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Doctor's Dissertation

The Sulphite Pulping of Douglas Fir

by Earl Bruce Brookbank, Jr.

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THE SULPHITE PULPING OF DOUGLAS FIR

A thesis submitted by

Earl Bruce Brookbank, Jr.,

B. S. in Ch. E., University of Washington, 1933,
M. S., Lawrence College, 1936

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INTRODUCTION

The pulping of Douglas fir (Pseudotsuga taxifolia Britt.) has been of interest to many investigators, one of the principal reasons for this being the tremendous amounts of this wood which occur along the Pacific coast in British Columbia, Washington, Oregon, Idaho, and northern California. At present the lumber industry is the sole consumer of this species, and because of the large size of the trees, much wood is lost as slabs, which are either burned or put through expensive cutting operations in making such articles as laths and broom handles. Greeley (1) in 1928 estimated the amount of slab wood available for pulping from this region as 4,000,000 units of chips (1 unit=200 cu. ft.) or 2,000,000 tons of pulp. Of this quantity, approximately five-sixths is Douglas fir. The supply is plentiful and will remain so for many years to come.

There are two types or varieties of Douglas fir, the coast type and the mountain type. The latter variety is of little interest at present from the pulping point of view, for its habitat is not accessible to pulp and paper mills, while the coast variety grows in extensive stands near the established mills of the Northwest.*

Young Douglas fir trees grow rapidly until competition with others in the stand for soil moisture and sunlight slows the growth rate considerably. Technical Note No. 218 of the Forest Products Laboratory at

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*Hereafter, the term Douglas fir will refer only to the coast type of wood.

Madison, Wisconsin, (2) gives the following figures for the density of this variety of fir:

Density....................34 lb. A.D./A.D. cu. ft.

Specific Gravity...........0.45 (O.D. Wt./green vol.)

These figures are averages for the whole tree. There are large variations in both density and growth rate, depending on the location of the sample with respect to the axis of the log. For example, the Douglas fir log used as raw material in the present work had a growth rate of 7 rings per inch at the center and 45 rings per inch at the periphery. The average density of the heartwood was 32.2 pounds per cubic foot, while the density of the sapwood was 47.0 pounds per cubic foot (Oven-dry weight and volume). These variations are to be expected in trees of this size, many of which are over 60 inches in diameter and 300, or more, feet tall.

Very little information is available as to the proportion of sapwood in Douglas fir. Hayward (3) states that there may be as much as four inches of sapwood in a 48-inch diameter log, corresponding to 31 per cent by volume. The specimen used in this work contained only 15 per cent sapwood by volume in a 27-inch log. The estimated amount of sapwood in slabs from the ordinary sawmill operation is 90 to 95 per cent by volume, corresponding to approximately 98 per cent by weight. Thus it may be seen that any pulping operation based on utilizing sawmill waste would be concerned principally with the sapwood of the tree.

Although it is a generally accepted fact that cooking Douglas fir with a calcium base sulphite liquor leads to an unsatisfactory pulp with regard to both yield and quality, very few references to laboratory or semi-commercial scale work on this point have been found in the literature. Wells and Rue (4) state that "...(Douglas fir) reduces with difficulty on account of the pitchy character of the wood." They report the following estimates of mill scale results when pulping this wood: "Yield, 45-50%; bleach requirement, 20 to 25%, with unbleached pulp of fair strength and poor color; probably somewhat pitchy."

English, Green, Mitchell, and Yorston (5) have reported on the rate of pulping of alcohol-benzene extracted Douglas fir sawdust, using calcium base liquor, but they consider their data only in its relation to an understanding of the physico-chemical factors which affect sulphite pulping in general. After seven hours' cooking, without digester relief, at 130°C., and using acid analyzing 5.59 per cent total and 0.80 per cent combined sulphur dioxide, a pulp containing 6.21 per cent lignin and 0.428 per cent sulphur was obtained.

Beuschlein (6) compared the pulping rate of alcohol-benzene extracted Douglas fir sawdust with that of white spruce, using soda base liquors. At 150°C. and using a liquor containing 4.00 per cent total and 1.00 per cent combined sulphur dioxide, a Douglas fir pulp which contained 22.9 per cent of non-cellulosic material was obtained. Under identical conditions, white spruce yielded a pulp containing only 3.8 per cent of non-cellulosic matter. The spruce cellulose yield was approximately 1 per cent higher than that from Douglas fir. Apparently it is not the extractives which make the pulping of Douglas fir difficult. The only other alternative is that there exist fundamental differences in the chemical or physical structure of the lignin. Working along similar lines, Bailey (7) investigated the ligninsulphonic acid from Douglas fir and concluded that lignin from this tree consists of four trimeric coniferyl aldehyde units, while spruce lignin is composed of three. This work was based on the theories of lignin structure advanced by Klason (8).

Benson, Erwin, Hendrickson, and Tershin (9) pulped young Douglas fir (3-inch diameter logs), using ammonia base liquor, and obtained a satisfactory pulp, similar in properities to pulp from spruce and hemlock. Old wood gave negative results, but old wood extracted with ammonia pulped satisfactorily. These experimental data are of limited value, as several independent variables were changed at one time, thus making close comparisons difficult.

The action of ammonia base sulphite liquor on spruce wood has been found by Doreé and Barton-Wright (10) to yield the same ligninsulphonic acid as is obtained when calcium bisulphite is used, indicating that successful pulping with ammonia base liquors is dependent on the reaction between ammonium bisulphite and the extractives of the wood, rather than on differences in behavior of calcium and ammonia base liquors toward the lignin.

The work mentioned in the foregoing paragraphs has resulted in a number of suggested pretreatments for use prior to regular sulphite cooking in reducing not only Douglas fir but also other resinous woods to pulp suitable for paper making.

Benson (11) has patented an ammonia pretreatment for the removal of resins, coloring matter, and other non-volatile compounds from uncrushed chips. When chips so treated are cooked by the usual sulphite process, pulp of good quality and low bleach consumption is obtained.

Another process (12) is based on a preliminary treatment of resinous wood chips with hydroxides of divalent metals. This partially removes the resins and saturates the wood with the base for subsequent cooking. This first step is followed by a short treatment with sulphur dioxide or chlorine compounds and the chips are then subjected to the usual type of sulphite cook. By-products, such as acetic acid, high grade rosin, turpentine oil, and lignin powder, may be recovered if desired.

Other pretreatments are based on the extraction of the chips with milk of lime (13), with liquid sulphur dioxide (14), and with sodium acetate formed by the reaction of sodium hydroxide on the acetic acid present in the wood (15). Richardson and Sherman (16) pulped resinous woods by using liquors containing both ammonium and calcium bisulphite.

With the exception of Benson and his co-workers (9), none of the above mentioned investigators have specified the source, growth rate, density, or location in the tree from which their wood was obtained. Benson obtained good pulp with no pretreatment when wood from young

Douglas fir trees was used. Although no definite information is available on the point, it seems reasonable to assume that trees of the size used by Benson were composed entirely of sapwood. There are considerable data in the literature pointing out differences in composition, colloidal nature, and pulping properties between sapwood and heartwood. Herty (17) obtained sulphite pulp of good quality from young southern pines as long as no heartwood was present.

Cohen (18) found that the ether extractives of Pinus radiata were more concentrated in the heartwood than in the sapwood. Sulphite pulp from heartwood was lower in yield and higher in screenings than that from sapwood. The sapwood pulp had 0.35 per cent ether extractives, while the heartwood pulp contained 3.84 per cent. The former would probably cause little or no trouble on the paper machine, while the latter could not be used. Pienkowski and Jurkiewicz (19) used x-ray methods in investigating the resin content of wood, their findings being in good agreement with those of Cohen.

Schwalbe and Ekenstam (20) investigated the colloidal properties of the sapwood and heartwood of pine and spruce in an attempt to explain the differences in behavior on cooking noticed by earlier investigators. Water adsorption from air of controlled humidity and temperature was used as a measure of the relative swelling of the various kinds of wood. It was found that pine heartwood adsorbed less water than the other types of wood studied. Green (freshly cut) sapwood adsorbed more water than green heartwood. On drying, the ability of sapwood to adsorb water decreased, while that of heartwood increased due to splitting and checking during the drying, which opened up a greater surface for adsorption. Starting with oven-dry wood, the final equilibrium moisture content of the two varieties was the same. This indicates that there are irreversible changes which take place on drying wood, the sapwood undergoing a syneresis and approaching the properties of heartwood. Whether this artificial mechanism of irreversible desiccation is analogous to the changes naturally taking place in a growing tree is unimportant, as long as the nature of the changes occurring after the tree is cut are known and understood.

These workers found pine to behave similarly to spruce in the adsorption of liquid from bisulphite liquor in bomb tubes at 110 deg. C., but the pine heartwood adsorbed the liquor in different concentrations than the other woods. The amount of free sulphurous acid adsorbed by the pine heartwood was so small that any cooking operation would have to be considered as a neutral sulphite process. Bergström (15) supported this finding, stating that the combined sulphur dioxide penetrates the wood much more rapidly than free sulphurous acid. No similar work on Douglas fir could be found, but it is probable that analogous behavior would be observed in the sapwood and heartwood of this species.

OBJECT OF INVESTIGATION

Since there is an almost unlimited supply of Douglas fir sawmill waste, consisting principally of slab wood which is 85 to 90 per cent sapwood, any pulping operation utilising this waste would deal almost exclusively with sapwood. It is the purpose of this investigation to attempt the sulphite pulping of Douglas fir, using calcium base liquors and no pretreatment. In the light of the success which has attended the pulping of pine sapwood, it is believed that the pulping of Douglas fir slab wood is possible and commercially feasible. The pulping of heartwood and sapwood will be attempted separately with the object of evolving cooking conditions within the present commercial limits for the reduction of both slab wood and entire logs.

An investigation of the lignin of Douglas fir sapwood and heartwood will be carried out for comparison with similar lignin derivatives prepared from spruce wood. Any differences found in amount or kind of lignin, between the several types of wood, will be used in attempting to explain their differences in behavior on cooking.

An investigation of the kind, amount, and distribution of extractives in Douglas fir heartwood and sapwood will be made for the purpose mentioned, viz., to explain differences in behavior on cooking of the various types of wood.

RAW MATERIAL

The Douglas fir log used in this work was received from the Weyerhaeuser Timber Co., Longview, Washington, and was cut in the spring of 1937. This log was chosen as representing the average from the Pacific Coast Douglas fir region with respect to size and growth conditions. The log was painted on the ends before shipment to minimize moisture loss and aging in transit. The log was received at the Institute with the bark intact.

A density disk, approximately one inch in thickness, was cut from the log at a distance of four feet from one end. This disk was barked, the average diameter calculated from the circumference, and the number of growth rings counted, a separate count of the growth rings in the sapwood being made.

The following method was used in making density measurements: The density disk was weighed, dried to constant weight at 105°C. and cut into pieces of a size convenient for use in the subsequent operations. The dry pieces were dipped in molten paraffin. After cooling, their volume was determined by immersion in water, the volume of water displaced being measured. In the case of Douglas fir, the density of the sapwood was determined by cutting away all heartwood and making separate volume and weight measurements on the sapwood pieces so obtained. From this data, the moisture content of the wood before chipping, and the growth rate and the density of both the heartwood and sapwood were calculated, and from the relative amounts of sapwood and heartwood in the log, the same values for the entire tree were calculated.

Similar measurements were made on the spruce and western hemlock logs used in making comparison pulps. The following results were obtained:

TABLE I

DENSITY, GROWTH RATE, AND MOISTURE CONTENT OF PULP WOOD LOGS

	Spruce		Douglas Fir		Western Hemlock
	No. 1	No. 2	Sapwood	Heartwood	
Moisture Content, %	36.9	40.2	29.4	21.3	36.1
Average Diam., in.	6.8	6.4	2.6	24.6	21.2
Growth Rate, rings per in.	19.4	10.3	36.0	17.0	23.0
Specific Gravity			0.76	0.48	0.47
Density (O.D. wt. and vol.) lb./ft.^{3}	32.6	30.4	47.0	30.0	29.1
Per cent of Whole Tree					
by Volume			14	86	
by Weight			20	80	

Wells and Rue (4) report the following average fiber lengths for spruce, Douglas fir, and western hemlock:

Spruce	2.8 mm.
Douglas Fir	4.8 mm.
Western Hemlock	3.3 mm.

A study of the data in Table I shows that Douglas fir sapwood has an unusually high density, due to its extremely slow growth rate. Because of this fact and the extra long fiber length, Douglas fir pulps should possess several unusual properties. The high density should be advantageous in increasing yields per digester, while the long fibers should result in high tearing strengths.

Since average values for the amount of sapwood to be expected in Douglas fir logs could not be found in the literature, data on the diameter and the sapwood thickness for a large number of commercial Douglas fir logs were obtained from a mill engaged in the manufacture of lumber from this species (21). All of the logs measured were from one region and are not necessarily representative of the logs found in other localities. Nevertheless, the data indicate the general range of values which might be expected. On logs ranging in diameter from 16 to 40 inches, the thickness of the sapwood layer ranged from 1.25 to 2.25 inches, corresponding roughly to about 15 per cent of the volume of the tree. This amounts to approximately 20 per cent by weight. It is the practice in this particular mill to allow two inches on the diameter of the logs for slab cuts, from which the average amount of sapwood to be expected in sawmill slabs has been estimated as over 90 per cent by weight. Thus, any pulping operation dealing with sawmill waste would involve the cooking of chips much richer in sapwood.

Samples of Douglas fir sapwood and heartwood were analysed for lignin, alcohol-benzene solubility, Cross and Bevan cellulose, pentosan content of the Cross and Bevan cellulose, pentosan content of the wood, acetyl groups and ash, using Institute methods. The analysis of the spruce wood used in this work was obtained from Institute files, while a representative analysis of western hemlock wood was obtained from the Forest Products Laboratory at Madison, Wisconsin (24). The data for these analyses are presented in Table II.

TABLE II

CHEMICAL ANALYSIS OF WOOD

Wood	Alcohol-Benzene Solubility, %	Lignin, %	Cross and Bevan Cellulose, %	Pentosans in Cross and Bevan Cellulose, %	Pentosans, %	Acetyl Groups, %	Ash, %
Douglas Fir Sapwood	2.17	29.4	60.8	5.9	8.54	1.15	0.36
Douglas Fir Heartwood	3.13	28.6	61.5	6.0	8.68	0.48	0.12
Spruce	4.01	29.3	61.9	10.4	13.90	1.59	0.31
Western Hemlock	2.80	30.5	59.3		9.60		

INVESTIGATION OF DOUGLAS FIR CELLULOSE

Object:

In the light of the poor quality of the pulps prepared from Douglas fir by other investigators, the question arose as to whether the cause was due to an inherent quality of the cellulose itself, or to the destructive action of the cooking liquor during the digestion process. It was believed that the isolation of cellulose from this species by a method milder in its action than any of the commercial processes would give data showing the characteristics of this pulp with respect to spruce pulp prepared in the same manner. Such a comparison would indicate whether or not there was some fundamental deficiency in the fiber of Douglas fir. If the properties of this pulp were near the range of those of spruce pulp, it would appear that cooking procedures were at fault and that a satisfactory commercial pulp could be produced if the cooking procedure was properly adjusted.

The mildest practical method of isolating cellulose from wood in the quantities desired for this work was by a modification of the Cross and Bevan cellulose determination. This involves the softening of commercial chips by a mild alkali treatment at low temperature, followed by chlorination in several stages with intermittent alkaline extractions.

Isolation:

A considerable quantity of sapwood and heartwood chips was converted to pulp by a modification of the chlorination method used by Kang and Libby (22). The following procedure was used:

In order to soften the chips, prepared as described later in this paper (page 22), they were charged into small gas-heated rotary autoclaves and treated with sodium hydroxide amounting to four per cent of the oven dry weight of the wood. A liquor ratio of five to one was used. After heating at 80°C. for one hour, an additional four per cent of sodium hydroxide was added, the temperature raised to 100°C. and held at this value until 80 per cent of the total chemical was consumed, as determined by titration of a sample of the liquor. The softened chips were then washed with cold water.

In order to obtain the wood in a form suitable for chlorination, the softened chips were passed once through a Bauer Pulper, using plates No. B980 set 0.021 (±0.005) inch apart. The product of this treatment resembled wood pulp, in that it consisted for the most part of individual fibers. On examination under the microscope, this material showed only a few more broken fibers than would be expected from the chipping operation, and while numerous shives were noticed, they consisted at most of six or eight fibers.

For chlorination, 800 grams (oven dry) of the defibered wood were treated with chlorine water in four or five stages at room temperature, as required for delignification. The pulp was washed with cold water and extracted with warm (35°C.) one per cent sodium hydroxide solution between stages. The caustic treatment was followed by an alkaline wash and then by a warm water wash. After the third stage caustic treatment, the pulp was screened through a 0.010 inch screen plate on a laboratory size Valley flat screen to remove shives and dirt. The end point of the chlorination was determined by the time required for exhaustion of the chlorine. If the chlorine in the last stage was not completely consumed after twenty minutes, as indicated by potassium iodide-starch paper, a sample of the liquor was taken for titration of the residual chlorine, and the pulp washed immediately.

In order to obtain a pulp having a good white color, it was found that a mild hypochlorite bleach was necessary after the last chlorination. Not more than 1.5 per cent of bleach (as standard 35 per cent bleaching powder) was used. The bleaching was carried out at room temperature (about 25°C.) at one per cent consistency. The bleach liquor contained an excess of lime, and the suspension was alkaline to phenolphthalein after complete exhaustion of the bleach. The resulting pulp was then washed thoroughly with cold water, pressed to a density of approximately 25 per cent and stored in moisture-tight containers pending their physical evaluation in the pebble mill. The pertinent data obtained in this isolation of Douglas fir cellulose are presented in Table III. A batch of spruce chips was treated in a similar manner, so that comparisons might be made between spruce and Douglas fir celluloses.

TABLE III

ISOLATION OF CELLULOSE FROM SPRUCE AND DOUGLAS FIR

Wood	Hours of Alkali Treatment	Amount of Chlorine used in Different Stages, %					Bleach % Bleaching Powder	Total Chlorine	
		1st	2nd	3rd	4th	5th		as % Chlorine	as % Bleaching Powder
Heart	2.83	22.0	5.9	7.8	2.7	1.5	0.8	40.2	114.8
Sap	3.42	20.7	7.1	6.3	2.5	1.0	0.5	37.8	107.9
Spruce	3.67	22.9	5.1	5.0	2.0		0.8	35.5	100.8

Evaluation:

The chlorine pulps prepared as described above were evaluated in the Abbé pebble mill, using the procedure described later in this paper (page). The physical tests made on the test sheets were: basis weight, burst ratio, M. I. T. folding endurance, tear factor, and apparent density. Institute procedures were used in evaluating all physical properties of the test sheets, and care was taken to approach the moisture equilibrium of 65 per cent relative humidity and 70°F. from the wet side. In every case, at least ten hours were allowed for conditioning before testing the sheets. These pulps were not evaluated chemically.

Comparison of Celluloses from Spruce and Douglas Fir:

The data obtained from the physical evaluation of these chlorine pulps are given in detail in Appendix A and are summarized in Table IV. Curves for these data are given in Figures 1, 2, and 3.

TABLE IV

PHYSICAL PROPERTIES OF CHLORINE PULPS AT 600 FREENESS

Wood	Burst Ratio	Tear Factor	M.I.T. Folds	Apparent Density	Time to 600 Freeness
Douglas Fir Heart	100	1.23	593	13.2	32
Douglas Fir Sap	112	1.60	587	13.2	36
Spruce	184	1.06	4300	17.4	46

Referring to Table III, it is seen that spruce required less chlorine than either Douglas fir heartwood or sapwood and that the heartwood required more chlorine than sapwood. Also, the time required for 90 per cent

FIGURE 1

BURST RATIO AND FREENESS DEVELOPMENT

Chlorine Pulps

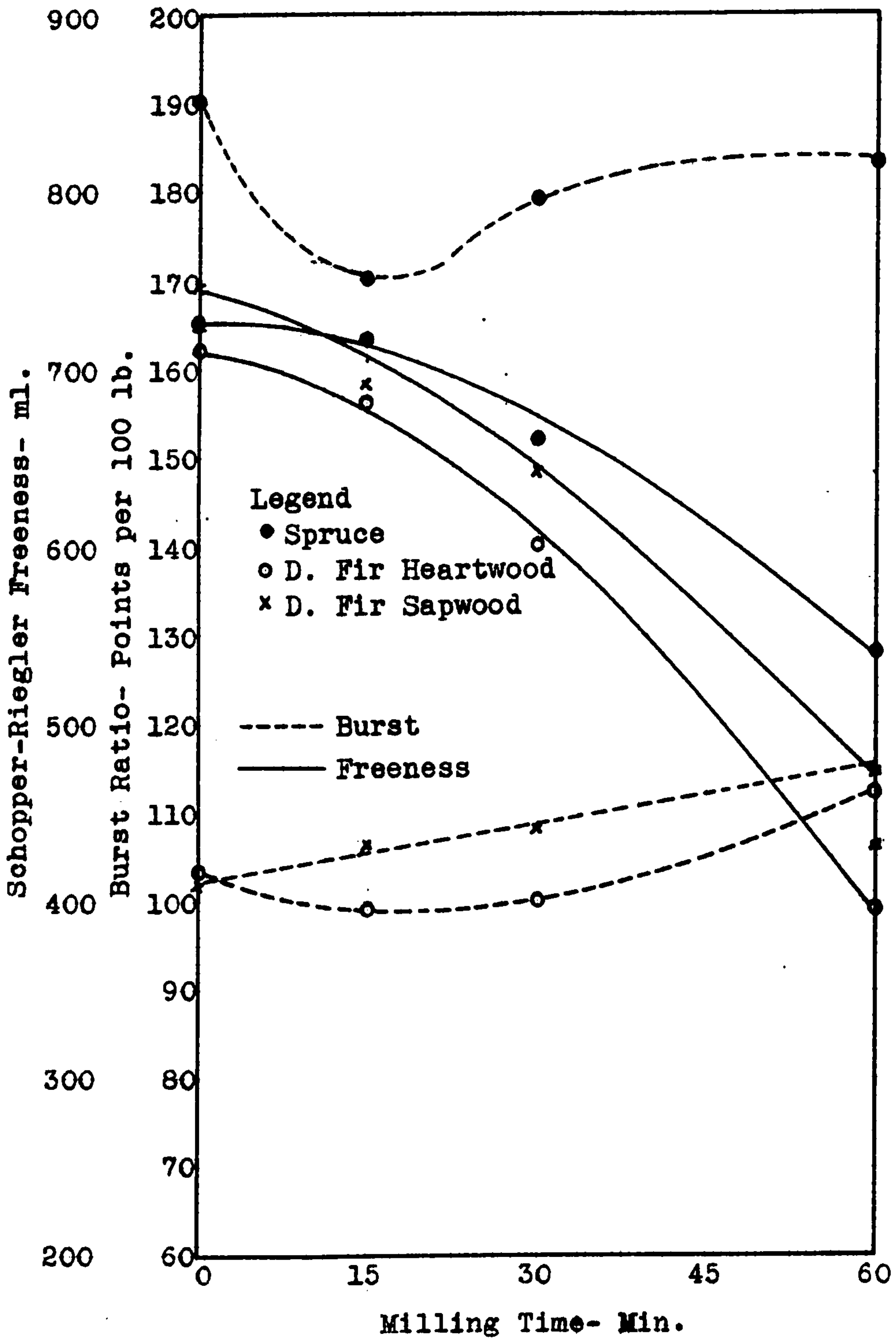


FIGURE 2

M.I.T. FOLDING ENDURANCE

Chlorine Pulps

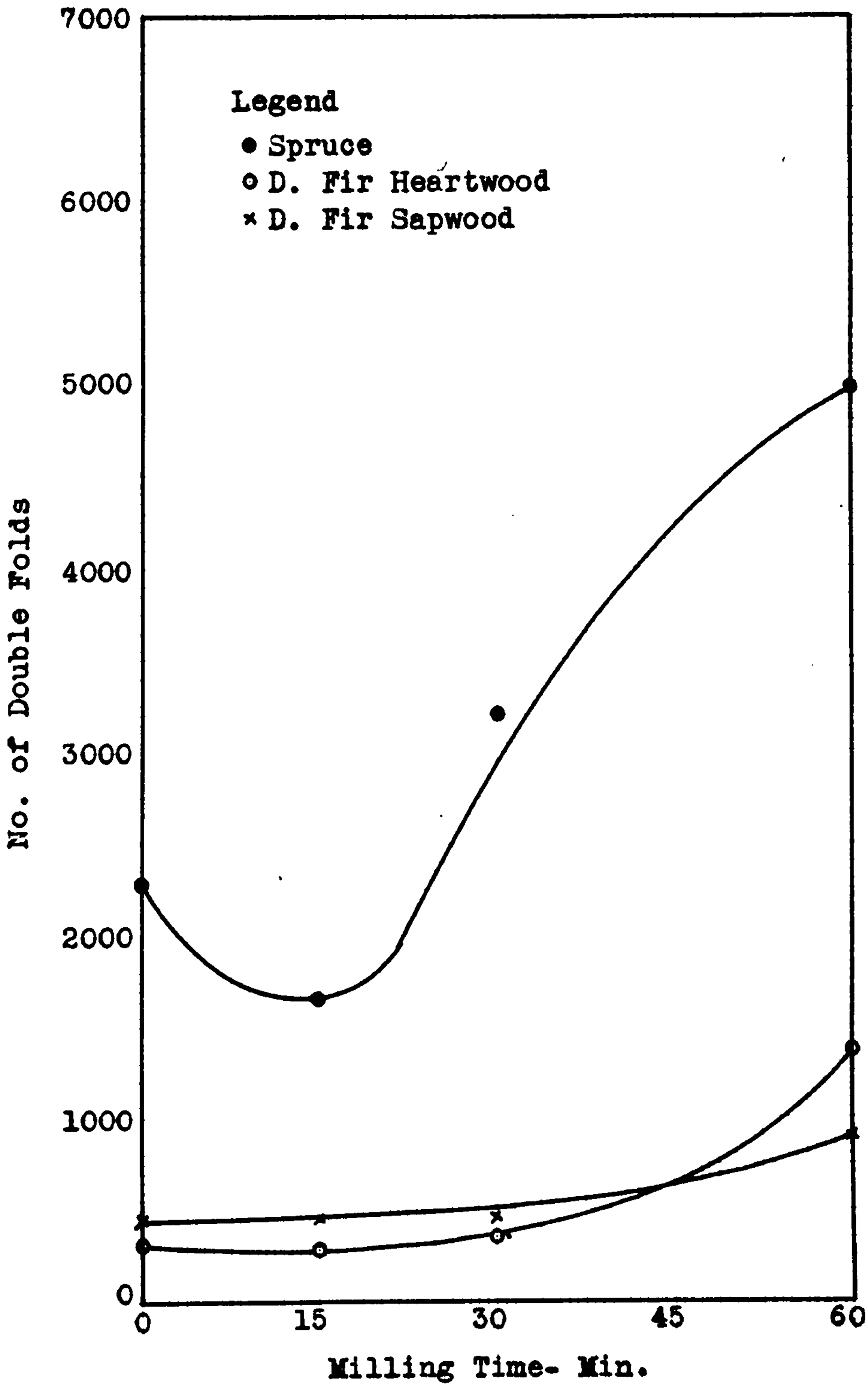
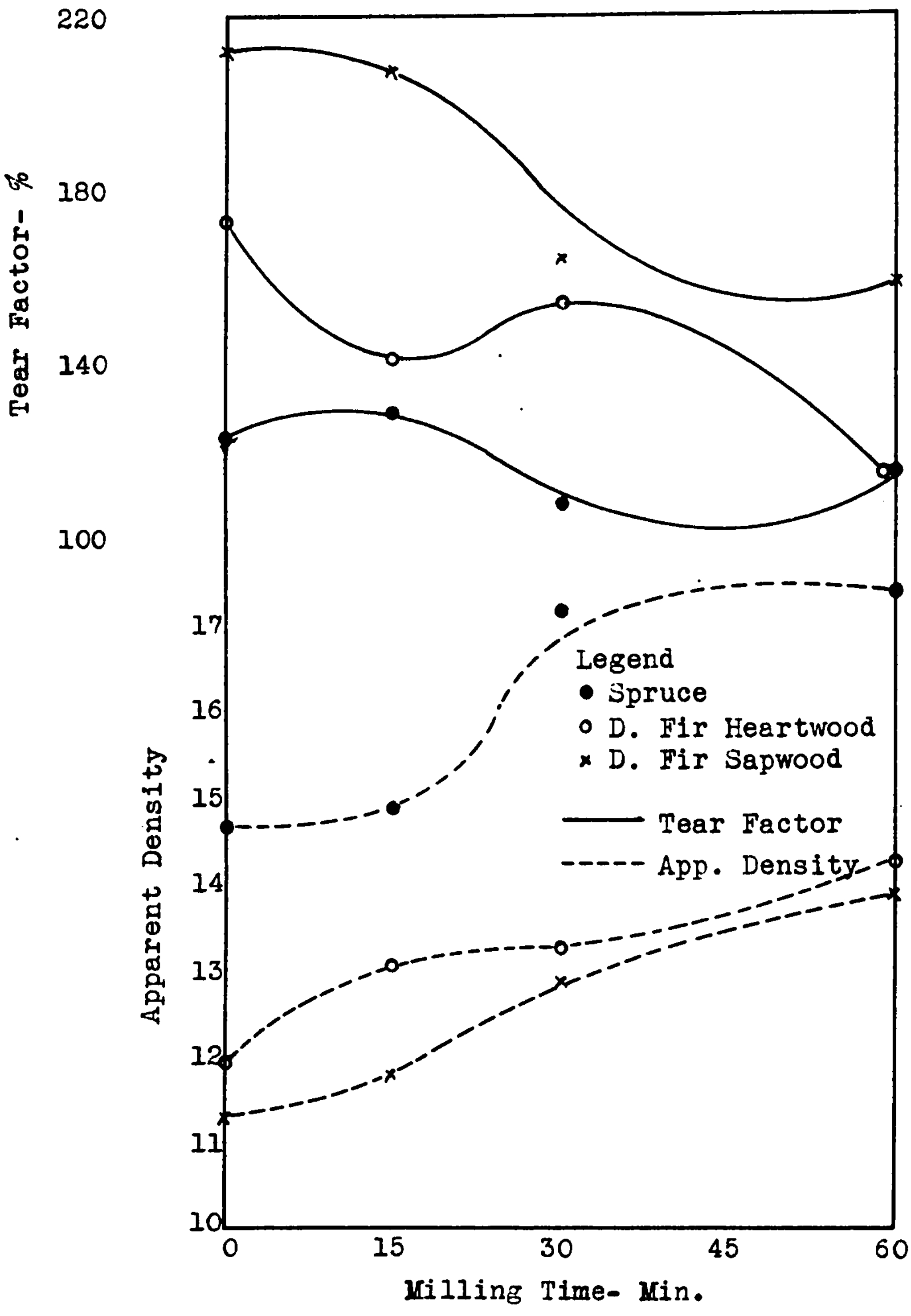


FIGURE 3

TEAR FACTOR AND APPARENT DENSITY

Chlorine Pulps



consumption of the alkali used in softening the chips was highest for spruce, while heartwood showed the most rapid consumption. The time required by sapwood was intermediate between the two. Thus it is apparent that heartwood (and to a lesser extent, sapwood) not only consumes alkali at a greater rate than spruce, but also requires a greater amount of chlorine for complete removal of encrustants from the cellulose. This would indicate the relative ease of pulping of these woods to be in the order: spruce, Douglas fir sapwood, and Douglas fir heartwood.

The strength properties of these chlorine pulps were arbitrarily compared at a freeness of 600 (Schopper-Riegler), this value being chosen as representative of the degree of hydration used in making a number of different grades of paper. In comparing the physical properties of the pulps obtained from the various wood samples, a number of interesting points are found (See Appendix A, Table IV and Fig. 1, 2, and 3).

Spruce showed a considerably higher burst ratio, folding endurance, and apparent density than the other two. Both Douglas fir heartwood and sapwood pulps showed higher tear factors than did spruce. A possible explanation of the low burst, fold, and apparent density observed in the Douglas fir pulps lies in the pentosan content of the Douglas fir wood. Spruce wood is considerably richer in this material, which is generally believed to be the source of at least a portion of the mucilaginous cementing matter which increases burst and fold in a sheet. Thus, a sheet made from Douglas fir pulp might be expected to be lower in burst and fold and bulkier than a similar sheet of spruce pulp. The high tear of the Douglas fir pulps may be attributed to the unusually long fibers of this species.

In general, pulps such as those from sapwood and heartwood would prove desirable in printing papers, and some grades of tissue, wrapping, and bond. The specific properties which make this pulp superior to spruce pulp for certain purposes are the high tear and low apparent density. Bleached Douglas fir pulps could probably not be used in a furnish without the addition of one or more blending pulps, in order to attain the balance of properties desired in the finished sheet.

Another possible application of Douglas fir pulps is in the field of chemical pulps for use in the manufacture of viscose, rayon, and other cellulose derivatives. While none of the data obtained in connection with the chlorine pulps have a direct bearing on this phase of pulp utilization, the sulphite pulps prepared in connection with this work have been evaluated chemically with this application in mind.

The physical data obtained in connection with these chlorine pulps show that Douglas fir cellulose as a paper has a definite field of application making fiber. This leads to the conclusion that the satisfactory pulping of this species by the sulphite process would not only add to the sphere of the industry in general, but also would open the way to the utilisation of a new and extremely large supply of cheap raw material which has heretofore been regarded as unsuitable for acid pulping.

PRACTICAL PULPING EXPERIMENTS

Procedures:

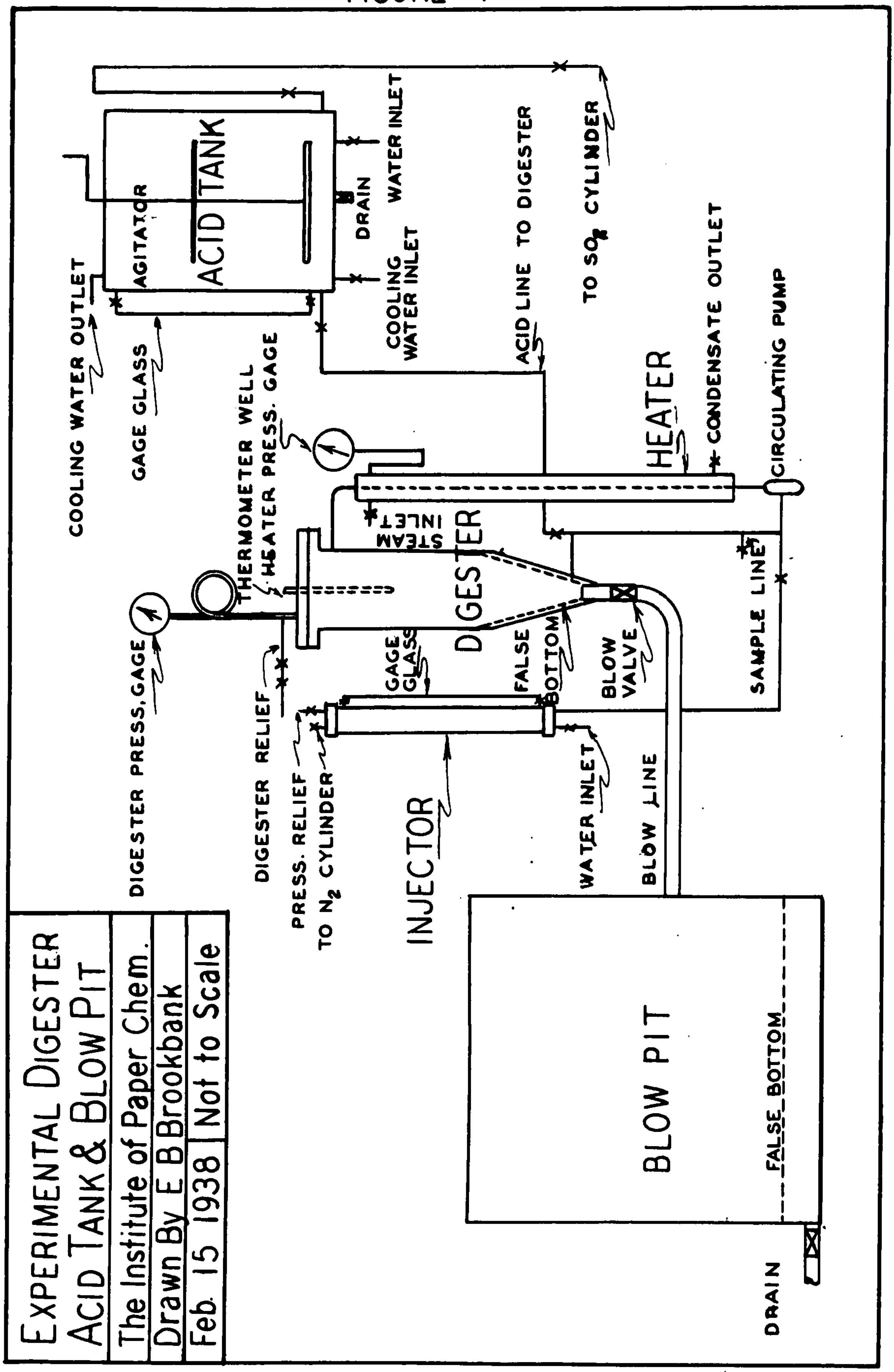
Wood Preparation: The chips used in the practical pulping work were prepared as follows: A section of the log, approximately eight feet long, was cut off and split into fairly narrow segments. The sapwood was split from these and barked by hand, while the heartwood for chipping was taken in complete segments, so as to be representative of the entire heartwood portion of the log. The two types of wood were chipped separately in a small Carthage chipper, using a knife setting to give chips five-eighths of an inch long. The chips were screened on a half-inch screen to remove fines, while the oversize knots and slivers were picked out by hand. The finished chips were then thoroughly mixed and stored in air-tight drums, a few milliliters of chloroform being added to prevent decay. Just before making a cook, a sufficient quantity of these chips for one digester charge (approximately 5500 grams of the wet chips) was wighed into paper bags. Representative moisture samples were taken as the bags were filled. These were weighed immediately and dried in an oven to constant weight at 105°C. From these data, the weight of oven-dry wood used in making the cook, as well as the amount of sap moisture present in the wood, were calculated.

Acid Preparation: Calcium base sulphite cooking acid was prepared from high calcium hydrated lime and refrigeration grade liquid sulphur dioxide. The acid was made in a small wood stave tank having a capacity of 220 liters and provided with a cooling coil, calibrated gage glass, agitator, and perforated copper pipe for the introduction of sulphur dioxide. The arrangement of the acid-making equipment with respect to the digesters is shown in Figure 4.

Cooking acid was made in the following manner: The acid tank was filled approximately half full of water and sulphur dioxide was bubbled in until the strength of the resulting sulphurous acid solution was somewhat in excess of the total sulphur dioxide content desired in the finished liquor. The calculated amount of hydrated lime for the total amount of acid to be made was then added as a slurry and the contents of the tank stirred vigorously. At this point, an analysis of the liquor in the tank was made, the results being used in calculating the amount of water or lime to be added to adjust the combined sulphur dioxide to the correct value.

After obtaining the correct lime content, sulphur dioxide was admitted from the cylinder with a small known valve opening for a measured time interval, and the acid was again analyzed. Thus it was possible to estimate the length of time for admission of sulphur dioxide to bring the free sulphur dioxide content up to the desired value. The following tolerances were arbitrarily chosen for the strength of the cooking liquor which was not varied in any of the cooks made.

FIGURE 4



EXPERIMENTAL DIGESTER
ACID TANK & BLOW PIT
The Institute of Paper Chem.
Drawn By E B Brookbank
Feb. 15 1938
Not to Scale
ACID TANK
AGITATOR
DRAIN
WATER INLET
COOLING WATER INLET
COOLING WATER OUTLET
GAGE GLASS
ACID LINE TO DIGESTER
TO SO₂ CYLINDER
THERMOMETER WELL
HEATER PRESS. GAGE
HEATER
CONDENSATE OUTLET
CIRCULATING PUMP
STEAM INLET
DIGESTER
DIGESTER PRESS. GAGE
DIGESTER RELIEF
GAGE GLASS
FALSE BOTTOM
BLOW VALVE
SAMPLE LINE
PRESS. RELIEF TO N₂ CYLINDER
INJECTOR
WATER INLET
BLOW LINE
BLOW PIT
FALSE BOTTOM
DRAIN

Total Sulphur Dioxide	7.25 ± 0.06%
Free Sulphur Dioxide	6.00 ± 0.04%
Combined Sulphur Dioxide	1.25 ± 0.02%

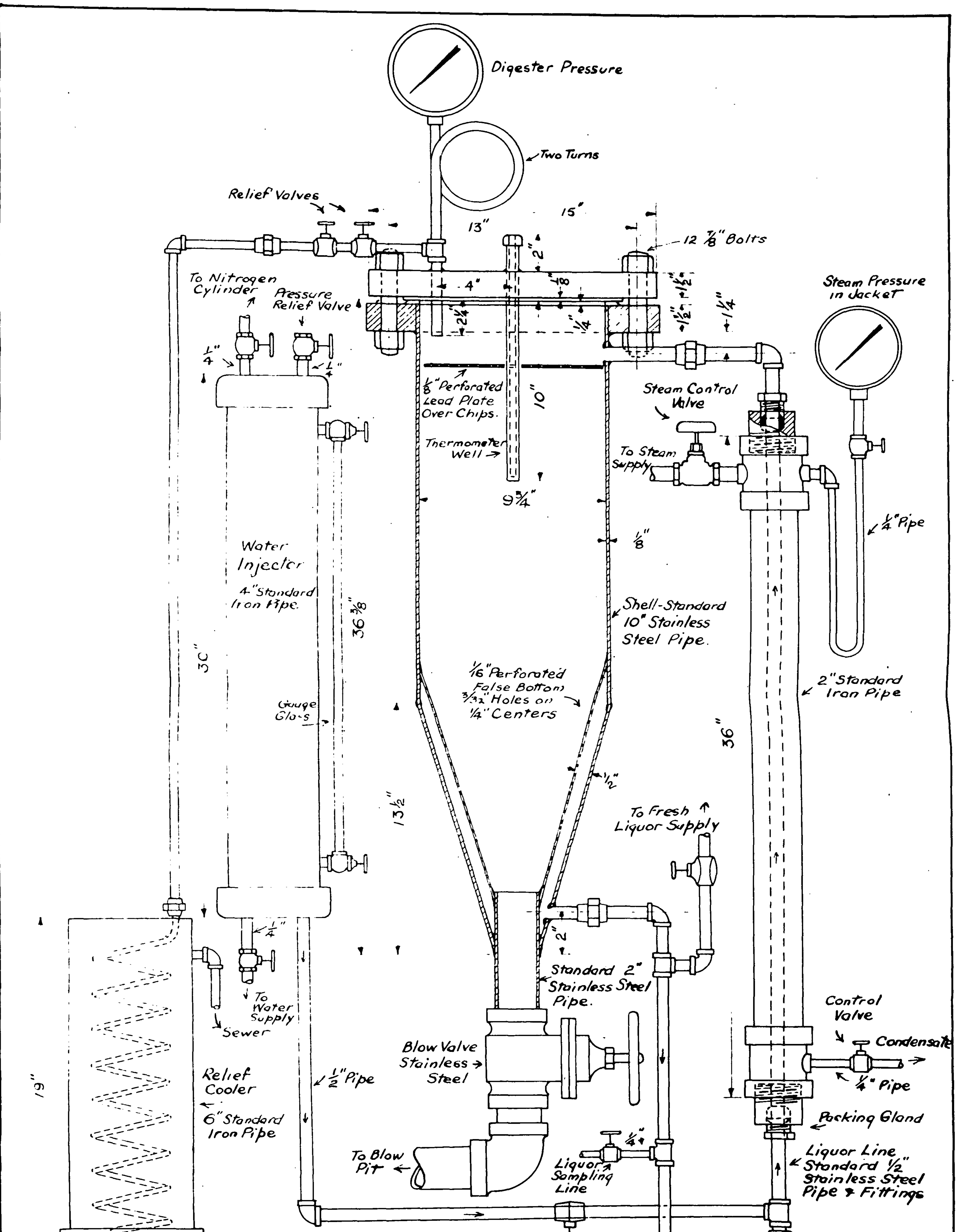
The terms total, free, and combined are used as customarily denoted in mill operation, the combined sulphur dioxide being the amount of sulphur dioxide equivalent to the lime present as calcium monosulphite, and the free being the difference between the total and the combined.

The acid was analyzed by the iodate method, as described by Palmrose (22), using the following sampling procedure: A small beaker of the acid was withdrawn from the tank after thorough mixing, and exactly 25 milliliters of this sample were diluted to 250 milliliters in a graduated flask. Twenty-five milliliters of the diluted acid, representing 2.5 milliliters of the original liquor, were used for titration.

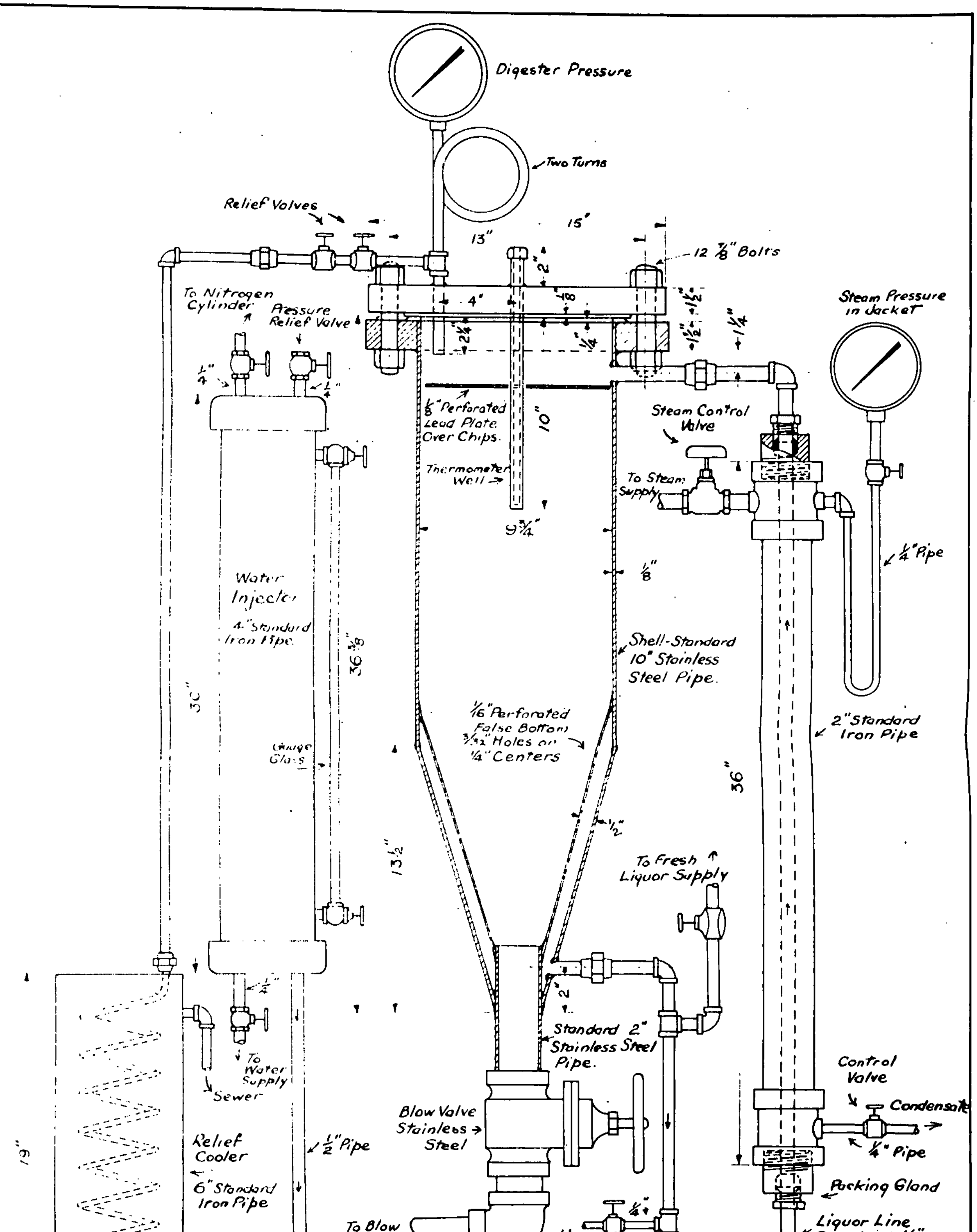
Digestion: The cooks were made in small stationary autoclaves provided with circulating pumps, indirect steam heaters, and injectors either for the application of gas pressure from a nitrogen cylinder or for the introduction of water, as desired. The digester and auxiliaries are shown in Figure 4. The autoclaves and heater piping, as well as any other piping or fittings which came into contact with the cooking liquor, were made of stainless steel. Connections were provided to the acid tank for filling the digesters by gravity flow, thus minimizing the loss of free sulphur dioxide during charging. The conical bottom of the digester was connected to the blow-pit by means of a two-inch stainless steel pipe in which was located a two-inch gate valve.

The blow-pit was a small wood stave tank containing a false bottom of stainless steel punched with 3/32-inch holes spaced 3/8-inch apart. The blow-pit was provided with a removable cover and vomit stack, the latter being made of six-inch copper pipe. The drain valve for the pit was located so as to draw off the liquid from under the stainless steel false bottom. A detailed drawing of the digester, heater, and injector is shown in Figure 5.

The following procedure was followed in making the experimental sulphite cooks. A weighed charge of air-dry chips of known moisture content was packed into the digester and covered with a perforated lead plate to prevent them from floating in the liquor. Cooking acid, prepared as above, was admitted through the bottom until the liquid level in the digester was two inches below the top flange. This was done regardless of the amount of acid required, as experience has shown that a slight variation in the gas volume above the liquor causes more variation in the pulp than does a slight variation in the liquor ratio. The quantity of acid required was measured by means of the calibrated gage glass on the acid tank. The greatest variation observed in the ratio of liquor to wood, using this method, was 7.1 $\pm$ 0.25. After charging the digester, the cover was bolted on and the digester pressure immediately raised to 85 pounds per square inch (gage) by means of the injector and a cylinder of commercial nitrogen gas. The



Experimental Sulphite Digester.
Institute of Paper Chemistry
March 4, 1937. J.R.L.



Experimental Sulphite Digester.
Institute of Paper Chemistry
March 4, 1937. J.R.L.

circulating pump was then started and steam was admitted to the heater at such a rate as to follow the predetermined temperature schedule. Gas was relieved from the digester, as necessary, to keep the pressure constant at the desired value of 85 pounds per square inch.

During the last fifteen minutes before blowing, the pressure was relieved at a constant rate to fifty pounds per square inch, at which point the digester was blown. The cover was removed and any pulp remaining in the digester and blow line washed into the blow pit with hot water. Before draining the waste liquor from the pit, a piece of unbleached muslin cloth was securely tied over the drain valve to catch any fibers which might have washed through the false bottom.

After draining off the waste liquer, the stock in the pit was flooded with hot water, stirred for fifteen minutes with a 1/4 h.p. "Lightnin Mixer" and then allowed to drain. The pit was filled a second time with hot water and the stock was stirred for another ten minute period, after which it was allowed to drain slowly overnight.

Determination of Yield: All pulps were screened on a small laboratory size Valley flat screen equipped with a plate cut with 0.010-inch slots. The rejections from the screening operation were dried to constant weight in an oven at 105°C. The screened pulp was pressed to a density of approximately 25 per cent, broken into small lumps and thoroughly mixed on a large table. It was then stored in an airtight container, triplicate moisture samples being taken as the can was filled. The net weight of the wet pulp in the can was determined and the oven-dry weight calculated from the moisture content. Pulp which passed through the false bottom of the blow-pit was washed out through the drain valve and, since it contained considerable dirt and fines, was dried separately. The oven-dry weight of this pulp (referred to as drainings) was added to that of the screened pulp in determining the screened yield. The weight of the screenings was added to that of the screened pulp to determine the total yield. All yield figures are reported on the basis of the weight of oven-dry wood charged to the digester.

<u>Bleaching Procedure</u>: Those experimental pulps which fell within the range of reasonable bleach consumptions were bleached by the following two-stage procedure:

The first stage consisted of a direct chlorination, using chlorine water in an amount equal to 35 per cent of the total requirement as indicated by the bleach consumption. The chlorination was carried out at 20°C. and at one per cent consistency until the chlorine was exhausted, provided this time interval did not exceed 30 minutes. The chlorinated pulp was thoroughly washed with cold water. The pulp was next neutralized with sodium hydroxide just sufficient to keep the suspension alkaline to phenolphthalein. This operation was carried out at two per cent consistency and was continued for a period of 30 minutes, followed by thorough washing with cold water. The temperature used was

20°C.

The second stage bleaching consisted of a hypochlorite bleach at 4.5 per cent consistency for a time interval of four hours at a temperature of 38°C. (100°F.). The amount of bleach used was determined by small scale bleach consumption determinations made on the pulp from the first stage after neutralization. The conditions of time, temperature, and consistency were maintained at the values given above for these determinations. The amount of bleach required to produce a brightness of 80 per cent as measured by the General Electric Reflection Meter was taken from the curve of bleach used versus brightness, and this amount was used in bleaching the balance of the pulp from the first stage. The bleach liquor was prepared from bleaching powder and was stabilized by the addition of an excess of lime. During bleaching, the pH of the liquor was maintained in the range from 8.0 to 8.5 by the addition of small amounts of hydrated lime. At the end of the bleaching period, the pulp was thoroughly washed with warm water, pressed to a density of approximately 25 per cent and stored in moisture tight containers pending physical evaluation.

Physical Evaluation: All of the unbleached and bleached pulps which had any promise of commercial value were evaluated in the pebble mill. The following milling procedure was used: Ninety grams (oven-dry) of pulp were disintegrated in two liters of water in the British disintegrator for 3000 units on the counter. The disintegrated pulp plus one liter of water (total volume of 3 liters) was placed in the jar of an Abbe' Pebble mill and 77 flint pebbles, weighing 5 kilograms and having a volume of 1892 milliliters, were added. The mill was then rotated at 60 r.p.m. for the desired time interval. At the end of the milling interval, the pulp was removed from the jar, diluted with 3 liters of water and the pebbles removed. Three liters of this suspension were diluted to 12 liters in a five-gallon crock and stirred with a small "Lightnin Mixer" for a period of 15 minutes. The consistency of the suspension was determined and test sheets made by the standard procedure (TAPPI Method T 205 m). The test sheets were conditioned at 65 per cent relative humidity and 70°F. for at least eight hours, care being taken to approach the equilibrium condition from the wet side. The sheets were evaluated for burst ratio, tear factor, basis weight, M.I.T. folding endurance, apparent density, and opacity, the last test being applied only to the bleached pulps. All physical tests were made by Institute methods.

In addition to the above physical evaluation, permanganate numbers and bleach consumptions were determined on the unbleached pulps, using Institute methods No. 410-6 and 409-2, respectively. The method of sampling for these two determinations was as follows:

Approximately 500 grams of the unbleached pulp, taken in a representative manner from the entire cook, were diluted to about two per cent consistency and stirred thoroughly with the "Lightnin Mixer" for 20 minutes. The sample used in making the determinations was taken in small amounts from this large average sample. It was felt that this method insured values for permanganate numbers and bleach consumptions more nearly representative of the entire lot of pulp than any method of grab sampling.

Chemical Evaluation: The best Douglas fir pulps from sapwood, heartwood, slab wood, and the entire tree were chosen for chemical analysis. The two comparison pulps, spruce and western hemlock, were also analysed. The following determinations, all made by Institute methods, were carried out on the unbleached pulps: Lignin, alpha-cellulose, cuprammonium viscosity, pentosans, alcohol-benzene solubility, ether solubility, and ash. These same pulps, after bleaching, were analyzed for the following: lignin, alpha-cellulose, cuprammonium viscosity, pentosans, copper number, ether solubility, and ash.

The data obtained in this work are presented later.

PROCESS VARIABLES:

Raw Material Variables: The raw material variables which influence sulphite pulping are: species, chip length, wood density, and wood moisture content. Of these, only the species variable was changed. The species of wood pulped were spruce, western hemlock, and Douglas fir heartwood and sapwood. Cooks were also made of mixtures of Douglas fir heartwood and sapwood in proportions representing slab wood and the entire tree. For the slab wood cook, 15 per cent of heartwood by weight was mixed with the sapwood. This value was chosen as richer in heartwood than would actually be found in sawmill waste. The mixture representing the entire tree was composed of 85 per cent heartwood by weight.

While the chip moisture content for the different kinds of wood varied, the moisture content of any one kind was constant over a very small range. The small variation which did occur was occasioned by the slight drying of the chips which took place during storage. It is felt, however, that this variation was so small as to be insignificant.

All chips used in this work were cut with the same knife setting on the chipper and were screened and sorted in the same manner. Thus, this variable was effectively eliminated.

Cooking Variables: As has already been stated, acid composition, digester pressure, gas volume in the digesters; and liquor ratio were constant within a very narrow range for all cooks made.

In selecting cooking conditions for the first cooks, an attempt was made to keep the action as mild as possible and still maintain the cooking variables within the commercial range. For this reason, a comparatively low maximum temperature of 134°C. was used. The temperature was brought up to this point very slowly after an initial rapid rise to 90°C. The temperature schedule used for the first few cooks is designated as Schedule No. 1, and is given in detail below.

Schedule No. 1 (see Figure 6):

From	To	Time-Hours	Total Elapsed Time-Hours
20°C.	90°C.	1.0	1.0
90	100	0.5	1.5
100	110	1.0	2.5
110	118	4.0	6.5
118	134	4.0	10.5

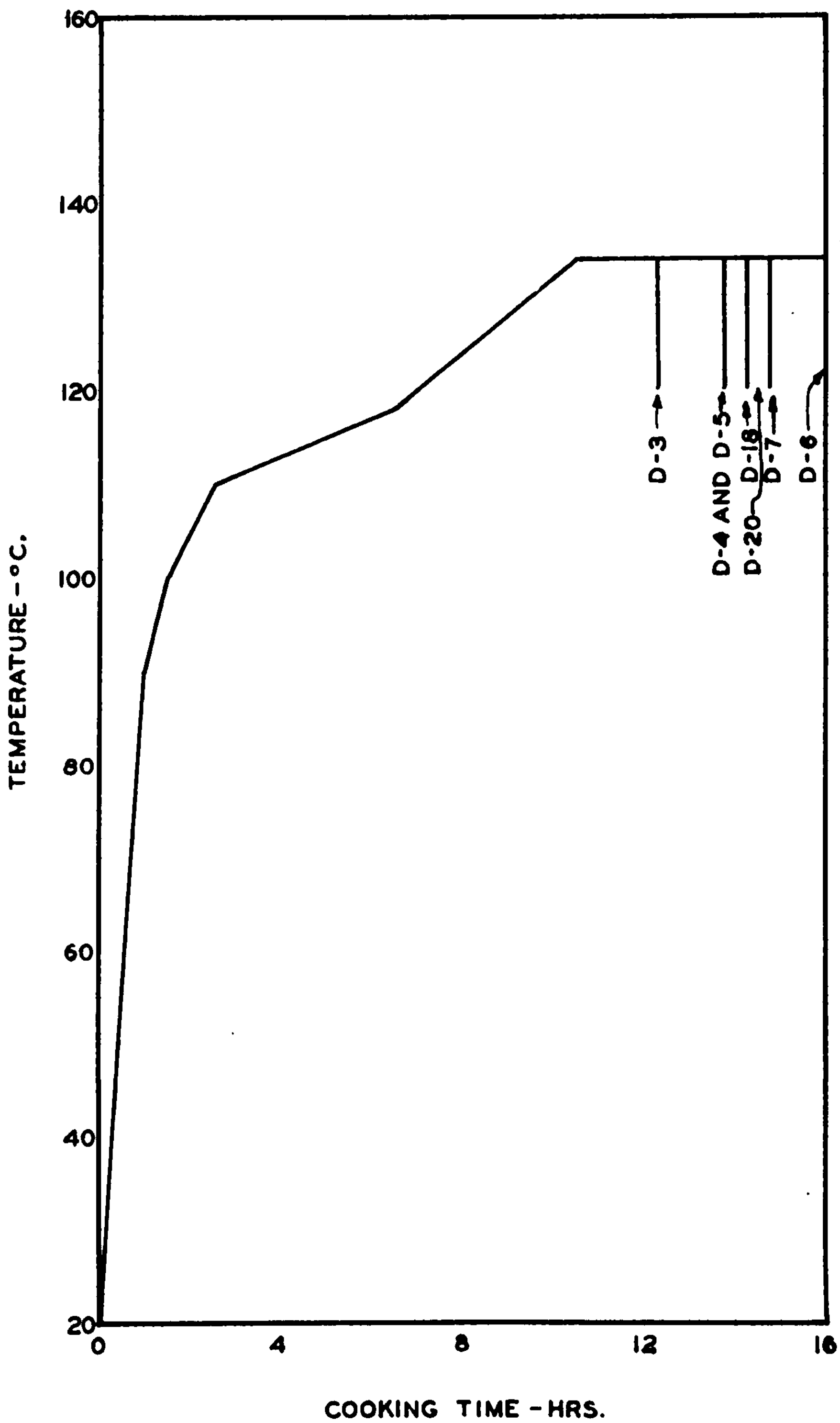
Hold 134°C. until end of cook.

This schedule was of the concave type in order to provide a greater time interval for sulphonation at comparatively low temperatures. Other schedules were used, and will be presented later.

The maximum digester pressure in all cooks was 85 pounds per square inch (gage). This pressure was applied by nitrogen gas at the very start of the cook and was maintained at this value throughout the cooking period by relieving gas to the atmosphere. The pressure was reduced uniformly to 50 pounds per square inch during the last 15 minutes of the cook, all cooks being blown at this final pressure. While this maximum pressure falls in the middle of the range of cooking pressures used in present commercial sulphite operations, the relief period at the end of the cook was much shorter than is usually the case. This short relief period was used in order that cooks might be blown on short notice. In most cases the endpoint of the cook was determined by the color of the cooking liquor; hence a short relief period made it possible to blow the

FIGURE 6

SCHEDULE NO. 1



cooks 15 minutes after the liquor sample indicated that cooking had progressed to the point desired. Any operation involving a longer relief period would therefore require an increase in the total cooking time in order to obtain a pulp with the same degree of delignification.

In most cases, as has been mentioned previously, cooking time was determined by the color of the cooking liquor. Precipitation of the cooking liquor was observed in several of the cooks, and as this was followed (within 15 to 30 minutes) by noticeable burning of the pulp, those cooks were blown as soon as possible after precipitation started in order to minimize this effect. The phenomena of burning and precipitation will be discussed in more detail at a later point in this dissertation.

Two sapwood cooks, one of 12.25 hours (D-3) and one of 13.75 hours (D-4), were made using Schedule No. 1. Both of these cooks produced satisfactory pulps from the standpoint of yield and quality. A heartwood cook (D-5), made under these same conditions with 13.75 hours total cooking time, was found to be quite raw. Using Schedule No. 1, two more heartwood cooks were made, the first of 16 hours' duration(D-6). This pulp was badly burned, and the waste liquor contained a considerable amount of an inorganic precipitate, which was found to contain both calcium sulphite and calcium sulphate. Free sulphur may have been present, but it was not identified. Cook No. D-7, made with heartwood chips under the above conditions, was blown after 14.75 hours. The liquor was cloudy when blown but still had an orange-red color. At a later date, another heartwood cook (D-18) was made under the same conditions as Cook No. D-7 and was blown after 14.5 hours. This was done to check the reproducibility of the cooking procedure as regards yield and quality of pulp. The waste liquor from this cook was clear at the time of blowing.

Several cooks were made at higher maximum temperatures. Cook No. D-8, using sapwood chips, was made according to Schedule No. 2 with a maximum temperature of 145°C. This schedule is given below.

Schedule No. 2 (see Figure 7):

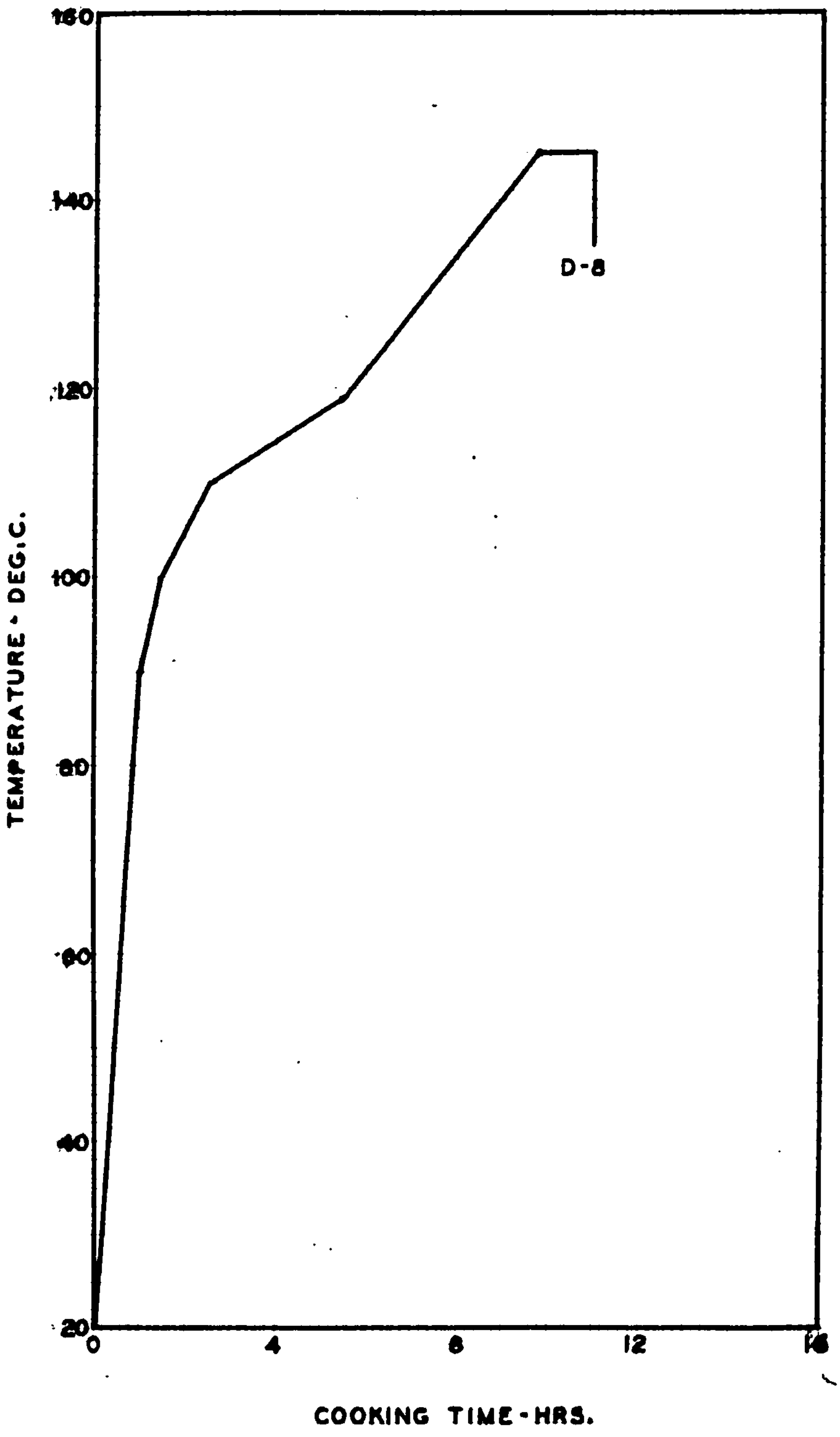
From	To	Time-Hrs.	Total Elapsed Time-Hrs.
20°C.	90°C.	1.0	1.0
90	100	0.5	1.5
100	110	1.0	2.5
110	119	3.0	5.5
119	145	4.25	9.75
Hold 145°C. until end of cook.			

The cook was blown after 11 hours, at which time the liquor had precipitated. This pulp was badly burned and was not evaluated except for yield, bleach consumption, and permanganate number.

It was felt that, with a more rapid temperature rise to 145°C., a satisfactory pulp might be obtained before burning occurred. For this reason, another sapwood cook (D-9) was made according to Schedule

FIGURE 7

SCHEDULE NO. 2



No. 3, given below. This cook resulted in a satisfactory pulp after 10 hours.

Schedule No. 3 (see Figure 8):

From	To	Time-Hrs.	Total Elapsed Time-Hrs.
20°C.	90°C.	1.0	1.0
90	100	0.5	1.5
100	110	1.0	2.5
110	120	2.0	4.5
120	145	4.0	8.5

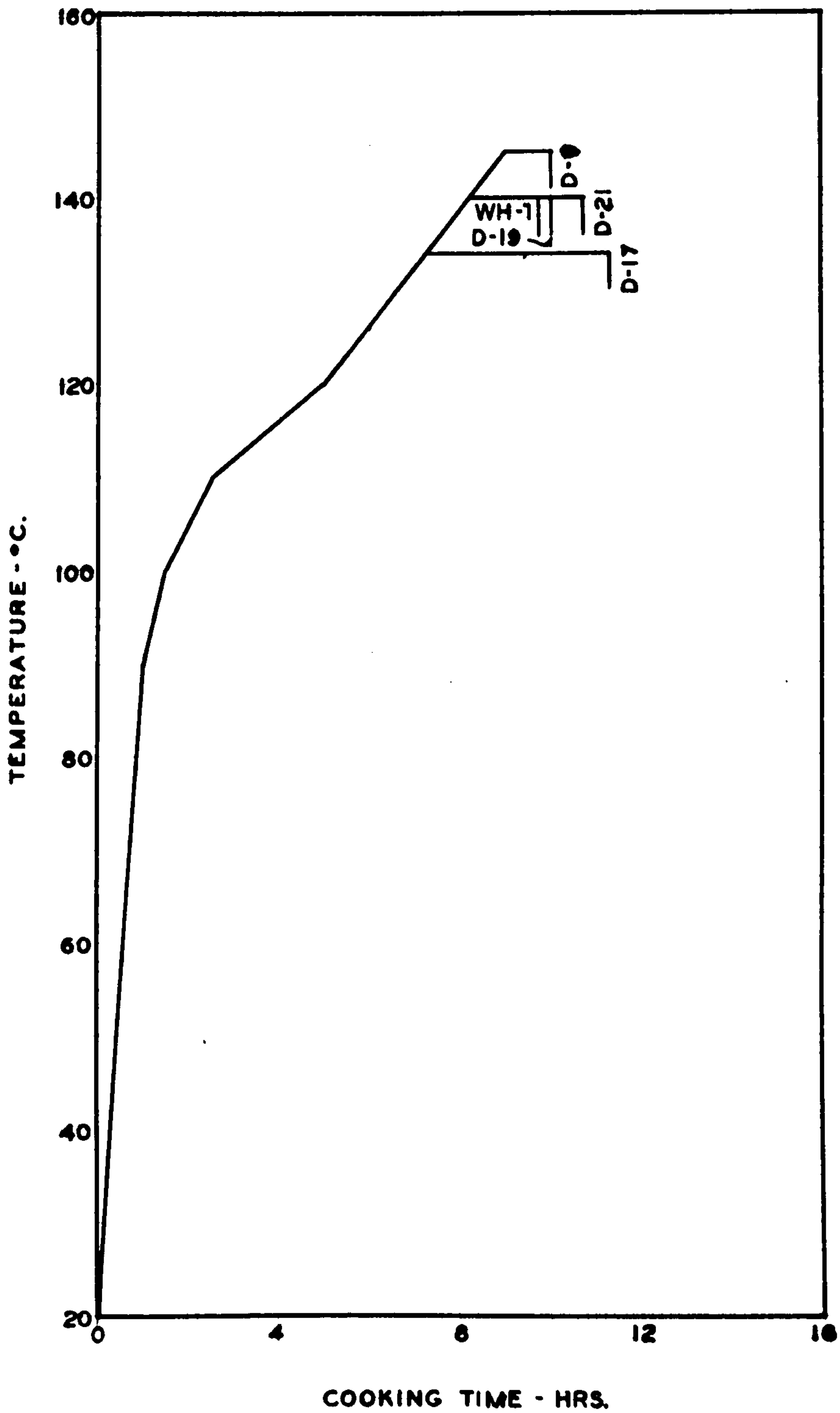
Hold 145°C. until end of cook.
Cooks made using Schedule No. 3, but at maximum temperatures below 145°C., had the same rate of rise from 120°C. to the final maximum temperature.

It was found that heartwood could not be pulped at 140°C. without precipitation of the cooking liquor with the attendant burning of the pulp. Cook D-21 illustrates this point. The cook was blown after 10.75 hours, at which time the liquor had precipitated. The digester charge was found to be incompletely pulped, the amount of screenings being very high. However, heartwood can be satisfactorily pulped using Schedule No. 3 with the faster temperature rise, provided the maximum temperature is low (134°C.). A satisfactory pulp was obtained from heartwood chips when cooked by Schedule No. 3 at a maximum temperature of 134°C., using a total cooking time of 11.25 hours (D-17).

Mixtures of sapwood and heartwood chips representative of slab wood and of the entire tree were cooked. The slab wood mixture (D-19), consisting of 85 per cent sapwood by weight, was pulped in 10 hours

FIGURE 8

SCHEDULE NO. 3



using Schedule No. 3 and a maximum temperature of 140°C. The mixture representative of the entire tree (D-20) consisted of 85 per cent heartwood by weight and was pulped at a maximum temperature of 134°C. in 14.25 hours, using Schedule No. 1.

For purposes of comparison, a western hemlock cook (WH-1) was made according to Schedule No. 3 and was blown after 9.75 hours, using a maximum temperature of 140°C.

A spruce pulp (S-2), cooked by the Institute pulping class in the Spring of 1937, was chosen as a comparison pulp from this species. This pulp was cooked at 75 pounds per square inch maximum pressure, the digester being relieved to 50 pounds per square inch during the last hour. No initial nitrogen pressure was used. The acid concentration in this case was 5.00 per cent free and 1.20 per cent combined sulphur dioxide. The temperature schedule for this cook is given below.

Concave Spruce Schedule (see Figure 9):

From	To	Time-Hrs.	Total Elapsed Time-Hrs.
20°C.	110°C.	3.0	3.0
110	120	2.5	5.5
120	140	1.5	7.0

Cook blown at end of 10 hours. Maximum temperature 140°C.

Summary of Experimental Pulping Data:

The data collected during the individual cooks are given in Appendix B and are summarized in Table V.

FIGURE 9

CONCAVE SPRUCE SCHEDULE

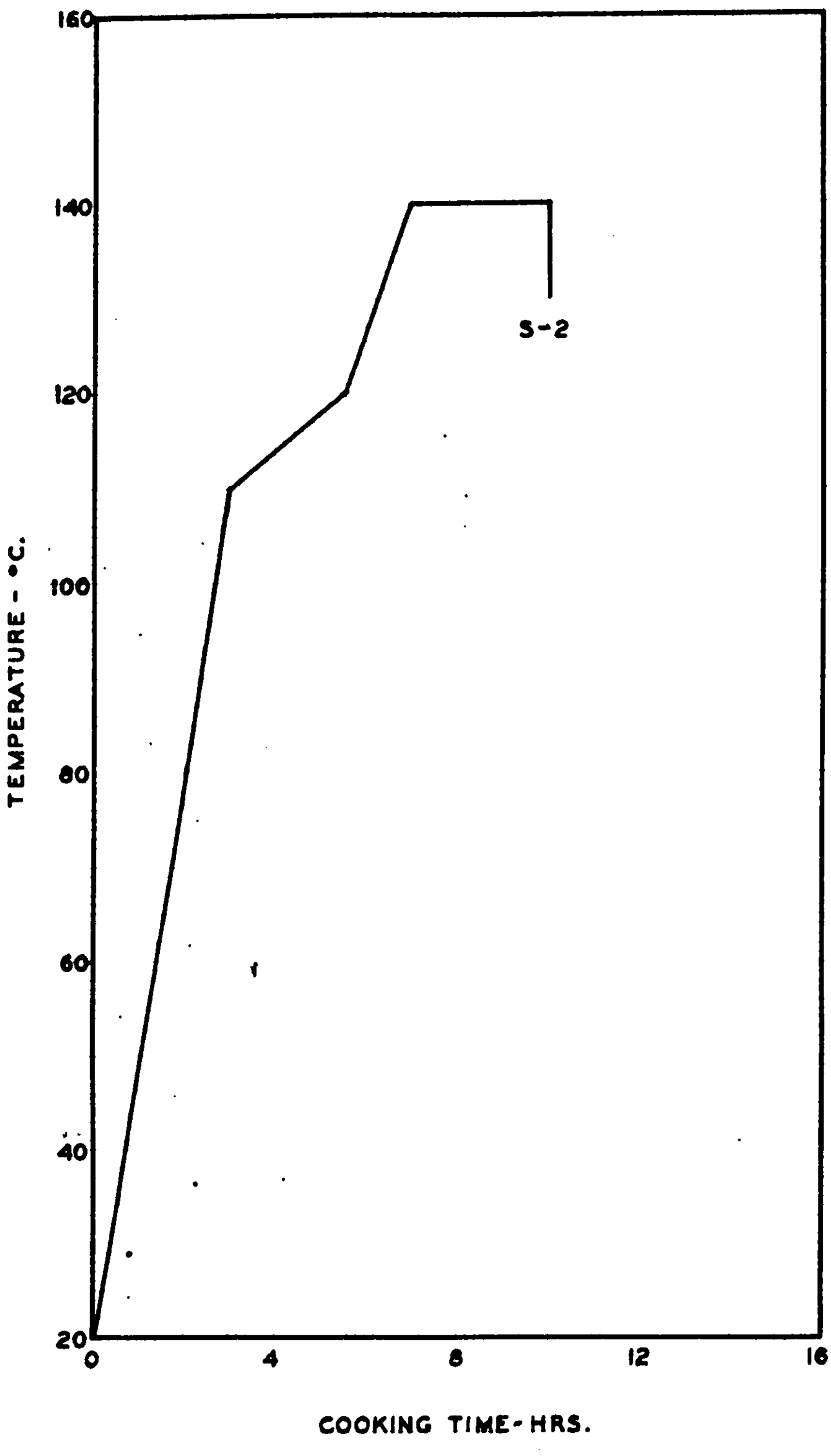


TABLE V

SUMMARY OF COOKING DATA

Cook No.	Dig. No.	Wood	Sched. No.	Max. Temp. Deg. C.	Total Time Hours	Liquor Ratio	Yields Total %	Yields Screened %	Yields Screenings %	$KMnO_4$ No.	Bleach Consumption %
D-3	2	Sapwood	1	134	12.25	7.33	52.3	51.9	0.78	16.1	21
D-4	2	Sapwood	1	134	13.75	7.33	46.0	45.9	0.31	11.2	13
D-5	2	Heartwood	1	134	13.75	6.95	56.9	56.7	0.37	27.2	37
D-6	2	Heartwood	1	134	16.00	6.95	47.8	47.7	0.10	8.0	6
D-7	2	Heartwood	1	134	14.75	6.94	51.2	51.2	0.18	15.2	19
D-8	2	Sapwood	2	145	11.00	7.02	47.9	47.9	0.06	9.0	5
D-9	2	Sapwood	3	145	10.00	7.17	50.0	49.7	0.66	12.4	17
D-17	2	Heartwood	3	134	11.25	7.19	53.3	52.5	1.39	21.3	30
D-18	2	Heartwood	1	134	14.50	6.85	52.0	51.7	0.50	17.1	23
D-19	4	Slab Wood	3	140	10.00	7.23	51.9	49.3	4.87	15.5	22
D-20	2	Entire Tree	1	134	14.25	7.00	49.1	49.0	0.14	11.3	13
D-21	1	Heartwood	3	140	10.75	7.27	58.2	51.5	11.40	38.4	—
S-2	2	Spruce	*	140	10.00	4.64	48.4	48.0	0.83	11.1	11
WH-1	1	Hemlock	3	140	9.75	6.33	50.5	50.3	0.41	11.4	15

* Concave Spruce Schedule

The detailed tabulation of physical testing data is given in Appendix C. A summary of the physical properties at a freeness of 600 is given in Table VI.

TABLE VI

PHYSICAL PROPERTIES OF UNBLEACHED EXPERIMENTAL PULPS AT FREENESS 600 SCHOPPER-RIEGLER

Pulp No.	Burst Ratio %	Tear Factor %	M.I.T. Fold Double Fold No.	Apparent Density	Time to 600 Freeness Min.
D-3	112	184	1483	12.7	90
D-4	110	195	1550	14.2	94
D-5	95	135	560	13.4	61
D-6	80	177	340	14.0	80
D-7	103	177	1080	13.8	76
D-8	Not Evaluated				
D-9	113	201	1330	14.0	92
D-17	112	145	980	13.3	108
D-18	107	134	1140	14.6	95
D-19	109	193	1360	14.1	104
D-20	100	165	620	13.6	85
D-21	Not Evaluated				
S-2	142	115	1040	15.1	74
WH-1	147	133	2740	15.7	84

The primary object of this work was to find cooking conditions which result in the satisfactory pulping of sapwood, heartwood, slab wood, and the entire Douglas fir tree. Cooks No. D-3, D-7, D-19, and D-20 were selected as fulfilling these objectives, and therefore the other pulps, while useful in determining the range of variables in which satisfactory pulps may be produced, were not evaluated as completely. Curves for the physical strength development of the selected pulps are shown in Figures 10, 11, 12, 13, and 14.

FIGURE 10

BURST RATIO

Unbleached Pulps

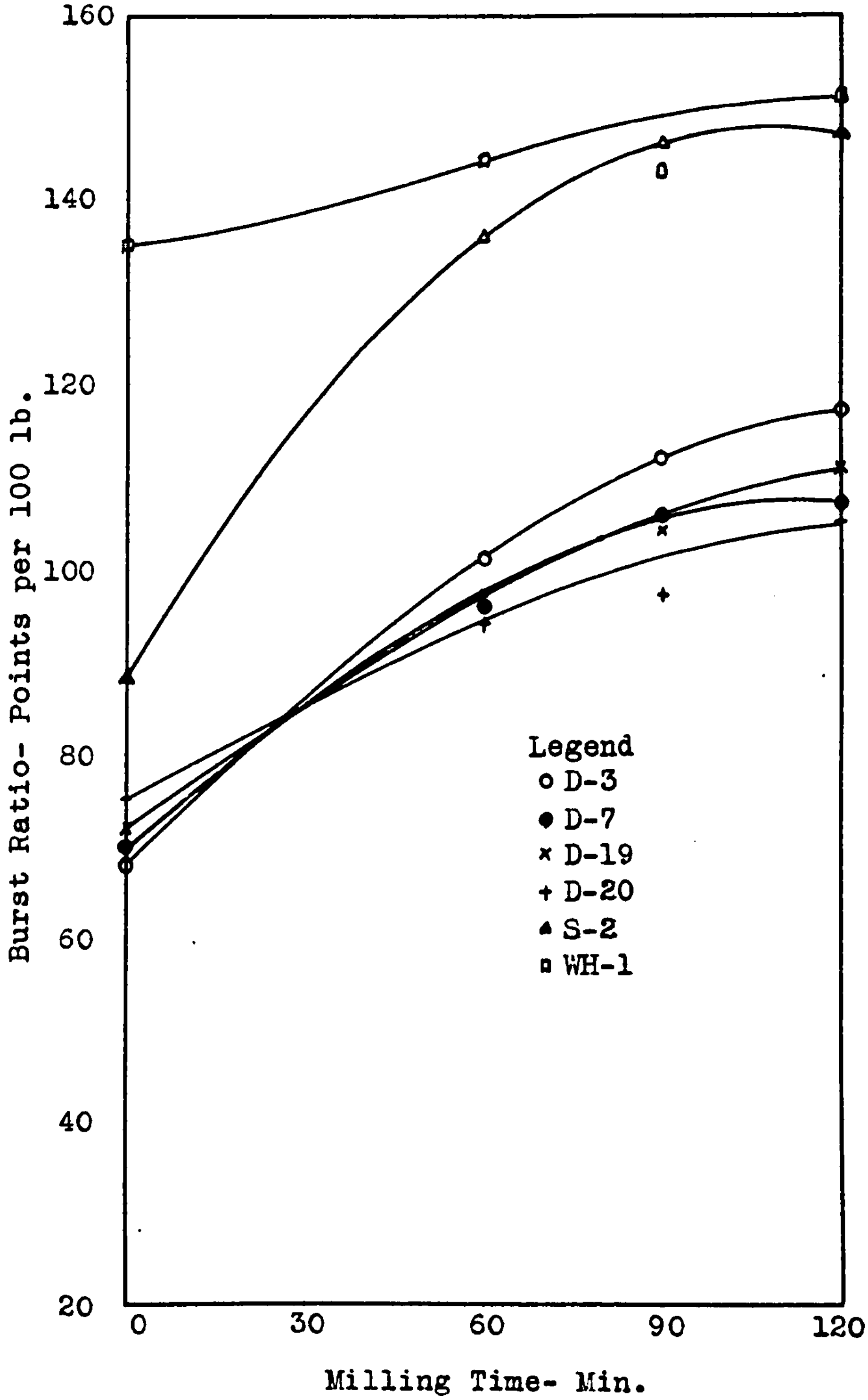


FIGURE 11

FREENESS DEVELOPMENT

Unbleached Pulps

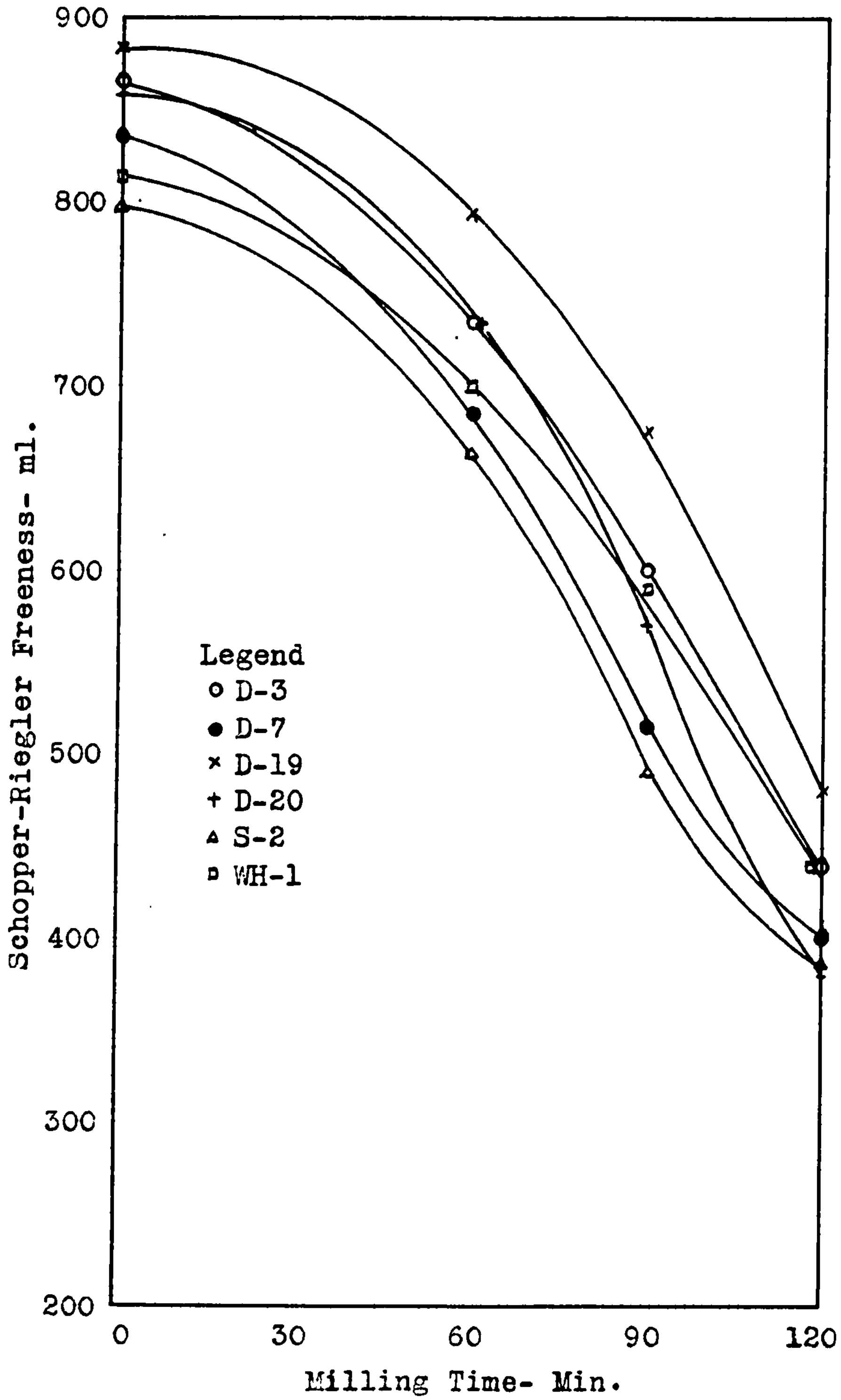


FIGURE 12

M.I.T. FOLDING ENDURANCE

Unbleached Pulps

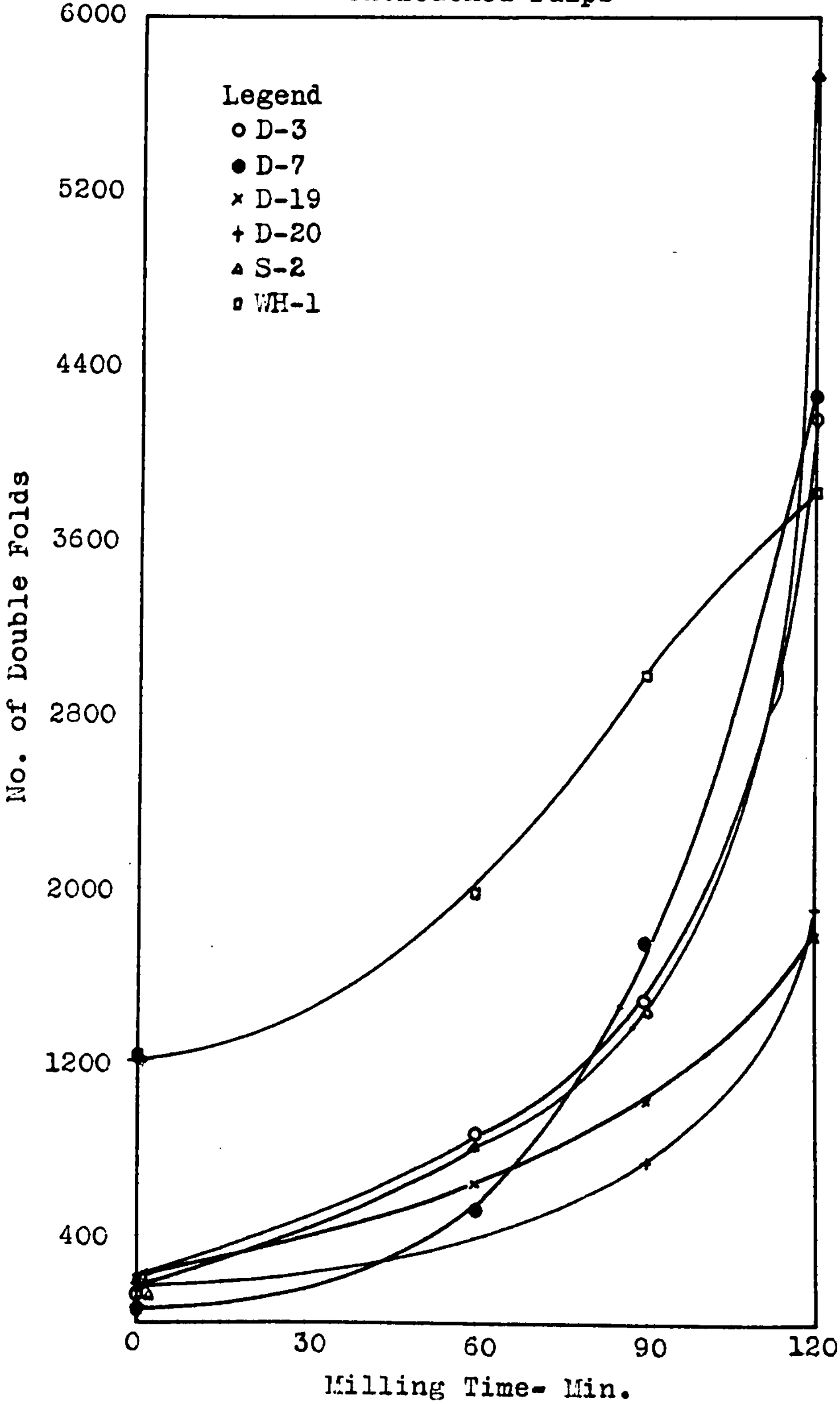


FIGURE 13

TEAR FACTOR

Unbleached Pulps

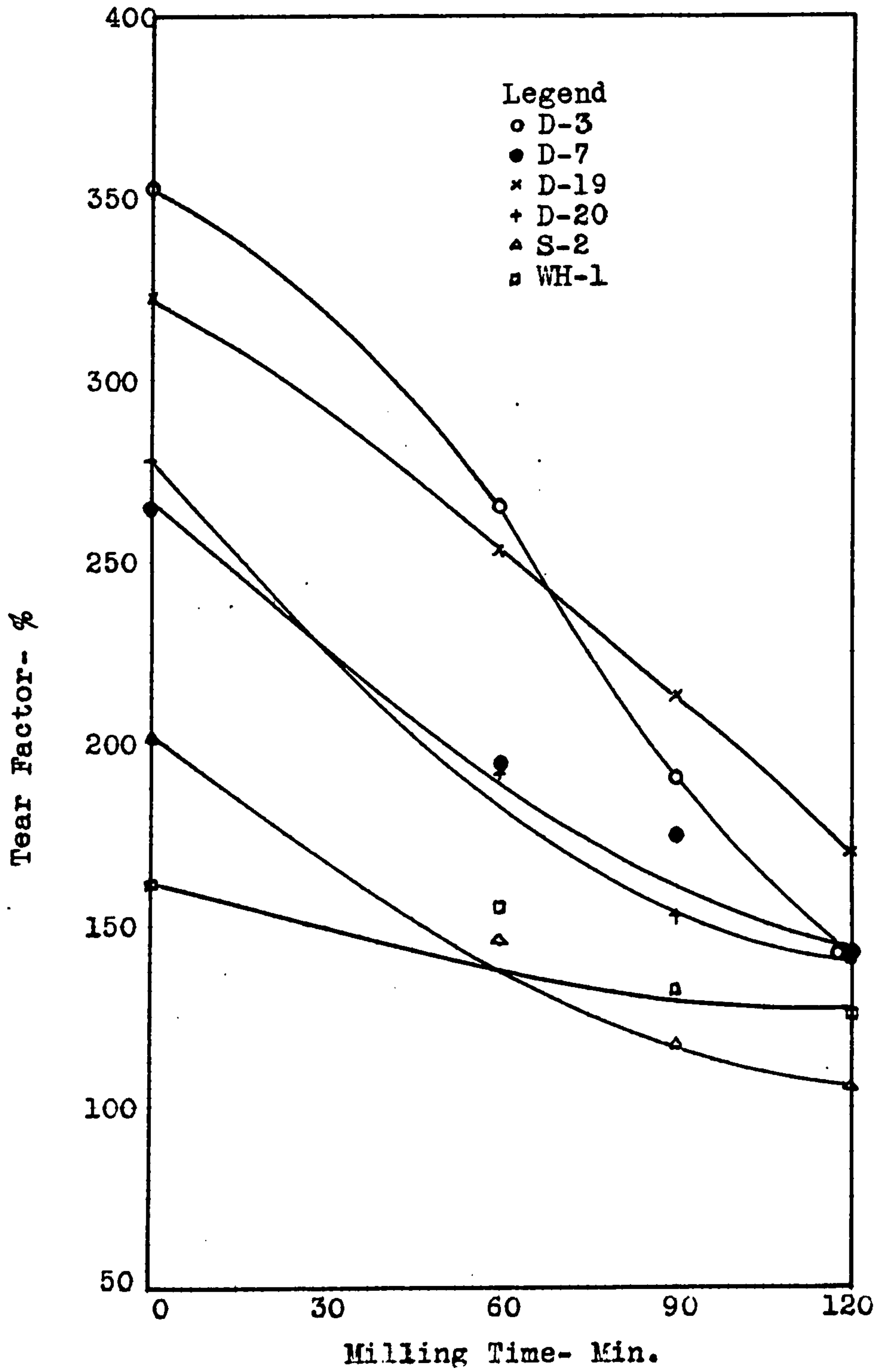
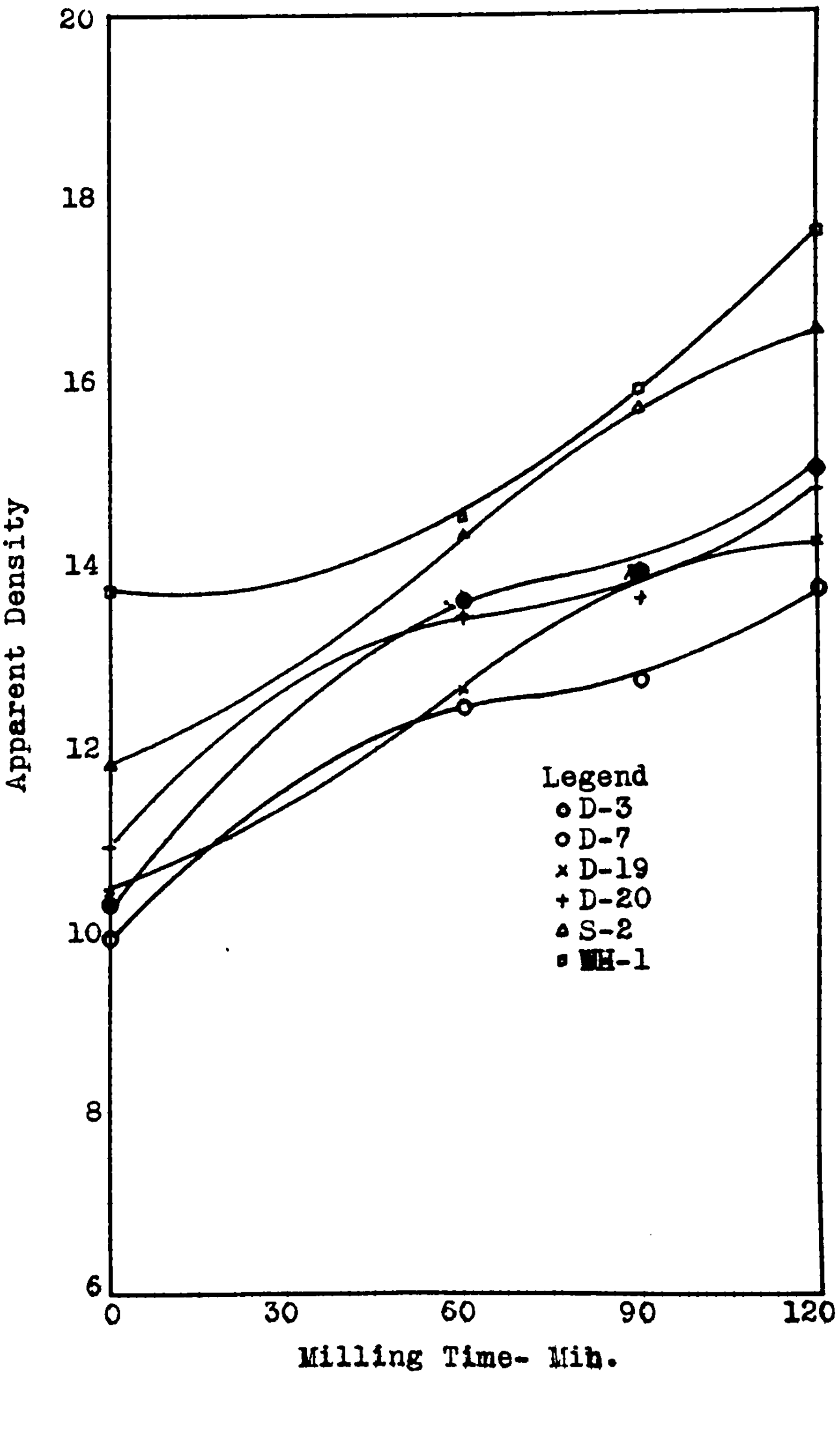


FIGURE 14

APPARENT DENSITY

Unbleached Pulps



Unbleached Douglas fir pulps, in general, had a poor color as compared with spruce and western hemlock pulps. The spectral reflectance curves of the six selected pulps were determined on the General Electric Recording Spectrophotometer. These curves are given in Figure 15. Table VII shows the I. C. I. tristimulus values for I. C. I. illuminant C as calculated from these curves by the selected ordinate method. Thirty ordinates were used.

TABLE VII

TRISTIMULUS VALUES OF UNBLEACHED PULPS

Pulp No.	Kind	x	y	Dominant Wave Length mμ	Excitation Purity %	Brightness (Visual Efficiency) %
D-3	Sapwood	0.344	0.345	580	17	51.0
D-7	Heartwood	0.357	0.366	576	25	45.8
D-19	Slab Wood	0.345	0.346	580	17	39.6
D-20	Entire Tree	0.351	0.357	578	22	46.4
S-2	Spruce	0.329	0.335	578	10	66.4
WH-1	W. Hemlock	0.333	0.332	584	10	54.0

Sample sheets of all the unbleached pulps will be found in Appendix E.

A number of the experimental pulps were bleached according to the procedure described earlier in this paper (page 28). The bleaching data are presented in Table VIII.

TABLE VIII

EXPERIMENTAL BLEACHING DATA

Cook No.	$KMnO_4$ No.	Bleach Cons.	First Stage		Second Stage		Total Bleach	Bright-ness, %
			% Cl_2	% NaOH	% Bleach	% CaO		
D-3	16.1	21	1.35	0.36	10	2.2	13.9	79.5
D-4	11.2	13	1.60	0.23	6	1.3	10.6	81.8
D-5	27.2	37	4.54	0.63	20	3.0	33.0	79.5
D-6	8.0	6	0.74	0.18	6	1.3	8.1	79.9
D-7	15.2	19	2.33	0.36	9	1.5	15.7	82.3
D-9	12.4	17	2.08	0.29	9	1.8	14.9	79.3
D-19	15.5	22	2.70	0.40	12	2.9	19.7	79.3
D-20	11.3	13	1.59	0.25	7	1.5	11.5	80.3
S-2	11.1	11	1.35	0.36	10	2.2	13.9	79.5
WH-1	11.4	15	1.84	0.29	10	2.1	15.3	79.8

The bleached pulps were evaluated for physical properties in the same manner as were the unbleached pulps, with the exception that opacity measurements were also made. The detailed data for these evaluations are found in Appendix D. Curves representing these data for the selected cooks are shown in Figures 16, 17, 18, 19, and 20. A summary of the physical properties at a freeness of 600 is given in Table IX. Sample sheets of the bleached pulps are to be found in Appendix E.

FIGURE 15

REFLECTANCE CURVES FOR UNBLEACHED PULPS

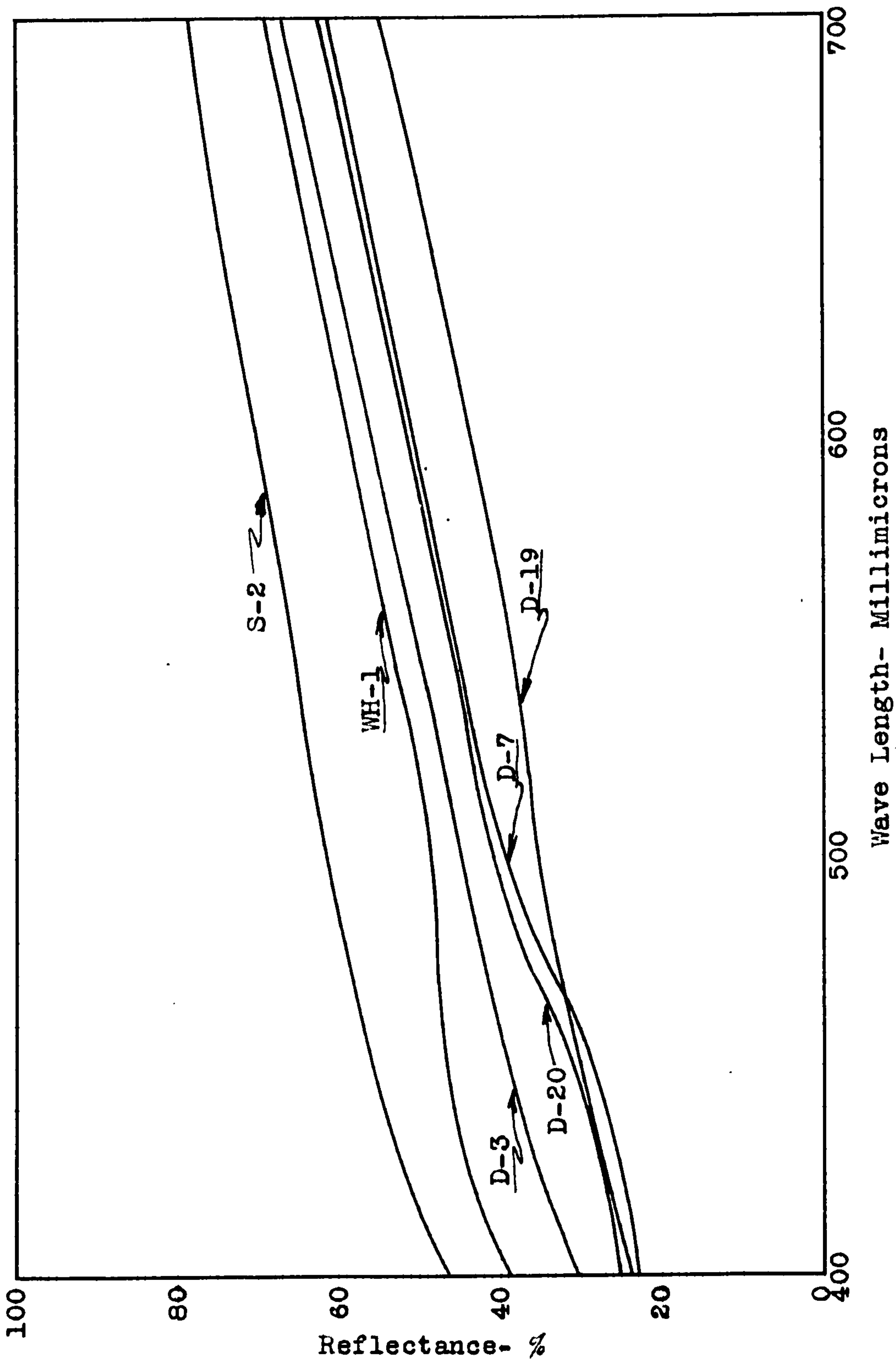


FIGURE 16

BURST RATIO

Bleached Pulps

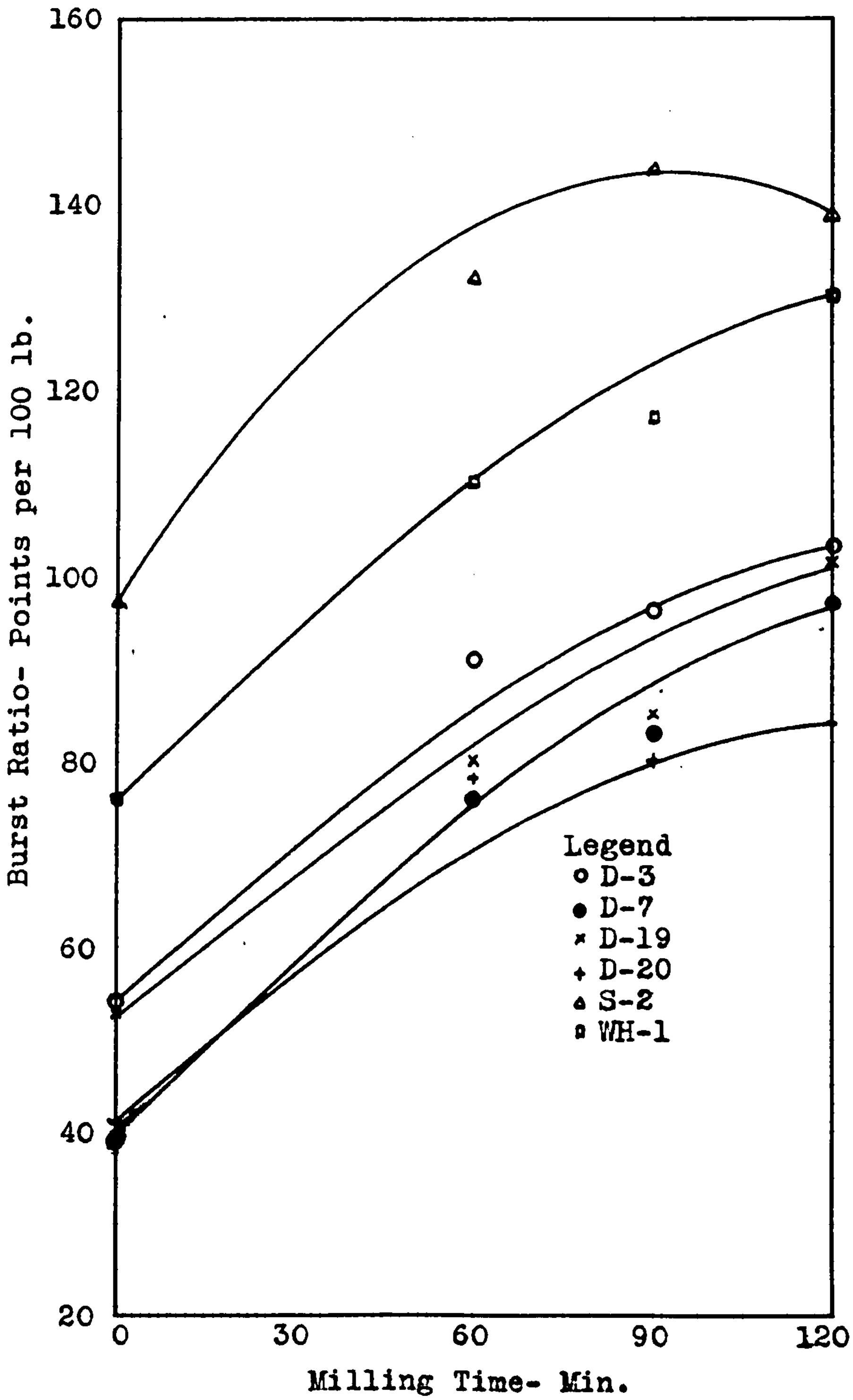


FIGURE 17

FREENESS DEVELOPMENT

Bleached Pulps

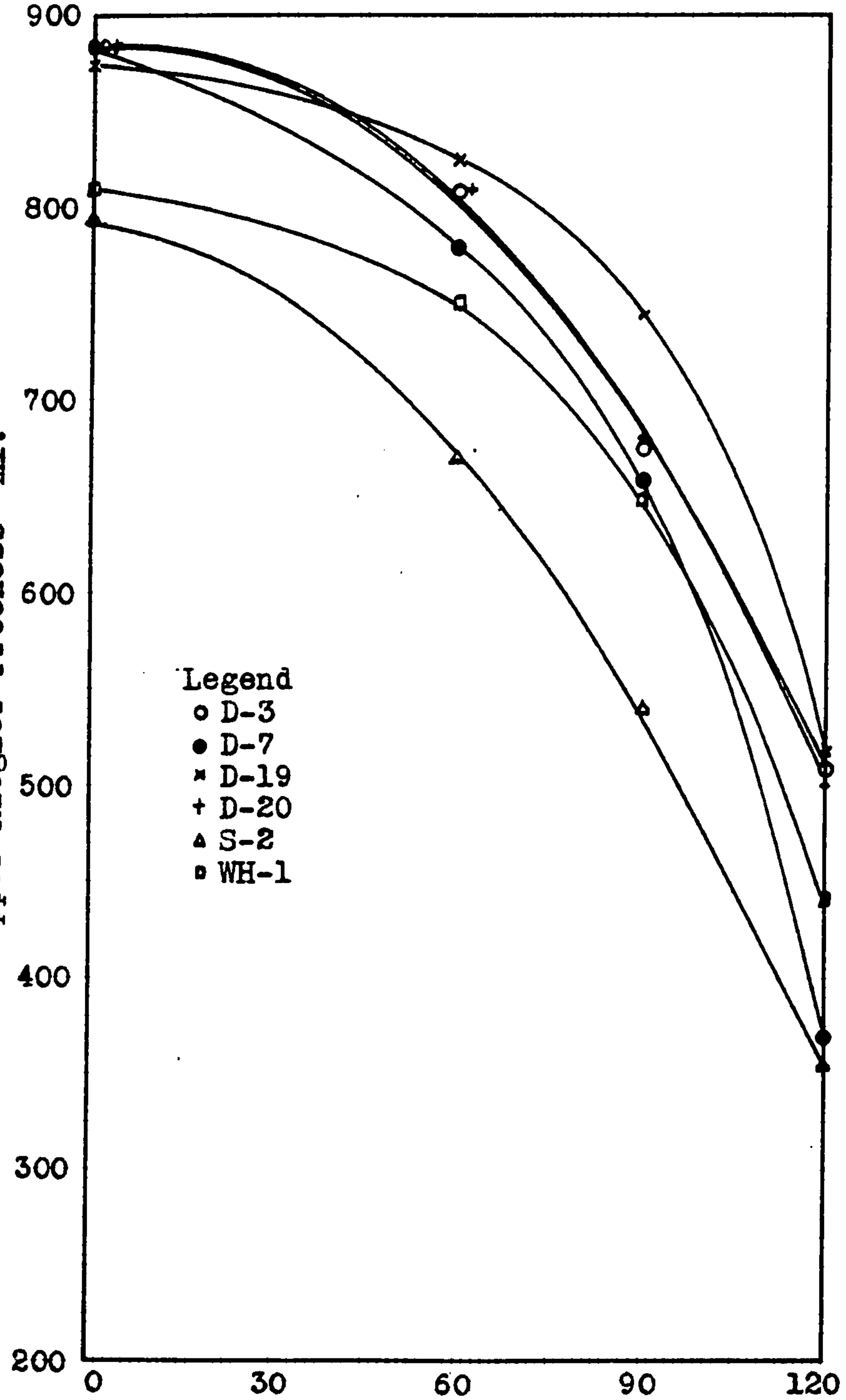


FIGURE 18

M.I.T. FOLDING ENDURANCE

Bleached Pulps

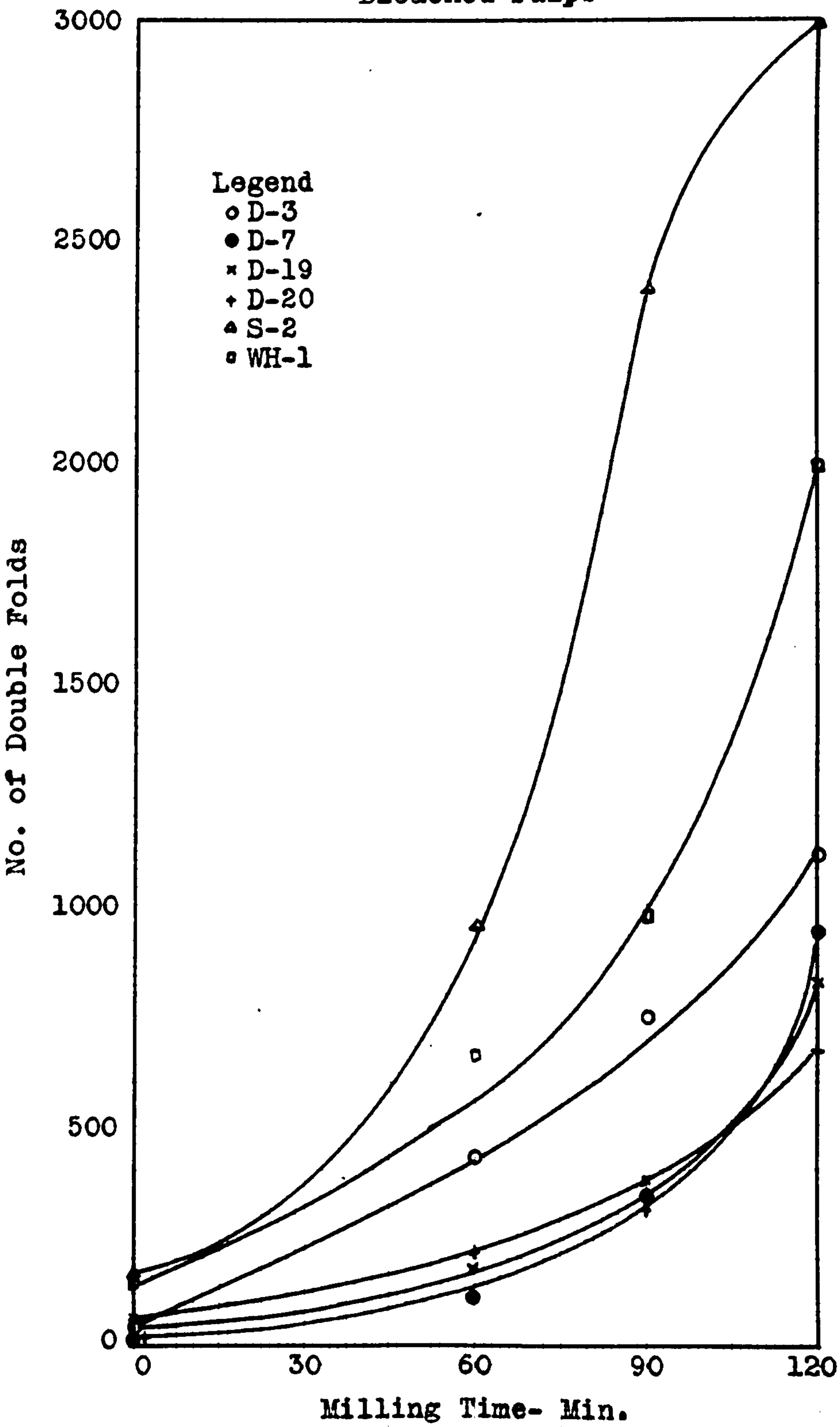


FIGURE 19

OPACITY AND APPARENT DENSITY

Bleached Pulps

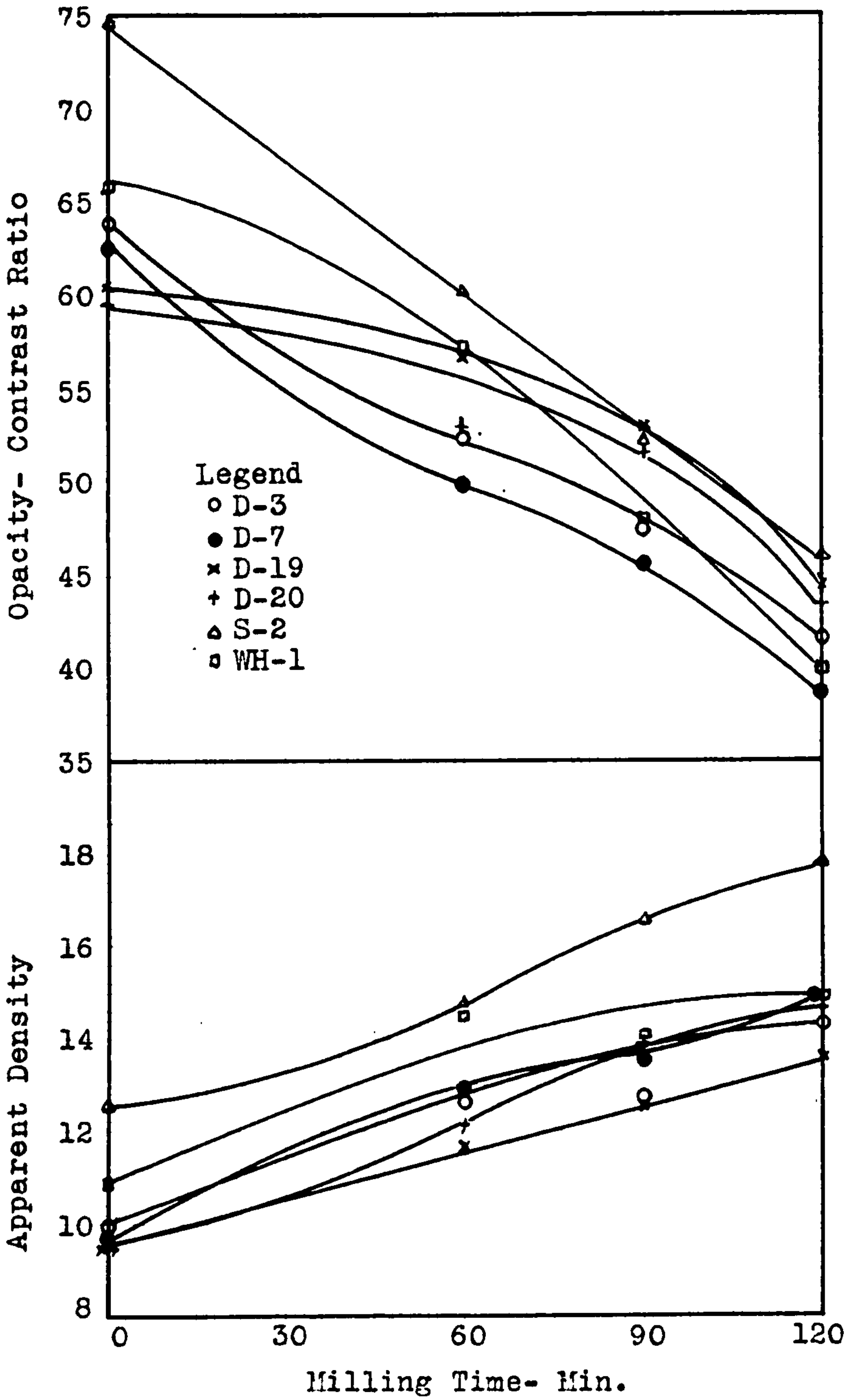


FIGURE 20

TEAR FACTOR

Bleached Pulps

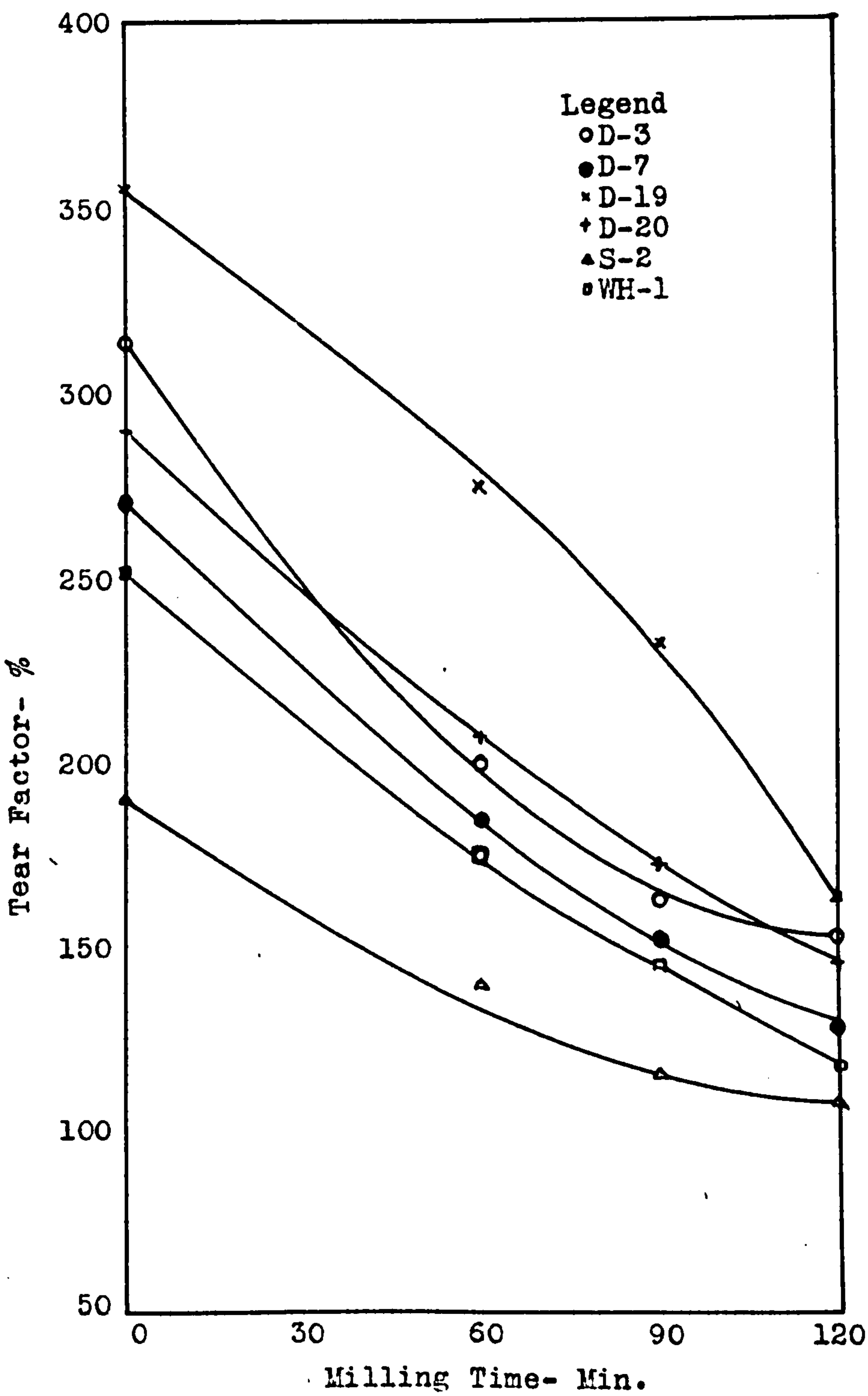


TABLE IX

PHYSICAL PROPERTIES OF BLEACHED EXPERIMENTAL PULPS AT FREENESS 600 SCHOPPER-RIEGLER

Pulp No.	Burst Ratio %	Tear Factor %	M.I.T. Fold DoubleFolds	Apparent Density	Contrast Ratio %	Time to 600 Freeness Min.
D-3	100	155	900	13.7	44.6	104
D-4	92	190	560	13.3	50.3	106
D-5	96	108	390	13.9	41.5	75
D-6	67	163	105	13.8	53.6	99
D-7	84	150	340	13.7	45.0	93
D-9	93	218	720	13.1	49.3	110
D-19	98	180	735	13.4	46.6	114
D-20	86	161	430	13.6	48.4	103
S-2	141	122	1900	15.9	55.4	78
WH-1	125	136	1275	14.3	45.8	99

Color curves of pulps S-2, D-3, and D-5 were determined on the General Electric Recording Spectrophotometer and are given in Figure 21. The I. C. I. tristimulus values for I. C. I. illuminant C were calculated, using ten selected ordinates. These data are given in Table X.

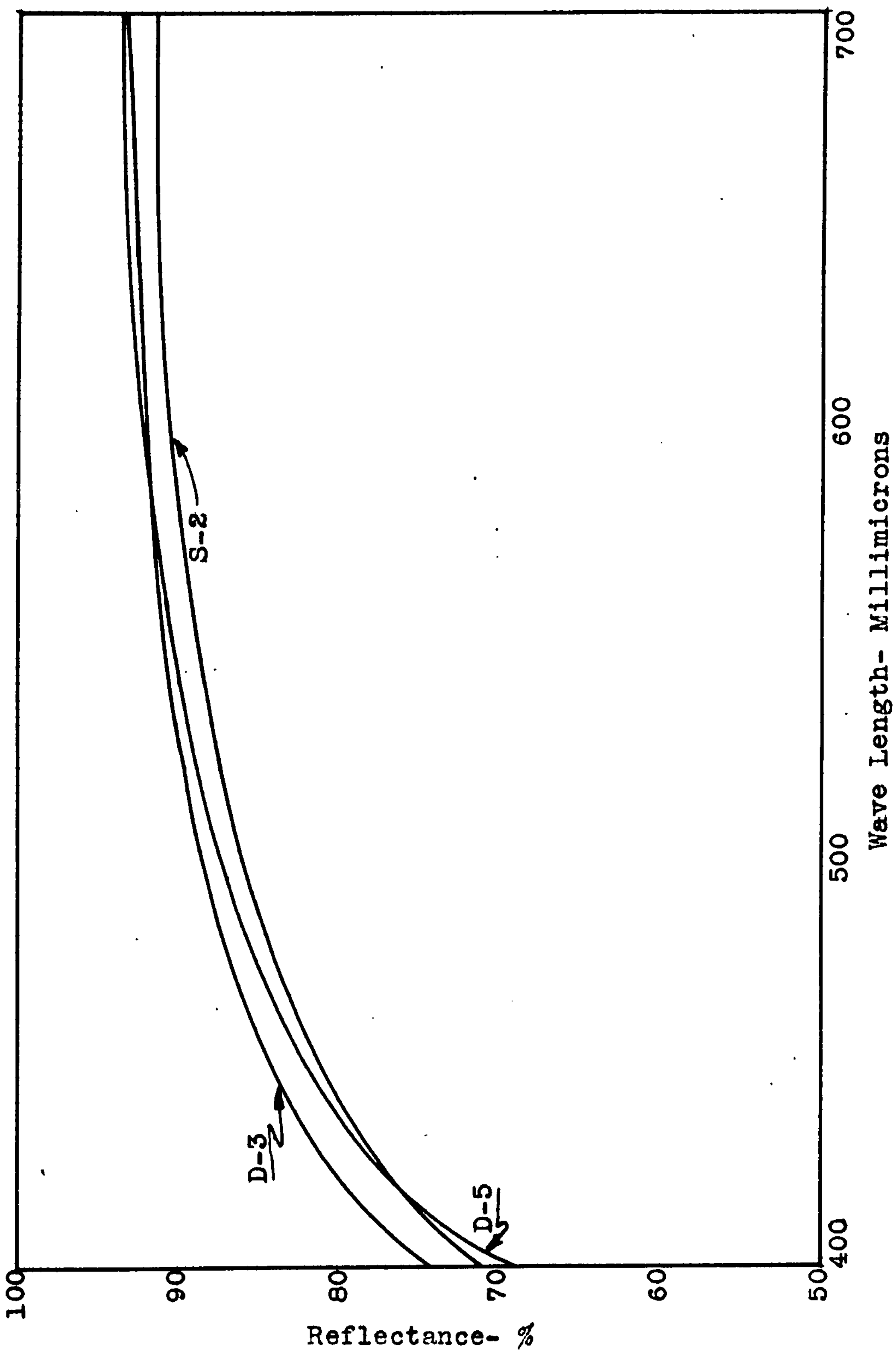
TABLE X

TRISTIMULUS VALUES OF BLEACHED PULPS

Pulp No.	Kind	x	y	Dominant Wave Length mμ	Excitation Purity %	Brightness (Visual Efficiency) %
S-2	Spruce	0.322	0.330	576	6.8	88.4
D-3	Sapwood	0.317	0.325	574	4.1	90.4
D-5	Heartwood	0.319	0.328	573	5.5	90.0

FIGURE 21

REFLECTANCE CURVES OF BLEACHED PULPS



The pulps which were selected for detailed evaluation (page 44) were subjected to chemical analysis, the results of which are given in Tables XI and XII.

TABLE XI

CHEMICAL PROPERTIES OF UNBLEACHED PULPS

Pulp No.	Alcohol-Benzene Soly. %	Ether Soly. %	Lignin	Pentosans	Alpha Cellulose %	Viscosity C.P.	Ash %
D-3	0.73	0.58	0.88	3.63	88.4	57.8	0.48
D-7	0.84	0.62	0.75	3.14	88.6	64.1	0.56
D-19	0.78	0.64	0.98	3.80	89.9	69.8	0.53
D-20	0.81	0.58	0.58	1.34	90.0	58.3	0.41
S-2	1.24	0.86	0.41	6.17	82.5	57.6	0.47
WH-1	0.95	0.80	0.64	4.75	84.1	[illegible].7	0.43

TABLE XII

CHEMICAL PROPERTIES OF BLEACHED PULPS

Pulp No.	Alpha Cellulose %	Lignin %	Copper No.	Viscosity C.P.	Pentosans %	Ether Soly. %	Ash
D-3	86.4	0.04	1.61	38.6	3.50	0.53	0.46
D-7	87.2	0.05	1.23	36.9	2.97	0.55	0.40
D-19	86.6	0.06	0.80	40.7	3.61	0.60	0.51
D-20	87.4	0.03	1.41	34.7	1.28	0.55	0.48
S-2	82.4	0.04	1.13	24.7	5.88	0.83	0.43
WH-1	82.8	0.05	1.29	28.8	4.63	0.75	0.47

Discussion of Data:

Pulping Data: It is apparent from the data presented in the preceding tables that Douglas fir wood, either as slabs or as whole logs, can be satisfactorily pulped by the sulphite process without any pretreatment and without deviating from the range of conditions used in present commercial operation. A study of Table V shows that both Douglas fir heartwood and sapwood are more difficult to reduce than either spruce or western hemlock. Heartwood is more difficultly pulped than sapwood. This increased resistance of the wood to pulping is more than offset, however, by the higher density of the wood, giving a higher yield of pulp per digester, and by the increased yield based on the weight of wood used. For example, Cook D-5 gave a screened yield of 56.7 per cent based on the weight of wood, and while the bleach consumption was 37 per cent, the amount of screenings based on the unscreened pulp was only 0.37 per cent. This is an increase of over 8 per cent in weight yield as compared with the spruce cook, S-2, and an increase of 6 per cent over the yield of pulp from western hemlock.

If a commercial digester were to be charged with the same volume of Douglas fir chips as is normally used when cooking spruce, the weight of Douglas fir chips required would amount to approximately 1.33 times the weight of spruce. Assuming the yield of Douglas fir pulp to be two per cent greater than that obtained from spruce, an amount which could easily be realised in commercial practice, the weight of pulp obtained from the Douglas fir cook would amount to 1.39 times the weight of spruce pulp. If the spruce were cooked ten hours and the Douglas fir fourteen hours, the pulp production per day for Douglas fir would be about 1.02 times that from spruce, assuming 1.25 hours for blowing and filling the digester. Thus, in spite of the longer cooking time required for Douglas fir, a change from spruce under the conditions described would result in a 2 per cent increase in pulp production at no extra cost. The cost for the Douglas fir cook would probably be lower than that for the spruce cook, as less steam would be required to cook at the lower temperature employed in pulping Douglas fir. These savings, coupled with the considerably lower cost of wood for the Douglas fir operation, show a decided economic advantage for this species.

It is possible to cook Douglas fir sapwood to almost any desired bleach consumption without danger of precipitation. The low screenings content of those pulps which were burned indicated that this occurred at the end of the cook, after precipiatation of the liquor started. If burning had taken place in the earlier stages, it would have been manifested by a fairly high amount of screenings containing burned chips. This was not the case. When dealing with the heartwood of this species, it was found that the major problem was to cook the pulp to the desired degree before precipitation of the liquor occurred. Using Schedule No. 1, precipitation was found to take place somewhere between 14.5 and 14.75 hours. The bleach consumption of Cook D-7, cooked 14.75 hours, was 19 per cent, while that for Cook D-18 after 14.5 hours was 23 per cent. Apparently this is the limit to which heartwood may be pulped under these conditions. Using Schedule No. 3, with a maximum temperature of 134°C., the bleach consumption of the pulp (D-17) was 30 per cent after cooking 11.25 hours, the point at which precipitation was first noticed in the liquor. This pulp had 1.39 per cent screenings as compared to 0.50 per cent for Cook D-18. This indicates that not only the maximum temperature, but also the temperature schedule, has some effect on the point where precipitation begins. Schedule No. 1 had a very low rate of temperature rise from 110°C., while Schedule No. 3 employed a much steeper temperature curve.

It was found that heartwood could not be completely pulped at 140°C. before precipitation of the liquor occurred. Cook D-21 illustrates this point. After 10.75 hours, this cook showed precipitation in the liquor, and was blown. The pulp was exceedingly raw, having a permanganate number of 38.4 with 11.4 per cent screenings. From this, it is seen that the problem of p lping Douglas fir hinges on a satisfactory pulping of the heartwood.

The problem in the pulping of Douglas fir heartwood is the precipitation of the cooking liquor and the resultant burning of the pulp. The cause of this phenomenon has not been definitely established, although several factors which influence its occurrence are known. Gishler and Maass (25), in their studies on the system calcium oxide-sulphur dioxide-water, observed the precipitation of calcium monosulphite from the solutions at temperatures considerably below the normal sulphite cooking range. They found that precipitation temperatures were increased by a decrease in lime content or by an increase in sulphur dioxide concentration of the liquor. With four per cent sulphur dioxide and two per cent lime, these workers found the precipitation temperature to be 70°C., while with four per cent sulphur dioxide and only one per cent lime, the temperature at which precipitation occurred was raised to 114°C. Similarly, with six per cent sulphur dioxide and two per cent lime, the temperature was 102°C., and with one per cent lime it was increased to 136°C. In the presence of spruce wood, these temperatures were increased considerably and, although the mechanism by which this increase was obtained was not determined, the possibility was advanced that the colloidal condition of the wood had a deterent action on the precipitation.

It may be possible, since calcium sulphite was observed in the precipitate found in the liquor from Cook D-6, that the precipitation temperature of the liquor was exceeded in those cooks which exhibited burning. If this were true, then Douglas fir wood does not elevate the precipitation temperature to the same extent as does spruce. Also, the heartwood of Douglas fir does not have the same elevating tendency as the sapwood.

Assuming this property of sulphite acid to be the cause of the precipitate, two remedies are suggested. First, the maximum temperature of the cook may be reduced. In comparing Cook D-21 with D-18, it is seen that a reduction in the maximum temperature from 140°C. to 134°C. does have a beneficial effect, in that precipitation occurs much later in the cook at the lower temperature. With the same maximum pressure, a reduction in temperature also results in an increase in sulphur dioxide concentration in the liquor, a factor which should be helpful in eliminating the difficulty. There is a limit to this reduction in temperature, however, in that a sufficiently high temperature must be used to complete the cook in a reasonable length of time.

The second remedy involves increasing the concentration of sulphur dioxide in the liquor by increasing the digester pressure. This was not tried in the present work, but is an interesting possibility.

Another factor which may have considerable effect on this tendency of the liquor to precipitate is the relative rates at which calcium oxide and sulphur dioxide are consumed by the wood. If the sulphur dioxide is removed from the liquor, by reaction with the lignin of the wood and by digester relief, at a much greater rate than lime is consumed, the precipitation temperature would be lowered. This may well be the case.

The effect of small amounts of impurities in the cooking acid in promoting the auto-oxidation and reduction of calcium bisulphite to calcium sulphate and free sulphur has been demonstrated. Selenium, for example, is very troublesome in this respect; other compounds having similar tendencies are the members of the pinene series. Since the oleo-resin of Douglas fir contains limonene, as well as other members of this series (30), this may be a contributing factor, if not the only cause, of the precipitation. If this is true, there is no possibility of completely eliminating precipitation by the adjustment of cooking variables alone. Also, a larger amount of these resins would be found in the heartwood than in the sapwood.

Since both calcium sulphite and calcium sulphate were found in the precipitate, it seems reasonable to conclude that precipitation in the case of Douglas fir heartwood is caused by a combination of two reactions: first, the precipitation of calcium sulphite caused by exceeding the precipitation temperature of the liquor, and second, the precipitation of calcium sulphate and sulphur caused by the presence of compounds of the pinene series. As has been demonstrated by Cooks D-5, D-7, D-18, and D-20, this precipitation can be avoided by the careful choice of cooking conditions. Using conditions reported for these cooks, pulps of a reasonable bleachability may be obtained before the precipitation point is reached.

While it is true that most of the Douglas fir cooks made had comparatively high bleachabilities, this fact is more than offset by the increased yields obtained from this species.

<u>Physical Properties</u>: A study of Table VI shows the same trends of pulp quality for Douglas fir pulps when compared with spruce as were

predicted from the physical evaluation of the chlorine pulps. Cook D-6, which was badly burned, showed a much lower burst and fold than did the other unbleached Douglas fir pulps. Cook D-5, a very raw cook, did not develop as high a burst as those pulps cooked to lower bleach consumptions. This point has also been observed in very raw spruce pulps, and cannot be considered as abnormal. Referring to Table XI, a general correlation between fold and pentosan content may be noted, and to a somewhat smaller extent, this is also true to burst. This tendency has been discussed in connection with the chlorine pulps, and will not be elaborated here.

Unbleached Douglas fir pulps develop their physical properties more slowly than do spruce and western hemlock (see Figures 10-14). The burst ratios were found to range from 80 to 113 per cent at 600 freeness, but in no case was a distinct maximum observed in the curve of burst ratio against milling time. Presumably, longer milling would result in increased burst ratios, although at freenesses below 400.

Folding endurance curves show these same tendencies. In general, Douglas fir pulps develop higher folding strengths than spruce at the higher freenesses, but they do not show as sharp a rise at the lower freenesses as is the case with the spruce pulp.

All Douglas fir pulps exhibited higher tearing strengths than either the spruce or western hemlock pulps. The curves of tear factor against milling time show the tear factor for Douglas fir pulps to decrease much more slowly with increased milling than was observed for the spruce pulp. This high initial tear and slow loss on milling is probably due in a large measure to the long fibers of this species.

A low apparent density is a typical property of these Douglas fir pulps. The pulps would therefore be useful in producing bulk in a sheet, a property which is very desirable in many grades of paper.

Because of their poor color, unbleached Douglas fir pulps would probably find application only in those grades of paper where color is not important, or in heavily dyed sheets. Their physical properties indicate their use in such grades as wrapping and cover papers.

Chemical Properties: The two most striking features shown by the chemical analyses of unbleached Douglas fir pulps (see Table XI) are the high content of alpha-cellulose and the low pentosan content. Values for alpha-cellulose (corrected for lignin) range from 88 to 90 per cent, 6 to 8 per cent higher than those for spruce and western hemlock.

Pentosan contents as low as 1.4 per cent were observed, while the maximum found was 3.8 per cent. Both spruce and western hemlock had pentosan contents considerably in excess of these values.

Although many investigators have described Douglas fir as a resinous wood, actual analysis of the wood shows this idea to be erroneous. The alcohol-benzene solubility of both the heartwood and sapwood of this species is lower than that found for spruce or western hemlock, and this fact is also observed in the analysis of the unbleached pulps. The ether solubility of unbleached Douglas fir pulps is also lower than that for spruce or western hemlock, indicating that unbleached Douglas fir pulps would cause little or no pitch trouble on the paper machines.

The lignin content of these pulps is about that expected from the bleach consumptions, although the correlation between bleachability and lignin content is only a general one.

The chemical analysis of these unbleached pulps indicate that they would probably serve very well as raw material for high quality rayon pulps.

Bleaching Data: The data obtained during the bleaching of the experimental pulps (see Table VIII) show that with only a two-stage procedure, it is possible to bleach Douglas fir sulphite pulp to a brightness as high as 80 per cent or more without any difficulty whatsoever. The amounts of bleach required in general were approximately the same as were required by the spruce pulp. Only the very raw Douglas fir pulps required more. Sapwood is no harder to bleach than either spruce or western hemlock, a finding which contradicts reports made in the literature. Further, the amounts of caustic soda and lime used in bleaching were not excessive for the Douglas fir pulps. A study of the data in Table VIII will show that in every case, with the exception of Cook D-6 which was burned, the amount of bleach required to reach a brightness of 80 per cent was appreciably less than the bleachability, while the spruce pulp required more to reach the same brightness. This seems to indicate that less chlorine is required for the removal of a given amount of coloring matter from Douglas fir pulps than is the case with either spruce or western hemlock.

Physical Properties of Bleached Pulps: The data in Table IX show that there was a small amount of degradation with respect to physical properties during bleaching. Burst and fold show a decrease over those found for the unbleached pulps, while the tear is somewhat higher. In general, the opacity of the Douglas fir pulps is lower than that for spruce but higher than that for western hemlock.

Bleaching tends to make the rate of hydration of all the pulps somewhat slower, but in the order as observed for the unbleached pulps. All strength curves for the bleached pulps possess normal shapes, as may be seen in Figures 16 to 20.

From a papermaking standpoint, bleached Douglas fir pulps would be desirable blending pulps for almost any grade of paper. The extremely long fibers of Douglas fir pulps may cause some difficulty on commercial paper machines from the standpoint of formation. The tendency of long fibers to flocculate into knots or bundles, giving rise to wild sheet formation, is known. It may be found necessary to use extreme jordan action in order to decrease the fiber length sufficiently for good sheet formation.

On the other hand, if used as a blending pulp, bleached Douglas fir sulphite would impart desirable properties to many grades of paper. Of primary interest in this respect are the high tear and low apparent density of these pulps. The use of Douglas fir pulps in such grades as mimeo, bond, ledger, printing, folding raw stock, wrapping, and cover would probably result in a cheapened furnish possessing improved physical properties.

Chemical Properties of Bleached Pulps: The chemical analyses indicate that the bleached Douglas fir pulps, while showing slight degradation from the bleaching operation, possess all the properties of spruce pulps after a mild alpha treatment. The viscosity is considerably higher than that for the spruce and western hemlock pulps, and the large increase in alpha-cellulose content over the other two species is again apparent. The pentosan content is somewhat lower than for the unbleached pulps, but not significantly so. The differences between the pentosan contents of the species are in the same order as was found for the unbleached pulps. The ether solubility of the bleached Douglas fir pulps is lower than that for spruce and western hemlock, while there are no significant differences between the lignin and ash values. Copper numbers are somewhat high for bleached pulps, indicating that some slight degree of degradation occurred during the bleaching operation. Nevertheless, the analytical data are comparative, as the same procedure was used for all pulps.

Because of their high alpha-cellulose content, the use of bleached Douglas fir pulps for the manufacture of cellulose derivatives is suggested. While no data are available on this point, the chemical analyses of the pulps indicate that any purification treatment need not be as extensive as is the case with other pulps. This particular application of Douglas fir pulps is certainly worth further investigation.

THEORETICAL WORK

INVESTIGATION OF DOUGLAS FIR LIGNIN

Object:

The object of this part of the work was to prepare a number of lignin derivatives from Douglas fir heartwood and sapwood in the hope that some significant difference might be found which would explain the difference in behavior of the two types of wood during pulping. All the lignin compounds were to be compared with similar compounds prepared from spruce lignin. It has been suggested by Bailey (7) that the lignin of Douglas fir differs from that of spruce only in the degree of polymerization, while Beuschlein (6) reports that the increased difficulty in pulping Douglas fir with soda base liquor can only be attributed to differences between the lignin of this species and that of spruce.

Wood Preparation:

Sawdust from both sapwood and heartwood was ground to pass a 40-mesh screen and be retained on a 60-mesh screen. The wood meal so prepared was then extracted with the constant boiling mixture of alcohol and benzene (1:2) until five milliliters of the solvent (in contact with the wood meal) left practically no residue on evaporation. The extraction was carried out in a large soxhlet extractor, as much as 300 grams of the wood meal being treated at one time. The extracted wood meal was then air-dried and placed in moisture tight Mason jars pending its use for preparation of the lignin compounds.

Sulphuric Acid Lignin:

The lignin residue from the determination of lignin in the wood samples was saved as sulphuric acid lignin. This was isolated according to Institute Method No. 13-1. A methoxyl determination was made on this lingin using the method of Viebock and Schwappach (26). The following data were obtained:

	Sapwood	Heartwood	Spruce
Yield, %	29.40	28.60	27.60
Methoxyl, %	14.00	14.40	14.80
Ash, %	0.52	0.48	0.55

As may be seen, the yield from the various types of wood is not significantly different. This may also be said of the methoxyl and ash values.

Willstätter Lignin:

Willstätter lignin was prepared from both heartwood and sapwood by the method of Kalb and Lieser (27). Ten grams of the wood meal were treated with 200 milliliters of hydrochloric acid (specific gravity 1.222 at 0°C.) at a temperature of 1 to 5°C. The suspension was shaken vigorously from time to time over a period of two hours, the temperature being allowed to rise to that of the room during this period. The mixture then was diluted with 65 grams of water in the form of finely cracked ice and allowed to stand at room temperature for 18 hours. At the end of this period, another 65 gram portion of water was added, and the lignin filtered off on a Buchner funnel. The precipitate was washed with 200 milliliters of hydrochloric acid (1:1), followed by a thorough washing with water. For purification, the lignin was boiled in 200 milliliters of water with the gradual addition of sodium carbonate until the supernatant liquor was neutral to litmus paper. The solution was removed by filtration. The precipitate was again treated with 200 milliliters of boiling water for a ten-minute period and filtered. This last treatment with boiling water was repeated three more times, and the product was then dried in a vacuum desiccator over sodium hydroxide and sulphuric acid. Methoxyl and ash determinations were made on the products, with the following results:

	Sapwood	Heartwood	Spruce
Yield, %	31.00	30.00	25-30
Methoxyl, %	14.40	14.20	14.5-15.5
Ash, %	0.21	0.22	

<u>Thioglycolic Acid Lignin:</u>

Thioglycolic acid lignins from Douglas fir heartwood and sapwood were prepared according to the method of Holmberg (<u>28</u>). Forty grams of the wood meal were treated with a mixture of 30 grams of thioglycolic acid and 400 milliliters of 2 <u>N</u> hydrochloric acid. The mixture was heated on a boiling water bath for a period of seven hours, at the end of which time the sapwood mixture had taken on a very red color, while the heartwood was a light orange. Spruce under these conditions shows only a very light buff color. The suspension was filtered and washed thoroughly with distilled water, and the filter cake was air dried. The sapwood meal had become a dark red, while the heartwood was a light orange color. The air-dried material was then mixed with 200 milliliters of ethyl alcohol and allowed to stand for 48 hours. The alcohol was then filtered off. The filtrate from the sapwood had a bright orange color, while that from the heartwood was a light straw color. The filter cake was again air-dried, then treated with 400 milliliters of distilled water containing 8 grams of sodium hydroxide, and allowed to stand over night. This dissolves the lignin. The pulp was filtered off and washed with distilled water until the volume of liquid reached 1 liter. The filtrate was then acidified with 20 milliliters of concentrated hydrochloric acid and the precipitated lignin removed by filtration. The lignin was washed with distilled water until acid free as shown by Congo red paper. The filtrate from the sapwood lignin was yellow in color, while that from the heartwood was almost colorless. The crude sapwood thioglycolic acid lignin was a deep red color, while that from the heartwood was a light buff.

The crude lignins were purified by dissovling them in dioxane, filtering, and precipitating the dioxane solution in ether. The precipitated lignins were washed twice with ether and twice with petroleum ether. The sapwood compound was a dull red powder, while the heartwood thioglycolic acid lignin was obtained as a cream-colored powder.

Since the solutions of the crude thioglycolic acid lignins appeared to be of widely different viscosities, it was thought that viscosity measurements of the dioxane solutions might give a rough idea of the relative degree of polymerization of the two lignins. For this reason, the viscosity of four per cent solutions of both heartwood and sapwood thioglycolic acid lignins in dioxane were determined by means of an Ostwald tube. However, the purified compounds were found to have identical viscosities, and no conclusions could be drawn from this data.

Methoxyl determinations were made on the purified thioglycolic acid lignins, which were found to be ash free. The data obtained follows:

	Sapwood	Heartwood	Spruce
Yield, crude, %	27.5	27.00	30.0
Yield, purified, %	24.0	24.00	25.5
Methoxyl, %	11.5	11.80	11.9

Phenol Lignin:

Phenol lignins were prepared from Douglas fir heartwood and sapwood according to the procedure described by Buckland, Brauns, and Hibbert (29). Pure anhydrous phenol (750 grams) was placed in a one-liter Claissen flask provided with a dropping funnel and a mechanical stirrer. The phenol was heated to 80°C. and 80 grams of wood meal added. An anhydrous ether solution (135 milliliters) of 5.4 grams of hydrogen chloride was added during a period of 10 minutes through the dropping funnel, the end of which was placed below the surface of the molten phenol. The mixture was stirred constantly during this time, and the temperature maintained between 80 and 90°C. The color of the reaction mass changed gradually from a light yellow to a deep greenish black. This same color change was noted for both the heartwood and the sapwood. At the end of one-half hour, the phenol was distilled off under reduced pressure, the temperature being slowly raised to 100°C. The residue was a brownish black tar, which became brittle at room temperature. This tar was dissolved in 150 milliliters of acetone and methyl alcohol (1:1) and the residual cellulose removed by filtration. The cellulose was washed thoroughly with methyl alcohol and acetone, and the washings combined with the filtrate. The solution so obtained was concentrated, and any phenol which was present was removed by distillation under reduced pressure. The residue from this operation was dissolved in 150 milliliters of the acetone-methyl alcohol solvent and the lignin precipitated by adding the solution dropwise, with vigorous stirring, to four liters of distilled water. The light gray solid which separated was filtered off and thoroughly washed with distilled water. The total yield of dry crude phenol lignin was 46 grams for the heartwood and 45 grams for the sapwood, equivalent to 57 and 56 per cent, respectively, of the original wood weight. Spruce, when treated as above, yields 67 per cent of a product having a methoxyl content of 6.4 per cent. The Douglas fir crude phenol lignins had methoxyl contents of 7.1 per cent for the heartwood product and 7.2 per cent for the sapwood product.

Fractionation of Crude Phenol Lignin

The crude phenol lignins were separated into an ether-insoluble fraction (Phenol Lignin A) and an ether-soluble fraction (Phenol Lignin B) by the following procedure:

Isolation of Ether-insoluble Phenol Lignin A: Twenty grams of the crude phenol lignins were dissolved in 200 milliliters of anhydrous, glycol-free dioxane, giving a very dark chocolate-brown solution. This was filtered and added dropwise to seven to eight times its volume of anhydrous ether with vigorous stirring. The lignin was precipitated as a fine light gray powder, while the supernatant ether solution was dark reddish-brown in color. It was then centrifuged, washed with three portions of dry ether and finally with two portions of petroleum ether. The ether-insoluble phenol lignin A was obtained as a very fine gray colored powder. Methoxyl contents after the first precipitation were 10.2 per cent for the heartwood compound and 10.8 per cent for the sapwood derivative.

These compounds were subjected to a second purification as described above. The methoxyl contents after this step were 10.5 per cent for the phenol lignin from heartwood and 10.7 per cent for the sapwood compound. The value reported for spruce was 10.4 per cent. Calculated for $C_{42}H_{32}O_6(OCH_3)_4(OH)_5(OC_6H_5)$. $3C_6H_5OH$ (1216.5): OCH_3, 10.2 per cent. The yields were 65 per cent of the crude product.

Isolation of Ether-soluble Phenol Lignin B: The dark brown ether solutions remaining after the removal of the ether-insoluble fraction A were evaporated to dryness under reduced pressure to remove the last traces of dioxane. The residues were redissolved in ether, leaving behind a small amount of a tarry substance. The ether solutions were again evaporated under reduced pressure to dryness, and the resultant tar dissolved in methyl alcohol. The lignin compounds were precipitated by dropping the methyl alcohol solutions into distilled water, the precipitates being obtained as very fine tan powders. They were separated by filtration and washed with distilled water, dried in a vacuum desiccator over sodium hydroxide and sulphuric acid, and weighed.

The final products were obtained in identical yields of 35 per cent of the crude phenol lignins. Methoxyl contents were 4.4 per cent for both the sapwood and heartwood compounds. Calculated for $C_{42}H_{32}O_6(OCH_3)_5(OH)_4(OC_6H_5)$. $15C_6H_5OH$ (2358.4): OCH_3, 5.4 per cent.

The methoxyl content of spruce phenol lignin B was found to be 5.3 per cent. However, this deviation from the values found for the Douglas fir preparations may not be significant, since the method of purification described above is not the same as that used for the spruce compound, which was precipitated from a dioxane solution by dropping it into dry petroleum ether.

Acetylation of Ether-insoluble Phenol Lignin A

Phenol lignin A (5 grams) was dissolved in 25 milliliters of dry pyridine and 15 milliliters of acetic anhydride added. The dark reddish brown solution was allowed to stand at room temperature for 36 hours, after which it was poured over finely cracked ice. After the ice had melted, the light brown precipitate was filtered off, washed free from pyridine and acetic acid, and dried. Yields obtained were 5.5 grams for the sapwood product and 5.5 grams for the heartwood compound. Methoxyl contents were 8.6 per cent for the sapwood product and 8.4 per cent for the heartwood product. Acetyl determinations showed 24.3 and 24.6 per cent, respectively, for the sapwood and heartwood compounds. A similar compound prepared from spruce by the same procedure showed 7.9 per cent methoxyl and 23.0 per cent acetyl. Calculated for $C_{42}H_{32}O_6$ $(OCH_3)_4$ $(OCOCH_3)_5OC_6H_5 \cdot 3C_6H_5OCOCH_3$ (1552.7): OCH_3, 8.0 per cent; $COCH_3$, 22.2 per cent.

Methylation of Ether-insoluble Phenol Lignin A with Diazomethane

Two grams of phenol lignin A were dissolved in 25 milliliters of dioxane and a slow current of diazomethane (prepared by slowly dropping 12 milliliters of a sodium glycolate solution, containing 6 per cent sodium, into 6 milliliters of nitrosomethylurethan) led into the solution. After about an hour, the evolution of nitrogen was observed, and the solution became noticeably lighter in color.

The reaction mixture was allowed to stand overnight and a small amount of the solution was taken for a methoxyl determination. This sample was filtered to remove a small amount of a gelatinous precipitate (presumably a polymer of diazomethane) and precipitated by dropping it into dry ether. The precipitate was washed twice with ether and twice with petroleum ether. Methoxyl contents after this first methylation were 20.2 per cent and 20.0 for heartwood and sapwood compounds, respectively.

The main portion of the reaction mixture was again methylated, using half as much diazomethane as in the first methylation and after standing overnight, was heated to 50°C. for 10 minutes, filtered, and precipitated as described above. Methoxyl contents after the second methylation were 21.7 and 21.8 per cent, respectively, for the heartwood and sapwood compounds. A third methylation of the heartwood product did not give a higher methoxyl content; hence it was concluded that these derivatives were completely methylated with respect to diazomethane after two methylations. Methylated spruce phenol lignin A prepared as above had a methoxyl content of 21.6 per cent. Calculated for $C_{42}H_{32}O_6(OCH_3)_6(OH)_3(OC_6H_5) \cdot 3C_6H_5OCH_3$ (1286.6): OCH_3, 21.7 per cent.

Summary:

The data collected on the lignin compounds described in the preceding pages are summarized in the following table.

TABLE XIII

SUMMARY OF DATA ON LIGNIN DERIVATIVES

Derivative	Douglas Fir		Spruce
	Sapwood	Heartwood	
Sulphuric Acid Lignin			
Yield, %	29.4	28.6	27.6
Methoxyl, %	14.0	14.4	14.8
Ash, %	0.52	0.48	0.55
Willstätter Lignin			
Yield, %	31	30	25-30
Methoxyl, %	14.4	14.2	14.5-15.5
Ash, %	0.21	0.22	----
Thioglycolic Acid Lignin			
Yield, crude, %	27.5	27.0	30
Yield, purified, %	24	24	25.5
Methoxyl, %	11.5	11.8	11.9
Ash, %	0.00	0.00	----
Phenol Lignin			
Yield, crude, %	56	57	67
Methoxyl, crude, %	7.2	7.1	7.0
Ash, crude, %	0.23	0.25	----
Ether Insol. A, %	65	65	75
Ether Sol. B, %	35	35	25
Methoxyl, A, %	10.7	10.5	10.4
Methoxyl, B, %	4.4	4.4	5.3
Methoxyl, A, acetylated, %	8.6	8.4	7.9
Acetyl, A, acetylated, %	24.3	24.6	23.0
Methoxyl, A, methylated, %	21.8	21.7	21.6

It may be seen from Table XIII that no differences exist between the lignin of Douglas fir heartwood and that of the sapwood. The data prove conclusively that the two lignins are at least isomers or polymers, if not identical. Elemental analyses for carbon and hydrogen were not made, but it would be a most unusualy coincidence if these showed any variation between the two types of wood, as six independent derivatives had identical yields and methoxyl contents, within the accuracy of the analytical methods used.

Similarly, while the agreement between the values for the Douglas fir lignins and those for the same compounds prepared from spruce is not as close as for the two Douglas fir types, the differences observed are not sufficient to indicate that native Douglas fir lignin differs from native spruce lignin. The one point of greatest difference observed, the methoxyl contents of the ether-soluble fractions of the phenol lignins, is of no significance, because two different methods were employed in purifying these compounds. Here, again, while no elemental analyses were made, the finding of significant differences in the carbon and hydrogen contents of compounds from the two species would be in the nature of an unusual and improbable coincidence. The same statement made with respect to the lignin of Douglas fir heartwood and sapwood may also be made in connection with the lignin of spruce and Douglas fir. The data obtained on six independent preparations show no difference between the two lignins. Therefore, the lignin of Douglas fir is either an isomer, polymer, or identical with the lignin from spruce wood.

INVESTIGATION OF THE EXTRACTIVES OF DOUGLAS FIR WOOD

Object:

In an effort to find some significant difference in the amount of tannins, pholobaphenes, or ether-soluble resins which might provide an explanation for the differences observed in the behavior of Douglas fir heartwood and sapwood when pulped by the sulphite process, the extractives of these two types of wood were separated into resins and fatty acids and waxes, tannins, and phlobaphenes. As was mentioned in the section on pulping data, it is possible that the presence of pinene and its homologues may catalyze the auto-oxidation and reduction of the cooking acid, thus promoting precipitation. Since heartwood gave more trouble in this respect, the presence of a greater amount of ether-soluble matter in the heartwood would give support to this possibility. The adverse effect of tannins and phobaphenes on the delignification of wood by the sulphite process has been described and, hence, any great differences in the amounts of these compounds in the two types of wood would provide additional data for explaining their behavior when cooked.

The distribution of the extractives across the face of the log was also investigated. This was done to demonstrate the uniformity of the extractive content throughout the log.

Procedure:

The extractives for use in this work were obtained from the extraction of the wood meal used in the preparation of the lignin derivatives.

This material was obtained, therefore, as a solution of the various extractive compounds in alcohol and benzene (1:2). The alcohol and benzene were removed as far as possible, by distillation under reduced pressure, and the gummy mass left in the distilling flask was taken up with water. The tannins went into solution, while the phlobaphenes and ether-soluble components of the mixture remained insoluble. This heterogenous mixture was then extracted with petroleum ether in order to remove the fats, waxes, and resin acids without dissolving any of the tannins or phlobaphenes. The petroleum ether extract was concentrated in a tared Pyrex dish, dried to constant weight, and weighed.

The water solution of tannins, which also contained carbohydrates and salts in solution, was separated from the phlobaphene material by filtration. The crude phlobaphenes were then further extracted with water, and this water extract added to the filtrate. The phlobaphenes were then dried to constant weight in a vacuum oven over concentrated sulphuric acid at 60°C.

The aqueous solution after removal of the phlobaphenes was extracted with ethyl acetate. This procedure removes the tannins, leaving the carbohydrates, salts and any other water and alcohol-soluble compounds present in the wood in solution. The ethyl acetate solution of the tannins was evaporated and dried to constant weight in a vacuum oven at 60°C. over concentrated sulphuric acid.

The weight of water-soluble carbohydrates and salts was determined by difference from the total extract and the weights of the other three fractions (see Figure 22).

Wood samples for the determination of the distribution of extractives across the face of the log were cut from the wood density disc (see page 10). The samples were ground in a disc mill and screened, the fraction passing 40-mesh but retained on 60-mesh being used. The samples were oven-dried to constant weight at 105°C. and then extracted with alcohol and benzene for a period of 12 hours, using Soxhlet extractors. The amount of soluble material was determined by evaporating the solvent, drying, and weighing. Samples were taken at the center of the log, 1/4, 1/2, and 3/4 of the distance to the sapwood boundary.

Data:

The data obtained are presented in Tables XIV and XV.

TABLE XIV

ANALYSIS OF DOUGLAS FIR EXTRACTIVES

	Sapwood		Heartwood	
	% of extractives	% of wood	% of extractives	% of O.D. wood
Resins, Fatty Acids, and Waxes	16	0.35	23	0.73
Phlobaphenes	25	0.54	22	0.69
Tannins	11	0.24	26	0.81
Carbohydrates, Salts, etc.	48	1.05	29	0.90
Total	100	2.18	100	3.13

FIGURE 22

SCHEME FOR SEPARATION OF EXTRACTIVES

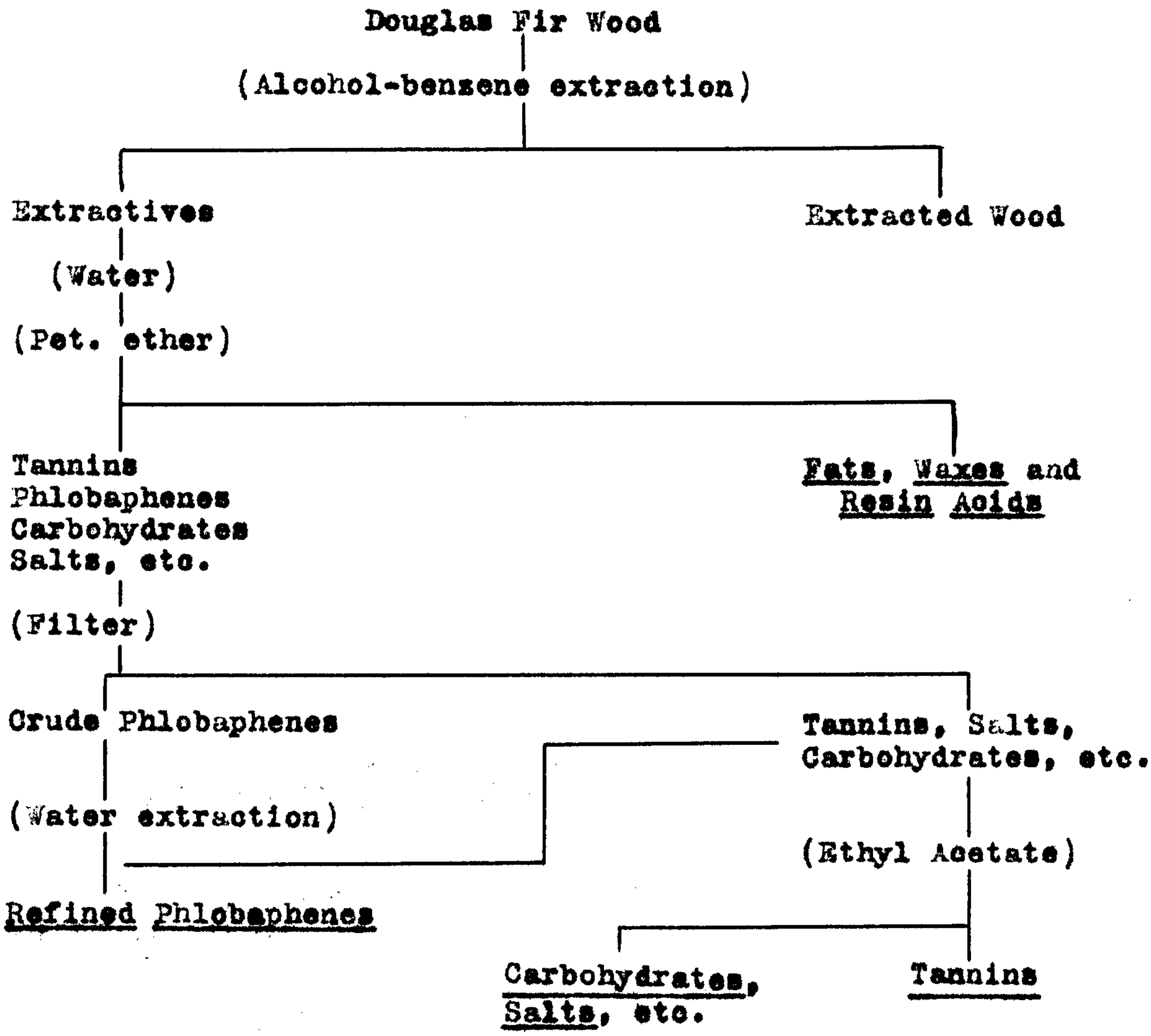


TABLE XV

DISTRIBUTION OF EXTRACTIVES IN DOUGLAS FIR

Location	Sapwood	3/4	1/2	1/4	Center
Extractives, %	2.18	3.25	2.90	3.10	3.13

Discussion of Data:

Referring to Table XIII, it is seen that the heartwood contains a larger quantity of both resins and tannins, while the phlobaphene content of the two types of wood does not vary greatly. While no effort was made to identify the members of the pinene series in this material, it is logical to assume their presence in view of published data.

In general, the amount of extractive matter found in this wood (both heartwood and sapwood) was very low. It is doubtful if the small amounts of tannins and phlobaphenes found would have any material effect on the rate of pulping of the wood. The differences observed in the resin contents of sapwood and heartwood do, however, lend support to the theory that members of the pinene series are responsible, at least in part, for the precipitation of the cooking liquor when heartwood is pulped.

The data on the distribution of the extractives (Table XIV), are not very helpful. About the only thing which may be said is that there is no great variation in the amount of extractive material across the heartwood section of the log. There is a noticeably lower amount in the sapwood, however.

GENERAL SUMMARY

Douglas fir cellulose, as isolated by chlorination, has been found to be satisfactory as a paper-making pulp when compared to spruce cellulose obtained in the same manner. Pulps satisfactory with respect to yield and quality were obtained from Douglas fir heartwood, sapwood, slab wood, and the entire tree by the usual sulphite process. It was not found necessary to use any chemical pretreatment or special base in the cooking liquor to accomplish this, contrary to the findings of other investigators as reported in the literature (4, 9, 11).

The principal points of difference between the cooking conditions used by these workers and those used in this investigation are: first, a higher concentration of free sulphur dioxide was used in these cooks, and second, all experimental pulping in this work was carried out at temperatures ranging from 134 to 140°C., values considerably lower than those reported in the literature. As some difficulty was encountered in pulping the heartwood of this species at 140°C. (because of precipitation of the cooking liquor and the resultant burning of the pulp), these variations in cooking conditions may well account for the success of this work and the poor results obtained by others.

The cooking conditions used in pulping Douglas fir were such as to give a high concentration of sulphur dioxide in the liquor until well into the cook. This was accomplished by using a very slow temperature rise from 110°C. up to the maximum temperature (134°C.) and a high initial pressure (35 lb. per sq. in.), thus favoring complete and rapid penetration of the chips and allowing a fairly long period at comparatively low temperatures for sulphonation of the lignin. The success of these measures may be judged by the very low screenings obtained. While cooking under these conditions requires a longer total cooking time, the higher wood density of Douglas fir and the somewhat higher pulp yields more than compensate for this extra cooking period, thus making it possible to obtain a slightly increased yield of pulp per day when using this species.

Theoretical investigations of the lignin and extractives of Douglas fir heartwood and sapwood as compared to spruce gave little help in providing an explanation of the differences observed in the behavior of these two types of wood when cooked. The lignin investigation showed that spruce lignin and the lignin of Douglas fir heartwood and sapwood are either identical, or are isomers, or polymers. The larger quantity of ether-soluble resin in Doublas fir heartwood does provide evidence that precipitation of the cooking liquor encountered when pulping this wood may be caused by the action of compounds of the pinene series in promoting auto-oxidation and reduction of the liquor to calcium sulphate and free sulphur.

As has been discussed in some detail previously, it is entirely possible that Douglas fir heartwood does not elevate the precipitation temperature of sulphite liquor to the same extent as does spruce, thus providing another explanation for this phenomenon. However, using the proper cooking conditions, precipitation in the liquor may be avoided.

The bleached and unbleached Douglas fir pulps upon beating gave development curves for physical properties which were similar to normal bleached sulphite pulps. While these development curves might be considered in the normal range of sulphite pulps, they did show a distinctly slower rate of development than the spruce and western hemlock pulps prepared in this study. In comparison with the spruce and western hemlock pulps the Douglas fir pulps, in general, were low in bursting and folding strengths and very much higher in tearing strength. Another outstanding difference is noted in the low apparent density; i.e., high bulk of the Douglas fir pulp.

Because of the much longer fiber length of Douglas fir pulps, it may be that difficulties in obtaining uniform formation would be encountered when these pulps were used as the complete fiber furnish for grades requiring good formation. However, it seems probable that Douglas fir pulp would impart valuable characteristics to sheets that are normally made of spruce or hemlock pulp if it were blended with the spruce or hemlock in the correct proportions. Higher bulk and tear than can be made from normal sulphite are often desired in many papers. Douglas fir pulp should improve these properties even when added in moderate amounts.

Unbleached pulps from Douglas fir were found to have considerably higher alpha-cellulose and lower pentosan contents than pulps from spruce or western hemlock. The low ether solubilities of the Douglas fir pulps indicates that very little or no pitch trouble would be occasioned by their use on the paper machine.

It was found that Douglas fir pulps from heartwood and sapwood, as well as those prepared from mixtures of the two, could be bleached to a relatively high brightness by a two-stage bleaching operation. While the method of bleaching employed caused some degradation of the pulp, the results are truly comparative, for as both spruce and western hemlock pulps were also bleached by this procedure.

The bleached pulps had physical properties similar in all respects to those of the unbleached pulps. Their application to papermaking has already been discussed.

The chemical analyses of the bleached Douglas fir pulps show decidedly higher alpha-cellulose and lower pentosan contents than were observed for spruce and western hemlock. This suggests their application in the manufacture of cellulose derivatives, and, while no data were taken in this respect, an investigation in this direction should prove worth while.

LITERATURE CITED

(1) Greeley, W. B., Pac. Pulp Paper Ind. 2, no. 12: 28-9 (November, 1928).

(2) Anon., Weights of various woods grown in the United States. Tech. Note No. 218, Forest Products Laboratory, Madison, Wis. Revised July, 1931.

(3) Hayward, P. A., "Wood, lumber and timbers." New York, Chandler Cyclopedia, 1930. Pages 360-8.

(4) Wells, S. D., and Rue, J. D., "The suitability of American woods for paper pulp." U. S. Dept. Agriculture Bull. No. 1485. Washington, U. S. Govt. Print. Off., 1927.

(5) English, H., Green, H., Mitchell, C. R., and Yorston, F. H., Forest Products Labs. Canada, Pulp Paper Lab., Quarterly Rev. no. 20: 15-21 (Oct.-Dec., 1934).

(6) Beuschlein, W. L., Paper Trade J. 88, no. 19: 66-7 (May 9, 1929).

(7) Bailey, A. J., Paper Ind. 16: 480-3 (1934).

(8) Klason, P., Ber. 64: 2733-9 (1931).

(9) Benson, H. K., Erwin, R. P., Hendrickson, J. R., and Tershin, J. A., Paper Trade J. 99, no. 12: 87-9 (Sept. 20, 1934).

(10) Dorée, C., and Barton-Wright, E. C., J. Soc. Chem. Ind. 48: 9-11T (1929).

(11) Benson, H. K., U. S. Patent 1,805,799 (May 19, 1931).

(12) L., Wochbl. Papierfabr. 62: 403 (1931).

(13) Rosen, G., U. S. Patent 1,857,695 (May 10, 1932); French Patent 715,353 (Aug. 7, 1930).

(14) McKee, R. H., and Cable, D. E., Paper Trade J. 80, no. 17: 41-6 (April 23, 1925).

(15) Bergström, C., Papier-Fabr. 30: 241-6, 255-7 (1932).

(16) Richardson, R. W., and Sherman, C. S., U. S. Patent 1,851,522 (March 29, 1932).

(17) Herty, C. H., Paper Trade J. 96, no. 13: 23-7 (March 30, 1933).

(18) Cohen, W. E., J. Council Sci. Ind. Research 8: 27-36 (1935): C. A. 29: 3829 (1935).

(19) Pieńkowski, S., and Jurkiewicz, L., Acta Phys. Polonica 3: 435-46 (1934); C. A. 30: 7364 (1936).

(20) Schwalbe, C. G., and af Ekenstam, A., Cellulosechem. 10: 1-11 (1929).

(21) Private Correspondence, J. H. Souliere of the Anacortes Lumber and Box Co.

(22) Kang, M. H., and Libby, C. E., Cellulose 1: 25-9, 50-1, 54-8 (1930).

(23) Palmrose, G. V., Paper Trade J. 100, no. 3: 38-9 (Jan. 17, 1935).

(24) Private Correspondence, Tabulation of data sent to H. F. Lewis by C. C. Curran.

(25) Gishler, P. E., and Maass, O., Can. J. Research B13: 370-9 (1935).

(26) Clark, E. P., J. Assoc. Agr. Chem. 15: 136-40 (1932).

(27) Kalb, L., Lieser, T., Hahn, R., Nevely, F., and Koch, H., Ber. 61: 1007-22 (1928).

(28) Holmberg, B., Svensk Pappers-Tidn. 33: 679-80, 685-6 (1930).

(29) Buckland, I. K., Brauns, F., and Hibbert, H., Can. J. Research B13: 61-77 (1935).

(30) Benson, H. K., and McCarthy, D. F., Ind. Eng. Chem. 17: 193-4 (1925).

APPENDIX A

CHLORINE PULPS

Data Sheets and Detailed Physical Data

DETAILED PHYSICAL DATA--CHLORINE PULPS

Cook No.	Basis Weight 24x36-500				Burst Ratio %				Tear Factor %			
	Milling Time--Min.											
	0	15	30	60	0	15	30	60	0	15	30	60
D-1	42.8	39.9	39.7	39.8	103	99	100	112	172	140	153	114
D-2	44.9	44.2	43.3	42.8	102	106	108	114	211	206	163	157
S-1	43.7	41.5	40.6	39.7	190	170	179	183	122	128	107	114

Cook No.	M. I. T. Fold No.				Apparent Density				Freeness ml.			
	Milling Time--Min.											
	0	15	30	60	0	15	30	60	0	15	30	60
D-1	329	292	346	1383	11.9	13.0	13.2	14.2	760	680	600	395
D-2	471	454	461	893	11.2	11.7	12.8	13.8	745	690	640	430
S-1	2290	1651	3198	5022	14.6	14.8	17.2	17.3	725	715	660	540

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

EXPERIMENT No. D-1 TYPE OF COOK Alkali DATE 8/9/'37

CHEMIST Brookbank DATA Brookbank BLEACH TESTER

COOK Brookbank SCREENMAN STRENGTH TESTER

REACTION VARIABLES				LIQUOR ANALYSIS	
ACTIVE CHEMICAL RATIO	0.08	AS	NaOH	SPECIFIC GRAVITY	
WATER RATIO	5 to 1			CAUSTICITY	98.8%
WGT. ACTIVE CHEMICAL	316 g	WGT. WATER	19750g	ACTIVE ALKALI AS NA OH	15.3 gpl
WGT. RAW MATERIAL O.D.	3950 g			Note: Initial chem. ratio 0.04	
LENGTH OF COOK	2 hr. 50 min.	SCHEDULE NO.		4% chem. injected after 1 hr.	
DIGESTER NO.	Rotary No. 1	WOOD	D. Fir Heartwood		

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
8:10	32								
20	50								
30	81								
40	83								
50	81								
9:00	81								
10	83	Inject 4% NaOH							
20	100								
30	105								
40	110	Alkali							
50	111	Cons.							
10:00	110	76%							
10	107								
20	106								
30	105	78							
40	105								
50	103								
11:00	102	80							
Burners off; cook washed									

YIELD CALCULATIONS

WGT. OF RAW MATERIAL A.D.	O.D.	
WGT. OF MOISTURE SAMPLES A.	B.	
WGT. OF SAMPLES O.D.		
PERCENT O.D.		AV.
WGT. OF PULP WET	O.D.	
WGT. OF MOISTURE SAMPLES A.	B.	
WGT. OF SAMPLES O.D.		
PERCENT O.D.		AV.
WGT. OF PULP SCREENED WET	O.D.	
WGT. OF SCREENINGS O.D.		
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES	A.		B.
WGT. SAMPLES O.D.			
PERCENT O.D.			AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

EXPERIMENT No. D-2 TYPE OF COOK Alkaline DATE 8/9/'37

CHEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank

COOK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES				LIQUOR ANALYSIS	
ACTIVE CHEMICAL RATIO	0.08 to 1	AS	NaOH	SPECIFIC GRAVITY	
WATER RATIO	5 to 1			CAUSTICITY	98.8%
WGT. ACTIVE CHEMICAL	320 g	WGT. WATER	20000	ACTIVE ALKALI AS NA OH	15.3 gpl
WGT. RAW MATERIAL O.D.	4000 g			Note: Active chem. added in two	
LENGTH OF COOK	3.5 hrs.	SCHEDULE NO.		portions. 4% at start and	
DIGESTER NO.	2 (Rotary)	WOOD	D. Fir Sapwood	4% at the end of 1 hr.	

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
8:15	34								
25	61								
35	83								
45	81.5								
55	83								
9:05	84								
15	83	4% chemical added							
20	100								
30	105								
40	106								
50	104	Alkali							
10:00	103.5	Cons.							
10	103	67%							
20	103								
30	103								
40	102	71							
50	102								
11:00	103								
10	103	76							
20	104								
30	103								
40	103	80							
Burners off,		cook washed.							

YIELD CALCULATIONS	BLEACH CONSUMPTION AND BLEACHING
WGT. OF RAW MATERIAL A.D. O.D.	STANDARD BL. CONS.
WGT. OF MOISTURE SAMPLES A. B.	COLOR
WGT. OF SAMPLES O.D.	PERMANGANATE NO. EST. BL. CONS.
PERCENT O.D. AV.	CHLORINE NO. EST. BL. CONS.
WGT. OF PULP WET O.D.	
WGT. OF MOISTURE SAMPLES A. B.	WGT. PULP BLEACHED WET O.D.
WGT. OF SAMPLES O.D.	WGT. BLEACHED PULP WET O.D.
PERCENT O.D. AV.	WGT. MOISTURE SAMPLES A. B.
WGT. OF PULP SCREENED WET O.D.	WGT. SAMPLES O.D.
WGT. OF SCREENINGS O.D.	PERCENT O.D. AV.
PERCENT SCREENINGS ON UNSCREENED PULP	YIELD BL. PULP ON UNBLEACHED ON RAW MAT.
PERCENT UNSCREENED PULP ON RAW MATERIAL	BLEACH LIQUOR ANALYSIS VOLUME
PERCENT SCREENED PULP ON RAW MATERIAL	BLEACHING TIME TEMP. CONS. VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

(E)PERIMENT No. S-1 TYPE OF COOK Alkali DATE 7/28/'37

(C)HEMIST Brookbank DATA Brookbank BLEACH TESTER

(C)OOK Brookbank SCREENMAN STRENGTH TESTER

REACTION VARIABLES				LIQUOR ANALYSIS	
(A)CTIVE CHEMICAL RATIO	0.08 to 1	AS	NaOH	SPECIFIC GRAVITY	
(W)ATER RATIO	5 to 1			CAUSTICITY	98.8%
(W)GT. ACTIVE CHEMICAL	350 g	WGT. WATER	21.9kg.	ACTIVE ALKALI AS NA OH	7.7 gpl
(W)GT. RAW MATERIAL O.D.	4380			Note: Initial chem. ratio 0.04	
(L)ENGTH OF COOK	3 hrs. 40 min	SCHEDULE NO.		4% chem. injected after	
(D)IGESTER NO.	Rotary No. 2	WOOD	Spruce	1 hr.	

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
9:20	25								
25	69								
30	80								
40	78								
50	77								
10:00	83								
10	83								
20	82	Inject	4% NaOH						
30	80								
35	100	Alkali							
40	101	Cons.							
50	103	51%							
11:00	104								
10	103	58							
20	103								
30	102	63.6							
40	103								
50	103								
12:00	102	71.1							
10	101								
20	100+								
30	100	77.3							
40	99								
50	98.5								
1:00	98	80							
Cook washed.									

YIELD CALCULATIONS		
WGT. OF RAW MATERIAL	A.D.	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP	WET	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP SCREENED	WET	O.D.
WGT. OF SCREENINGS	O.D.	
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING			
STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES	A.		B.
WGT. SAMPLES	O.D.		
PERCENT	O.D.		AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

APPENDIX B

Data Sheets

Experimental Sulphite Cooks

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

(EX)PERIMENT No.	D-3	TYPE OF COOK	Sulphite	DATE	8/23/'37
(C)HEMIST	Brookbank	DATA	Brookbank	BLEACH TESTER	Brookbank
(C)OOK	"	SCREENMAN	"	STRENGTH TESTER	"

REACTION VARIABLES				LIQUOR ANALYSIS	
(A)CTIVE CHEMICAL RATIO		AS		SPECIFIC GRAVITY	
(W)ATER RATIO	7.33			CAUSTICITY	
(W)GT. ACTIVE CHEMICAL		WGT. WATER	31.31	ACTIVE ALKALI AS NA OH	
(W)GT. RAW MATERIAL O.D.	4272 G.			Total SO_2	7.22
(L)ENGTH OF COOK	12.25 hrs.	SCHEDULE NO.	1	Free	5.96
(D)IGESTER NO.	2	WOOD	D. Fir Sapwood	Combined	1.26

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
9:55	23	85	0	85	4:10	117.5	85	12.0	73.0
10:10	37	85	0	85	25	117.5	85	12.0	73.0
25	58	85	0	85	40	118.5	85	12.9	72.1
40	77	85	0	85	55	120	85	14.1	70.9
55	87	85	0	85	5:10	121	85	15.0	70.0
11:10	93.5	85	0	85	25	122	85	16.0	69.0
25	100	85	0	85	40	123	85	16.9	68.1
40	102.5	85	1.4	83.6	55	124	85	17.9	67.1
55	105	85	2.8	82.2	6:10	125	85	19.0	66.0
12:10	107.5	84	4.4	79.6	25	126	85	20.0	65.0
25	110	85	6.1	78.9	40	127	84.5	21.0	63.5
40	110+	85	6.4	78.6	55	128	85	22.0	63.0
55	111	85	6.8	78.2	7:10	129	85	23.1	61.9
1:10	111	84	6.8	77.2	25	129.5	84	23.7	60.3
25	111.5	85	7.2	77.8	40	131	85	25.7	59.3
40	112.5	85	7.9	77.1	55	132	85	26.9	58.1
55	113	85	8.3	76.7	8:10	133	85	28.1	56.9
2:10	113.5	85	8.7	76.3	25	134	85	29.4	55.6
25	114	85	9.0	76.0	40	134	85	29.4	55.6
40	114.5	85	9.4	75.6	55	133.5	84	28.8	55.2
55	115	85	9.8	75.2	9:10	134	85	29.4	55.6
3:10	115.5	85	10.2	74.8	25	134	85	29.4	55.6
25	116	85	10.6	74.4	40	134	85	29.4	55.6
40	116	85	10.6	74.4	55	134	85	29.4	55.6
55	117	85	11.6	73.5	10:10	134	50	29.4	20.6
					Blown at 10:10 P. M.				

YIELD CALCULATIONS		
WGT. OF RAW MATERIAL A.D.	O.D.	
WGT. OF MOISTURE SAMPLES A.	B.	
WGT. OF SAMPLES O.D.		
PERCENT O.D.	AV.	
WGT. OF PULP WET	O.D.	
WGT. OF MOISTURE SAMPLES A.	B.	
WGT. OF SAMPLES O.D.		
PERCENT O.D.	AV.	
WGT. OF PULP SCREENED WET	O.D.	
WGT. OF SCREENINGS O.D.		
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING			
STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES A.			B.
WGT. SAMPLES O.D.			
PERCENT O.D.			AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

EXPERIMENT No. D-4 TYPE OF COOK Sulphite DATE 8/26/'37

CHEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank

COOK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES		LIQUOR ANALYSIS	
ACTIVE CHEMICAL RATIO	AS	SPECIFIC GRAVITY	
WATER RATIO 7.33		CAUSTICITY	
WGT. ACTIVE CHEMICAL	WGT. WATER 31.3kg	ACTIVE ALKALI AS NA OH	
WGT. RAW MATERIAL O.D. 4272 g.		Total SO_2	7.29
LENGTH OF COOK 13.75 hrs.	SCHEDULE NO. 1	Free	6.04
DIGESTER NO. 2	WOOD D. Fir Sapwood	Combined	1.25

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
7:55	25	85	0	85	55	119.5	85	13.7	71.3
8:10	45	85	0	85	3:10	121	85	15.0	70.0
25	62.5	85	0	85	25	122	85	16.0	69.0
40	71	85	0	85	40	123	85	16.9	68.1
55	90	85	0	85	55	124	85	17.9	67.1
9:10	95	85	0	85	4:10	124.5	85	18.5	66.5
25	100	85	0	85	25	126.5	85	20.6	64.4
40	102+	85	1.2	83.8	40	127	85	21.0	64.0
55	105	85	2.8	82.2	55	128	85	22.0	63.0
10:10	107.5	85	4.4	79.6	5:10	129	85	23.1	61.9
25	110	85	6.1	78.9	25	130	85	24.3	60.7
40	110.5	85	6.5	78.5	40	131	85	25.7	59.3
55	111	85	6.8	78.2	55	132	85	26.9	58.1
11:10	111	85	6.8	78.2	6:10	133	85	28.1	56.9
25	112	85	7.5	77.5	25	134	85	29.4	55.6
40	112.5	85	7.9	77.1	40	134	85	29.4	55.6
55	113	85	8.3	76.7	55	134	85	29.4	55.6
12:10	113.5	85	8.7	76.3	7:10	134	85	29.4	55.6
25	114	85	9.0	76.0	25	134	85	29.4	55.6
40	114.5	85	9.4	75.6	40	134	85	29.4	55.6
55	114.5	85	9.4	75.6	55	134	85	29.4	55.6
1:10	115.5	85	10.2	74.8	8:10	134	85	29.4	55.6
25	116	85	10.6	74.4	25	134	85	29.4	55.6
40	116.5	85	11.1	73.9	40	134.5	85	30.0	55.0
55	117	85	11.5	73.5	55	133.5	85	28.9	56.1
2:10	117.5	85	12.0	73.0	9:10	134	85	29.4	55.6
25	118	85	12.4	72.6	25	134	85	29.4	55.6
40	118.5	85	12.9	72.1	40	134	50	29.4	20.6

YIELD CALCULATIONS

WGT. OF RAW MATERIAL A.D.	O.D.	
WGT. OF MOISTURE SAMPLES A.	B.	
WGT. OF SAMPLES O.D.		
PERCENT O.D.		AV.
WGT. OF PULP WET	O.D.	
WGT. OF MOISTURE SAMPLES A.	B.	
WGT. OF SAMPLES O.D.		
PERCENT O.D.		AV.
WGT. OF PULP SCREENED WET	O.D.	
WGT. OF SCREENINGS O.D.		
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES A.			B.
WGT. SAMPLES O.D.			
PERCENT O.D.			AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

PERIMENT No. D-5 TYPE OF COOK Sulphite DATE 9/3/'37

IEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank

OK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES		LIQUOR ANALYSIS	
CTIVE CHEMICAL RATIO	AS	SPECIFIC GRAVITY	
~~XXXXXXX~~ Liquor Ratio 6.95		CAUSTICITY	
GT. ACTIVE CHEMICAL	WGT. WATER 29.7kg	ACTIVE ALKALI AS NA OH	
GT. RAW MATERIAL O.D. 4272		Total SO_2	7.23
ENGTH OF COOK 13.75 hrs.	SCHEDULE NO. 1	Free	5.97
IGESTER NO. 2	WOOD D. Fir Heartwood	Combined	1.26

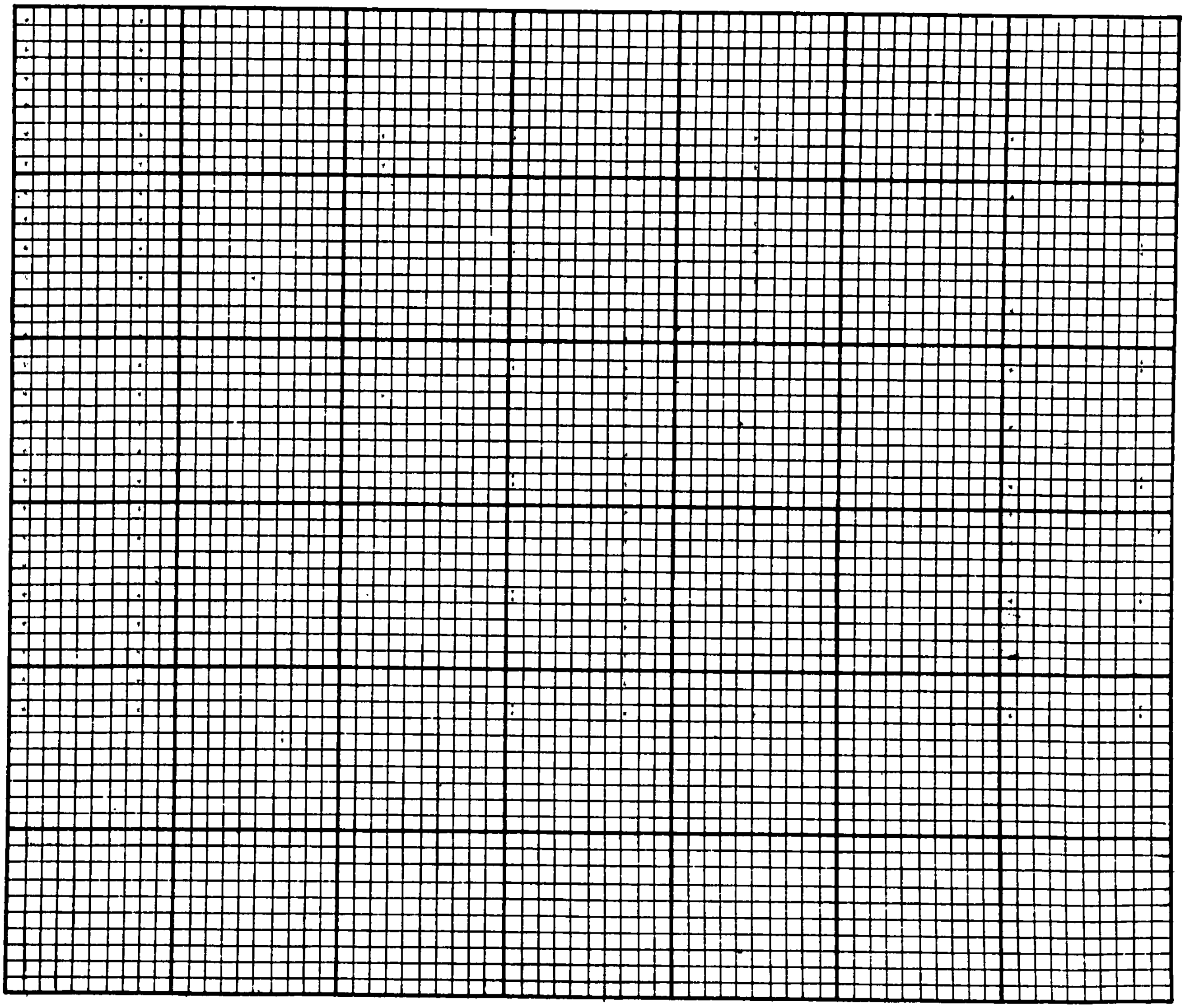
TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
9:25	29	85	0	85	4:25	120	85	14.1	70.9
40	48	85	0	85	40	120	85	14.1	70.9
55	64	85	0	85	55	122	85	16.0	69.0
10:10	75	85	0	85	5:10	123	85	16.9	68.1
25	88	85	0	85	25	124	85	17.9	67.1
40	95	85	0	85	40	125	85	19.0	66.0
55	100	85	0	85	55	126	85	20.0	65.0
11:10	102.5	85	1.4	83.6	6:10	126.5	85	20.6	64.4
25	105	85	2.8	82.2	25	127.5	85	21.6	63.4
40	107.5	85	4.4	79.6	40	129	85	23.1	61.9
55	110	85	6.1	78.9	55	130	85	24.3	60.7
12:10	110+	85	6.4	78.6	7:10	131	85	25.7	59.3
25	111	85	6.8	78.2	25	132	84.5	26.9	57.6
40	111.5	85	7.2	77.8	40	133	85	28.1	56.9
55	112	85	7.5	77.5	55	133	85	28.1	56.9
1:10	112.5	85	7.9	77.1	8:10	134	85	29.4	55.6
25	113	85	8.3	76.7	25	133.5	85	28.7	56.3
40	113.5	85	8.7	76.3	40	134	85	29.4	55.6
55	114	85	9.0	76.0	55	134	85	29.4	55.6
2:10	114.5	85	9.4	75.6	9:10	134	85	29.4	55.6
25	115.5	85	10.2	74.8	25	134	85	29.4	55.6
40	115.5	85	10.2	74.8	40	134	85	29.4	55.6
55	116	85	10.6	74.4	55	134	85	29.4	55.6
3:10	116.5	85	11.1	73.9	10:10	134	85	29.4	55.6
25	117	85	11.5	73.5	25	134	85	29.4	55.6
40	117.5	85	12.0	73.0	40	134	85	29.4	55.6
55	118	85	12.4	72.6	55	134	85	29.4	55.6
4:10	118.5	85	12.9	72.1	11:10	134	50	29.4	20.6
					Cook blown at 11:10				

YIELD CALCULATIONS		
WGT. OF RAW MATERIAL	A.D.	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP	WET	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP SCREENED	WET	O.D.
WGT. OF SCREENINGS	O.D.	
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING			
STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES	A.		B.
WGT. SAMPLES	O.D.		
PERCENT	O.D.		AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES



THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

EXPERIMENT No. D-6 TYPE OF COOK Sulphite DATE 9/9/'37
CHEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank
COOK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES		LIQUOR ANALYSIS	
ACTIVE CHEMICAL RATIO	AS	SPECIFIC GRAVITY	
Liquor Ratio 6.95		CAUSTICITY	
WGT. ACTIVE CHEMICAL	WGT. WATER 29.71	ACTIVE ALKALI AS NA OH	
WGT. RAW MATERIAL O.D. 4272 g.		Total SO_2	7.24%
LENGTH OF COOK 16 hrs.	SCHEDULE NO. 1	Free	5.97
DIGESTER NO. 2	WOOD D. Fir Heartwood	Combined	1.27

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
7:05	25	85	0	85	3:20	124.5	85	18.5	66.5
20	45	85	0	85	35	126	85	20.0	65.0
35	59.5	85	0	85	50	126.5	85	20.6	64.4
50	75	85	0	85	4:05	128	85	22.2	62.8
8:05	87	85	0	85	20	129	85	23.1	61.9
20	94	85	0	85	35	130	85	24.3	60.7
35	99.5	85	0	85	50	131.5	85	26.3	58.7
50	103	85	1.4	83.6	5:05	132	85	26.9	57.6
9:05	105	85	2.8	82.2	20	133	85	28.1	56.9
20	107.5	85	4.4	80.6	35	133	85	28.1	56.9
35	110	85	6.1	78.9	50	134	85	29.4	55.6
50	110	85	6.1	78.9	6:05	134	85	29.4	55.6
10:05	110.5	85	6.5	78.5	20	134	85	29.4	55.6
20	111.5	85	7.2	77.8	35	134.5	85.5	29.9	55.6
35	112	85	7.5	77.5	50	134	85	29.4	55.6
50	112.5	85	7.9	77.1	7:05	134	85	29.4	55.6
11:05	113	85	8.3	76.7	20	134	85	29.4	55.6
20	113.5	85	8.7	76.3	35	134	85	29.4	55.6
35	114	85	9.0	76.0	50	134	85	29.4	55.6
50	114.5	85	9.4	75.6	8:05	134	85	29.4	55.6
12:05	115	85	9.8	75.2	20	134	85	29.4	55.6
20	115	85	9.8	75.2	35	134	85	29.4	55.6
35	116.5	85	11.1	73.9	50	134	85	29.4	55.6
50	116.5	85	11.1	73.9	9:05	134	85	29.4	55.6
1:05	117	85	11.5	73.5	20	134	84	29.4	54.6
20	117.5	85	12.0	73.0	35	134	81.5	29.4	52.1
35	118	85	12.4	72.6	50	134	80	29.4	50.6
50	119	85	13.2	71.8	10:05	134	79	29.4	49.6
2:05	120	84	14.1	70.9	20	134	78.5	29.4	49.1
20	121.5	84	15.5	69.5	35	133	77.5	28.1	49.4
35	122	84	16.0	69.0	50	134	78.5	29.4	49.1
50	123	85	16.9	68.1	11:05	134	50	29.4	20.6
					Cook blown at 11:05				

YIELD CALCULATIONS
WGT. OF RAW MATERIAL A.D. O.D.
WGT. OF MOISTURE SAMPLES A. B.
WGT. OF SAMPLES O.D.
PERCENT O.D. AV.
WGT. OF PULP WET O.D.
WGT. OF MOISTURE SAMPLES A. B.
WGT. OF SAMPLES O.D.
PERCENT O.D. AV.
WGT. OF PULP SCREENED WET O.D.
WGT. OF SCREENINGS O.D.
PERCENT SCREENINGS ON UNSCREENED PULP
PERCENT UNSCREENED PULP ON RAW MATERIAL
PERCENT SCREENED PULP ON RAW MATERIAL

BLEACH CONSUMPTION AND BLEACHING
STANDARD BL. CONS.
COLOR
PERMANGANATE NO. EST. BL. CONS.
CHLORINE NO. EST. BL. CONS.
WGT. PULP BLEACHED WET O.D.
WGT. BLEACHED PULP WET O.D.
WGT. MOISTURE SAMPLES A. B.
WGT. SAMPLES O.D.
PERCENT O.D. AV.
YIELD BL. PULP ON UNBLEACHED ON RAW MAT.
BLEACH LIQUOR ANALYSIS VOLUME
BLEACHING TIME TEMP. CONS. VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

PERIMENT No. D-7 TYPE OF COOK Sulphite DATE 9/20/'37

EMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank

OK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES		LIQUOR ANALYSIS	
CTIVE CHEMICAL RATIO	AS	SPECIFIC GRAVITY	
Liquor Ratio 6.92		CAUSTICITY	
GT. ACTIVE CHEMICAL	WGT. WATER 29.71	ACTIVE ALKALI AS NA OH	
GT. RAW MATERIAL O.D. 4292		Total SO_2	7.24%
ENGTH OF COOK 14.75 hrs.	SCHEDULE NO. 1	Free	5.98
GESTER NO. 2	WOOD D. Fir Heartwood	Combined	1.26

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
9:55	24	85	0	85	5:25	121.5	85	15.5	69.5
10:10	50	85	0	85	40	123	85	16.9	68.1
25	64	85	0	85	55	124	85	17.9	67.1
40	75	85	0	85	6:10	124.5	85	18.5	66.5
55	88	85	0	85	25	125	85	19.0	66.0
11:10	95	85	0	85	40	126.5	85	20.6	64.4
25	100	85	0	85	55	126.5	85	20.6	64.4
40	102.5	85	1.4	83.6	7:10	128.5	85	22.8	62.2
55	105.5	85	3.1	81.9	25	130	85	24.3	60.7
12:10	107.5	85	4.4	80.6	40	131	85	25.7	59.3
25	109	85	5.4	79.6	55	132	85	26.9	57.6
40	110.5	85	6.5	78.5	8:10	133	85	28.1	56.9
55	111	85	6.8	78.2	25	134	85	29.4	55.6
1:10	111.5	85	7.2	77.8	40	134.5	86	30.1	55.9
25	112	85	7.5	77.5	55	135	86	30.7	55.3
40	112.5	85	7.9	77.1	9:10	133.5	84.5	28.7	55.8
55	113	85	8.3	76.7	25	134	85	29.4	55.6
2:10	113.5	85	8.7	76.3	40	134.5	85.5	30.1	55.4
25	114	85	9.0	76.0	55	134	85	29.4	55.6
40	114	85	9.0	76.0	10:10	134	85	29.4	55.6
55	115	85	9.8	75.2	25	134	85	29.4	55.6
3:10	115.5	85	10.2	74.8	40	134	85	29.4	55.6
25	116	85	10.6	74.4	55	134	85	29.4	55.6
40	116.5	85	11.1	73.9	11:10	134	85	29.4	55.6
55	117+	85	11.5	73.5	25	134	85	29.4	55.6
4:10	117.5	85	12.0	73.0	40	134	84.5	29.4	55.1
25	118	85	12.4	72.6	55	134	82.5	29.4	53.1
40	119	85	13.2	71.8	12:10	134	81.0	29.4	51.6
55	120	85	14.1	70.9	25	134	80.3	29.4	50.9
5:10	120.5	85	14.6	70.4	40	134	50	29.4	20.6
					Cook	blown at	12:40		

YIELD CALCULATIONS

WGT. OF RAW MATERIAL A.D. O.D.

WGT. OF MOISTURE SAMPLES A. B.

WGT. OF SAMPLES O.D.

PERCENT O.D. AV.

WGT. OF PULP WET O.D.

WGT. OF MOISTURE SAMPLES A. B.

WGT. OF SAMPLES O.D.

PERCENT O.D. AV.

WGT. OF PULP SCREENED WET O.D.

WGT. OF SCREENINGS O.D.

PERCENT SCREENINGS ON UNSCREENED PULP

PERCENT UNSCREENED PULP ON RAW MATERIAL

PERCENT SCREENED PULP ON RAW MATERIAL

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.

COLOR

PERMANGANATE NO. EST. BL. CONS.

CHLORINE NO. EST. BL. CONS.

WGT. PULP BLEACHED WET O.D.

WGT. BLEACHED PULP WET O.D.

WGT. MOISTURE SAMPLES A. B.

WGT. SAMPLES O.D.

PERCENT O.D. AV.

YIELD BL. PULP ON UNBLEACHED ON RAW MAT.

BLEACH LIQUOR ANALYSIS VOLUME

BLEACHING TIME TEMP. CONS. VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

EXPERIMENT No. D-8 TYPE OF COOK Sulphite DATE 9/24/'37
CHEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank
COOK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES				LIQUOR ANALYSIS	
ACTIVE CHEMICAL RATIO		AS		SPECIFIC GRAVITY	
Liquor Ratio 7.03				CAUSTICITY	
WGT. ACTIVE CHEMICAL		WGT. WATER	30.1	ACTIVE ALKALI AS NA OH	
WGT. RAW MATERIAL O.D.	4285 g.			Total SO_2	7.29
LENGTH OF COOK	11 hrs	SCHEDULE NO.		Free	6.04
DIGESTER NO.	2	WOOD	D. Fir Sapwood	Combined	1.25

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
10:00	28	85	0	85	5:00	128	85	22.2	62.8
15	54	85	0	85	15	129.5	85	23.9	61.1
30	63	85	0	85	30	130.5	85	25.1	59.9
45	74	85	0	85	45	132.5	85	27.5	57.5
11:00	88	85	0	85	6:00	133.5	85	28.8	56.2
15	96	85	0	85	15	135	85	30.7	54.3
30	100	85	0	85	30	137	85	33.4	51.6
45	102.5	85	1.4	83.6	45	138	85	34.8	50.2
12:00	104.5	85	2.5	82.5	7:00	139.5	85	37.0	48.0
15	107.5	85	4.4	80.6	15	141.5	85	39.9	45.1
30	109.5	85	5.7	79.3	30	143	85	42.3	42.7
45	110.5	85	6.5	78.5	45	145	85	45.5	39.5
1:00	111.5	85	7.2	77.8	8:00	146	86	47.2	38.8
15	112	85	7.5	77.5	15	145	85	45.5	39.5
30	113	85	8.3	76.7	30	145	84	45.5	38.5
45	114	85	9.0	76.0	45	145	83	45.5	37.5
2:00	114.5	85	9.4	75.6	9:00	145	50	45.5	4.5
15	115	85	9.8	75.2	Blown at 9:00 P. M.				
30	116	85	10.6	74.4					
45	117	85	11.5	73.5					
3:00	117.5	85	11.9	73.1					
15	118	85	12.3	72.7					
30	119	85	13.2	71.8					
45	120.5	85	14.6	70.4					
4:00	122	85	16.0	69.0					
15	123.5	85	17.4	67.6					
30	125	85	19.0	66.0					
45	126	85	20.1	64.9					

YIELD CALCULATIONS

WGT. OF RAW MATERIAL	A.D.	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP	WET	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP SCREENED	WET	O.D.
WGT. OF SCREENINGS	O.D.	
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES	A.		B.
WGT. SAMPLES	O.D.		
PERCENT	O.D.		AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

PERIMENT No. D-9 TYPE OF COOK Sulphite DATE 10/7/'37

IEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank

OK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES				LIQUOR ANALYSIS	
CTIVE CHEMICAL RATIO		AS		SPECIFIC GRAVITY	
~~RATIO~~ Liquor Ratio 7.05				CAUSTICITY	
GT. ACTIVE CHEMICAL		WGT. WATER	28.5	ACTIVE ALKALI AS NA OH	
GT. RAW MATERIAL O.D.	4285 g.			Total SO_2	7.25
ENGTH OF COOK	10 hrs.	SCHEDULE NO.	3	Free	6.00
IGESTER NO.	2	WOOD	D. Fir Sapwood	Combined	1.24

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
10:30	22	85	0	85	5:00	128.5	85	22.8	62.2
45	53	85	0	85	15	130.5	85	25.1	59.9
11:00	72	85	0	85	30	132	85	26.9	58.1
15	78	85	0	85	45	133	85	28.1	56.9
30	90	85	0	85	6:00	135.5	85	31.4	53.6
45	96	85	0	85	15	137	85	33.4	51.6
12:00	100	85	0	85	30	138	85	34.8	50.2
15	103	85	1.6	83.4	45	139	85	36.5	48.5
30	105	85	2.8	82.2	7:00	141	85	39.5	45.5
45	107.5	85	4.4	80.6	15	142.5	85	41.9	43.1
1:00	109.5	85	5.7	79.3	30	145	85	45.5	39.5
15	111	85	6.8	78.2	45	145	85	45.5	39.5
30	112	85	7.5	77.5	8:00	145	85	45.5	39.5
45	113	85	8.3	76.7	15	145	85	45.5	39.5
2:00	114	85	9.0	76.0	30	145	50	45.5	4.5
15	115	85	9.8	75.2	Blown at 8:30 P. M.				
30	116	85	10.6	74.4					
45	117	85	11.5	73.5					
3:00	119	85	13.2	71.8					
15	118.5	85	12.8	72.2					
30	120.5	85	14.5	70.5					
45	121	85	15.0	70.0					
4:00	123	85	16.9	68.1					
15	125	85	19.0	66.0					
30	125.5	85	19.5	65.5					
45	127.5	85	21.6	63.4					

YIELD CALCULATIONS

WGT. OF RAW MATERIAL A.D.	O.D.
WGT. OF MOISTURE SAMPLES A.	B.
WGT. OF SAMPLES O.D.	
PERCENT O.D.	AV.
WGT. OF PULP WET	O.D.
WGT. OF MOISTURE SAMPLES A.	B.
WGT. OF SAMPLES O.D.	
PERCENT O.D.	AV.
WGT. OF PULP SCREENED WET	O.D.
WGT. OF SCREENINGS O.D.	
PERCENT SCREENINGS ON UNSCREENED PULP	
PERCENT UNSCREENED PULP ON RAW MATERIAL	
PERCENT SCREENED PULP ON RAW MATERIAL	

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.	
COLOR	
PERMANGANATE NO.	EST. BL. CONS.
CHLORINE NO.	EST. BL. CONS.
WGT. PULP BLEACHED WET	O.D.
WGT. BLEACHED PULP WET	O.D.
WGT. MOISTURE SAMPLES A.	B.
WGT. SAMPLES O.D.	
PERCENT O.D.	AV.
YIELD BL. PULP ON UNBLEACHED	ON RAW MAT.
BLEACH LIQUOR ANALYSIS	VOLUME
BLEACHING TIME TEMP. CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

EXPERIMENT No. D-17 TYPE OF COOK Sulphite DATE 11/15/'37
CHEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank
COOK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES				LIQUOR ANALYSIS	
ACTIVE CHEMICAL RATIO		AS		SPECIFIC GRAVITY	
WATER RATIO	7.19			CAUSTICITY	
WGT. ACTIVE CHEMICAL		WGT. WATER	30.2	ACTIVE ALKALI AS NA OH	
WGT. RAW MATERIAL O.D.	4196 g.			Total SO_2	7.25%
LENGTH OF COOK	11.25 hrs.	SCHEDULE NO.	3	Free	6.01
DIGESTER NO.	2	WOOD	D. Fir Heartwood	Combined	1.24

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
11:00	18	85	0	85	4:45	124.5	85	18.5	66.5
15	35	85	0	85	5:00	125.5	85	19.5	65.5
30	61	85	0	85	15	127	85	21	64
45	79	85	0	85	30	128	85	22	63
12:00	90	85	0	85	45	129.5	85	23.7	61.3
15	95	85	0	85	6:00	132	85	26.9	58.1
30	99	85	0	85	15	133.5	85	28.8	56.2
45	103	85	1.6	83.4	30	134	85	29.4	55.6
1:00	105.5	85	3.1	81.9	45	"	"	"	"
15	107	85	4.1	80.9	7:00	"	"	"	"
30	109	85	5.3	79.7	15	"	"	"	"
45	112	85	7.5	77.5	30	"	"	"	"
2:00	113	85	8.3	76.7	45	"	"	"	"
15	113	85	8.3	76.7	8:00	"	"	"	"
30	114	85	9.0	76	15	"	"	"	"
45	115	85	9.8	75.2	30	"	"	"	"
3:00	116	85	10.6	74.4	45	"	"	"	"
15	117	85	11.5	73.5	9:00	"	"	"	"
30	118	85	12.4	72.6	15	"	"	"	"
45	119	85	13.2	71.8	30	"	"	"	"
4:00	120	85	14.1	70.9	45	134.5	85	30.0	55.0
15	121.5	85	15.5	69.5	10:00	133.5	80	28.8	51.2
30	123	85	16.9	68.1	15	133	50	28.1	21.9
					Cook blown at 10:15				

YIELD CALCULATIONS

WGT. OF RAW MATERIAL A.D.	O.D.
WGT. OF MOISTURE SAMPLES A.	B.
WGT. OF SAMPLES O.D.	
PERCENT O.D.	AV.
WGT. OF PULP WET	O.D.
WGT. OF MOISTURE SAMPLES A.	B.
WGT. OF SAMPLES O.D.	
PERCENT O.D.	AV.
WGT. OF PULP SCREENED WET	O.D.
WGT. OF SCREENINGS O.D.	
PERCENT SCREENINGS ON UNSCREENED PULP	
PERCENT UNSCREENED PULP ON RAW MATERIAL	
PERCENT SCREENED PULP ON RAW MATERIAL	

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.	
COLOR	
PERMANGANATE NO.	EST. BL. CONS.
CHLORINE NO.	EST. BL. CONS.
WGT. PULP BLEACHED WET	O.D.
WGT. BLEACHED PULP WET	O.D.
WGT. MOISTURE SAMPLES A.	B.
WGT. SAMPLES O.D.	
PERCENT O.D.	AV.
YIELD BL. PULP ON UNBLEACHED	ON RAW MAT.
BLEACH LIQUOR ANALYSIS	VOLUME
BLEACHING TIME TEMP. CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

PERIMENT No. D-18 TYPE OF COOK Sulphite DATE 12/1/'37

HEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank

OOK 〃 SCREENMAN 〃 STRENGTH TESTER 〃

REACTION VARIABLES				LIQUOR ANALYSIS	
CTIVE CHEMICAL RATIO		AS		SPECIFIC GRAVITY	
ATER RATIO	6.85			CAUSTICITY	
GT. ACTIVE CHEMICAL		WGT. WATER	29.2 l	ACTIVE ALKALI AS NA OH	
GT. RAW MATERIAL O.D.	4269 g.			Total SO_2	7.24%
ENGTH OF COOK	14.50 hrs.	SCHEDULE NO.	1	Free	5.99
IGESTER NO.	2	WOOD	D. Fir Heartwood	Combined	1.25

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
10:15	22	85	0	85	5:45	122	85	15.7	69.3
30	40	〃	〃	〃	6:00	123	〃	16.9	68.1
45	55	〃	〃	〃	15	124	〃	18	67
11:00	75	〃	〃	〃	30	125.5	〃	19.5	65.5
15	88	〃	〃	〃	45	127	〃	21	64.0
30	95	〃	〃	〃	7:00	128	〃	22	63.0
45	100	〃	〃	〃	15	129	〃	23.1	61.9
12:00	104	〃	2.4	82.6	30	130	85	24.3	60.7
15	105	〃	2.8	82.2	45	131	85	25.6	59.4
30	108	〃	4.6	80.4	8:00	132	85	26.9	58.1
45	110	〃	6.1	78.9	15	132.5	85	27.7	57.3
1:00	111	〃	6.8	78.2	30	133.5	85	28.8	56.2
15	112	〃	7.5	77.5	45	133.5	85	28.8	56.2
30	112	〃	7.5	77.5	9:00	133.5	85	28.8	56.2
45	112	〃	7.5	77.5	15	134	85	29.4	55.6
2:00	112.5	〃	7.9	77.1	30	134	85	29.4	55.6
15	113	〃	8.3	76.7	45	134	85	29.4	55.6
30	113.5	〃	8.7	76.3	10:00	134	85	29.4	55.6
45	114	〃	9.0	76.0	15	134	85	29.4	55.6
3:00	114.5	〃	9.4	75.6	30	134	84	29.4	54.6
15	115	〃	9.8	75.2	45	133	83	28.1	54.9
30	115.5	〃	10.2	74.2	11:00	133.5	83.5	28.8	54.7
45	116	〃	10.6	74.4	15	134	84.5	29.4	55.1
4:00	116	〃	10.6	74.4	30	134	85	29.4	55.6
15	118	〃	12.4	72.6	45	134	85	29.4	55.6
30	118	〃	12.4	72.6	12:00	134	85	29.4	55.6
45	118.5	〃	12.8	72.2	15	133.5	84	28.8	53.2
5:00	119	〃	13.2	71.8	30	134	84	29.4	54.6
15	120	〃	14.1	70.9	45	134	50	29.4	20.6
30	121	〃	14.6	70.4	Cook blown at 12:45				

YIELD CALCULATIONS

WGT. OF RAW MATERIAL	A.D.	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP	WET	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP SCREENED	WET	O.D.
WGT. OF SCREENINGS	O.D.	
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES	A.		B.
WGT. SAMPLES	O.D.		
PERCENT	O.D.		AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

PERIMENT No. D-19 TYPE OF COOK Sulphite DATE 12/1/'37
IEMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank
OOK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES		LIQUOR ANALYSIS	
CTIVE CHEMICAL RATIO	AS	SPECIFIC GRAVITY	
ATER RATIO	7.23	CAUSTICITY	
GT. ACTIVE CHEMICAL	WGT. WATER 31.8 1	ACTIVE ALKALI AS NA OH	
GT. RAW MATERIAL O.D.	633 g. HW; 3765 g. SW	Total SO_2	7.24
ENGTH OF COOK	10 hrs. SCHEDULE NO. 3	Free	5.99
IGESTER NO.	4 WOOD D. Fir Slab Wood	Combined	1.25

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
11:45	25	85	0	85	5:00	120.5	85	14.5	70.5
12:00	35	85	0	85	15	123.5	85	17.4	67.6
15	63	85	0	85	30	130	85	24.3	60.7
30	77	85	0	85	45	129.5	85	23.8	61.2
45	88	85	0	85	6:00	127.5	85	21.6	63.4
1:00	95	85	0	85	15	131	85	25.7	59.3
15	100	85	0	85	30	134	85	29.4	55.6
30	102	85	1.2	83.8	45	134.5	85	30	55
45	105	85	2.8	82.2	7:00	134.5	85	30	55
2:00	107	85	4	81	15	134	85	29.4	55.6
15	110	85	6.1	78.9	30	136.5	85	32.5	52.5
30	112	85	7.5	77.5	45	138.5	85	35.4	49.6
45	113	85	8.3	76.7	8:00	139	85	36.5	48.5
3:00	113.5	85	8.8	76.2	15	140	85	38	47
15	114.5	85	9.3	75.7	30	140	85	38	47
30	115	85	9.8	75.2	45	140	85	38	47
45	115	85	9.8	75.2	9:00	140	85	38	47
4:00	116.5	85	11.1	73.9	15	140	85	38	47
15	119	85	13.2	71.8	30	140	85	38	47
30	119	85	13.2	71.8	45	140	50	38	12
45	120	85	14.1	70.9	Cook	blown at	9:45		

YIELD CALCULATIONS

WGT. OF RAW MATERIAL A.D.	O.D.	
WGT. OF MOISTURE SAMPLES A.	B.	
WGT. OF SAMPLES O.D.		
PERCENT O.D.		AV.
WGT. OF PULP WET	O.D.	
WGT. OF MOISTURE SAMPLES A.	B.	
WGT. OF SAMPLES O.D.		
PERCENT O.D.		AV.
WGT. OF PULP SCREENED WET	O.D.	
WGT. OF SCREENINGS O.D.		
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES A.			B.
WGT. SAMPLES O.D.			
PERCENT O.D.			AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

PERIMENT No. D-20 TYPE OF COOK Sulphite DATE 12/21/'37
EMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank
OK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES				LIQUOR ANALYSIS	
TIVE CHEMICAL RATIO		AS		SPECIFIC GRAVITY	
ATER RATIO	7.00			CAUSTICITY	
GT. ACTIVE CHEMICAL		WGT. WATER	30.2 l	ACTIVE ALKALI AS NA OH	
GT. RAW MATERIAL	O.D. 3663 g. HW; 657 g. SW			Total SO_2	7.29%
NGTH OF COOK	14.25 hrs.	SCHEDULE NO.	1	Free	6.03
GESTER NO.	2	WOOD	D. Fir Entire Tree	Combined	1.26

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
8:00	17	85	0	85	3:15	121	85	15	70
15	37	85	0	85	30	121.5	85	15.5	69.5
30	56	85	0	85	45	122.5	85	16.5	68.5
45	76	85	0	85	4:00	123.5	85	17.5	67.5
9:00	88	85	0	85	15	125	85	19	66
15	93	85	0	85	30	127	85	21	64
30	98	85	0	85	45	127	85	21	64
45	102.5	85	1.4	83.6	5:00	128	85	22	63
10:00	105.5	85	3.1	81.9	15	129	85	23.1	61.9
15	108	85	4.7	80.3	30	130	85	24.3	60.7
30	110	85	6.1	78.9	45	130.5	85	24.9	60.1
45	110	85	6.1	78.9	6:00	132.5	85	27.5	57.5
11:00	112	85	7.5	77.5	15	133.5	85	28.7	56.3
15	112.5	85	7.9	77.1	30	134.5	86	30	56
30	112.5	85	7.9	77.1	45	134.5	86	30	56
45	112.5	85	7.9	77.1	7:00	134.5	86	30	56
12:00	113	85	8.3	76.7	15	134	85	29.4	55.6
15	114	85	9.0	76.0	30	134	85	29.4	55.6
30	114	85	9.0	76.0	45	133.5	85	28.7	56.3
45	114.5	85	9.4	75.6	8:00	134	85	29.4	55.6
1:00	115	85	9.8	75.2	15	134	85	29.4	55.6
15	115	85	9.8	75.2	30	133.5	85	28.7	56.3
30	116.5	85	11.1	73.9	45	133.5	85	28.7	56.3
45	117.5	85	12	73	9:00	134	85	29.4	55.6
2:00	117.5	85	12	73	15	135	85	30.6	54.4
15	118	85	12.4	72.6	30	134	84	29.4	54.6
30	118	85	12.4	72.6	45	134	83	29.4	53.6
45	119	85	13.2	71.8	10:00	134	80	29.4	50.6
3:00	119.5	85	13.7	71.3	15	134	50	29.4	20.6
					Cook	blown at	10:15		

YIELD CALCULATIONS

WGT. OF RAW MATERIAL A.D. O.D.

WGT. OF MOISTURE SAMPLES A. B.

WGT. OF SAMPLES O.D.

PERCENT O.D. AV.

WGT. OF PULP WET O.D.

WGT. OF MOISTURE SAMPLES A. B.

WGT. OF SAMPLES O.D.

PERCENT O.D. AV.

WGT. OF PULP SCREENED WET O.D.

WGT. OF SCREENINGS O.D.

PERCENT SCREENINGS ON UNSCREENED PULP

PERCENT UNSCREENED PULP ON RAW MATERIAL

PERCENT SCREENED PULP ON RAW MATERIAL

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.

COLOR

PERMANGANATE NO. EST. BL. CONS.

CHLORINE NO. EST. BL. CONS.

WGT. PULP BLEACHED WET O.D.

WGT. BLEACHED PULP WET O.D.

WGT. MOISTURE SAMPLES A. B.

WGT. SAMPLES O.D.

PERCENT O.D. AV.

YIELD BL. PULP ON UNBLEACHED ON RAW MAT.

BLEACH LIQUOR ANALYSIS VOLUME

BLEACHING TIME TEMP. CONS. VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

PERIMENT No. D-21 TYPE OF COOK Sulphite DATE 12/21/'37

EMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank

OK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES		LIQUOR ANALYSIS	
TIVE CHEMICAL RATIO	AS	SPECIFIC GRAVITY	
ATER RATIO 7.27		CAUSTICITY	
GT. ACTIVE CHEMICAL	WGT. WATER 29.0 l	ACTIVE ALKALI AS NA OH	
GT. RAW MATERIAL O.D. 3991 g.		Total SO_2	7.29%
NGTH OF COOK 10.75 hrs.	SCHEDULE NO. 3	Free	6.03
GESTER NO. 1	WOOD D. Fir	Combined	1.26
	Heartwood		

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
9:30	20	85	0	85	3:00	122.5	85	16.4	68.6
45	55	85	0	85	15	124	85	17.9	67.1
10:00	70	85	0	85	30	125	85	19	66
15	75	85	0	85	45	126	85	20	65
30	89	85	0	85	4:00	127.5	85	21.6	63.4
45	94	85	0	85	15	129.5	85	23.8	61.2
11:00	98	85	0	85	30	131	85	25.7	59.3
15	101	85	0.7	84.3	45	132.5	85	27.5	57.5
30	104	85	2.1	82.9	5:00	134.5	85	30	55
45	107.5	85	4.3	80.7	15	137	85	33.4	51.6
12:00	110	85	6.1	78.9	30	138.5	85	35.6	49.4
15	111.5	85	7.1	77.9	45	139	85	36.5	48.5
30	112	85	7.5	77.5	6:00	140	85	38	47
45	113	85	8.3	76.7	15	140	85	38	47
1:00	114	85	9.0	76.0	30	140.5	85	38.9	46.1
15	114.5	85	9.3	75.7	45	140	85	38	47
30	115.5	85	10.1	74.9	7:00	140	85	38	47
45	116.5	85	11.1	73.9	15	140	85	38	47
2:00	118	85	12.4	72.6	30	140	85	38	47
15	119.5	85	13.6	71.4	45	140	85	38	47
30	120	85	14.1	70.9	8:00	140	85	38	47
45	121	85	15	70	15	140	50	38	12
					Cook	blown at	8:15		

YIELD CALCULATIONS		
WGT. OF RAW MATERIAL	A.D.	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP	WET	O.D.
WGT. OF MOISTURE SAMPLES	A.	B.
WGT. OF SAMPLES	O.D.	
PERCENT	O.D.	AV.
WGT. OF PULP SCREENED	WET	O.D.
WGT. OF SCREENINGS	O.D.	
PERCENT SCREENINGS ON UNSCREENED PULP		
PERCENT UNSCREENED PULP ON RAW MATERIAL		
PERCENT SCREENED PULP ON RAW MATERIAL		

BLEACH CONSUMPTION AND BLEACHING			
STANDARD BL. CONS.			
COLOR			
PERMANGANATE NO.			EST. BL. CONS.
CHLORINE NO.			EST. BL. CONS.
WGT. PULP BLEACHED WET			O.D.
WGT. BLEACHED PULP WET			O.D.
WGT. MOISTURE SAMPLES	A.		B.
WGT. SAMPLES	O.D.		
PERCENT	O.D.		AV.
YIELD BL. PULP ON UNBLEACHED			ON RAW MAT.
BLEACH LIQUOR ANALYSIS			VOLUME
BLEACHING TIME	TEMP.	CONS.	VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

THE INSTITUTE OF PAPER CHEMISTRY

PULPING LABORATORY

PERIMENT No. WH-1 TYPE OF COOK Sulphite DATE 12/1/'37

EMIST Brookbank DATA Brookbank BLEACH TESTER Brookbank

OK " SCREENMAN " STRENGTH TESTER "

REACTION VARIABLES				LIQUOR ANALYSIS	
CTIVE CHEMICAL RATIO		AS		SPECIFIC GRAVITY	
ATER RATIO	6.33			CAUSTICITY	
GT. ACTIVE CHEMICAL		WGT. WATER	27.4 l	ACTIVE ALKALI AS NA OH	
GT. RAW MATERIAL O.D.	4326 g.			Total SO_2	7.24
NGTH OF COOK	9.75 hrs.	SCHEDULE NO.	3	Free	5.99
GESTER NO.	1	WOOD	W. Hemlock	Combined	1.25

TEMPERATURE AND PRESSURE RECORD

TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS	TIME	TEMP. °C.	PRESSURE (LBS. GAUGE) DIGESTER	STEAM	GAS
11:15	22	85	0	85	4:15	120	85	14.1	70.9
30	45	85	0	85	30	121	85	15	70
45	60	85	0	85	45	122.5	85	16.4	68.6
12:00	75	85	0	85	5:00	123	85	16.9	68.1
15	87	85	0	85	15	125.5	85	19.5	65.5
30	93.5	85	0	85	30	126.5	85	20.5	64.5
45	98	85	0	85	45	128	85	22.3	62.7
1:00	102	85	1.1	83.9	6:00	130	85	24.4	60.6
15	105	85	2.8	82.2	15	132.5	85	27.5	57.5
30	106	85	3.5	81.5	30	133.5	85	31.4	53.6
45	110	85	6.1	78.9	45	136	85	32.1	52.9
2:00	110.5	85	6.5	78.5	7:00	138	85	34.8	50.2
15	111.5	85	7.1	77.9	15	140	85	38	47
30	112.5	85	7.9	77.1	30	140.5	86	38.9	47.1
45	114	85	9.0	76	45	140	85	38	47
3:00	115	85	9.8	75.2	8:00	140	85	38	47
15	116	85	10.6	74.4	15	140	85	38	47
30	116.5	85	11.1	73.9	30	140	85	38	47
45	118	85	12.4	72.6	45	140	85	38	47
4:00	119	85	13.2	71.8	9:00	140	50	38	12
					Cook	blown at	9:00		

YIELD CALCULATIONS

WGT. OF RAW MATERIAL A.D. O.D.

WGT. OF MOISTURE SAMPLES A. B.

WGT. OF SAMPLES O.D.

PERCENT O.D. AV.

WGT. OF PULP WET O.D.

WGT. OF MOISTURE SAMPLES A. B.

WGT. OF SAMPLES O.D.

PERCENT O.D. AV.

WGT. OF PULP SCREENED WET O.D.

WGT. OF SCREENINGS O.D.

PERCENT SCREENINGS ON UNSCREENED PULP

PERCENT UNSCREENED PULP ON RAW MATERIAL

PERCENT SCREENED PULP ON RAW MATERIAL

BLEACH CONSUMPTION AND BLEACHING

STANDARD BL. CONS.

COLOR

PERMANGANATE NO. EST. BL. CONS.

CHLORINE NO. EST. BL. CONS.

WGT. PULP BLEACHED WET O.D.

WGT. BLEACHED PULP WET O.D.

WGT. MOISTURE SAMPLES A. B.

WGT. SAMPLES O.D.

PERCENT O.D. AV.

YIELD BL. PULP ON UNBLEACHED ON RAW MAT.

BLEACH LIQUOR ANALYSIS VOLUME

BLEACHING TIME TEMP. CONS. VOLUME WATER

PRESSURE AND TEMPERATURE CURVES

APPENDIX C

Detailed Physical Data

DETAILED PHYSICAL DATA--UNBLEACHED SULPHITE PULPS

Cook No.	Basis Weight 24x36--500				Burst Ratio %				Tear Factor %			
	Milling Time				Milling Time				Milling Time			
	0	60	90	120	0	60	90	120	0	60	90	120
D-3	40.0	48.2	37.4	38.8	68	101	112	117	353	266	184	142
D-4	42.0	41.8	40.5	39.9	73	97	108	117	329	238	208	161
D-5	38.3	41.4	38.0	39.2	60	80	94	105	193	171	138	104
D-6	36.5	35.6	39.8	36.2	55	73	83	84	246	197	165	138
D-7	39.8	42.1	40.4	39.2	70	96	106	107	262	195	176	142
D-9	39.1	39.9	42.2	40.4	75	104	111	115	312	237	208	172
D-17	38.4	40.3	38.0	41.6	49	86	94	117	303	200	163	133
D-18	42.0	41.3	42.0	40.1	77	99	103	110	233	164	130	114
D-19	40.1	42.5	43.0	40.8	72	97	104	111	323	253	213	170
D-20	41.8	40.9	40.0	41.8	75	94	97	105	278	188	152	139
S-2	41.7	39.5	41.4	43.1	88	136	146	147	202	146	117	106
WH-1	42.6	39.5	40.0	39.0	135	144	143	151	161	154	132	125

Cook No.	M. I. T. Fold No.				Apparent Density				Freeness ml.			
	Milling Time				Milling Time				Milling Time			
	0	60	90	120	0	60	90	120	0	60	90	120
D-3	205	858	1483	4165	9.9	12.4	12.7	13.7	865	735	600	440
D-4	272	1184	1299	2585	10.7	13.3	13.9	15.1	880	770	625	490
D-5	71	346	566	1627	10.1	12.3	13.3	14.4	855	800	615	445
D-6	27	125	521	1711	10.1	12.7	14.5	15.7	860	710	525	385
D-7	77	535	1745	4261	10.3	13.6	13.9	15.0	835	685	515	400
D-9	270	836	1257	2377	10.1	13.0	14.0	14.0	880	745	615	480
D-17	17	309	415	1528	9.5	12.2	12.7	14.1	870	795	725	370
D-18	296	751	975	1921	11.1	13.6	14.5	14.8	870	735	615	450
D-19	179	644	1019	1791	10.4	12.6	13.9	14.2	885	795	675	480
D-20	219	631	743	1898	10.9	13.4	13.6	14.8	860	735	570	380
S-2	176	818	1445	5744	11.8	14.3	15.2	16.5	800	665	490	385
WH-1	1246	1994	2989	3833	13.7	14.5	15.9	17.6	815	700	590	440

APPENDIX D

DETAILED PHYSICAL DATA--BLEACHED SULPHITE PULPS

DETAILED PHYSICAL DATA--BLEACHED SULPHITE PULPS

Cook No.	Basis Weight 24x36--500				Burst Ratio %				M. I. T. Fold No.			
	Milling Time				Milling Time				Milling Time			
	0	60	90	120	0	60	90	120	0	60	90	120
D-3	39.0	49.6	38.2	45.3	54	91	96	103	145	424	741	1106
D-4	39.8	38.3	41.8	39.8	44	72	84	98	16	88	257	956
D-5	43.2	39.8	40.0	40.1	56	89	100	103	33	395	515	962
D-6	38.4	39.8	41.8	39.8	26	52	63	79	3	21	69	240
D-7	41.3	39.3	40.0	43.6	39	76	83	97	9	105	325	933
D-9	39.4	40.6	41.6	39.8	49	68	82	98	23	191	319	914
D-19	39.6	45.3	44.8	40.4	53	80	85	102	26	167	371	823
D-20	37.9	40.5	44.1	43.2	41	78	80	94	10	201	309	659
S-2	40.8	42.1	41.5	40.5	97	132	144	139	157	949	2382	2991
WH-1	39.0	45.2	41.4	39.8	76	110	117	130	144	651	970	1982

Cook No.	Tear Factor %				Apparent Density				Opacity %			
	Milling Time				Milling Time				Milling Time			
	0	60	90	120	0	60	90	120	0	60	90	120
D-3	316	200	163	153	9.9	12.7	12.8	14.4	64.0	52.6	47.3	41.8
D-4	381	293	214	180	9.1	11.5	12.8	13.8	64.3	55.6	53.4	45.6
D-5	179	119	107	83	10.7	13.4	14.5	15.9	65.7	47.1	38.0	36.2
D-6	198	211	179	114	8.7	12.0	13.2	15.5	63.7	58.2	56.2	43.8
D-7	272	185	152	127	9.7	12.8	13.6	15.0	62.9	50.1	45.9	39.0
D-9	351	325	257	199	9.2	11.1	12.5	13.4	62.6	55.8	53.8	46.9
D-19	356	276	232	168	9.4	11.6	12.5	13.6	60.6	57.7	53.2	44.9
D-20	291	207	173	145	9.4	12.1	13.8	14.6	59.6	53.2	51.9	43.8
S-2	191	140	115	108	12.6	14.8	16.6	17.9	75.5	60.5	52.4	46.2
WH-1	253	176	146	117	10.8	14.5	14.1	14.9	65.9	57.4	48.3	40.2

Cook No.	Freeness ml.			
	Milling Time			
	0	60	90	120
D-3	885	810	675	510
D-4	870	810	720	420
D-5	875	680	525	405
D-6	870	810	625	335
D-7	885	780	660	370
D-9	865	830	740	470
D-19	875	825	745	515
D-20	885	810	680	500
S-2	795	670	540	355
WH-1	810	750	650	440

APPENDIX E

SAMPLE SHEETS

Bleached and Unbleached Experimental Pulps

D-3

Sapwood

D-4

Sapwood

D-5

Heartwood

D-6

Heartwood

D-7

Heartwood

D-9

Sapwood

D-17

Heartwood

D-18

Heartwood

D-19

Slab Wood

85% Sapwood
15% Heartwood

D-20

Entire Tree

85% Heartwood
15% Sapwood

S-2

Spruce

WH-1

Western Hemlock

D-3B

Sapwood

D-4B

Sapwood

D-5B

Heartwood

D-6B

Heartwood

D-7B

Heartwood

D-9B

Sapwood

D-19B

Slab Wood

85% Sapwood
15% Heartwood

D-20B

Entire Tree

15% Sapwood
85% H eartwood

S-2B

Spruce

WH-1B

Western Hemlock