# RADIAL PERMEABILITY OF TIMBER

by

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## ABSTRACT

A procedure is described for monitoring the uptake of liquids under pressure, into small blocks of dry wood. Tests with eight softwood species showed that rates of uptake in the radial direction correlated well with the known treatability of these species in relation to commercial pressure impregnation with wood preservatives.

Several different patterns of radial penetration were observed and it was found that the most important morphological feature influencing these was the nature and condition of the ray parenchyma cells, the crossfield pits in particular.

Examination of the structure of water-stored spruce showed that the bordered pit membranes and tori had been destroyed, probably by bacteria. It is suggested however, that the main factor causing increased permeability in this material was the partial destruction of some of the crossfield pit membranes.

The possibility of developing biological pre-treatment for the preservation of poles of refractory species is discussed.

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APPENDIX

#### Introduction

(a) General Background to Current Preservation Practices and Problems

Throughout history, wood has provided man with one of his most valuable basic materials and even today the per capita consumption of wood products (except for fuel) is probably higher than at any stage in the past. In undeveloped countries wood is normally in abundant supply and tree species producing wood with any desired characteristic (strength, durability, workability, beauty, etc.) can be obtained by selective logging without trouble, and without the need for much thought of conservation. As countries become more developed, however, the forested area is invariably depleted and sooner or later this necessitates some measure of conservation. As the need for conservation becomes greater, and the value of land and labour increase. forestry becomes more of an economic excercise, and it is no longer possible to produce a wide variety of timber species. regardless of cost, to meet all the market requirements. Foresters tend to concentrate on those species which are the easiest to handle silviculturally, and which will produce the greatest volume of generally useful wood in the shortest time. In the temperate countries of Europe and South America, where this development has taken place to the greatest extent, the woody plants which best fulfil these requirements are the

conifers, the so-called softwoods, of the timber trade. Even in the sub-tropical countries of South Africa, South America and Australasia, forest management has concentrated on softwoods to supply the bulk of timber requirements, and where fast growing conifers have not occurred naturally, exotic species have been introduced, often with spectacular results in terms of wood volume production. The main advantages of softwoods for general utilization are their ease of sawing and machining, their good strength to weight characteristics, and their long fibre, making them particularly suitable for pulping for the production of newsprint and paper containers. From the forestry point of view the fastest growing conifers tend to be pioneer species in the ecological sense, and this makes them amenable to treatment as a crop which can be planted or regenerated in pure stands. Although such monocultures carry inherent risks of soil deterioration and epidemics of pathogens, the economic benefits in establishment, tending and harvesting are so great that this is, and continues to be. the preferred silvicultural system for production forestry. In such a system the seedlings are planted (or regenerated) in close proximity to each other so that in their early stages they grow in a slim upright form with little development of lateral branches. Successive tending operations remove a high percentage of the slower growing and worst formed trees

so that the full production potential of the site is concentrated on the eighty or so trees per acre which will form the final crop. These are harvested at the completion of the economic rotation (usually when the current annual increment of timber volume falls below the mean annual increment for the rotation) and this is generally well before the trees have reached full biological maturity. One result of this is that the final crop trees, which have been kept growing at maximum rate throughout their entire life, contain much less heartwood than would be found in naturally occurring trees of the same species at full maturity; the intermediate yields from thinning operations produce virtually all sapwood. While this is a desirable characteristic for the manufacture of pulp, it renders the product quite unsuitable, in its native state, for use as solid wood which may be exposed to biological agents of deterioration such as decay fungi, wood boring insects, or marine boring animals. Even where heartwood is present. the type produced by most of the fast growing conifers has very little natural dumbility. The heartwood of such species as larch, Douglas fir, and some of the slower growing Pinus species will last for several years exposed to the elements provided it is not in contact with the ground, but only a few relatively slow growing conifers (e.g. the western red cedar

quantities of a suitable, insoluble, toxic material in the heartwood to allow it to be used under the demanding conditions of ground contact.

To overcome this the forester looks to the scientist and technologist to find methods of altering the properties of the sapwood (and the non-durable heartwood) so that it can no longer be destroyed with ease by fungi and insects.

With the exception of a few animals which bore into wood for protection (e.g. marine borers of the family Pholadidae, and some insects such as carpenter bees) most organisms which destroy wood do so in order to utilize, the cellulosic and/or lignin fractions as nutrients. Thus it should be possible theoretically, to protect wood from these organisms by chemically modifying it so that it is no longer suitable as a food source. This approach is being investigated currently by several laboratories around the world but so far there is no indication that it is a feasible commercial proposition. External coatings serve a two-fold purpose in providing a physical barrier against the entry of organisms, and also by preventing or minimizing the entry of moisture which is necessary for their metabolism. The disadvantages of external coatings (e.g. a good paint system) are firstly expense, and secondly the fact that protection is lost completely if the coating wears off or becomes ruptured in service. The third alternative is to impresnate the wood to some depth with a chemical that is toxic or repellent to the organisms of deterioration. The

desirable properties of such a chemical are often listed in preservation literature as follows:- It should be toxic (or repellent) to all agents of biodeterioration but harmless to man and domestic animals. It should not adversely affect wood strength or other physical properties, or impair its machining, painting, gluing, etc. It should be chemically stable, non-volatile, and resistant to leaching. It should reduce, rather than increase inflammability, and should not be corrosive to metal fasteners. Finally it should be plentiful, cheap, and easy to impregnate into the wood.

No single chemical possesses all these attributes and it is most unlikely that one ever will, but there is no doubt that considerable progress has been made since the days when the ancient Chinese attempted to preserve wood by soaking it in strong saline solutions. Perhaps the most generally effective preservative used in the past has been creosote distilled from coal tar, and this is still widely used for the preservative treatment of marine piles, poles, and railway sleepers. The main disadvantages of creosote are its increasing cost, lack of availability in some areas, its colour and oily nature (creosoted wood cannot be painted and is dirty to handle) the high cost of creosote impregnation plant, and the fact that because of its odour and "dirtiness" it is unsuitable for timber to be used in dwellings except for such uses as foundation piles. As wood used in building construction is protected from

the elements by the exterior sheathing of the structure, or by a paint system, resistance to leaching is not of paramount importance and this simplifies the choice of chemical which can be used. Boron compounds have been shown to give excellent protection against wood boring beetles (the greatest hazard to wood when liquid water is excluded) and treatment methods which are cheap and effective have been developed for these in Australia (mainly for protecting hardwoods from <u>Hyotus</u> species) and New Zealand. In New Zealand diffusion treatment methods were developed by Harrow, (1954) & Carr, (1955) for hardwoods and softwoods up to four inches in thickness. This so-called momentary immersion process requires only very simple treating equipment, is effective for all species, and produces treated timber that is safe and clean to handle and use.

The great disadvantage of boron treatment is its leachability, and in addition to limiting its use to wood which
will be continually protected in service, care must also be
taken in storing the treated timber at the treating plant,
merchant's yard, and building site. Care must also be
excercised during the seasoning of boric treated timber as
kiln schedules designed to give optimus drying times will
cause migration of the salts to the surface layers and also
an over-all loss by steam volatilization.

Thus even if a wood utilization enterprise has the financial resources to install both creosoting and boron treating facilities, there remain several areas of incomplete technical fulfillment.

Probably the greatest single advance in wood preservation was the discovery by S. Kamesan of Dehra Dun Forest Research Institute in 1933 that a solution containing arsenate, copper and hexavalent chromium could be injected into wood to form a highly stable and effective wood preservative. This original copper-chrome-arsenate formulation (named 'Ascu' by its inventor) proved so superior to other water-borne preservatives which had been developed up to that time (chromated zinc chloride, zincmeta-arsenate, acid copper-chromate, fluor-chrome-arsenatephenol. mercuric chloride to mention a few) that with minor modifications it has become the most universally used 'Ascu', copper-chrome-arsenate formulations are now well known by such proprietary names as 'Greensalt' 'Tanalith C'. 'Boliden K33', and 'Celcure A'. The principal toxic ingredients and mode of action is the same for each of these preservatives and they differ only in the relative proportions of copper: chromate:arsenate, the chemicals used in the formulations (sodium or potassium salts in some and oxides in others), and in the nature of the commercial product (pastes - free flowing salts - twin packs).

The great advantage of copper-chrome-arsenate formulations is the coupling of high resistance to leaching by water, with a broad spectrum of biological efficiency against marine boring molluses and crustaceans, wood boring insects including termites, and decay fungi including soft rots. The one main disadvantage is that with normal commercial methods of impregnation, it is difficult to obtain satisfactory penetration into some species of wood. This is especially the case with large dimension timbers and particularly so with natural rounds e.g. piles, poles and fence posts, the only really economic outlet for the thinnings of softwood plantations where pulping facilities do not exist in close proximity to the forests.

Particular reference will be made in this introduction to the conditions pertaining in New Zealand as the author has worked in timber preservation in that country and is familiar with these conditions.

Also, New Zealand has the highest per capita consumption of treated timber in the world (over 11 cubic feet per head of population per year) using approximately 12 times as much as the next highest country, Sweden, and 30 times as much as the United States of America. In addition, New Zealand is the only country where all timber preservation is controlled by Government regulation and strict inspection procedures exist

to ensure that all treatment conforms to standards laid down by an independent Government-sponsored Timber Preservation Authority. The full requirements of the Timber Preservation Authority (TFA) are set out in a book "Timber Preservation in New Zealand - Specifications" (Revised 1969).

# (b) Vacuum/Pressure Impregnation.

The vacuum/pressure, or full cell, method of impregnating wood with preservatives has remained practically unaltered since it was patented by Bethell in 1838. The equipment consists basically of a pressure cylinder (usually 4-6ft. diameter and 50-100ft. long) with a door at one or both ends so that the wood to be treated can be wheeled in on some form of trolley. The only other essentials areavacuum pump, a pressure pump, a storage tank for the treating solution, and a mixing tank for preparing treating solutions of the desired concentration. Timber to be treated is dried to a moisture content below fibre saturation point (about 30% of the oven dry weight with most species) so that the lumena of the cells are devoid of free water, and then evacuated in the treating cylinder. A vacuum (22 - 26 inches of mercury at sea level) is drawn and maintained for a period of 15 - 30 minutes. The treating solution is then admitted and the pressure brought up to a pre-determined figure (usually 200 p.s.i. gauge pressure) and held until no further flow from the storage tank occurs. This so-called "treatment to refusal" is normally taken to be satisfied at virtual refusal i.e. when the rate of flow falls to a specified figure such as 2 gallons per 100cu.ft. wood over a period of 10 minutes.

The treating cylinder is then emptied of treating solution and a final vacuum is applied to remove surplus liquid.

The plant and ancilliary equipment required for this process is relatively inexpensive (o.f. crecesting plant requiring air pressure and heating), wood that is not too refractory can be impregnated uniformly and well, and the process is generally regarded as being the easiest preservation process to control.

In specifying required concentrations of waterborne preservatives in wood, the normal method is to state a weight of dry preservative salt in relation to a volume of wood. e.g. lbs/cu.ft. or Kg/cu.m., and this is attained in practice by adjusting the concentration of the treating solution in accordance with the expected liquid uptake of the wood being treated. The final retention of preservative is then calculated easily, by dividing the weight of salt used (volume of treating soln. x conc.) by the volume of wood, which is measured by the displacement of treating solution in the cylinder of known volume. Because wood shrinks when dried below fibre saturation point, the volumes of untreated wood will vary slightly with moisture content, so for uniformity the volumes are always calculated on the swollen volumes i.e. the displaced volume at the completion of treatment. The uptake of solution will vary with the density of the wood and its moisture content at the time of treatment and this can be calculated with a fairly high degree of accuracy as will be

shown later in this thesis. In commercial practice the expected uptake is estimated from general experience with the species and type of wood; allowances being made for the presence of any impermeable heartwood in the charge.

### c. Problems arising with various softwood species.

With copper-chrome-arsenate preservatives it has been shown that the ultimate location of the salts is within the wood cell walls (Belford et al. 1959; Petty and Preston, 1966) and in this location the compounds formed are resistant.to leaching by water. The exact mechanism by which the chemicals become deposited throughout the cell wall is not yet clear but it is certain that entry and fixation take place within a very short time of treatment. For this reason it is essential that all decay susceptible wood must be penetrated, for unlike the case with oily preservatives such as creosote (which remain as liquids in the cell lumena), untreated wood that is exposed by checking in service cannot gain protection from preservative which may migrate over the exposed surface from adjacent heavily treated areas.

Experience has shown that normal vacuum/pressure impregnation with CGA solutions does not always achieve this desired amount of uniform and even penetration, particularly in natural rounds such as piles, poles and posts where a deep envelope of well treated wood is essential. In the 1969 revision of the New Zealand T.P.A. specifications the species which may be treated with CCA preservatives for these commodities are limited to the following:-

- Marine Files Pine species, except that treatment by process specification P9 (oscillating pressure) is restricted to Corsican and radiata pine.
- (ii) Round poles <u>Pinus</u> species (Austrian, Corsican, loblolly, lodgepole, longleaf, maritime, muricata patula, ponderosa, radiata, shortleaf, slash and strobus) and kawri (Agathis australis).
- (iii) Posts and sawn timbers for use in ground contact:. As for (ii) i.e. kauri and Pinus species except that some Pinus species (lodgepole, miricata, strobus, slash and patula) are not approved for treatment as low durability and untreatability. Several other species of softwoods have been critically examined by the T.P.A. and some of these have been approved for treatment at various times in the past. These include Douglas fir (Pseudotsuga menziesii), larch (Larix decidua), macrocarpa (Cupressus macrocarpa), redwood (Sequoia sempervirens), Lawsons cypress (Podocarpus totara), tanekaha (Phyllocladus trichomanoides) rimu (Dacrydium cupressinum).



#### Photograph 1

Sections cut from the centre of Douglas fir fence posts after pressure treatment with copper-chrome-arsenate solution.

None of these is now approved for the treatment of wood to be used in the sea, or for ground contact, or other high decay hazard use. The exotic species listed (Douglas fir, larch, macrocarpa, Lawsons cypress, redwood and sugi) are proscribed because treatment trials have shown that penetration of the treating solutions is either very shallow, (even in the sapwood) or is very irregular and varies from one charge to another or even from one piece to another in a seemingly uniform charge, (see photograph 1). With the New Zealand indigenous species listed the uptake of treating solution is normally good (approaches saturation, albeit at a slower rate than with Pinus species), but the distribution of the salts in the treated wood is irregular; copper in particular appears to be "screened out" and concentrated in a narrow peripheral zone. Several other exotic species such as the spruces (Picea abies and P. sitchensis) are excluded from the approved list because their reputation for treatment in their countries of origin is so notorious that no-one in New Zealand has even bothered to attempt treating them.

The main purpose of the research covered by this thesis has been to try and find some reasons for the differences in treatability exhibited by these various species, and to suggest where possible how the treatability of the refractory species might be improved.

#### PART I

# Permeability

#### a. Background and literature survey.

Although there has been a considerable amount of research over the last sixty years on the subject of wood permeability. very little of practical value has emerged, and the basic reasons for variations in ease of treatment are almost as obscure as ever. One possible reason for this lack of success is that the problem has been approached from two completely different angles with little nutual appreciation of the factors involved. On one hand practical wood treaters have attempted to improve the treatment of different species and commodities by purely empirical methods such as variations of time, pressure, vacuum, temperature etc. with little knowledge of the wood structure problems involved, while on the other hand laboratory research workers have studied fine structures, pore size, and directional permeability with little thought of the practicalities of commercial treatment.

The first approach has resulted in some improvements in treatment but these have been small in comparison with the large amounts of time, trouble and expense that have been consumed. The worst espect of this approach is that there may be a continuing expenditure on problems that are insoluble with

the equipment being used. The second approach has suffered mainly through over generalization in attempting to apply the findings of investigations carried out with small specimens under abnormal physical conditions. Because of this, promising theories have often given contradictory results in practice and much time has probably been lost in pursuing irrelevant details. An example of this may be the vast amount of time spent on studying the bordered pit pairs in coniferous tracheids and their relationship to longitudinal flow rates. From the time of the first work by I. W. Bailey (1913) this structure has been studied by numerous research workers in attempts to show its precise structure, function, behaviour, and effect on permeability. This work has included several studies on the mechanism and effects of pit aspiration (I. W. Bailey, 1913, 1916; Griffin, 1919, 1924; Scarth, 1928; Sutherland, 1932; Phillips, 1933; Stone, 1936, Johnston and Maass, 1930; Henriksson, 1957; Kishima and Hayashi, 1962: P. J. Bailey, 1966: Krahmer and Côté, 1963; Thomas, 1967, 1969: Liese and Bauch, 1967: Comstock and Côté, 1968). From these it is now accepted that pit aspiration is caused by surface tension forces exerted by the cell water as wood is seasoned, and that it can be avoided, or at least minimised, by drying by solvent exchange through a series of water miscible liquids with low surface tension. It has been

demonstrated also that solvent exchange drving results in greater longitudinal permeability to both gastes and liquids under laboratory conditions but it has not been shown conclusively that pit aspiration is an important factor in commercial treatment. Furthermore, no-one has yet put forward a feasible suggestion as to how pit aspiration could be overcome on a commercial scale. Henriksson (1952) considered the "value action" of unaspirated bordered pit pairs in green wood and from equations of cabulated closing pressures, developed the theory of the Oscillating Pressure Method for the pressure New Zealand (McQuire, 1964) showed however that these equations were not valid, and that with the only species in which treatment was completely satisfactory (Pinus species) the penetration was madial and not axial. Pre-treatment of green wood with mercuric chloride has been claimed to fix the torus of the bordered pit pairs of trachieds in an unaspirated condition, but this was tried by the author with no were treated by vacuum sap replacement using a solution of HgCl, and then were peeled and seasoned. Vacuum/pressure treatment with CCA solution at 200 p.s.i. resulted in an unsatisfactory standard of treatment (photograph no. 2) which was not detectably superior to the similar posts that had not been pre-treated. According to Stone (1936), pre-steaming green



# Photograph 2

Sections out from the centre of Douglas fir posts pre-treated with  ${\rm Hg\,Cl}_2$  and pressure treated with CCA preservative.



the state of the same of

# Photograph 3

Sections out from the centre of Douglas fir posts which had been pre-steamed, air dried, and pressure treated with CCA preservative.

wood causes a slight increase in the percentage of pits aspirated during seasoning but in the same series of trials with Douglas fir in New Zealand this treatment resulted in a noticeable improvement in the uniformity of preservative penetration (Photograph no. 3).

The pre-occupation with bordered pits has come about probably because most laboratory studies on the permeability of wood in different grain directions have shown that flow rates in an axial direction are greater (and often very much greater) than in either of the transverse directions. This has led to the assumption that it is axial penetration which is important in preservative treatment. What has been generally neglected or overlooked in these considerations is an assessment of the effectiveness of any such penetration, and also a comparison of the areas offered and distances involved for various directional flows in commercial treatment.

A simple definition of "effective" penetration" is impossible because it depends on the type of commodity (its size, shape, importance, unit value, use to which it is put, ease of replacement eto.) and the preservative being used.

In general it may be said that decay susceptible timber must be penetrated, and particularly with highly fixed preservatives such as CCA salts, this penetrated zone must be uniformly and evenly treated. With large section members an envelope of

treated wood will normally suffice provided this is deep enough that the underlying wood will never be exposed during normal service life. Thus preservation specifications with CCA salts generally require an over-all retention of salt (lbs dry salt per cubic foot) plus a minimum penetration at some fixed point such as the mid point or ground line in poles or posts. Recently there has been a swing towards endresults type specifications in which a definite minimum retention of one or more of the toxic components that can be checked by chemicalanalysis is required within any part of the treated zone. A pole specification of this type might read "Penetration must be continuous and uniform to a depth of 1 inch (or 80% of the sapwood depth whichever is the greater) and the retention must be such that chemical analysis of any part of a zone from 1 inch to 1 inch from any surface must show a loading of at least 0.24% CuO equivalent as a percentage of the oven dry wood weight in 90% of samples taken.

A specification such as this places a great premium on radial penetration in round poles. Considering a pole (which for illustrative purposes can be taken as a cylinder) of length 20 feet, diameter 9 inches, with a required depth of penetration of 1.0 inches, the areas available for penetration during treatment, and the distances the solution will have to travel in an axial and mdial direction are as follows:-

Area for radial penetration =  $\pi$  x 9 x 20 x 12 = 6,800 sq. ins. " " axial " =  $2(\pi x 4.5^2 - \pi x 3.5^2)$  = 50 sq. ins. Ratio of radial area : axial area =  $\underline{136}$  : 1 The distances the preservative solution would need to travel

Axial from both ends) = 120 inches

Ratio of axial distance to radial distance = 120 : 1

are - Radial = 1 inch

Thus the advantages of radial penetration over axial penetration are great, but nevertheless there has been very little attention given to this and many workers have dismissed it out of hand. Stamm, 1967 in surveying the literature on directional flow in softwoods quotes Johnson and Maass (1930) as stating that flow in the fibre direction is 50 to 100 times as great as across-the-fibres (without differentiating between radial and tangential flow). He then summarises his article by stating that flow is 100 to 200 times greater along the fibre than in the transverse direction. No mention is made here of species, whether this applies to heartwood or sapwood . or what the moisture content or method of seasoning was. Hunt permeability at that time by stating that "even under the best conditions, the rays probably play only a subordinate role in the impregnation of softwoods." This general opinion has persisted until the present time and the third edition of Hunt and Garratt (1967) states that "except in softwoods that possess unobstructed resin ducts ( in fusiform rays) preservatives must pass through many cell walls and pits in moving a relatively short distance across the grain either radially or tangentially." Some workers have not agreed with this contention however and the experimental results of others do not support it.

Scarth (1928) considered that radial resin canals were important "trunk routes" for penetration into sapwood and this was also the opinion of Cox and Irwin (1953) and Kishima and Hayashi (1960). The latter acknowledged however that resin canals are not important pathways for preservative treatment because of their small number in relation to over-all wood volume. Schulze and Theden (1948) considered the rays of Pinus sylvestris wood to be permeable but Buro and Buro (1959) stated that only the ray tracheids and radial resin canals were permeable; not the ray parenchyma. Kolio (1953) showed radial penetration in pine to be ten times as great as in spruce, and Schulze (1960) found a higher percentage of ray area in easier treated spruce. Fleischer (1950) compared wood from sections of Douglas fir that had proved easier and harder to treat than normal and found that there were more rays and more fusiform rays in the esier material. He also found more rings per inch. larger tracheids. and more "apparently unaspirated" pits, but as the standard of treatment of even the "easier" samples was not very good, these correlations may be meaningless. Wardrop and Davies (1961) examined the flow paths in various grain directions in

<u>Pinus radiata</u> and considered that ray parenchyma cells were preferred to the ray tracheids.

Koran (1964) examined permeability in various grain directions in Douglas fir using \$ inch cubes, and found that permeability in different directions changed somewhat with different temperatures and pressures. At 100 p.s.i. the sapwood was almost as permeable tangentially as axially and these were twice as great as radial permeability at 70°F but only about 10% higher at 212°P. At atmospheric pressure and 70°F longitudinal permeability was three times that of the radial direction which was slightly more than tangential, while at 212°F redial permeability was double tangential and only 25% below axial. Different permeabilities under different conditions were also shown by Erickson and Estep (1962) who found the relative flow rates in unseasoned Douglas fir heartwood in the directions tangential : radial : axial to be about 1: 14: 63. When this wood was seasoned the axial flow rate was unaltered while madial decreased by 50% and tangential increased. Sargent (1960) examined radial penetration in Douglas fir. hemlock, sitka spruce, balsam fir, western white pine (soft nine) and loblolly nine (hard nine). His general conclusions were that the degree of ray penetration obtained correlated closely with the accepted treatability of the species concerned. He considered ray tracheids to be generally more permeable than ray parenchyma and ascribed this to the "resin-like" material

which often covered the end walls of the ray parenchyma cells. An attempt to correlate depth of penetration of ray parenchyma with cell lengths showed a periodicity of approximately the right order but did not prove conclusively that the end walls were a barrier to penetration. A barrier apparently existed at the late wood - earlywood boundary in some rays but no explanation of this was found. A visual dynamic microscopic observation of creosote movement under low pressure in an axial direction demonstrated the intercommunicating nature of rays and axial tracheids, and showed that axial tracheids were sometimes penetrated in a direction opposite to the general flow direction, via a ray. This is demonstrated also in a film made by Hickson's Timber Impregnation Co., showing a dyed aqueous solution moving through Scots pine sapwood under pressure.

Liese and Bauch (1967) examined the radial permeability of Douglas fir, spruce, larch, and Scots pine, and found a correlation between enertability and percentage of ray tracheids present. Hayashi and Kishima (1965) compared permeability in the axial and radial directions for Pinus densiflora.

Chamaecyparis obtuse and Cryptomeria japonica and found that radial penetration in the Pinus samples was twelve times that in the other two. Radial penetration in Pinus was as great as axial penetration in the other two species. They were unable to find any difference in penetrability between ray tracheids

and ray parenchyma. Choong, Tesoro and Skarr (1966) examined the transverse air permeability of six softwoods (and nine hardwoods) and obtained a good correlation between this and treatability with creosote. In these experiments, transverse movement of creosote could be either radial or tangential. Pinus echinata and Taxodium distichum were found to have much higher transverse permeability than the other softwoods viz. Larix occidentalis, Picea sitchensis, Pseudotsuga menziesii, and Abies balsamea.

#### (b) Methods used in determining permeability.

two or more samples of wood is to submerge the samples in a liquid and remove them after a set time, weigh them and compare liquid pick-up as either an absolute quantity or in relation to the weight or volume of the sample. If the solution is coloured some information on the depth of penetration can be obtained also from this. The method is suitable for dry wood only, but in the context of work aimed at finding information relevant to vacuum/pressure treatment this limitation is quite acceptable. By sealing all but one of the faces of the wood sample it is simple to limit penetration to one grain direction and this was the method used by Hayashi, Nishimoto and Kishima attaching the wood sample to a weighted platform and suspending this from a direct reading balance. With this apparatus they

were able to plot uptake as mg. liquid per cm<sup>2</sup> of surface area against time. The disadvantage of this technique is that uptake can be measured only at normal atmospheric pressure and the blocks are filled with air which must diffuse out from the cells before the liquid can penetrate.

Graham (1964) used a simple "sink-float" test to compare of dry wood (e.g. 2 inch cubes or samples made longer in the axial direction) are evacuated in a vacuum desiccator and vacuum. A perforated plate is used to prevent the samples underside of this. Air is then admitted to the jar and the time taken for individual samples to sink is noted. This uptake will not represent total saturation of the wood because of the higher density of wood substances but if the basic density (oven dry weight) and initial weight and moisture content are known, the actual uptake can be calculated. For most wood species the sinking point in water will correspond to an uptake of 75 - 85% of the maximum possible uptake. Unidirectional penetration can be obtained in this method also by sealing all but one (or two) of the faces. This technique is an improvement on that used by Hayashi and coworkers in that by using an initial vacuum to remove the air in the wood, the conditions come nearer to those pertaining in

pressure treatment. Although it would not be possible to increase the pressure above stmospheric pressure in a vacuum desicator it would be a relatively simple matter to construct a chamber (with a siting window, or wholly from perspex) so that positive air pressures could be exerted after the vacuum was released, and noting the "sink time" of individual samples when subjected to various pressures. The disadvantage of this technique is that, even with refinements of pressure, there is only one uptake/time observation possible i.e. when the sample just commences to sink.

Koran (1964) used 3 inch cubes (of Douglas fir) sealed on five sides with several coats of a solution of cellulose acetate in acetone to determine unidirectional uptake of creosote under various pressures and at various temperatures. No mention is made of an initial vacuum so presumably this was not used; crecsote is normally applied by the Lowry or Rueping process where the liquid is injected against atmospheric or raised air pressure in the wood in order to recover surplus oil at the completion of the treatment. No siting or uptake monitoring devices were used and treatment was finished after a pre-determined time (8 hours) whereupon the blocks were removed, weighed, and the uptakes calculated as a percentage of the uptake in those blocks which were considered to be saturated. The obvious disadvantage of this method is that there is no means of judging how the penetration is proceeding during treatment, and numerous experimental runs must be made for every

size and species to determine an optimum time for each set of conditions.

A completely different school of thought has attempted to determine the permeability of wood by measuring the flow rate of liquid or gas through wood specimens. The simplest of these utilizes a hydrostatic head of water with the wood sample fixed in a tube leading from the bottom of this reservoir. Water emerging from the other end of the sample is collected in a graduated vessel and volumes collected are plotted against time. These volumes can be expressed in absolute terms for standard sized specimens or related to the length and cross-sectional area of the specimen. Hayashi et al. (1966) increased the pressure differential across the specimen by connecting the outflow end to a vacuum pump; a desirable consequence of this modification is that the sealing sheath is forced more tightly around the specimen. They measured "permeation rates" as cc/cm2 against time. Hartmann-Dick (1954) further increased the pressure differential by using a pressure of two atmospheres applied to a Thiessen bacterial pressure filtering apparatus in which the porcelain filters were replaced with discs of wood. He obtained "filtration values" for various wood samples using an equation

Filtration Value = mls. liquid x disc width (mm.) sectional area (cm<sup>2</sup>) x time (min).

P. J. Bailey used regulated water mains pressure and compressed

nitrogen to force water through round discs of Douglas fir clamped into a metal holder with O-rings. He also used a back pressure (compressed nitrogen) to vary the differential in relation to the applied pressure. The wood discs were all made to a standard size and flow rates were measured as mls/min and plotted against time. The same apparatus was used by Petty (1967) but in this case the main object of the investigation was to calculate pore sizes in bordered pit membranes using non-swelling liquids and gasses.

Choong, Tesoro and Skaar (1966) used gas flow through thin sections of dry wood to calculate the permeability of six softwoods (see Part 1(a)) and nine hardwoods. In their apparatus the wood sections were cemented to the end of a cylindrical glass tube and all the exposed wood except the area corresponding to the orifice of the tube was coated with paraffin wax. The pressure differential was obtained by a vacuum on the exit side of the specimen and flow rates were measured by the rate of rise of liquid in an inverted graduated cylinder (in an open liquid tank) on the entry side. Measurements could only be made until the air initially present between the liquid and the specimen was exhausted. Permeability was expressed as Darcy units from the equation:

$$K = \frac{QL}{A(P_1 - P_2)}$$

where K is the permeability constant of the specimen.

- Q = vol. air ccs/sec
- L = thickness of specimen (cm)
  - A = area (cm<sup>2</sup>)

P<sub>1</sub> - P<sub>2</sub> = Pressure drop across the specimen in atmospheres.

Smith and Banks (1970) used sas flow through wood specimens.

Smith and Eanks (1970) used gas flow through wood specimens to determine permeability and pore size and were able to separate the viscous (Poiseuille) component of the total flow from the slip (Knudsen) flow of the gas molecules. They used hydrogen, nitrogen, helium, neon, and krypton, and checked the pore sizes obtained by using artificial pore-sized constrictions made by sandwiching various micropored filters between sections of finely pored diffuse-porous hardwoods.

Although the results from these flow rate investigations provide a general measure of the permeability of the wood specimens used, and there is some agreement between the permeabilities obtained and the known treatability characteristics of the species, the approach has several serious disadvantages. Where gases are used to measure flow rates the moisture content of the wood will alter with time until it reaches equilibrium with the system and this will depend on the relative humidity and temperature of the gas. Progressive changes will alter the flow rates and the only practical way to avoid this is to use dry gas and oven dry wood, a condition that is never encountered in normal wood preservation. Smith and Banks state that liquid flow can be predicted from these (gas flow) data with some qualifications:

- (1) Liquid pressure may cause pit sealing with some species.
- (2) Swelling liquids may alter the characteristics of the medium.

A very real problem with flow rate measurements is the difficulty of effectively sealing the wood specimens so that the flow is restricted to one grain direction and short circuiting is prevented. In this respect the systems used by Hayashi and co-workers, and Choong and co-workers are the most effective in that external pressures tend to reinforce the seal by pressing it tightly against the surfaces of the wood. Where pressure is exerted against the seal it seems almost impossible to prevent some short circuiting by the liquid. When sections of very refractory wood were prepared for radial and tangential penetration in the apparatus designed by Bailey, the use of dyed solutions showed that movement often took place around the 0-rings instead of directly across the specimen.

Perhaps the greatest criticism of flow rate measurements is that once a flow path has been established liquid will continue to move through this (unless it becomes blocked by pressure on pits, particles in the solution, or air embolisms) and large areas of the wood may remain unpenetrated. If any artificial openings, (e.g. checks or splits caused by drying or specimen preparation) exist, the readings will be abnormally high and this will also be the case with species that have

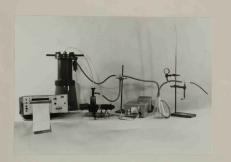
large unobstructed resin canals running in the same direction as the liquid movement. Even where resin canals or artifacts do not exist the flow may not be uniform over the whole area of the specimen; Petty (1967) (using dyed styrene which was later polymerised in the cells) showed that even in green wood having a minimum number of aspirated pits, longitudinal flow was not uniform and a considerable number of the tracheids appeared to be non-conducting. In air dry spruce wood, where the majority of the earlywood bordered pits were aspirated, longitudinal penetration of reduced basic fuchsin solution was almost wholly through the latewood tracheids and even in this region not all of the cells were conducting, (Fetty 1970).

For these reasons, permeability as determined by flow rate measurements may not bear a very close relationship to treatability with preservative solutions such as copper-chrome-arsenate where uniform and even penetration to a condition of virtual saturation of the wood is essential.

It was therefore decided to develop a piece of apparatus which would measure uptake of solution into solid blocks of wood, in any desired grain direction, and record the rate of uptake continuously from the time of initial penetration right through to complete saturation.  (c) Uptake monitoring Apparatus - Development, use and method of assessing results.

In commercial vacuum/pressure treatment uptake is monitored and measured by periodic readings of the solution level in a graduated storage vessel. This method was examined as a possibility for a laboratory - size piece of equipment but rejected because of difficulties in making accurate measurements on that scale. When passing from initial vacuum to pressure after admitting the solution, the level in the fine, calibrated tube, was found to fluctuate violently for some considerable time, and with permeable samples about 60 - 80% of the uptake had taken place before the level settled down enough for an accurate reading to be made. Expansion and contraction of the apparatus under pressure and vacuum was another factor mitigating against making accurate uptake readings to the desired scale of ± 0.1 mls. of solution.

The only other possibility was to develop a piece of equipment in which the wood sample itself could be weighed continually during impregnation, and the weights recorded. Attempts to find a mass transducer, displacement transducer, or strain gauge, which might perform this function were unsuccessful as none could be located that would be guaranteed to stand up to the environmental conditions of vacuum and pressure while immersed in a liquid, especially one which might have corrosive properties.



## Photograph 4

Uptake monitoring apparatus - complete apparatus except for vacuum pump and nitrogen cylinder.



Photograph 5

Impregnating vessel with block attached to the supporting platform.

#### Uptake measuring apparatus

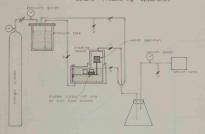
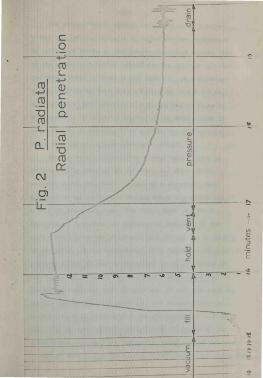


Fig. 1



The system finally adopted uses light beams directed across the treating cylinder to signal changes in buoyancy in small wood blocks as they absorb liquid. A general view of the complete apparatus (except for the nitrogen cylinder and vacuum pump) is shown in photograph 4, and a close-up of the treating cylinder with a wood block (2 x 2 x 2 cm.) held on the support platform by a rubber band is shown in photograph 5. A schematic diagram of the whole system is shown in Fig. 1, and Fig. 2. is a photocopy of a recorder trace covering a complete treatment schedule.

The blocks are supported on a metal spring which is attached to the back of the cylinder, and which can be adjusted before treatment by means of a centreing screw. Attached to the spring is a shutter which cuts across one of a pair of twin light beams (the object beam) and which increases or decreases the intensity of that beam as the spring moves downwards or upwards with changes in the density of the block. At the commencement of a treatment, the initial position of the shutter is determined by the dry weight of the block, and as every block is different an adjustment of the spring centreing screw is necessary to zero the scale on the electronic box (photograph 4). The recorder trace is zeroed also by an adjustment to the galvanometer. Thus the zero line on the recorder represents the dry weight of the block before treatment and during the vacuum part of the schedule. When liquid is admitted to the chamber containing the block, the block is raised until the upward movement is restrained by the spring. This movement of the spring (and shutter) decreases the intensity of the object beam light and hence increases the differential signal to the recorder. By adjusting the output signal from the electronics the trace deflection from this signal is made to cover more or less the full scale deflection of the recorder, i.e. to the uppermost point on the trace in Fig. 2. As the block absorbs liquid, it increases in density and this increasing downward force on the spring increases the object beam intensity. Thus a gradual uptake of liquid is registered on the chart as a gradual decrease in the differential signal to the galvancester.

The light source is a microscope lamp powered by a smoothed DC supply of 6V. In the earliest trials AC power was used but this resulted in a very broad indistinct trace on the UV recorder so the supply was changed to DC. The light beam is divided into two idential beams before it enters the chamber by a pair of shielded perspex rods; similar rods on the opposite side of the chamber receive the beams and focus then on to a pair of COP 71 germanium phototransistors. The electronics consist of a zero control on the phototransistors, a differential amplifier with emitter follower, and a 100 chm variable resistor output control. The amplifier provides a 200ww beam differential signal to the galvanometer of a variable speed UV recorder. Chart speeds range from 0.2 inches per minute to 5 inches per second in eight steps which can

be changed by push button during operation.

When first used, the wood specimen was evacuated in a completely dry chamber, but this was unsatisfactory because the differential scale altered drastically through absorption of light by the liquid when the chamber was filled. Modification of the electronics to give a ratio instead of differential signal would have been a major undertaking, so the apparatus was modified. In the modified form the light beams always pass through liquid and the block is held above the liquid level during the vacuum period on a platform attached to a vertical extension of the spring (see photograph 5.). A vertical cylinder was added to the apparatus to accommodate this. This meant that it was no longer possible to draw a very high vacuum, but as the vacuum in commercial treatment does not exceed about 26" Hg at sea level, this was not thought to be a serious loss.

Because the germanium phototransistors are affected by temperature, and also because they have a sharp absorption peak in the infra-red part of the spectrum (100% transmission at 1.55µm but only 10% at 1.6µm) the apparatus was very sensitive to any temperature changes. Although it has been housed in a temperature controlled room it has been necessary to zero the phototransistors frequently when carrying out treatments. For future work the germanium phototransistors are to be replaced with silicon phototransistors (EFX 25)

which will not be affected by fluctuations in temperature and which have a much broader spectral response (40% at 0.4 µm and 60% at 1.0 µm with a broad peak at about 0.9 µm). This will give a much steadier output, and although linearity of response may be reduced slightly, preliminary trials indicate that this will be negligible over the range used.

For a complete treatment the sequence of operations is as follows:-

- i) The wood specimen is fixed to the platform, the top section of the treating vessel is screwed on, and valves 1, 2, 3, 6, and 7 are closed. Valves 4 and 5 connecting the pressure tank and the top of the treating vessel to the vacuum pump are open.
- The vacuum pump is switched on and the vacuum modulated to 26" Hg by adjusting venting valve 7.
- iii) At the completion of the vacuum cycle, valve 2 is opened and the liquid siphons from the pressure tank to the treating vessel while the vacuum is maintained throughout the system.
- iv) When the treating vessel is full (as shown by liquid passing through a siting glass in the line by valve 5) valves 4 and 5 are closed and the vacuum pump is switched off.
- v) When the phototransistors have been zeroed and the output dignal is stable, valve 3 is opened and the system is vented to atmosphere.

- wi) When the venting to atmospheric pressure is complete (about \$\frac{1}{2}\$ min.) valve \$\frac{3}{2}\$ is closed and valve \$1\$ is slowly opened to admit nitrogen to the pressure tank and bring the system up to the required pressure.
- vii) At the completion of the pressure period (i.e. when the recorder shows the wood is saturated, or when no further uptake is being obtained, or at a predetermined time) valve 1 is closed and valve 3 opened to vent the system to atmosphere.
- viii) When venting is completed valve 2 is closed, valves
  5 and 6 are opened and the liquid drains from the
  top cylinder containing the specimen. If it is
  desired to ascertain that no further penetration
  takes place, the vacuum pump can be switched on
  before valves 5 and 6 are opened so that the treating
  vessel can be drained under vacuum.

## Specimen preparation

Specimens to be used in the uptake measuring apparatus were prepared from flat sawn, even grown, sapwood boards (free from knots, compression wood or other defects) that had been conditioned to an equilibrium moisture content of about 10% in a constant temperature and humidity room set at 25°C and 50%RH. The boards were rip-sawn and thicknessed into sticks 2cm x 2cm in cross-section, and these were then sawn into 2cm cubes with a very fine tooth cross-cut saw. Every block was numbered so that the board, stick, and position

within the stick were known. After a further period in the conditioning room to ensure constant weight had been reached, the blocks were weighed to the nearest 0.01 gramme. Every third block from each stick was then oven dried at 105°C to determine oven dry weight and moisture content (moisture present as a percentage of the oven dry wood weight). These blocks were then saturated with water in a vacuum desiccator, the swellen dimensions were measured with a micrometer, and the swellen volumes were calculated to 0.01cos. Noisture contents and basic densities (oven dry weight), were recorded for these blocks, and then for the other blocks interpolated along the sticks. The blocks which had been oven dried and saturated were then discarded and the remainder were stored in the conditioning room until required.

When the blocks were required for uptake tests they were weighed and then sanded down on all faces except those to be exposed to the liquid. Edges and corners were rounded off slightly to give better adhesion of the sealing compound at these points. In the early trials, four faces were sealed so that liquid could penetrate from two opposing faces, but in all later treatments (as reported in the results section of this and future chapters) five faces were sealed so that penetration was restricted to one face. In the case of radial penetration the outer face (that which was furthest from the

pith in the tree) was the one left unsealed.

The first sealing compound tried was exproxy resin, but although this provided a strong, waterproof coating with good adhesive properties, it was rejected because curing times proved too critical. Curing at elevated temperatures was undesirable because this would have affected the moisture content and may also have altered the permeability. Room temperature curing took at least a day, and if it proceded too far the resin became brittle and fractured when the wood absorbed water and began to swell. Cellulose acetate dissolved in acetone (as used by Koran, 1964) provided a much quicker setting seal which did not become brittle, but was found to be insufficiently strong to withstand high liquid pressures when applied over the end grain of species with large diameter early wood tracheids e.g. Sequoia sempervirens. The best sealing compound for the purpose was ABS polymer dissolved in methyl ethyl ketone. This compound adhered well to the wood (provided the first coat was applied in a fairly dilute, low viscosity condition) and set very quickly so that the whole process of applying three coats took only about half an hour. The final seal was strong and elastic enough to withstand liquid pressures of 100 p.s.i. over the largest tracheids and resin canals, and to accommodate tangential swellings of up to 7% without rupturing. All species were tested with the seal by coating all six faces of test blocks and submitting them to a

vacuum cycle followed by 100 p.s.i. liquid pressures.

#### Assessment of uptake

Because blocks of different species to be examined in the series would vary in density, and possibly moisture content, (both of which factors would influence the amount of liquid that could be absorbed) it was decided that all assessment would be in relation to the maximum possible uptake of each block. This was calculated from the simple basic equation:maximum volume volume of volume of swollen volume of liquid which = of block can be absorbed substance initially present density of wood - (W - ODW) substance

where ODW = oven dry weight of the block  $ED = Basic \ density = \frac{oven \ dry \ weight}{swollen \ volume}$ 

W = weight of the block.

The density of wood substance is a subject that has been debated for many years and figures ranging from 0.73 (Jayne and Kause, 1963) to 1.54 (Wangaard, 1969) have been proposed. Values below 1.0 cannot be entertained seriously and most workers accept values of 1.44 to 1.54. Lower values of 1.44 to 1.47 are found when the density is measured in non-polar displacing media, and the general conclusion is that these liquids cannot penetrate the small void spaces in the dry cell wall, but that water can. With water as the displacing medium,

the wood substance density values obtained by several workers have been consistently around 1.53 - 1.54 and as the treatments in these trials were all with water or dilute aqueous solutions, it was decided to use the figure of 1.54.

Thus the maximum possible uptake of a block of oven dry wood

$$=$$
 ODW  $(\frac{1}{BD} - \frac{1}{1.54})$ 

From this:

max. saturated weight of an oven dry block

$$= ODW \left(1 + \frac{1}{BD} - 0.65\right)$$

As the oven dry weight and basic density were calculated for every block, the maximum saturated weights were known, and it was a simple matter to weigh a block just before treatment, and subtract this from the maximum saturated weight to determine the maximum possible uptake. This was complicated slightly by the necessity to sand the blocks to obtain a perfect seal with the ABS compound, and the complete procedure and calculations required were as follows:-

- a) BD = basic density | recorded for each block.
- c) W<sub>1</sub> = weight of block immediately before preparation for treatment.
- d) MC = moisture content =  $\frac{\text{W}_1 \text{ODW}}{\text{ODW}}$  x 100
- e) W1(b)= weight after sanding
- f) ODW(b) = oven dry weight after sanding (calculated from e) and d).

- g) saturated weight of sanded block = ODW(b)  $(1 + \frac{1}{ED} 0.65)$
- h) maximum uptake of sanded block =(g) -(e)
- i) W2 = weight of block after coating with sealer
- j) W3 = weight of block after treatment
- k) Uptake =  $W_3 W_2$
- 1) Uptake as a percentage of maximum possible =  $\frac{(k)}{(h)}$  x 100

Two sources of error are possible in these calculations. The first results from the original interpolation of basic densities and moisture contents from along the sticks. However, as the blocks are fairly small, and every third one along the stick was used to determine these values accurately, the error is unlikely to be large. The second possible error lies in the assumption that neither moisture content nor basic density are affected by the sanding.

Although errors from either, or both, these sources should be small, they could have a significant effect on calculations made on blocks of only 8ccs total volume, and in practical terms it is probably reasonable to consider uptakes anywhere in the region of 90% of the alculated maximum as representing virtual saturation.

# Calibration of Uptake monitoring apparatus

The initial calibration of the recorder was made with a series of weights on a lever attached to the platform which

supports the block in the treating vessel. By adjusting the output control to the galvanometer it was possible to calibrate the recorder to ensure that with the largest blocks and uptakes the scale deflection would not be too great for the chart width; a suitable scale proved to be approximately one chart division per gramme weight. A permanent calibration was impossible because with blocks of different initial weight the zero-control had to be adjusted and this slightly altered the calibration.

At the completion of each treatment the block was
weighed and this provided an accurate determination of the
over-all uptake. Uptake calibration was then made simply by
dividing the total movement of the trace during the uptake
period by the total uptake weight. Uptakes as absolute units
of weight and as percentages of the maximum possible uptake
could then be calculated for any time scale point on the trace.

These calculations are valid only if it is certain that the calibration did not shift during the course of the treatment and to check this, calibration checks were made comparing density changes in the block during filling and during treatment.

# a) Filling calibration

Exposed weight - (W1) = weight of block + weight of exposed part of support

submerged weight-  $(W_2) = W_1$  - (volume of block + volume of previously exposed holder)

change in weight = W1 - W2

 Volume of block + exposed holder (measured in ccs, expressed as grammes)

Calibration = divisions on chart/change in weight

#### b) Treating calibration

Weight before absorption  $(W_{\frac{1}{2}})$  = weight of block - volume Weight after absorption  $(W_{\frac{1}{4}})$  = treated weight - treated volume Change in weight - W. - W.

change in weight = W<sub>3</sub> - W<sub>4</sub>

Calibration = division on chart/change in weight

As the calculations in these check calibrations take account of changes in block volume as well as changes in weight the results from a) and b) should be identical. In practice they were generally within † 2% and the treatment was rejected if the difference was greater than 10%.

The calibration for determining weight uptake by the block at various times does not take swelling into account and this introduces an error into the calculations. A comparison of uptake and swelling rates showed these to be very close, however, (Appendix II) and as the change in volume (maximum measured about %) is small compared with the change in weight (up to 120%) the error at any particular time could not be great enough to affect the results to any marked extent.

### d) Species used in Permeability Tests

In selecting the type of material to be examined it was decided to restrict this to the sapwood of softwood species that are commonly used for poles etc. and those which have fairly well established treating characteristics. It was desired also to cover as many different structural types as possible, particularly with regard to ray structure. The species finally chosen were Pinus radiata, Pinus nigra (Corsican), Pseudotsuga menziesii, Sequoia sempervirens, Cryptomeria japonica, Cupressus macrocarpa, Podocarpus dacrydioides, Agathis australis. Air dried sapwood of all these species was obtained from New Zealand and details of the source, seasoning and gross physical characteristics (density, rings per inch etc.) are shown in Appendix I. Table 1 (derived from Greguss, 1955 and Patel, 1967) shows details of the microscopic structure of these species and also of Picea sitchensis which is introduced in a later part of this thesis. The treatability ratings are based on Timber Preservation Authority assessments in New Zealand and on the author's personal experience of treatment trials with these species at the commercial level.

It is stressed that it was never intended that these
trials should provide a treatability classification of the
species being examined. To do this would necessitate testing
numerous samples of different origin, age, seasoning and preparation;

this was quite beyond the scope of the present work. It was hoped however that this range of species would exhibit permeability characteristics which would be correlated in a general way with the known treatability of the species, and which could be explained by a detailed examination of the structure of the samples themselves.

#### e) Results of Permeability Tests.

Tests were made on the three grain directions for each of the eight mecies, and at least 2 - 3 replicates were used for each treatment. In almost every case the replicated treatments gave nearly identical uptakes with time. The only times when there was a marked divergence in the results was on rare occasions when penetration had taken place between the seal and the wood or through an area that had not been adequately sealed. When this occurred further replicates were treated until it was certain that the results were reliable. The treatment scheduled used in each case was as follows:-

- i. Initial vacuum 15 minutes at 26" Hg.
- ii. Filling under vacuum. Filling took approximately \$\frac{1}{2}\$ minute, and the system was held under vacuum for a further period of about \$\frac{1}{2}\$ - \$\frac{1}{2}\$ minute to stabilize and to zero the phototransistors.
- iii. Venting to atmosphere 4 minute

- iv. Pressurizing. The rate was adjusted so that the total time from vacuum to 100 p.s.i. was 1 min.
  - v. Pressure period. All treatments were terminated after 1 hour if saturation had not been achieved by that time.

Results are shown graphically in Figs. 3, 4, and 5.

#### Longitudinal Penetration

As expected, penetration in this direction was very rapid and with each of the 8 species virtual saturation was reached in under 3 minutes. (Fig 3.). Acathie, Podocarpus, Pinus nigra and Pinus radiata are shown to reach saturation slightly faster than the other species but this may not be significant and it would be necessary to use longer specimens to establish the validity of this observation.

#### Tangential Penetration

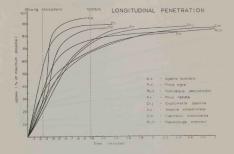
Penetration was very much slower in this grain direction (Fig. 4.) and only with <u>Pinus nigra</u> was saturation achieved within the hour. It would be expected that tangential penetration would follow the same pattern as longitudinal if the accepted pattern of penetration (i.e. from tracheid to tracheid via the bordered pits) is correct. The results do not conform to this pattern; nor are they in line with the order of treatability as shown in Table 1.

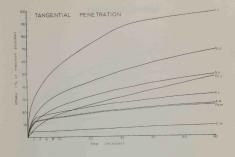
#### Radial Penetration

Uptake rates in the radial direction were much more variable than in either of the other two grain directions (Fig. 5) and results fall into three distinct groups

- A Wirtual saturation reached in under five minutes <u>Pinus</u> radiata and <u>Pinus nigra</u>.
- B Thirty percent saturation reached in under ten minutes 
  <u>Agathis australis</u> and <u>Podocarpus dacrydioides</u>.
- C Thirty percent saturation not reached in one hour.
  Sequoia sempervirens, Pseudotsuga mengiesii,
  Cryptomeria japonica, and Cupressus macrocarpa.

The most striking aspect of the radial penetration results is the close agreement between these and the treatability ratings in Table 1. Group A contains the only two species which are rated as very easy to treat. Group B contains the two indigenous New Zealand species A australia and P.dacrydioides, the former being the only softwood species (except Pinus species) that is approved by the New Zealand T.P.A. for treatment as poles, posts etc. with waterborne preservatives, and the latter being the species which was removed from the approved list only in 1966 because of copper screening. Group C contains the other four species and these have all been subjected to commercial treatment trials but not approved because of poor or variable treatment results.





Pig. 4

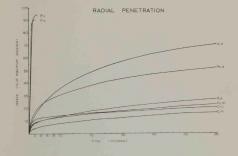


Fig. 5

## Discussion of Results.

Table II shows the relative amounts and sizes of some of the component tissues which might affect penetration in the various grain directions. Agathis australis and Podocarpus dacrydioides are known to have the same type of bordered pit structure (i.e. with no well defined or thickened torus) and the length to diameter ratio of the tracheids of these two species may account (at least partially) for the relative differences in longitudinal and tangential penetration. The large size of Sequoia tracheids, both length and diameter, may assist longitudinal and tangential penetration in this species, but from Table II it would be expected that Cryptomeria would be much less permeable, particularly in the tangential direction. The results do not support this and obviously some other factor such as the percentage of pits aspirated, or resistance of the bordered pit membranes to rupturing under pressure must be important. Pinus nigra has tracheids of a large average diameter and this could contribute to its greater tangential permeability, particulary as the density and percentage latewood are both high, indicating many thick-walled cells probably with unaspirated bordered pits. Furthermore, P.nigra has very high radial permeability and as pathways in different grain directions have been shown to be inter-connecting in some Pinus species by Sargent (1960) and in the film

made by Hicksons Timber Impregnation Co., this may have some influence on penetration in both the longitudinal and tangential directions. The relatively poor permeability of <u>Pinus radiata</u> in the tangential direction was unexpected, although the small average tracheid diameter would tend to mitigate against high permeability in this direction. <u>Pseudotsuga menzicesii</u> and <u>Cupressus macrocarps</u> both show some correlation between tracheid size and uptake rates in the tangential direction.

The most interesting results were those obtained for radial penetration, and there is no correlation at all between these and any of the data in Table II. Nor is there any correlation between radial uptake and the presence or absence of horizontal resin canals as has been postulated by some workers. The two <u>Pinus</u> species both possess horizontal resin canals but <u>Agathis</u> and <u>Podocarpus</u> do not. <u>Pseudotsuça</u> on the other hand does have resin canals. The only feature listed in Table I which is common to the four most radially permeable species, and not the others, is the possession of ray parenchyma with thin horizontal walls.

	Order			Re	Resin Canals Ray & Epithelial Cells Trachei				,	13	Ray Parenchyma														The state of				
	Coniferales	Ac	Rays								Horizontal Tangential Walls				1000		Crossfield Pits								10000				
7333	1					Thick	2				-		This	-1-	-	-	-	ing	14 1		Type				e				
Species	Family	length (in cells)	(in cells) width (in cells)	Thin			7-12 cells	ic nt nt	17.	Thin & smooth (less then 1.5µ)	peq	smooth	Thin & smooth	Bead-like	Dentste	number per tracheid crossing	Diameter (microns)	Arauceroid	Podocerpoid	Decrydioid	Cupressoid	Piceoid	Texodicid Glyptostroboid Pincid Benestriform	Treatability reting / = ersy x = difficult					
Agathis australis	Araucare- aceae	8 <b>-1</b> 0 (2 <b>-</b> 20)	1 (2)	-	-	-	-	-	-	200		+		-	+	-	-	3-6-10	6-11	+	-	-	-	-	-	-	-	-	11
Podocarpus dacrydioides	Podocarp- aceae	1-50	1 (2)	3	-	-	-	-	E			+	×	×	+	-	-	1-2	5-8	-	+	+	-	-	-	-	-		1-11
Cupressus macrocarpa	Cupress- aceae	1-12 (30)	1 - 2	-	-	-	-	(14)	-		-	×	+	( <b>x</b> )	-	+	(m)	1-2-(6)	4-9	-	-	-	+	-	-	-	-	-	**- <del>X</del> -√
Cryptomeria japonica	Taxodiae- eae	1-8	1 (2)	-	-	-	-	-	-	-	-	-	( <b>*</b> )	+	+	-	-	1-2-(4)	6-8	100	-	-	+	-	+	ж	-	-	XX
Sequoia sempervirens	Teatre Tea	14-20 (1-35)	1 (2)	-	-	-	-	×	×	-	-	-	×	×	+	×	-	1-8	6-12	-	-	-	-	-	+	+	Ī	1	ж
Pinus radiata	Pinaceae	5-7 (1-10)	1-2 (R)	+	-	-	-	+	+	+		+	×	-	+	×	-	1-4	8-12	1	-	-	-	-	ж	-	+	-	JJJ
Pinus nigra	0.300	1-10	1 (R)	+	-	-	-	+	-	+	-	+	-	-	+	-	-	1-2	10-20	-	-		-	-	-	-	-	+	111
Pseudotsuga menziesii	11	1 <b>-</b> 5 (16)	1 (R)	-	+	×	-	+	-	+	-		+	-	7		+	2-6	4-6	-	-	1	-	+	-	-	-	-	ж
Picea sitchensis	11	10-12	1 (R)	-	+	-	-	+	-	+	-		+	-	-	-	+	2-6	3-8	100	-	-		+	×	-	-		ЖK

<sup>=</sup> Present # = occasional

<sup>- =</sup> Absent

<sup>(\*) =</sup> uncertain structures

Table II Some physical characteristics of the wood used in

Uptake tests

	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
(4) heroteken	ic ty	per	age	entage tissue	Trac		chei	of tra- is to be trated
Species	Basic	Rings	Percentage latewood	Percentage ray tissue	length (mm)	diameter (µ)	longi.	tang.
Pinus nigra	0.432	7.6	43	4.7	4.8	41	4.2	490
Pinus radiata	0.419	6.0	38	4.7	5.3	30	3.8	670
Pseudotsuga menziesii	0.400	6.3	48	4.4	4.7	35	4.3	570
Sequoia sempervirens	0.256	3.6	15	6.4	5.8	45	3.4	450
Cryptomeria japonica	0.296	13.0	26	3.2	4.8	27	4.2	750
Cupressus macrocarpa	0.338	4.6	(20)	5.7	4.0	35	5.0	570
Podocarpus dacrydioides	0.358	13.0	(15)	8.6	4.5	46	4.4	440
Agathis australis	0.440	13.0	22	9.8	6.5	34	3.1	580

- Basic density This figure is the mean of the basic densities calculated for every third block along the sticks.
- (2) Rings per inch- From a ring count on the 2 cm. cube blocks.

- (3) Percentage latewood. A very approximate figure obtained by measurement of the denser zones on a sanded crosssection. Figures in brackets represent very indefinite latewood zones.
- (4) Fercentage ray tissues. Taken from low power photomicrographs of tangential sections. Ray tissue including parenchyma, tracheids and resin canals were cut out and weighed.
- (5) & (7) Tracheid lengths. From measurements of tracheids isolated by macerating with tricthylene glycol and phenol sulphonic acid (Burkart 1966). The phenol sulphonic acid was increased from 0.5% to 3.5% to reduce macerating times to 10 minutes.
- (6) & (8) Tracheid widths. From counting the number of tracheids over one annual ring (or 5 mm. whichever was the greater) in a radial row in a microscopic crosssection.

#### PART 2

## Pathways of Radial Penetration

### (a) Discussion of methods for tracing pathways

The most desirable method for tracing the pathways of liquid penetration would be some dynamic system whereby the actual flow could be followed visually. This method was used by Sargent (1960), and also by Hickson's Timber Impregnation Co. in following the course of longitudinal flow through Pinus wood. In each case a radial face of a block of wood was machined to provide a smooth surface which was coated with a film of clear plastic. The other faces were then sealed so that penetration had to take place through one end and the liquids (creosote in Sargent's experiments and dved aqueous solutions in Hickson's) were forced through under a low hydrostatic pressure. The movement of the liquid could then be observed through the plastic film with the aid of a microscope, and if desired it could be recorded on cine film. This technique was tried in the course of this work and the results quoted by Sargent and shown in Hickson's film, were reproduced with longitudinal flow in pine wood. It demonstrated clearly that the movement was predominantly along the axial tracheids in the direction of the applied pressure, but in many instances the flow was arrested and then moved laterally through the rays, apparently through the ray parenchyma cells. From the rays the flow would then

return to axial flow through ther tracheids although occasionally in a direction opposite to that of the applied pressure.

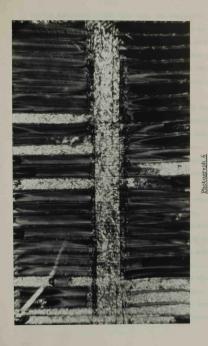
Unfortunately, this technique did not give satisfactory results when used with less permeable wood species, or when the flow was other than in the longitudinal direction. Under these conditions the seal was inadequate for the required pressures.

Smith and Redding (1964) solved this problem by constructing a small pressure cylinder in which the normal door was replaced by a transparent viewing door. Specimens were fixed to the inside of this door with clear resin, and the penetration of preservative could be observed while the whole system was under pressure. Low power magnification could be used, but the thickness of the double perspex door precluded the use of magnifications greater than about X 30. More detailed study could be made when the specimen was removed from the cylinder after various degrees of penetration had been achieved however, and this is undoubtedly a very useful technique when such equipment is available.

The other alternative is to impregnate the wood with a solution that can be detected in <u>situ</u> when the sample is subsequently sectioned. Provided the impregnation has been only partial, an examination of the sections may demonstrate the route the solution was taking. This approach was attempted first by I. W. Eailey (1913) who used a suspension of carbon

black. This work showed that longitudinal penetration followed the natural pathways which exist in the living trees, i.e. along the tracheids and through the bordered pits.

Dyed aqueous solutions have been used by several workers in the field including Buro and Buro (1959). Kishima and Hayashi (1960), Wardrop and Davies (1961), Hayashi and Kishima (1965) and Liese and Bauch (1967). Buro and Buro used a blue filtering dye to trace the pathways of radial penetration in vacuum treated pine wood and found the dye to be located in the ray parenchyma cells when the wood was sectioned and examined under a microscope. They claimed however, that penetration had been through the ray trachieds and that the dye had moved from these cells to the parenchyma cells only after impregnation had been effected. The basis for this assertion was their finding that when blocks of wood were treated with hot paraffin wax, subsequent examination showed this to be located only in the tracheids, and that when the ray tracheids were blocked in this manner no penetration of the ray parenchyma cells was possible if the blocks were re-treated with dyed aqueous solutions. When this experiment was repeated during the course of the current work however. the results quoted by Buro and Buro were not observed and photograph 6 (Pinus radiata radially impregnated with paraffin wax and section examined between crossed polars) shows clearly that the wax has penetrated the ray parenchyma.



Plans radiate RLS. Examined between crossed polars to show the location of paraffla wax in may presubly an oalis.  $\chi$ 

Wardrop and Davies (1961) used dyes, and also double treatments so that the two solutions would react and form insoluble precipitates which would not be removed or altered during specimen preparation, and which could be detected under the microscope. Treatments used were copper sulphate followed by dithio-oximide, ferric chloride followed by potassium ferrocyanamide, and Coppick and Fowler reagents.

The same approach was used by P. J. Bailey (1965) who treated with silver nitrate followed by hydrazine hydrate. This treatment had the advantage that the precipitate was visible under both the optical and electron microscopes.

Double treatments are not so suitable however when impregnation is to be halted at a particular point so that the course of penetration at that time can be studied.

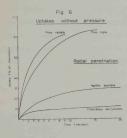
Kishima and Hayashi (1960) examined several dye solutions including red ink, blue-black ink, acid fuchsin, eosin, safranin, methylene blue, and malachite green, and concluded that the most satisfactory was a 1% solution of acid fuchsin. This dye showed no tendency to be filtered out by the wood, and did not creep over unpenetrated areas after treatment.

With aqueous dyes the problem is to cut sections thin enough for the microscopical examination, and mount them, without using any liquids which might re-dissolve the dye.

This was overcome by using a modification of a dry sectioning

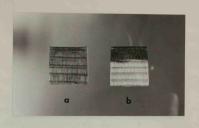
technique suggested by Fleischer (1950). The technique is to stick cellotape on to a microtomed surface and then cut a section with the cellotape attached. With this method it was found possible to cut quite good sections down to about 6p thickness without them breaking or curling. Fleischer examined his sections while they were still attached to the cellotape but this results in a considerable loss of clarity. It was found possible to remove the cellotage by first drving the sections completely over a desiccating agent, and then immersing them in xylene. Xylene does not dissolve fuchsin dyes at all but after a few minutes the sections float free from the cellotage and can be transferred to a slide and mounted in Canada balsam. The optimum thickness for radial a tangential sections prepared in this manner was found to be about 15p and 25 p respectively.

To determine the pathways of penetration it was essential that only partial saturation of the test blocks should be obtained. Preliminary tests indicated that an uptake in the vicinity of 20% of the maximum would be the most generally satisfactory. From Fig. 5. it can be seen that with the permeable species this would be achieved with only a very short pressure period which would be difficult to control with any precision. Trials were therefore carried out with the two pine species, and also Agathis and Podocarpus, to see if a similar, but slower, uptake could be obtained by venting



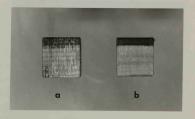
to atmosphere after vacuum but not applying any positive pressure. The results of this test, Fig. 6, showed that both the pine species could be treated quite adequately by this method and that the absence of applied pressure, and the slightly longer time to reach 20% saturation, would make it much easier to stop the treatment at the desired point. As the acid fuchsin dye solution absorbed more light in the permeability apparatus, the output to the recorder was reduced considerably, and it was difficult to calibrate the recorder so that the point corresponding to 20% uptake could be marked accurately. In an endeavour to overcome this, basic fuchsin decolourised with sodium metabisulphite was tried and the first full series of dve treatments used this. The dye markings were not as positive or clear as with acid fuchsin however. As the uptake times were the same as those established earlier with distilled water, a further series was treated with a 1% acid fuchsin solution and in this series the uptake period was controlled largely by the times indicated in Figs. 5 and 6. The actual uptakes were checked by weighing when the blocks were removed and the results are shown in Table 3. (overleaf).

The majority of the photographs used in the foregoing section were taken with samples and sections prepared from the acid fuchsin treatments, but a few are included from the first series using basic fuchsin. There was no discernible difference between the two dyes with either total uptake or penetration pathways.



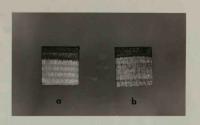
Photograph 7

Radial penetration of dye solution into blocks of (a) Pinus radiata, (b) Pinus nigra.



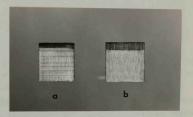
Photograph 8

Radial penetration of dye solution into blocks of (a) Agathis australis, (b) Podocarpus dacrydioides.



Photograph 9

Radial penetration of dye solution into blocks of (a) Pseudotsuga menziesii, (b) Sequoia sempervirens.



Photograph 10

Radial penetration of dye solution into blocks of (a) <u>Cryptomeria</u> japonica (b) <u>Cupressus macrocarpa</u>.

Table 3- Treatment details and radial uptakes with dye solutions

Species	Pressure	Antici- pated time	Actual time	Uptake as % maximum
Pinus nigra	Atmospheric	0.6 mins.	1.Omins.	23
Pinus radiata	Atmospheric	0.5 mins.	0.5mins.	19
Agathis australis	100 p.s.i.	4.0 mins.	5 mins.	22
Podocarpus dacrydioides	100 p.s.i.	3.0 mins.	4 mins.	13
Pseudotsuga menziesii	100 p.s.i.	42.0 mins:	35 mins.	26
Sequoia sempervirens	100 p.s.i.	28.0 mins.	35 mins.	20
Cryptomeria japonica	100 p.s.i.	52.0 mins.	45 mins.	24
Cupressus macrocarpa	100 p.s.i.	60 <sup>+</sup> mins.	45 mins.	15
at the Delivery of Securior	sta terrore	the two past	pri bert the	Land Street

# (b) Results of Radial Penetration. - Trials with Dye Solutions.

Photographs nos. 7 - 10 inclusive show the gross penetration of acid fuchsin into the blocks when penetration was restricted to the radial direction. The most striking result of this trial is the complete penetration of the <u>Pinus radiata</u> block, photograph 7(a). This was achieved with an uptake corresponding to only 19% of the maximum possible uptake. A high proportion of the rays are penetrated even at the face most distant from the exposed face, but only the outer millimeter shows complete and uniform penetration of both rays and tracheids. The precentage of rays and tracheids

which show dye penetration falls off gradually as the distance from the exposed face increases but it is notable that the outermost layers of trachedds in each growth ring show penetration. Two axial resin canals which have been exposed on the sectioned surface also show dye penetration.

In photograph 7(b) the pattern of penetration in Gorsican pine appears quite different although the pressures, times, and uptakes were similar. There is obviously some penetration of the rays from the advance face but this does not exceed 1-2mm. As with Pinus radiata, the latewood bands appear to become penetrated more easily but the difference is less pronounced, and the outer 5 - 6mm. show complete penetration of all tissues.

Photograph 8(a), (Agathis australis), shows a penetration pattern somewhat intermediate between the two pines, but the rate was considerably slower, and higher pressures were used.

Penetration appears uniform to a depth of four millimeters, and as this represents 20% of the total depth, the uptake in this region must have been virtually complete. The extra 2% would be accounted for in the advance penetration in the rays.

In photograph 8(b), (<u>Podocarpus dacrydicides</u>), there appears to be very little ray penetration in advance of the heavily penetrated area, although examination with a lens shows that there is a definite penetration to about 0.4mm. in almost all the rays. The apparently uniformly penetrated area extends to a distance of about 3.8mm. from the exposed face and this represents a depth of penetration equivalent to

19% of the total penetration. As the uptake was only 19% of the maximum possible, and the depth of penetration was fairly uniform throughout the block, this indicates that complete saturation was not obtained in the visually penetrated area at this stage.

In photograph 9(a), it appears that the Douglas fir block has been penetrated completely to a depth of 6 mm. and that this zone is preceded by a zone of ray penetration extending a further 1 - 1.5mm. Six millimeters represents 30% of maximum penetration and this figure is only slightly greater than the uptake precentage. As the penetration was particularly even in this sample however, this difference could mean that some of the tracheids in the penetrated area were not completely filled with solution.

Photograph 9(b), Sequoia sempervirens shows very little ray penetration even when magnified, and the depth of penetration and uptake figure indicate that the wood is virtually saturated right to the extremity of the penetration. A slightly greater depth of penetration at one edge of the block suggests that some solution may have forced a path between the wood and the seal at this point but this is unlikely to have altered the general picture very much.

In photograph 10(a) <u>Cryptomeria japonica</u> shows a pattern of penetration very similar to that of the preceding redwood. The depth of penetration indicates complete saturation of the

penetrated area but this is slightly uneven and there is slight evidence of penetration between the wood and the seal at one point.

Photograph 10(b) <u>Cupressus macrocarpa</u> shows very even penetration but the depth indicates incomplete saturation; 20% penetration as against only 15% uptake. The margin of the penetrated area shows a faint and indistinct advance penetration.

# Microscopical study of dye penetration.

## Pinus radiata.

The penetration of dye along the ray parenchyma can be seen clearly in the radial longitudinal sections of photographs 11 and 12. Photograph 11 shows a small ray with two rows of parenchyma cells and two rows of ray tracheids, one on either side of the parenchyma. In this section the dye can be seen passing through what appear to be parenchyma cell end walls without any obvious impedence. One of the four parenchyma cells shown has thickened walls but this also appears to be penetrated as readily as the thin walled cells surrounding it. It is notable that there is no trace of dye in the ray tracheids in this area, nor is there any evidence of movement of the liquid into the earlywood axial tracheids through which the ray is passing at this point. Photograph 12 shows a ray consisting of only one parenchyma and several ray tracheid cells immediately beyond a heavily penetrated latewood area. The lower permeability of the ray tracheids is demonstrated very clearly in this photograph. Photograph 15 is a tangential longitudinal section cut from near the face most distant from the exposed face. Fenetration of the dye (in this case basic fuchsin) has taken place in the majority of the mys but there has been little transverse movement into the axial tracheids.

### Pinus nigra.

In photographs 14 and 15 radial penetration along the ray parenchyma but not the ray tracheids is again obvious. Photograph 14 shows the limit of penetration in a ray and a feature of note here is the uniformity of penetration of the solution in the individual ray cells. Photograph 15 shows a ray at a point a little further removed from the limit of penetration and in this the movement of solution from the ray parenchyma to the adjacent axial tracheids is demonstrated. At the limit of penetration, a tangential longitudinal section (photograph 16 - basic fuchsin) shows that although not all the rays have been penetrated at this stage, there is still a lateral movement from a ray to an adjacent axial tracheid.

### Agathis australis

Photograph 17 is a radial longitudinal section showing a penetrated ray in the region of a latewood-earlywood boundary. The ray tissue (sarenchyma only in this species) appears to be very disordered, with no distinct end walls to the individual cells. The dye solution has apparently penetrated the ray
quite readily but the penetration from ray to axial tracheids
is irregular and there is no indication that movement is any
easier into the latewood than the earlywood.

### Podocarpus dacrydioides

Photographs 18 and 19 are radial longitudinal sections at the limit of penetration showing that in this species, advance penetration of the rays is reduced considerably. In photograph 18 advance penetration covers only eight tracheids and in photograph 19 the number covered is eleven. These photographs also demonstrate two other features which were not found in the pines or in kauri. The first is the apparent penetration along the intercellular spaces between the ray parenchyma cells. Photograph 20, a tangential longitudinal section cut across the limit of penetration, shows that the penetration through intercellular spaces is not very pronounced however, and that penetration also takes place through the lumena of the cells. The second feature is the apparent preferential penetration of axial parenchyma over axial tracheids. This is seen in both the radial longitudinal sections and also in the low magnification tangential section - photograph 21.

# Pseudotsuga menziesii.

Douglas fir is generally considered to have an intermediate permeability rating in the family <u>Pinaceae</u>, being more readily treated than the spruces, firs and larches, although considerably more refractory than the pines. This was attributed by Liese and Bauch (1967) to its more abundant and more easily penetrated ray tracheids.

Photograph 22 certainly indicates that in some cases penetration can take place along a ray tracheid and from this can move into both adjacent axial tracheids and also (either directly or via the axial tracheid) into adjoining ray parenchyma. Photograph 23 shows that in some instances the opposite is apparently true and here penetration along the ray parenchyma is well in advance of ray tracheid penetration. In some cases penetration seems to move uniformly along the ray parenchyma cells while in others it appears to move along the ray parenchyma intercellular spaces, and then back into a parenchyma lumen further along the ray. The resinous contents which Sargent (1960) considered as a major barrier to ray parenchyma penetration in this species, particularly when the end walls were coated, can be seen in these photographs but there is not an excessive amount present.

## Cryptomeria japonica.

In this species, advance penetration along the rays is still further reduced and covers only three or four tracheids as shown in photographs 24 and 25. At the limit of penetration the dye appears to be located within the parenchyma cell walls or in the intercellular spaces so a series of tangential longitudinal sections were cut to resolve this question. In photograph 26 ray penetration is advance of the area of heavily treated tracheids, but the parenchyma walls are solidly stained. Further slong the rays the amount of staining becomes less, (photograph 27), and at the extreme limit of penetration photograph 28 illustrates conclusively that the initial ray penetration is through the intercellular spaces between the ray parenchyma cells and the tracheid walls.

#### Sequoia sempervirems.

The pattern of radial penetration in Sequoia is very similar to that in Cryptomeria but the amount of advance penetration is still less, and the penetration through intercellular spaces is even more pronounced in the radial sections - see photograph 29. In photograph 30 (a tangential longitudinal section cut near the limit of penetration) two rays are shown. In one the penetration has just commenced through the intercellular spaces while in the other it is fairly advanced and the dye solution appears to have moved from it into the adjacent axial cell. Transverse movement from this cell to adjacent tracheids appears to be impeded by the pit membranes. In photograph 31 the bordered pits appear to be unaspirated and dye solution is shown moving from one tracheid to its neighbour. It is impossible to tell from this section whether the solution has penetrated the tracheid from the heavily stained ray, or from the tracheid on the other side,

or from another ray not in the field of view. The other ray shown towards the bottom of the photograph is in an early stage of intercellular space penetration and its adjacent axial cell must have been penetrated from some other source. In photograph 32 the ray cell, which is only partially penetrated, lies adjacent to a heavily penetrated axial cell. From the nature of the stained contents of this cell it is presumed that it, and the similar cell in photograph 30 are axial parenchyma cells. Dye has penetrated the openings of the crossfield pits (if this term is applicable to pits between ray and axial parenchyma cells) but appears to be prevented from entering the lumena of the ray cells by intact membranes.

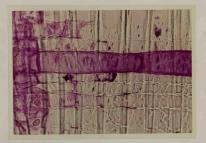
### Cupressus macrocarpa

Penetration of the intercellular spaces is still more pronounced in <u>Cupressus</u> and there appears to be practically no advance penetration except along these passages, see photographs 35 and 34. In photograph 34, and in the two tangential sections (photographs 35 and 36) which were cut near the limit of penetration, it is seen that solution not only penetrated the ray intercellular spaces, but also moved out into the intercellular spaces between the axial tracheids.



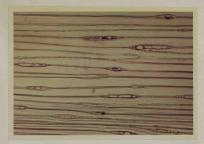
Photograph 11

Pinus radiata RLS. Acid fuchsin dye in ray parenchyma X 400



Photograph 12

Pinus radiata RLS. Acid fuchsin dye penetrating ray parenchyma cells but not ray tracheids. X 440



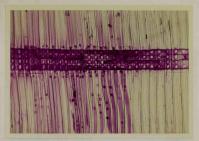
Photograph 13

Pinus radiata TLS. Basic fuchsin dye in the region of extreme penetration. X 150



photograph 14

Pinus nigra RIS. Limit of penetration of acid fuchsin solution in a ray. X 200



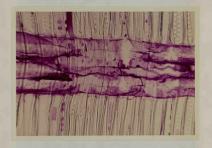
Photograph 15

Pinus nigra RLS. Acid fuchsin solution penetrating axial tracheids from ray parenchyma. X 140



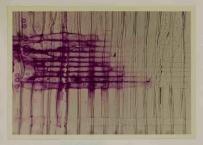
Photograph 16

Pinus nigra TIS. Limit of radial penetration of basic fuchsin solution. X 200



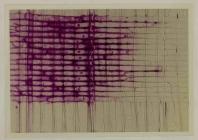
Photograph 17

 $\underline{\mathtt{Agathis}}$  australis RLS. Acid fuchsin penetration through ray parenchyma. X 220



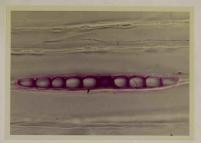
Photograph 18

Podocarpus dacrydioides RLS. Radial penetration of acid fuchsin solution. X220



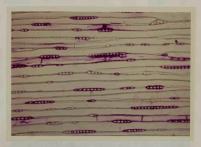
Photograph 19

Podocarpus dacrydioides RIS. Radial penetration of acid fuchein solution. X 220



Photograph 20

Podocarpus dacryiodies TIS. Uniform penetration of acid fuchsin in a ray at the limit of penetration. X 700



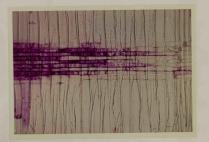
Photograph 21

Podocarpus dacrydicides TIS. Acid fuchsin in rays and axial parenchyma near the limit of penetration. X 140

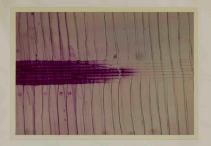


Photograph 22

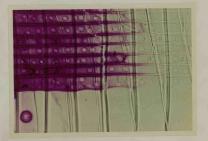
Pseudotsuga menziesii RLS. Radial penetration of acid fuchsin, X 11.0



Pseudotsuga menziesii RIS. Radial penetration of acid fuchsin. X 140



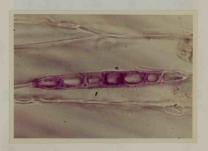
<u>Photograph 24</u> <u>Cryptomeria japonica</u> RLS. Limit of radial penetration X 14.0



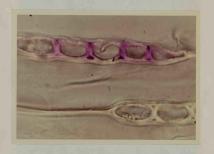
Photograph 25
Cryptomeria japonica RLS. Fimit of radial penetration X 440



Photograph 26
Cryptomeria japonica TLS. Section through tay near the limit of penetration. X 900

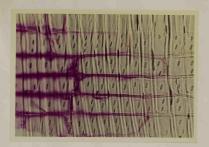


<u>Photograph 27</u> <u>Cryptomeria japonica</u> TLS. Similar to Photograph 26, but further removed from heavily treated area. X 900



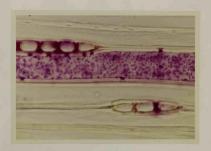
Photograph 28

Cryptomeria japonica TLS. Extreme limit of penetration. Dye located mainly in intercellular spaces. X 1450

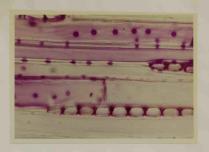


Fhotograph 29
Sequoia sempervirens RLS. Limit of radial penetration.

X 4440

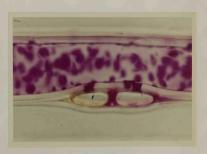


Photograph 30
Sequoia sempervirens TLS. Section near the limit of radial penetration. X 450.



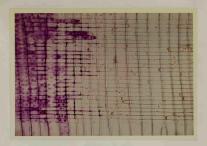
Photograph 31

Sequoia sempervirens TLS. Redistribution of dye solution from ray cells. X 360

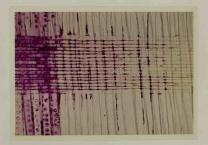


Photograph 32

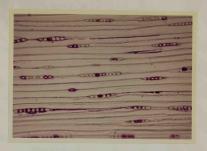
Sequoia sempervirens TLS. Penetration of an axial cell in advance of an adjacent ray. X 900



Fhotograph 33.
Cupressus macrocarpa RLS. Limit of radial penetration X 140

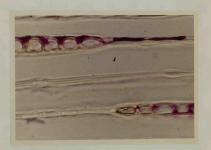


Photograph 34
Cupressus macrocarpa RLS. Limit of penetration showing dye in intercellular spaces. X 140



Photograph 35

Cupressus macrocarpa TLS. Section near the limit of radial penetration. X 140



# Photograph 36

<u>Cupressus macrocarpa</u> TLS. Section at the limit of penetration showing dye in the intercellular spaces of ray parenchyma cells and axial tracheids.

### PART 3

Factors affecting penetration of aqueous solutions in the Radial Direction

The following methods and techniques were used to examine
the structures which might have some bearing on the radial
penetration of liquids into the wood specimens used in the
previous permeability and penetration studies.

## (a) Optical microscopy

As it was essential to preserve the wood structure with as little damage as possible, no preparation techniques such as boiling in water or other liquids were used. Blocks were made with a cutting face of no more than 4mm square and these were soaked in distilled water until more or less saturated. Sections were cut with a sledge microtome, stained with safranin, and either mounted in 20% glycerol or dehydrated through ethanol and mounted in canada balsam. To protect the ray structure of some species the blocks were embedded before sectioning. For ordinary sections of 12-25u polyethylene glycol (PEG) was used; blocks were saturated. transferred through a series of PEG solutions to pure PEG and then left to harden. After microtoming, the PEG was dissolved out and the sections were stained and mounted as usual. Thin sections of 1-4m were cut on an ultramicrotome or pyramitome after embedding in resins as described for

electron microscopy. For this material, staining was usually carried out on sections of about 100  $\mu$  thickness before they were embedded in the resin, and was normally a one hour treatment with 0.% Klin 0<sub>4</sub>. Sections embedded in methacrylate or methacrylate mixtures were removed from the resin with xylene and mounted in Canada balsam for viewing. With epoxy resins, the technique of Mayer, Hampton and Posario (1961) was used to remove the resin in some cases, but more generally the sections were mounted in lens immersion oil and viewed with the resin still intact.

## (b) Interference microscopy

The development and use of this technique for examining the crossfield pits has been published (McQuire 1968) and only a brief account will be given here. Samples were soaked in distilled water as for (a) and radial longitudinal sections of 6 u were cut with a sledge microtome. Sections were then trimmed with a sharp scalpel (under a binocular microscope) to expose suitable rays on the edges of the sections. This was necessary as the interference microscope used in the work was of the laterally sheared beam type, and observations free from distortion could be made only on the edges of specimens. Sections were mounted in 20% glycerol (refractive index 1.3683) and with a 40% objective it was quick and simple to determine whether the crossfield pit membranes were intact or ruptured. Observations were made most easily with white light from a tungsten lamp; here the presence of membranes

caused interference colours which contrasted strikingly with the background colour. For photography in black and white, best results were obtained with a mercury vapour lamp and monochromatic filter; either the pits or the background were extinguished to give maximum contrast. The only occasion upon which this technique can give apparently false results is when the membrane is of such a thickness that, with a particular difference in refractive index between membrane and mounting medium, a path difference of almost exactly one whole wavelength is obtained. This happened with Douglas fir as seen in Photographs 65 and 66 . Here the section has been mounted successively in 20% glycerol ( $\gamma = 1.3683$ ) and liquid paraffin ( $\eta = 1.47$ ). Double mounting in this manner makes it possible to measure both the membrane thickness and its refractive index and with the Douglas fir a thickness of 2.1 µ and refractive index of 1.60 was obtained.

Cutting sections of 6µ from un-embedded wood with a sledge microtome must impose a considerable strain on relatively thin tissues such as the crossfield pit membranes and it cannot be stated definitely that ruptured membranes are not preparation artefacts. However, it is still possible to draw comparisons between species and samples. With some species, e.g.Cupressus macrocarpa and Sequoia sempervirens, the crossfield pits were never (or exceedingly rarely) found open, and this indicates that the membranes are strong enough to resist any such preparation forces. With the pine species the crossfield pits

in the earlywood were often open, indicating that the membranes were either broken previously or were easily ruptured during preparation because of their large diameter and relative thinness. The universality of open pits in pine latewood indicates that they are very easily ruptured, or more probably, were already broken, possibly by the stresses set up as the thick-walled latewood tracheids dried out when the wood was seasoned originally.

## (c) Electron microscopy using replicas

(i) Two stage replicas - The method developed and most commonly used in the Astbury Department of Biophysics is the cellulose acetate film technique. A piece of cellulose acetate sheet moistened with acetone is pressed on to a clean split or microtomed wood surface and allowed to set. It is then peeled off, shadowed with heavy metal (at an angle of 20 - 30°), and coated with a film of evaporated carbon about 200% thick. The carbon surface is scored with a razor blade into squares about the size of an electron microscope grid, and the whole replica is transferred to an acetone bath to dissolve the cellulose acetate. The carbon replicas float free and are gathered on grids. Some of the replicas shown in the accompanying photographs were prepared in this manner.

Dr. Seehan (personal communication) of the Forest Research Institute, Reinbek, uses methyl methacrylate for forming the first replica by brushing partially polymerised resin monomer on to the surface and then allowing polymerisation to complete at room temperature. After shadowing with metal and coating with carbon, the replica is then backed with a layer of fairly viscous gelatin solution, and a strip of paper is attached to one edge with the gelatin . The gelatin is allowed to dry completely (over night) and then the methacrylate is dissolved be suspending the replica in three successive baths of solvent, (over night in the first bath). The gelatin backed carbon replica is then cut into small squares with scissors, (particular areas can be selected easily at this stage) and the gelatin is removed by floating the replicas, carbon side up, on a bath of water at 50°C. The carbon replicas continue to float on the surface and to ensure complete removal of the gelatin, are transferred with a glass rod to two further water baths at temperatures of 55°C and 60°C. After about 15 minutes in each water bath, the replicas are picked up from the surface on a grid. The Agathis bordered pit replica shown in photograph 55 was prepared in this manner. Gelatin backing, and the subsequent procedures

described above, are just as applicable to cellulose

acetate as methacrylate replicas, and the majority of the cellulose acetate replicas shown in the photographs were prepared using this hybrid technique. The advantage over the non-backed cellulose acetate replica technique is that the final cleared replica is much easier to pick up from a water surface than from an acetone bath and fewer are lost.

The main disadvantage of any of the two-stage replica techniques is that they are suitable only for solid surfaces. Where there are holes in the surface (e.g. perforated crossfield pits, or the spaces between the fibrils of the margo of bordered pit membranes), the liquid plastic will penetrate and solidify. When the film is peeled off these protrusions remain as elongated whiskers which interfere with uniform shadowing, tend to weaken the replica, and make interpretation of the structures difficult.

(ii) <u>Direct Replicas</u> - With this technique (in which the wood surface is shadowed and coated directly) small holes and spaces do not become coated, and thus show their true identity in the replica. In the technique described by Côté, Koran and Day (1964) the replica is backed with high melting point paraffin wax after shadowing and coating, and the wood is then removed by successive treatments with 72% sulphuric acid and macerating mixture (equal parts of

10% chromic acid and nitric acid). The replica is then mounted on a grid and the wax dissolved away by condensed benzene vapour in a solvent boiler. Attempts to use this technique resulted in successive failures mainly because the molten wax penetrated openings in the carbon film and around its edges, thus coating the wood and protecting it from the acids. On Côte's recommendation polystyrene was tried instead of wax and this resulted in some improvement.

The most successful direct replica technique used was that described by Puritch (1970). In this technique thin slices of wood (about 100 u thick) are shadowed and coated, and then placed, wood side down, on a 2% solution of cellulase enzyme for two days. They are then transferred to the chromic/nitric acid macerating mixture for 2 days, washed in water, and picked up on grids. Theoretically cellulase enzyme will not attack lignified tissues and this is apparently borne out if the replicas are examined under crossed polars after the cellulase treatment. Modifications were therefore tried in which the cellulase was replaced by dilute organic acid, pure water, or was ommitted completely. Although all these modified procedures produced some satisfactory replicas, and the problem of mould growth on the enzyme was overcome, the results were generally inferior and less reproducable than when the cellulase was used.

All the two-stage and direct replica techniques discussed above were used with varying degrees of success on radial wood surfaces, but none was satisfactory for examining either transverse or tangential surfaces. This lack of success was caused by the large surface holes made by the tracheid lumens in the transverse sections, and the ray cell lumens in the tangential sections. One possible means of overcoming this difficulty was to use freeze-etching, but attempts to use this technique were completely unsuccessful. The main reason for this may have been the impossibility of obtaining a clean fracture across frozen lignified wood cells with the quipment available.

All the replicas reproduced in this thesis were shadowed with gold/palladium.

# (d) Electron microscopy using thin sections

As very high magnification and resolution were not required in this study, section thicknesses in the region of 700 - 1000% were generally satisfactory. This still required the use of an ultramicrotome and resin embedding however, and methyl/butyl methacrylate, styrene/butyl methacrylate, epon, and araldite were all tried. The usual procedure adopted was soaking the specimens in distilled water, cutting 100 µ sections on a sledge microtome, fixing/staining in cold 0.3% KMm 0<sub>4</sub>) dehydrating in ethanol or isopropyl sloohol, and embedding in

resin. Where this procedure gave satisfactory contrast, no additional staining procedures were used. When contrast was poor, post staining with uranyl acetate and lead citrate was tried but this did not produce a very noticeable improvement. Although the later sectioning was with a diamond knife, the bulk of the work was done with glass knives and with these a much improved performance was obtained when the knives were made from 80° rhoubi rather than from squares.

As with the optical microscopy specimen preparation, no softening procedures that night have affected the wood microstructure were permitted and this reinforced the inherent difficulty of producing good thin sections of wood. Structural features of each species examined in relation to penetration.

#### Pinus radiata

From the photographs of the penetrated block, and the coloured photomicrographs of dye penetration, it is clear that liquids can pass along the ray parenchyma cells for considerable distances with very little apparent obstruction. The cross-section photograph (photograph 37), and also photograph 11, show parenchyma cell end-walls, but either these must be perforated, or the liquid by-passes them though simple pits or discontinuities in the thin cell walls, into adjacent parenchyma cells. Thin optical sections (2m), cut through the ray cells of epoxy resin embedded material, show that the non-thickened walls are very fragile and would be ruptured easily. Photographs 38 and 39 show the extremely thin walls and absence of birefringence in these cells. Even in the thickened cells, the area in the vicinity of the crossfield pits remains thin, as seen in photograph 39. The electron micrograph of a section cut through a crossfield pit (photograph 40) shows that in this instance the crossfield pit membrane is intact but is very thin and the ray parenchyma wall has partially collapsed and pulled away. In the interference micrographs (photographs 41 and 42) it is seen that some of the earlywood pit membranes, and all the latewood membranes are ruptured. As stated in the section on method.

ruptured membranes may be artefacts but nevertheless, their presence demonstrates that the membranes are weak and easily broken. The speed of liquid uptake in the radial direction, particularly when only a vacuum to atmospheric pressure treatment is used, supports the hypothesis that the crossfield pit membranes in the latewood are open in the dry wood, while in the earlywood some are open and the remainder are easily ruptured.

In Pinus radiata therefore, the pattern of penetration appears to be primarily along the ray parenchyma cells with lateral movement through open crossfield pits into the adjacent latewood tracheids. When these cells are more or less full of solution, the pressure begins to build up and membranes between the ray parenchyma cells and earlywood tracheids rupture allowing penetration of these cells to be effected. Although the permeability of heartwood was not included in this study it is known from commercial experience that the treatability of radiata pine heartwood can be extremely variable. In some cases it can be treated completely (albeit with more difficulty than sapwood), but in other cases penetration can be effected into the rays alone, or not at all. According to Balatinez and Kennedy (1967) the rays in hard pines do not mature completely until heartwood formation has commenced so there may be a structural difference between sapwood and heartwood ray parenchyma cells. Bauch, Liese and

Scholz (1968) show that the torus of the bordered pits of softwoods becomes lignified in the heartwood and it is probable that some similar chemical change takes place in the crossfield pit membranes. Thus the development of heartwood could alter the permeability in three gradual steps.

- The thickened parenchyma cells would have a greater
  resistance to seasoning strains, and fewer crossfield
  pit membranes would be ruptured during drying. Another
  factor influencing this would be the generally slower
  drying and reduced shrinkage of heartwood. At this stage,
  impregnation would still be possible, but it would be
  slower as more of the membranes would have to be
  ruptured.
- As the crossfield pit membranes became lignified, they
  would become more difficult to rupture, until the stage
  was reached where it would be possible to force liquid
  along the rays but not into the adjacent tracheids.
- 5. In the final stage of mature heartwood formation a build up of resin in the rays would preclude all penetration. It should be relatively simple to test this hypothesis with radiata pine heartwood of varying age and from different sites, and this will be done in future work.

## Pinus Nigra

Radial penetration in Corsican pine seems to take exactly the same pathways as in radiata pine but with even less

obstruction between the ray parenchyma cells and the axial tracheids. Radial and tangential longitudinal sections cut from sanwood embedded in polyethylene glycol (photographs 43 and 44) show a considerable degree of disorder in the thinwalled parenchyma cells, with no apparent barriers to movement along the rays. The ray tracheids, however, are much more nearly intact and the encrustion of the bordered pits (as shown in photograph 45) would explain why the ray tracheids are less permeable than the ray parenchyma in this species. The crossfield pits in Corsican pine are of the large fenestriform type, and it would be expected that these would be easily ruptered. Photograph 46 shows the very large pit openings with only fragments of membrane visible in the earlywood region. In photographs 47 and 48 a very characteristic pattern of rupture is evident. in the latewood, and it seems highly probable that this has taken place during the drying of the adjoining thick-walled axial tracheids.

The pattern of radial penetration in <u>Pinus nigra</u> therefore seems to be along the ray parenchyma cells and directly into the axial tracheids of the latewood band. As penetration progresses from the latewood to the earlywood, fewer open crossfield pits are encountered but a slight pressure rise, caused by the liquid having to force its way through a somewhat disordered ray parenchyma system, is sufficient to rupture the thin membranes covering the very large crossfield pit apertures.

In this manner, complete saturation of all the tissues would be achieved with only a relatively small amount of advance penetration along the rays as shown in photograph 7(b). Agathis australis (Kauri)

This species is far removed phyllogenetically from the Pinus species but the pattern of radial penetration appears to be almost identical to that found in Pinus nigra. Kauri does not have ray tracheids hence any penetration of the rays can take place only through ray parenchyma. In photographs 49, 50, 51 and 52, the structure of the rays is seen in transverse, radial longitudinal, and tangential longitudinal sections. From these it is seen that the cells are thinwalled, non-birefringent, and disorganised to a considerable extent. The interference micrographs 53 and 54 indicate that the crossfield pits are open in both the earlywood and latewood but these may well be artefacts as the thin and partly collapsed parenchyma cell walls could be torn away from the pit apertures very easily during the sectioning of unembedded material.

When radial penetration takes place in this species it appears likely that the liquid could be forced free from the confines of the parenchyma cells and fill the ray spaces between the partially collapsed parenchyma cells. From here it would penetrate the axial tracheids throughthe unobstructed crossfield pit apertures in the radial walls of the tracheids. The bordered pits between adjacent tracheids of <u>Agathis</u> species

do not have a well-defined and thickened torus as in most conferous species (see photograph 55), and it is possible that these membranes could be ruptured by normal treating pressures. If liquid moved from a ray into one axial tracheid as suggested above, the collapse of the membranes of the bordered pits on the radial walls would permit transverse and vertical movement into other tracheids in the same tangential plane. This mechanism almost certainly operates in the latewood bands of the pines, where the majority of the pits are unaspirated, but the relatively low tangential permeability of kauri (see Fig. 4) suggests that the bordered pit membranes are not very easily ruptured and that most of the tracheids are probably penetrated directly from an adjacent ray.

#### Podocarpus dacrydioides

Podocarpus daorydioides exhibits the next highest radial permeability after the two Pinus species and kauri, but in this case there is no marked penetration along the rays. Photograph 56, a tangential longitudinal section, shows that the ray parenchyma cells are all intact, and this is illustrated even more clearly in photograph 57 in which a T.L.S. is viewed between crossed polars. In radial longitudinal section (photograph 58) further proof is seen that the ray parenchyma cells are entire and without any suggestion of disorder.

Parenchyma cell end walls which look yery robust and impermeable

are also clearly visible in this photograph. When 6 - 7p radial longitudinal sections were examined under the interference microscope (photograph 59) it appeared that all the crossfield pits were open and this was established as factual by remounting the section in a medium of different refractive index.

When the crossfield pits were studied by electron microscopy however, the picture appeared to be quite different. Ruptured crossfield pit membranes were found occasionally, as seen in photograph 60, but in most cases the membranes were intact as shown in photograph 61. In this photograph, the encrusting layer (formed on the inside of parenchyma cells from the dried remnants of the cytoplasm and other cell contents) is partly stripped off revealing the membrane as a continuation of the parenchyma cell wall. Thin sections of araldite embedded wood also showed the crossfield pit membrane to be intact (photograph 62) in all of several sections examined. In this photograph it is seen that the membrane is exceedingly thin and flimsy, and as the crossfield pits have a relatively large diameter it is not surprising that the membranes were ruptured when 6 - 7µ sections were cut from unembedded material with a sledge microtome. If the crossfield pit membranes are so easily ruptured by sectioning it is feasible to consider that they would be ruptured easily by the pressure involved in treatment. The mechanism for radial penetration might

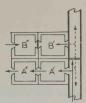
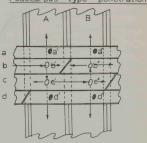
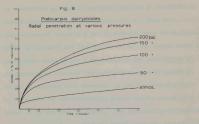


Fig. 7 Diagrammatic representation of a T.S. & R.L.S. showing

Podocarpus - type penetration





therefore be as shown in Fig. 7 i.e. solution passing along ray cell "b" is impeded by the end wall of that cell and pressure builds up. At a certain point the membrane covering crossfield pit "b' ruptures, and the liquid flows into axial tracheid A. When this is filled and pressure again builds up crossfield pit 'C' is ruptured and flow is initiated in both directions in ray parenchyma cell c. From here, penetration of tracheid B can be effected when the membrane of c" is ruptured and finally the liquid can regain entry to parenchyma cells of row b by rupturing the membrane at b". If pressure is required for penetration in this manner it would be expected that increases in the treating pressure would result in correspondingly higher uptake rates; this is shown to be the case in Fig. 8. The possibility of lateral re-distribution from penetrated axial tracheids depends upon the degree of pit aspiration, and the strength of the seal in the aspirated pits. When unsealed blocks of all species were vacuum impregnated for density measurements it was found that Podocarpus dacrydioides was the slowest to sink, but in pressure impregnation it had much faster uptakes than most species particularly in the tangential direction. These facts suggest that a large proportion of the bordered pits are aspirated but the membranesis easily ruptured by pressure. Members of the podocarpaceae have bordered pit membranes without thickened tori and photographs 63 and 64 show that in

both the latewood and the earlywood, the pits are aspirated. Although these pits are aspirated it is obvious that the membranes are not strong structures and very little pressure would be required to rupture them, particularly in the case of the earlywood pits, most of which showed the partial fracture (seen in photograph 64) around the margin of the pit orifice. Lateral re-distribution as shown in the transverse section diagram in Pig. 7. would therefore be highly probable.

If the pattern of radial penetration in <u>Podocarpus</u> is as suggested here, it follows that the treating solution must follow a fairly tortuous path through axial tracheids and bordered pits, as well as through ray parenchyma cells and crossfield pits. During this time the solution is in intimate contact with the cell walls of many different tissues and this could possibly account for the filtering or screening of the metallic constituents of copper-chrome-arsenate solutions during vacuum/pressure treatment.

## Pseudotsuga menziesii (Douglas fir)

Radial penetration is restricted to such an extent in Douglas fir that this species is generally considered refractory in relation to vacuum/pressure treatment. In photographs 22 and 23 it is shown that the ray parenchyma cells are intact and have end walls which tend to form barriers to penetration. Ray trachedia apparently constitute an important pathway for radial penetration but this is not invariably so; photograph 65

shows two rows of ray tracheid/axial tracheid pits that are all aspirated and P. J. Bailey (1969) showed that these pits were often encrusted. When the crossfield pits were examined under the interference microscope it appeared that they were ruptured (photograph 66) but when the sections were remounted in a medium of different refractive index (as described in 3b) it was obvious that the membranes were still intact, (see photograph 67). Replicas of ray tissue viewed in the electron microscope confirmed that the crossfield pit membranes were generally intact, and photograph 68 shows the aperture of one such pit from the axial tracheid side. The random orientation of microfibrils which is characteristic of primary cell walls is seen clearly in this replica.

Thus it would appear that radial penetration through the rays is restricted considerably in this species with only the intercellular spaces and some ray tracheids forming more or less unobstructed pathways. Rovement from the rays to the axial tracheids is also impeded by the relatively thick crossfield pit membranes, and the aspirated, often encrusted, bordered pits between ray and axial tracheids. To examine the possibility of lateral re-distribution of liquids if and when there was a breakthrough from rays to axial tracheids, the bordered pits were studied in both the earlywood and the latewood. In the earlywood tracheids, the observations of other workers were confirmed, and photograph 69 shows a typical

tightly aspirated bordered pit. Latewood pits are normally less aspirated and this was found to be the case in the material used in this study. Photograph 70 is a highly magnified electron micrograph of the pit margo just beyond the border of the torus : the spaces between the fibrils are shown distinctly. Although large particles of solids, and air embolisms, could be trapped by such a network, water or solutions could pass through quite readily. Lateral redistribution would therefore be quite possible in the latewood bands but not in the earlywood, and a penetration pattern such as this is frequently seen in commercially treated rounds of Douglas fir. In commercially treated wood the initial radial penetration is more likely to be through radial seasoning checks than through the rays however, and the resulting pattern of penetration is irregular, as seen in photograph 1. Partial penetration in which only the latewood bands are treated is acceptable with creosote under some specifications (provided that the penetrated bands are continuous and uninterrupted) but it is not acceptable with waterborne preservatives.

### Cryptomeria japonica

This species was not studied in detail as it was thought that the work done with <u>Sequoia</u> would suffice for the family <u>Taxodiaceae</u> to which both species belong. Table 1 shows that both <u>species</u> have a similar general structure, and the electron micrographs of Harada (1964) in which the nature of the crossfield

pit was first revealed, show a structure in <u>Cryptomorta</u>
<u>japonica</u> which is almost identical to that found in the
<u>Sequoia sempervirens</u> studied here. The uptake patterns shown
in Pigs 3, 4 and 5, and the patterns of penetration shown in
photographs 24 to 32 are also very similar for the two species.

Sequoia sempervirens (Californian redwood)

In photograph 71 the general structure of Sequoia rays is seen in both latewood and earlywood regions. The horizontal and end walls of the ray parenchyma cells are all intact with no sign of collapse or disorder, and there is no change in wall thickening or pitting at the latewood/earlywood boundary. Some ray tracheid tissue is also visible in this section but it is of the discontinuous and rudimentary type reported by Greguss (1955) for this species. When the crossfield pits were examined with the interference microscope it was found that the membranes were all intact in both the earlywood and the latewood, - see photographs 72 and 73. This was confirmed by electron microscope studies using both replicas (photograph 74) and thin sections of resin embedded material (photograph 75).

In photographs 29 and 30 it is seen that radial penetration in advance of the heavily treated area takes place along the intercellular spaces. These intercellular spaces (see pgotograph 76) appear to form relatively unobstructed passages of considerable length, but they are of small size (less than 2µ across) and liquid movement would be retarded by friction. Back (1969) has suggested that these intercellular spaces may form part of the gas canal system of living trees, allowing interchange of oxygen and carbon dioxide from the parenchyma cells. He states that there are pits connecting the intercellular spaces and the parenchyma lumena, but that these are often blind pits. No intercommunicating channels of any type were found in the Sequoia wood, so presumably the gases must diffuse across the relatively thin parenchyma cell walls. If this is so, and there are no open pathways from the intercellular spaces to the parenchyma lumena, then liquid must also diffuse through the cell wall in order to fill the parenchyma cells. Liquid movements from the ray parenchyma cells to the axial tracheids would follow if the pressure built up to a sufficient extent to rupture the crossfield pit membranes. This does not seem to occur however, as sections cut from the middle of the heavily treated area were found to have the crossfield pit membranes still intact when examined under the interference microscope, see photograph 77. This would also appear to rule out any possibility of radial penetration of the type suggested for Podocarpus. The other possibility is that liquid moves through the ray parenchyma cell wall itself and through the crossfield pit apertures in the tracheid walls without rupturing the membranes. Photograph 30 could be interpreted as showing

this type of liquid movement from the intercellular space to the parenchyma wall and hence to the tracheid lümen.

The prospects for much lateral redistribution also seem poor in <u>Sequeia</u> as the latewood percentage is low, the earlywood bordered pits are aspirated, and the tori relatively thick and impermeable - see photographs 78 and 79.

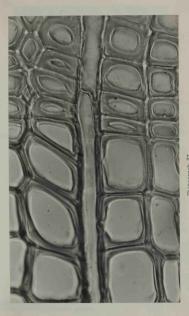
In photographs 30 and 32, cells having the general appearance of axial parenchyma are seen to be penetrated in advance of nearby axial tracheids and it is possible that these could be penetrated directly from the intercellular spaces. From these cells liquid could then move to other axial cells, and also to other rays, as suggested in photographs 31 and 32. There is no indication in the photographs of dye penetration, that the liquid moves from one axial cell to another through the bulk of the cell wall but this possibility cannot be ruled out.

## Cupressus macrocarpa

The samples of <u>Cupressus macrocarpa</u> were the least permeable of any used in this series of tests. The ray tissue of this species consists of only parenchyma cells but these are very robust and thick-walled as shown in photograph 80, and the electron micrographs. The simple pits between adjacent ray cells are only constrictions in the wall with almost one micron of cell wall left intact, see photograph 81.

The end walls of the parenchyma cells are intact, and while they do not appear to be particularly robust (see photograph 82), they would not permit an easy passage of liquids and would require considerable pressure to be ruptured. Photograph 83 shows an intact crossfield pit membrane of considerably greater thickness than any seen in the other species. The membrane appears to have been damaged during specimen preparation but even allowing for this it is seen to be thick and somewhat convex. Photograph 84 is a replica of the inside wall of a ray parehchyma cell and the outline of a crossfield pit can be seen on the surface which is covered with the dried remnants of the cell contents. When radial longitudinal sections 6µ thick were examined under the interference microscope, the crossfield pit membranes stood out very clearly (see photograph 85) and at higher magnification (photograph 86) the convex nature of the membrane is indicated by the variations in phase difference across the membrane.

From these considerations of ray structure it would not be expected that liquids could move easily along the rays, and in photographs 35 and 34 it is seen that advance penetration is virtually non-existent except for that which takes place along the intercellular spaces. A feature of this penetration not eVident in the other species, is the penetration of the intercellular spaces between the axial tracheids; this appears to originate directly from ray intercellular space penetration. It is not possible to see however whether the liquid which has penetrated the spaces between the tracheids is able to move into the lumens of these cells through either the pits or the walls. Interal redistribution would be minimal in this species because of the small amount of latewood tissue and the tightly aspirated pits in the earlywood as seen in photographs 87 and 88,



Pinus radiata TS. showing ray perenchyma end wall.



Pinus radiata TIS. Epon embedded 2u section X 480.



Photograph 39
Pinus radiata TLS. Same section as in Photograph 38
viewed between crossed polars. X 480



Photograph 40

Pinus radiata TIS. Through crossfield pit. Electron micrograph of epon embedded section. X 13,800



Photograph 41

Pinus radiata RIS. latewood. Interference photomicrograph. X 750

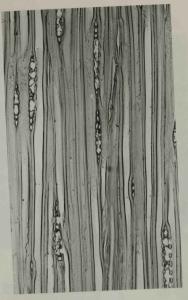


Photograph 42

Pinus radiata RLS earlywood. Interference photomicrograph.

X 750

Photograph 43



Photograph 44

<u>Pinus nigra</u> TLS latewood. Section out after embedding in polyethylene glycol. X 240



Photograph 45

Pinus nigra. Encrusted ray tracheid bordered pit. Cellulose acetate replica. X 18,800



Pinus nigra RLS. earlywood. Interference photomicrograph. X 960



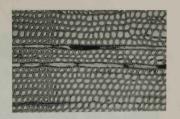
Photograph 47

Pinus nigra. RLS latewood. Interference photomicrograph. X 480



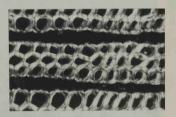
Photograph 48

Pinus nigra RLS latewood. Interference photomicrograph. X 750



Photograph 49

Agathis australis TS showing rays passing through an earlywood/latewood boundary. X 120



Photograph 50

Agathis australis T.S. viewed between crossed polars. X300

The Assessed



2

Agathis australis ILS. Ray parenchyma cells. Section cut from material embedded in

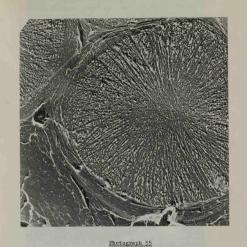


Photograph 53
Agathis australis RLS. Interference photomicrograph. X600



# Photograph 54

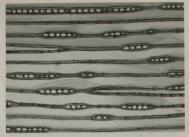
Agathis australis RIS. Interference micrograph of very narrow crossfield pit (width 0.8 \mu). Background colour, blue. X 2300



Agathis australis.

Bordered pits. Methylmethacrylate two-stage replica.

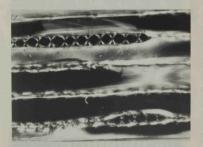
X 11,500



Photograph 56

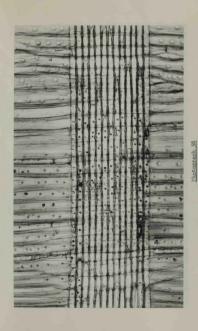
Podocarpus decrydioides TLS.

X 240

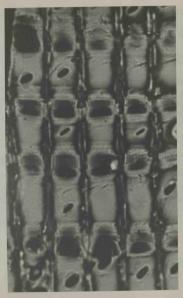


Photograph 57

Podocarpus dacrydioides TLS. viewed between crossed polars.



odocarpus dacrydioides RLS. through ray.



Photograph 59

Podocarpus dacrydioides RLS. Interference micrograph with dark background.



Photograph 60

Podocarpus dacrydioides. Direct replica showing a ruptured crossfield pit membrane. X 23,000



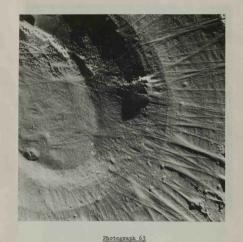
Photograph 61

<u>Podocarpus dacrydioides</u>. Direct replica. The outline of a crossfield pit can be seen through the membrane and the encrusting layer which has been partly stripped off.

X 18,600



Red<u>cerpus</u> decrydioides. Electron micrograph of a section through a crossfield pit.  $\text{KinO}_4$  stained and avaidite embedded. X 22,500



Podocarpus dacrydioides. Direct replica of a latewood bordered pit. X 23,000



Podocarpus dacrydicides. Direct replica of an earlywood bordered pit. X 12,500



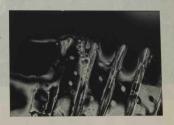
Photograph 65

Pseudotsuga menziesii. Aspirated bordered pits between ray tracheids and axial tracheids. Direct replica. X 5,500



Photograph 66

Pseudotsuga menziesii RLS. Interference micrograph of 6m section mounted in 20% glycerol. X 750



Photograph 67

<u>Pseudotsuga menziesii</u> RIS. Same material as in Photograph 65 but remounted in liquid paraffin. X 750



Photograph 68

Pseudotsuga menziesii. Crossfield pit. Direct carbon replica. X 12,500



Photograph 69

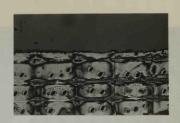
Pseudotsuga menziesii. Aspirated bordered pit in the earlywood. Direct replica. X10,000



Photograph 70

Pseudotsuga menziesii. Part of the margo of an unaspirated latewood pit. Direct replica. X 75,000

Sequoia sempervirens RLS. Resin embedded 5u section.



Photograph 72

Sequal sempervirens RLS. Interference micrograph of earlywood. X 480



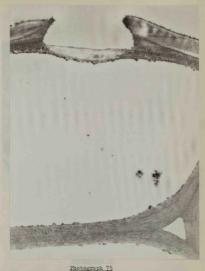
Photograph 73

Sequoia sempervirens RLS. Interference micrograph of latewood. X 480

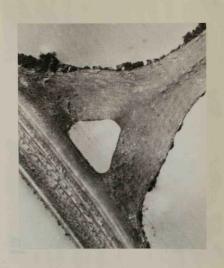


## Photograph 74

Sequois sempervirens. Cellulose acetate replica of a crossfield pit showing the border and the warty layer of the tracheid wall.



Sequoia sempervirens. Section through a ray parenchyma cell showing the crossfield pit membrane. Mino fixed/stained, epon embedded. X 8,600.



Photograph 76

Sequoia sempervirens. Section through a ray showing the intercellular space between two ray parenchyma cells and the wall of an axial tracheid. Stained with MMnO<sub>A</sub>, X 14,000



Photograph 77

Sequoia sempervirens. Interference micrograph showing intact crossfield pit membranes in the heavily treated area. X 750.



Sequois sempervirens. Section through an aspirated bordered pit. Stained with MinO<sub>4</sub>, embedded in epon. X 18,000.

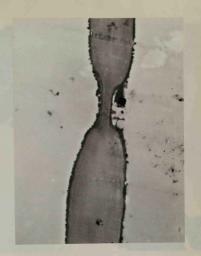


Photograph 79

Sequoia sempervirens. Bordered pit aperture and the surface of the appressed torus. Cellulose acetate replica. X 18,500.



Cupressus macrocarpa TLS. Section through a ray and a bordered pit.



Photograph 81

Cupressus macrocarpa RLS. Section showing a simple pit in horizontal wall between two ray parenchyma cells. Stained with KMnO<sub>4</sub>, embedded in styrene/methacrylate. X 10,800



Photograph 82

Gupressus macrocarpa RIS. Section through a ray parenchyma end wall. Preparation as for Photograph 80. X 10,800.



Photograph 83

Cupressus macrocarpa. TLS. Section through a crossfield pit with membrane intact. Preparation as for Photograph 80. X 11,500



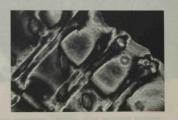
Photograph 84

<u>Cupressus macrocarpa</u>. Cellulose acetate replica of the inside well of a ray parenchyma cell showing the outline of a crossfield pit. The remmants of cell contents litter the surface. X 12,000



Photograph 85

Cupressus macrocarpa RIS. Interference micrograph. X 750



Photograph 86

Cupressus macrocarpa RLS. Interference micrograph. X 1,900



Photograph 87

Cupressus macrocarpa. Replica of an earlywood bordered pit. X 9,000



Photograph 88

Cupressus macrocarps. Section through an aspirated bordered pit. Preparation as for Photograph 80. X 10,800.

## PART 4

## Possibilities for improving the penetration of preservative into Refractory species

Nost attempts to improve the permeability of wood have relied on mechanical or physical methods and these have not been notably successful.

One of the oldest techniques is mechanical incising, and this is still used with a number of commodities such as railway sleepers (which are incised on all faces) and poles (which are normally incised only in the ground line area). The incisions are made with a truncated triangular steel knife in most cases, and the resultant incisions are about 3 inch deep and \$ - 1" long in the direction of the grain. Incising can be done by hand, using a hammer with several knives protruding from the face, or mechanically, by forcing the timber between revolving drums, into which the knives are set. During preservative treatment the preservative penetrates the incisions easily, and is then forced out. along and across the grain, to produce elongated lensoid pockets of penetrated wood around each incision. The incisions must therefore be spaced so that the treated pockets coalesce to form a completely treated envelope. This will happen only with species which have a reasonably high tangential permeability. With species of low tangential permeability such as Cupressus

macrocarpa, the incisions would have to be very closely spaced and this would not be practicable. If the incident were made across the grain instead of along it, fewer cuts would be required because longitudinal penetration would cover a much greater area from each incision. Incisions in this direction would be much more difficult to make, however, and as they would out across the fibres the strength of the pole would be drastically reduced.

Thus for incising to be effective, safe, and economic, it is essential that the species being treated should have a considerably greater permeability in the tangential than the radial direction. From Figs. 5 and 4 it is seen that this applies to only <u>Podocarpus dacrydioides</u>, and to a lesser <u>Sequoia</u> and <u>Cryptomeria</u>. Even with these species it would be necessary to establish uniform tangential penetration of both the latewood and earlywood bands, especially if the treatment was with a highly fixed waterbourne preservative such as copper-chrome-arsenate.

Higher treating temperatures and pressures usually improve penetration but the nature of the wood and the preservatives limit the extent to which these can be increased. It has been demonstrated that the radial permeability of Podocarpus dacrydicides is improved progressively as the pressure is increased from 0 - 200 p.s.i. (see Fig 8 ) but it is unlikely that this species could stand much higher pressure without the cells collapsing. Herdwoods such as

Eucolyptus species are creosoted at pressures of up to 1000p.s.i. in Australia but with Douglas fir it has been found necessary to restrict treating pressures and temperatures to about 160 p.s.i. and 180° to prevent damage to the wood. With preservatives such as creosote or pentachlorophenol-in-oil, penetration is greatly improved at elevated temperatures because the viscosity is lowered and the hot oil tends to dissolve resins and other impeding substances. The temperature of copper-chrome-arsenate preservatives cannot be increased by any significant degree however, because solutions containing chromates become unstable at temperatures above about 120°F.

The Queensland Forest Products Laboratory (Australia) has claimed that penetration and uptake are improved if a very high vacuum is maintained for an extended period at the commencement of vacuum/pressure treatment, but this would not be expected from the results of Petty(1969), and semi-commercial trials carried out by the Australian Commonwealth Scientific and Industrial Research Organisation did not confirm it.

Fre-steaming, either before seasoning, or before treatment, is another technique which has been tried in an attempt to improve treatability. McQuire (1964) found that pre-steaming freshly out radiata pine produced much better treatment by the Oscillating pressure method, and while a slight reduction in the moisture content was the main beneficial result, it was suggested that some physical or chemical effects may also have been important. Eamber and Johnstone (1968) used pre-steaming to

improve the permeability of radiata pine heartwood, and showed that this treatment affected the ray structures. Photomicrographs of steamed and unsteamed rays showed that the steaming had caused a partial collapse of the parenchyma cells leaving large irregular intercellular spaces. As mentioned in Part 1 of this thesis, steaming produced a slight, but definite, improvement in the treatment of Douglas fir posts, but the standard obtained was still unsatisfactory. Prolonged steaming at higher temperatures and pressures would probably improve subsequent treatment but this would be achieved at the expense of wood strength. In the relatively mild steaming schedules used to prepare wood for OPM treatment it was found that there were losses of up to 20% in some strength properties (McQuire 1966) and greater losses than this could not be tolerated in load-bearing commodities such as poles.

In the last two decades there has been considerable interest in biological methods of improving permeability, but the organisms responsible have not always been identified, and hypotheses put forward to explain their mode of action have been inconclusive and generally unproved.

Attention was first focussed on the possibility of improving permeability biologically by Blew (1952) and Lindgren and Harvey (1952). Blew showed that fungus infected pine posts absorbed considerably more preservatives than control posts which had been treated with an anti-stain chemical before

seasoning, but the relative merits and descrits of the various fungal species was not known. Lindgren and Harvey used a preseasoning spray with % sodium fluoride and this resulted in a profuse growth of the mould Trichederma which inhibited the development of other fungal species, particularly the wood decaying basidiomyceftes. Even so it was found that the Trichederma infected pine posts absorbed approximately 10 times as much light oil preservative during a 5 minute soak as did control posts treated with sodium pentachlorophenate and ethyl mercury phosphate to prevent all fungal growth. Lindgren (1952) obtained similar increases in the permeability of pine blocks innoculated with Trichederma in the laboratory but attempts to increase the permeability of other species were less successful.

Graham (1954), and Lindgren and Wright (1954) carried out a series of scaking, hot and cold bath, and pressure treatment trials on Douglas fir posts which had been given various pretreatments to encourage or to prohibit mould growth. In general the infected posts treated much better than the clean controls but those specifically innoculated with Trichoderma were less permeable than some others which had been naturally infected. Wicroscopical examination of the heavily moulded posts showed an almost complete breakdown of the ray tissue and as these cells are lignified in Douglas fir it is possible that some degradation of the wood tracheids might also have taken place.



Photograph 89

Douglas fir post sections. Pre-trea

Pre-treatment - NaF spray.
Treatment - Vacuum/Pressure
with CCA preservative.

Perry (1955) carried out similar laboratory tests with blocks of Douglas fir wood and found that treatment with NaF plus a spore suspension of Trichoderma increased the uptake of solution in dip tests. When a large scale test with Douglas fir posts was carried out however the results were unexpected. Three basic treatments were used, - no treatment, sprayed with 2% NaF, and sprayed with 2% NaF plus Trichoderma spore suspension. Each of these was subdivided into two groups - covered with tarred paper and not covered, and each of these was again subdivided into two groups - close piled and open piled. Both the treated groups which had been close piled and covered, treated much better than usual, but the surprising result was that the group which had no treatment but had been close piled and covered, treated twice as well as any other. There was no visible sign of the presence of any decay fungi and the increased absorptiveness was ascribed to some hitherto undetected native mould. With hindsight, it seems almost certain that this increase was caused by bacterial action.

Schulz (1956) improved the uptake of creosote/fuel oil in pressure treatment of spruce posts by a factor of 3 - 5 X by inducing <u>Trichoderma</u> growth, but a similar test with Douglas fir in New Zealand failed to produce a marked improvement (photograph89).

The reason for this difference was probably that the Douglas fir posts were put on seasoning racks after spraying and although under shade, dried too quickly for the fungi to become well established. Schulz incubated his spruce posts at 80°P and 97% relative humidity initially, and then gradually reduced the humidity to allow them to season.

Thus although fungal infection was shown to increase permeability with several wood species, the increase was not always sufficient to ensure really adequate treatment, and there was always the risk that damaging decay fungi night become established. When subsequent treatment was with creaset, the heat and volatile constituents of the preservative would sterilize the wood beyond the linit of actual penetration, but this would not be so with waterborne salts.

Ille (1957) published the first report dealing with permeability increases caused by ponding and stated that improved creosete treatment was obtained with spruce, silver fir and Douglas fir. Stutz and Stout (1957) found an increase in porosity in ponded <u>Pinus ponderosa</u>, <u>Pinus lambertians</u> and <u>Pinus monticola</u>, and stated that this was brought about by bacterial action on the ray cells. Since 1957 several workers have examined the treatability of water-stored wood and in every case a considerable increase in the permeability of sapwood has been reported.

A very full bibliography, with abstracts, has been prepared by Unligil (1969) covering all forms of water storage and <u>Trichoderma</u> infection in relation to the penetrability of wood,

and only the most pertinent papers will be referred to here. The majority of these published works deal with Pinus species but some cover other more refractory species, and these also report marked increases in permeability. Forest Products Research Laboratory (Princes Risborough) 1960 examined sitka spruce. Douglas fir, and Japanese larch that had been stored in a log pond for periods of 3, 6, and 20 months, and found that uptakes improved considerably for ponding periods up to six months ; after 6 months the improvement was much less marked. Klem and Halvorsen (1963) found that a complete and uniform sapwood penetration was obtained in Norway spruce that had been sunk in rivers and lakes for periods of 18 - 150 years : no significant changes were found in any other properties of the timber. Liese and Karnop (1968) studied pine and fir permeability after summer and winter ponding, and found that increases of ten times were obtained after four weeks summer storage. Winter storage caused only a slight increase in the permeability of pine, and had practically no effect on fir. Vakim et al. (1968) carried out large scale trials with spruce poles, and concluded that water storage without bark was one of three treatments that would guarantee subsequent preservative treatment to the standard required for transmission poles in Russia. The other two treatments recommended were infection with "safe" fungi, and moist storage with bark still on. Apart from the first published paper by Ille (1957), no mention is

made of alternative types of water storage (such as under sprinklers) for the refractory species. Pine wood held under sprinklers has been examined by some workers however, and these (including Suclahti 1961, and MacPeak 1965) have reported permeability increases quite comparable with fully ponded material.

In 1967 the Irish Institute for Industrial Research and Standards pended some Sitks and Norway agrace poles to examine the effect of this treatment on say replacement treatment. The results of this test were disappointing but when some of the material was pressure treated with creesete the results were so spectacular that further thals were initiated. After hearing of these results from Dr. Dunleavy (Head of the Forest Products Department I.I.R.&D.) the author requested that samples of pended and unponded sitks spruce be made available for laboratory permeability measurements and detailed micoscopical examination.

## History of the sitka spruce test material

The trees were from Killeshandra Forest, County Cavan.
They were planted in 1954 (at a spacing of 6' x 4') and were
felled in 1968 during a fourth thinning operation; at that
time the stand was considered to be slightly overstocked at
about 500 stems per sore and the current annual increment was
beginning to fall off. The trees were cut to the size of
"standard" poles, 9 metres long with a minimum top diameter
(inside bark) of 15cm., and minimum butt diameter of 20.4cm

inside bark. After peeling, the poles were made into rafts of about 10 poles each, and ponded in one of the small Killeshandra lakes. The lake forms part of a river system, and the water is fresh and apparently well aerated judging from its popularity for fishing. The poles were removed from the lake in March 1969 (after ponding periods of 4 - 11 months) and were open stacked on well ventilated racks to season.

Freshly felled trees were peeled and seasoned at the same time.

Samples cut from the sapwood and heartwood of one ponded and one unponded pole were forwarded to Leeds University in August 1969. The exact ponding time of the pole from which the sample was cut was not known, but it was somewhere between 6 - 11 months.

#### Commercial treatment trials

Two charges of poles (both of which contained ponded and fresh poles) were treated by P.D.W. Ltd, Kilteel Polefield, in October 1969. Treatment was with creosote by the Rueping process and charge details were as follows:)

	Charge A	Charge B
moisture content (sapwood) fresh poles	25%	25%
ponded poles	35%	35%
charge volume (cu.ft.)	342	237
Initial air pressure	40p.s.i.	40p.s.i.
Storage tank temperature	190°F	190°F

Treating temperature	150°F - 165°F	150°F-173°F
Treating pressure	147p.s.i.(max)	140p.s.i.(max)
Pressure period	140 minutes	120 minutes
Final vacuum (over night)	24 ins.	24 ins.

Charge A

22.261bs/cu ft.

Charge B

15.541bs/cu ft

The appearance of the poles after treatment was the same for both charges and was stated on the charge sheets as "Ponded poles-wery dry on surface, penetration full saywood. Unponded poles-oil on surface, penetration approx à "toà" even penetration blus irrecular patches around seasonine cracke".

The charge sheets do not mention the number of ponded and unponded poles in each charge, and as no poles were weighed before and after treatment, there is no way of knowing the comparative retentions of the two groups. The nett absorptions given seem abnormally high even for well treated sapwood and, if correct, must indicate a very heavy loading in the treated zone. The uniformity and depth of penetration are illustrated in ubotocrauks 90 and 91.

#### Jaboratory tests on Permeability

Nett absorption

Using the same apparatus and methods described in Part 1 of this thesis, the permeability was determined in all three grain directions for the sapwood and heartwood of both the ponded and fresh samples. Results of these tests are shown graphically in Figs. 9, 10 and 11, (longitudinal, tangential



### Photograph 90

Picea sitchensis.

Cross sections cut from the centre of ponded and fresh poles after pressure treatment with creosote.



Photograph 91

# Pices sitchensis.

Bolts cut from creosoted ponded poles and ripped down the centre. Sapwood penetration has been complete and even.

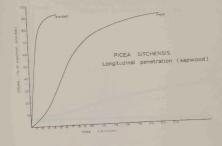


Fig. 9

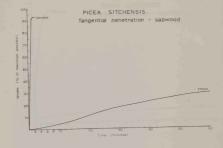


Fig. 10

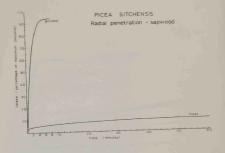
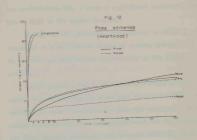


Fig. 11



and radial penetration for ponded and fresh saywood) and Fig. 12 (penetration in all three directions for ponded and fresh heartwood).

As with the other species examined, the longitudinal penetration in the small 2 x 2 x 2 cm. blocks was very rapid, but nevertheless Fig. 9 shows a significant increase in the uptake rate in the ponded material.

In Fig. 10 the tangential permeability is shown to be increased to a very marked extent by ponding and is comparable in fact to the longitudinal permeability of the unponded material.

Radial permeability is increased by similar amount in the ponded saywood and Fig. 11 shows that virtual saturation was achieved in under five minutes. From the correlation between radial permeability and treatability which was established for the other species examined, this increase would emerge as the most important benefit derived from the ponding.

From Fig. 12 there is no indication that ponding has had any effect on heartwood permeability in any grain direction. The permeability of the fresh material is slightly higher in fact, due probably to the slightly lower density and greater ring width in these particular samples. The absence of any increase in heartwood permeability after ponding is in accord with the findings of other investigators - Vasilev (1965).

Forest Products Research Laboratory (1965), and Liese and Karnop (1968).

## Pathways of Penetration and the effect of structural factors on liquid movement

It is generally agreed that in all softwoods, longitudinal penetration takes place along the axial tracheties, with the bordered pits as the main communicating pathway between one trachetid and the next in series. The same general pattern of movement also applies to tangential penetration, i.e. from one trachetid to the next through the bordered pits in the radial walls. In this case the rate of movement is much slower however because the distance across the trachetid lumen is much shorter than the longitudinal distance. Thus the bordered pits constitute the main obstruction to smooth liquid flow in both the longitudinal and tangential directions and if large numbers of the bordered pits are aspirated, the flow is impeded.

In latewood, pit aspiration is usually prevented by the thickness of the cell walls, and the torus remains in a more or less central position when the wood is seasoned. This fact explains the greater tangential and longitudinal penetrability of the latewood bands of most softwood species. Photograph 92 shows an unaspirated latewood bordered pit in the sapwood of the unponded sitks spruce. In the earlywood of the fresh sapwood the pits were aspirated with the tori pressed firmly

over the pit apertures as shown in the electron micrograph of a direct carbon replica (photograph 93) and in the stereoscan electron micrograph (photograph 94). In the ponded material however the tori and entire pit membranes were absent. Careful examination of the earlywood of many preparations of ponded sapwood failed to locate any trace of a torus or membrane. The techniques used to examine these pits included interference microscopy with thin sections of epoxy resin embedded wood (photograph 95), the Stereo-scan (photograph 96) and electron microscopy with direct and cellulose acetate replicas. Knuth (1964) states that bacteria destroy the tori and disrupt the remaining membrane of pine wood; Greaves (1969) shows examples of pit attack in pine by bacteria; Knuth. McCoy and Duncan (1965) show bacterial attack on pine pit membranes; Lutz, Duncan and Scheffer (1966) mention bacterial activity in the pits of water stored southern pine; and Liese and Karnop (1968) state that in ponded Scots pine and fir the tori become granulated, or disintegrate, or occasionally are seen lying eccentrically in the pit chamber because (presumably) of the destruction of the border fibrils. Thus most workers who have studied water stored wood and bacterial action report some measure of attack on the pit membrane and torus, and the complete destruction of these elements, in the sapwood of the ponded spruce examined in this study, is without doubt the cause of the phenominal

increase in tangential permeability. The destruction of the torus, which consists mainly of pectin in the sapwood region, is believed to be caused by the action of pectinase producing bacteria. Microscopical examination of sections cut from spruce wood after ponding for two months in lake Killeshandra, showed large numbers of bacteria in the cavities of almost all the pits in the sapwood (see photographs 97 and 98). Liese and Karnop (1968) note that one of the first manifestations of bacterial attack is the reduced staining ability of the tori to pectin indicators. Suolahti and Wallen (1958) found a correlation between pectin decrease, and permeability increase, in pine that had been exposed to bacteria and fungi, and Knuth and McCoy (1962) considered hydrolysis of pectic compounds by bacteria to be the major factor responsible for increased longitudinal permeability in ponded pine. Vasilev (1965) isolated bacteria from ponded pine and Norway spruce and found that these were capable of utilizing sugars with a low degree of polymerization, pectins, and (apparently) stored starch, but they did not affect the ligno-cellulose complex. This could explain the lack of any increase in permeability in the ponded heartwood as Bauch, Liese and Scholz (1968) have shown that lignin (and additional aromatic compounds) are formed in the tori during heartwood formation. Nicholas and Thomas (1968) were able to degrade the torus of pine sapwood pits with pectinase solution, but found that enzymes had no

effect on tori in the heartwood. They considered the reason to be poor accesibility caused by extractives forming an incrusting layer over the pit membranes.

Destruction of the bordered pit membranes would not be expected to affect radial permeability and the reason for the marked increase in this direction probably lies in changes in the ray tissues. Several workers have remarked on the early bacterial colonization of rays in ponded wood and photographs 99 and 100 show concentrations of bacteria in the rays of sitka spruce after 2 months ponding. Damage to the ray parenchyma cells of pine wood has been reported by Ellwood and Ecklund (1959), Knuth (1964), Greaves and Levy (1965), Lutz, Duncan and Scheffer (1966), and Liese and Karnop (1968). With pine sapwood, the final outcome of bacterial attack on the ray parenchyma cells is an almost complete disintegration of these tissues, particularly in the multiseriste rays containing radial resin canals. This would certainly improve radial permeability but nothing approaching this state of disintegration was found in the ray cells of the sitks spruce examined in this work. Photographs 101, 102, 103, 104, 105, 106, 107, 108, 109 and 110 are comparable pairs of photomicrographs of fresh and ponded sapwood seen in radial, transverse, and tangential section. These photographs show that there has been no noticeable degradation of the ray tracheids, or parenchyma cells, or of the radial resin canals, and certainly no collapse or disintegration as has been reported for pine rays. Liese and Karnop (1968) found that bacterial attack on pinewood rays commenced at the crossfield pits and then proceded to the remainder of the parenchyma cell walls until all these were eventually destroyed. With fir samples however, only the crossfield pit membranes were attacked, the remainder of the cell walls being unaffected so that the structure of the rays was preserved. The crossfield pits of both fresh and ponded sapwood were therefore studied using the various microscopic techniques described in Part 3, and also the "Stereo-scan" scanning electron microscope with small blocks of solid wood which had been split to expose radial faces. In photographs 111 and 112 the interference micrographs indicate that in the ponded material the majority of the crossfield pits have been ruptured. In 111 (freshwood) only one pit in the field is open and from the immediate surrounds it is obvious that this has been cut during specimen preparation. In the ponded material over 50% of the pits are open, and in only one of the five rows of ray cells shown in the field are all the membranes intact. The Stereo-scan electron micrographs of fresh and ponded wood demonstrate this partial destruction of the crossfield pit membranes even more clearly. In photograph 113 only one pit in the field is open and this could be the result of specimen preparation. In photographs 114, 115 and 116 the majority of the pits are open and while some of these holes could be made by specimen preparation, it is obvious that

others are not. Photographs 117 and 118 show higher magnification micrographs of such pits where the parenchyma wall is
complete and undamaged around the pit orifice. That not all
ponded crossfield pit membranes are ruptured is shown in
photograph 119. Photographs 120 and 121 (fresh and ponded wood
respectively) are direct replicas showing the pit apertures and
membranes viewed from the direction of an axial tracheid, the
secondary wall of which has been split during sample preparation.
The nature of the perforation in the membrane of the ponded
wood is similar to that seen when using other techniques.

To determine the actual pathway of liquid movement during radial penetration, blocks of fresh and ponded sapwood were treated with acid fuchsin, and sectioned as described in Part 2. Photograph122 shows the general pattern of penetration in the solid blocks. In (a) (fresh material - 20% uptake after 40 minutes at 100 p.s.i.) the penetration is seen to be very even with no advance penetration of the rays. In (b) (ponded sample - 17% uptake after 2 mins. at Atmospheric pressure) slight penetration of the rays can be seen.

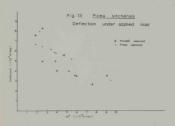
Photographs 123 and 124 show dye penetration in fresh sapwood and these confirm the opinion of Liese and Bauch (1967) that the parenchyma cells are difficult to penetrate and that radial penetration tends to be by way of the ray tracheids.

Even so, the ray tracheids do not appear to be very easily penetrated and several show little or no advance penetration of

the dye solution. Photograph 125 demonstrates that the end walls of the parenchyma cells in the pended wood still constitute a barrier to penetration but photograph 126 shows that there is a general penetration of the ray parenchyma and ray tracheid cells in advance of the region of heavily treated axial tracheids. These photographs suggest wery strongly that the radial penetration in the pended wood is of the same type that was proposed for Pedecarpus dacrydicides. As the berdered pit membranes and a large number of the crossfield pit membranes have been destroyed by bacteria and do not have to be ruptured by pressure, it would be expected that penetration and redistribution would be sore rapid in pended spruce than in Pedecarpus and this was borne out by the radial permeability measurements shown in Figs. 5 and 11.

The lack of any increase in the redial permeability of ponded heartwood must be due to some change that takes place in the crossfield pits during heartwood formation. Falatinees and Kennedy (1967) have shown that in spruce the ray parenchyma cells mature and become lignified in the sapwood at a fairly early stage, but it is possible that lignification does not extend to the crossfield pit membranes which may remain largely pectin. During heartwood formation, some change may take place similar to the heartwood lignification of the tori (as described by Bauch, Liese and Scholz, 1968) so that bacterial enzymes are no longer able to attack the membrane.

The greatest danger with fungal pre-conditioning to increase permeability is that basidiomycete fungi may become established, or that the moulds may themselves act as soft rot organisms and cause a reduction in the mechanical strength of the wood. This danger does not appear to be present when wood is subjected to bacterial attack by ponding for the length of time necessary to bring about an adequate increase in permeability and treatability. The photographs of radial, tangential, and transverse sections of ponded spruce show no sign of attack on the axial tracheids, and preliminary mechanical tests showed no loss of strength. Fig.13 shows the result of a small laboratory-scale test for stiffness; this property being one which is markedly affected by even the earliest stages of biological degradation. Tiny beams cut from the centre of the growth rings of both fresh and ponded wood, were supported at each end and tested for stiffness by applying a set load to the centre. The deflection was measured to the nearest 0.001 inches on a dial gauge micrometer. By plotting deflection against bd3 it is seen that there does not appear to be any notable difference between the two samples.

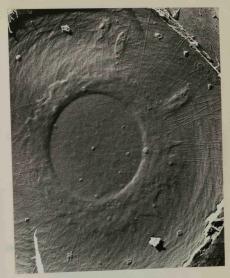




Photograph 92

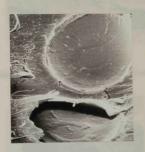
Picea sitchensis. Unaspirated latewood bordered pit from fresh wood.

Direct Replica. X 19,000



Photograph 93

Pices sitchessis Aspirated earlywood bordered pit from fresh wood.
Direct Replica. X 13,800



Photograph 94

Picea sitchensis Stereo-scan photomicrograph of earlywood bordered pits from fresh wood. X 3,200



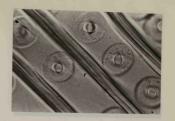
Photograph 95

Interference photomicrograph of resin embedded Picea sitchensis ponded material X 750



Photograph 96

Stereo-scan photomicrograph of an earlywood bordered pit from ponded sapwood. X 3,800 Picea sitchensis.



Photograph 97

Picea sitchensis.

R.L.S. showing bacteria in sapwood bordered pits after two months ponding. X 1,200



Photograph 98



Photograph 99

<u>Picea sitchensis.</u> R.L.S. Bacteria in ray parenchyma cells after two months ponding. X 1,890



Photograph 100

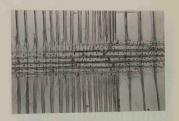
Picea sitchensis. R.L.S. Bacteria around the crossfield pits of ponded sapwood. X 1,890



Photograph 101

Picea sitchensis. R.L.S. Fresh Sapwood

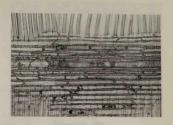
X 375



Photograph 102

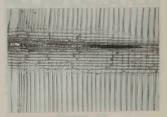
Picea sitchensis. R.L.S. Ponded Sapwood.

X 190



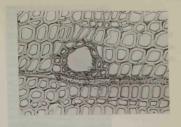
Photograph 103

Picea sitchensis. R.L.S. Fresh Sapwood. Section through a multiseriate ray with resin canal. X 190



Photograph 104

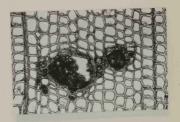
Picea sitchensis. R.L.S. Ponded Sapwood. Section through a multiscriate ray with resin canal. X 120



Photograph 105

Picea sitchensis. T.S. Fresh Sapwood.

X 290



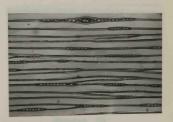
Photograph 106

Picea sitchensis. T.S. Ponded Sapwood.

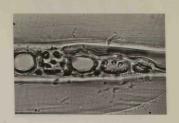
X 190



Photograph 107
Picea sitchensis.
T.L.S. Earlywood.
Fresh Sapwood.
X 120

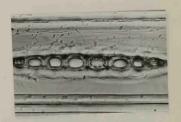


Photograph 108



Pices sitchensis. T.L.S. Presh Sapwood. Section through ray.

X 1,150



Photograph 110

Picea sitchensis. T.L.S. Ponded Sapwood. Section through ray.



Photograph 111

<u>Picea sitchensis</u>. R.L.S. Interference photomicrograph with light background. Fresh sapwood. X 750



Photograph 112

Picea sitchensis. R.L.S. Interference photomicrograph with dark background. Ponded sapwood. X 750



Photograph 113

<u>Ficea sitchensis</u>. Stereo-scan photomicrograph of fresh wood ray.

X 740



Photograph 114

Picea sitchensis. Stereo-scan photomicrograph of ponded wood ray. X 880



Photograph 115

Picea sitchensis. Stereo-scan. Ponded wood ray. X 440



Photograph 116

Picea sitchensis. Stereo-scen. Ponded wood ray. X 440



Photograph 117

Picea sitchensis. Stereo-scan. Ruptured crossfield pit in ponded sapwood. X 1,100



Photograph 118

Picea sitchensis. Stereo-scan. Ruptured crossfield pit in ponded sapwood. X h,300



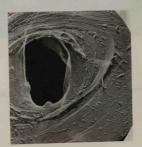
Photograph 119

Picea sitchensis. Stereo-scan. Intact crossfield pit membranes in ponded sapwood. X 1,700



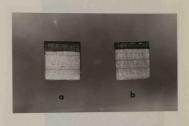
Photograph 120

Picea sitchensis. Direct replica of a crossfield pit in fresh sapwood, X 11,300



Photograph 121

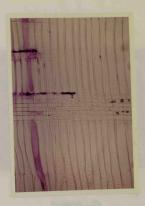
Picea sitchensis. Direct replica of a crossfield pit in ponded saywood.



## Photograph 122

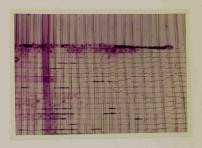
Picea sitchensis. Penetration of acid fuchsin into blocks.

- (a) Fresh. 20% uptake in 40 minutes at 100p.s.i.g.
- (b) Ponded. 17% uptake in 2 minutes at 0 p.s.i.g.



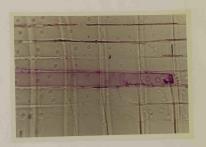
Photograph 162

Picea sitchensis. R.L.S. Radial penetration of acid fuchsin in fresh sapwood. X 175



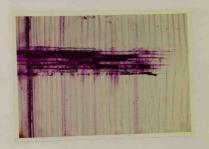
# Photograph 124

Picea sitchensis. R.L.S. Radial penetration of acid fuchsin in fresh sapwood. X 140



Photograph 125

Picea sitchensis. Penetration impeded by a parenchyma cell end wall, ponded saywood. X 600



Photograph 126

Picea sitchensis. R.L.S. Radial penetration of soid fuchsin into ponded sapwood. X 140

#### PART 5

#### Conclusions

The species of wood used in these investigations were selected because they covered arange of known treatability characteristics. The first objective was to examine laboratory techniques for determining wood permeability to see if any system could be found which would give results in line with known treatability characteristics, particularly in the treatment of natural rounds (such as poles) with water-borne preservatives. This was achieved with a piece of apparatus which recorded rates of uptake in 2 centimetre cubes of sapwood that were sealed to restrict penetration to one grain direction. Rates of uptake in the radial direction showed great variation between the eight species tested but these could be grouped into three classes of radial permeability which correlated well with a grouping based on treatability. Group I High permeability. Pinus radiata and Pinus nigra. These two species are known to treat very readily by vacuum/pressure and in these tests virtual saturation

was obtained in under 5 minutes with a treating pressure of 100 p.s.i.g. (pounds per square inch gauge pressure). Group II Intermediate permeability. Acathic australis and <u>Podocerpus dacrydicides</u>. Both these species had uptakes exceeding 50% of the possible maximum in under ten minutes treatment at 100 p.s.i.g. Both species are known to treat reasonably well with waterborne preservatives although <u>Podocarpus</u> dacrydioides is not approved for treatment with copper-chrome-arsenate preservatives in New Zealand because experience has shown that the final distribution of the preservative components is unsatisfactory.

Group III Refractory species. This group included the
remainder of the species - Facudotauga menziesii, Secucia
sempervirens, Cryptomeria japonica, Cupressus macrocarpa.
These species are all classed as refractory in commercial
treatment, and in the laboratory tests none had uptakes
exceeding 30% of the possible maximum after treatment for
one hour at 100 p.s.i.g.

These results could be grouped more simply by taking the uptake as a percentage of the possible maximum after a set pressure period. A suggested procedure for evaluating treatability in this manner is to coat 2 x 2 x 2 cm. blocks on five sides so that penetration is restricted to the radial direction, weigh the blocks, subject them to 15 minutes vacuum followed by 10 minutes treatment in water at a pressure of 100 p.s.i.g., remove the blocks and re-weigh them, and finally calculate the uptake as a percentage of the possible maximum. The classification would be made on the following basis. Group I Easily treated. Uptakes in excess of 50%.

Group II Intermediate treatability, Uptakes of 25 - 50%.

A system such as this would require only very simple apparatus consisting of a vacuum pump, a small pressure vessel, and a cylinder of compressed nitrogen. Several blooks of the same, or different species, could be treated simultaneously and a comparison of the treatability, or an assessment of the variability, could be made very quickly and simply. The same apparatus and procedure would be quite satisfactory in comparing the effectiveness of various pre-treatments designed to increase the permeability of pole-type wood.

When the pathways of radial penetration were studied by using interrupted treatments with dye solutions, it was found that several quite distinct systems operated with the different species. In the most permeable species (Pinus radiata, Pinus nigra and Agathis australis, the solution moved freely along the parenchyma cells of the rays. These cells are thin-walled and were often partially collapsed in the material used in these tests. Movement from the ray parenchyma cells to the axial tracheids took place through the crossfield pits which were easily ruptured by pressure, or were already ruptured in the dry wood. In the two pine species there was strong evidence that the crossfield pit membranes of the latewood region were already ruptured (probably as a result of stresses set up during drying) and this would account for the preferential penetration of the latewood bands. In Podocarpus dacrydicides

the parenchyma cells of the mays were thicker-walled and had end walls intact so that a free movement of liquid along the rays was not possible. The crossfield pit membranes were thinand easily ruptured however and it is thought that liquid moved under pressure from ray cells to tracheids, and then back again to other ray cells. The bordered pit membranes of Podocarpus were also thin and easily ruptured and this would undoubtedly assist penetration by allowing rapid tangential re-distribution. This pattern of penetration is much slower and more tortuous than that seen in the Pinus species and Agathis, and it is possible that it contributes to the premature precipitation of CCA salts (particularly copper) in Podocarpus dacrydicides wood. The same pattern of radial penetration is suggested for sitka spruce that had been stored in water for several months. With ponded spruce the uptake rate was much faster than in Podocarpus and this is explained by the fact that a considerable proportion of the crossfield nits were already ruptured, and the membranes of the bordered pits were entirely absent. Work is still proceding on the nature and mechanism of this pit membrane degradation but there seems little doubt that it is brought about by bacterial action. Water storage did not improve the permeability of the heartwood but the effect on the sapwood was so spectacular that it is a definite commercial possibility for improving the trestability of poles and other round wood commodities. Similar improvements in permeability have been claimed for other types of biological pre-treatment but ponding is considered superior to these because it is easier to control and safer. Bacteria do not appear to cause any loss of strength in the timber during the required ponding period, and the maintenance of virtual saturation precludes infection by any wood destroying fungi. Full ponding may not be possible in some areas because of a lack of suitable lakes and the cost of excavating artificial log ponds, but it may be possible to obtain the same results by the use of sprinklers. Other workers have shown that sprinklers have the same effects as ponding on the permeability of Pinus species and there seems no reason not to expect similar results with other more refractory species. Unpublished work by Princes Risborough Porest Products Laboratory indicates that temperature is important for initial colonization by bacteria but not so much for their later development. If this is so it would appear economically feasible to devise a system whereby poles could be stacked first under a sprinkler using re-circulated warmed water (possibly enriched with bacterial culture) and later transferred to ambient temperature sprinklers or even into block stacks well covered with polythene.

The Institute for Industrial Research and Standards of Ireland is currently investigating optimum ponding times at different seasons for sitks and Norway spruce poles, but so far there is no information on the minimum period required under ideal conditions. Wood samples taken from sitka spruce poles which had been ponded in Ireland for only two winter months (January and Pebruary) were found to have heavy bacterial infestation in the bordered pits and mys, but permeability measurements have not yet been carried out on this material. If the effect is as rapid as these samples indicate, it is possible that a considerable amount of the wood produced by normal commercial operations becomes infested, and in warm humid climates this could well explain the reputation some species have for variable treatability.

In unpended spruce and <u>Pseudotsuga mensicsii</u>, radial penetration was sevemiy restricted and the preferred pathways were usually the ray tracheids. The crossfield pits of both species were found to be obstructed by pit membranes and the bordered pits of the earlywood tracheids were aspirated. The latewood pits were unaspirated and this would allow lateral redistribution of liquid if some of the crossfield pitsin this region were ruptured. It is possible that the membranes of crossfield pits in the latewood region are sore easily ruptured because of seasoning stresses and this (aided by lateral redistribution) would explain the apparent barrier to ray penetration at the latewood boundary remarked on by Sarsent (1960).

In <u>Cryptomeria japonica</u>, <u>Sequoia sempervirens</u>, and <u>Cupressus macrocarpa</u>, radial penetration was still further restricted and the only advance penetration of the rays was in the intercellular spaces between the ray parenchyma cells. In Cryptomoria and Sequois the liquid appeared to move through the parenchyma cell wall, to the cell lumen, and also to the region of the crossfield pit aperture. From here it apparently penetrated the axial tracheids without rupturing the crossfield pit membranes. In Sequois it appears possible that axial parenchyma cells may have played mose role in redistribution from the rays but it was not possible to establish this definitely. In Cupressus there was a movement of liquid from the intercellular spaces in the rays, to the intercellular spaces between the axial tracheids, but there was no evidence seen of movement from these to the tracheid lumens.

Thus in these three refractory species, the pathways of radial penetration are still somewhat obscure but it appears that some movement through cell walls may be involved and this would probably explain the relatively slow rates of uptake. As far as is known no ponding or other biological pre-treatments have been attempted with these species. The pit membranes are fairly robust, but not appreciably more so than in spruce, and if bacterial attack could destroy these there seems no reason why water storage could not affect a similar improvement in treatability.

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## APPENDIX I

### Origin, Preparation, and Physical properties of the Wood Samples.

## . Pinus radiata

Origin - Kangaroa Forest, New Zealand.

Tree age - 35 years.

Sawing and seasoning - Cut from butt log, air seasoned completely

Mean basic density - 0.419.

Mean moisture content - 13.0% (after conditioning)

Number of rings per inch - 6.0

Approximate percentage of latewood - 38%

### 2. Pinus nigra (Corsican)

Origin - Kaingaroa Forest, New Zealand.

Tree age - 30 years

Sawing and seasoning - Air dried, details unknown.

Mean basic density - 0.432

Mean moisture content - 10.7%

Number of rings per inch - 7.6

Approximate percentage of latewood - 43%

## Pseudotsuga menziesii

Origin - Kaingaroa Forest, New Zealand.

Tree age - 40 years

Sawing and seasoning - as for Pinus radiata

Mean basic density - 0.400

Mean moisture content - 10.9%

Number of rings per inch - 6.3

Approximate percentage of latewood - 48%

## Sequoia sempervirens

4.

Origin - Compartment 20, Whakarewarewa Porest, New Zealand.

Tree age - 58 years

Sawing and seasoning - as for Pinus radiata

Mean basic density - 0.256

Mean moisture content - 10.1%

Number of rings per inch - 3.6

Approximate percentage of latewood - 15% (plus a distinct zone of intermediate type of 45%)

### 5. Cryptomeria japonica

Origin - Compartment 3, Whakarewarewa Forest, New Zealand.

Tree age - 55 years

Sawing and seasoning - as for Pinus radiata

Mean basic density - 0.2%

Mean moisture content - 8.9%

Number of rings per inch - 13

Approximate percentage of latewood - 26%

#### 6. Cupressus macrocarpa

Origin - Compartment 12, Whakarewarewa Forest, New Zealand.

Tree age - 31 years

Sawing and seasoning - as for Pinus radiata

Mean basic density - 0.338

Mean moisture content - 11.5%

Number of rings per inch - 4.6

Approximate percentage of latewood - 20%

#### . Agathis australis

Origin - Herekino State Forest, Kaitaia, New Zealand.

Tree age - 150 - 200 years

Sawing and seasoning - Air dried, other details not known.

Mean basic density - 0.440

Mean moisture content - 11.7%

Number of rings per inch - 13

Approximate percentage of latewood - 22%

## 8. Podocarpus dacrydioides

Origin - Punakitere S.D., Northland, New Zealand.

Tree age - 40 - 50 years.

Sawing and seasoning - Trees cut and air dried as fence posts.

Seasoned partly under cover.

Mean basic density - 0.358

Mean moisture content - 12.9%

Number of rings per inch - 13

Approximate percentage of latewood - 15% (very indistinct)

### 9. Picea sitchensis

Origin - Killeshandra Forest, Cavan, Eire.

Tree age - 36 years

Sawing and seasoning - Cut as poles. half of which were ponded for 6 - 11 months. Air seasoned on racks outside.

	Fresh Sap.	Ponded Sap.	Fresh Heart.	Ponded Heart.
Mean basic density	0.317	0.348	0.328	0.352
Mean moisture content	12.9%	13.4%	14.8%	13.4%
No. of rings per inch	5.0	6.4	2.9	8.0
Approx. % of latewood	30%	30%	27%	29%

APPENDIX II

Uptake rate compared with swelling rate for vacuum treated unsealed blocks of Pinus radiata.

	Time (minutes)	Uptake as % of total	Swelling as % of total in the tan- gential direction
Vacuum 26"Hg	0	0	0
Water admitted	10	0	0
Block covered	10.5	0	5
Vented to atmosphere	11	0	20
	11.5	44	36
	12	73	49
	12.5	81	66
	13	88	89
	14	92	95
	15	94	98
	16	96	98
	18	98	100

Total Uptake represented approximately 125% of the untreated weight of the block.

Total swelling in the tangential direction represented approximately 6% of the total swelling which represented approximately 9% of the untreated volume of the block.

Presumably blocks increase in weight as soon as they are wetted but because swelling commences immediately the density does not alter significantly, and the change does not register on the recorder.