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The Effects of Starch on Pickup, as Influenced by Solids, Temperature, Viscosity and Depth of Penetration on Sheet Properties

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THE EFFECTS OF STARCH ON PICKUP,
AS INFLUENCED BY SOLIDS, TEMPERATURE,
VISCOSITY AND DEPTH OF PENETRATION
ON SHEET PROPERTIES.

A Thesis Submitted by

James W. Rann

In partial fulfillment for the re-
quirements of a Bachelor of Science Degree
from Western Michigan University
Kalamazoo, Michigan

To The Department of Paper Science
and Engineering
Western Michigan University

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ABSTRACT

This report is a review of the current status of knowledge in the area of size press variables. It delves into the sheet properties affected by the application of surface size.

A pilot plant study was conducted which evaluated the effect of Penford Gum 280. This starch was applied to the 46 pound 50% hardwood, 50% softwood, 15% clay filled sheet being formed on the 30 inch wide pilot papermachine at Western Michigan University. Starch application was made at various temperatures and solids concentrations to obtain different amounts of pickup and variable depths of penetration.

The results of this study include a plot of the intimate relationship of solids, temperature, viscosity, and pickup. There is also a plot of solids, temperature and depth of penetrations influence on the amount of pickup. Sheet properties of burst, pile, tear, TEA, elongation, stiffness, wax pick, W&N ink holdout, smoothness, and air permeability are evaluated. All properties were found to have maximum benefit from a 3% pickup range at different application temperatures. An attempt was made to evaluate the influence of penetration on the sheet properties. This evaluation was difficult because the increased depth of penetration was only half again as deep as the original depth.

LITERATURE REVIEW

INTRODUCTION

An investigation of published literature on surface sizing and coating operations was carried out. Primary objectives were to find which properties of the base sheet are influenced by surface size, how to alter them, and their ultimate effect on the printing qualities of coated paper. An abundance of material was found on each individual topic, but very little relating the three. A shortage of man hours was foreseen and therefore a condensed study was decided upon.

HISTORY (1)

Starch sizing agents have probably been around as long as paper, beginning with the Chinese in 312 A.D. It is believed that Arabs used starch for surface sizing and uncooked starch for filler. Starch, however, was largely displaced by animal size about the fourteenth century, in a tub size apparatus, which remained until modern times. Chemical modifications of starch improved properties so that animal sizing agents have been virtually eliminated.

DEFINITIONS OF SURFACE SIZING

There are probably as many definitions for surface sizing as there are papermakers in the world. Some are

very general like, "anything added to paper." (2) A little less generally, sizing may be defined as the addition of materials to the furnish for paper or board in such a manner that these materials increase the resistance of the sheet to penetration by liquids or vapors, with surface sizing being the addition of the sizing material to the surface of the sheet after the sheet has been formed (3).

Size can be defined as any chemical, other than bleach, fillers, pigments, and dyes, which are added to the paper-making furnish, or subsequently applied after the web is formed, which alter those characteristics of the sheet that relate to its resistance to the transudation or absorption of liquids which come into contact with the web.

Sizing, then, is the chemistry and processing of paper products to alter their resistance to the passage of liquids or gases into and through them (4). There is a calender stack treatment used on heavier grades of paper. Application requires generally higher viscosity, size, and temperatures.

REASONS FOR SURFACE SIZING

There exists a variety of reasons for applying surface size. Surface sizing of paper improves the finish, produces a better surface for printing, minimizes scuffing, controls air permeability, prevents excessive or undesirable penetration of other finishing agents, decorates or improves appearance, and improves strength characteristics (5,6).

In addition to the above reasons for surface sizing the following are included:

- 1) Increase pick resistance
- 2) Increase smoothness
- 3) Improvements in erasability
- 4) Improved physical strength
- 5) Improved resistance to the passage of liquids and gases
- 6) Paste lint or fuzz to the sheet
- 7) Decreases ink absorption
- 8) Modify handle - touch or feel of the sheet
- 9) Enhanced appearance by uniform cockle finish
- 10) Increase hardness or rattle for quality impression

TYPES OF SIZE PRESSES

Basically there are two main types of size presses in use today, a recently developed one and another on machine application methods available for use. At the present time the vertical size press is the most used in the industry. (28) This press design is shown in figure 1.

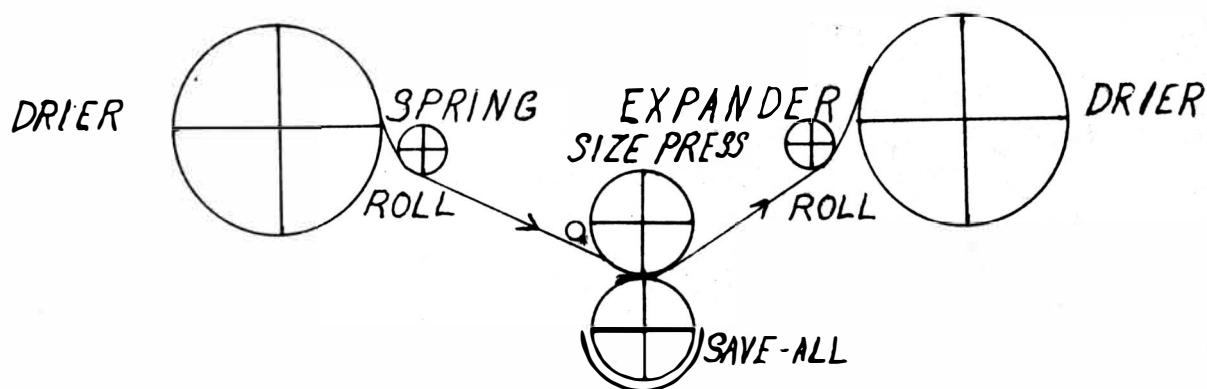


FIGURE 1

The rolls are arranged on a vertical centerline with the movable roll arm connected to a lifting-load device. Under the bottom roll is a save-all pan which extends slightly above the horizontal centerline. The size outlet from the save-all can be varied in height to control the depth of the size pond. The sheet is led into the press at a slight downward angle to hold the size at the nip of the press. Size is flowed onto the sheet in front of the press and pressed into the sheet. The bottom of the sheet is either sized by dipping the roll into the save-all pan or by spraying the size on the bottom roll and allowing it to deliver the size at the roll velocity (12,14).

The second type press that has recently received attention is the horizontal size press shown in figure 2.

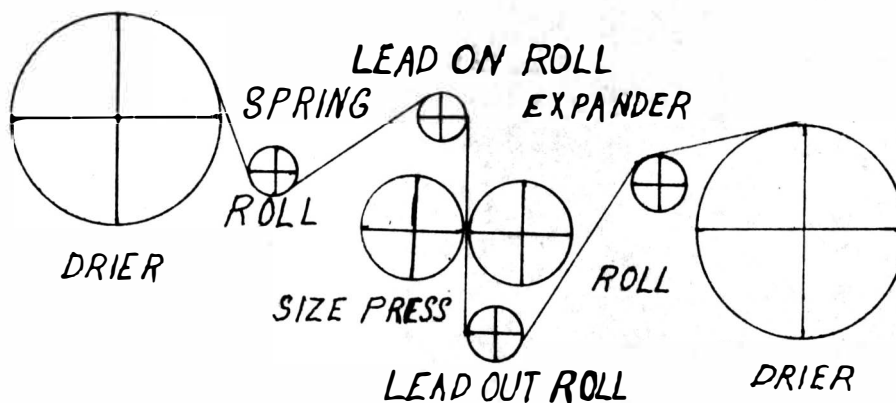


FIGURE 2

This type of press is basically the same as the vertical with a pair of rolls and a loading device. Size is usually applied, with one spout on each side of the sheet, located at the center of the sheet. A sufficient quantity of starch

is applied to spill over the ends of the rolls. Sheet entrance is critical but tension is not as critical as it is for the vertical press (12,14,15,27). There is no heavy load of a size pool on the web. Size applied in this press is less likely to produce the two sided effects seen in the vertical press. (27)

The latest press on the market is the inclined size press. It is structured like the two above presses except the press rolls are arranged on an incline. The size is applied to the sheet just in front of the press. The angle of incline on the press has been adjusted so that the paper can be fed directly into the dryers. This press eliminates the problem of wrinkles, which is caused in the horizontal press because the sheet expansion can not be fully compensated for by expander rolls.

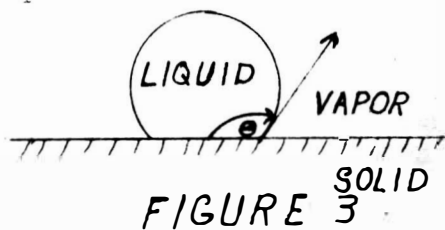
Tub sizing is a method whereby the paper is submerged into the size for about seven feet in a long pan. The excess size is squeezed out by a reverse lead around the bottom soft roll, and a wrap of the wire side against the top hard roll. This process requires slow machine speeds and therefore is usually done off the paper machine.

SURFACE SIZING THEORETICAL

Wetting and penetration of all solids is dependent upon a number of basic principles, which are best expressed by the classic Dupre and Washburn equations.

In the contact angle phenomenon of wetting the angle between the liquid and solid determines whether the sheet is

wettable or non-wettable. For this paper the non-wettable properties are important.



If the sheet is to resist spreading then it is desired to have the free energy of the solid vapor interface less than or equal to the free energy of the liquid plus that of the solid liquid interface. The angle θ , when this situation is obtained, is above 90° , and the sheet is referred to as non-wettable.

Dupre (29) further developed a mechanism for penetration of the liquid into the sheet which is related to the driving force.

$$\Delta P = 2(S_{sv} - S_{sl})/r$$

ΔP = pressure differential driving force

S_{sv} = free energy of solid vapor interface

S_{sl} = free energy of solid liquid interface

r is the pore radius

Resistance to wetting therefore would require a small driving force. This would be obtained by making the value of the free energy of the solid-liquid interface close to that of the solid vapor interface. In addition to the driving force, the rate of penetration of liquid entry into the sheet is important.

Washburn (30) presents the following equation for the rate of entry.

$$v = l/t = (rs_1/4\eta)\cos\theta$$

v = velocity, cm/sec

l = depth of penetration, cm

t = time, sec

r = pore radius

s_1 = surface to area of the liquid, dynes/cm²

η = viscosity, poises

θ = contact angle of liquid on surface, degrees

This equation gives a measurement of the penetrating power of the liquid in a given situation.

Cobb (13,20,21) modified Washburn's equation to the following:

$$l^2 = rs(\cos\theta t)/(2\eta)$$

All parameters are detailed above. This modification measures the depth of penetration at any given time.

The parameters from above that can be changed include: free energy of the solid interfaces by the addition of internal size and other compounds; the pore radius which can be plugged by fillers, surface sizing compounds, and other additives, or by the use of small fibers and fiber fines. It should be pointed out that the pores are not necessarily round, but could be blocked at one end, squares, cracks, and many other shapes. The viscosity of the fluid will inversely alter the velocity and depth of penetration of the fluid.

VARIABLES IN SURFACE SIZING

There are three sets of variables in the surface sizing process: material variables, machine variables, and web variables.

Material Variables

Due to its uniformity of quality, supply, its wide range of viscosities, and in particular its low price, starch has been the most used product. (1) Corn, potato, and tapioca are the predominant types of starches on the market. In the U.S., corn is the most widely used of the three. In addition to starch, other agents are presently being used for surface sizing. Some of these agents include; animal glue, polyvinyl alcohol, alginates, and carboxymethyl cellulose (CMC). (27)

Shirley (5) classified starch products used in surface sizing into the following groups: enzyme converted; medium to high fluidity thin boiling; low soluble dextrines and gums; oxidized starches; and starch derivatives and chemical condensation with other products. Because of lesser solution instability, exceedingly good film properties, and little or no tendency to foam, Strasser (8) and Casey (1) claim the oxidized starches are best suited for this operation.

A second variable in the materials category is a group of interrelated factors. Temperature, solids concentration, and viscosity are the components of this group variable.

The effect of the starch temperature on its penetration into the sheet was found contested by six authors. Half (3,11,14) felt that an increase in the temperature of the starch would cause an increase in penetration into the sheet. The other half (5,14,18) felt the increased temperature would decrease the penetration.

Looking at the theoretical discussion above, both groups could be correct. If the temperature affects the free energy of the liquid more than it does the viscosity, then penetration would be increased, as in Cobb's equation. The increase could be due to an unbalancing of the forces holding the contact angle. The driving force ΔP may have increased enough to cause it.

Temperature increases will increase the rate of penetration (3). There is some dispute as to how viscosity and penetration are related. It is contended by some (5,12,19) that if there is an increase in viscosity the penetration will decrease. They feel for the most part that penetration is equal to one over the square root of the viscosity. Two authors (10,18) feel that penetration will increase when the viscosity increases.

The first argument, that the penetration depth is equal to one over the square root of the viscosity, relates directly back to Cobb's modified equation where the depth of penetration squared varies inversely with the viscosity. The second argument could be due to the particle size, sheet formation, core radius, or possibly the pressure applied at the nap caused the driving force ΔP to be high enough to increase the penetration. 2e

Temperature's effect on pickup is also questioned as some authors (3,14,16) felt increasing the size temperature would increase the pickup. However, other (17,18) felt that

the size temperature increase would cause a decrease in pickup. This is possible if less solids reach the sheet. The first argument could be due to more penetration holding more starch on the sheet.

Viscosity is decreased by temperature within a range as the particles are able to move farther apart. Outside this range, evaporation of water would proceed at a sufficient rate to increase the viscosity. The viscosity is affected by the molecular size of the compound. It depends on the network structure of the molecule, how spread it is, and how well hinged together. The viscosity will increase the amount of starch picked up, when the viscosity is increased. Increasing the solids content will increase the viscosity which will decrease the depth of penetration according to Cobb's equation. Increasing the solids content will increase the pickup is the belief of some authors (12, 15, 16, 18). One other important variable of the size press solutions is its solution stability characteristics. Some products have a retrogradation problem, sometimes setting up like a jell, other times crystallization caused incomplete cooking.

Machine Variables

In the size press, sizing operations are affected by some variable and some non-variable factors. The major variables include: the roll loading, because of its ability to squeeze the size out of the sheet; roll diameter influen-

tial in the amount of pickup; and the crown required for the roll. Today controlled crown rolls are used to minimize the roll diameter and therefore the necessary crown. Small crowns are desirable because larger nip pressures can be applied. Roll hardness and material are the design features presently in dispute (12,15). The only feature agreed upon here is that one roll should be harder than the other. Some of the materials in dispute are bronze, stainless, or gunmetal versus stonite or microrock. Different hardnesses used include those in the plastometer ranges of fifteen to fifty-five. Most people agree that the hard roll should be used on the side needing the better finish, but they question whether the better finish is due to hardness of the roll or surface smoothness. The hardness of the roll will change the pickup. By purchasing a softer roll one could increase the surface size pickup (12).

The speed at which the paper machine is running is important because of the dwell time which influences the penetration (14). The larger t is in Cobb's equation, the deeper the penetration (1^2). The sheet leaving the press on a slight angle will provide clean pull away and a minimum of sheet waver (12,15). Other factors are: dryer temperatures; sheet tension going through the press; teflon dryer rolls; and bowed rolls.

Increasing the nip pressure which is influenced by the above machine variables will increase the penetration. This factor can be directly explained in the theoretical discussion. The nip pressure causes a large ΔP or driving force causing the fluid to penetrate the sheet. (Nip pressure when increased will in fact decrease the percent pickup of the paper. (12,14,15,17,18).)

Web Variables

This classification can be broken down into two subclasses; surface and sheet structure.. One surface property of interest here is smoothness which aids in regulating the size pickup (5,12,13). Another key property is wettability of the surface. The penetration of size is based on relationships which have already been discussed. Another factor is the amount of internal size that has been added to the sheet. The larger the amount in the sheet the less will be picked up at the size press. The distribution of the internal size is also likely to affect the size picked up. The consensus seemed to be that increasing the internal size of the sheet will decrease the pickup (5,12,15).

The sheet structure consists of density, pore size, moisture content, and formation. In the density considerations, the amount of beating time (3) which aids fiber packing; the type of wood which influences individual fiber density, the amount of broke added to the stock slurry which affects filler, fiber length, and fines; and other additives

must be considered. These sheet density affects are agreed upon by a number of authors (9,12,15,26). Increasing sheet density will decrease pore size which, from the previous discussion of Washburn's equation, should reduce the penetration. The density could also have an effect on the free energy of the sheet, which would change the contact angle and therefore the wettability of the sheet. Increases in the sheet density due to the above reasons will decrease the pickup (15).

The pore size which is partly due to refining and furnish as an influential factor is agreed upon by Clarke (22), Dappen (23), Durfy (14), and Lee (20). The pores perform the disservice of letting water into the sheet so the surfact size is added to reduce the number and/or size of the pores to reduce wettability.

Increasing the radius in the Dupre equation decreases the driving force which should lower penetration. It should however, from the Washburn equation, increase the rate of penetration. Since the ΔP in Dupre's equation is altered by the nip pressure larger pore sizes should increase the penetration.

The moisture content next in line for discussion has many stated ranges, including 3-35% (17), and 5-12% (5,10,15,18). Optimum moisture was stated as 12% by a couple of the authors. The moisture would possibly affect the free energy of the solid, thus increasing the contact angle.

It may reduce the driving force, and inhibit the rate of penetration through the cosine of the angle as previously discussed.

Pickup is increased by increasing moisture within the above stated ranges. The last variable is sheet formation, under which comes uniformity, desired in this study by some authors (13,24). Reif (25) says that the presence of fines on a sheet surface gives maximum printing characteristics. He also says their distribution in the sheet is of utmost importance because of their relationship to pore size.

SHEET PROPERTIES AFFECTED BY SURFACE SIZING

In the strength properties category the burst and tensile and fold will increase with increases in the surface size (4,8,10,14,17,18). Tear is not really agreed upon by all. Durfy (14) claims it decreases with surface size increases. Dreshfield (4) states tear increases with surface size increases. Walker (20) argues that tear strength is not improved, but it benefits indirectly from reduced stock treatment providing drying is controlled. Chilson and Fahey (18) concluded there is no affect on tear.

Included in the surface properties is smoothness, which many agree is improved by surface starch (4,2,10,11,13,17). Fuzz, a big problem to the industry is decreased by applications of starch on the surface of the sheet (4,8,11). Improvement in erasability is gained by the increasing of the surface size (4,9,12). Pick resistance is improved by

increasing the amount of starch on the surface of the web (7,10,13,16). Scuff resistance, Dreshfield (4) claims, is improved by increasing the surface size.

Ink absorption and water absorption, factors considered as sheet wettability, are both considered here, and both are altered by surface sizing (12,18).

Optical tests reduced by increasing the surface size are brightness (11) and opacity (18). Gloss is improved by surface size (18). Two summary tables have been compiled. The first relates penetration to each of the material, machine, and web variables, and the second relates pickup to each variable.

TABLE I

INFLUENCE OF VARIABLES ON PENETRATION

TYPE OF VARIABLES	PENETRATION INCREASES	PENETRATION DECREASES
<u>MATERIAL</u>		
Higher application temperature	3,11,14	5,17,18
Higher viscosity	10,18	5,12,19
Higher solids content		author
Solution instability		5,7,8,17
<u>MACHINE</u>		
Increasing nip pressure	18	
Increasing roll loading	12,15	
Increasing roll hardness	12,15	12,15
Type of material (roll)	12,15	12,15
Increasing speed		14
Increasing drier temperature		12,15
<u>WEB</u>		
Increasing internal size		author
Increasing beating time		3
Increasing pore size	14,20,22,23	
Increasing moisture (5-12% range)		author

TABLE II

INFLUENCE OF VARIABLES ON PICKUP

TYPE OF VARIABLES	PICK UP INCREASES	PICK UP DECREASES
<u>MATERIAL</u>		
Increasing temperature	3,14,16	17,18
Increasing viscosity	author	
Increasing solids content	12,15,16,18	
<u>MACHINE</u>		
Increasing nip pressure		12,14,15,17,18
Increasing roll hardness		12,15
<u>WEB</u>		
Increasing moisture (5-12% range)	3,5,10,15,18	
Increasing internal size		5,12,15
Increasing smoothness		5,12,15
Increasing sheet density		3,15

Table 3
Sheet Properties

TYPE OF PROPERTY	INCREASE	DECREASE	UNCHANGED
<u>Strength</u>			
Burst	4, 8, 10, 14, 17, 18		
Tensile	4, 8, 10, 14, 17, 18		
Fold	4, 8, 10, 14, 17, 18		
Tear	4	14, 20	18
<u>Surface</u>			
Smoothness	4, 7, 10, 11, 13, 17		
Fuzz		4, 8, 11	
Erasability	4, 9, 12		
Pick Resistance	7, 10, 13, 16		
Scuff Resistance	4		
Wettability		12, 18	
<u>Optical</u>			
Brightness		11	
Opacity		11	18
Gloss	18		

EXPERIMENTAL

The objectives of this laboratory study, conducted on the pilot paper machine at Western Michigan University, using Penford Gum 280 as the surface size, being applied to a 46# sheet in production consisting of 50% hardwood, 50% softwood, and 15% clay, fall into two basic categories: to study the effects of starch pickup on sheet properties and to relate the degree of penetration to these properties. More specifically, the sheet properties to be evaluated will include the strength properties of tensile, elongation, tensile energy absorption (TEA), tear, burst, and wax pick. The surface properties of K&N Ink holdout, smoothness, and wax pick and the structural properties of stiffness and air permeability will also be evaluated. In examination of increased pickup, the factors of temperature, solids, and viscosity will be looked at in terms of their influence on the pickup, sheet properties, and penetration.

There are four groups of procedures followed in this report, consisting of; preliminary, run conditions, starch application, and testing.

PRELIMINARY PROCEDURES

Sixteen five-gallon buckets were coded each to receive starch at the correct solids, which were adjusted to the right temperature before running. Sixteen jars were also used in order to save starch solutions for viscosity and solids testing.

Stock preparation was the next order of business required before the papermachine run. The furnish consisted of 50% bleached, draft hardwood, 50% Samoa softwood, 15% B.W.W. clay filler, and 2% alum. A Hollander type beater was used which handled a charge of 220 pounds of dry fiber. The following weights were added in oven dry quantities for the fiber and other additives; 110 pounds of hardwood pulp, 110 pounds of softwood pulp, 33 pounds of clay, and 4.25 pounds of alum. Each of five beaters was charged and beaten to a Canadian Standard Freeness (CSF) of 440 ± 15 ml.

The starch was prepared as follows: 41.5 pounds of Penford gum 280 were added to the starch cooker and heated to 195° F at 20% solids, where it was held for fifteen minutes. This solution was diluted to forty gallons, and four of the five gallon containers with corresponding code for solids were filled.

The remaining starch was diluted to 13% solids, and the temperature fell to about 180° F at this point. Again the appropriate four buckets were filled with this starch solution. Dilution to 10+% solids was the next step, and the starch was reheated to 180° F with steam. The appropriate four buckets were then filled with starch. In order to obtain the last four samples, seventeen pounds of starch were added to the cooker and diluted to 7% solids, where it was cooked for fifteen minutes at 195° F.

The solids I had anticipated I did not reach, so an alternative cooking method is suggested here. Remove the samples

from the cooker at about 2% more than desired and draw off only about $4\frac{1}{2}$ gallons. Then when the run is started, steam can be added to raise the starch temperature to about 15° above the desired run temperature. The solution should then be diluted to the five gallon mark with hot water of equal temperature. Cooling water can also be added to reduce to the desired temperature for the run. This will give a much better solids control.

RUN CONDITIONS

Paper was made continuously on the thirty-inch wide Louis Calder Paper Machine at Western Michigan University. The sheet was adjusted to 47 pounds basis weight (25x38-500) with a machine speed of eighty feet per minute. The average dryer surface temperature was 255° - 260° F with a $4\frac{1}{2}\%$ moisture entering the size press and a final moisture of about 5%. The pH was trimmed to 4.8 with sulfuric acid. Rosin was added continuously at the mixbox at a rate of .2% based on the fiber. This was obtained by dissolving five pounds of rosin in fifty gallons of soft water.

STARCH APPLICATION

The temperature of the starch was raised to the desired range by direct steam heating or cooled to range by pouring water on the outside of the bucket. One feed tank of the size press was filled, and the starch was circulated to the tub and shower of the size press. The size press on the pilot paper-machine is a verticle size press with a rubber bottom roll and steel top roll. While the temperature was being adjusted with

the steam coils in the bottom of the storage tank, the next starch was prepared. When each starch solution reached the desired temperature, the reel was flagged as the start of the run, and shortly after a twenty foot sample was saved from the roll. The next starch sample was then added to the second tank, adjusted to temperature. The roll was flagged as the end of this run. The new starch was then circulated through the system, and the above steps were repeated. Table IV gives the sequence of temperature and solids used. The solids were checked by evaporation of a sample of each starch solution.

TESTING OF SAMPLES

For these tests dependent on basis weight, average test values were divided by a ratio of the average weight of each type paper divided by the average weight of the run (46.4 pounds).

For the tensile, elongation, and TEA tests, four-inch by twenty-five-millimeter samples were cut. The Instron tester was used for elongation, tensile, and TEA tests. The TEA was calculated according to the equation $TEA = 3.774$ times the integrator reading. The Scheffield Smoothcheck was used to test smoothness, with the average of five tests per side being recorded as the smoothness for that sheet. Porosity was tested using the Scheffield Porosimeter with the $3/8$ -inch diameter holes. Again, five readings were used as the average. The bursting strength tests were recorded as the average of three readings per sheet, with one test felt side and two wire side on the Perkins Mullen tester.

TABLE IV
SAMPLE TESTING SEQUENCE

<u>SEQUENCE #</u>	<u>SAMPLE CODE</u>	<u>SOLIDS %</u>	<u>TEMPERATURE °F</u>
1	AW	18.0	185
2	BW	13.1	185
3	CW	10.8	185
4	DW	7.1	185
5	AX	14.0	150
6	BX	13.1	150
7	CX	10.2	150
8	DX	7.2	150
9	AY	---- ^a	120
10	BY	11.6	120
11	CY	9.9	120
12	DY	6.8	120
13	AZ	---- ^b	92
14	BZ	13.0	92
15	CZ	10.4	92
16	DZ	6.6	92

a This starch solution ran long enough to collect a 16 foot sample but then became too viscos to be circulated. It was diluted before a solids sample could be obtained.

b This starch solution was too viscos to ren at all. No sample was saved for a solids sample.

The Dennison Wax test was run on each side of five samples. No difference was found between the felt and wire sides of the samples.

The Elmendorf Tear tester was used to test samples in both machine and cross machine direction.

The Gurley Stiffness tester was used to test samples in both directions. The average of the two sided swing was used for the value reported for the sheet.

For the K&N ink test, ink was spread on a glass plate. A rubber roller was rolled through the ink, which picked up an ink film, and the film was then transferred to the sheets of paper being tested. The ink was allowed to penetrate for two minutes then scraped off with a metal spatula and wiped clean with a cotton cloth. The brightness was measured on the uninked and the inked portions. The inked brightness was divided by the paper brightness and multiplied by 100 to give the reported value. A high value is equivalent to good hold-out of ink. The Elrepho was used to test these brightnesses.

The percent pickup by the sheet was determined by analyzing for the total starch in paper using TAPPI Standard T 419 su-70. The samples were corrected for turbidity due to the suspended filler by standardizing against extract from standard samples of paper which contained no starch. Table V and Figure 4 present the calibration curve values and curve respectively for the starch determinations.

The last test was a tapergrinding process, conducted according to a procedure of Wink (31), to study the depth of

penetration. A vacuum plate was made by the machine shop in the Physics Department at Western Michigan University which held the paper samples to be ground to its face. A .004 brass shim was placed under one end of the plate, and the plate was magnetically held to the grinding table. A vacuum was pulled using an industrial type vacuum cleaner with dual motors. The samples were ground with a course stone by the surface grinder in the Physics Department. Mr. Richard Dourbin performed the grinding and is here thanked for his contribution. A 3/4-inch grind was made on each side of the sample. The samples were measured for depth of grind on a caliper tester and marked according to remaining caliper. The samples were then stained with potassium iodide, iodine solution. The solution was applied by a paint brush on the ground area. Where the starch could be detected as partially removed, a line was drawn. The original caliper minus the caliper at this line determined the depth of penetration.

Table VI is a chart summarizing the number of samples that were tested and the tests that were evaluated in this laboratory study.

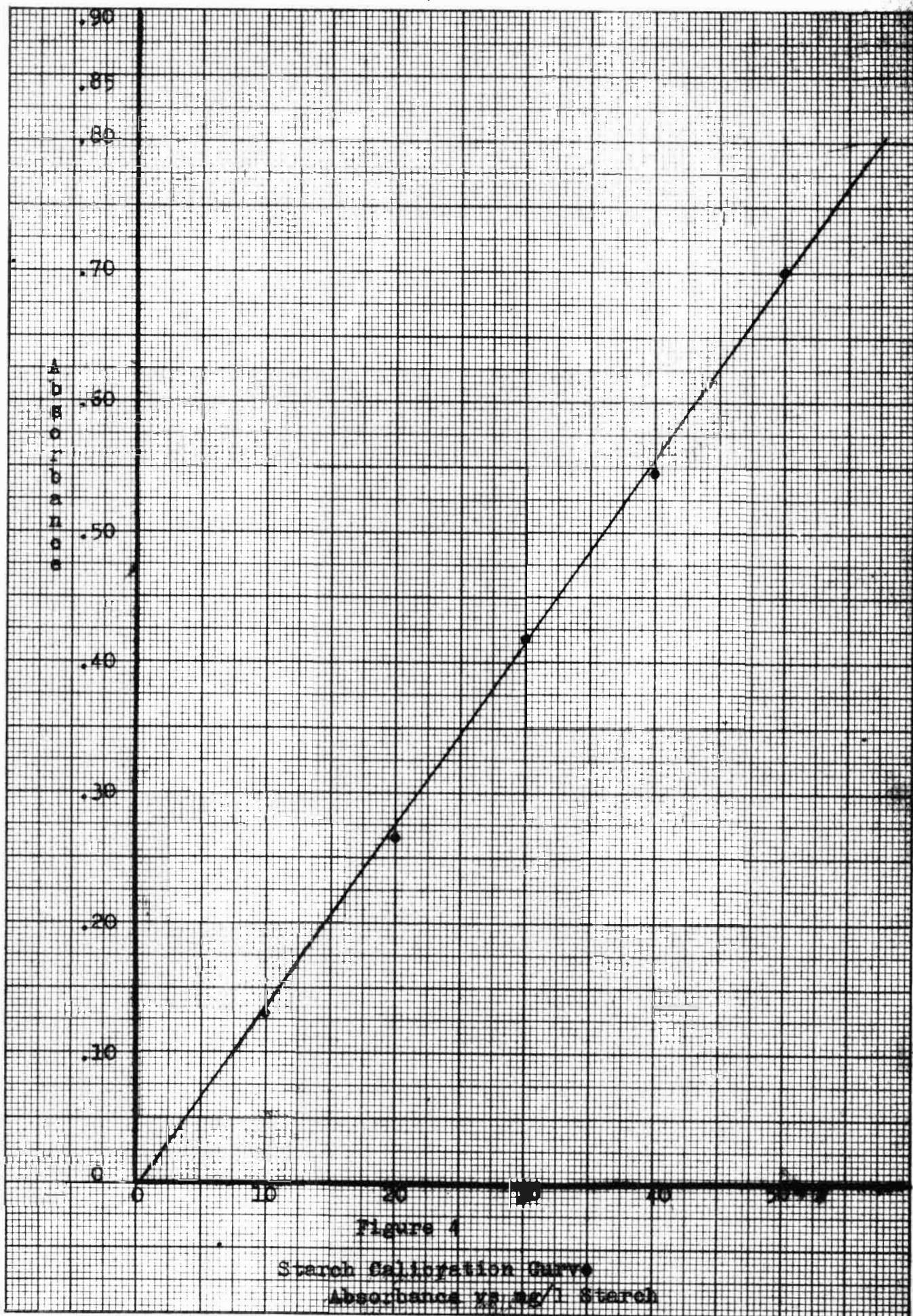
TABLE V
CALIBRATION CURVE DATA FOR RELATING STARCH
CONCENTRATION AND ABSORBANCE

milliliters starch solution used at 2 mg/l	starch concentration milligrams per liter	Absorbance
5	10	.130
10	20	.265
15	30	.417
20	40	.545
25	50	.700

TABLE VI
NUMBER OF SAMPLES TESTED AND AVERAGED
TO BE PLOTTED AS ONE VALUE

TEST	NUMBER OF TESTS	CROSS MACHINE MACHINE	FELT WIRE
Stiffness	20	X	
Tensile	25	X	
TEA	25	X	
Elongation	25	X	
Tear	20	X	
Smoothness	25		X
Porosity	25		X
Burst	25		X
Dennison Wax	10		X*
K&N Ink	10		X*
Depth of Penetration	4		X*

* Each test is the evaluation of one side



PRESENTATION OF RESULTS

Table VII summarizes the test results by listing significant differences between types of paper for their stated property. The temperature of starch application exhibiting the best performance for each paper property is listed. The best performing sheet is listed above the slash line, and the samples of paper over which it showed the most significant differences is listed below the slash line. For example, the tensile tests most improved by starch application in the low range (1%-2%) were the 185⁰F, 150⁰, and 120⁰ (all others), and the least improved tensile value was found to be the 92⁰F starch application. Tests like this with more than one value above and/or below the line mean that the multiple samples showed the same or nearly the same results for this test.

The following discussion is an in-depth look at the results of the testing summarized in Table VII. Each area discussed will be followed by the figures appropriate to discussion.

SOLIDS, TEMPERATURE, VISCOSITY, PICKUP RELATIONSHIPS

Figure 5 shows the relationship of the solids to the viscosity at constant temperatures. The starches at less than 10% solids in the 120⁰ F to 185⁰ F range all maintain about the same viscosity. From here they spread out with the high temperature rapidly increasing in % solids but slowly

TABLE VII

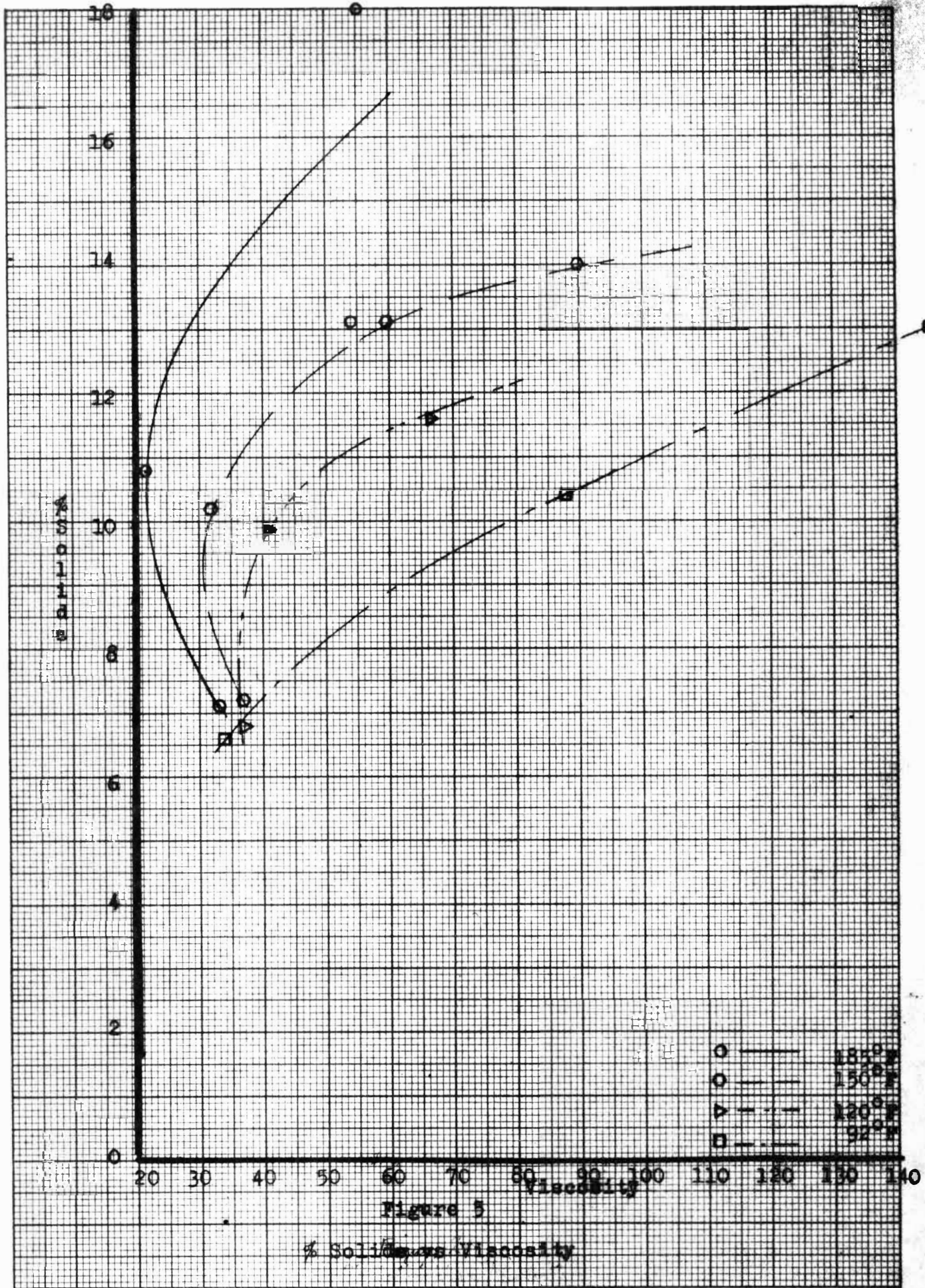
SHEET PROPERTIES EVALUATED
BEST PERFORMER / WORST PERFORMER

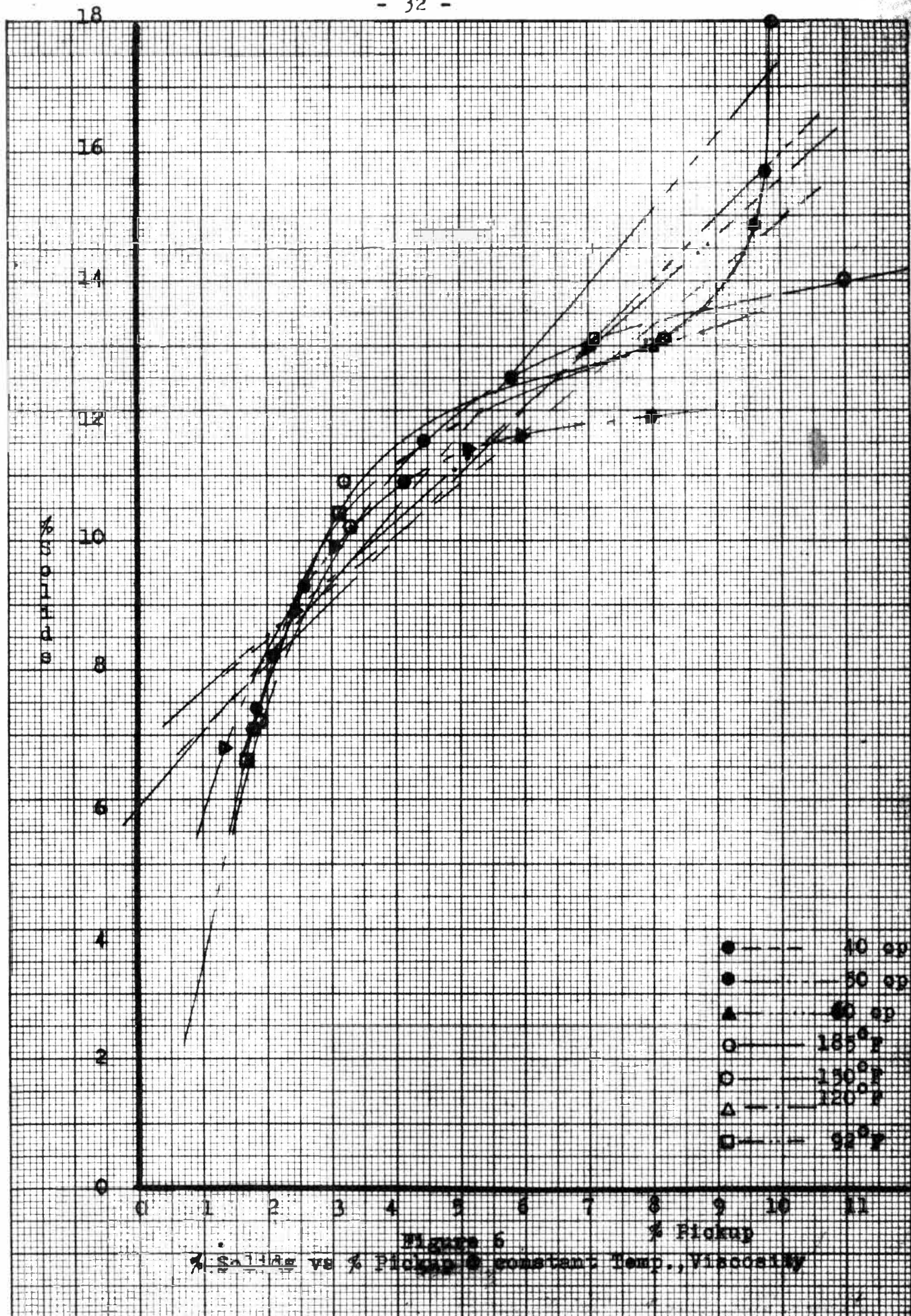
<u>TEST</u>	<u>1-2% Pickup</u>	<u>3-6% Pickup</u>	<u>6-8% Pickup</u>
STRENGTH			
Mullen	120/92	150,92/120	92/others
Tensile	others/92	185,150/120	-----
Tear	120/185,150	120/185,150	92/150
TEA	185/others	185/120	92/150
Elongation	185/150	185/120	185/120
Wax	120/92	-----	-----
SURFACE			
K&N Ink Absorbance	185/92	185/120	150/120
Smoothness	185/150	92/120	150,120/185,92
STRUCTURE			
Stiffness	92/185	92/185	92/150
Air Permeability	185,150/120,92	185,120	185,150/92

increasing in viscosity. The 150⁰ F and 120⁰ F starches follow about the same slope of solids increase over the viscosity increase. The incremental increase in solids produces more of an increase in viscosity. The 92⁰ F starch has a nearly linear relationship with increasing solids versus increasing viscosity. From here then viscosity can be held lower with increasing solids by increasing the temperature.

Figure 6 shows solids versus pickup at constant temperature and constant viscosity. From here it is apparent that a sharp increase in % solids will not give a large increase in pickup at any temperature until the 10-11% range of solids is reached. From here small increases in % solids will sharply increase the pickup in all temperature ranges, with the largest effect on the 120⁰ F starch. At constant viscosity solids can be doubled by doubling temperature, and pickup will increase about four times its previous value.

Figure 7 is an operating curve for Penford Gum 230. From this plot it is possible to establish a desired pickup and then choose a set of operating conditions which will give this pickup. For example, for a 3% pickup, 185⁰ F could be applied at 8% solids and a viscosity of about 25 cp. At 150⁰ a 9% solids would be needed, and the viscosity would be about 32 cp. 10% solids would be needed with a viscosity of 42 cp for operating conditions at 120⁰ F. At 92⁰ F the viscosity would be about 84 cp, and something over 10% solids would have to be used.





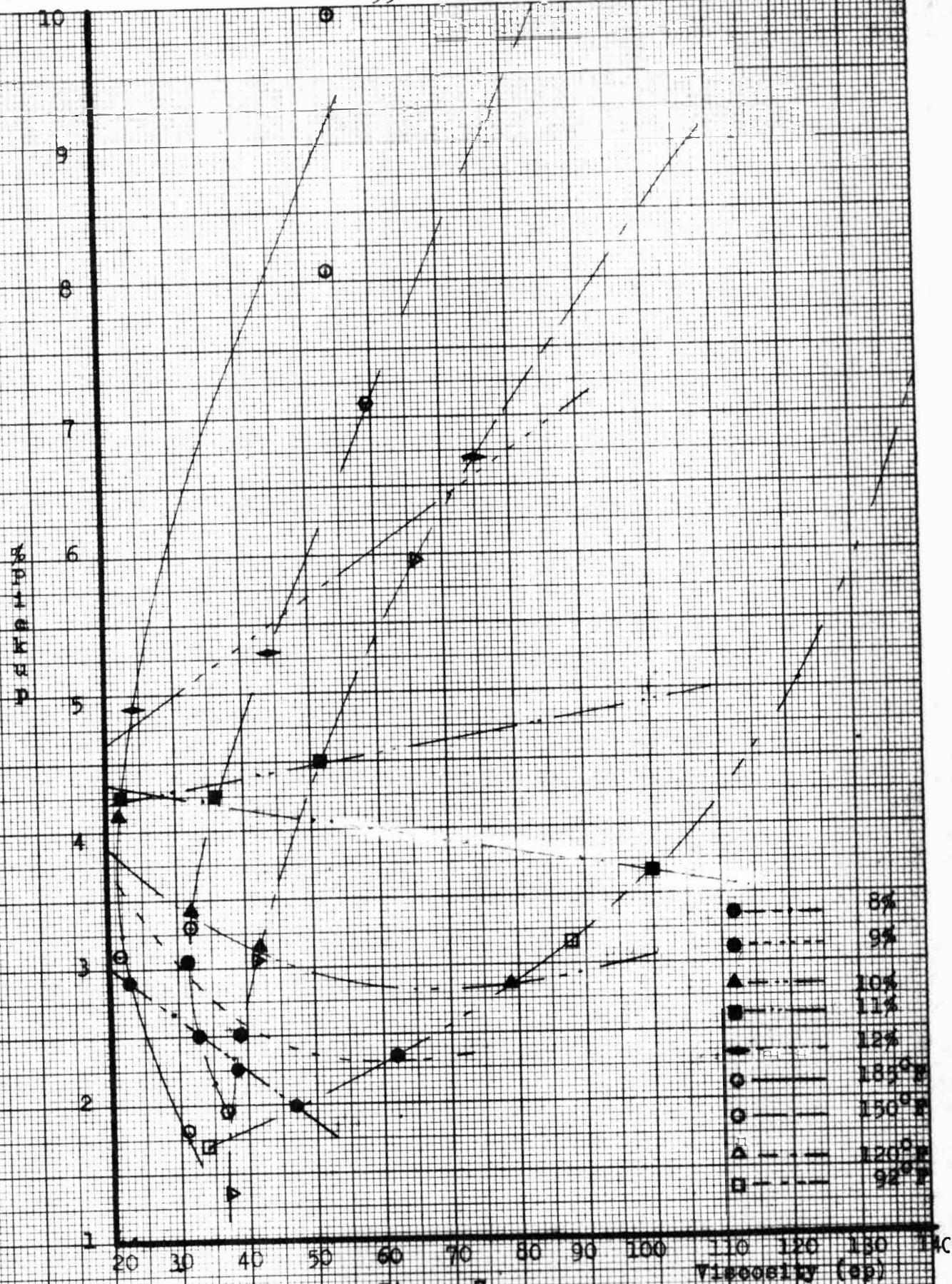


Figure 7

% Pickup vs Viscosity @ constant Temp. Solids

These three figures show that there is an intimate relationship between viscosity, % solids, temperature, and the % pickup which is inseparable. According to Figure 6, below the 10-11% solids range, neither solids nor temperature have a very great effect on pickup because of changing viscosity. At a constant viscosity, however, pickup can be increased almost linearly with simultaneous temperature and solids increases. Viscosity, therefore, has no influence of its own on the amount of pickup, but the solids and temperature have a significant combined relationship to pickup.

PICKUP, PENETRATION, TEMPERATURES, SOLIDS RELATIONSHIP

Figure 8 shows that below 10% solids and above 11% solids, the amount of pickup will vary directly with the depth of penetration. As the depth of penetration increases, the % pickup increases. In the 10-11% solids range, increasing the depth of penetration has little effect on the % pickup. This is the range from Figure 6 that showed a sharp increase in pickup for a small increase in solids. At this range also, from Figure 5, the viscosity began to increase rapidly. For this range then, it appears that pickup increases as the penetration increases, due to an increase in viscosity. Above the 11% range, % pickup increases rapidly in relation to depth of penetration, with rapid increases in viscosity. This starch seems to undergo some type of transition state in the 10-11% range which alters the relationship of pickup penetration, and viscosity.

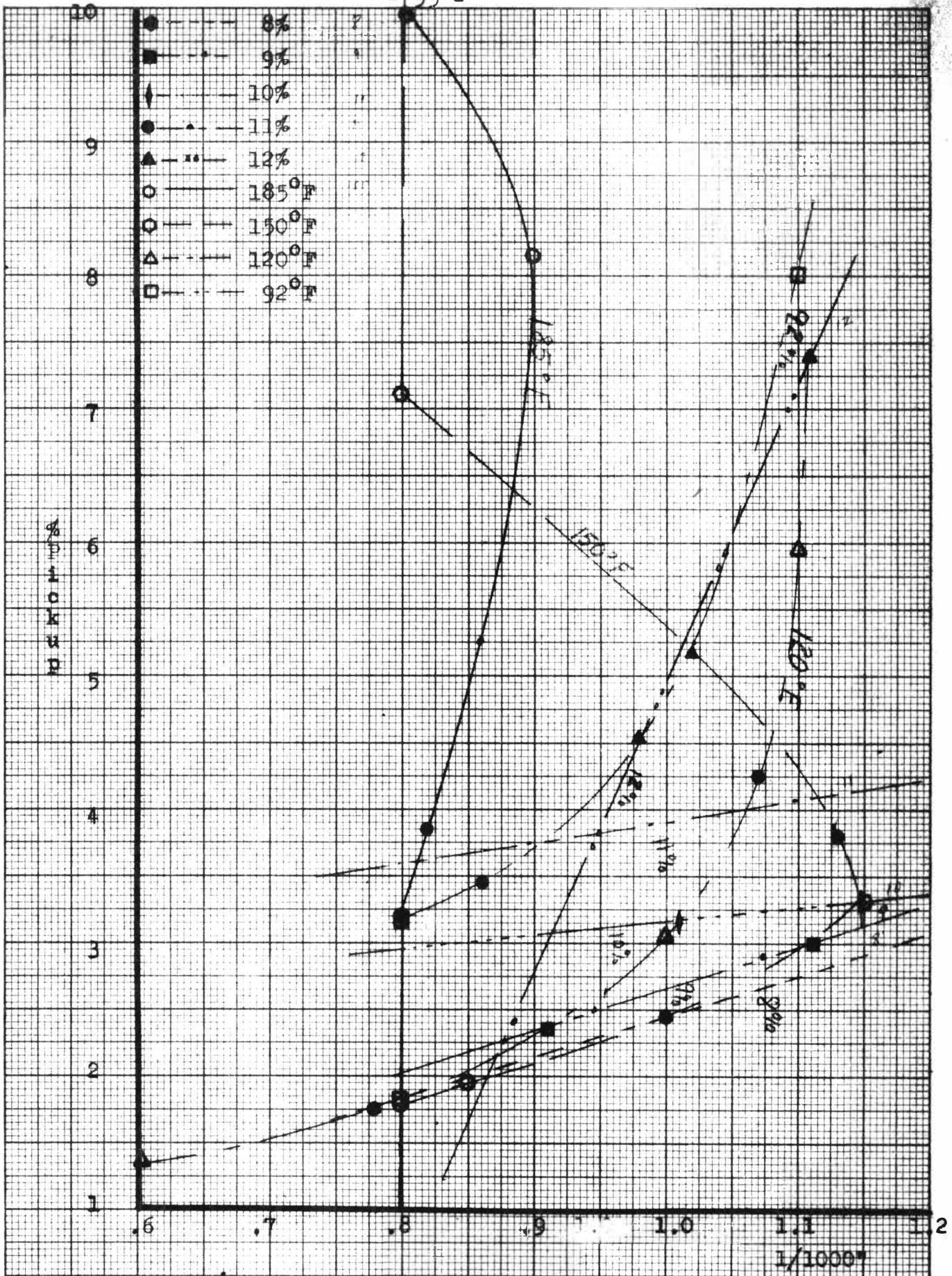


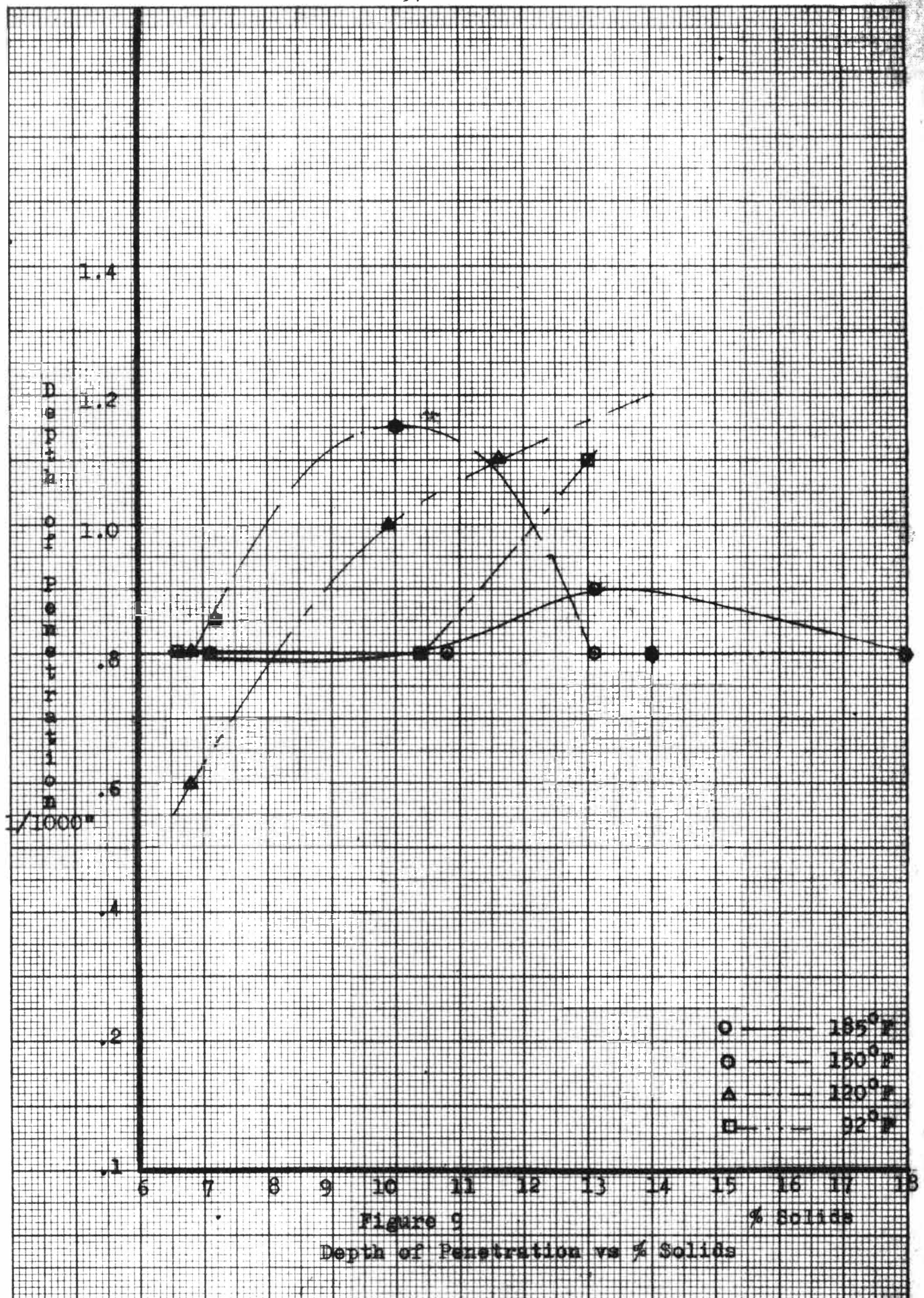
Figure 3
% Pickup vs Depth of Penetration @ const. Temp., Solids

Figure 9 shows that temperature has a marked effect on penetration at a constant solids. The depth of penetration reaches a peak in a different solids range for each temperature. This graph then tells that the depth of penetration will vary according to solids level of each different temperature.

Figure 10 shows the relationship of solids and pickup at constant temperature and depth of penetration. This indicates that small changes in pickup at constant depth of penetration requires changes in % solids. The relationship between % solids and temperature is so dependent that neither can be isolated as the most influential on the depth of penetration.

BURST

For the low pickup range (1-2%), from Figure 11, all values were close except the 120⁰ F application, which gave the most improvement. The starch for this sample was found closer to the surface than the other samples. It could be assumed that the starch nearer the surface will improve burst more rapidly than penetrated starch. This theory is supported by the 185⁰ F application, which after its increase in penetration and return towards the surface, it regains the initial slope increase. The 150⁰ F starch reacted completely differently as the starch penetrated rapidly, but the strength improvement was the same as for the 185⁰ and 92⁰ F applications. The 92⁰ F starch maintained the most constant increase in burst strength above the 3% range. The penetration at this point began to increase gradually which caused the change in slope, possibly in proportion to the amount of increased depth



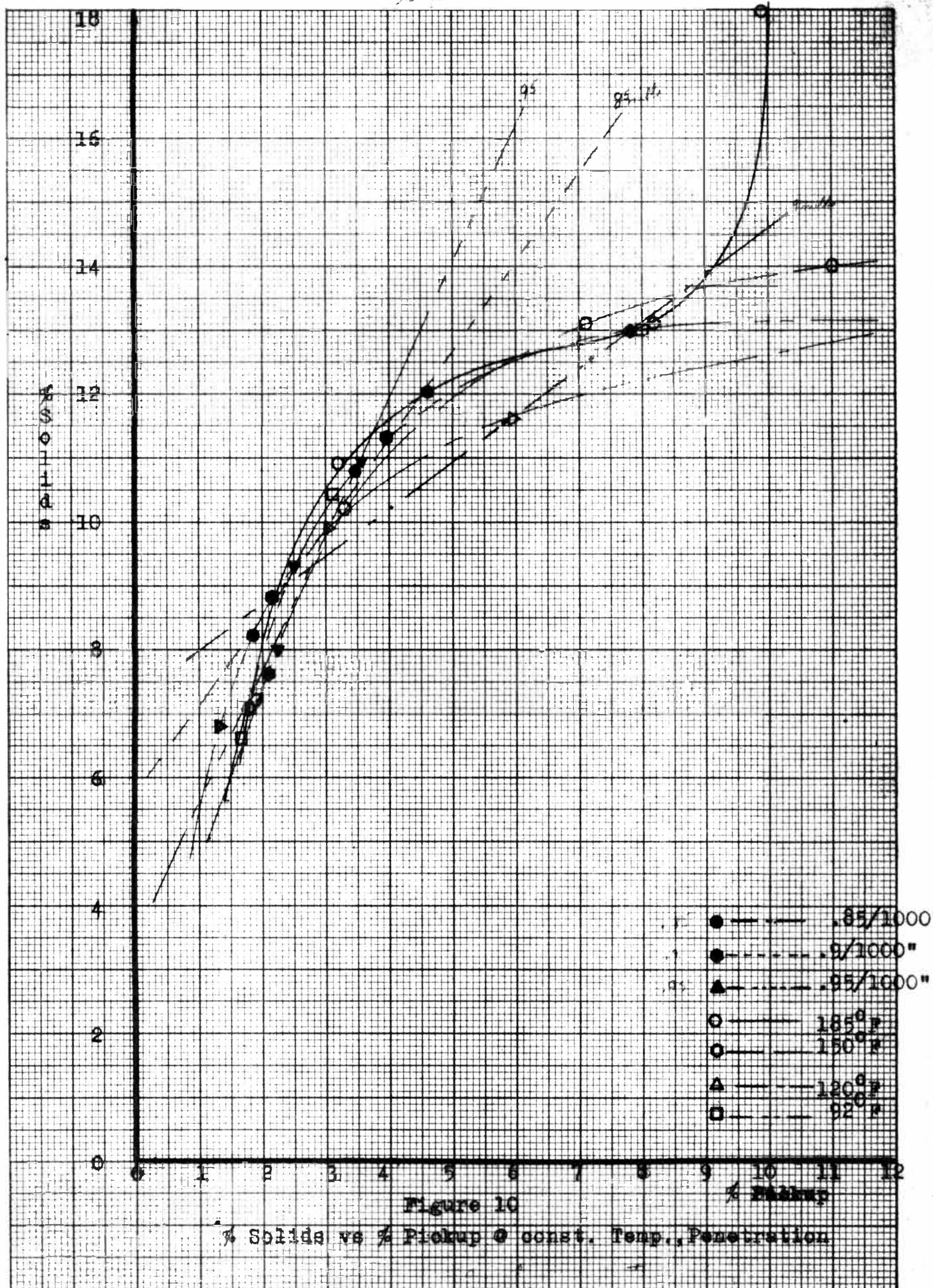
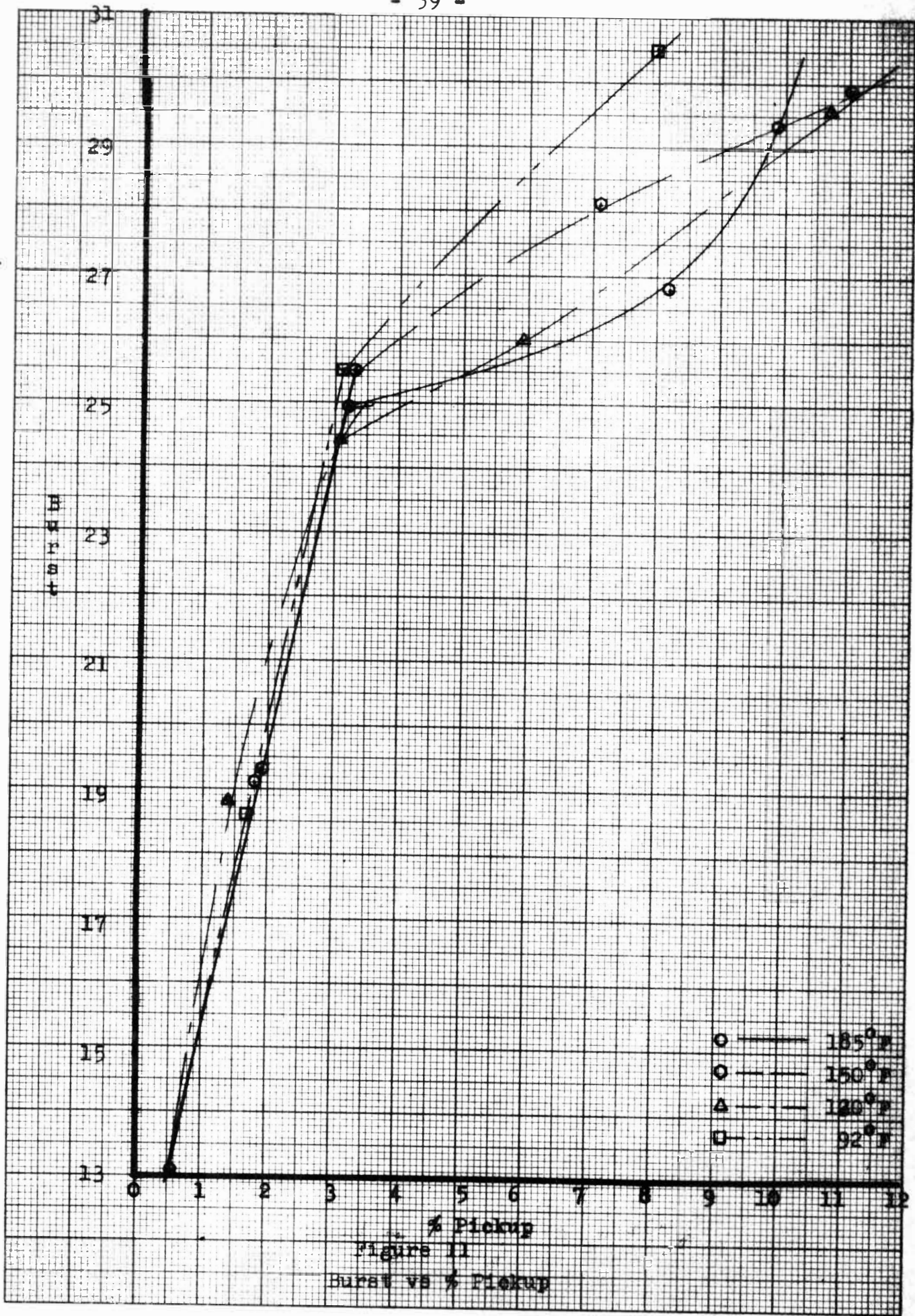


Figure 10
% Solids vs % Pickup @ const. Temp., Penetration



of penetration. There is a linear increase in burst strength to the 3% range which seems to be optimum for strength improvement per pound of pickup. It could be concluded that starch closer to the surface gives the best improvement in burst strength.

TENSILE

For the tensile in the low pickup range (1-2%), from Figure 12, the 120⁰ F starch gave the best improvement, as it did in the case of burst. As the starch began to penetrate more, its strength improvement began to increase less rapidly. In the 3% range, the 135⁰ starch has a far superior strength than the 92⁰ starch. Both of these starches were at the same depth of penetration, which tends to discount the surface starch theory. The best improvements are seen in the 3% range, again on a per pound of pickup basis. For tensile strength improvement, it would be best to aim for 3% pickup and apply the starch at 135⁰ F.

TEAR

Figure 13 shows that any application of starch will decrease the tear strength. This is probably due to the fact that increased bonding will decrease the tear strength. It may also be related to the stiffness of the sheet. Some feel that the stiffer the sheet is, the lower the tear strength will be. This factor does not appear to be more influential than the bonding factor. This is seen in Figure 14 as the stiffest sheet, the 92⁰ F sheet, does not have the lowest tear strength in the 3% pickup range. From Figure 13 it is seen

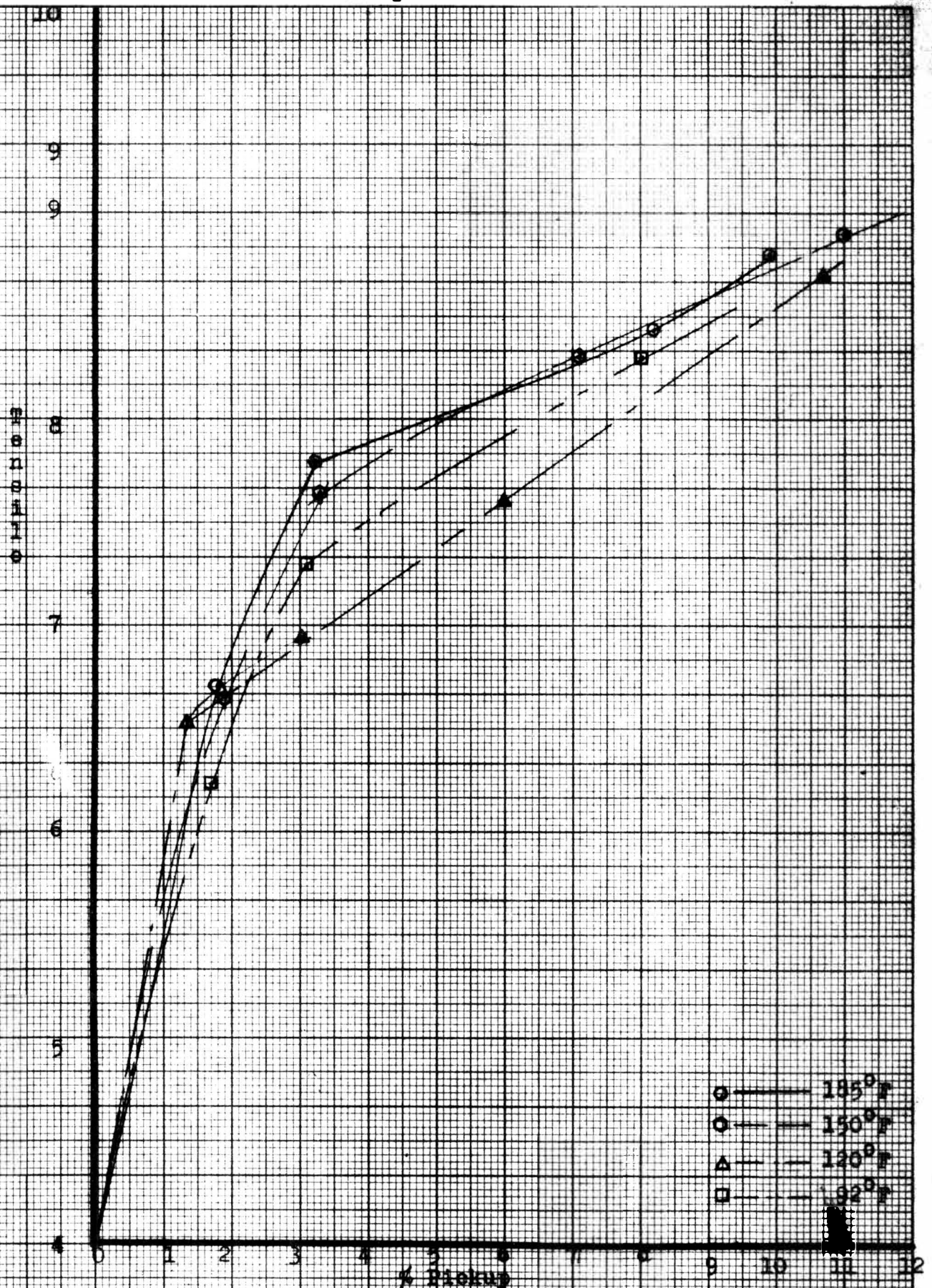


Figure 12
Tensile vs % Pickup

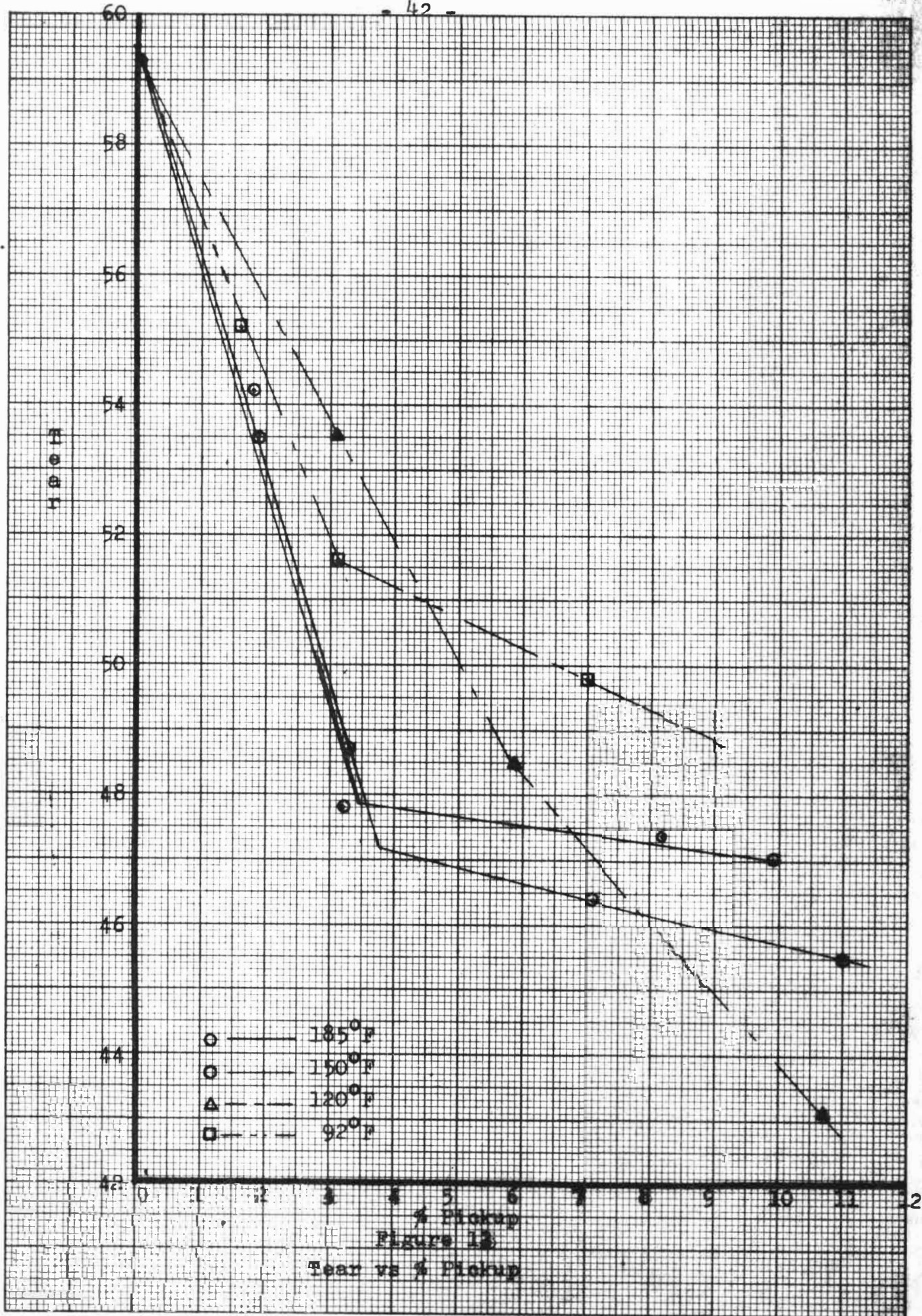


Figure 13

Tear vs % Pickup

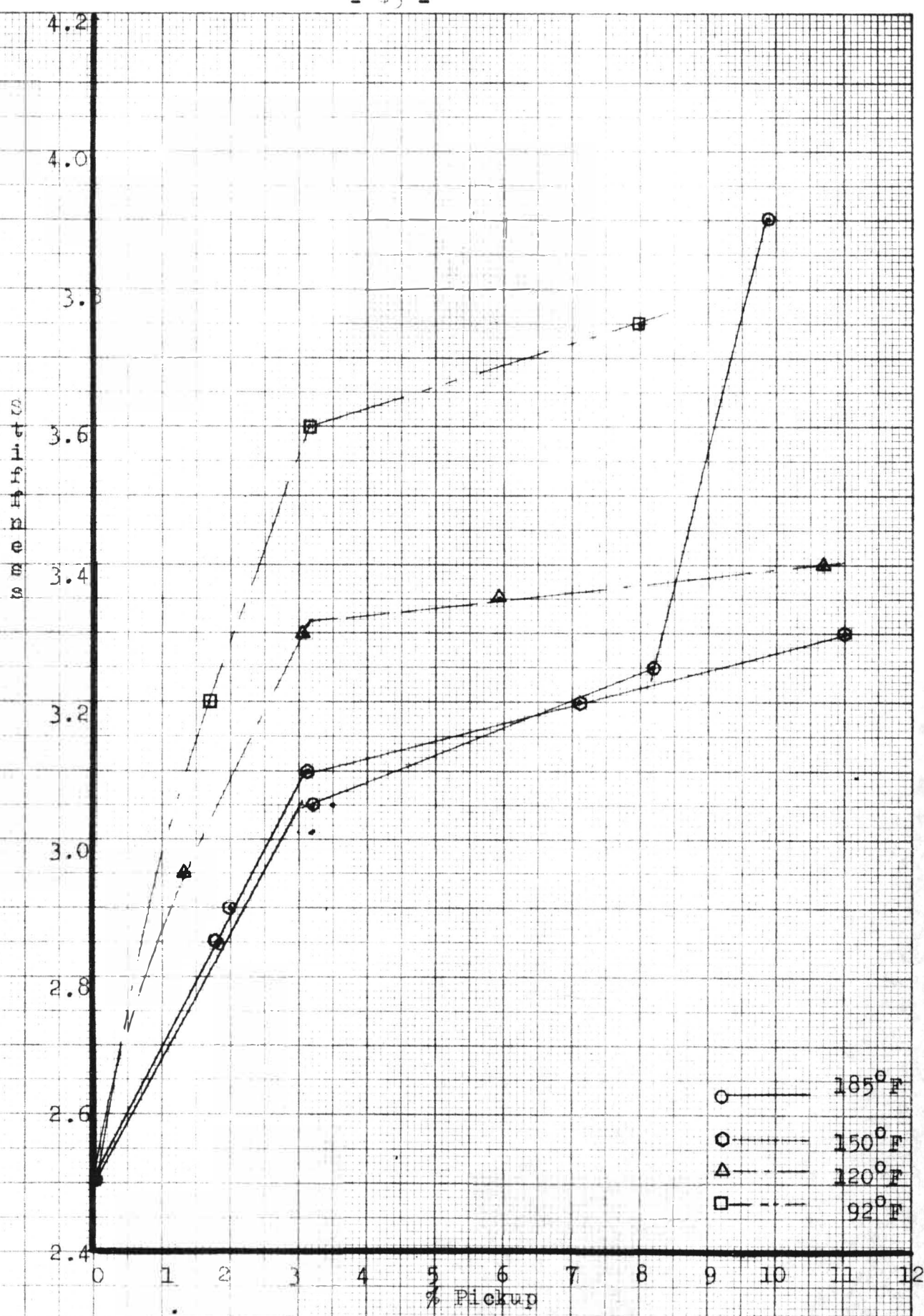


Figure 14
Stiffness vs % Pickup

that starch application at 120⁰ F is best for least strength loss in the 3% range. Because strength is continually lost from starch application, the less starch applied is better. It is necessary to apply starch for its other benefits; and therefore, it should be applied at 120⁰ F in the 3% pickup range.

STIFFNESS

Figure 14 shows that the 92⁰ F starch application is best for the most rapid increase in stiffness up to the 3% range. The 185⁰ F starch, which has the same penetration as the 92⁰ F starch, has the lowest increase in stiffness in the 3% range. The stiffness apparently is not related to penetration in this range. For the most part, again the stiffness increase is best up to the 3% pickup range. In this range, the 92⁰ F starch is the best to use. It is seen that past the 3% pickup range for the 185⁰ F starch, there is a marked increase in strength almost equal to the increase in the 3% range. What this means I do not know.

TEA

From Figure 15, the most rapid increase is obtained by the 185⁰ starch application in the 3% range. These results parallel exactly the tensile strength results. For starch application at 185⁰ F and in the 3% pickup range, the most rapid increase in strength can be found. It is therefore best to use this range and temperature for optimum strength improvement.

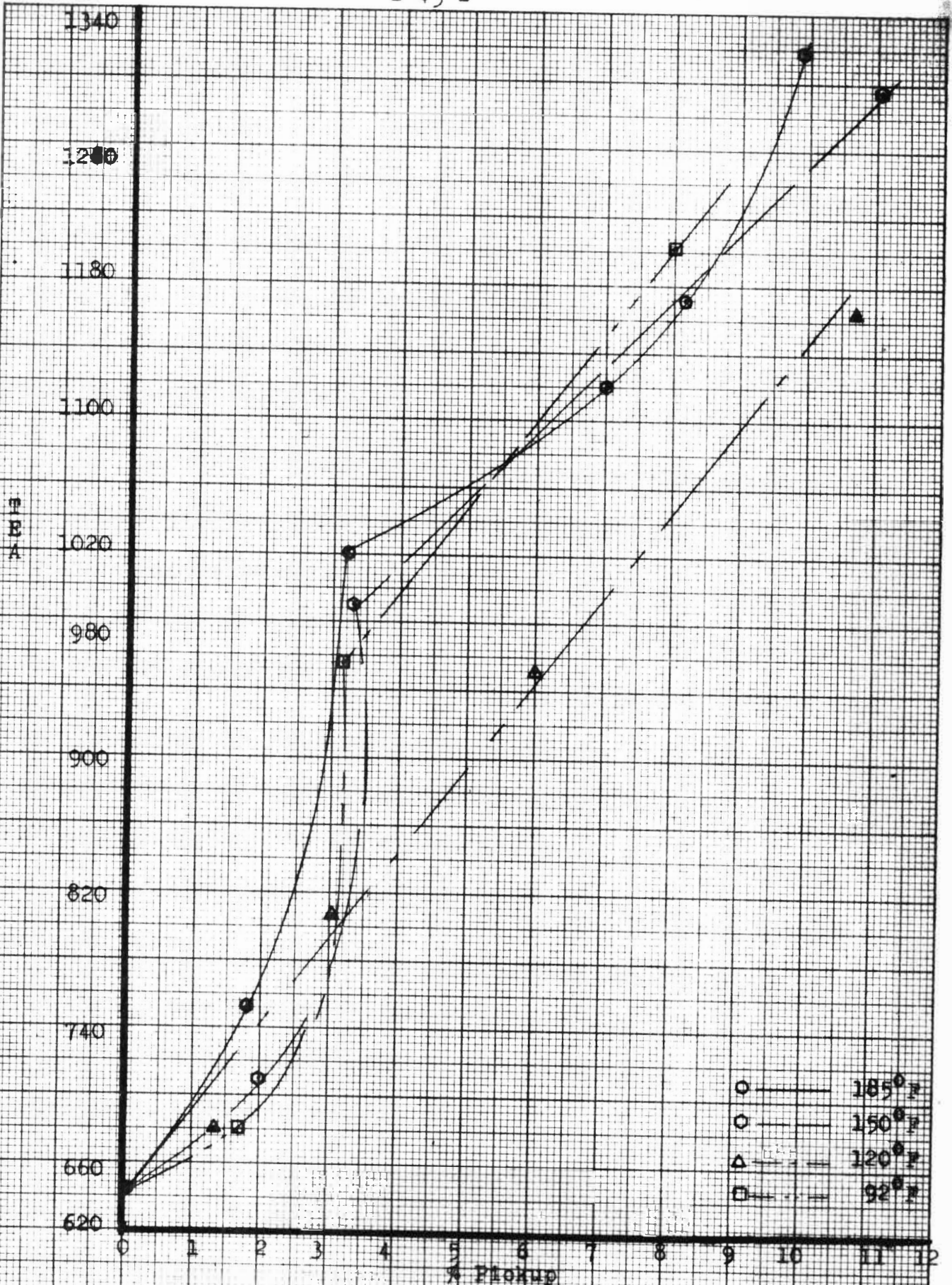


Figure 13
TEA vs % Pickup

ELONGATION

The $\frac{1}{2}$ elongation from Figure 16 is almost an exact replica of the tensile plot. In fact, placing one over the other would almost cause the second one to vanish. It does not appear that any correlation can be found between $\frac{1}{2}$ elongation and the depth of penetration. It can again be said that the 3% pickup range gives the maximum increase in elongation using starch at 135⁰ F.

WAX

This property is actually a strength and surface property and is shown in Figure 17. It is seen here that applying starch at 120⁰ F will most rapidly improve this test. A maximum wax was found for this starch of 18, reached by the 3% range for all samples. It appears that there would be no benefit from adding more starch to the sheet for this property. The 3% pickup range and any starch application will give optimum results.

K&N INK ABSORBANCE

Figure 18 indicates that the more penetrated starch, in this case the 150⁰ starch, will give the best holdout in ranges of 1-2% and above 6% pickup. This indicates that the starch distribution is more important than starch lumped in one area of the sheet for holding out ink. For best results in ink holdout, it would be most advantageous to apply starch at 150⁰, and a per point improvement would be maximized in the 3% pickup range.

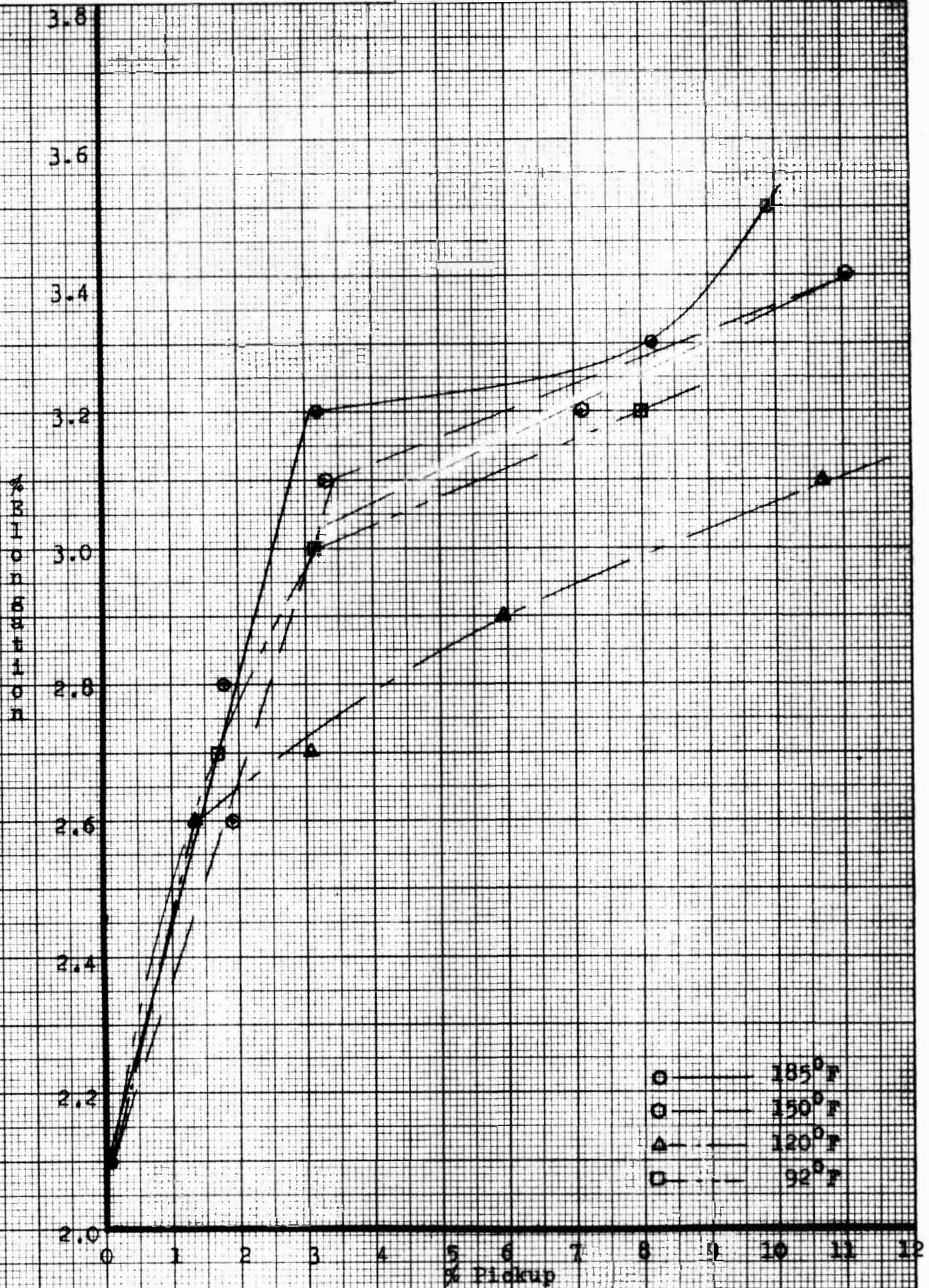
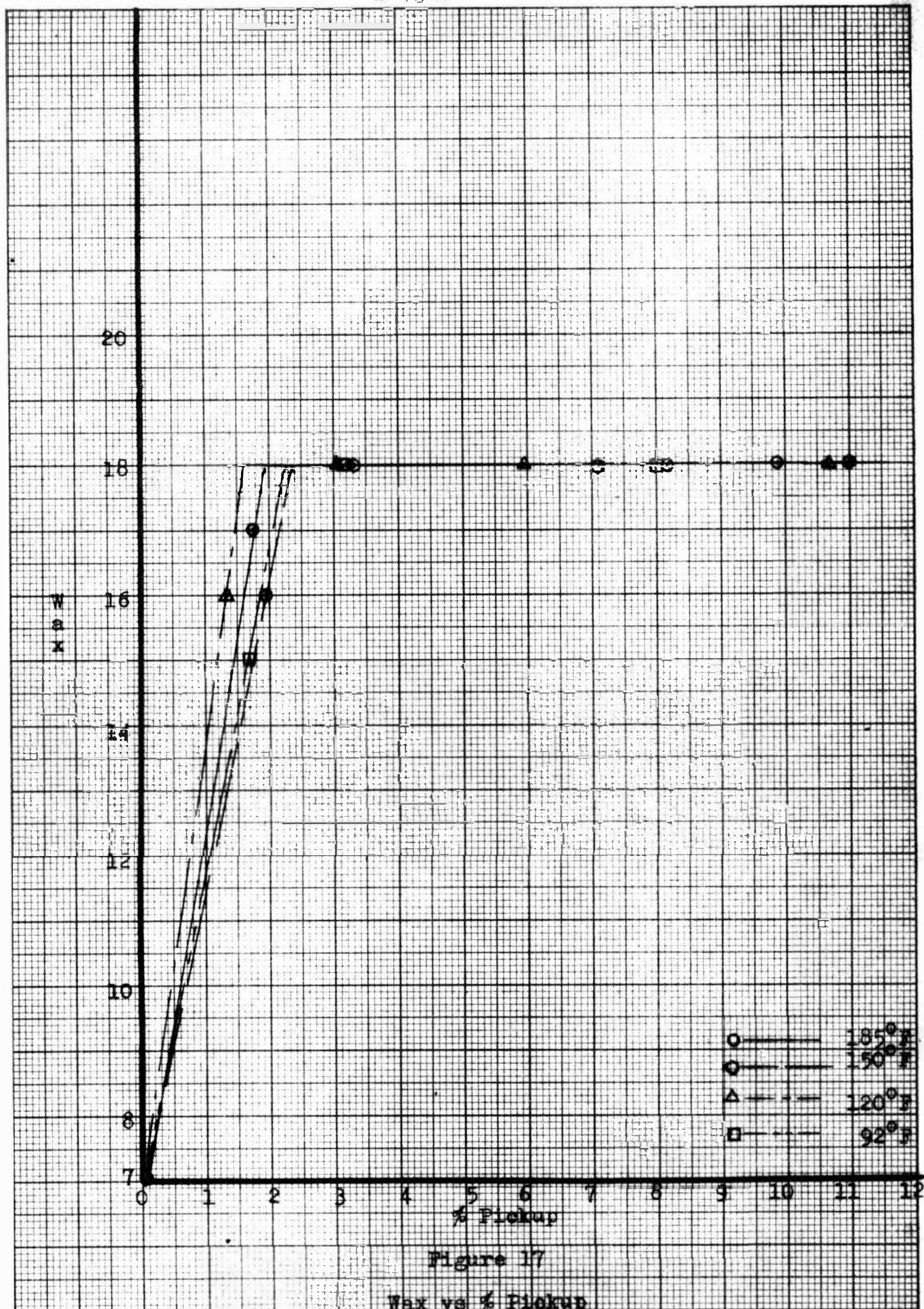
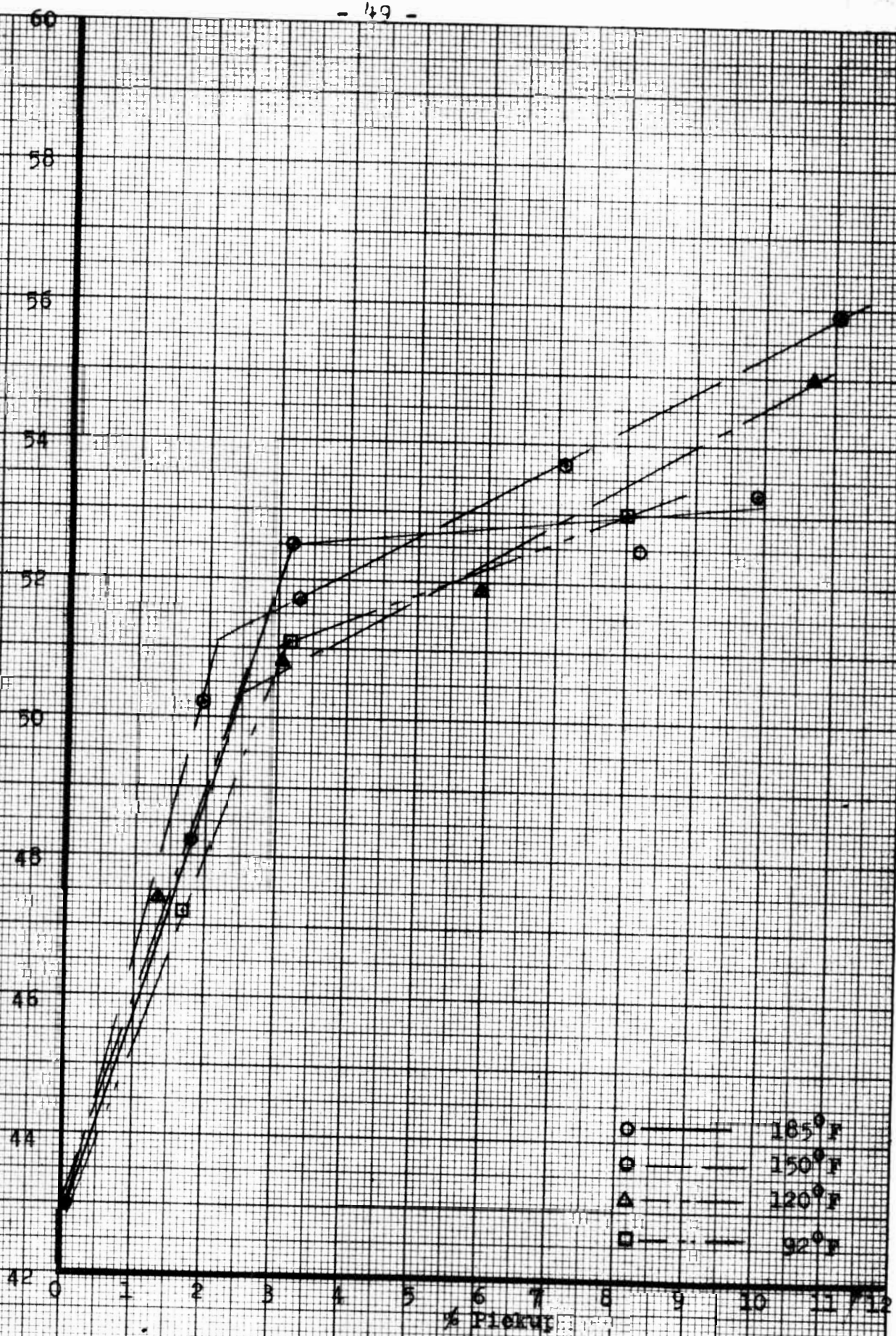


Figure 16

% Elongation vs % Pickup



K & N
I n k
A b s o r b a n c e



K&N Ink Absorbance vs % Pickup

Figure 18

SMOOTHNESS

The lowest values represent the smoothest sheets, from Figure 19. The smoothness decreases rapidly for all cases, but the 150⁰ starch application has the least loss in smoothness after the 3% pickup range. The 185⁰ or 92⁰ starch has the lowest decrease in smoothness up to this point. Penetration can not really be related to smoothness, probably because of the initial depth of penetration of the samples. For optimum smoothness, use as little starch as possible; but if using starch in the 3% pickup range, apply it at either 92⁰ or 185⁰ F.

AIR PERMEABILITY

For permeability, the lowest values are the best, as seen in Figure 20. There is a strong improvement in lowering permeability in the 185⁰ and 150⁰ F starches in the first 3% pickup ranges. These taper off in the 3-6% ranges, but then decline rapidly again. In the 3% ranges, the 185⁰ and 150⁰ represent opposites in depth of penetration, the 185⁰ being least penetrated and 150⁰ most penetrated. There may be a more uniform distribution in these areas which is causing these to be less permeable than the 92⁰ or the 120⁰ starches. For maximum reduction in air permeability, either 185⁰ or 150⁰ starch should be applied in the 3% pickup range.

Throughout all these tests the 3% pickup range was found to be optimum for rates of improvement. More improvement could be obtained, but at a sharp increase in costs. The optimum temperature was indicated each time for the test. From these values then, one could refer to Figure 7 and see the needed

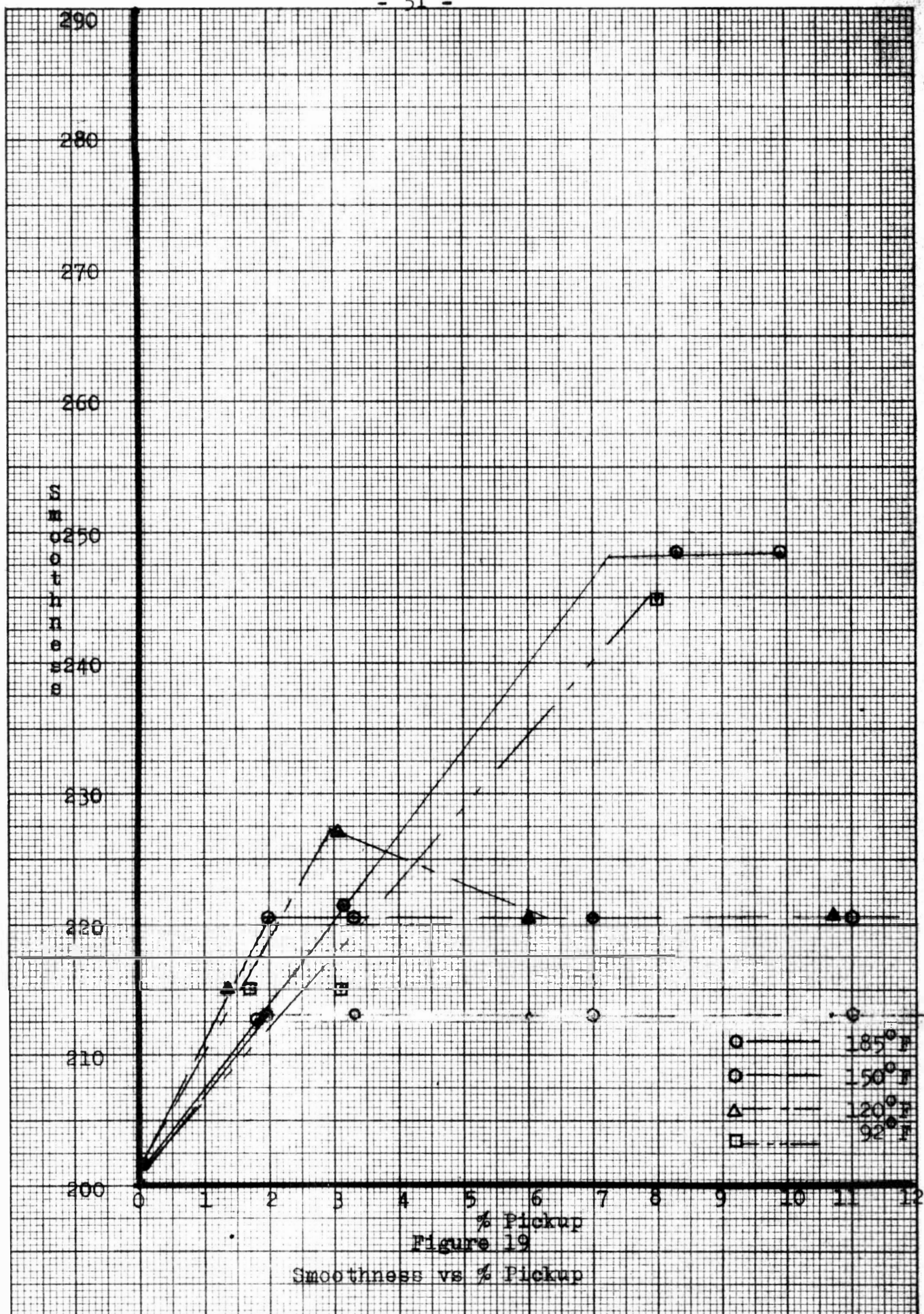


Figure 19

Smoothness vs % Pickup

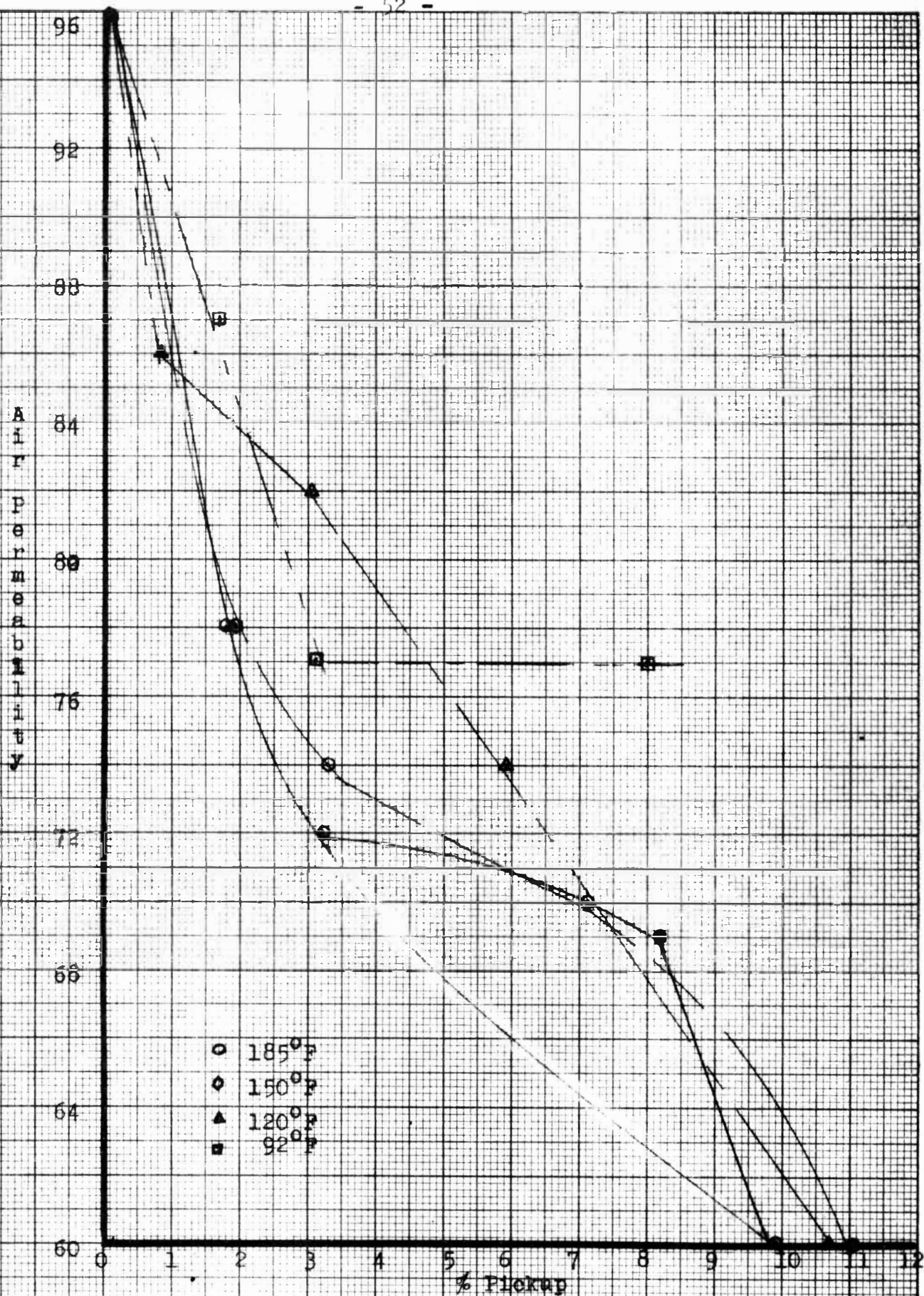


Figure 20

Air Permeability vs % Pickup

solids to run at to get these sheet properties. The properties and temperatures are summarized below.

SUMMARY

1) Burst strength is improved most by the starch closer to the surface, and the most rapid improvement comes up to the 3% pickup range at 92⁰.

2) Tensile does not support the surface theory, but does support the 3% pickup range. It also shows the 135⁰ F temperature to be best for strength improvement in this range.

3) Tear strength does not improve with starch application. It is necessary to improve other strengths by application of starch; and therefore, the 120⁰ F starch is recommended in the 3% pickup range because it gives the least loss in strength.

4) Stiffness is increased most by 92⁰ F starch application in the 3% pickup range.

5) TEA parallels the results of tensile, and the 135⁰ F application at the 3% pickup range is best.

6) Per cent elongation is a duplication of the tensile and TEA results. This means that 135⁰ and 3% pickup is most desirable for improvement of this test.

7) Wax is best improved by a 3% pickup at any temperature application.

8) The 150⁰ F application of starch is best for improving ink holdout in most pickup ranges.

9) Applying starch at 135⁰ or 92⁰ and keeping it below the 3% pickup range will minimize smoothness loss due to starch application.

10) The optimum temperatures to apply starch at in the 3% pickup range for best reduction in permeability are 185⁰ and 150⁰ F.

CONCLUSIONS

Viscosity has no influence of its own on the amount of pickup, but the solids and temperature have a significant, inseparable relationship on the amount of pickup.

There is a transition state in the 10-11% solids range for this starch which alters the pickup, penetration, and viscosity relationship. The depth of penetration is also inseparable from the solids-temperature relationship.

Each sheet property has a maximum rate of improvement up to the 3% pickup range for this type paper and this starch. The most beneficial temperature has been reported in the summary, and the necessary solids can be read from Figure 7 to give these results.

It can not be proven from this study whether the depth of penetration has any strong influence on sheet properties or not. From the results herein it appears that penetration has little effect on most properties. It depends on one's definition of "surface" starch. For the most part, the penetration started at .8/1000 inches, so can this be considered surface starch?

SUGGESTIONS FOR FURTHER STUDY

Make a study similar to this for other types of starch to see if there is a transition state in all starches or just this one.

Take paper with the base qualities discussed in this paper and coat them to study how surface sizing influences coating.

APPENDIX

TABLE VIII
VALUES OF POINTS ON GRAPHS

<u>CCODE</u>	<u>BASIC WEIGHT</u>	<u>PERMEABILITY</u>	<u>WAX</u>	<u>BURST</u>	<u>MOOETHNESS</u>	<u>TENSILE</u>	<u>ELONGATION</u>
AW	50.25	60	18	29.3	246	8.8	3.5
BW	46.53	69	18	26.8	247	8.4	3.3
CW	45.32	72	18	24.9	223	7.8	3.2
DW	44.48	78	17	19.1	215	6.7	2.8
AX	48.39	60	18	29.9	221	8.6	3.4
BY	46.31	70	18	28.1	221	8.3	3.2
CX	45.15	74	18	25.5	223	7.6	3.1
DX	45.24	78	16	19.3	221	6.6	2.6
AY	47.64	60	18	29.6	221	8.7	3.1
BY	46.55	74	18	26.0	221	7.6	2.9
CY	46.76	82	18.	24.4	237	6.9	2.7
DY	44.74	86	16	18.8	215	6.5	2.6
BZ	49.59	77	18	30.5	245	8.3	3.2
CZ	48.75	76	18	25.5	215	7.3	3.0
DZ	46.21	87	15	18.6	215	6.2	2.7
PM	45.43	95	7	9.8	203	3.8	2.2
STD							
AM	47.08	83	7	8.9	197	4.0	2.2
STD							

<u>TEA</u>	<u>TEAR</u>	<u>STIFFNESS</u>	<u>K&NINK HOLDOUT</u>	<u>PICKUP</u>	<u>VISCOSITY</u>	<u>%SOLIDS</u>	<u>DEPTH OF PENETRATION</u>
1318	47	3.9	53.3	9.94	55	18.0	.8/1000
1171	47.4	3.25	52.4	8.17	54	13.1	.9/1000
1020	47.8	3.05	52.5	3.21	21.6	10.8	.8/1000
754	54.2	2.9	48.2	1.78	31.3	7.1	.8/1000
1297	45.5	3.3	55.9	11.05	90	14.0	.8/1000
1120	46.4	3.2	53.7	7.10	59.5	13.1	.8/1000
991	48.7	3.1	51.7	3.30	32	10.2	1.15/1000
710	53.5	2.9	50.2	1.96	37.2	7.2	.85/1000
1163	43.1	3.4	55	10.67			
953	48.5	3.35	51.9	5.95	66.2	11.6	1.10/1000
806	53.5	3.3	50.8	3.03	41.3	9.9	1.00/1000
678	53.1	2.95	47.4	1.36	37.0	6.8	.6/1000
1202	49.8	3.75	53	8	146	13.0	1.10/1000
957	51.6	3.6	51.1	3.14	88.4	10.4	.8/1000
681	55.2	3.2	47.2	1.69	34.2	6.6	.8/1000
665	59.3	2.55	42.8				
623	59.3	2.45	43.2				

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