G. Project 1685

PROJECT REPORT FORM

PROJECT NO. 1102-10
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REPORT NO. 8
DATE October 11, 1951
NOTE BOOK 885
PAGE 113 to
SIGNED Frans Vaurio
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Penetration Tests

TAPPI - ASTM ROUND ROBIN TESTING
Wax Penetration Test

Wax	ASTM-D5-25	MICRO PENETRATION
A	25.3	20.7
B	15.2	11.0
C	8.9	9.0
D	26.0	24.0
E	37.7	33.2

Note: The shrinkage of A was very high. There was no reflection visible. Left a 1/4-inch diameter hole in the center of the micropenetration sample. "A", "B" and "E" were poured at 180°F. The melting point of "C" is so high that the wax was chilled too soon leaving a poor surface. "C" was poured at 215°F. The wax "D" was tested without using the transfer dish as it was not possible to get a good zero setting under water. "D" was poured at 195°F.

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MEASUREMENT OF TACK IN LAMINATING WAXES

INTRODUCTION

A problem which frequently arises in the use of microcrystalline type waxes for laminating is the evaluation of their ability to remain adhesive at elevated temperatures or temperatures just under their application temperatures. This problem arises, for example, in the use of glassine or greaseproof laminations to boxboard for powdered detergent or soap containers. In this application, the containers are filled with the powders at temperatures up to 150°F. and in such cases the laminate is subject to separation if the thermal characteristics of the adhesive are not properly chosen. The problem of laminate performance at elevated temperature also exists in normal use and storage conditions of many other laminated products--such conditions as, for example, in poorly ventilated freight cars in abnormally hot weather or in the holds of ships in tropic climates. These latter conditions are frequently met in packaging applications for the armed forces. In the selection of a laminating material for some application it is therefore important to know or predict its behavior at elevated temperatures. It is the purpose of this project to develop means of predicting such performance in regard to microcrystalline waxes by means of a relatively simple test on the bulk material.

Microcrystalline waxes are used as laminating materials because of their excellent moisture resistance, flexibility, and low cost. These materials, unlike paraffin, do not have sharp melting points, and at temperatures below

that at which they are true liquids, they pass through a condition of unhomogeniety characterized by a soft gel-like consistency. This slush-like state may exist over a broad or narrow range of temperature depending on the particular wax and may be further characterized by a large or small degree of a loosely defined property called tack or stickiness. Tack is a desirable property from the standpoint of laminating in that it allows a withdrawal of pressure during the setting or freezing period of the adhesive. Such a withdrawal of pressure cannot in fact be avoided in a continuous laminating where pressure is applied at the nip between rolls for only a brief instant. With materials which have a sharp melting point such as paraffin wax this brief instant should coincide with the transition of the wax from liquid to solid as nearly as possible; otherwise the plys being laminated will separate after passing through the nip. With microcrystalline laminating waxes the timing is less critical because the tack of these materials holds the plys together for the brief cooling or setting period following the pressure nip.

On the other hand, the performance of a well adhered laminate at elevated temperatures is adversely affected by a change from a solid material to a tacky slush because the tendency for ply separation in the latter state is greater than it is in the former state. Therefore, the difference between the application temperature and the optimum performance temperature is increased which is undesirable.

It is therefore of importance both from the standpoint of the laminator and the consumer to know how broad a temperature range the intermediate state between a liquid and a solid exists and also what degree of tack this region possesses.

Many workers have considered tack to be primarily a function of viscosity[1] or in fact to be the same as viscosity. Others contend that tack is a combination of several properties such as viscosity, yield point, elasticity, relaxation time, wettability, cohesion, and adhesion.[2] Where a Newtonian liquid is involved and the meaning of tack is restricted to the restraining force involved in the separation of two surfaces wetted by the material being tested, then it is true that tack and viscosity are the same property. However, the measurement of tack usually involves more than measuring only viscosity, or plastic flow properties if the material is not Newtonian. On less fundamental terms, tack can be considered to be the apparent adhesion developed when the nonsolid material being investigated is brought into contact with a solid material or when two surfaces of the material being investigated are brought into contact. The term "apparent" adhesion is used here to differentiate between true adhesion, the meaning of which is limited to the attractive forces between two surfaces. Apparent adhesion may involve both true adhesion, rheological behavior, wettability and other factors.

The above concept of tack would not usually appear to have a counterpart in the performance of a laminating wax in that here a change of temperature is usually involved after initial contact is made. In other words the material upon which the tack is being measured is brought into contact with a solid material at one temperature during the laminating process and the apparent adhesion is of interest at other temperatures. However, it is possible that in actual performance, the contact between the laminating

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[1] J. J. Bikerman, J. Colloid Science, 2 (1947) 163

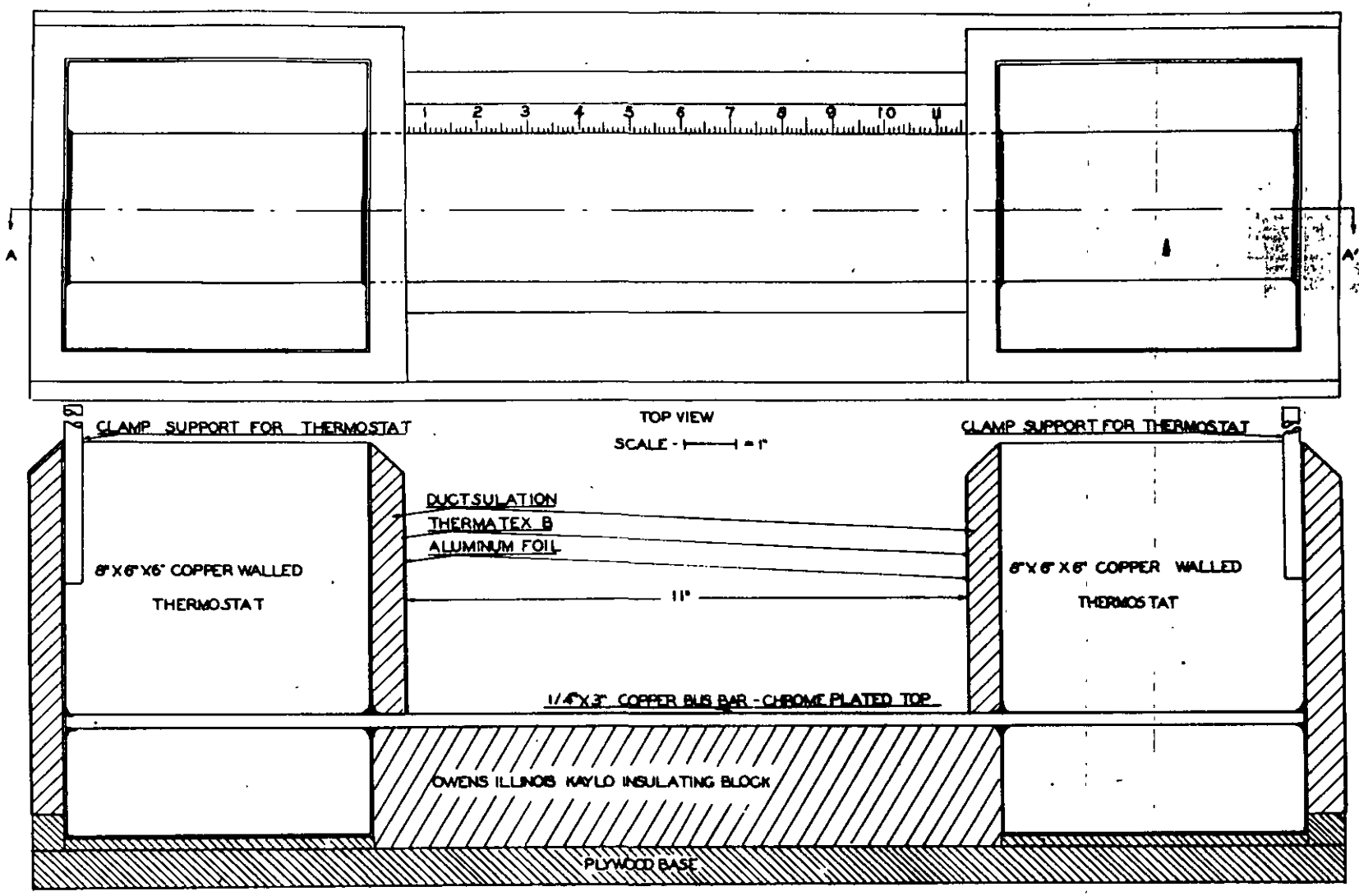
[2] British Rheoligists Club. Patra J., 6 No. 1:12-16 (1942).

material and a solid or between two surfaces of the laminating material would be made in the region in which the apparent adhesion would normally be of interest (i.e., the region between the liquid and solid state). Three possibilities therefore exist for tests on laminating waxes to determine the degree of tack in the region between liquid and solid mentioned above. One is to make the initial contact with solid surfaces with the temperature of the laminating material in the liquid region (to insure wetting of the surface) and to test the apparent adhesion in the region below which the laminating materials are liquid. The others are to make the initial contact between the laminating material and a solid surface or between two surfaces of the laminating material at the same temperature that the resultant apparent adhesion is tested.

The following paragraphs describe a test intended to determine the degree and broadness of the tacky region of a microcrystalline wax by the method in which contact with a solid surface (in this case, a dielectric) is made and the apparent adhesion tested at the same temperature.

EXPERIMENTAL PROCEDURE

In order to provide a surface of a microcrystalline laminating wax at a multiplicity of temperatures so that the regions between liquid and solid states could be accurately determined as well as determining the tack within that region, a gradient bar was constructed as shown in Figure 1 below.



SECTION THRU AA'

TEMPERATURE GRADIENT BAR

In Figure 2 the calibration curve was obtained with one thermostat at 70°F. and the other at 188°F.* The measurements of surface temperature on the gradient bar were made with a thermocouple filed down to provide good metal to metal contact. Pressure was applied to the couple by means of a pencil eraser to insure good contact. The position along the gradient was established by means of a scale marked off in 0.1" divisions as shown in Figure 1.

The following microcrystalline laminating waxes were available for preliminary testing. These represent typical commercially used materials.

1. 2300 wax, Socony Vacuum Oil Company, Inc.
2. Magnawax Brown, Socony Vacuum Oil Company, Inc.
3. Jancoat, Bareco Oil Company.
4. Multiwax W-835, Petroleum Specialties, Inc.

Accurate spherical nylon balls 1/4" and 1/2" in diameter were obtained from the Ace Plastics Fabricators to use as dielectric solid surfaces.

The test used to determine the tack characteristic of the wax surface over a temperature gradient was to let a nylon ball roll onto the wax coated gradient bar in a direction of constant temperature (transversly across the gradient bar) at various points along the gradient bar representing various temperatures. Kinetic energy was imparted to the ball by releasing it from the top of a small ramp 7/8" long at a 52° angle. In this test it was assumed that the tack of the wax is proportional to the

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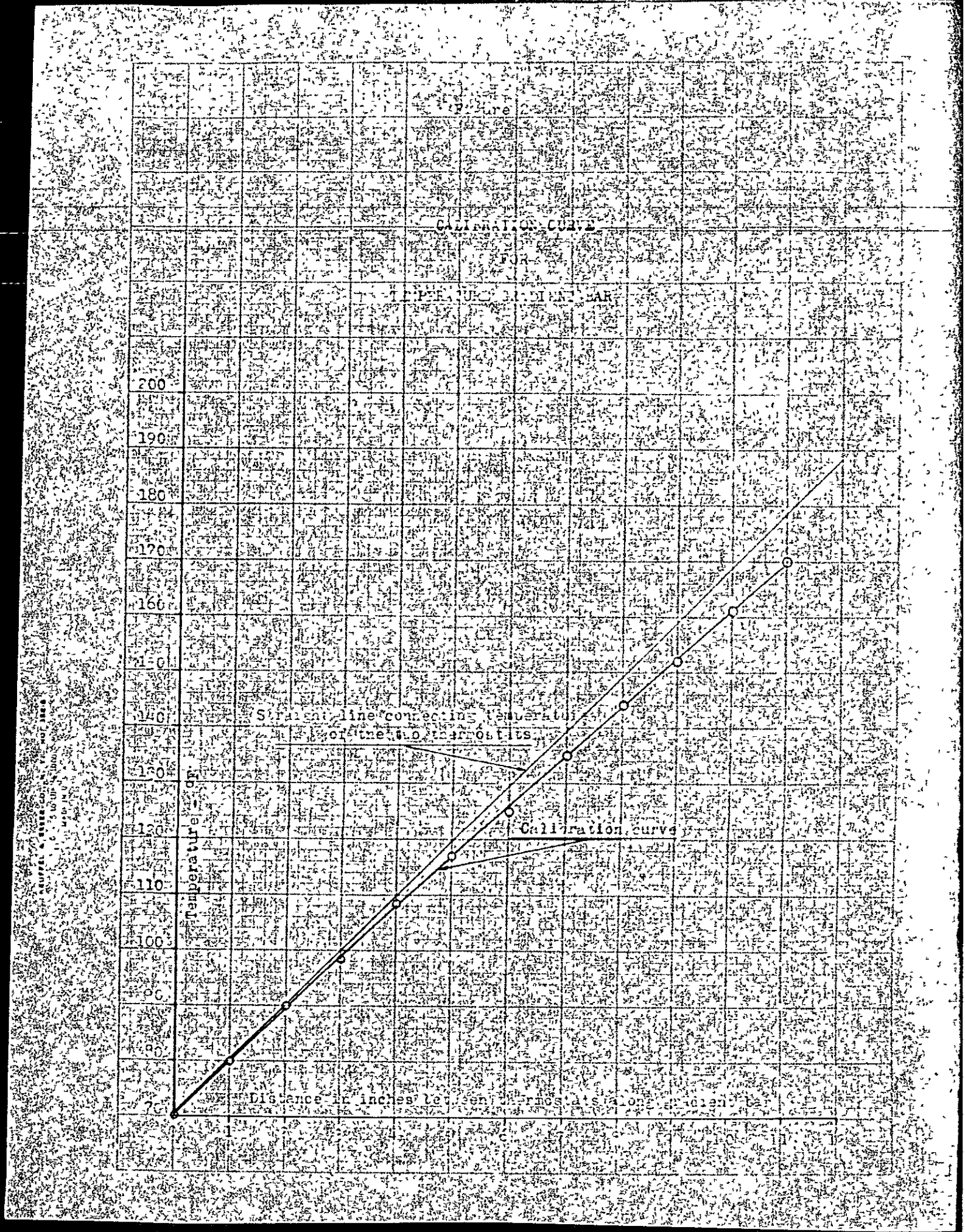
* Actually reads 190°F. with one thermometer; 187° with another. Thermocouple used for surface measurement of gradient bar read 188°F.

Figure 2

CALIBRATION CURVE

FOR

TEMPERATURE GRADIENT BAR



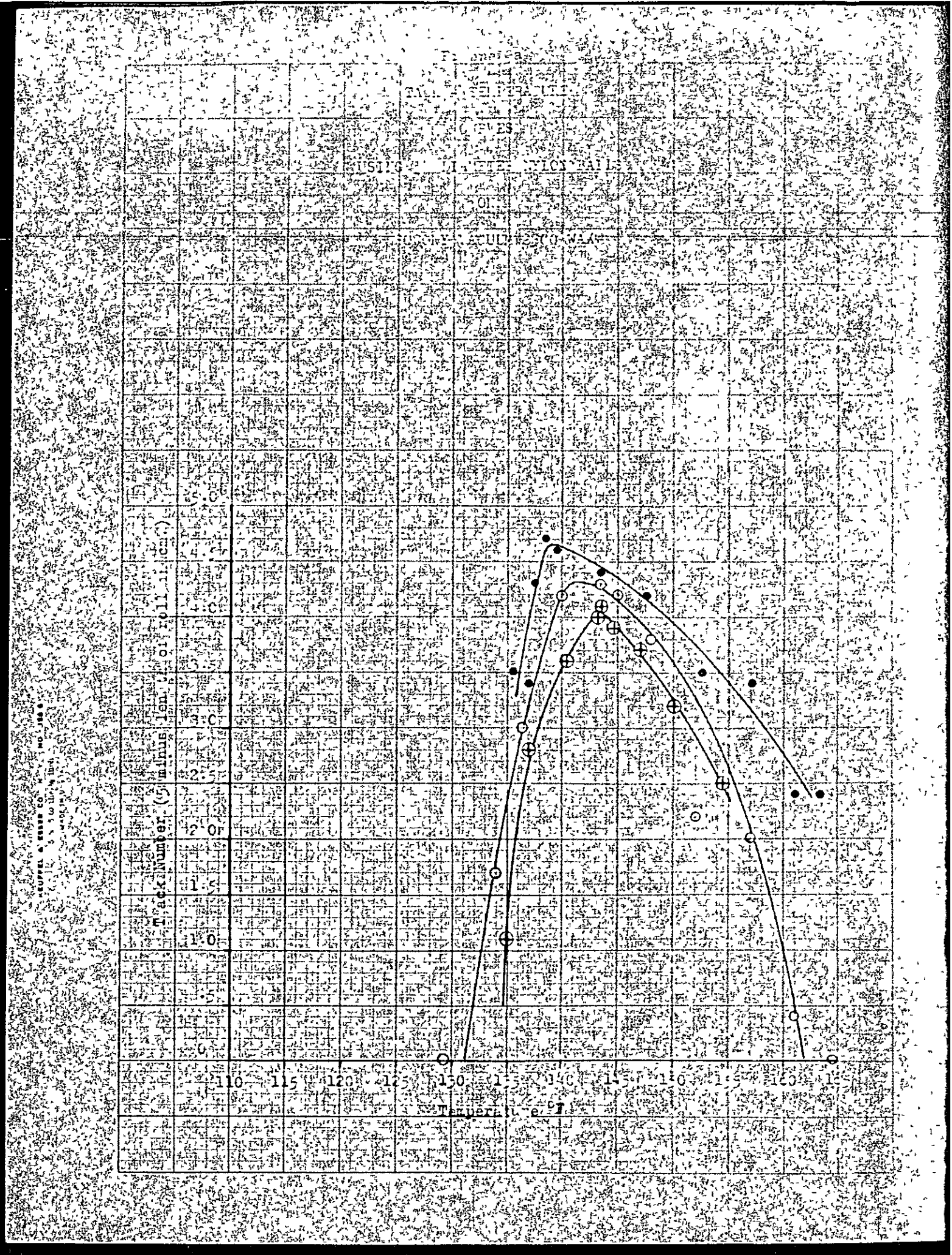
retarding effect on the rolling ball so that the further the ball rolled the less tacky the wax. For example, at a temperature high or low enough for the wax to be either liquid or solid, only a small retarding effect would be produced whereas at in-between temperatures in which the wax is in a quasi-solid or tacky state, the retarding effect would be larger. The cooling effect of the balls on the wax was neglected since the balls were nylon and have quite poor heat conducting properties.

It was noticed in attempting to duplicate the results obtained with one of the waxes that the thickness of the wax coating was very critical. Therefore a procedure was adopted for casting reproducible coating thicknesses. This consisted of masking off the edges of the gradient bar with Scotch Tape, and then with the gradient bar at a fairly uniform temperature above the melting point of the wax, the masked-off area was coated with molten wax by means of a heated smoothing bar. The reproducibility of this procedure as shown in the accompanying data in Figure 4 was dependent on the constancy of the temperature of the smoothing bar at the time of application.

RESULTS

In all of the curves in Figures 3, 4 and 5 below, the distance which the nylon balls rolled in centimeters was subtracted from five (arbitrarily chosen so that a roll of 5 cm. or more would be considered zero tack), and the resulting "tack number" plotted against the temperature along the path of roll on the gradient bar.

The curves in Figure 3a and 3b were obtained with the 1/4" diameter balls on two waxes. In these curves no effort was made to control the smoothing


Track Number (5) minus Temperature (°F)
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
110
115
120
125
130
135
140
145
150
155
160
Temperature °F

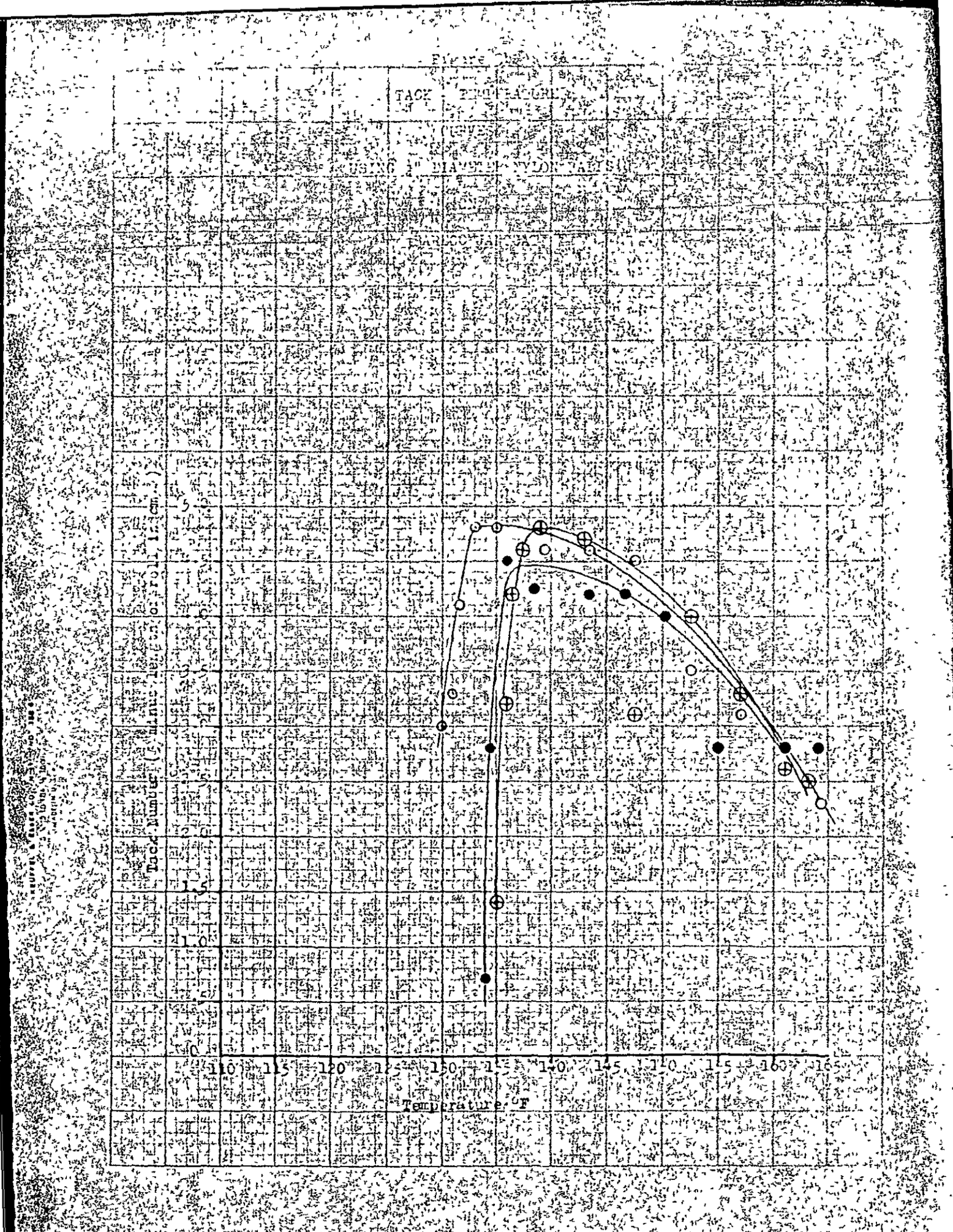
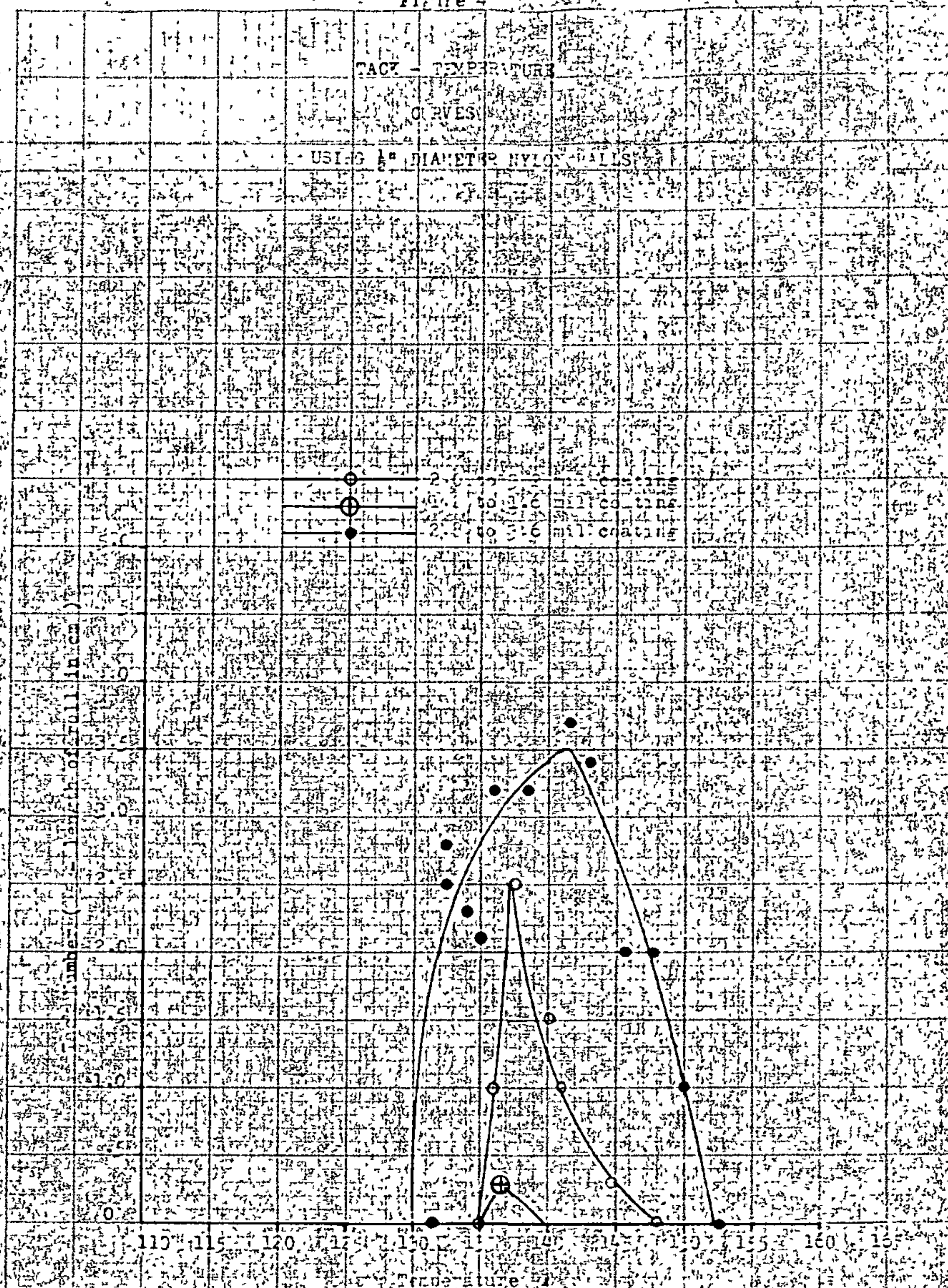

TACK
Tack, Number of Grams
Temperature, °F

Figure 4


TACK - TEMPERATURE
CURVES
USING ½" DIAMETER NYLON BALLS
mil coating
to mil coating
to mil coating
0
1.0
2.0
110
115
120
130
140
150
155
160
Temperature

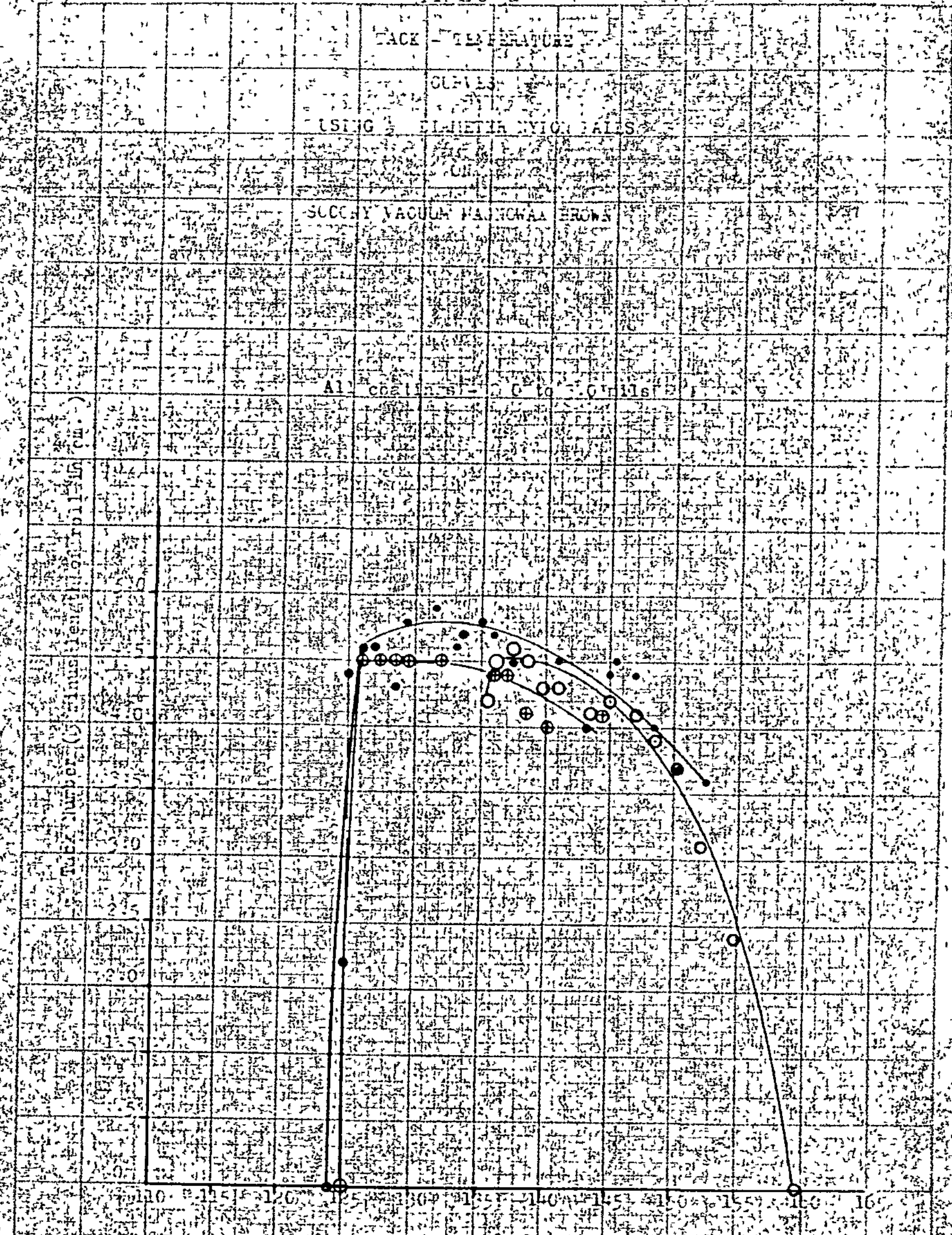
bar temperature and consequently the wax coatings were not of equal thickness and the 3 curves for each wax, therefore, did not coincide.

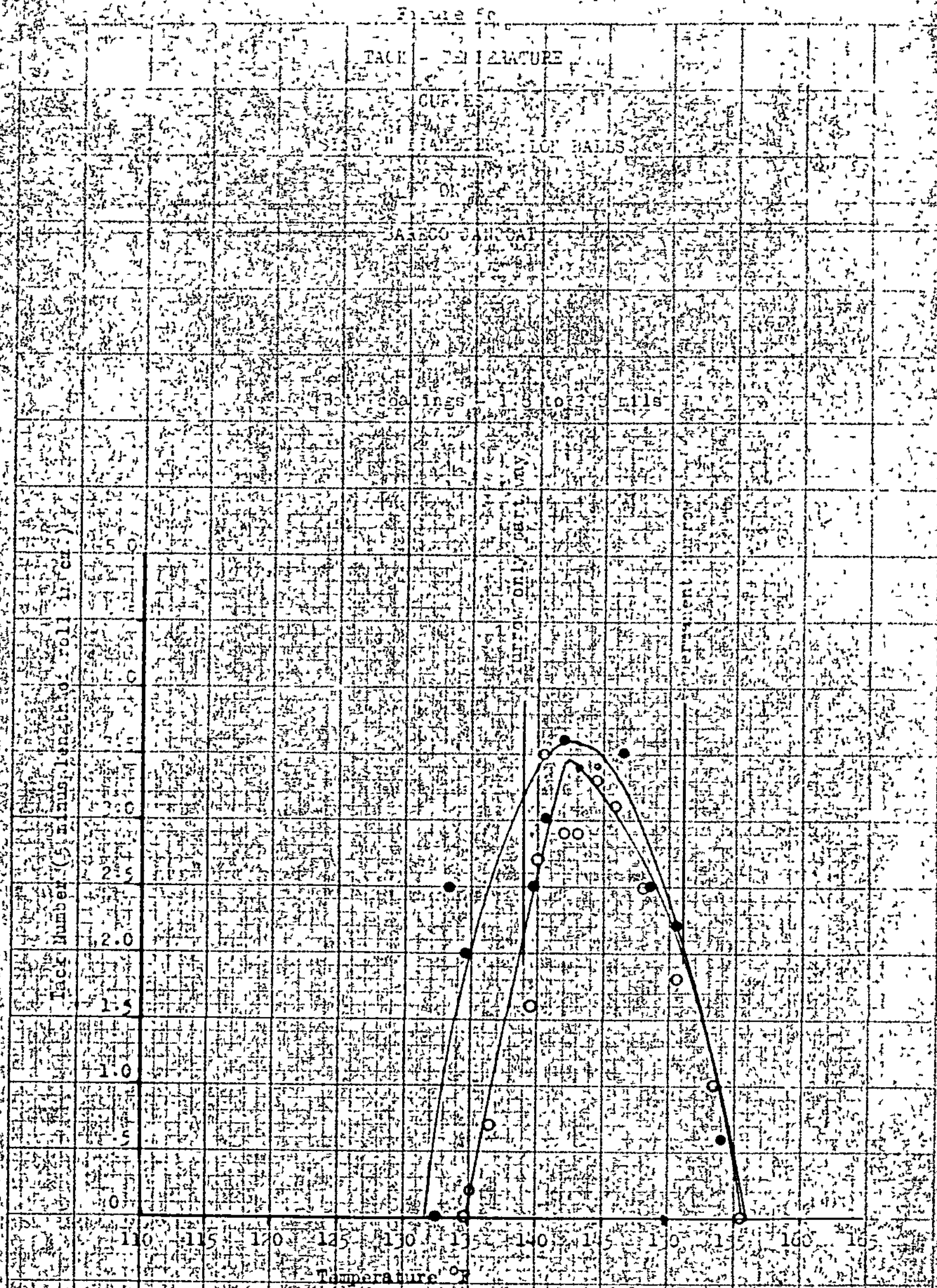
The curves in Figure 4 were obtained with the 1/2" diameter nylon balls on the Socony Vacuum 2300 wax. The 2.0 to 3.2 mil. coating was obtained with the smoothing bar heated only slightly over the melting point of the wax by means of a steam jet. The 1.2 to 1.6 mil. coating was obtained with an unheated smoothing bar, and the 2.8 to 3.6 mil. coating was obtained with the smoothing bar heated far above the melting point of the wax by means of a gas flame. All thickness measurements were made by means of an Interchemical thickness gage.

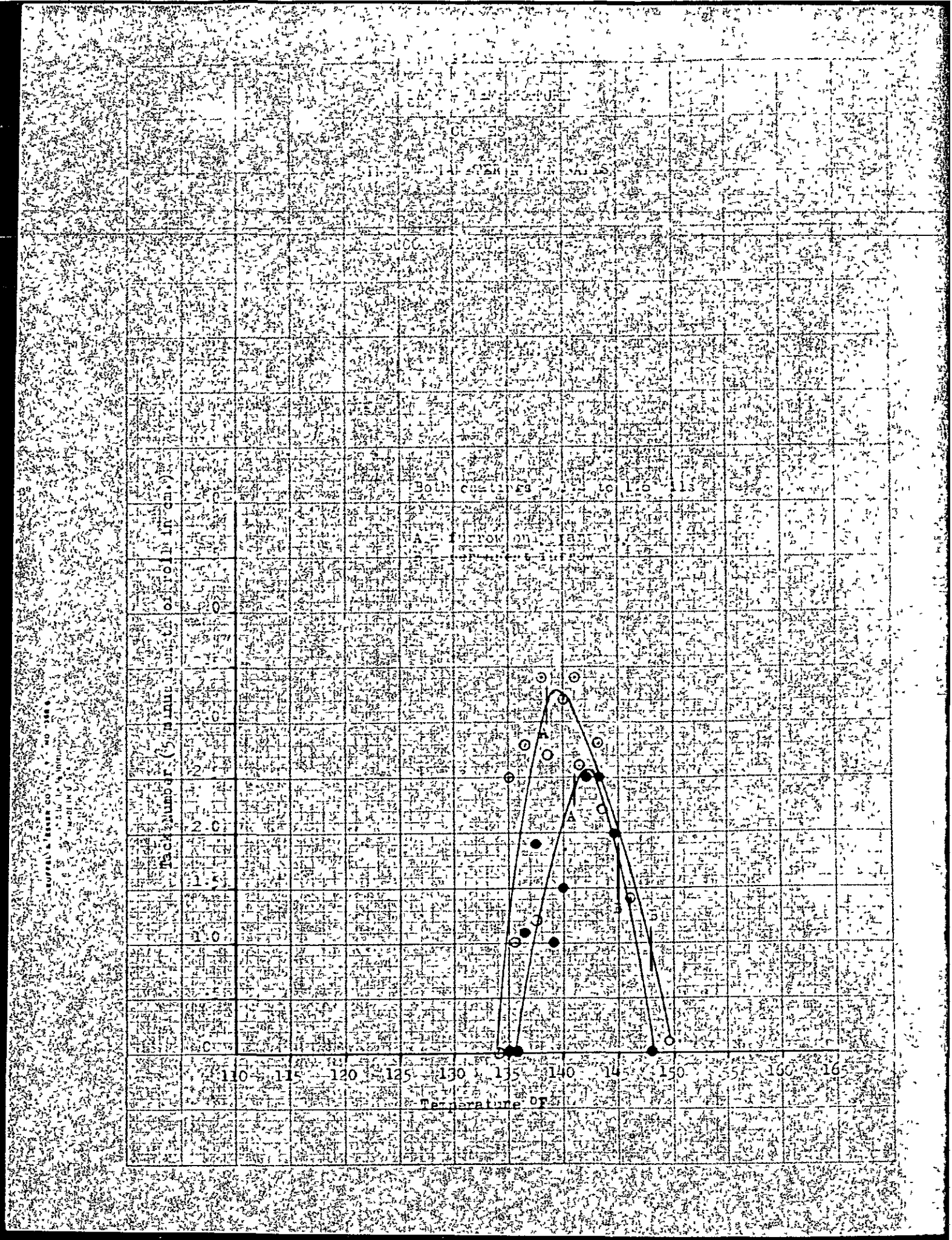
In Figures 5a, 5b, 5c, and 5d, the coatings represented by all the curves were made with the smoothing bar held at as nearly a constant temperature as possible. This was done by immersing the bar, a 1/4" diameter rod, in the 188°F. thermostat for a substantial interval prior to the application of the coating. The coating thicknesses were again determined by an Interchemical thickness gage.

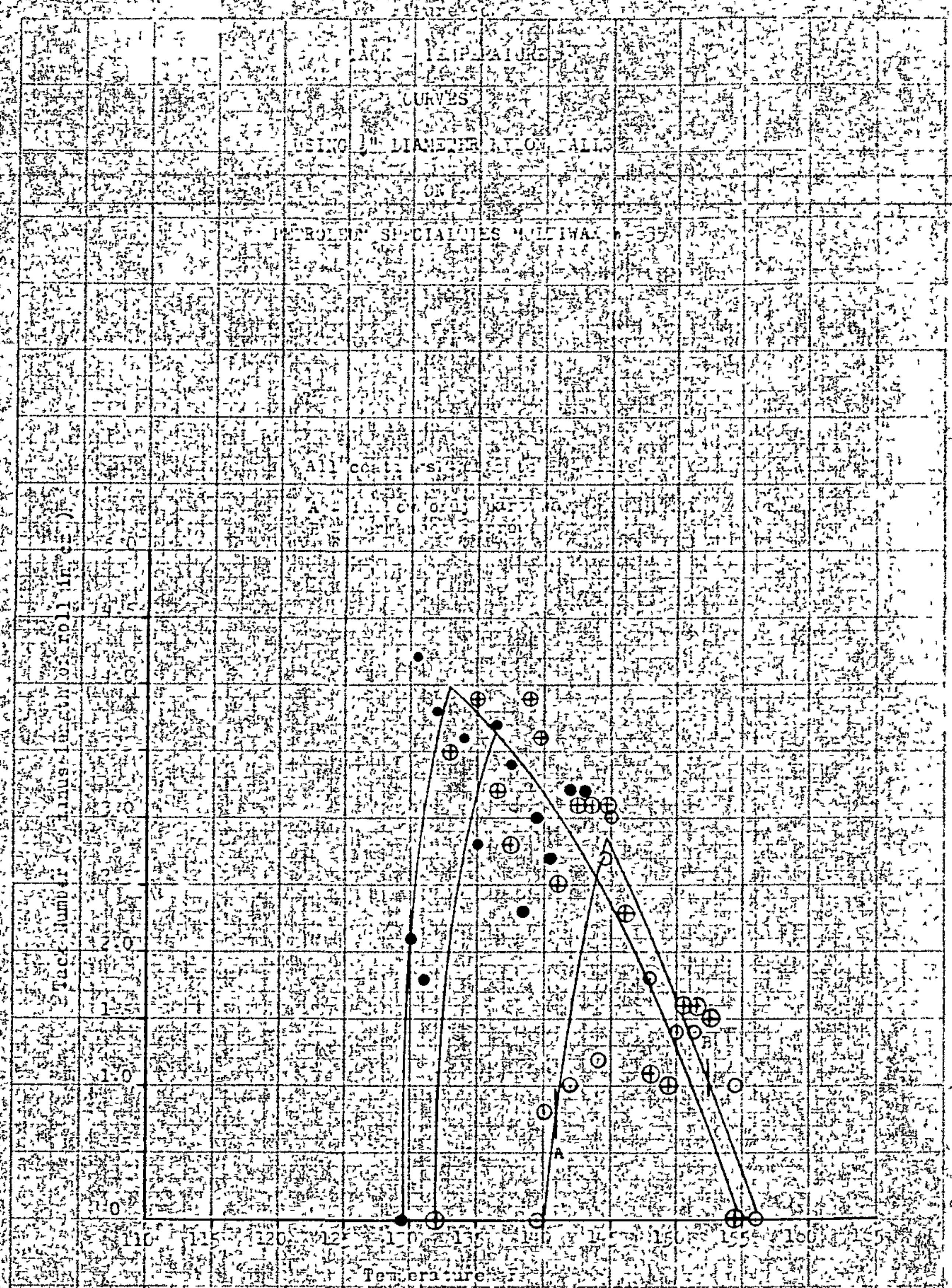
Besides the retarding effect data of the various waxes tested as represented by the curves in Figures 3, 4, and 5, several other observations were made. These observations are in reference to the effects of the rolling ball on the wax coating. Three general effects were noted and might be used to establish an arbitrary demarkation between the liquid state, the solid state, and the intermediate quasi-liquid -solid state. The effect in the liquid state might better be considered a lack of a permanent effect. In other words, the rolling ball in such cases left no permanent evidence on the

Figure 5a


TACK - TEMPERATURE
CURVES
USING ½ DIAMETER NYLON BALLS
SCOTCH VACUUM FASHOWAA BROWN
All coatings - 0.0 to 3.0 mils
Temperature °F.


TACK - TEMPERATURE
CURVES
ON
Both coatings
mils
Tack Number
5.0
2.0
1.0
0
110
115
130
140
160
Temperature °F


Both curves
to 1.6
Temperature °F


CURVES
110
115
120
130
150
155
160
1.0
0
A
Temperature

wax surface. In the solid state the effect was a furrow in the wax for only part of its travel and a dull line or trace for the remainder with a sharp line of demarkation between the end of the furrow and the beginning of the trace. In the region between these states the furrow in the wax surface extended the entire length of ball travel. In all cases where a furrow was made its depth was down to the chrome plated wax casting surface.

DISCUSSION OF RESULTS

It appears that the method employed above produces results which are not reproducible, although the reproducibility varies from one wax to another; Magnowax Brown being the best and Multiwax W-835 the poorest. The principal reason for the lack of reproducibility is the depending of the test on wax film thickness. Other factors which might be at least partly responsible are variations in the amount of kinetic energy imparted to the ball as a result of the release mechanism (see Appendix A), foreign material or lumps on the surface of the wax, slight variations in the temperature of the wax from the calibration curve for the gradient bar as a result of local cooling by convection currents, etc., and snowballing or building up of wax on the ball after traveling a distance more than the circumference of the ball. The effect of variation of weight from ball to ball was eliminated as a possible reason for lack of reproducibility by running curves in Figure 5 with only one ball which was washed in heptane after each roll.

This method of rolling a ball over a tacky surface and measuring the energy absorbing capacity of the surface is used in the pressure sensitive adhesive tape field for measuring the tack of these adhesives. A more

direct and perhaps more flexible and more reproducible method might be to use a procedure described by Busse, Lambert, and Verdery[1,2] in which a solid surface is brought into contact with the tacky material by means of drive mechanism which provides positive contact over a regulated time interval and a positive withdrawal. The resistance to separation is measured as tack. This test was designed for measuring the tack of GR-S rubber.

Another test which might be applicable to microcrystalline wax is similar to the above test except that it is used for printing ink where the wettability of the ink for the separating surfaces is not a factor. This test measures primarily the flow characteristics of the ink and was designed by Green[3] as a replacement for the old subjective "finger tab out" test by which the finger is pressed upon a thin film of the tacky material then withdrawn suddenly.

Busse, Lambert and Verdery's test would be applicable for microcrystalline waxes only in the case cited in the introduction in which contact is established at the same temperature as the "apparent adhesion" is measured. Green's test on the other hand would be applicable in both cases; the one just mentioned and also the one in which contact is made at one temperature and measurement of "apparent adhesion" at another temperature. However, there are better tests which might be used for the latter case. These are stripping tests of which many are described in the literature and the reason they might be considered better is that they more closely resemble actual use conditions.

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1. W. F. Busse, J. M. Lambert and R. B. Verdery "Tackiness of GR-S and other Elastomes". J. App. Phys. 17, 376-385(1946)

2. J. M. Lambert and R. A. McDonald, Rev. Sci. Inst., 19 (1948)119.

3. H. Green. Ind. Eng. Chem., Anal. Ed., 13 (1941)632.

rbg/geh

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TRANSPARENTIZING OF PAPER

The possible use of epoxy resins for transparentizing of paper was investigated.

For the initial tests the treating solution consisted of:

Araldite No. 102	15 parts (by wt.)
Hardner No. 951	1 part (by wt.)

Regular lampshade stock and an absorbent saturating stock were tested by a float-dip process and by a vacuum impregnation.

The viscosity was too high to allow penetration into the machine-finished dense lampshade stock. However, the absorbent paper was impregnated by the vacuum treatment.

The resin impregnated sheets were cured overnight at room temperature. The fully impregnated sheet was fairly transparent.

This idea appears to have possible value for use for lampshades, envelope windows, and packaging applications.

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Frans Vaurio

HYGROEXPANSIVITY

This report covers part of the work in a study of means of reducing the hygroexpansivity of paper. Resorcinol was used as a resin forming chemical capable of entering the amorphous portion of the cellulose molecule. It was reacted with formaldehyde using para-toluene sulphonic acid as a catalyst.

Handsheets were prepared by disintegrating beaten (450 C.S. freeness) and dewatered St. Mary's Kraft pulp with a Williams disintegrator until free of knots. This took about 20 minutes. The consistency of the wet pulp was 27.89% and 163.2 grams were diluted with 8937 grams of demineralized water, and 910 grams of this stock were used per sheet. The sheets were made on an 8-1/2 by 8-1/2 inch recirculating handsheet mold. The wet sheets were pressed at 65 p.s.i. for 3 minutes then dried for 10 minutes at about 215°F. Some of these sheets were used for the following treatments and some of the untreated sheets were used as controls. One set of controls was treated with formaldehyde but without the resorcinol.

Treating With Resorcinol

The following standard procedure was used in treating a number of the sheets. (1) The raw weight of each sheet was determined. (2) Resorcinol was dissolved in alcohol in various percentages. (3) Specific gravity of the resorcinol alcohol solution was noted. (4) The sheets were dipped in the resorcinol solution and drained for 15 seconds. (5) The wet weight was obtained. (6) The

sheets were allowed to air dry in a hood. (7) After the air drying the sheets were treated with formaldehyde. (8) The sheets were treated with para-toluene sulphonic acid in water solution to give approximately 1/2 per cent on the amount of resorcinol. Another procedure used for treating with resorcinol was to make up a mixture 25 grams resorcinol, 400 grams of 37 per cent commercial formaldehyde, 8.65 grams of para-toluene sulphonic acid and 425 grams of water. A number of sheets were dipped in this and then dried as before. This mixture tends to gel in a couple of hours so the treatment was done immediately after the mixture was made. Several modifications of the formaldehyde procedure were tried, and the following appeared to work the best. About 500 ml. of commercial formaldehyde were placed in a liter round bottom flask with a single hole stopper. The flask was heated with a bunsen burner. A glass tube led to the bottom of a 2 liter glass graduate in which the sheet of paper was placed in a form of a cylinder. A metal screen was used to support the sheet of paper above the bottom of the cylinder to prevent it from becoming too wet. The vapors from the formaldehyde were passed into this large cylinder through a glass tube extending to the bottom of a two liter graduate. The excess vapor was absorbed in an erlenmeyer flask containing a solution of sodium thiosulfate to pick up the excess formaldehyde. Approximately 5 minutes of boiling was used in each case to obtain formaldehyde for treating of the sheets. Table I gives the treatment used with the standard procedure. Table II gives the treatment used with the mixture of resorcinol, formaldehyde and catalyst. The sheets were sent to the humidity room for testing for basis weight, caliper, wet and dry tensile, and hygroexpansivity on the Neenah instrument. The results of the basis weight, caliper, wet and dry tensile are given in Table III. The results of the hygroexpansivity will be reported when they are finished.

TABLE I

RESORCINOL TREATMENT OF HANDSHEETS*
(St. Mary's Unbleached Kraft 450 C.S.)

No.	Raw Wt.	Wet Wt.	Resorcinol Solution s. g.	% solids	Pickup %(solid on fiber)	Catalyst** g/liter	ml./applic
29-1	5.24	9.90	.860	15	13.3	2.5	3.1
2	5.30	10.75	.860	15	15.4	2.5	3.6
3	5.30	10.72	.863	15	15.3	2.5	3.5
4	4.79	9.70	.824	5	5.1	.25	11
5	5.22	10.20	.824	5	4.7	.25	11
6	5.05	9.68	.824	5	4.5	.25	11
7	4.55	9.15	.815	2	2.0	.25	4
8	4.32	9.05	.815	2	2.2	.25	4
9	4.49	9.02	.815	2	2.0	.25	4
10	4.39	9.09	.812	.67	.70	.025	13
11	4.55	8.80	.812	.67	.61	.025	13
12	5.12	9.19	.812	.67	.52	.025	13
13	4.90	9.10	.810	.33	.28	.025	4.4
14	4.85	9.88	.810	.33	.34	.025	8.8
15	4.62	9.22	.810	.33	.31	.025	8.8

* Handsheets 8-1/2 x 8-1/2 inches. Treated with formaldehyde vapor after resorcinol treatment.

** para-toluene sulphonic acid. Applied by spraying water solution.

TABLE II

TREATMENT OF HANDSHEETS WITH RESORCINOL FORMALDEHYDE AND CATALYST MIXTURE*
(St. Mary unbleached kraft 450 C.S.)

No.	Raw Wt.	Wet Wt.	Dry Wt.	Pickup, % (fiber basis)
1020-32-1	4.87	14.31	5.31	9.3
1020-32-2	4.92	14.30	5.31	7.9
1020-32-3	4.89	13.83	5.25	7.3
1020-32-4	4.73	13.42	5.10	7.8
1020-32-5	4.65	13.14	5.08	9.2
1020-32-6	4.40	12.70	4.85	10.2

Note: 8-1/2 x 8-1/2 handsheets dipped in the resorcinol mixture

* Formula

25 g. Resorcinol
400 g. Formaldehyde (37%)
8.65 g. para-toluene sulphonic acid
425 g. water

TABLE III

EVALUATION OF HANDSHEETS RESORCINOL TREATMENT

Treatment*	Code	Basis Wt.	Caliper	Tensile Dry[a]	Tensile Wet**
Control none	151590	77.7	.0084	27.6	1.0
Alcohol and formaldehyde 1/2	151591	76.1	.0086	21.5	1.1[b]
5	151592	76.8	.0086	22.7	4.9
15	151593	83.7	.0091	30.9	11.9
	151594	100.1	.0107	25.4	28.7

* Per cent resorcinol - fiber basis
** 16 hour soak
[a] Tested at 50% R.H., 73°F.
[b] The average of only five determinations instead of ten

fv/ak

Further tests will be conducted along this line, possibly treating the fiber in the anhydrous state and also in the wet state, then subsequently treating with formaldehyde and catalysts and curing the sheets. We will also try to cure the resorcinol in the fiber before sheetmaking, using formaldehyde and catalyst.

HYGROEXPANSIVITY OF RESORCINOL TREATED PAPER

Treatment* Code		Hygroexpansivity, % Change from 65-50% R.H. Max.	Min.	Ave.**	Change from 65-33% R.H. Max.	Min.	Ave.**
Control none	151 590	.180	.174	.177	.352	.332	.341
Alcohol and formaldehyde	151 591	.176	.162	.169	.348	.324	.335
1/2	151 592	.242	.178	.200	.470	.354	.394
5	151 593	.140	.132	.135	.282	.266	.274
15	151 594	.092	.066	.080	.204	.130	.173

* Per cent resorcinol - fiber basis

** Average of three determinations

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HYGROEXPANSIVITY

This report covers the results of tne hygroexpansivity tests on the sheets treated with resorcinol, formaldehyde vapor and finally with a catalyst. The tests on the sheets treated with the mixture of resorcinol, formaldehyde and catalyst are still under way, and will be reported when they are available. The sheets tested are described in Project Report No. 11. The tests were performed on the Neenah expanismeter in two different ranges. One from 65% to 50% relative humidity, and the other from 65% to 33% relative humidity. The specimens were conditioned to relative humidities of 50% and 86% respectively for obtaining expansivity values presented in this report. The values shown represent single determinations on different sheets. The average of the tests on three sheets are then shown. The results appeared to indicate that the treatment must be up in the range of 5 to 15% before any real reduction in hygroexpansivity is expected. At .5% resorcinol based upon the fiber, the results were actually poorer than with the control sheets. With 5% there was an appreciable drop in hygroexpansivity, and with 15% the results are better than one half of the hygroexpansivity of the control sheet. The work with the wet tensile strengths indicates that a goodly percentage of the normal fiber to fiber bonding has been replaced by some form of a resin type of bonding, since the wet strength was very great. It is hoped that we can trace this down still further by trying to get the resorcinol into the fiber prior to the sheet-making operation in order to assure the location of the resorcinol within the fiber.

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Darwin Bork

HYGROEXPANSIVITY

This report covers part of the work in a preliminary study of means of reducing the hygroexpansivity of paper. Resorcinol was used as a resin forming chemical, selected for the possibility of being capable of entering the amorphous portion of the cellulose molecule. It was reacted with formaldehyde using paratoluene sulfonic acid as a catalyst. St. Mary's southern kraft pulp (unbleached) was beaten to a 450 c.s. freeness. Handsheets were made as described in Report No. 11. The treatment of these sheets with a resorcinol solution containing formaldehyde and paratoluene sulfonic acid is described in Report No. 11. These sheets were tested for basis weight, caliper, hygroexpansivity, and wet and dry tensile. The results obtained are shown in Table I.

The results appear to be quite comparable to those obtained by adding the resorcinol and the formaldehyde treatments separately. Apparently we have not differentiated as to the location of the resin in the sheets. There appears to be a rather wide variation between the minimum and maximum wet tensile, indicating that the treatment was not too uniform. The wet tensile was not quite as high as with previous treatment, but was still appreciable amounting to approximately 23% of the dry tensile. The resin pickup ranged from 7.3 to 10.2% of the fiber weight. Further work is contemplated with the aim of trying to get more

of the resin into the fiber by having the fiber in a more open or expanded form at the time of introduction of the resorcinol.

TABLE I

USE OF RESORCINOL TO CONTROL HYGROEXPANSIVITY OF PAPER
(Use of a Single Dip Method)

Code No.	Resin %	Basis Wt. lb. (25 x 40/500)	Caliper inch	Hygroexpansivity Change from 65-50% R.H.	Hygroexpansivity Change from 65-33% R.H.		Schopper Tensile lb./inch Dry	Schopper Tensile lb./inch Wet
1162-32-1	9.2	82.3	.0091	0.106	0.218	Max.	30.0	17.8
1162-32-2	9.9	83.1	.0089	0.084	0.182	Min.	24.7	2.5
1162-32-3	7.3	81.8	.0089	0.094	0.190	Av.	27.2	6.2
1162-32-4	7.8	79.5	.0088	0.086	0.172			
1162-32-5	9.2	79.0	.0087	0.100	0.204			
1162-32-6	10.2	75.3	.0081	0.080	0.172			
Av.	8.9	80.2	.0088	0.092	0.190			

Dictaphone fv/dl

PROJECT REPORT FORM

PROJECT NO. 1102-10
COOPERATOR Institute
REPORT NO. 14
DATE October 12, 1954
NOTE BOOK 1162
PAGE 86 TO 90
SIGNED Betty John
Betty John

Copies to: Files
Howells
John

FLAME RESISTANT TREATMENTS FOR PAPER WITH DIETHANOL AMINE PHOSPHATE AND TRIS BETA CHLOROETHYL PHOSPHATE

INTRODUCTION

Samples of tris beta chloroethyl phosphate and diethanol amine phosphate were received from the Mathieson Chemical Corporation to be considered as possible flameproofing agents for paper. No suggestions for application to the paper or technical literature accompanied the samples. All correspondence dealing with the samples is found in the letters dated April 30, 1954, April 8, 1954, and March 22, 1954 with the Mathieson Chemical Corporation.

MATERIALS AND METHODS

The base sheet for impregnation was the kraft flame resistant base stock from the International Paper Company. It has a basis weight of 31.7 (24 x 36/500). [See Project 1742].

The flame resistance test methods and apparatus are those utilized for Project 1742. They are as follows:

A wooden test cabinet 12 inches wide, 12 inches deep, and 30 inches high was constructed inside a draftproof hood. The cabinet front is provided with a sliding glass door, and the top is constructed with a hooded opening 8 inches in diameter. The cabinet is equipped with a Bunsen burner and a pilot light.

The specimen being tested (a strip of paper 12 inches by 2-1/2 inches) is supported vertically above the Bunsen burner by means of a clamp which covers the upper 0.5 inch of the specimen. Steel clips, 0.25 inches wide, extend down the sides of the specimen to prevent curling away from the flame. The lower edge of the specimen is adjusted to 0.75 inches above the top of the burner which produces a flame height of 1.5 inches with the air supply completely shut off. A flame is applied to the lower edge of the specimen for 12 seconds and then shut off. Afterglame and afterglow time are noted.

The one-minute leach test consists of immersing the treated paper in running tap water (rate of flow -- 31 liters per hour) for one minute. The specimens are held in the water current by means of paper clips to assure exposure of all surfaces.

Solutions of known concentration of the samples in appropriate solvents were prepared and used for impregnation. Impregnation was accomplished by dipping 5 preweighed sheets into the solution and passing the sheets through squeeze rolls to remove the excess. The sheets were reweighed and wet pickup was determined. From this data add-on was calculated as a percentage of the raw weight of the paper. The specimens were dried on a drum drier for 15 minutes at 230° F. and subjected to the flame resistance test 15 - 30 minutes after removal from the drier.

RESULTS

Diethanol amine phosphate:

Diethanol amine phosphate was found to be miscible with water and a 30% solution was prepared and used for impregnation. Successive solutions for impregnation were prepared by dilution of the above solution to a lower solids content. Table I shows the amount of initial flame resistance at various add-ons.

TABLE I

% Add-on	Char Length, Inches *	
25	4	(0)
24	4-1/4	(0)
18	5	(0)
14	10-3/4	(4)
9	12	(5)

* Figures in parentheses indicate the number of specimens out of five which burned completely.

For convenience of tabulation and the computation of averages, 12 inches (the length of the sheet) was used when the sheet had burned completely.

As indicated by the table, adequate flame resistance will result in the range of an 18% add-on. No afterglow or afterglame was noted for any of the specimens. No noticeable deterioration of the physical properties of the paper was noted.

A one-minute leach was carried out on the specimens showing an add-on of 25% with the result that all flame resistance was lost during leaching.

It was considered that a durable treatment could be accomplished by the addition of appropriate agents which would react with one of the active groups of the sample (possibly the alcohol or amine) and thereby insolubilize it with respect to water. Therefore formaldehyde was added to the impregnation solution and a 15 minute cure at 300° F. followed initial drying of the samples in an effort to permanentize the treatment. No leach resistance resulted. Further efforts to permanentize the treatment were not carried out since adequate diethanol amine phosphate was not available to evaluate the possibilities further.

Tris Beta Chloroethyl Phosphate:

Tris beta chloroethyl phosphate was found to be immiscible with water, slightly miscible with carbon tetrachloride with cloudiness, and miscible with methyl ethyl ketone. A solution of tris beta chloroethyl phosphate in methyl ethyl ketone was prepared and used for impregnation. No initial flame resistance resulted with an add-on in the range of 50%, but a lower rate of burning appeared. However, in view of the large add-on further treatments were not considered.

bj/mk