# PERMEABILITY OF A MOUNTAIN-TYPE DOUGLAS FIR STEM CONTAINING INCLUDED SAPWOOD BANDS

by

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# A THESIS SUBMITTED IN PARTIAL FULFILMENT OF

# THE REQUIREMENTS FOR THE DEGREE OF

# MASTER

in the Faculty

of

# Forestry

We accept this thesis as conforming to the required standard

THE UNIVERSITY OF BRITISH COLUMBIA

April, 1961

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# ABSTRACT

Permeability to creosote of sapwood, included sapwood, and normal heartwood of a mountain-type Douglas fir stem was correlated with specific gravity, growth rate, percent summerwood, tracheid length, number of longitudinal resin ducts, alcohol-benzene, acetone and ether-soluble extractive contents of the corresponding zones. The effect of pressure and temperature on creosote retention was tested on creosote retention in true sapwood, included sapwood (abnormal heartwood), and normal heartwood. Test specimens were extracted in different solvents and ease of penetration tested by creosote impregnation.

Among the factors investigated in the present study, specific gravity, tracheid length, growth rate, and number of longitudinal resin ducts did not have a measurable influence on creosote retention. Percent summerwood did not vary significantly at the five positions tested.

Pressure had the greatest effect on creosote retention at 212°F. for heartwood, less for included sapwood and least for sapwood. The influence of temperature on creosote retention in Douglas fir heartwood was greater at 100 psi pressure than at atmospheric pressure. The effect of alcohol-benzene and acetone-soluble extractives on wood permeability was not proven statistically significant. A visual hyperbolic relationship was obtained between ether-soluble extractives and wood permeability. The higher the extractive content, the greater the retention. Pre-treatment of samples with different solvents, in order to remove some of the extractives, improved the permeability of heartwood and included sapwood significantly but caused only a slight improvement in sapwood.

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#### ACKNOWLEDGEMENTS

The author wishes to express his gratitude to Dr. R.W. Wellwood and Mr. R.W. Kennedy, of the Faculty of Forestry, The University of British Columbia, for their constructive direction and helpful criticism.

Special thanks are due Mr. R. Buff, Manager of the Buff Lumber Company, Westwold, British Columbia for supplying the material used in this project.

The author would also like to gratefully acknowledge the help given by the Vancouver Laboratory, Forest Products Research Branch, Canada Department of Forestry, for the free use of their facilities.

#### INTRODUCTION

The movement of liquids in wood is of great interest to several branches of the wood industry. It is particularly so in wood preservation, fire-proofing, dimensional stabilization, pulp and paper manufacturing, and also in the drying, gluing, and finishing of wood.

During the past 60 years a considerable amount of work has been done on the evaluation of the factors affecting the penetration of preservatives, especially creosote, into Douglas fir timber. As a result of these experiments a great improvement can be seen in the techniques for impregnating Douglas fir with oil- and water-borne preservatives. However, there still remains a great deal of research work to be done in this field.

It is a well known fact that Douglas fir sapwood can be impregnated with preservative liquids more easily than the heartwood. In addition, preliminary tests performed by the author this year indicated that the permeability of certain portions of Douglas fir heartwood is superior to that of others. These more permeable parts of the heartwood, interspersed among the zones of normal heartwood, have the color of sapwood, and are in fact zones of included sapwood.

This recent observation of the superior permeability of included sapwood (abnormal heartwood) over that of the normal heartwood, offers a completely new approach to the investigation of some of the factors affecting the penetration of creosote into Douglas fir.

The present investigation may be divided into three major parts. The object of the first part was to determine the pattern of absorption of creosote at different points in a section of a log of interior Douglas fir containing included sapwood bands. Various treatments were used to investigate the effect of pressure and temperature on the penetration of creosote into this material. The purpose of the second part was to determine some of the physical and chemical properties of the end-matched specimens used for the absorption studies. The physical properties investigated were specific gravity, growth rate, percent summerwood, number of longitudinal resin ducts per unit of cross section area, and fibre length. Of the chemical properties, the quantities of alcohol-benzene, acetone and ether solubles were determined. This enabled a correlation to be made of the pattern of absorption with the physical and chemical properties of the appropriate wood specimens.

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In the third part of the study, wood blocks, similar to those used in the first part of the study, were extracted with several organic solvents for a standard period of time. Following extraction the specimens were treated with creosote and the ease of penetration determined in order to test the hypothesis that the extractive content of the material affected its penetrability.

#### LITERATURE REVIEW

## 1. Physical and Chemical Structure of Douglas Fir Wood

(a) Physical properties (7)

Douglas fir wood is composed of two major types of elements, longitudinal and transverse. The longitudinal elements consist primarily of wood tracheids and secondly of epithelial parenchyma cells of the resin canals. These longitudinal elements constitute over 90 percent of the volume of most softwoods.

The tracheids are hollow cellulosic tubes tapered and closed at both ends, and somewhat rectangular or elliptical in cross-section (13). The individual tracheids are connected with each other by bordered pits, which are hence important for the movement of liquids both in the living

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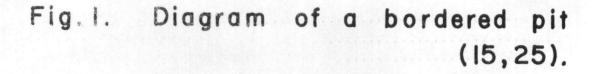
and dead tree. A diagram of such a bordered pit is shown in Figure 1. Great importance is attached to the fine structure of the pit membrane of bordered pits since the preserving liquid must pass through the permanent pores of this membrane. The pits are located on the radial walls of the springwood tracheids, and are concentrated towards the ends. They occur mainly in a single vertical row on the cell wall, but occasionally form double rows. The pits in the summerwood zone are less numerous, smaller in size and may be located on both the tangential and radial walls, depending upon the particular species.

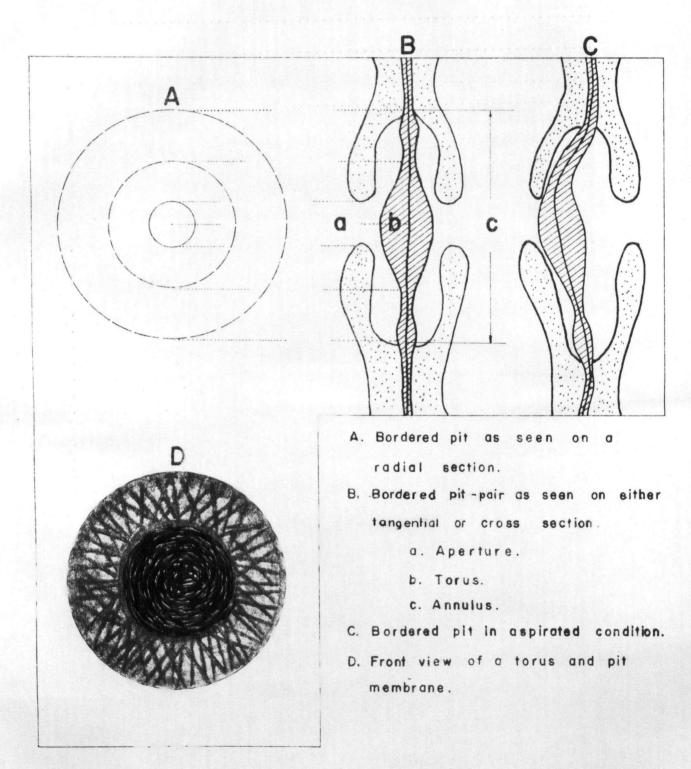
The bordered pit has an overhanging rim, more or less circular in surface view, and the pit is divided by the closing apparatus. The closing membrane consists of the torus and the pit membrane. The torus is circular in outline, and is of sandwich construction. The thin middle lamella is held between, or holds together, the two primary wall thickenings. The pit membrane consists of a network of cellulosic filaments radiating from the torus to the margin of the pit cavity. The filaments appear to arise from the two faces of the torus and to be made of microfibrils joined into coarser strands (15, 17, 25, 26, 33, 34, 36). Springwood cells are characterized by relatively thin walls, with rather long overhanging pit borders, whereas the summerwood cells have thick walls, small pit cavities and thick, short tori. Stamm (48) measured the size of the permanent pores in the pit membrane by physical methods, and reported an average pore diameter of 28.2 millimicrons.

Buckman and associates (10) found that the effective diameters of the permanent pores in the pit membrane vary with the moisture content of the wood. Below the fibre-saturation point the effective pore diameter

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decreases with increasing moisture content. Recent results obtained by Smith (46) indicated that the average size of the openings controlling flow through certain softwoods is equivalent to a diameter of about 2-3 microns. Marts(36) reported an average diameter of 23 microns for the border, 11 microns for the torus and 7 microns for the aperture of the Douglas fir bordered pits.

In Douglas fir, longitudinal parenchyma cells are quite sparse and scattered throughout the growth rings. Their function in the living tree is to store reserve food and extractives. The parenchyma cells can be identified by their flat, blunt ends and the presence of simple pits in the cell wall. Overhanging rims, pit cavities, and tori are lacking in these simple pits. Thus simple pit pairs are merely round holes in the contiguous cell walls, with a dividing membrane between.

Epithelial parenchyma cells lining the vertical resin ducts are found in Douglas fir. The resin ducts are postcambial in formation and occur only as intercellular spaces in the wood.

The transverse or radial wood elements are the wood ray cells. Wood rays are of two different types, uniseriate and fusiform. The uniseriate rays are generally one cell in width and from one to many cells in height. In Douglas fir, ray tracheids form the marginal cells of the ray, and the other cells are parenchymatous. The fusiform ray consists of the same elements as the uniseriate with the addition of epithelial cells that surround a transverse resin duct. As a result, these rays are several cells wide in the middle and taper to one cell in width at the margins. The main functions of wood rays are food storage and translocation from the inner bark to the living cells in the tree stem.

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The stem is built up of growth rings or annual rings, normally one being formed every year. Each growth ring contains two distinct zones, namely, springwood and summerwood. These zones are the result of rapid growth at the beginning of each growing season. During the period of fast growth, when conduction of water and raw materials to the crown is important, thin-walled tracheids with large lumens are formed. As growth slows down in the latter part of the growing season, thick-walled tracheids are formed. The abrupt transition between the thick-walled summerwood tracheids of one year's growth, and the larger thin-walled springwood tracheids of the next year, makes the annual ring distinct in appearance.

A Douglas fir stem can be divided into two major zones, namely, the sapwood and heartwood. All cells in the heartwood portion of the stem are dead and their function is mechanical support. In the sapwood the tracheids die shortly after they are formed, but still function as conducting elements. As new cells are continually formed by the cambium, and added to the sapwood, a proportionate number of the old sapwood cells are converted to heartwood. These cells, as they become part of the heartwood, possess a higher resistance to the entry of fungi and preserving liquids than the sapwood cells. In addition, they lose all their functions except mechanical support.

In the case of the so-called included sapwood, the conversion of sapwood to heartwood appears to be incomplete. This assumption is confirmed by the low extractive content, and by the lack of coloring matter in the included sapwood zones.

Certain changes take place in wood during the conversion of sapwood to heartwood. Numerous pits become aspirated. A bordered pit becomes aspirated when the torus moves to one side of the pit cavity and closes the

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pit aperture. In addition, resins and other extractives present in the sapwood usually become hard and remain deposited within the resin ducts and cell lumina in the heartwood. However, no basic change takes place in the structure of the wood.

# (b) <u>Chemical structure</u>

Chemically, the fully matured cell wall consists of varying amounts of cellulose, lignin and non-cellulosic polysaccharides. Cellulose is the skeleton around which the other substances are deposited. This substance is considered to consist of long molecular chains of glucose residues. The long chains of cellulose molecules in the cell walls are parallel over at least part of their length. In these zones of parallelism the units of glucose anhydride are bonded lengthwise as well as crosswise. The parallel replication of the cellulose chains builds up the whole crystalline structure of cellulose.

Between the crystallites in the amorphous regions, the cellulose chains are only partially parallel. They are somewhat disorganized and thus cross valences are lacking or greatly reduced. The advent of electron microscopy and its application to cell wall studies revealed the presence of well defined units called microfibrils, which in different cellulosic materials average approximately 200  $\mathring{A}$  in breadth and vary from 25 - 100  $\mathring{A}$  in thickness (15). These microfibrils are of indefinite length and are apparently somewhat rectangular in cross-section. Frey-Wyssling, as stated in Dadswell (16), further suggested that a microfibril, with a cross-section of 100 x 200  $\mathring{A}$ , consists of four so-called elementary fibrils each containing a crystalline core, separated from each other by regions of lower order of crystallinity. Most of the non-cellulosic polysaccharides, and lignin, are packed between the microfibrils.

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Extraneous components can be subdivided into two groups. The first group, called "extractives", is composed of chemicals which can be removed easily by neutral solvents. Among these extractives are substances such as resin acids, colouring matter, and waxes. The second group consists of miscellaneous components such as starch grains, silica, and calcium oxalate crystals. These are substances which cannot easily be removed by solvents, but nevertheless are quite distinct from the cell walls.

Extractives are generally found in the cell cavities. They may also be present in very fine capillaries of the cell wall, thereby making their complete removal impossible.

The over-all percentage composition of Douglas fir heartwood was determined by Graham and Kurth (23). The sample was taken from a wideringed, freshly-cut, second-growth Douglas fir. The extractive contents were based on the oven-dry weights of the unextracted wood and were determined successively for ether, alcohol, and hot-water-soluble extractive. Other components of wood were based on the weights of oven-dry extracted wood. Their results are shown in Table 1.

TABLE 1. The over-all composition of Douglas fir heartwood (23).

Extractives and constituents	Percentage		
Moisture	9.10		
Ether solubility	1.32		
Alcohol solubility	5.46		
Hot-water-solubility	2.82		
Total extractives:	9.60		
Ash	0.175		
Lignin (total sample)	30.15		
(40-60 mesh sample)	29.35		
Holocellulose	71.40		
Pentosan	10.11		
Methoxyl	4.75		

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2. General

In the past a great deal of experimental work has been done on the penetration of preservative liquids into wood. Most of these studies included the investigation of the pathways through which preservatives can enter wood.

Tiemann (51), in 1909, explained the permeability of wood on the basis that seasoning or drying causes the formation of narrow, spiral, microscopic checks in the tracheid walls, thus producing means of penetration. In addition, he stated that the larger these openings are, the more permeable the wood is to preservatives.

Weiss (52) confirmed the above theory, and explained the superior permeability of summerwood over springwood by the fact that the thickwalled summerwood cells check more than the thin-walled springwood cells, thus providing larger channels for liquid movement. On the other hand, Gerry (21) attributed no significance to these microscopic splits, in the penetration of creosote into larch.

Bailey (2) was the first investigator to realize the importance of bordered pit membranes in the impregnation of coniferous woods by liquids. The positions of the tori in the bordered pits was studied by Griffin (25). Shefound that the majority of the tori were in the aspirated condition in those wood specimens which showed poor penetration of preservatives. However, in the specimens that obtained good treatment, most of the tori were in central position. She reported that the aspiration was caused by drying.

The relation of the aspirated bordered pits to the unaspirated ones, as a percentage, was determined by Stamm (47). He found that this ratio was 40 percent for coast-type Douglas fir and only 14.6 per cent for mountain-type Douglas fir.

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Phillips (38) substantiated Griffin's findings and believed that the aspiration was due to the loss of the last trace of free water in the cell. He found that all the springwood pits became aspirated upon drying, but a portion of the summerwood pits remained unaspirated. This was explained by the difference in rigidity of the summerwood and springwood pit membranes, and by the size of the pit apertures.

In 1936, Stone (49) investigated the bordered pits of Douglas fir. No relationship was found between the degree of aspiration of the pits and the penetrability of liquids into the wood. The majority of the tori appeared to be in a completely aspirated position when he observed 2-micronthick Douglas fir sections under a compound binocular microscope at 440 X magnification. However, by means of photomicrographs taken in ultra violet and polarized light, the same tori were observed as not being fully aspirated at magnifications up to 9000 diameters. Stone explained that the tori were not completely aspirated because their surfaces were not smooth, but were quite irregular in nature. This was confirmed by other investigators. He also stated that liquid would have little difficulty in passing through the space between the over-hanging lamella and the edge of the torus.

These findings were confirmed by Erickson, Schmitz and Gortner (18). They concluded that either pit aspiration does not occur as extensively as reported in the literature, or else it does not greatly influence permeability.

In his recent study with water-borne preservatives, Preston (40) concluded that the major portion passes through the transient cell-wall capillaries, and only a small portion goes through the bordered pits. He supports this statement by the fact that the number of transient cell-wall

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capillaries far exceeds the number of permanent pores in the pit membranes. According to Stamm (48), the fractional cross-sectional area of the transient cell wall capillaries is of the order of 0.1, whereas that of the permanent pores of the pit membrane is of the order of 0.004. Taking the above factors into consideration, he attributes a minimum importance to the aspiration of bordered pits in determining the permeability of wood to water or similar liquids.

Proctor and Wagg (41) found that the number of resin ducts per unit area in the coast-type Douglas fir was seven times that of the mountaintype Douglas fir. They also found more longitudinal resin ducts in the wider growth rings and concluded that there may be a relationship between treatability and the number of resin ducts. Fleisher (19) related permeability of Douglas fir to lumen cross-sectional area and fibre length. He found the lumen cross-sectional area to be larger in permeable Douglas fir than in the impermeable type.

Summerwood is generally more permeable than springwood in Douglas fir heartwood. Earlier investigators (25, 26, 30) explained this by the displacement of tori to a greater degree in springwood than in summerwood. The following reasons may account for the difference in permeability of springwood and summerwood:

- (1) The resin ducts were found to be localized mainly in the summerwood zones of the growth rings.
- (2) The bordered pits in the summerwood tracheids are smaller and fewer in number than in the springwood fibres, but they often occur as pit canals with the torus in the dividing membrane apparently lacking (7).

- (3) The summerwood lumina are also smaller than springwood lumina, and capillary action of the preservative may account for greater penetration.
- (4) Weiss (52) suggested that the dense, thick-walled summerwood tracheids check more readily than the light, thin-walled springwood cells, thus accounting for the greater penetration of creosote in summerwood.

It was suggested by Buro and Buro (12) that the position of the torus is not the only factor responsible for penetrability. They attribute some significance to the substances deposited in the cell wall. Miller (37), based on his studies of the permeability in Douglas fir, arrived at a similar conclusion. He states that permeability may be associated with both the minute structure and the chemistry of wood.

#### MATERIAL AND METHODS

#### 1. Material

A four-foot section of a green Douglas fir (<u>Pseudotsuga menziesii</u> (Mirb.) Franco) log, 20 inches in diameter, and about 340 years old, was used for this investigation. The log, as viewed in the cross section, displayed a "target ring" pattern of heartwood and included sapwood zones (Figure 2). The material was free from surface defects and had a slope of grain less than one in twenty. The tree was cut approximately 12 miles southeast of Kamloops, at an elevation of 3500 feet, and therefore represents the mountain-type (or interior-type) of Douglas fir.

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# Figure 2. A section of an interior-type Douglas fir timber.

# 2. Methods

#### A. Absorption Studies

(a) <u>Preparation of test specimens</u>. Since the Douglas fir log section contained two included sapwood bands, one set of three side-matched specimens of  $\frac{3}{4}$ " x  $\frac{3}{4}$ " x 36" was prepared from the true sapwood, one set from each of the two included sapwood zones and one each from two normal heartwood zones (Figure 2). The side-matched specimens were carefully prepared in such a way that each would contain the same annual rings. Eighty  $\frac{3}{4}$ -inch cubes were then prepared from each of the five sets and labelled simultaneously.

(b) <u>Conditioning to 14 percent moisture content</u>. Following preparation, the test specimens were placed in an electrically-controlled conditioning

chamber. A constant relative humidity of 74 percent was maintained in the conditioning chamber, at 74°F. dry-bulb and 68°F. wet-bulb temperature. Test samples were removed from the chamber periodically, and their moisture contents determined. Six weeks was adequate to obtain a uniform equilibrium moisture content of 14 percent in all test specimens.

(c) <u>Sealing</u>. To measure the amount of creosote absorbed in radial, tangential, and longitudinal directions, the appropriate face of the cubes was left unsealed and the remaining five faces sealed. Five sides of the test specimens (rather than four) were sealed, in order to provide a more reliable measure of longitudinal penetration in the relatively short specimens. The control specimens were sealed on three neighbouring sides and left open on the opposite sides. It was necessary to sand the blocks before sealing in order to prevent the formation of channels which would permit the movement of liquid under the sealer. The test specimens were numbered with a red wax pencil before the application of the sealer, for easy identification after treatment.

The sealer was made by dissolving a plastic material (in this case a plastic ruler) in acetone. The viscosity of the solution was regulated by the addition of solvent. The sealer was spread onto the blocks with paint brush. Several coats were applied in order to provide a continuous film over the end-grain surfaces of the test specimens. The first coat of sealer applied was a solution of low viscosity. The main reason was to provide adequate penetration for the establishment of a good mechanical bond between the sealer and the wood. The second reason was to aid in the escape of air from the surface of the wood, which would otherwise appear under the sealer in the form of air bubbles. The following coats were of a higher viscosity solution. The evaporation of acetone from each coating took approximately five minutes.

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# (d) Treatment Conditions

# (i) Pressure

Two pressures were employed to treat the test specimens - 100 psi and atmospheric pressure. The application of pressures greater than 100 psi was not considered because the combined effect of high pressure and temperature might have caused collapse in the wood specimens. The pressure treatment was done at the Vancouver Laboratory, Forest Products Research Branch, Canada Department of Forestry, using the small pressure retort shown in Figure 3.

# (ii) Temperature

Half the blocks were treated at room temperature  $(70^{\circ}F.)$  and the other half at the boiling point of water  $(212^{\circ}F.)$ . The  $212^{\circ}F.$  temperature of the preservative was maintained by keeping the treating apparatus in boiling water for the entire treating period.

#### (iii) Duration of treatment

An 8-hour time period was applied in all treatments. This duration was determined from a preliminary treatment performed at room temperature and atmospheric pressure.

A sapwood control specimen was attached to the arm of a scale in such a way that the specimen could be immersed in creosote. Immediately after the immersion, the submerged weight of the specimen and attachment was determined to the nearest centigram. Weight readings were then taken at 15-minute intervals for the first 2 hours and hourly for the next 8 hours. Since the volume of the specimen presumably remained constant, the increase in weight was equal to the amount of creosote absorbed by the wood. Absorption values, reported in Table 6, were plotted against time and the 8-hour treating period determined from the graph on Figure 4.

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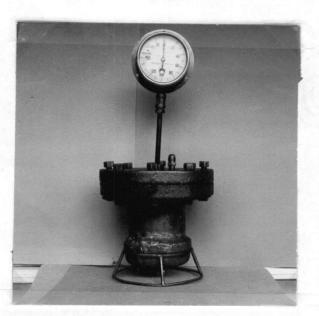
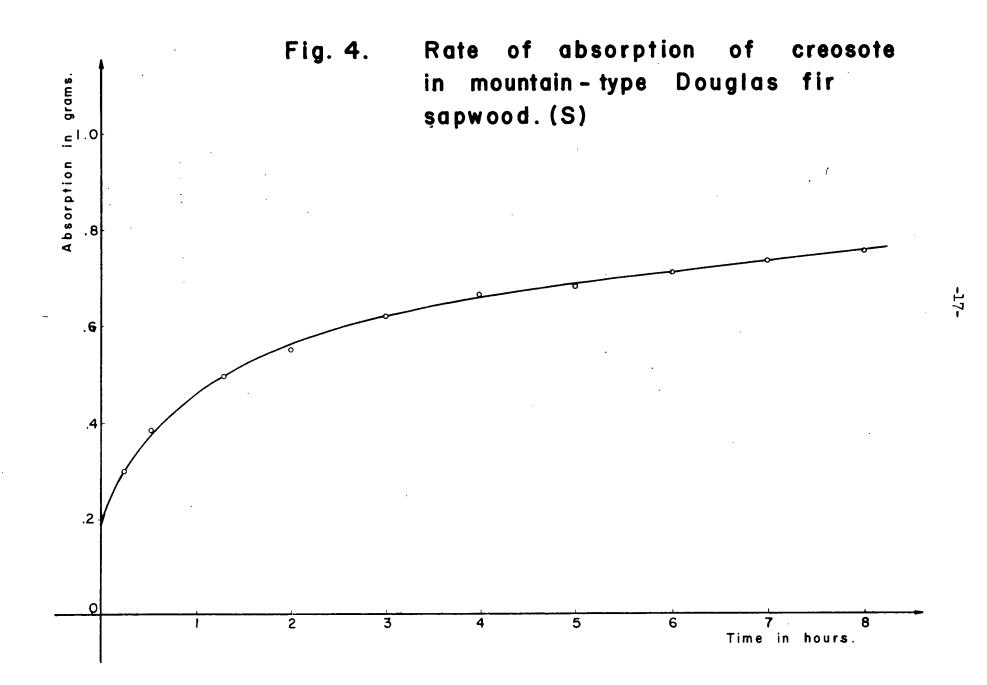


Figure 3. A small pressure retort.



#### (e) Preservative

Coal-tar creosote was used in this investigation. Creosote was taken from the same source throughout the experiment in order to eliminate errors introduced due to viscosity and specific gravity differences.

# (f) Measurement of absorption

Following the sealing process the test specimens were weighed and separated into four treatment groups. The first group was treated at atmospheric pressure and room temperature (70°F.), the second at atmospheric pressure and  $212^{\circ}$ F temperature, the third at 100 psi pressure and  $70^{\circ}$ F., and finally the fourth group at 100 psi pressure and  $212^{\circ}$ F. The excess creosote was removed from the specimens and their weight redetermined. The amounts of creosote, retained in the wood specimens in grams, were calculated and recorded (See Table 7).

B. <u>Determination of Some of the Physical and Chemical Properties of Wood</u>
(a) <u>Physical properties</u>

(i) <u>Specific gravity</u>. Four test specimens were taken from each of the five groups of specimens and their specific gravities determined using the water displacement method (7).

(ii) <u>Percent summerwood</u>. Five end-matched wood blocks were taken from the test specimens used in the absorption studies. Each block was aspirated for approximately 10 hours before 30-micron transverse sections were cut on a sliding microtome. Slides were then prepared from the sections. Mork's definition, which states that summerwood starts where twice the double radial tracheid wall thickness equals the radial diameter of the lumen, was considered the point of initiation of summerwood (45).

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(iii) <u>Growth rate</u>. The slides prepared for percent summerwood determinations were used to measure the number of rings per radial inch.

(iv) <u>Tracheid length</u>. Match-stick size pieces were split from each of the five samples in such a way that each included the full range of growth rings. The samples of each group were boiled in separate test tubes for a period of four hours in a solution of equal volumes of glacial acetic acid and hydrogen peroxide. Following the maceration, the pulp was washed overnight in running water. The fibres were stained, then dehydrated, using a slow alcohol series. Two slides from each of the five groups were prepared for tracheid length determination. Ten tracheids were measured on each slide and 20 from each group. An inverted microscope was used in this experiment. Both springwood and summerwood tracheids were randomly measured, no attempt being made to separate the two types.

(v) <u>Longitudinal resin ducts</u>. The total number of longitudinal resin ducts was determined on each slide prepared for percent summerwood determination. They area of each section was then measured and the number of resin ducts per square inch computed.

#### (b) Chemical properties

In the course of this part of the investigation, the same type of end-matched specimens were used as in the previous studies. Sample "S" was taken from the true sapwood, samples "A" and "D" from the true heartwood, and samples "B" and "C" from the included sapwood (Figure 2). Alcohol-benzene, acetone, and ether-soluble extractive contents of the five sets of samples were determined in accordance with Tappi Standards T6-m 59 and T5-m 59, respectively (1).

The extractive content of wood was reported as percentage by weight of the soluble matter in the moisture-free wood. Two determinations were performed in each case, and the results recorded as the averages of the two

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values.

#### C. Extraction Prior to Impregnation

# (a) Preparation of test specimens

Samples were taken from the sapwood (S), included sapwood (C), and true heartwood (D) zones (Figure 2). End-matched specimens,  $\frac{3}{4}$ -inch in cross-section and  $\frac{1}{2}$  inch along the grain, were prepared from each zone and numbered simultaneously. Each sample was then sanded on a belt sander and inspected for natural defects. A total of 58 specimens was prepared (See Table 13).

# (b) Extractions

One-third of the test specimens from each zone (eight) were treated with various organic solvents, using the Soxhlet extraction method; another third by the hot extraction method; while the remaining third controls were not treated.

For the Soxhlet-type extraction, the wood blocks were placed in the Soxhlet apparatus, and the solvent in the flask boiled briskly in order to ensure six to eight siphonings per hour.

In the hot extraction method, the wood blocks were boiled in various solvents, which were changed periodically to maintain their effectiveness.

The detailed set-up of the extraction study is presented in Table 13. A standard extraction period of 240 hours, or 10 days, was used for both types of extraction. The 240-hour period was chosen in order to ensure that a sufficient length of time was allowed for the solvent to reach the middle portions of the relatively impermeable heartwood. The dimension of the test specimens along the grain was reduced from  $\frac{3}{4}$  inch to  $\frac{1}{2}$  inch, in order to improve the longitudinal penetration of the solvent. These solvents were

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alcohol-benzene (1:2), acetone, ether, sodium hydroxide (0.1%) and water. The effect of the extraction period was not undertaken in this study.

Two specimens from the included sapwood (C) and two from the heartwood (D) were treated by the hot extraction method and two from each by the Soxhlet-type extraction. Thus, the number of wood specimens extracted by each procedure described below amounted to eight (See Table 13). Only the hot water type of extraction was employed in the treatment of sapwood. A total of three sapwood specimens received extraction treatment.

(i) <u>Alcohol-benzene (1:2), ether, acetone, and hot water extraction</u>. The extraction procedure was started with alcohol-benzene, followed by ether, acetone and water. The period of extraction in each solvent was 60 hours, giving a total of 240 hours.

(ii) <u>Alcohol-benzene</u>. The test specimens were extracted in alcoholbenzene (1:2) for 220 hours, in alcohol for 10 hours and finally boiled in water for another 10-hour period. The purpose of the 10-hour extraction in ethyl alcohol was to remove the benzene from the wood, and the water extraction, to replace the alcohol with water.

(iii) <u>0.1% Sodium hydroxide extraction</u>. This solvent was used to remove or change some of the carbohydrates and lignin in the wood in order to improve its permeability. The specimens were extracted in the solvent for 230 hours, and in water for an additional 10-hour period. The purpose of water extraction was to remove the residual sodium hydroxide from the wood.

(iv) <u>Water</u>. The total extraction time in water was 240 hours, or 10 complete days.

## (c) Conditioning to 14% moisture content

Following extraction, both the extracted and unextracted test specimens were placed in an electrically-operated conditioning chamber.

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This chamber was set at 74°F. dry-bulb and at 68°F. wet-bulb temperature, which provided a 74 percent relative humidity. This in turn brought about 14 percent equilibrium moisture content in the wood.

## (d) Sealing

Following conditioning, the end grains of the test specimens were sealed with plastic sealer in the manner previously described.

# (e) Measurements of absorption after extraction

Both the extracted and unextracted test specimens were weighed. They were then submerged in creosote at room temperature and atmospheric pressure, for an eight-hour period. After this treatment the excess creosote was removed from the surface of the blocks and their weights determined. Retention values were then calculated and recorded (see Table 13).

#### D. Microscopic Studies

## (a) <u>Preparation of slides</u>

Twenty-micron thick tangential sections were cut on a sliding microtome from each of the five zones. The sections were stained with analine safranin stain, taken through the alcohol series, cleared in xylene and mounted in Canada balsam.

# (b) <u>Microscopic observations</u>

A monocular microscope was employed to study the degree of aspiration in the 20-micron tangential sections. Approximately 600 X magnification was used in this study.

# RESULTS

#### A. Absorption Studies

Creosote retention values of the specimens are included in Table 7. The average values are presented in Table 2. The effect of direction of

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penetration on retention values for one set of test conditions is shown in Figure 5. The influence of pressure and temperature on creotote retention in samples tested is given in Table 3, and in Figures 6, 7, and 8.

An analysis of variance for all the retention data is given in Table 8. Standard methods of analysis, as outlined by Cochran and Cox (14), were used to determine the significance of each factor in this study.

## B. Physical and Chemical Properties of the Wood

(a) <u>Physical properties</u>

(i) <u>Specific gravity</u>. The specific gravities of the sapwood, included sapwood, and the heartwood zones are presented in Tables 4 and 9, and in Figure 9D. Sapwood had the lowest specific gravity of the five zones tested, whereas the specific gravity of heartwood was fairly constant. No major difference was found between the specific gravities of heartwood and included sapwood zones.

(ii) <u>Percent summerwood</u>. Percent summerwood values are shown in Tables 4 and 10, and in Figure 9C. The average value remained fairly constant through the five zones. Results varied from 22 to 26 percent. The accuracy of the method used to determine percent summerwood was approximately ± 3 percent. The apparent relationship between permeability and percent summerwood was not accepted as being significant.

(iii) <u>Growth rate</u>. Table 4 contains the average growth rate values of the five zones. It can be observed from Table 4 and Figure 9B that the growth rate decreased from pith to bark.

(iv) <u>Resin ducts</u>. The number of longitudinal resin ducts per unit area in each zone is recorded in Table 4 and in Figure 9G. No definite pattern could be observed in the cross section.

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(v) <u>Fibre length</u>. The tracheid length values are presented in Tables 4 and 11, and in Figure 9B. Tracheid length increased from pith to bark at a fairly steady rate. A slight drop could be observed in the "D" heartwood zone near the bark.

## (b) <u>Chemical properties</u>

Alcohol-benzene (1:2), acetone, and ether-soluble extractives are given in Tables 4 and 12 and Figure 9F. Correlation between the extractive contents and creosote retentions are presented in Figures 10, 11, and 12. Each of the five wood samples contained a greater amount of alcoholbenzene-soluble extractives than either acetone or ether solubles. In all three cases, more extractives were removed from the D heartwood zone than any other zone.

#### C. Extraction Studies

Creosote retentions of the extracted and unextracted specimens, following an eight-hour treatment at room temperature and atmospheric pressure, are presented in Table 13. Average values calculated from Table 13 are entered in Table 5.

The ratios between the retention values of extracted and unextracted specimens for sapwood, included sapwood, and heartwood were 1.2, 6.5, and 8.1 respectively. Greatest improvement in penetrability resulted from extraction with water. Permeability increase was slightly less with 0.1 percent sodium hydroxide, least with extraction in alcohol-benzene alone.

#### DISCUSSION

#### 1. Effect of pressure on creosote retention

At a temperature of  $212^{\circ}$ F. and pressure of 100 psi, an average retention of 1.11 grams was obtained for heartwood (D) (Table 2). At the

-24-

same temperature, but at atmospheric pressure, the retention was only 0.17 grams. Consequently, the specimens treated at 100 psi pressure absorbed 553 percent more creosote than those at atmospheric pressure. Similar calculations were performed for the other zones and the results, in percentages, entered in Table 3. An increase in retention of 208 percent was obtained in included sapwood (C) and 115 percent in sapwood. From the above data, and from Figures 6,7 and 8, it may be observed that the effect of pressure on creosote retention is greatest for heartwood (D), less for included sapwood (B and C), and least for sapwood.

The influence of pressure at  $70^{\circ}$ F. on creosote retention is somewhat different than at  $212^{\circ}$ F. Only 150 percent increase in retention was obtained for heartwood (D), 78 percent for included sapwood (C) and 216 percent for sapwood. This would indicate that the influence of pressure on creosote absorption in heartwood is greater at higher temperatures than at lower ones. The opposite holds for sapwood. Pressure has a greater effect on retention at lower temperatures than at higher ones. Consequently, the application of high (treating) pressures and temperatures appears to be more advantageous for heartwood than for sapwood. In sapwood the influence of pressure on retention is far greater than that of the temperature. Hence in the treatment of sapwood, the treating pressure is the dominant factor, with little significance attached to the temperature. In the case of heartwood, however, both the treating pressure and temperature are of major importance.

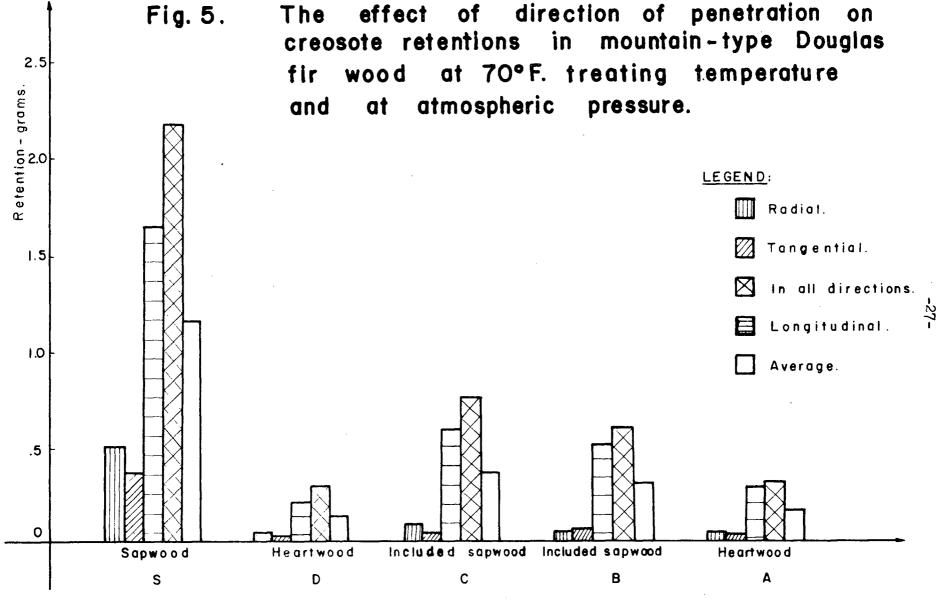
When treating sapwood under high pressure, a rise in temperature does not seem to be economically justified. In practice, however, the narrow sapwood band is rarely, if ever, treated without the heartwood. On the other hand, often the wood preserving industry is only interested in impregnating the sapwood.

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Position	Pressure	Atmosp	heric	100 p <b>si</b>		
in the	Temperature	70°F.	212 <sup>0</sup> F.	70 <sup>0</sup> F.	212 <sup>0</sup> F.	Averages
Cross- section	Direction of Penetration	(	Retention	1 - grams	;)	·
True Sapwood S	Radial Tangential Longitudinal All (control) A v e r a g e	0.49 0.31 1.63 2.17 1.15	1.50 0.80 2.04 2.65 1.75	2.12 4.15 4.15 4.09 3.63	3.36 3.90 3.82 4.00 3.77	1.87 2.29 2.91 3.23 2.58
True Heartwood (outer) D	Radial Tangential Longitudinal All (control) A v e r a g e	0.05 0.03 0.21 0.28 0.14	0.08 0.07 0.24 0.28 0.17	0.10 0.08 0.55 0.68 0.35	1.02 0.09 1.29 1.23 1.11	0.31 0.23 0.57 0.62 0.43
Included Sapwood C	Radial Tangential Longitudinal All (control) A v e r a g e	0.09 0.05 0.59 0.76 0.37	0.15 0.07 0.95 1.29 0.62	0.22 0.15 1.28 0.98 0.66	1.65 1.30 2.55 2.14 1.91	0.53 0.39 1.34 1.29 0.89
Included Sapwood B	Radial Tangential Longitudinal All (control) A v e r a g e	0.06 0.07 0.52 0.61 0.32	0.17 0.17 0.96 1.02 0.58	0.21 0.16 0.93 1.03 0.58	1.22 1.39 2.00 2.21 1.71	0.42 0.45 1.10 1.22 0.80
True Heartwood (inner) A	Radial Tangential Longitudinal All (control) A v e r a g e	0.05 0.04 0.28 0.31 0.17	0.05 0.07 0.36 0.50 0.25	0.12 0.09 0.61 0.75 0.39	0.92 1.27 1.64 1.68 1.38	0.29 0.37 0.72 0.81 0.55
AVERAGE:		0.43	0.67	1.12	1.98	1.05

o

TABLE 2. Average creosote retentions of the five zones of a mountain-type Douglas fir stem under different conditions of treatment.



Position in the cross section.

Sutherland (50) attributes the improved absorption at high pressures to an enlargement of the pores in the pit membrane.

# 2. Influence of temperature on creosote retention

An average retention of 1.38 grams was obtained for heartwood (A) at 100 psi pressure and 212° F. (Table 2). Under the same pressure, but at 70°F., the average retention was only 0.39 grams. Thus a temperature change of 142°F. resulted in a retention increase of 254 percent in heart-(B) wood, 195 percent in included sapwoo<u>d</u>/ and only 4 percent in sapwood (Table 3). This indicates that temperature had the greatest influence on heartwood, less on included sapwood, and least on sapwood.

The above order is different at atmospheric pressure. Temperature appears to have very little influence on creosote retention of heartwood. It may be concluded, therefore, that the application of high temperature, without elevated pressure, does not have a significant influence on creosote retention. In other words, in treating heartwood, the application of both high pressure and temperature is essential.

In sapwood, approximately the same retentions were obtained at 70°F. and atmospheric pressure as in heartwood at 212°F. and 100 psi pressure. This clearly demonstrates the superior permeability of sapwood over heartwood.

The influence of temperature on creosote retention in Douglas fir heartwood is greater at 100 psi pressure than at atmospheric pressure. The reverse is true of sapwood. Here temperature has a greater influence on retention at atmospheric pressure than at 100 psi pressure (Figure 8).

The effect of temperature on the absorption of creosote can be explained by a change in viscosity of creosote with a change in temperature. Creosote has higher viscosity at 70°F., but becomes increasingly thinner and

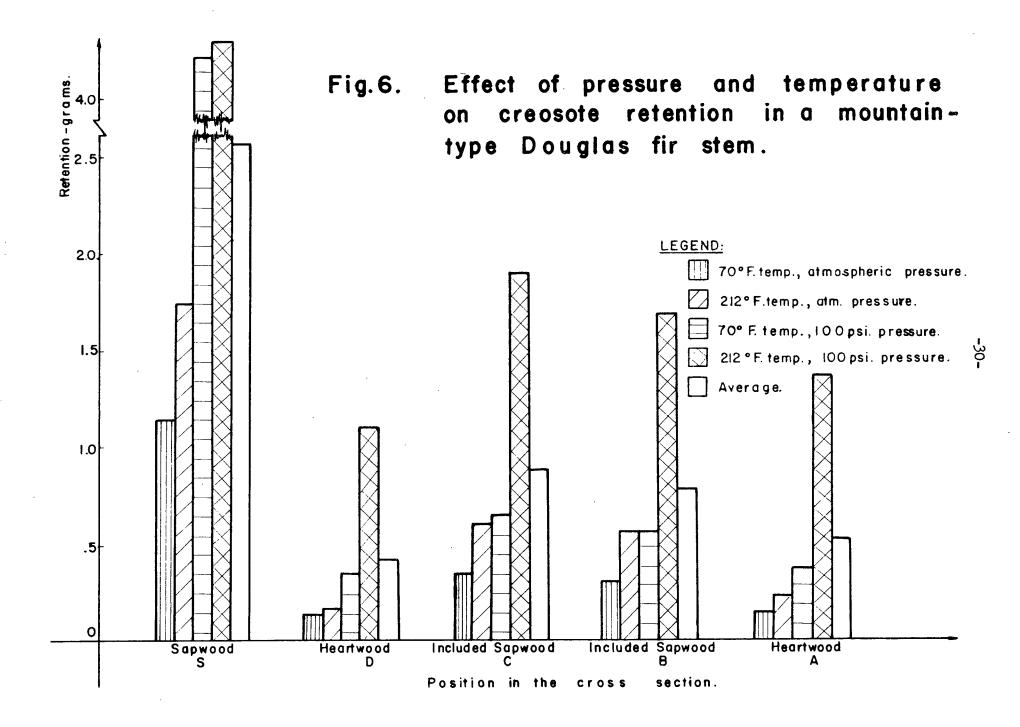
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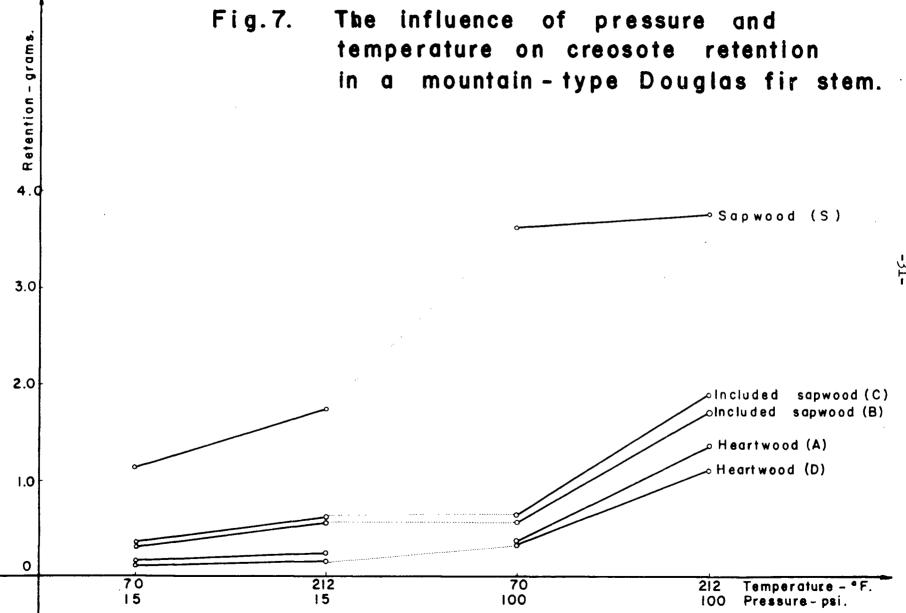
	Increase in Creosote Retention - Percent								
Kind of Wood		ncrease from c to 100 psi :	Temperature Increase from 70 <sup>°</sup> F. to 212°F. at:						
	70°F.	212 <sup>0</sup> F.	Atmospheric Pressure	100 psi					
Sapwood (S)	216*	115	52	4					
Heartwood (D)	150	553	21	217					
Included Sapwood (C)	78	208	68	189					
Included Sapwood (B)	81	195	81	195					
Heartwood (A)	129	452	47	254					
AVER AGE :	131	305	54	172					

TABLE 3. Effect of pressure and temperature on creosote retention in mountain-type Douglas fir.

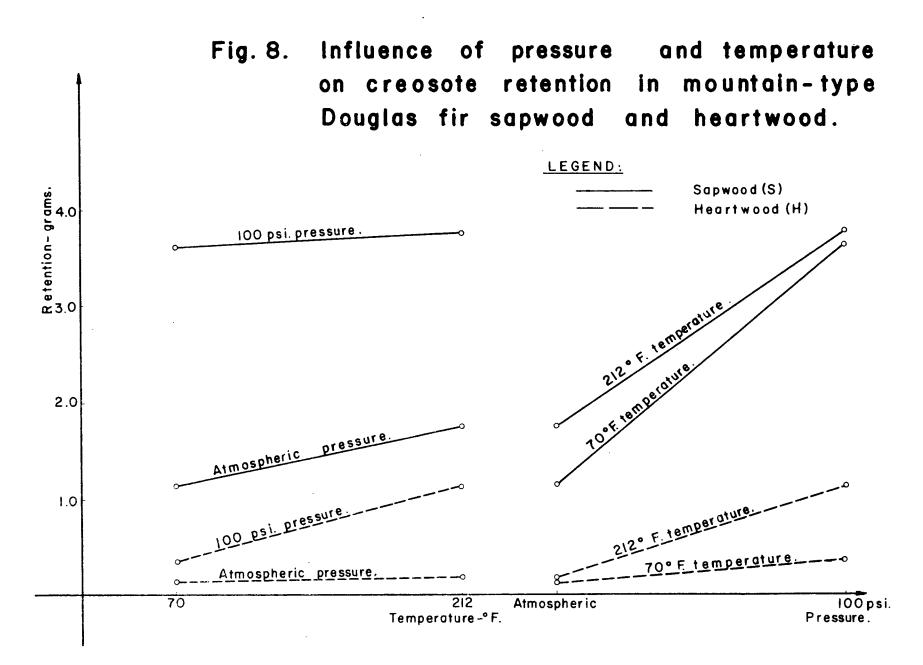
\*Increase in retention = 100  $\left[\frac{R70^{\circ}F.,100 \text{ psi}}{R70^{\circ}F.,15 \text{ psi}} - 1\right] = 100 \left[\frac{3.63}{1.15} - 1\right] = 216\%$ 

R  $70^{\circ}$ F., 100 psi = average retention value of 16 test specimens at  $70^{\circ}$ F. treating temperature and 100 psi. Average R values were taken from Table 2.





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more fluid at higher temperatures. Consequently, it penetrates wood more readily at higher than at lower temperatures.

A definite relationship between viscosity and penetrance, which could be expressed by empirical equations for specific conditions, was found by Bateman (4).

A great importance is attributed by Howald (28) to the presence of peptized colloids in oil preservatives. The colloids are believed to exert their influence by changing the capillary relationship between oil and wood.

Raphael and Graham (42) confirmed Bateman's conclusion, stating that oils with low viscosity and specific gravity are the best penetrants. High-viscosity, low-specific gravity oils penetrate better than high-viscosity and high-specific gravity oils.

It was concluded by Liese (32) that, although the depth of penetration of oily wood preservatives is influenced by their viscosity, surface tension, and specific gravity, even greater importance must be ascribed to chemical factors, such as the chemical composition of preservative.

#### 3. Correlation between specific gravity and permeability

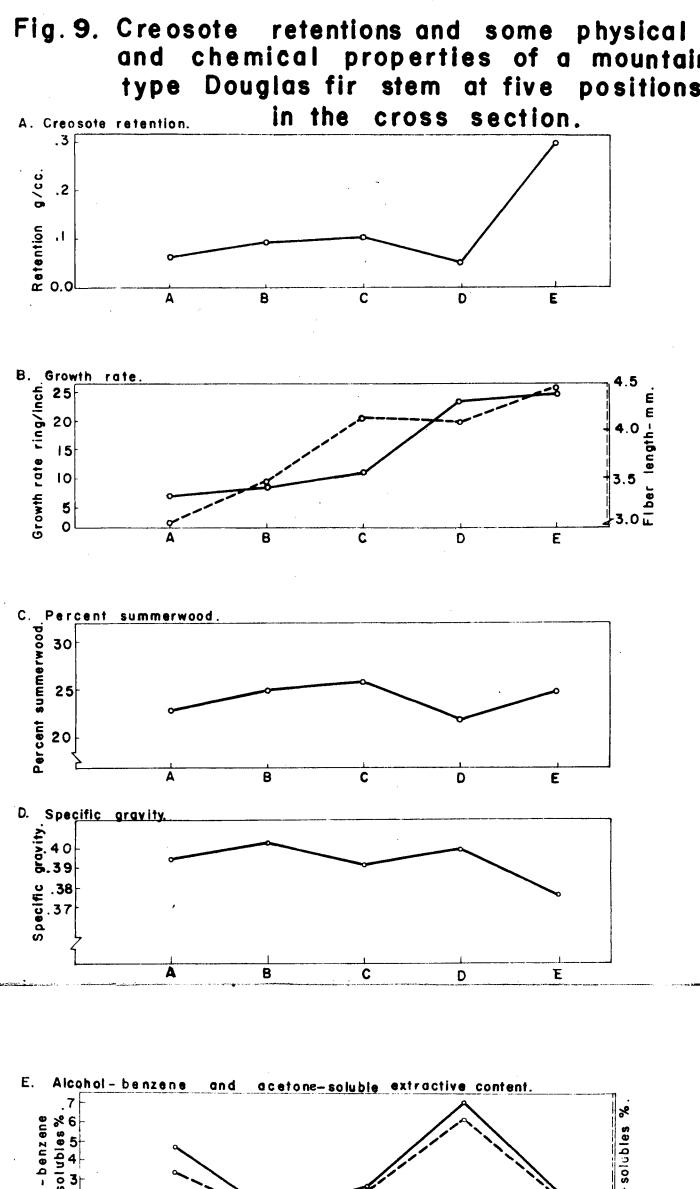
The average specific gravity of the five zones remained fairly constant (Table 4 and Figure 9D), while the permeability of the corresponding zones varied significantly. Consequently, there was no definite correlation between specific gravity and ease of penetration.

Miller (37) recently investigated the influence of specific gravity on the penetrability of Douglas fir, but found no correlation between the two variables. In their absorption studies of ponderosa pine with oil-base preservatives, Brown, Moore, and Zabel (8) concluded that an increase in the

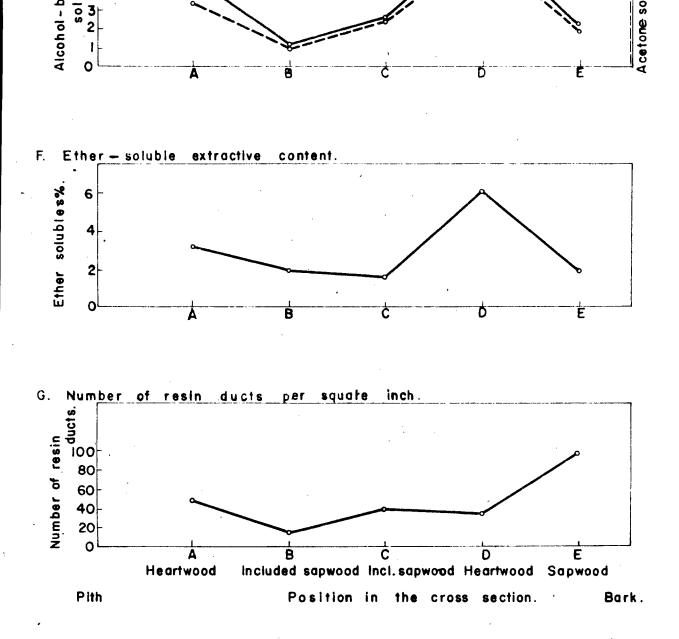
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Position in the cross section	Heartwood A	Included Sapwood B	Included Sapwood C	H <b>eartwoo</b> d D	Sapwood S
Properties	l	2	3	4	5
Specific gravity	•395	.403	•392	•400	.376
Percent summerwood	23	25	26	22	25
Growth rate (rings per inch)	18.1	22.0	285	60.2	63.8
Fibre length (mm.)	3.03	3.48	4.15	4.11	4.14
Number of longitudinal resin ducts per sq. in.	316	90	325	226	632
Alcohol-benzene-soluble extractives (%)	4.66	1.21	2,68	7.01	2.27
Acetone-soluble extractive content (%)	3.34	1.13	2.40	6.10	1.83
Ether-soluble extractive content (%)	3.17	1.87	1.57	6.14	1.90
Creosote retention g/cm <sup>3</sup>	0.065	0.094	0.105	0.051	•304

TABLE 4. Some physical and chemical properties of a mountain-type Douglas fir stem.



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specific gravity of wood causes a slight decrease in absorption. This effect decreases with an increase in moisture content.

#### 4. <u>Influence of percent summerwood on permeability</u>

There appears to be a linear correlation between percent summerwood and permeability (Table 4 and Figure 9C). The apparent relationship, however, can not be accepted as being significant, for the following reasons:

(i) The average percent summerwood values of the five zones vary only from 22 to 26 percent. This 4 percent variation between the five zones is smaller than the 7.4 percent standard deviation of the entire data.

(ii) The accuracy of the method used to determine percent summerwood was approximately ± 3 percent.

(iii) An analysis of variance revealed no significant differences between the percent summerwood values of the different zones.

Specimens representing a much wider range of percent summerwood must be selected to investigate the influence of this factor on treatability. The effect of percent summerwood on penetration of preservatives into Douglas fir was investigated by Miller (37), who found no correlation between the two variables.

#### 5. Effect of growth rate on creosote retention

No significant correlation between growth rate and the ease of penetration is revealed in Figure 9B and Table 4. The growth rate decreases from pith to bark, but the creosote retention values do not follow the same pattern. Other factors seem to be of more importance in the determination of wood permeability.

Neither Bryan (9) nor Miller (37) found any relationship between growth rate and treatability of wood when impregnating Douglas fir with creosote. In their investigation of longitudinal penetration of creosote in Douglas fir, Raphael and Graham (42) concluded that wood with high ring count showed a more uniform distribution of preservatives than wood with a low ring count.

#### 6. Relationship between fibre length and creosote retention

In longitudinal penetration, the preservative must pass through fewer cell walls in woods with long fibres than in woods with short fibres per unit length. Theoretically, a deeper penetration and a higher retention would be expected in the first type of wood than in the second.

The results of this study, given in Table 4 and Figure 9, indicate that fibre length is not an important factor in the determination of the permeability of wood. Any minor influence which fibre length may have is dominated by the effect of more important factors.

## 7. Influence of longitudinal resin ducts on penetrability (Table 4 and Fig. 9G)

Within this mountain-type Douglas fir stem no correlation was found between the number of longitudinal resin ducts and longitudinal penetration. It was observed, however, that resin ducts in heartwood contained more resin than those in included sapwood. Consequently, the resin ducts in included sapwood may have aided penetration, whereas those in the heartwood are less likely to have done so.

Hunt and Garratt (29) stated that in mountain-type Douglas fir heartwood resin ducts may be penetrated to a greater or lesser extent, but so little preservative enters the adjacent wood cells that the resultant penetration is little improved. On the other hand, maximum penetration along the resin ducts of Douglas fir was observed by Scarth (43), who attributed great significance to this factor.

#### 8. Correlation between extractive content and treatability

There appears to be inverse relationship between alcohol-benzenesoluble extractives and creosote retention in Douglas fir heartwood (See Figure 10). An increase in alcohol-benzene solubles results in a proportional decrease in wood permeability. Thus, heartwood having a high extractive content is more difficult to impregnate with creosote than wood relatively low in extractive content. This correlation between the two variables did not prove to be statistically significant.

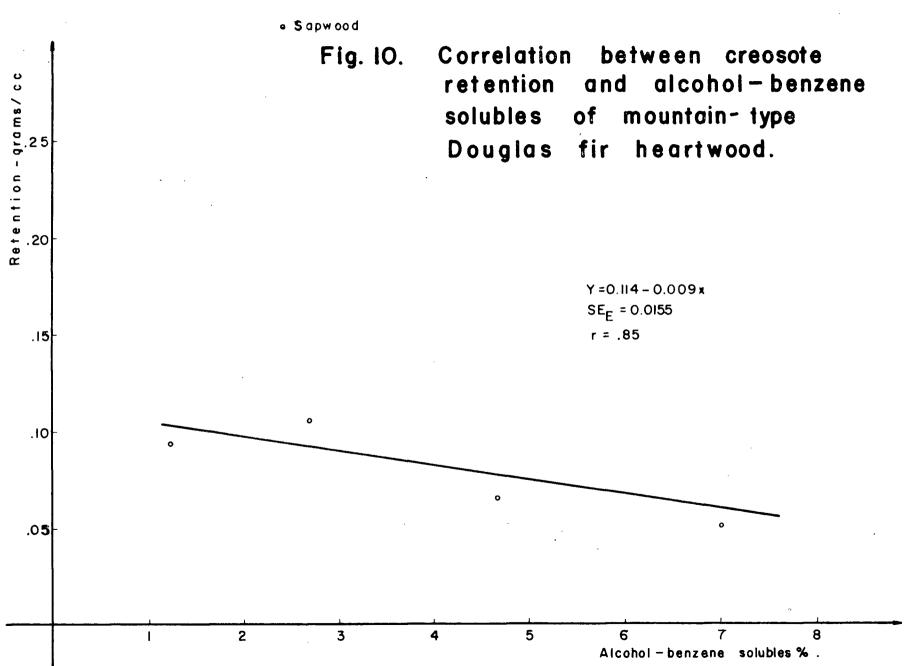
A similar relationship was obtained between acetone-soluble extractives and creosote retention. The slope of the straight line, shown in Figure 12, does not significantly differ from zero.

The results were somewhat different with ether-soluble extractives. A hyperbolic, rather than a straight-line, relationship can be observed in Figure 11. The curve indicates that the influence of extractive content on wood permeability decreases with increasing extractive content. Thus, it may be assumed that only a small quantity of extractives is required to cause major changes in the treatability of wood. This small amount of extractives in the cell wall structure appears to be adequate to close most of the channels otherwise available for liquid movement. The channels affected are probably the numerous cell wall capillaries and the permanent pores in the pit membrane.

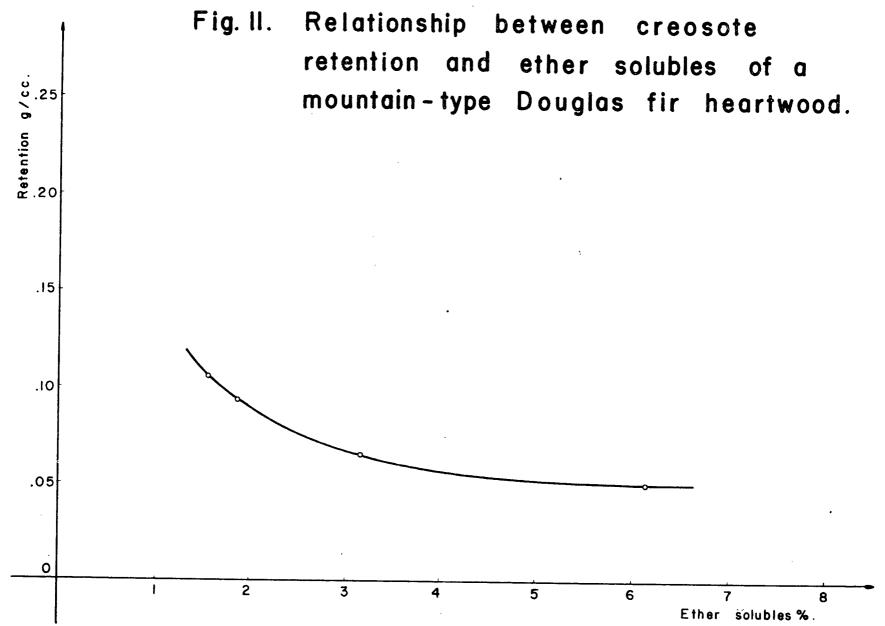
Included sapwood contained a similar amount of extractives to true sapwood, and approximately one-third of that of the heartwood (Table 4). Creosote retention of included sapwood was only one-third of that of the sapwood and not quite twice as much as that of the heartwood. The amount of extractives, as well as their location within the cell-wall structure, must be considered when studying their effect on wood permeability.

Based on the fact that sapwood and included sapwood contained similar amounts of extractives but possessed different permeability character-

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-39a-

istics, it may be assumed that the locations of the extractives are not the same in the two types of wood. This assumption is confirmed by the fact that the major function of sapwood in the living tree is conduction, while that of the included sapwood is mechanical support. It is probable that the extractives in both the included sapwood and heartwood are located in such a way as to partially or fully seal most of the capillaries present in the cell walls.

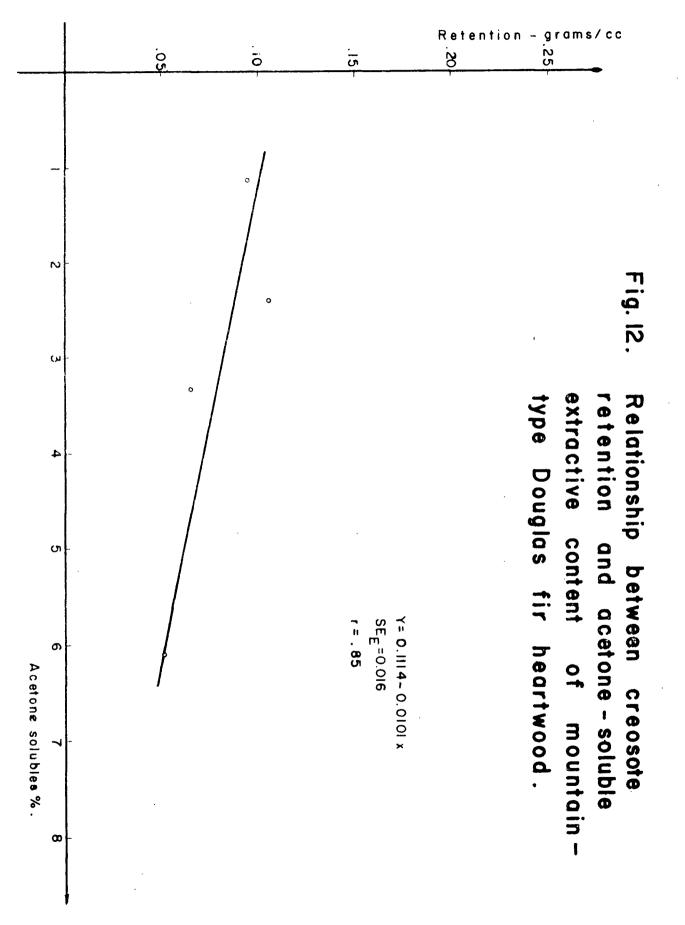
The difference between included sapwood and normal heartwood, as far as permeability is concerned, may be due to the extent to which the cell wall capillaries are blocked with extractives. In included sapwood the degree of deposition of extractives in the cell wall capillaries is expected to be lower than in the heartwood. This would be expected since the former tissue has apparently only partially undergone the process of conversion from sapwood to heartwood. In addition, included sapwood contains, smaller percentage of extractives available for deposition than heartwood. This theory may be supported by the fact that creosote retention in heartwood was approximately half of that in included sapwood.

Stone (49) found spaces between the tori and the edges of the pit aperture, when analyzing photomicrographs of aspirated pits at high magnifications. From this finding he concluded that the surface of the torus was too rough to completely seal the aperture to the flow of most liquids. There is a possibility that these openings did not exist in the original wood, but were caused during the preparation of the slides by the removal of some of the extractives which might normally plug the openings.

### 9. Improvement in treatability due to extraction

In the previous part of this study an inverse relationship was found to exist between extractive content and permeability. If this correlation is

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-17-

true, an improvement in retention may be expected upon the removal of some of the extractives from the cell wall structure. The results given in Table 5 support this assumption. Improved permeability was obtained following extraction. The extent of the improvement in permeability varied in the different types of wood. The average ratios between the creosote retentions of extracted and unextracted test specimens were 1.2 for sapwood (S), 6.5 for included sapwood (C), and 8.1 for heartwood (D) (Table 5).

From the above figures, and from Table 4, it may be observed that heartwood, having the highest extractive content, on extraction improved in permeability to the greatest extent. Sapwood, containing low percentages of extractives, increased in treatability very slightly. Included sapwood, with similar extractive contents to that of sapwood, improved greatly in permeability.

Since the improvement in the permeability of sapwood and included sapwood was very much different, in spite of their similar extractive contents, either the location or types of extractives or the degree of aspiration of the bordered pits must differ in the two kinds of wood. This observation supports the previous assumption which attributes a great significance to the location of extractives in the cell wall structure.

Heartwood, having the highest extractive content, as well as the greatest improvement in treatability on extraction, confirms the fact that the amount of extractive present in wood is another major factor in determining its permeability.

The data reveal no major differences between the hot and Soxhlet extraction methods. Therefore the temperature of the solvent appeared to have no measureable influence on the degree of improvement in permeability. This is probably due to the long extraction period employed. Of solvents used

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TABLE 5. Average creosote retentions of mountain-type Douglas fir heartwood, included sapwood, and sapwood, following a 240-hour extraction in different solvents.

Duration of Extraction	Type of Solvents	Included C	<u>Kind of w</u> Sapwood	ood: Heartw D	rood	Average	
(Hours)		Soxhlet	Extraction Hot		<b>TT </b>		
		Soxiffet		I Soxhlet	Hot		
60 60 60 60	Alcohol-benzene ) Ether ) Acetone ) Water )	0.67	0.66	0.46	0.56	0.58	
220 10 10	Alcohol-benzene ) Alcohol ) Water )	0.69	0.53	0.56	0•45	0.55	
230 10	Sodium hydroxide ) Water )	0.67	0.68	0.57	0.66	0.64	
240	Water	0.65	0.67	0.67	0.62	0.65	
None	Control (No extraction)	0.10	0.10	0.07	0,07	0.08	
Avera	g e:	0.55	0.53	0.46	0.47		
Avera	Average:		54	0.	0.47		
	Ratio of retention of extracted specimens to unextracted:		6.3	<b>8.</b> Ó	8.2		
Average ra	tio:	6.	5	8.	1		

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Water extraction, 240 hrs.; Average retention, extracted = 0.48 g.; unextracted, 0.39 g.; Ratio = 1.2

in the extraction studies, water gave most improvement in permeability (Table 5). The reason for this may be that the water relieved, partially or fully, the aspiration in the bordered pits, thus providing a greater number of channels for liquid movement. This is, of course, only an hypothesis which could be proved or disproved by a detailed study of the degree of aspiration in extracted and unextracted matched specimens.

This aspect of the study may have some practical significance in the wood preserving and pulp industries. A method of pre-treatment could be developed to improve the permeability of wood.

According to the results obtained in this investigation, water would appear to be the most effective, and the most economical solvent for extraction. A problem in the wood preserving industry would originate from the large sizes of the material to be extracted, and the long periods of time required for solvent to reach the middle portions of what is a relatively impermeable material. Furthermore, the use of higher temperatures may be necessary to increase the effectiveness of the solvent.

The pulp industry, however, would not face this problem as the size of the chips is sufficiently small to enable thorough extraction in a relatively short time.

#### 10. Effect of solvent on wood

Water, at low temperatures, does not react chemically with wood. Its action is confined to the removal of some of the water-soluble extractive content. At elevated temperatures, water has a marked chemical effect on wood. It influences strength properties by breaking down some of the pentosan and cellulose components. The amount of these materials removed from the wood depends on the severity of the conditions (temperature, pressure and duration of treatment).

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Neutral solvents, such as alcohol, benzene and acetone, do not affect the strength properties of wood. These solvents, having larger molecular sizes than water, cannot enter the small capillaries in the amorphous region, and therefore do not rupture the secondary valence forces between the cellulose molecules.

Sodium hydroxide reacts with lignin and wood carbohydrates, breaking down the basic components and causing delignification. The degree of delignification depends on the concentration of the solvent and on the treating conditions. In the extraction of Douglas fir wood with 0.1 percent sodium hydroxide, it was observed from the reddish color of the solution, and the appearance of the extracted specimens, that a large portion of the lignin had been removed. The amount of lignin removed from the test specimens was not determined.

#### 11. Bordered pit aspiration

Most of the tori in the bordered pits of sapwood, heartwood, and included sapwood appeared to be in an aspirated condition when observed in 20-micron-thick sections under a binocular microscope. The magnification used was approximately 600 X. These observations may not be reliable for two reasons. One of these is that much thinner sections, 2 microns in thickness, are required for the accurate investigation of the degree of aspiration. Another reason could be the possible effect of sectioning and slide preparation on the degree of aspiration observed. The latter cause of error probably exists in many of the studies of this type. Observation of pit aspirations have usually been made on wood sections cut on a microtome. The disruption of the wood structure during sectioning and slide preparation should not be overlooked as a possible source of error due to mechanical forces and chemical solvents.

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If the uniform aspiration of the bordered pits observed in heartwood, sapwood, and included sapwood is accepted as valid, it leads to an important conclusion. This is that the aspiration of bordered pits has no effect on the permeability of mountain-type Douglas fir.

#### 12. Direction of penetration (Figure 5)

Much better penetration was obtained from the ends than from the sides of the test specimens. In fact, in many specimens, complete impregnation of the material was obtained when only the end grain was exposed to the creosote. The ratios between longitudinal and side penetration of the included sapwood (B,C) were higher than those for heartwood (A,D). This means that the permeability of mountain-type Douglas fir included sapwood, along the grain, was greater than that of heartwood. These ratios (Figure 5), however, were considerably smaller than those given by MacLean (35). He reported that in the very refractory, Rocky Mountain-type Douglas fir, the longitudinal penetration ranged from about 25 to 35 times as great as the side penetration, when the wood was impregnated with creosote. This difference may perhaps be explained by the fact that the specimens used in the present study were too short to obtain a true measure of longitudinal penetration, and thus of the ratio of longitudinal to side penetration.

In the four zones (A,C,D,S) of this mountain-type Douglas fir stem the radial penetration was superior to the tangential penetration (Figure 5). In order to find an explanation for this, an intensive anatomical study would be required. It may be assumed, however, that the rays and/or the radial resin ducts facilitated the radial penetration (from the flat-sawn face) of creosote. No count was made of the number of fusiform rays, nor a study of their condition.

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#### SUMMARY

- Permeability of included sapwood is superior to that of normal heartwood, but inferior to true sapwood.
- 2. The effect of pressure on creosote retention at 212°F. is greatest for heartwood, less for included sapwood, and least for sapwood. The influence of pressure on creosote absorption in heartwood is greater at higher temperatures than at lower ones. Pressure has a greater effect on the retention of sapwood at lower temperatures than at higher ones.
- 3. The influence of temperature on creosote retention in Douglas fir heartwood is greater at 100 psi pressure than at atmospheric pressure. The reverse is true for sapwood. Temperature has a greater influence on retention at atmospheric pressure than at 100 psi pressure.
- 4. There was no definite correlation between specific gravity of wood and ease of penetration.
- 5. Percent summerwood did not vary significantly within this mountain-type Douglas fir stem.
- Tracheid length had no measurable influence on creosote retention in mountain-type Douglas fir wood.
- 7. Growth rate was not found to be an important factor in the determination of wood permeability.
- 8. No relationship was found between the number of longitudinal resin ducts and longitudinal penetration of creosote, probably due to lack of length of specimens, as noted.

9. (i) A statistically non-significant correlation was obtained between alcohol-benzene-soluble extractives and creosote retention.

(ii) The slope of the straight line obtained for acetone-soluble extractives and creosote retention did not significantly differ from zero.

(iii) A hyperbolic, rather than a straight-line, relationship was found between ether-soluble extractives and creosote retention. The higher the extractive content, the greater the retention.

(iv) Within this mountain-type Douglas fir stem the amount as well as the location of the extractives is considered to be important in the determination of wood permeability.

- 10. Pre-treatment of samples with different solvents in order to remove some of the extractives improved the permeability of heartwood, included sapwood and sapwood. The increases were respectively 8.1, 6.5 (all solvents), and 1.2 (only water) times, compared with the controls.
- 11. No definite conclusion can be drawn from the limited number of observations on the degree of bordered pit aspiration.
- 12. It would be desirable to know the effect of extraction treatment on the cell walls and on the mechanical properties of the wood. A chemical analysis of the solvents after the extraction treatment would show which wood components had been extracted.
- 13. The development of an extraction pre-treating technique is suggested for the wood preserving and pulp industries, to improve wood permeability. Type of solvent, temperature, duration of treatment, and economics should be determined for specific conditions.

14. The specimens used in the present study were too short to provide a true measure of the ratio of longitudinal to side penetration. In the four zones of the log section, higher retentions were obtained in the specimens when exposing the tangential faces than the radial faces.

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APPENDIX

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Time (Hours)	Absorption (Grams)
0	0
l minute	0.07
0.25	0.30
0.50	0.38
1.25	0.50
2	0.56
3	0.62
4	0.66
5	0.69
6	0.71
7	0.73
8	0.75

# TABLE 6. Creosote absorption values of mountain-type Douglas fir sapwood.

Position			A	tmospl	neric	pressu	ire				Pre	essure	e at l	00 ps:	L		
in the	Direction		70 <sup>0</sup>				2120	°F.			70	°F.			212°1	F.	
Cross Section						μ		Re	eplica	ates			ſ				
Fig.1		1	2	3	4	1	2	3	4	1	2	3	4	1	3	3	4
							Reter	tion	value	es (gi	rams)						
True Sapwood S	Radial Tangential Longitudinal All (Control)	0.43 1.38	0.30	0.38	0.68 0.14 1.90 2.36	0.52 2.01	0.66 2.01	1.12 2.06	1.18 0.88 2.08 2.13	4.26 4.22	3.66 4.13	4.46 4.09	4.21 4.12	4.29	4.30 4.06	3.69 3.42	3.34 3.31 3.70 3.90
True Heartwood D	Radial Tangential Longitudinal All (Control)	0.02 0.21	0.03 0.23	0.04 0.21	0.07 0.03 0.20 0.31	0.04	0.06	0.09 0.25	0,10	0.03 0.54	0.02 0.48	0.07 0.56	0.61	0.87	0.74	0.98	1.25 1.21 1.40 1.32
Included Sapwood C	Radial Tangential Longitudinal All (Control)	0.07 0.56	0.05 0.64	0.01 0.65	0.11 0.08 0.52 0.71	0.02	0.07 0.89	0 <b>.09</b> 0 <b>.</b> 86	0.98	0.25	0.14 1.30	0.10 1.30	0.18 0.11 1.21 0.88	0.70 2.75	0.98	1.95 2.29	1.40 1.56 2.63 2.08
Included Sapwood B	Radial Tangential Longitudinal All (Control)	0.07	0.05	0.08	0.07 0.08 0.45 0.57	0.25	0 <b>.</b> 17 0 <b>.</b> 90	0.11 0.97	0.13	0.10	0.18 0.93	0.20 0.86	0.24 0.15 0.92 1.07	1.10 2.05	1.20 2.07	1.82	1.36 1.42 1.59 2.22
True Heartwood A	Radial Tangential Longitudinal All (Control)	0.01 0.27	0.01 0.30	0.06	0.05 0.06 0.27 0.26	0.06	0.02 0.44	0.08 0.35		0.05	0.05 0.59	0 <b>.1</b> 3 0 <b>.</b> 62		0.72	1.07	1.80	1.58 1.49 1.75 1.61

TABLE 7. Creosote retentions in mountain-type Douglas fir sapwood, included sapwood, and heartwood under different conditions of treatment.

-55-

S	Source	Sum of Squares	D.f.	Mean Squares	F
1.	Pressure	79.65	l	79.65	1632**
2.	Temperature	23.87	l	23.87	489**
3.	Position	193.48	4	48.37	991**
4.	Direction	35.70	3	11.90	244**
5.	Pressure x temp.	7.48	1	7.48	153.3**
6.	Pressure x position	31.46	4	7.86	161.1**
7.	Pressure x direction	1.68	3	0.56	11.5**
8.	Temp. x position	1.81	4	0.45	9.2**
9.	Temp. x direction	0.37	3	0.12	2.5 <sup>n.s.</sup>
10.	Position x direction	7.31	12	0.61	12.5**
11.	Pressure x temp. x position	5.82	4	1.45	30.0**
12.	Pressure x temp. x direction	0.55	3	0.18	3.7*
13.	Pressure x position x direction	8.30	12	0.69	14.1**
14.	Temp. x position x direction	3.24	12	0.27	5•5**
	Error	12.35	253	0.048814	•
	TOTAL	413.07			
	C	351.77			

TABLE 8. Analyses of variance of creosote retentions in mountain-type Douglas fir as affected by pressures, temperatures, position, and direction of penetration.

n.s. = non-significant

\*significant

\*\*highly significant

Replicates		Position i	in Cross Sec	tion	
webileapes	Heartwood (inner) A	Included Sapwood B	Included Sapwood C	Heartwood (outer) D	Sapwood S
l	0.399	0.399	0.383	0.394	0,352
2	0.397	0.406	0,398	0.392	0.397
3	0.407	0.402	0.394	0.408	0.389
4	0.376	0.403	0•394	0.406	0.367
AVERAGE ;	0.395	0.403	0•392	0.400	0.376

TABLE 9. Specific gravity values\* of a mountain-type Douglas fir stem.

\*Based on green volume, oven-dry weight of unextracted specimens.

$\begin{array}{c c c c c c c c c c c c c c c c c c c $	Position Ring number	Heartwood A	Included Sapwood B	Included Sapwood C	Heartwood D	Sapwood S
Sum of squares         7172         9827         15,529         24,108         34,180           Averages         22.9         25.3         26.3         21.5         25.1           Standard         deviations         5.3         4.2         6.9         7.0         8.4           Number         13         15         21         47         49           Source         Degrees         Sum of Square         Mean Square         Variance ratio	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	30 20 27 24 29 26 22 17 15 21 31	29 25 30 21 23 33 22 23 18 25 21 23 27	27 32 22 33 38 27 25 20 15 20 15 20 14 30 5 21 30 31	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	25       33         33       25         17       25         25       50         33       9         40       10         33       22         25       29         25       17         20       26         20       20         33       25         33       25         33       25         33       25         33       17         33       22         17       12         33       33         8       33         21       29
deviations5.34.26.97.08.4Number1315214749SourceDegreesSum of SquareMean SquareVariance ratio	Sum of squares Averages	7172	9827	15,529	24,108	34,180
Number         13         15         21         47         49           Source         Degrees         Sum of Square         Mean Square         Variance ratio		5.3	4.2	6.9	7.0	8.4
	Number	13	15	21		49
Total 144 7,775		Degrees S	um of Square	Mean Squar	e Variance 1	ratio
		144	7,775	100.0	<u> </u>	<b>T</b> 1 <b>A A</b>
Between         4         493         123.3         2.37         Not significan           Within         140         7,282         52.0					2.37	NOT SIGNIFICAN

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TABLE 10. Percent summerwood values of a mountain-type Douglas fir stem and analyses of variance.

Position in the cross section Number	Heartwood A	Included Sapwood B	Included Sepwood C	Heartwood D	Sapwood S
		(Units)			· ·
$ \begin{array}{c} 1\\ 2\\ 3\\ 4\\ 5\\ 6\\ 7\\ 8\\ 9\\ 10\\ 11\\ 12\\ 13\\ 14\\ 15\\ 16\\ 17\\ 18\\ 19\\ 20\\ \end{array} $	51 69 85 80 70 75 78 95 82 67 92 85 66 75 63 100 70 87 78 76	106 100 83 97 74 75 87 78 86 93 95 99 97 107 82 99 97 107 82 99 58 85 87 90	$     \begin{array}{r}       120 \\       83 \\       112 \\       109 \\       77 \\       98 \\       85 \\       107 \\       111 \\       108 \\       107 \\       122 \\       125 \\       84 \\       130 \\       101 \\       105 \\       112 \\       119 \\       98 \\     \end{array} $	77 115 97 86 92 130 115 123 70 100 132 110 90 120 100 132 85 95 129	85 125 95 87 118 105 110 111 132 112 76 122 112 125 132 112 123 140 122 120
TOTAL:	1544	1771	2113	2094	2264
Average:	77.2	88.6	105.7	104.7	113.2
mm :.	3.03	3.48	4.15	4.11	4.44

TABLE 11. Fibre length values for various sections in a mountain-type Douglas fir stem.

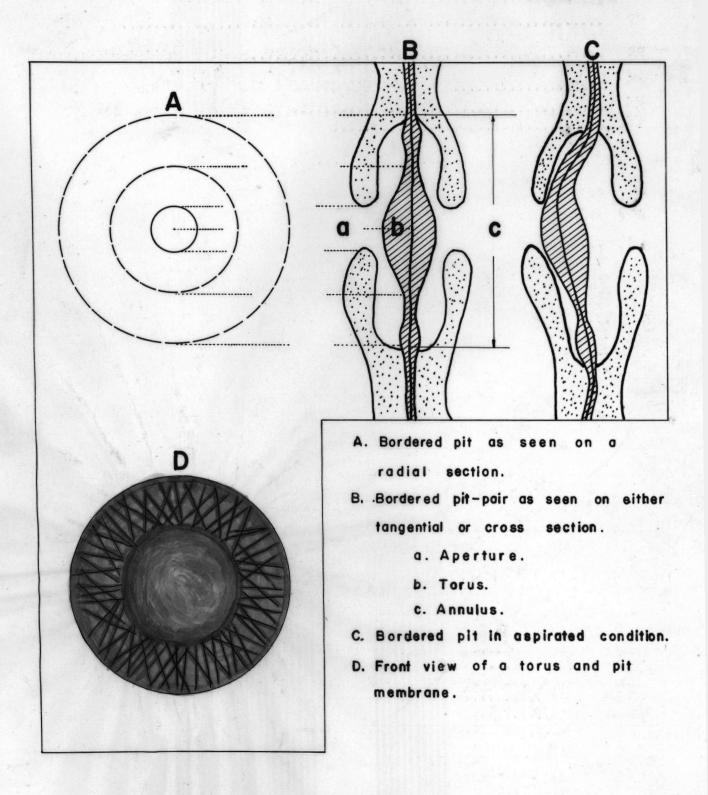
Position in the cross section		A		В		C		D		S
Extractive content	1	2	1	2	1	2	1	2	1	2
Alcohol-benzene solubles - %	4.61	4.71	1.06	1.36	2.60	2.75	6.96	7.06	2.25	2.29
Acetone solubles - %	3.23	3.45	1.16	1.10	2.32	2.48	6.04	6.16	1.91	1.76
Ether solubles - %	3.14	3.20	1.96	1.78	1.61	1.53	6.08	6.20	1.95	1.85

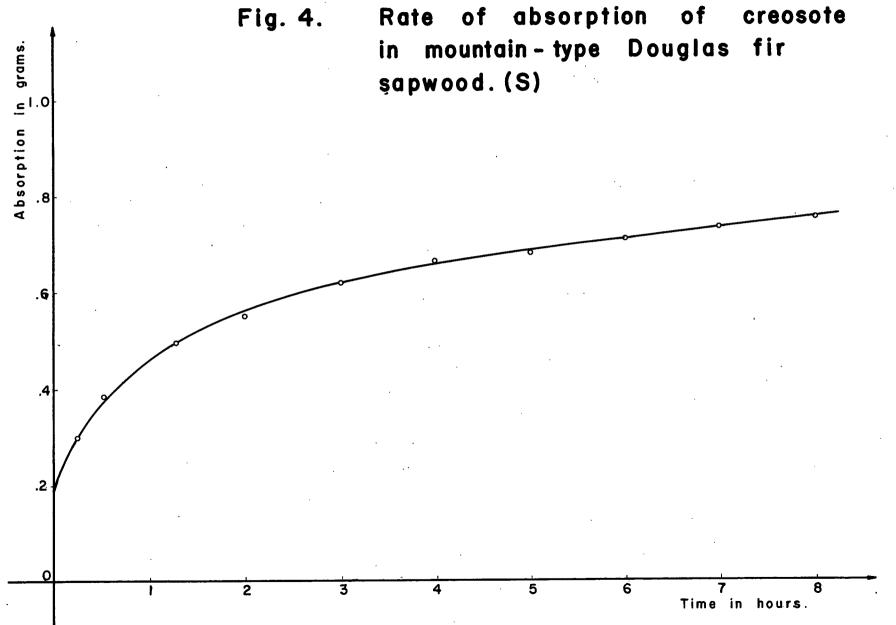
TABLE 12. Alcohol-benzene, acetone, and ether solubility of mountaintype Douglas fir wood.

	Duration of Extraction	Type_of Solvent	Replicates	Inclu	Kind of Muded Sapwood	Heart	
					<u> </u>	<u> </u>	)
	(Hours)			_	••	xtractions:	
				Soxhlet	Hot	Soxhlet	Hot
		<u>.</u>			Retentio	ons (grams)	
	60	Alcohol-benzene				1	
	60	Ether	1	.69	•59	•45	•49
	60	Acetone	2	.64	.73	.46	.62
	60	Water					
	220	Alcohol-benzene					
	10	Alcohol	1	.64	•55	.64	.42
	10	Water	2	.73	.51	.47	•48
	230	0.1% Sodium	1	.64	.70	• <b>5</b> 5	.61
	10	hydroxide	1 2	.69	.65	.58	.70
		Water			•		
			1	•69	.67	58	.63
	240	Water	2	.61	.66	.76	.60
			1	.12	•09	.07	.0'
• .			2	.10		.08	.00
	None	Control (no extraction)					
· .		extraction	3	•09	•08	.06	.09
			4	.10	.11	.06	.06
			4 5	•09	.12	.09	.0
						1	
:		1	AVERAGE:	0.10	0	0.0	27
		240-hour hot -	Control	Extracted		-	
	water ext Retention	raction	•395 •354	.448 .498			

TABLE 13.	Creosote	retentions	of mountai	in-type Dou	ıglas fir	heartwood,	included	sapwood,	and	
	sapwood,	following a	a 240-hour	extraction	n <mark>in dif</mark> f	erent solve	nts.			

Fig.1. Diagram of a bordered pit (15,25).

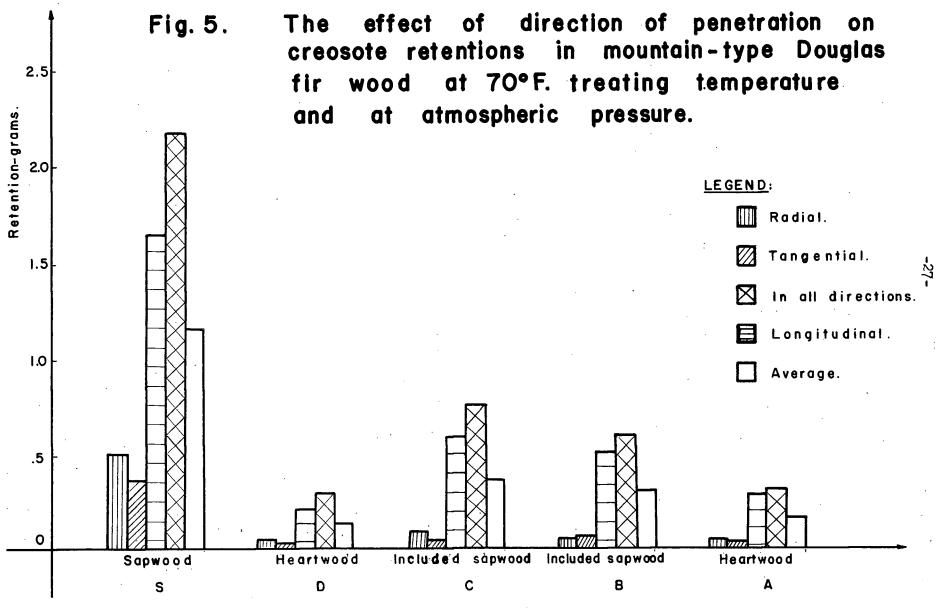




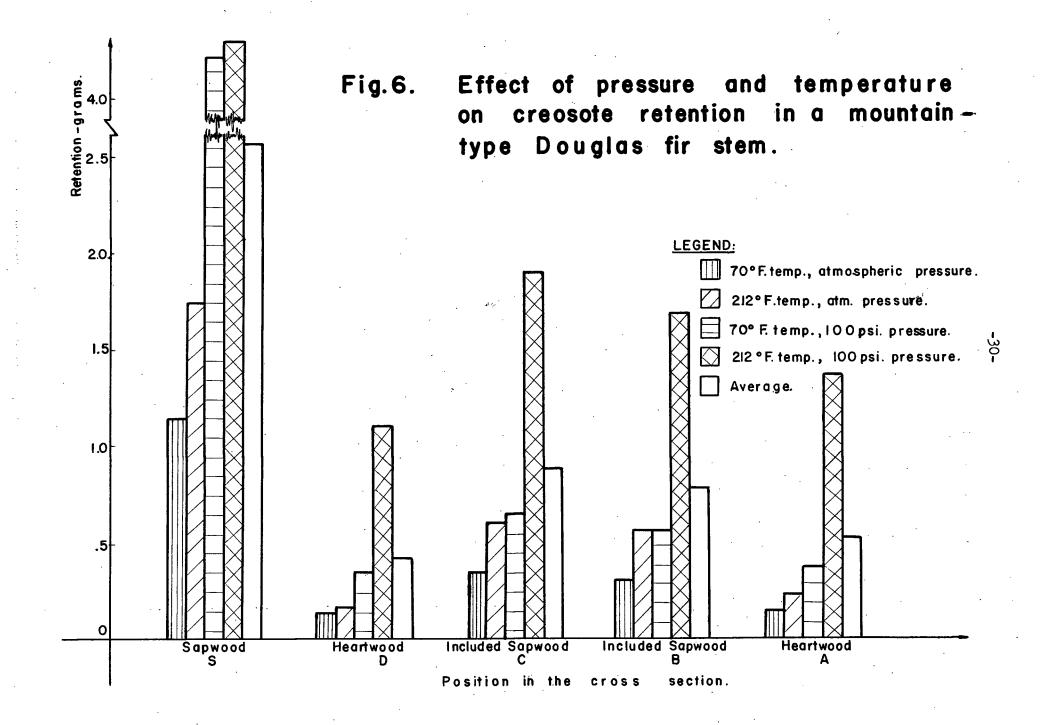
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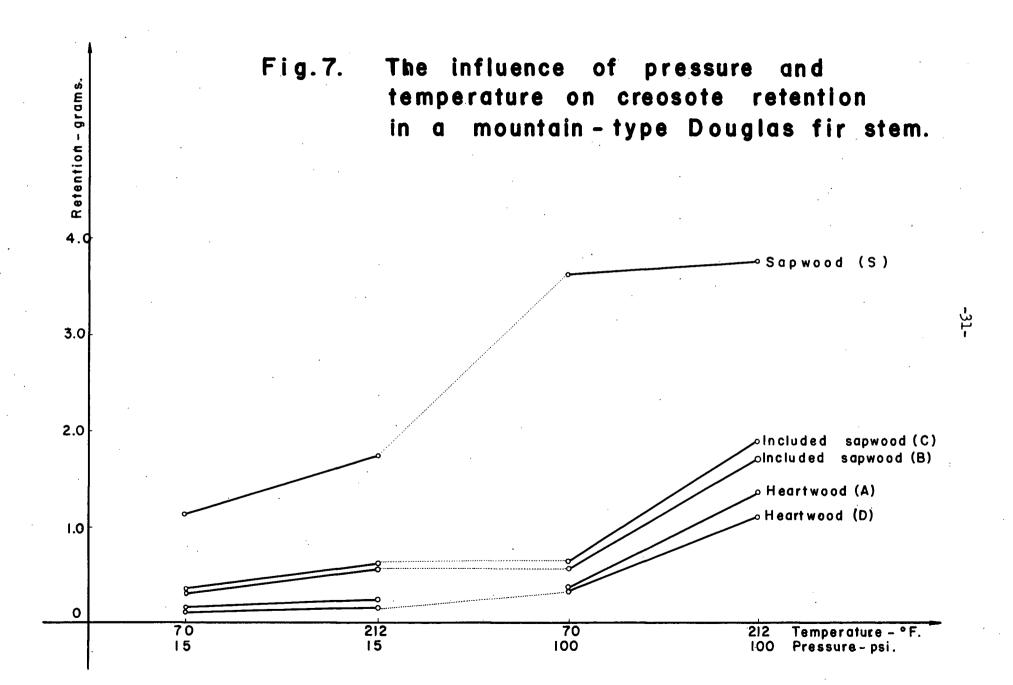
, <sup>1</sup>

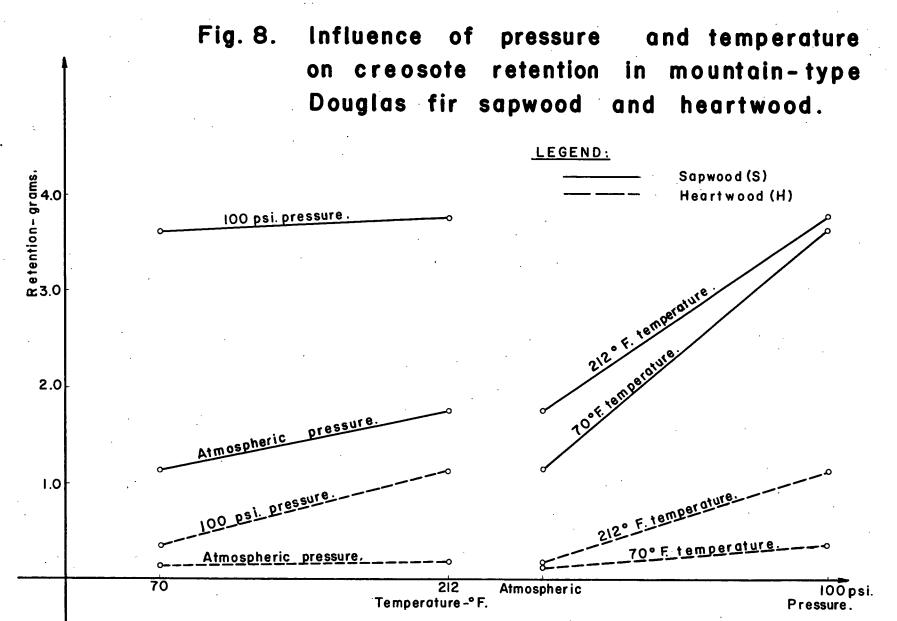
-17-



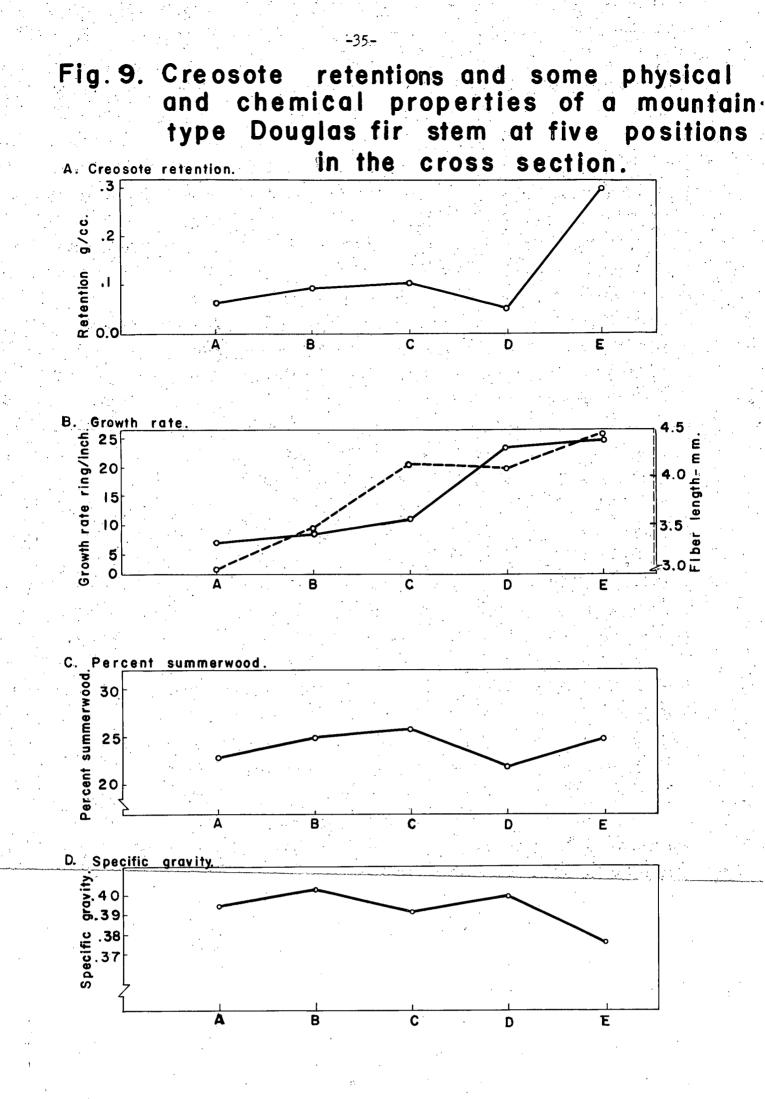
Position in the cross section.



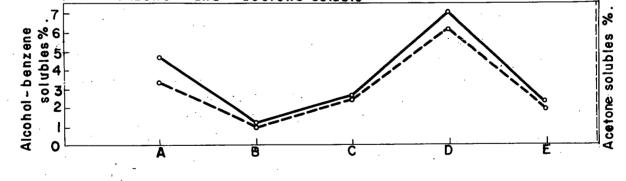


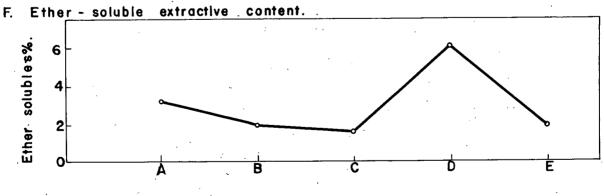


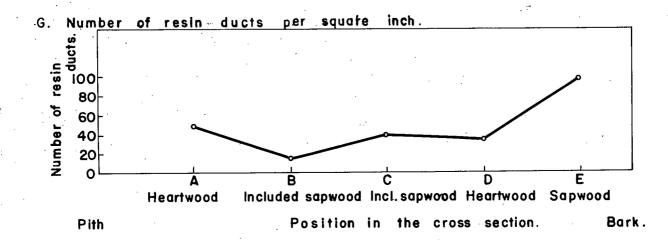
-32-



Ε. Alcohol – benzene acetone-soluble extractive content. and





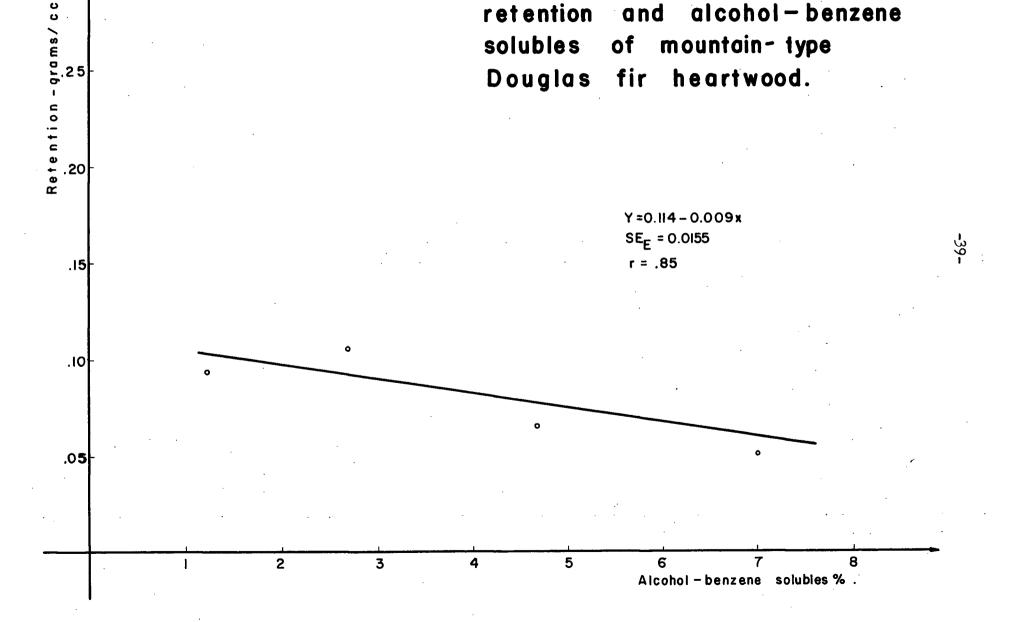


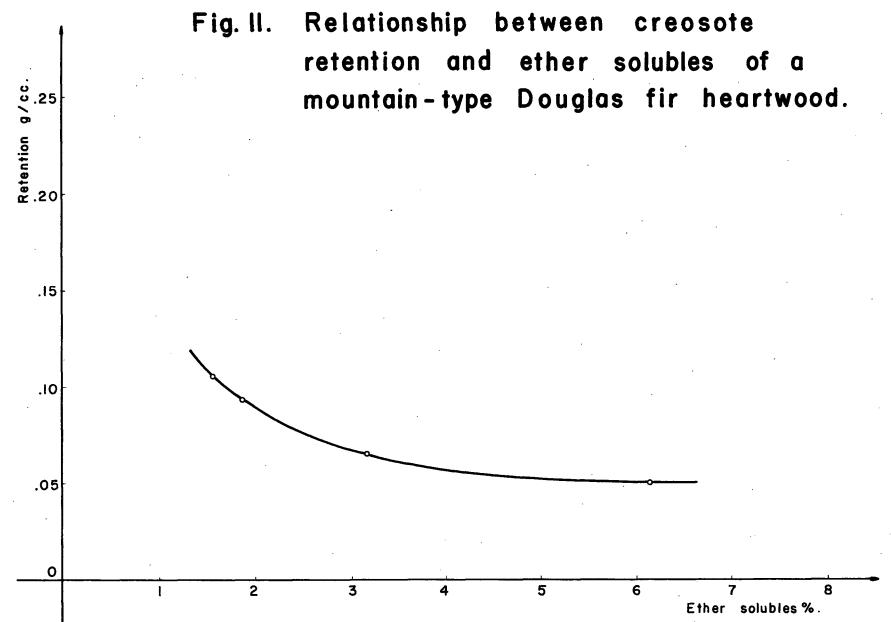


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Correlation between creosote retention and alcohol-benzene of mountain-type solubles fir heartwood. Douglas





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