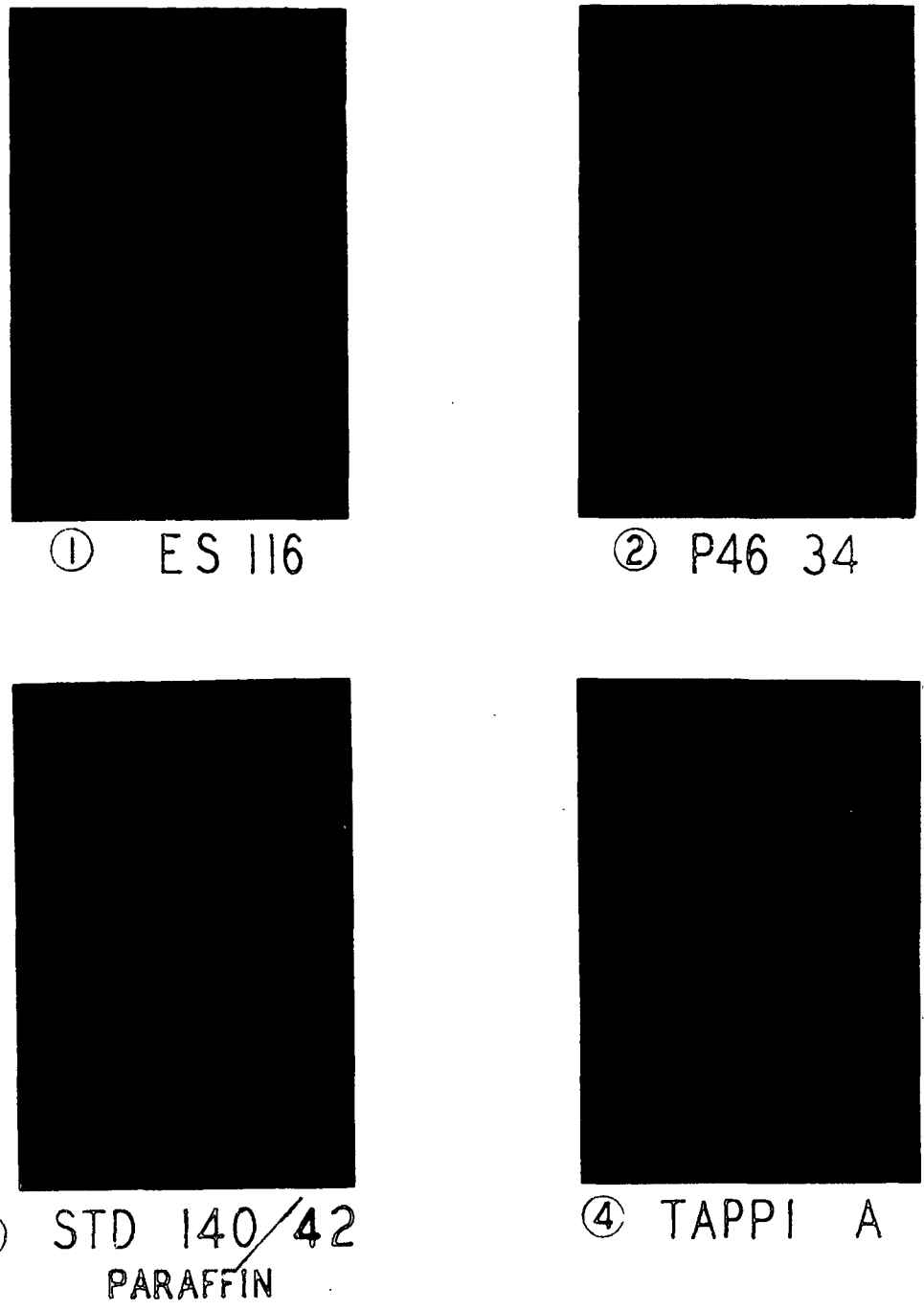

Figure 1. Scuffing Tests of Waxes

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SURFACE WAX DETERMINATION

INTRODUCTION

Some question exists as to the validity of the quantitative results obtained by our routine method of determining the surface wax weight of waxed strips prepared for blocking point determination. Specific conditions have been set down by TAPPI-ASTM Committee VI as test coating procedure. Under these conditions the standard sheet is saturated in a molten wax bath, well doctored on one side and coated with four to six pounds per ream on the test surface. The method in question pertains to the determination of the surface wax weight and its control within the limits specified. One by 9.52-inch strips are cut from the center of the coated web, weighed, placed upon a glass plate and scraped with a sharp razor blade in long even strokes until the very slightest fiber drag is noted. The strip is then brushed to remove particles of wax, again weighed and the weight loss calculated to read in pounds per ream.

The method would seem valid although lacking somewhat in precision. Indeed the basic question may find terminus only in serious consideration of the more academic question, "Where lies the surface of a saturated and coated sheet of paper?"

P

The purpose of this report is to explain two methods that have been set up in order to gain further information as to the validity of our present routine method and possible improvement of it.

METHOD I

Description

The residual removed by the scraping process is dissolved in toluene in order that the fiber content may be determined. The fiber-in-toluene dispersion is then centrifuged at high speeds to attain a measurable column of packed fibers. Standard 15-ml. centrifuge tubes were found to be too large in bore for the small quantities of fiber concerned, but on the other hand, it was necessary to use rather large volumes (about 15 ml.) of toluene to assure that the fibers are completely washed into the tube. Special tubes were made to suit the purpose from Pyrex glass tubing. The most satisfactory were 4-3/4 inches in over-all length, with a 15 mm. diameter three-inch long reservoir on top, tapering to a thick wall 0.05-inch capillary stem 1-3/4 inches long. The packed fibers attained a measurable height in the capillary portion of the tube. Some difficulty occurred in securing a uniformly packed column particularly at the higher percentages of fiber recovery. The hand-made centrifuge tubes were not calibrated to read on a pounds per ream basis but simple scraping of known weights from the standard base sheet and intermittent centrifuging would seem the most simple method to accomplish this calibration.

Data:

Waxed Sheet Tested - Marathon submitted calibration strip W - 3613

Surface Wax Weight - 5 lb./ream (Marathon)

Surface Wax Removed (lb./ream)	Packed Fiber Recovered (cm. column length)
2.4	0.42
5.0	3.74

METHOD II

Description

The residue was washed into a standard 15-ml. centrifuge tube with toluene. The fibers were washed three times by decantation with toluene in order to remove all wax from the fibers. The fibers were then flushed from the inverted centrifuge tube with the aid of a small curved pressure pipet and acetone, onto a six-cm. watch glass. The acetone was evaporated at 180°F. in a platen heated circulating air oven. The fibers were partially covered with another watch glass and:

1. Preliminary Run - Cooled, desiccated over calcium chloride for 45 minutes.
2. Runs 1 and 2. - Cooled, and allowed to stabilize at room temperature and humidity for 30 minutes.

The samples were then weighed, using a two-cover glass tare and the weight stated in pounds per ream.

Data:

A - 1st scraping (to feel of fiber)

B - 2nd scraping (to app. 5 lb./ream)

Run	Surface Weight Removed (lb./ream) A	A Plus Surface Weight Removed (lb./ream) B	Fiber Recovered (gm.)	(lb./ream)
Pre. A	3.17		0.0052	0.52
" B		5.59	0.0077	0.77
1 - A	3.42		0.0023	0.23
1 - B		5.14	0.0054	0.54
2 - A	2.07		0.0025	0.25
2 - B		4.93	0.0059	0.59

CONCLUSION

Although rough, the results would seem to demonstrate that considerable fiber is removed in continued scraping of the strip to remove a full five pounds per ream of surface material. In fact, it would seem that only about two pounds per ream could be classified as surface wax. Our present routine method would seem satisfactory within the limits of accuracy required.

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James M. Throne

POTENTIOMETER AND THERMOMETER CALIBRATION

INTRODUCTION

The recently received Electronik Potentiometer and a good grade thermometer were calibrated against a National Bureau of Standards Certified Thermometer. The potentiometer will be used to record the temperature gradient along the long axis of the blocking point apparatus. The major purpose of the calibration was a careful check of the instrument, as received, against the standard thermometer. Correction factors or possible internal adjustment of the instrument will assure accurately recorded temperatures.

The laboratory thermometer was calibrated for use in such routine calibration checks as the potentiometer may require.

A preliminary check of the range maximum calibration with a boiling water bath indicated that considerable error does exist.

Measuring Equipment:

A. Potentiometer

Name—Type 153 Electronik Recorder
Manufactured by—Minneapolis Honeywell Regulator Company
Features—Single point, fast sweep, strip chart recording potentiometer
Calibration—0 to 100°C. in 0.5°C. for Copper-Constant thermal-couples
Used with—Alnor, six point, double pole sweep switch (1 r.p.m.)

Remarks--The ball point recording pen furnished with the instrument does not give a continuous record at slow chart speeds. Another has been ordered.

B. Thermometer

Type--Liquid in glass
Name--Wilkens Andersen Company, Germany
Length--70 centimeters
Range-- -5 to +104°C. in 0.1°C.
Immersion--Total
Ice point--+0.05°C.
Conditions: Stored a minimum of 3 days at 72°F. Total immersion in saturated ice bath in Dewar flask. Read in one hour, read and recorded at two hours.
Remarks--Reacts slowly
a. To measure to nearest 0.1°C.--age minimum of 5 minutes
b. To measure to nearest 0.02°C.--age minimum of 10 minutes
c. For maximum precision on slowly rising temperature--allow temperature to rise no faster than 0.05°C. per minute.

Standard Measured Against:

National Bureau of Standards Certified Thermometer

Type--Liquid in glass
Name--Wesco 3406149 NBS 60809
Length--60 centimeters
Range-- -5 to +105°C. in 0.1°C.
Immersion--Total
Tested--August 1934 and 1944 by National Bureau of Standards
Ice point--+0.06°C. (1944--NBS)
+0.10°C. (1953--This laboratory)

Calibration:

A. Constant Temperature Equipment

1. Well insulated mineral oil bath
2. Knife blade immersion heaters, 250 watts--Variac controlled.
3. Lightning mixer--Variac controlled.
4. Small cooling coil
5. Mercury to platinum switch.
6. Precision scientific relay
7. Potentiometer operational pen--to record relay action.

B. Conditions

1. Constant temperature room
2. Potentiometer calibrated <u>in situ</u>
3. Bath held to constant temperature for a minimum time lapse of 50 minutes.
   a. Maximum precision of bath temperature control--0.01°C.
   b. Average precision of bath temperature control--0.03°C.
4. Thermometer used to indicate mean temperature of emergent stem.

C. Immersion

Both thermometers were immersed to a depth of 10°C. and the thermal-couple well to a depth of 6 inches. Since both thermometers were of the total immersion type, the observed temperature readings were corrected for emergent stem error according to the relation:

$$\text{Stem Correction} = 1.6 \times 10^{-4}\ n(T^\circ - t^\circ)$$

where n = number of degrees emergent from bath
T° = temperature of the bath
t° = mean temperature of the emergent stem

Data collected under the "Reading of Thermometer" heading of Table I has been corrected for emergent stem error and ice point elevation.

Should good approximations be required without the necessity of making emergent stem corrections, under conditions similar to the calibration, use Table II.

Data :

TABLE I

Reading of Thermometer (see above)	Correction to Thermometer Reading	Temperature	Correction to Potentiometer Reading	Reading of Potentiometer
+0.05°C.	-0.05°C.	0.00°C.	---	---
27.11	-0.03	27.08	-0.1°C.	27.2°C.
34.40	-0.09	34.31	+0.2	34.1
42.10	-0.07	42.03	+0.2	41.8
50.02	-0.05	49.97	+0.5	49.5
57.97	-0.04	57.93	+0.4	57.5
64.82	-0.03	64.79	+0.6	64.2
72.57	-0.06	72.51	+0.7	71.8
79.18	-0.05	79.13	+0.7	78.4
86.38	-0.06	86.32	+0.9	85.4
96.04	-0.00	96.04	+1.1	94.9

TABLE II

Thermometer Observed Temperature*	Temperature	Correction to Observed Temperature
+0.05°C.	0.00°C.	-0.05°C.
27.10	27.08	-0.02
34.36	34.31	-0.05
42.02	42.03	-0.01
49.88	49.97	+0.09
57.73	57.93	+0.20
64.50	64.79	+0.29
72.13	72.51	+0.38
78.64	79.13	+0.49
85.69	86.32	+0.63
95.13	96.04	+0.91

* Note calibration, immersion, par. 3

jmt/rm

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BLOCKING POINT DETERMINATION

INTRODUCTION: The only logical manner to begin and proceed with this report would seem to be a chronologic sequence of its several phases. Accordingly we shall discuss the final calibration of the thermal couples used to sense gradient temperature conditions of the blocking point apparatus. Next, we shall describe our experience with various methods of thermal contact of the couples with the gradient plate and last we shall attempt to describe factors of gradient bar stability, coating variables and conditions adopted for the TAPPI-ASTM Section VI Round Robin Tests.

CALIBRATION OF THERMAL COUPLES

Six 28-gage copper constantan thermal couples were calibrated against an ASTM thermometer (64 cm. long reading from -10 to 104°C. with 0.1 degree unit scale divisions) to be used as temperature sensing elements along the long axis of the gradient temperature plate. The calibration was accomplished with the use of a large volume, carefully controlled constant temperature bath. Control of the bath was accomplished with the use of a three-wire De Khotinsky relay and as many as three emersion-type knife blade heaters for an average precision to 0.16°C. The bath was held to temperature for a minimum period of one hour to allow couples and thermometer to stabilize. A Student's potentiometer and galvanometer (Leeds and Northrup), a student constructed standard cell furnished by the physics department, and a single pole radio

waffer type selector switch comprised the major measuring equipment. A curve was plotted for temperature vs. millivolts and found on analysis to give a relation of the type F.°=42.5 mV / 34.7 within the limited range calibrated. Average deviation of the calibration points was ± 0.6°F, with a maximum deviation of 3.10°F. An attempt to re-establish the calibration points gave an even greater deviation. The measuring equipment was looked to as the major cause of our lack of precision.

RECALIBRATION OF THERMAL COUPLES

A Type-K potentiometer, a galvanometer with a sensitivity of 16.4×10^{-8} amp/mm. (20 ohm internal resistance) and a commercial standard cell were used in our attempt at a more precise calibration. This equipment gave a reproducibility of readings under actual test conditions of ± 0.002 mV. The average deviation of points from the calibration curve was ± 0.09°F.; the maximum deviation was 0.81°F. This equipment was considered to give an over-all precision in the region needed for final blocking point determinations to be stated, and precise to, one degree Fahrenheit.

DESCRIPTION OF THE GRADIENT TEMPERATURE PLATE

The description is essentially as previously stated, per Report I dated February 27, 1953, with the following additions:

1. The plate was etched with acid along either edge in 0.5 cm. divisions.
2. Foam rubber pads--1-1/2" by 5/8" by 24" cream foam rubber.
3. Surface resting thermal couple elements, to be more fully explained in the next section.

THERMAL CONTACT

A preliminary study of gradient temperature conditions along the long axis of the gradient bar indicated that the actual method of contact was very critical. An extended study was made to determine the optimum temperature conditions as a reference of good thermal contact. Theoretical consideration of the matter assumes the gradient condition to give a near linear relation when plotted as temperature against distance from the hot portion of the plate. No inversion of temperatures is expected but was in fact demonstrated by empirical evidence when contact was made by simply placing the reference couples under the weighted rubber pads. Factors were thought to be: thermal conduction along thermal couple wires, intimate contact of couple with plate and insulating properties of the pressure pad. It is noted that good thermal contact, in this sense, implies good electrical contact. We must therefore guard against such stray currents as might disrupt the very sensitive thermal electric circuit. Resting couples retained by such substances as sponge rubber, firm rubber, pressed paperboard and many others were found to give unsatisfactory results even though considerable pressures were used. Cork was found to have good thermal insulating qualities as well as satisfactory placement retaining qualities. It gave fair reproducibility of temperature when recorded, removed, replaced, and again recorded. Permanent deformity of its surface was seen to develop under pressure and was considered to be its major drawback. Saran plastic pressure pads were decided upon as the best available material to secure the couple to plate contact.

Consideration was given to the use of couples tapped into the plate. This could be accomplished by tapping into the side of the plate or tapping from the bottom surface to near top surface. Either method would have the advantage of allowing deeper immersion of the reference couples, and thus reduce possible conduction of heat along the thermal couple wires. This might be particularly important because of the fact that the couples were calibrated in a deep well immersed into a constant temperature bath. Considered opinion was that while this method has the several important advantages mentioned as well as being easier to maintain from a purely routine mechanical viewpoint, it would not seem to be as sound an approach to the temperature existing between the weighted pad and the gradient plate, i.e. surface temperature, this being the actual temperature to which the waxed strips are subjected during the operation of the test. We decided to perfect our method of surface resting thermal contact.

It was found that pressure of considerable magnitude was necessary to attain maximum surface temperature. It would seem that enough pressure must be used to deform the surface of the pressure pad to such a degree as to envelop the couple and thus protect it from possible convective losses of heat. Pressure of higher magnitudes was necessary for the higher temperature thermal couples than for the couples near the median of the plate (nearer room temperature) which would seem to substantiate the above assumption. A device capable of spring loading each of six separate saran pressure

pads was constructed. The springs are mounted on a 2-inch channel iron which is supported over the plate in a rigid manner. Supports were placed under the table for additional stiffness. The gradient plate itself is thus free of distortion. Each pressure fixture is equipped with a threaded bolt that allows varying amounts of applied pressure. The springs were calibrated to the nearest pound pressure in terms of number of turns applied to the threaded bolt. (Twenty pounds of pressure was delivered to the 3/4-in. diameter Saran pads by 6-1/3 turns.) No change in thermal e.m.f. has been noted above sixteen pounds applied pressure.

It is noted that the above method of contact in attaining maximum thermal contact also attains good electrical contact. This fact presented a unique circuit difficulty with the single-pole selector switch. An extraneous circuit was shown by empirical evidence to exist between the reference couples in contact with the plate and the common lead within the switch. Another waffer disk was added to the selector, giving a double pole switch, and the difficulty overcome.

Consideration was given to possible 'stray' currents that might exist because of a difference in potential between the gradient plate and the measuring apparatus. The couple leads were shielded and the shield grounded to a cold water pipe. More extensive shielding may be necessary in the future.

GRADIENT PLATE STABILITY

In order that the gradient plate establish and maintain a time stable gradient, the constancy of such factors as water temperature and pressure, room temperature and the voltage fed to the heater must be maintained. The cooling water source is a direct connection to the well water supply, found to run 50.1°F.with an observed variation of ± 0.1°F. A simple constant head device assures constant flow rate within very close limits. The apparatus is maintained in a constant temperature room to negate difficulties due to ambient temperature conditions. The voltage, as supplied by a multitap transformer, is recorded at regular intervals during the test sequence to determine normal variations. A 48-hour recording was made of the standard line voltage and found to vary in a more or less orderly fashion (day to night) by four volts. This degree of variation was considered as a possible source of error but not too serious at this time. More precise temperature control of the hot portion of the gradient plate is anticipated in the near future.

COATING VARIABLES

Figure 1 is a photograph of the Institute modified Standard Oil Development Laboratory type waxer. An extensive study was made of its characteristics with thought to understanding, first, its versatility and secondly its ability to produce and reproduce the specific coating conditions required by the proposed TAPPI-ASTM test standard. Several things might be mentioned. As expected, the larger the diameter of the wire of the wire wound rod, the heavier the coating weight of the surface wax. A wire diameter of 0.013 inch was found to give a test surface wax

weight of 4 to 6 pounds per ream with but slight changes in tension required to maintain the coating weight within these limits for all waxes under consideration at this time. All coating was done at a paper speed of 67 feet per minute to conform with the 0.5-second time delay from last doctoring rod to cooling bath surface as specified in the round-robin method. Air cooled coating was accomplished under the same conditions with the addition of an off-the-machine rewind roll at a distance of 13 feet from the last doctoring rod (delay--11.6 seconds). No sticking occurred on the additional roll and the air cooled wax paper did not block at the wind-up roll.

Some question exists as to the validity of the surface wax weight determination as it has been run in the past. It would seem to be largely a question of interpretation of the word "surface." By our present method, the test surface of a 1 by 9.52-inch strip is scraped with a sharp single edge razor blade to "feel" of paper fiber. The strip is weighed on the analytical balance. The weight loss in grams is multiplied by 100 to express the weight of wax removed in pounds per ream. The surface weight of the Marathon calibration strip by this method was found to be 2.2 pounds per ream. A telephone conversation with Mr. Daninbrink of Marathon gave the surface wax weight to be five pounds per ream. We decided to attempt recovery of the fiber removed by the hand scraping method for the Marathon sample so that some agreement might be reached on this determination. By the first method, the scrapings were washed with toluene into a capillary centrifuge tube of our own construction and centrifuged at high speed. The recovered fibers were packed

into the capillary and the height of the column measured in centimeters.

For the second method, the fibers were handled as before except that they were thoroughly washed with toluene by decantation, oven dried at 180°F. and weighed on the analytical balance. Data varied considerably for the two methods. The second method gave 0.84 pound per ream of recovered fiber when the test surface was scraped to remove a full five pounds per ream and 0.23 pound per ream recovered when 3.4 pounds per ream were removed. The first method gave 3.7 and 0.4 centimeters respectively, for the two conditions. The height of the packed column was not calibrated to give results on a pound per ream basis but nevertheless the ratio of values should have been preserved.

A possible explanation might be that the fibers bound within the small capillary (0.05-inch diameter) and did not pack in uniform manner. If this were true, the second method might more closely represent the actual amount of fibers recovered.

In either case it would seem that in light of the considerable variations possible by the hand scraping method of surface wax determination, more study of the effect of large variations of surface wax weight on the blocking temperature is indicated. Several committee members have reported that surface wax weight would seem to be independent of the blocking temperature and in fact suggest that sufficient wax be used in each case to obtain maximum gloss. Should further study disclose the surface wax weight to be critical, more consideration must be given to a standard method of surface wax determination.

TABLE I

RESULTS OF ROUND-ROBIN BLOCKING POINT TESTS

Committee on Blocking, Section VI
TAPPI-ASTM Technical Committee
on Petroleum Wax

Test Conditions

Wax bath temperature--175°F. ± 2°F.
Water bath temperature--50°F. ± 3°F.
Time from last doctor rod to water bath surface = 0.5 sec.
Specimens aged--24 to 36 hr. (50% R.H., 73°F.)
Blocking plate used--Modified Marathon Blocking Plate
Foam rubber pads--S.O.D. Soft Sponge
Thermocouple placement: on surface under 20 p.s.i. spring pressure
No. specimens of each Wax--Five
Ream size--24 inches x 36 inches--500 sheets

Sample	Cooling	Blocking Temp. °F. Av. Dev.	Reported Blocking Temp. °F.	Wax Weight Test Surface lb./ream	Type of Blocking Point
Sinclair P-259	Water	114.1 ± 0.09	114	4.4	Sharp
	Air	114.3 ± 0.05	114	5.1	Sharp
Sinclair P-258	Water	101.6 ± 0.25	102	5.3	Fair
	Air	103.8 ± 0.16	104	5.3	Fair
A. R. C. #383570	Water	107.0 ± 0.10	107	5.3	Sharp
	Air	107.7 ± 0.15	108	4.6	Fair
S. O. D. Esso W-4314	Water	109.2 ± 0.58	109	4.4	Fair
	Air	110.2 ± 0.02	110	4.3	Sharp
S. O. D. Esso W-4317	Water	118.8 ± 0.85	119	5.1	Poor
	Air	121.8 ± 0.15	122	4.1	Fair
Sun Oil X-9883	Water	109.3 ± 1.84	109	5.6	Poor
	Air	117.3 ± 0.05	117	5.4	Fair
Cities Service CS-B-3	Water	120.2 ± 0.18	120	5.3	Poor
	Air	122.3 ± 0.04	122	5.1	Sharp
Marathon Calib. Stp. W-3613	Water	101.5 ± 0.03	102	2.2	Sharp

TABLE II

Results of Round-Robin Blocking Tests--Second Run

Committee of Blocking, Section VI
TAPPI-ASTM Technical Committee

Test Conditions

As per Table I
No. specimens of each Wax -- Three
New wax samples used in each case.

Sample	Cooling	Blocking Temp. °F.	Av. Dev.	Reported Blocking Temp. °F.	Wax Weight Test Surface lb./ream	Type of Blocking Point
Sinclair P-259	Water**	114.4	0.04	114	5.7	Sharp
	Air	114.9	0.03	115	5.5	Sharp
Sinclair P-258	Water	101.9	0.30	102	4.4	Fair
	Air	104.2	0.26	104	5.3	Fair
A. R. C. No. 383570	Water	107.3	0.20	107	4.1	Sharp
	Air	107.6	0.04	108	5.4	Fair
S. O. D. Esso W-4314	Water	110.0	0.08	110	6.3	Sharp
	Air	110.4	0.04	110	5.6	Fair
S. O. D. Esso W-4317	Water*	121.6	0.80	122	6.0	Poor
	Air	122.0	0.00	122	5.8	Fair
Sun Oil X-9883	Water	113.8	0.28	114	6.0	Poor
	Air	116.3	0.10	116	4.9	Fair
Cities Service CS-B-3	Water	119.7	0.40	120	5.7	Poor
	Air	123.1	0.08	123	5.1	Fair
Marathon Calif. Stp. W-3613	Water	101.4	0.02	101	2.8	Sharp

Note: * -- 14 Strips run and averaged.
** -- 2 " " " "

The small waxer was found to give satisfactory coatings on an experimental basis but will need redesigning with thought to greater structural strength in order to adapt it to more routine use.

ROUND ROBIN TESTING

Seven waxes and a Marathon submitted calibration strip were tested by procedure outlined per letter from Mr. H. F. Hitchcox, Chairman Section VI, Blocking Committee, dated May 13, 1953. Five test strips of water- and air-cooled waxed paper were tested for the first run; three test strips of each for the second run. Results were reported on a straight average of total strips run. Temperatures were given to the nearest degree Fahrenheit and the average deviation indicated. The character of the blocking point was judged by a visual qualitative method as sharp, fair, or poor. See Tables I and II for reported results. We noted good correlation between the visual character of the blocking point and the precision of the visual method of selecting the blocking point as expressed by the average deviation.

Air cooled samples exhibited higher blocking temperatures in each case and in general 'sharper' blocking character but as a standard test procedure, the method suffers several difficulties of standardization. It may, for example, be hard to control ambient air conditions.

Water cooling of specimens imparted superior gloss to the wax surface in each of the seven waxes. This quality, however, is of little aid in selecting a visual blocking point. In fact, these specimens often exhibited 'spotted' blocking which caused great difficulty in selecting a reproducible blocking point. The TAPPI-ASTM method specifies that the blocking point is to be selected at the point where fifty per cent

of the test strip exhibits the blocking phenomenon. The spotting effect was quite reproducible for each individual wax. More could be done to possible instrumentation of blocking point selection. This would probably proceed along the lines of a photoelectric method of recording light sent at a small angle to the axis of the paper. Possibly it could be zero adjusted to the normal gloss of the paper and a standard unit of loss of gloss selected as indicating the blocking point. The evident question is whether or not the industry is interested in such a method of instrumentation.

The ASTM suggested procedure caused two practical difficulties. They are, first, the specification that a one-inch strip be cut from the center of the machine waxed sheet. As our waxer is best suited to prepare a five-inch waxed sheet this specification causes considerable waste of material. The Marathon research department uses a two-inch waxer for round robin testing. A specification to the effect that one-inch strips be cut from the prepared sheet in such a manner that the sample is secured no closer than one-half inch from the edge would seem more expedient. Secondly, the suggested procedure requires a 24-30 hour aging period at 72°F. 50% R.H. Our experience in retesting specimens aged for a period of one week indicates a general elevation of the blocking temperature. Water cooled sample blocking points were elevated by 1.5°F.; air cooled by 0.5°F. (average). Difficulty arises in routine procedure when testing several waxes in concurrent fashion. Three separate specimens at most can be tested in a five-day week. Also of mention is the fact that evident confusion exists among the committee members as to the number of specimens to be tested for each wax.

Clarification of this matter and possible selection of an aging period such as one week ± 2 days would expedite a standard test procedure. It is noted that the suggested aging period embraces but little increase in time variation as compared with the present aging requirement. It would, however, allow a minimum of five separate tests per five-day week and more if a limited number of strips were tested for each wax.

We have adopted the use of two sheets of glassine (12 by 24-inch) to cover the test strips when placed upon the gradient plate rather than the separate one-inch strips to enclose each test strip. This procedure allows greater ease in keeping wax from the foam rubber strips. Greater precision of placement of the test strips could be accomplished if the method allowed a technique of attaching the test strips to the large bottom sheet of glassine. They could easily be secured with ordinary wire staples, the whole 'set-up' placed upon the blocking plate, the top sheet placed, and the pads and weighted bars lowered into position.

IMPROVED TEMPERATURE CONTROL

The blocking plate has recently been equipped with a deep immersion type Fenwal thermoswitch (maximum sensitivity under optimum conditions is ±0.1°F.) in the hope of securing more precise control of the temperature at the hot end of the plate. It was inserted into a 5/8-inch drilled hole so centered as to give a 3/16-inch thick wall to the cartridge type heater. A sensitive relay activates the current

to the multitap transformer. We should be able to control such factors as overshoot and lag by use of this equipment. We expect very close control of the hot-end temperature by this method.

RECORDING POTENTIOMETER

We have recently received a Brown type 153 Electronik Recorder (single point fast sweep potentiometer) for use with this equipment. It is calibrated to record temperature in degrees Centigrade (finest scale division--0.5°C.). It will be used in connection with a six-point double pole sweep switch to record all pertinent data concerning gradient plate stability throughout the 17-hour test period. It is expected that this equipment will do much to streamline our test procedure and will probably produce more consistent results.

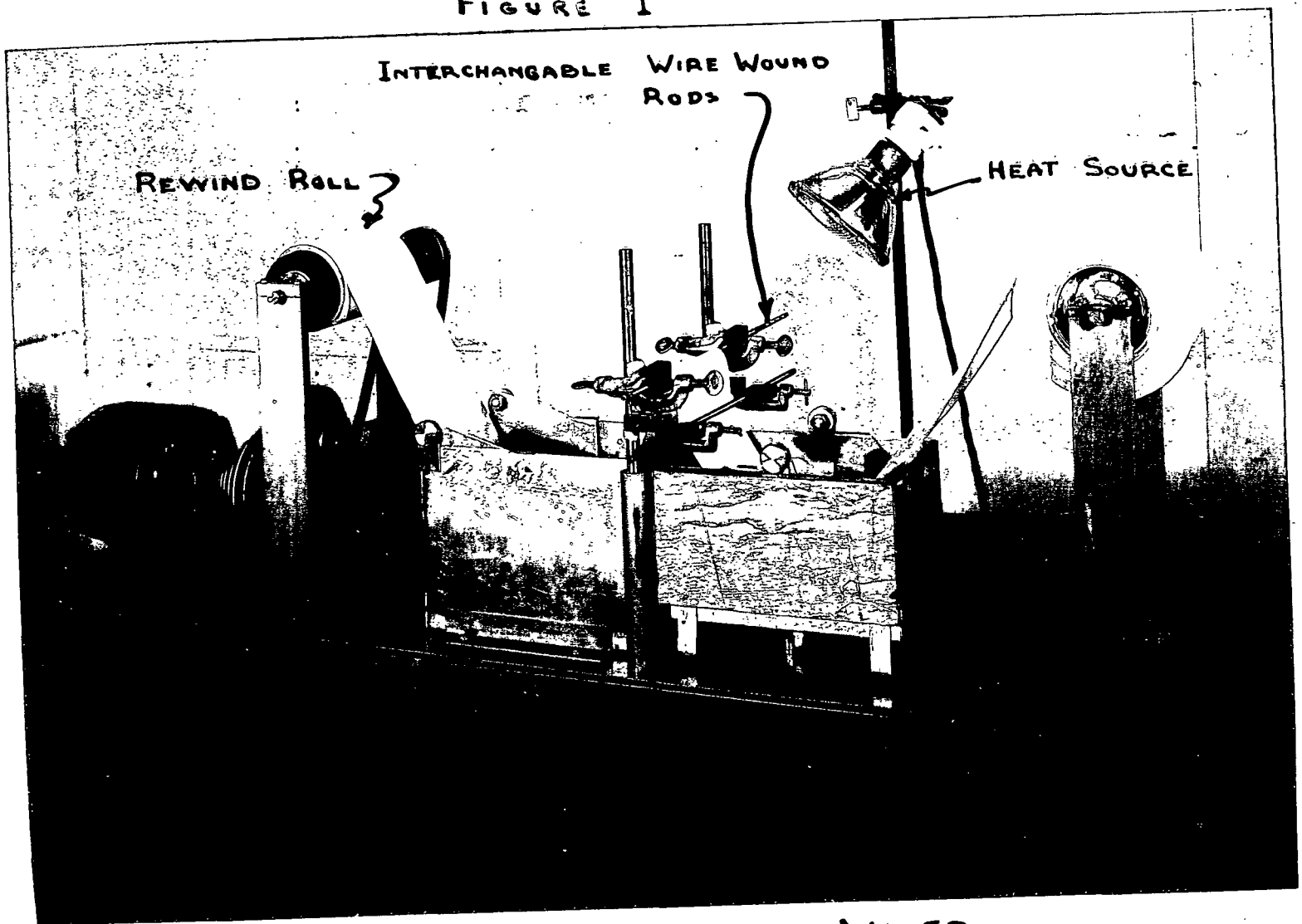
REFERENCES FOUND USEFUL

"TEMPERATURE - Its Measurement and Control in Science and Industry" by the American Institute of Physics. Reinhold Publishing Company, 1941.

"Advanced Electrical Measurements" by W. R. Smythe and W. C. Michels. D. Van Nostrand Co., 1932.

jt/ma

FIGURE I



INSTITUTE LABORATORY WAXER

PROJECT REPORT FORM

PROJECT NO. ✓ 1685
COOPERATOR The Institute of Paper Chemistry
REPORT NO. 7
DATE November 16, 1953
NOTE BOOK 1233
PAGE 118 TO 124
SIGNED James M. Throne

James M. Throne

Copies to: Files
Howells
Vaurio
Krueger
Throne

LABORATORY WAXER SPECIFICATIONS: WEB SPEED, TENSION AND MISCELLANEOUS

Introduction

A preliminary study was made to determine the drive requirements for our laboratory waxer. This was accomplished by selecting conditions of speed and torque that would approach the maximum feasible during a coating operation.

We first determined the maximum feasible operating speed with the use of Mayer rods as metering devices.

Next, we sought the maximum feasible web tensions under conditions similar to those of the waxing operation.

Such engineering considerations as appeared under the strenuous conditions of the test were noted as a guide for general improvement of the waxer itself.

Experimental Drive Unit:

A 1-1/2 h.p. Veridrive giving direct delivered speeds of 300 through 1400 r.p.m. for a near 5:1 ratio was used for the appraisal so that the waxer could be evaluated at maximum conditions of speed and torque.

The present drive system incorporates a 1725 r.p.m. 1/4 h.p. General Electric type KH motor with a 48:1 gear reducer by use of a pair

of 5:4:3:2-inch step pulleys. Speeds delivered to the 3-1/2 inch rewind role were 136:69:38 and 18 feet per minute.

Additional Equipment:

1. Prony Brake--used on unwind roll.
   a. 60-pound capacity spring scale.
   b. Heavy felt web.
   c. Heavy springs as counter balance.
2. 48:1 gear reducer.
3. 1:8 sheave increase.

Speeds Obtainable:

Veridrive

Gear case and pulley system--52 through 271 feet per minute.

Direct drive and pulley adjustment--200 to 1500 feet per minute.

Maximum Feasible Web Speed:

The Mayer rod method of metering the dip roll coated sheet seemed to fail at speeds near 200 feet per minute whether or not the Mayer rods were supported and baffled to prevent mechanical failure (actual runs--213 and 196 ft./min.). The reason would seem to be an "edge effect" inherent in dip roll coating of a narrow sheet. Kerosene was used in the dip tank to eliminate the practical difficulties of using molten wax during high speed coating operations. The viscosity of kerosene at room temperature was found to be 3.5 cp. as compared to 7.0 cp. for molten wax. It would seem a suitable substitute.

Maximum Feasible Web Tension:

Braking force was applied to the unwind stand with a Prony brake at speeds varying from 50 to 900 feet per minute. Twenty-five lb. sulfite

breadwrapper , and 25 and 60 lb. unbleached Kraft webs were threaded into the waxing machine in normal position and used as the motive force for the unwind roll. Torques of 16.5-in. lb. and 18.9-in. lb. at speeds of 288 and 834 feet per minute respectively, caused breakage of the 24 lb. sulfite sheet. It should be noted that aligning difficulties inherent in the present waxing machine caused breakage at a point before the actual tensile strength of the sheet could be attained. When using the heavier webs, slightly greater torques caused a variety of machine failures which made true measurement of the applied torques impossible. Twenty-in. lb. torque would seem to be about the maximum practical limit of required torque delivery for a driving unit with a several-fold safety factor. It is estimated that braking torques of 2 to 5 in. lb. applied to the unwind roll would be sufficient for normal low viscosity coating operations, e.g. samples for blocking point determination.

Calculated Horsepower:

The horsepower required was calculated according to the relation:

$$P_o = \frac{(F_1 - F_2)\, g\, \pi\, rn}{10 - 7}$$

where: $(F_1 - F_2)$ = gram difference in spring gages
g = 980.665
r = radius in cm.
n = revolution per second

P_o = watts dissipated in drum and web.
1 hp = 746 watts

The calculated power requirements were 0.50 cp. at 834 feet per minute and 0.28 h.p. at 288 feet per minute.

It should be noted that these calculated values were obtained from data obtained by braking the unwind stand and thus are liable to serious error as caused by friction throughout the threaded sequence of the machine.

Drive Requirements--Summary:

1. Motor should be 1/4 to 1/2 h.p. (General)
2. Drive should possess adequate torque output to allow operation through full range of required speeds. Calculated torque (maximum) was 20 in. lb.
3. Drive should deliver continuously variable speeds of 0 to 250 ft./min. to the rewind roll.
4. Reversing--not desired.
5. Constant speed and/or constant torque--not required.
6. On-off and variable while running speed reproducibility within 5% of total speed variation.
7. Full speed adjustment while running and from stop position.
8. Method of maintaining constant web speed. This will necessitate speed adjustment during the waxing operation so that web speeds may be maintained at a predetermined value. This may be accomplished by:
   a. Manual adjustment of the speed control throughout the coating operation. An indicating tachometer would indicate the adjustment required. This method would be satisfactory but would require considerable attention of the operator.
   b. A rheostat controlled by the buildup of the rewind roll which would automatically maintain constant web speeds. This feature can be secured as an optional feature of several electronic converter and oil type variable speed drives. This method would be of advantage in requiring a minimum of operator attention.
   c. The use of a "take-up" roll ahead of a slip clutch driven rewind roll would provide the necessary constant web speed. Possible difficulty is foreseen, however, in designing a take-up roll that would give positive web contact (no slippage) without marring the soft waxed surface. Realizing that this problem can be overcome by such machine refinements as powered web carrying rolls and the use of several large take-up drums, this method would seem impractical for our purposes.

Drives for Consideration:

1. Graham Drive
   1/4 h.p. 0-450 r.p.m. 40 to 80 in. lb. torque output Price $306
   1/2 h.p. 0-450 r.p.m. 50 to 100 in. lb. torque output Price $311.

   It Offers:

   Full torque output over entire range.

   Speed variable when stopped and while running.

Zero to maximum speed.
Micrometer remote control offered could probably be adapted to control web speed to constant value. Cost $13.50
Size and construction should allow ease in mounting.

Expect:
Some maintenance
Noisy at very low output speeds.
No institutional discount.
Single phase motor--$3.00 extra.

2. Precision Controlled Thy-Mo-Trol Drive:
Recent information from Mr. Rae sets its price at $224, institutional discount included, for a 1/2-h.p. 20-1 speed range non-reversing drive. In his opinion the tachometer feedback offered as optional feature could control web speed from the buildup of the rewind roll or a mechanical linkage to the standard rheostat control could be furnished by his department.

It Offers:
Constant torque as standard feature.
Precision speed control.
1750-88 r.p.m.
Variable when stopped and while running.
Almost vibrationless delivered power.
Control station includes operating buttons and speed adjustment.
Good speed control during line voltage changes.

Expect:
Gear belt or chain reduction of 7-1 necessary to attain speeds of 12-250 r.p.m. to the rewind roll.
Will not be able to secure speeds from 0 r.p.m.
Maintenance--tube replacement.
Four separate units to install.
About one minute delay for initial starting.

3. General Radio Company Variac
1/3 h.p. (control only) list price $160
1/2 h.p. (control only) list price $230

It Offers:
5-10 r.p.m. to 15% above rated speed range.
Instant starting.
High starting torque.
Continually variable while running and while stopped.
Very simple control mounting.

Expect:
Does not include motor--use any d.c. motor of required h.p.

4. Veri-Speed Motodrive
   1/2 h.p. 26-262 r.p.m. lists at $354
   1/3 h.p. 26-262 r.p.m. lists at $311
   1/4 h.p. 26-262 r.p.m. lists at $288

It Offers:
   1/10 speed range
   Adequate torque output
   No additional speed reduction necessary
   Optional controls offered may possibly be adequate for web speed control.

Expect:
   It cannot be adjusted from stopped position.

5. Adjusto Speed Drive
   Eddy Current clutch design
   10-1 and 17-1 ratio of speeds from 1100 and 3400 r.p.m. respectively

6. Franklin Drive:

   1/2 h.p. without motor lists at $642
   50-1 range from 46 to 2300 r.p.m.

7. Leland Drive:

   Offered in 3-1 and 5-1 speed ratios.
   This drive would not in itself present the necessary speed variation.
   One of the major advantages of this drive is quick reversibility, a feature not required for this application.

8. Vickers Drive

9. Gast Air Motor

The use of a 42K Edgemont V-belt pulley disc clutch in conjunction with various of the drives listed above would allow:

1. Variable speeds while the waxer was stopped from those drives not constructed for this operation, e.g. Veri-Speed Motodrive.

2. Less shock to any of the driving motors when starting the waxer at high preset speeds. The drive unit could idle at high speed while final adjustments were made. It is noted that this would also serve to conserve web and coating materials.

The order of drive listings above is roughly that of preferance by this operator. The Graham drive would seem to have the definite advantage of giving speeds from zero to the maximum. It would also deliver directly to the rewind roll without the use of a reduction unit. Some question exists as to whether it will deliver "vibrationless" power to the rewind roll. The use of a V-belt rather than a chain should aid in this respect. The use of a clutch would probably cut maintenance costs considerably.

Mr. Krueger informs me that we could evaluate a 1-1/2 h.p. Graham drive which he expects delivered within the next three weeks in conjunction with another project.

Mechanical Difficulties and Considerations:

1. Slippage between rubber compression roll and drive shaft of the rewind roll caused serious torque limitations and chattering.

Suggest that larger washers be used on compression roll and the washers be keyed to the drive shaft. Cutting the compression roll to 5-1/2 inches long would prevent "squeeze-out." This would make it necessary to extend the threaded portion of the shaft.

2. Dip tanks should be enlarged to allow:
   a. Processing of a full 5-1/2 inch web.
   b. Greater capacity in the wax bath.
   c. Space for fixtures and baffle area within water cooling dip tank.

Suggest that dip tanks 7 inches wide would be necessary to allow clearance for proposed dip roll support, pin bearings, and a minimum 6-inch dip roll. In conjunction with this the construction of the wax tank to 11 rather than 9 inches would allow the tank to be 1-1/2 inches deeper; thus greater wax capacity. This would mean that the unwind stand width should be increased to 8 inches (inside diameter) to facilitate removal of the wax dip

tank and allow use of the maximum roll width. An 11-inch long water cooling dip tank would allow greater water mass and greater clearance between water inlet and outlet and the moving waxed web. The tank is deep enough and its increased length and width would not seem to cause dependent structural difficulties.

3. Present techniques of controlling temperature of the water cooling dip tank are not satisfactory. The temperature rises through the full allowable range (b.p. determination waxing) in 25 to 30 seconds. This greatly impedes extended coating of the water cooled type.

Suggest the new tank be fitted with standard pipe fittings to allow cooling from an outside source. It is necessary to stay clear of the region occupied by the web. Fittings could be attached to the back of the tank and piped to the center. Three-fourths-inch fittings should be satisfactory. The level should be controlled to 5-1/2 inches.

4. Considerable difficulty occurs because of poor alignment control of the web throughout its threaded sequence. Present dip roll bearings are not satisfactory.

It is suggested that the roll supports be fastened to the table top--free of the dip pans. This will assure positive alignment and eliminate the tendency of the pans to "lift." Bearings of the pin type would seem most satisfactory. Alignment could be controlled by horizontal adjustment of the dip roll support along the top of the table top stanchion. A gear rack would be of aid in vertical adjustment of the dip roll.

5. Present radiant method of heating the Mayer rods is not altogether satisfactory.

Suggest that heating of the rods by cartridge heaters would permit more satisfactory control of the temperature. In addition , the holder would give added support to the metering rods.

We have one holder, acceptable for the lower rod. Another of basically the same design incorporating a smaller incident angle to the rod itself would be satisfactory for the upper Mayer rod.

6. A simple brake will be required for the unwind stand.

Suggest that a Prony type of braking system would allow the constant web tensions required. This could be accomplished by retaining a weighed soft felt material over the unwind roll.

7. Dependent upon the method of mounting the driving unit,, the rewind stand will require stiffening.

Suggest that strapiron braces welded to small steel plates bolted to the table top would be satisfactory.

8. Provision is required for alignment of the unwind and rewind stand.

Suggest that slotted screw-set bearing blocks attached to the upright stands would provide the necessary alignment.

Summary:

The suggestions and considerations incorporated in this report are not complete or all-inclusive but will require modification and improvement as time progresses. This report should, therefore, be considered as preliminary in scope and involvement.

jmt/rm

PROJECT REPORT FORM

PROJECT NO. 1685
COOPERATOR I.P.C.
REPORT NO. 8
DATE February 15, 1954
NOTE BOOK 1233 1278
PAGE 134 - 149 pp 18 - 53
SIGNED James M. Throne

James M. Throne

Copies to: Howells
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Vaurio
Throne

BLOCKING POINT DETERMINATION

INTRODUCTION

The following is a report of various improvements that have been made on the laboratory waxer and the blocking point apparatus prior to participation in the current round robin testing program for determination of the blocking point of paraffin wax. It will include a report of work in testing a new set of possible variables and finally a report of the final data forwarded to the committee chairman.

BLOCKING PLATE IMPROVEMENTS AND CALIBRATION

A Brown Electronik recording potentiometer, one second full scale deflection, 0 to 100°C., calibrated for Copper-Constantan thermocouples, was calibrated with the use of a constant temperature bath against a National Bureau of Standards calibrated thermometer prior to its use as a continuous recording instrument of the temperature gradient along the long axis of the blocking plate. Since the recorded temperatures are expected to remain constant throughout the test period, a double pole 1 revolutions per hour sweep switch was installed to select each of the gradient points in order (8 cm. from the hot end and spaced at 9 cm. intervals to 52 cm. for a total of six points) and record them as a function of time. Chart speed was 2 inches per hour.

While the constant temperature bath method of calibrating the recording potentiometer was satisfactory for more fundamental inspection, it was tedious when consideration was given to such factors as thermocouple aging time, thermometer corrections etc., as a routine method of calibration. We decided to try to calibrate it with the use of the Type-K potentiometer, a commercial standard cell and a galvanometer with a sensitivity of 16.4 by 10^{-8} amp./mm. A copper-constantan cold junction was used to oppose the in-machine cold junction compensating coil so that classic mv. vs. temperature tables could be used directly in the calibration. Care was necessary to protect the galvanometer and Type-K potentiometer. If the Type-K potentiometer was brought up to value slowly the recording potentiometer was actuated smoothly and no galvanometer deflection was noted. Whereas if the Type-K potentiometer was preset to a given value and the circuit switch closed, it was some time before the recording potentiometer settled to a value and considerable swing of the galvanometer was noted. Current draw during the imbalance was thought to be very low so that little danger exists in overloading the potentiometer or galvonometer but since the very sensitive potentiometer is being used to give an accurate current source rather than its more usual use as a null point instrument, every precaution should be taken. As noted by the character of the galvanometer swing the recording potentiometer is seen to be at least as sensitive as the galvanometer. The values obtained by this method of calibration follow.

TABLE I

Mv.	Observed Temperature -C°
0.000	0.5[1]
0.077	2.0
0.198	5.0
0.390	10.0
0.992	25.0
1.19	30.0
1.61	40.0
2.03	50.0
2.47	60.0
2.91	70.0
3.36	80.0
3.72	88.1[2]
3.81	89.9[3]
4.00	94.0

The values indicated by superscripts 1, 2 and 3 above deviate from the classic relation. The other values conform with the expected values.

[1]This value is 0.5°C. high.
It was probably caused by mechanical failure of the instrument to reach the 0.0°C. position on the scale.

[2]This value is 0.1°C. high.

[3]This value is 0.1°C. low
The two above values could deviate because of a small error in the scale but this is not likely because the deviations are of opposite sign. A more likely explanation is that the mv. values are not given in enough significant figures to determine temperatures accurate to 0.1°C. The mv. vs. temperature values were secured from "Service Manual 15019 M" by Minneapolis Honeywell Regulator Company.

The above method of calibration varies from that suggested by the instrument manual (Section 1550, Part II) only in the use of the external cold junction. The manual suggests subtracting the compensated mv. (calculated from data in the table) from the classic value to obtain the setting for the Type-K potentiometer. Our method allows a check of in-machine cold-junction compensation and is less time consuming in that no calculations are required.

After an interim of conversion to a 0 to 1 mv. range in connection with another application, difficulty was encountered in calibrating by the above method. The recording potentiometer standard cell was tested with the Type-K potentiometer and its voltage found to be 1.01913 volts. This is within the factory specifications (1.0190 $\pm$ 0.0005 v.) as given by the manual. The difficulty was finally found to be in the setting of the sensitivity adjustment. If the sensitivity was damped too heavily the instrument is slow in behavior and in fact indicated low by 2 to 5°C. throughout its range. Very careful adjustment was necessary to a point just below that at which a rapid continuous fluctuation of the indicator occurs.

BLOCKING PLATE CALIBRATION FOR ROUND ROBIN TESTING

Procedure, Paragraph 2 of a "Suggested TAPPI Method for Blocking Point of Paraffin Wax" by H. F. Hitchcox dated January 8, 1954 suggests a method for calibration of a blocking point apparatus and recorder. A "test" couple is immersed in a beaker of water together with a calibrated thermometer. The water is carefully heated to 110°F. The mv. of the test couple is measured

with a Type-K potentiometer and the deviation from the classic value is carefully noted as a correction factor. The test couple is then placed directly adjacent to the couples in normal position along the full gradient of the plate, covered with the sponge pads and weighted bars, aged, and the test couple mv. equivalent (Type-K potentiometer) compared with the recorded couple. If this difference is more than one degree fahrenheit, less the correction factor of the test couple, certain procedures are prescribed.

The suggested method was used in the final calibration of the blocking plate with the exception that the test couple was connected to the recording potentiometer at 110°F. and a scale change was made to compensate for the deviation of the test couple value from the classic one. The recorder was then used to measure the temperatures of both the test and normally placed couple. The test couple was so positioned that it crossed essentially the full width of the blocking plate and was secured as near as possible and in direct line with the pressure-loaded surface-resting thermocouples. It was then secured in place with pressure sensitive tape and weighted with the six pads and bars used in the routine procedure. Listed below are the values obtained after a minimum aging period of one hour. The values indicated by superscripts 1, 2 and 3 were checked by removing the test couple, replacing, aging, and again reading the temperature.

TABLE II

Distance Hot End, (cm.)	Test Couple, (C°)	Correction, (C°)	Recorded Couple, (C°)
8	70.2[1]	-1.1	71.3
17	60.2[1]	-1.1	61.3
26	50.4[1]	-0.9	51.3
35	41.3[1]	-0.4	41.7
44	32.0[1]	-0.2	32.2
53	22.8	+0.6	22.2

TRANSVERSE CORRECTIONS

The pressure bar was removed from its normal position and placed so that the temperature was measured at the center and 9 cm. toward either edge along a line 13, 26, 39, and 52 cm. from the hot end of the blocking plate. Below are the values obtained after aging each couple a minimum of one hour:

TABLE III

Distance Parallel To Hot End, (cm.)	Center, (C°)	9 cm. Toward Water--In	9 cm. Toward Water--Out
13	64.3	65.2	64.8
26	50.2	50.6	50.3
39	36.4	36.6	36.6
52	23.2	22.8	23.5

TOTAL CORRECTIONS

The values of Tables II and III were plotted in such a way that longitudinal and transverse gradient corrections could be made from a single graph of three independent variables, on the basis of the position of the blocking point expressed in cm. distance from the hot end and strip position on the gradient plate.

D. C. RELAY--HEATING PLATE

A single-pole double-throw (Cutler Hammer 10.000 ohm coil, capacity 15 amp.) relay system employing a d. c. power supply from a 150 volt d. c. Selenium rectifier was installed to provide the cartridge heater of the blocking plate with either of two voltages selected from the multitap transformer. The voltages selected are dependent upon the span of the gradient required with consideration to minimizing the voltage difference. This method allows superior control of lag and overshoot as compared with the limited selection of voltages allowed by a single throw relay--zero and selected. Fifty-five and 95 volts, respectively, to the high and low sides of the relay would seem the optimum condition for the gradient established for the round robin testing program. This condition gives a 2.0 degree fahrenheit gradient per centimeter, with 160°F. maximum temperature. Relay action as activated by the Fenwell Thermoswitch was 60% on, 40% off in one complete cycle for two-minute intervals. The temperature variation at the

8 cm. position as measured by the recording potentiometer was 0.3°C. A 2.3°F. per centimeter gradient, 176°F. maximum, established for the blocking point of latex coatings, required 75 and 100 volts respectively, for comparable relay action.

A second d. c. relay, used as a single throw switch, in parallel with the first activates the operational pen of the recording potentiometer.

A standard laboratory relay and general household thermostat have been installed as a safety device. The thermostat, mounted under spring pressure on the cold end of the plate will disconnect the power source to the multitap transformer if the temperature should rise appreciably at the lower end of the plate during the test period, as during failure of the well water used to cool the low temperature portion of the plate.

LABORATORY WAXER

A 1-1/2 h. p. 0-650 r.p.m. Graham drive has been installed for temporary use as a controlled speed drive unit on the laboratory waxer. It is equipped with a 100-turn, 1000-division micrometer remote control. This gives very good speed control throughout its full range. The r.p.m. delivered to the rewind roll (an approximate 1 to 2 V-belt reduction) was obtained with a one second sweep electric timer and a commercial Productimeter (one division per revolution) throughout the full range. Distinct micrometer

settings gave reproducible speed in r.p.m. within 1 r.p.m. (the finest division of the Productimeter) through the entire range. This data was graphed. The speed required (r.p.m.) to maintain constant web speed (ft./min.) through a range of rewind role build-up diameters was calculated on the basis of standard formulas for the circumference and web delivery rate of the rewind roll. The necessary micro settings were then interpolated from the above mentioned graph. Actual web speeds were specified in the test method on the basis of the interval of time required for the web to travel from the last doctor rod to the water bath surface. The adjustments mentioned were made at intervals of 1/2-inch roll diameter increase during a continuous waxing procedure. The maximum error calculated at the highest web speed (225 ft./min.) and minimum roll size (3-1/2 inch diameter) was 14%. This error diminished to about 8% for normal waxing procedure and could be further diminished by adjusting the speed at smaller ingredients of roll diameter.

One change was required in the standard adapted as waxing procedure. The sheet coated at 225 ft./min. exhibited a "cockled" appearance. In an attempt to prevent this effect an air knife was used to doctor the cooled sheet and thus prevent the accumulation of water in the rewind roll. This was not successful largely because of the restricted space in which to doctor and unsupported web. A web which was partially doctored in this manner exhibited the same appearance. It is possible that the difficulty lies not in the presence of water in the rewind roll but rather occurs because water

is trapped in the sheet since the wax has not sufficiently set to prevent water absorption by the sheet during the cooling operation. It is noted that the distance from last doctor rod to water bath surface is fixed so that the wax must reach the water bath surface at higher temperatures proportional to the increase in web speed for the various coating conditions. In either case, and it may be that the phenomenon can be explained only by a combination of the above observations, No simple routine method was found to escape the difficulty while maintaining web speed. For the last two of the four waxes tested, a machine change was made which decreased the distance from the last doctor rod to the water bath surface from 9 to 5-1/2 inches, thus cutting the web speed from 225 to 137 ft./min. and eliminating the difficulty.

The test method asked for an observation of the gloss secured by the several test conditions. The results are included in the attached table.

The waxer was fitted with a low head, large flowing volume, baffled, water-cooling dip tank. The use of tap water and a steam mixing faucet allowed control of the water temperature to 50 $\pm$ 3°F. The bath construction assured that the temperature was held within these limits for web speeds as high as 225 ft./min. This however was possible because the temperature of full flowing tap water during this season is approximately 41°F. During the summer months the minimum temperature obtainable without

artificial cooling would be about 60°F. Well water would probably be a satisfactory, though somewhat limited, means of cooling throughout the full year; 50°F. would be the minimum temperature.

Mayer rod heaters were installed to give support as well as temperature contol to the metering devices. Each rod is heated with two 150-watt 3/8-inch cartridge heaters. They have been calibrated for heat delivered to the wire surface as measured with a 0 to 500°F. Alnor hand pyrometer, with various voltages as delivered by a 7.2 amp. Variac. For round robin testing, a solid steel drill rod was used to meter the reverse (opposite test surface) surface rather than Mayer rods, as we were striving for a noncoated surface. The heated supports were so designed that the percentage contact of the web with the rod could be widely varied. In this way several adjustments are possible in the metering process. The sheet may be scarcely grazed by the rod or contact it for as many as 290 degrees. An alternate metering rod made of 3/8-inch stainless steel pipe into which the cartridge heaters were inserted can be used where applications require more than 290-degrees wrap around. The adjustments mentioned together with a break that would allow careful control of web tension should give us a well controlled metering process.

Dip rolls for both water and wax tanks equipped with hardened pin bearings and so mounted as to permit edge control have been installed on the waxer. The rolls are designed so that they may be lowered to the tank

bottom or elevated to or above the fluid surface by simply adjusting a gear and rack of teeth by means of a crank.

The machine has been designed to accommodate a maximum 6-inch wide web in roll sizes to 12 inches in diameter.

SUMMARY AND FINAL DATA

No great difficulty was encountered in attaining the conditions prescribed for the current round robin testing program with those exceptions mentioned in the pertinent sections above. Testing was started January 20 and finished January 31, 1954.

In connection with the interpretation and selection of the actual blocking point, it was difficult to select a definite blocking point in the case of Cities Service wax CS-B-3. An area characterized by haze or spotting was noted considerably below the actual picking and blocking temperature. Its existence caused difficulty in selecting the final pick and blocking points. It was therefore reported to the committee chairman in terms of the temperature at which it occurred.

It is difficult to draw valid generalizations as to the effect of the variables studied in our allotted portion of the round robin test program from so limited a sample of data. It would not appear, from the

limited sampling accomplished in this laboratory, that coating weight or waxing speed are significant variables of blocking point determinations. More careful analysis of data secured from the four parties participating in this phase of the test program will most certainly yield more certain knowledge of the factors concerned.

The results reported to the committee chairman follow.

jt/mab

4-6 lb./ream wax on one side
other side scraped

Wax	Time from last doctor rod to water (sec.)	Haze and spotting °F	Pick/ Block °F.	Character of blocking point	Gloss	Coating Speed (ft./min)	Time of immersion in water bath (sec.)
S.O.D. W-4317	0.8		116/116	sharp	fair	56	1.1
	0.5		113/115	fair	good	90	0.7
	0.2		116/116	poor	good*	225	0.3
Sun oil X-9883	0.8		106/108	fair	fair	56	1.1
	0.5		107/109	fair	good	90	0.7
	0.2		107/110	poor	good*	225	0.3
Cities Service CS-B3	0.8	(116)	128/138	poor	good	56	1.1
	0.5	(117)	127/137	poor	good	90	0.7
	0.2	(117)	130/138	poor	good	137	0.5
Sinclair P-258	0.8		97/103	fair	good	56	1.1
	0.5		95/101	fair	good	90	0.7
	0.2		97/103	fair	good	137	0.5

*Specimen exhibited cockled effect.

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PAGE 150 - 160 TO 7 - 18
SIGNED James M. Throne

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PENETRATION OF PETROLEUM WAX--ROUND ROBIN TESTING

INTRODUCTION

The following is a report of the evaluation of wax by the Needle Penetration Test under the current round robin testing program. The tests were carried out according to the "Suggested TAPPI Method for the Consistency of Petroleum Wax-Needle Penetration Method" (a copy of which accompanies correspondence from W. J. Yates, Section II secretary, dated November 14, 1952) and a letter to committee members by H. Schindler dated October 21, 1953, except as noted in the pertinent paragraphs below. The major variables under consideration at this time are (a) the effect of several intervals of conditioning time upon penetration and (b) the evaluation of a larger mold, two inches in diameter, as a possible means of eliminating voids, to try to get more reproducible penetrations in the high (above 50 tenths millimeter) penetration region. In addition, an examination was made of the surface and internal wax characteristics.

APPARATUS AND EQUIPMENT

Constant Temperature: The constant temperature equipment consists of a 300 by 300 mm. glass jar equipped with a laboratory stirrer, three 250-watt knife blade immersion heaters, two of which were used intermittently, being

switched on and off by the thermoregulator, and one as an auxiliary heater, which was "on" continuously. Two Variacs were used to proportion voltage to the intermittent and auxiliary heaters for control of "lag" and "overshoot."

A small bilge pump (IPC DB-523) furnished water to a 5-1/2 inch diameter, 2-1/2 inch high, Lucite dish upon the penetrometer table. A gravity overflow returned the water to the main bath.

It is noted that the test method states that the thermostat (Lucite dish) "shall be connected with the main water bath so that its temperature is equal to that of the water bath." This is true of our method except as greater refinement of temperature control should prove necessary. When testing at elevated temperatures a gradient exists between the temperature of the main bath and the thermostat because of heat loss to the air, and from the thermostat walls to the penetrometer table. This heat loss is dependent on the rate of circulation and the elevation of the test temperature above room temperature. This difference was found to be approximately 0.6°C. when testing at 115°F. (46°C.) with normal circulation rate. This fact was found useful in adjusting the temperature at the thermostat. Adjustment of the circulation rate by means of a clamp on the hose connecting the main bath to the thermostat gave a fine adjustment of the temperature not possible by the adjustment of the regulator itself.

Because of our difficulty in obtaining the above mentioned test conditions, we have conditioned our samples the required time in the thermostat dish rather than the main bath. We believe that the temperature variation

during the conditioning time is thus considerably less than that encountered by transferring the sample from the main bath to thermostat just prior to testing, as the method specifies. Test temperatures were thus measured at the thermostat rather than at the main bath and a perforated shelf within the main bath to support the mold specimens was not used.

Fundamental difficulty still exists in that the temperature within the dish may vary by more than the allowed variation when several test specimens are immersed in it, because of the small mass of circulating fluid it contains as compared to the mass of fluid contained in the main bath.

It is difficult to state how great an effect these considerations have on temperature control and ultimately on the penetration value but further refinements would seem necessary to obtain the required conditions if in fact the "Suggested Method" is not changed by further use and examination.

Thermometer and Corrections: Because of the shallow immersion depth allowed by the thermostat dish for temperature measurements, emergent stem corrections were made by classic formulae to give corrected temperatures. A -10 to 104°C. Wilkens Anderson thermometer with 1.0°C. finest division was used.

Testing at 100°F. (37.78°C.)

Ice point and characteristic thermometer corrections: -0.08°C.
Emergent stem correction (Room Temp. 25°C. Immersed to -7.0°C.) +0.09°C.
Total correction: +0.01°C.
Nominal temperature: 37.79°C.

Testing at 115°F. (46.11°C.)

Characteristic and ice point thermometer corrections: -0.06°C.
Emergent stem correction (as above): +0.18°C.
Total correction: +0.12
115°F. = 46.11°C.
Nominal temperature: 46.23°C.

Sample Container: The standard sample container consists of a brass cylinder open at both ends having a 1-1/16 inch inside diameter, 1-1/4 inch height and 1/8 inch wall thickness. To prevent slippage of a very hard wax a few coarse screw threads were cut into the center part of the inside wall of the cylinder. This container will be referred to as a 1-inch mold for the purposes of this report.

We had not received a copy of the proposed 2-inch sample mold from the Atlantic Refining Company in time for use by our engineering department.

We submitted our own design to engineering and the molds made according to our design were finished before hearing from the Atlantic Refining Company. Slight modification was made of the design to best use the materials on hand. The Atlantic Refining Company design has since been received and appears to be quite similar to our design except for the use of pins to retain the wax.

The molds used are two inches in inside diameter and 1-1/4 inches high with a 5/32 inch wall. The material is brass. A 3/16-inch deep shoulder, 3/32-inch thick was left in the center of the inside wall to retain the wax casting. The bottom edge of the mold was lapped to give a "leak-proof" contact with the brass plate.

Penetrometer: A "Precision" Penetrometer, 1/10 mm. divisions (IPC DC-87) was used to measure the depth of penetration.

The standard needle for this test is the ASTM D-5 penetration needle.

The "Esso" needle was also used by this laboratory for the current test. A drawing and specifications are included in a letter from H. Schindler to Mr. Vaurio dated March 21, 1952.

PROCEDURE

Sample Preparation: The pouring temperature is specified as 30°F. above the melting point. The ASTM methods used and modifications thereof for determination of the melting point follow:

Wax - Shell Oil P-417
Type - Microcrystalline
Method - ASTM D-127-49
Modifications:
1. 5-7/8 inch long test tube
2. Lanco Thermometer - 15 to 110°C. in 1.0°C. Laboratory Calibration No. 5

Results: 60.3°C. (140.5°F.)
Molds poured at 77.0°C. (170.5°F.)

Wax - Moore and Munger - 100
Type - Paraffin Wax
Method - D 87-42
Modifications:
1. 5-7/8 inch long test tube
2. CACO Thermometer 0 to 225°F. in 2.0°F. Laboratory calibration No. 6
3. Temperature recorded at 30 second intervals

Results: 134.2°F. (56.8°C.)
Molds poured at 164.2°F. (73.5°C.)

The wax was heated in a large test tube (8 inches long, 1-1/2 inch diameter) by immersing it into a beaker of warm water and heating with a bunsen burner. A calibrated laboratory thermometer was used as a stirring

rod. A clean single-weight glass plate was wet with a 50% glycerine solution and the excess removed with a cotton swab. The plate was placed on a paper towel resting on the bench top. The wax was poured immediately upon reaching the correct temperature.

Conditioning: The poured sample was left to cool one hour at room temperature. It should be noted that the method prescribes placing the poured molds "in an air bath at 77°F. ± 2.0°F. for one hour." A more likely estimate of our working temperature would be 77°F. ± 5°F. Thus some error may be incorporated during the critical crystallization period of air bath conditioning. However, the method does not prescribe conditioning of the brass molds prior to pouring, so that some variation in conditioning is possible under the present wording on the "Suggested Method."

The method states that the water bath temperature is to be constant to ± 0.2°F. (± 0.1°C.). Testing started with a 3 wire De Khotinsky relay and bi-metallic thermoregulator. Temperature control by this means was found to be marginally adequate. The sensitivity of temperature control was limited by "chatter" at the contacts of the regulator when set to give the control needed. Penetrations for the Moore and Munger 100 wax in the two-inch molds were repeated because temperature control was inadequate.

For testing at 115°F. a "Precision" scientific relay and mercury to platinum thermoregulator were used to control temperature. It gave control at the thermostat to ± 0.03°C.

Penetrations were run on all specimens with the "Esso" needle immediately after testing with the standard D-5 needle. In addition,

consecutive penetrations, of five second duration, were made with both needles to augment our understanding of void formation and other variables (data not included in this report).

See also Apparatus, constant temperature, paragraphs 3 and 4.

EXPERIMENTAL RESULTS

Attached are copies of data prepared for distribution to committee members.

EXAMINATION OF MOLDED SPECIMENS

Surface Character

Shell P-417: Both the one and two inch molds gave slightly rough surfaces when removed from the glass plate over all of the surface except for a circular area (approximately 5/8 inches in diameter) near the mold center, which was smooth. When placed in the water bath, bubbles formed over the rough surface causing difficulty in placing the needle. The circular area, however, presented a smooth reflective surface, almost bubble free, so that the needle was easily placed. The bubbles were more troublesome at the 115°F. test temperature.

Suggested explanation - May be either:

1. The molten wax, as poured from the test tube into the mold, may wash the glycerine from the glass plate permitting a circular smooth area.

2. The molten wax, as poured from the test tube into the mold, may heat a portion of the glass plate to a higher temperature than the surrounding area, thus allowing a longer time for the surface to cool. This might

conceivably produce a smoother, more reflective surface.

Moore and Munger - 100: This wax presented a smooth reflective surface over the entire molded surface irrespective of mold size or conditioning temperature.

The penetration surface became slightly concave at 115°F. immersion but presented no difficulties in needle placement.

Internal Structure:

Shell P-417:

1-inch mold (1 hour air, 5 hours in water at 115°F.)
Deep cavity - Average 9/16 inch deep.
11/16 inch (1.74 cm.) from cavity to penetration surface at mold center.

2-inch mold (as above)
Cavity - Average 7/16 inch deep
13/16 inch (2.06 cm.) from cavity to penetration surface at mold center.

Moore and Munger - 100:

1-inch mold (1 hour air, 5 hours in water at 115°F.)
Cavity at bottom surface - Average 5/16 inch deep
Cavity leads to 3/8 inch diameter, 11/16 inch long void
3/8 inch (0.95 cm.) from void to penetration surface at mold center.

2-inch mold (1 hour air, 5 hours in water at 100°F.)
Cavity - Average 9/16 inch deep
11/16 inch (1.74 cm.) from cavity to penetration surface at mold center.

SUMMARY AND CONCLUSIONS

The two-inch diameter molds provided test specimens free of hidden voids, whereas the one-inch diameter molds gave specimens with hidden cavities or voids with the Moore and Munger wax.

The Esso needle, according to our results, was superior to the ASTM-D5 needle when comparing higher penetration values.

The results are too variable and too few in number to use as a basis for drawing any valid conclusions as to the effect of increasing the conditioning time. The composite group results will be more meaningful, it is hoped.

Our 2-inch mold design seems as satisfactory as the design proposed by Atlantic Refining Company.

At elevated temperatures it is not possible to maintain the same temperature in the test dish and the main water bath, as required by the present suggested method, because of the heat losses from the hose and the plastic dish walls and from evaporation of water in the "thermostat" or test bath.

jt/mb

TAPPI-ASTM TECHNICAL COMMITTEE ON PETROLEUM WAX
ROUND ROBIN TESTS ON NEEDLE PENETRATION
EFFECT OF MOLD DESIGN

Standard ASTM-D5 Needle Penetration

		Shell P-417 Wax										Moore and Munger (100 Melting) Wax											
	Condition	100°F.				115°F.						100°F.						115°F.					
	Hours	1	2	3	Date	1	2	3	4	5	Date	1	2	3	4	5	Date	1	2	3	4	5	Date
One-Inch Mold	1	40	40	40	12-29	185	196	179	177	180	1-8	60	58	55	53	55	12-31	142	152	173	182	188	1-6
	3	39	39	40		184	176	173	183	180		57	58	58	—	—		165	157	140	169	181	
	5	39	39	40		186	178	175	178	180		62	63	59	61	61		312	150	185	148	184	
Two-Inch Mold	1	42	42	43	12-29	179	149	155	152	155	1-11	58	56	56	56	56	1-13	139	161	169	139	142	1-7
	3	41	41	40		174	165	167	166	163		66	57	60	61	63		159	199	158	147	138	
	5	40	41	41		175	171	173	169	180		57	58	61	63	60		179	168	178	159	172	

Note: All tests were run on the date poured, ~~except the 100°F. two-inch mold with the P-417 wax which was tested on the next day~~.

ESSO NEEDLE (Continuous Cone)

Mold	Condition Time, Hours	Shell P-417 Wax 100°F. 1	2	3	Shell P-417 Wax 115°F. 1	2	3	4	5	Moore and Munger (100 Melting) Wax 100°F. 1	2	3	Moore and Munger (100 Melting) Wax 115°F. 1	2	3
One-Inch Mold	1	—	--	--	139	131	130	129	128	56	53	54	—	--	--
	3	—	--	--	130	129	127	128	127	56	57	58	100	104	102
	5	39	40	40	139	136	134	135	130	59	57	57	98	102	104
Two-Inch Mold	11	42	42	42	148	125	124	127	122	68	62	60	109	105	103
	3	39	40	40	132	125	123	122	127	57	57	56	109	105	107
	5	41	42	41	131	126	125	125	127	64	61	64	--	--	--

Note: All tests were run on the date poured, ~~except the 100°F. two-inch mold with the P-417 wax which was tested the next day.~~ The tests with the Esso needle were made immediately after those with the ASTM-D5 needle.