Unsteady-state flow of light organic solvent wood preservatives into refractory softwood

A thesis presented in part fulfilment of the requirements for the degree of Doctor of Philosophy of the University of London

by

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Volume 1, of 2

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Abstract

European whitewood comprises a significant proportion of the world's resource of structural timber, but is susceptible to biological degradation. Many similarly susceptible timbers can be protected by impregnation with a solution of biocide(s) in an organic solvent such as white spirit. European whitewood cannot satisfactorily be impregnated deeply with such preservatives, wherefore this investigation was undertaken, having the following aims:

Development of techniques to monitor the fluid flow phenomena which occur during the process of timber treatment.

Examination of the 'standard' technique for the impregnation of whitewood with organic solvent preservative to obtain precise information about its performance.

Investigation of means whereby the techniques for the industrial impregnation of whitewood may be improved.

All three aims were fulfilled to a substantial extent. Four techniques were developed to examine aspects of flow and these yielded results which, in several respects, were not consistent with established theory.

Investigation of variation in the rate of flow during the immersion phase of "industrial" treatments revealed flow rate to decrease less rapidly than expected, and work using a purpose built flow meter did not support the classic proposition that flow is impeded by air embolisms.

Measurement of pressures within whitewood yielded new information about the behaviour of fluids within this timber.

Examination of samples treated using a range of schedules generated information about the characteristics of spruce and about the performance of the 'standard' spruce treatment technique. It also indicated how this might be improved by reducing uptake without producing a corresponding drop in penetration.

Overall, the results indicate that flow of organic solvent into whitewood does not necessarily proceed by sequential filling of successive voids, but may involve mechanisms which result in the walls of lumena becoming coated with impregnant liquid without the lumena becoming totally filled.

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Dedication

to

Colin Kyte

Jean Taylor

Professor John Levy

and especially

my wife, Jan

1 INTRODUCTION

1.1 Liquid flow into timber - its importance

Throughout recorded history timber has been a commodity highly prized as a structural and building material. Sumerian poetry dating back probably to the 2nd millenium B.C. records the adventures of the young King Gilgamesh of Uruk in Mesopotamia (now Iraq). Soon after acceding to the throne Gilgamesh went to war with a neighbouring kingdom in order to obtain timber for building in his capital city (Anon ca. 2500 B.C.).

It passes unrecorded precisely how Gilgamesh utilised his hard-won timber. It is possible he may have taken some active measures to conserve it because there are scattered references in other ancient manuscripts which imply a rudimentary knowledge of timber preservation, or at least of timber conservation (Genesis 6, I Kings 6). In any event it is beyond reasonable doubt that he was vastly less extravagant with it than were the Scottish chieftans who, about a thousand years later in around 55 B.C., frustrated Caesar's forces by encamping within fortifications which typically involved 6,400 linear metres of 23 cm square section timber (Clark & Piggott 1965).

Such extravagance with timber was of little consequence in areas where it was abundant and, until the Industrial Revolution, builders and architects throughout Europe were able to compensate

for imperfectly developed civil engineering skills by using timbers in oversized dimensions.

The growth of European Navies in the 17th century brought with it what appears to have been Europe's first experience of demand for suitable timber locally outstripping supply. This situation resulted not only from the need for more and more ships to enlarge fleets, but more from the fact that, once built, these ships seldom had a working life of more than seven years. East India Company vessels rarely made more than four return trips to India before becoming useless through decay (Richardson 1978). Even so, the realisation that Europe's timber resources were exhaustible was slow to dawn and Defoe, writing in 1724, dismissed fears that smelting works were using timber faster than it could be replaced, asserting that Kent, Sussex and Hampshire were "one inexhaustible storehouse of timber" (Richardson 1978). History has of course shown him to have been wrong. As the social and economic fabric of Britain was changed by the first industrial revolution, the demand for timber vastly outstripped local supply and increasing quantities of softwood were imported from the Baltic and Canada.

On land, as at sea, new constructional timber was required not only for new sites, but also for the replacement of decayed timber in existing structures. Consequently the desirability of minimising decay became increasingly clear - a fact evidenced by the offer made by the Royal Society in 1784 of a gold medal 'for the

discovery of the various causes of dry rot in timber and a certain method of its prevention' (Richardson 1978).

Although this medal was awarded ten years later, the 'various causes of dry rot' were not satisfactorily discovered. The issue was inevitably clouded by the then extant theory of spontaneous generation and wood technologists of the day debated whether timber decay could be prevented by such precautions as 'precipitation of the albumens' (Boulton 1884).

Today, however, timber decay is recognised to be due to the action of saprophytic organisms which, by breaking down timber, help to maintain the ecosystem in dynamic balance. It follows therefore that if timber is to be 'removed' from the ecosystem for a time while it is utilised by man as a constructional material, 'a certain method of decay prevention' is to interrupt this natural process and arrest the action of these organisms.

Simply keeping timber 'dry' (i.e. below 20% moisture content expressed on a dry weight basis) is in itself an important facet of timber protection because wood destroying fungi are able to degrade only 'wet' timber (Cartwright & Findlay 1958). However, timber is a hygroscopic substance and takes up an equilibrium moisture content with its surroundings. Where its end use is, for example, as fence posts, transmission poles or railway sleepers, maintenance of a dry state is not (yet) a very practical approach to prolonging service life.

When timber is used out of ground contact in building components such as window frames and external doors, keeping it dry is theoretically a more feasible proposition. Regrettably, common experience is that, in the context of current U.K. building practice, it is seldom possible to predict with adequate confidence that dryness of the timber in such components will continuously be maintained throughout the 20 - 50 years of their required service life (Tack 1968). It is thus frequently necessary to modify the timber itself so that despite the existence of favourable physical conditions, fungi and insects are unable to thrive.

One of the oldest (e.g. Kyan 1832), and still to date the most widely employed, techniques for effecting this modification is to deposit within timber fungicidal and/or insecticidal chemicals. These are almost invariably carried into place in liquid solution and left behind as the carrier solvent evaporates. However, this simple, broad summarisation of the 'art of timber preservation' belies the many technical difficulties associated with transporting suitable chemicals into place in liquid solution. Although liquid movement occurs readily through timber 'in-vivo', maturation and post-harvesting changes in the timber often result in physical impediments to the flow of preservative solutions through harvested timber. The nature of these impediments is such that timbers such as the Spruces (Picea spp) and many Eucalypts (Eucalyptus spp) cannot yet be reliably impregnated to impart deep and long lasting protection against micro organisms in high hazard situations (Morgan 1975, Cockcroft 1976, Aston & Watson 1976).

The object of the work described in this thesis was to investigate what happens during attempts to impregnate the relatively impermeable timbers with preservative solutions. More specifically the work was carried out to attempt to gain an insight into the flow of liquid carrier solvents, especially the non-polar 'white spirit' type, into refractory softwood.

1.2 Anatomy of timber

Much is already known about the flow of liquids through permeable timbers. However, preparatory to reviewing the details of this, it is necessary to consider the nature of timber, particularly in terms of the functional and spatial relationships of its component parts.

1.2.1 Timber - its origins and types

Timbers used for building purposes are derived from the accumulated woody tissue, or secondary xylem, produced annually by many perennial plants. During the growing season a band of meristematic tissue, termed the cambium, which is located just below the bark, divides repeatedly by the formation of a longitudinal, ca. 1 μm thick layer (the middle lamella) in the mid tangential plane (Fig 1.1). Down either side of this is deposited a ca. 0.1 μm layer of cellulose (the primary wall). Some of the new cells so formed develop into 'phloem', a transient tissue which translocates metabolites to the roots of the tree. The remainder give rise to a succession of more permanent 'xylem initials' at the periphery of the existing woody tissue. Most of these xylem initials develop a three-layered secondary reinforcing wall within the wall (Fig 1.2). The cell contents are then lost and functional secondary xylem results (Jane 1970).

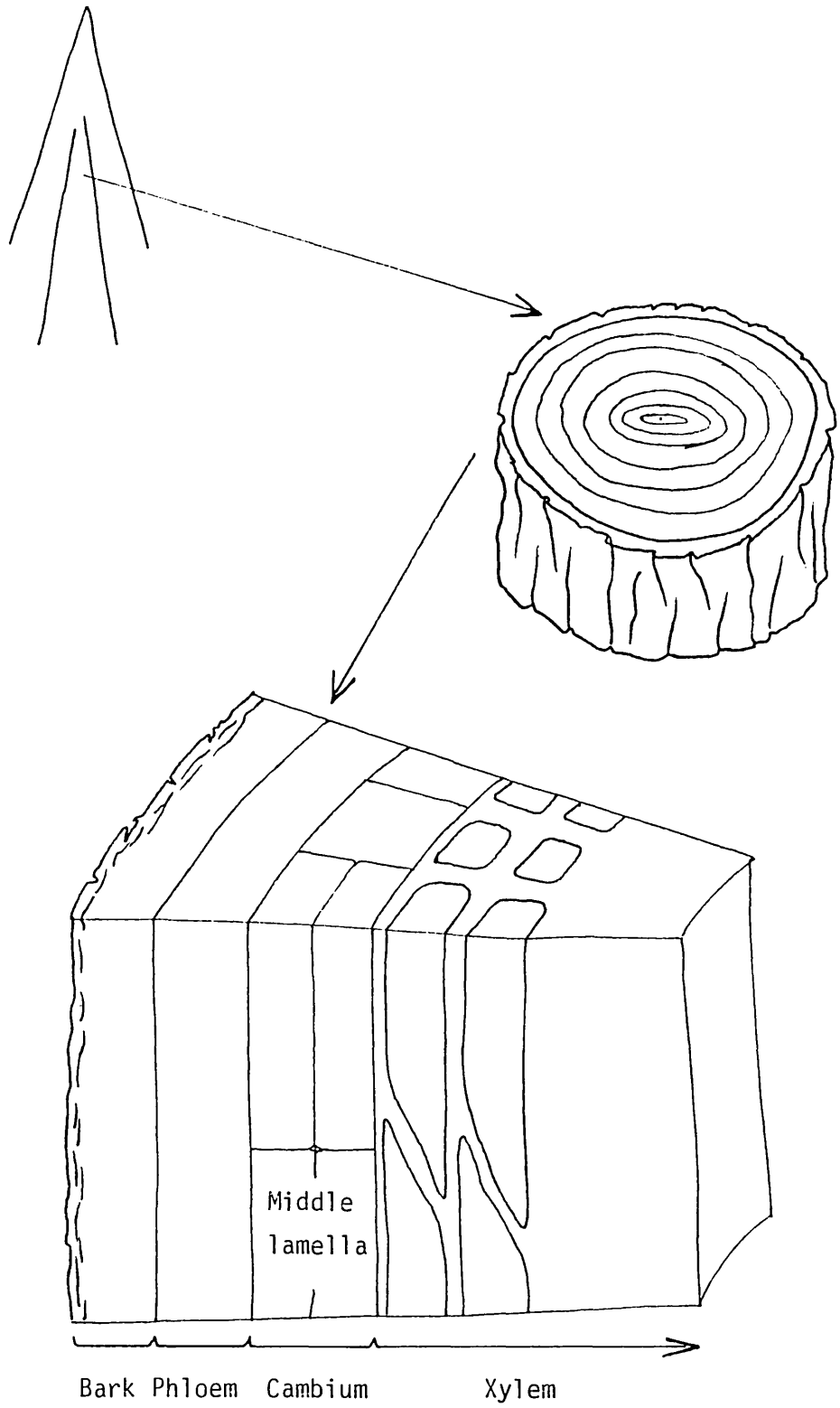


Figure 1.1 Schematic diagram (not to scale) showing position of cambium and middle lamella in the growing tree

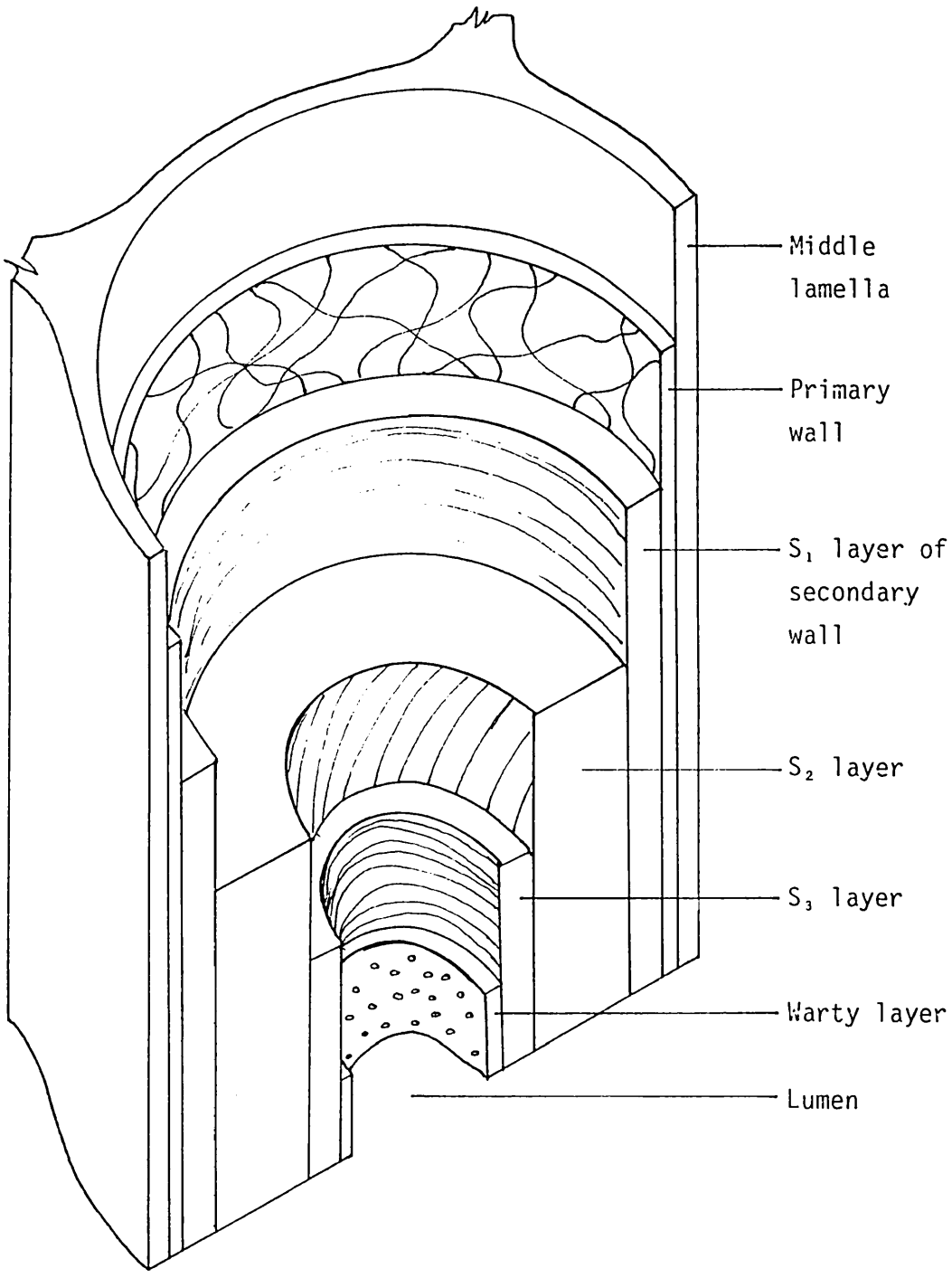


Figure 1.2 Generalised schematic diagram (not to scale) of the composition of the wall of a woody xylem cell after secondary thickening (After Ward et al, 1964)

By convention, timber from gymnosperm trees, examples of which are pines, spruces and firs, is termed 'softwood'. Conversely, timber from woody angiosperms i.e. broad leaved trees such as oak, obeche and eucalyptus is termed 'hardwood'. Softwoods and hardwoods are distinguished solely on the basis of their origin and anatomical structure and not, as might reasonably be supposed, by their mechanical characteristics.

In living softwood tree species, the functions of support and water transport are both serviced by a single cell type. In hardwoods, on the other hand, two distinct tissues fulfil these functions. This basic difference has important implications in relation to timber impregnation and it is thus apposite to describe the microscopic structure of softwoods and hardwoods more fully.

1.2.2 Softwood structure

The basic units of softwood structure are termed 'tracheids'. These serve for both support and liquid transport and their generalised morphology and size are indicated in Fig 1.3. From Fig 1.3 it may be noted that the tracheids are closed, roughly cylindrical structures with tapered ends. There is only a negligibly small amount of space between tracheids and so water movement occurs through their lumena. Passage of liquid from one lumen to another must occur via the pits (q.v. 1.2.2.1). One cubic metre of softwood may contain between 10^{10} and 10^{14} tracheids, the vast majority (>90%) of which are axial, i.e. they are each aligned with their long axis parallel to the longitudinal axis of the tree or branch of which they form a part. Their ends overlap somewhat (Fig 1.4) and they are bonded together such that timber has its greatest tensile strength in the longitudinal direction.

Running radially from the centre of the tree to its periphery, at right angles to the axial tracheids is a small amount of radial tissue which may comprise either fairly undifferentiated cells (parenchyma), or more tracheids. Like the axial tracheids, the radial tissue also serves for the transport of water and materials in solution.

The collective term for each discrete group of radial tissue is a 'ray'. The number and arrangement of tracheids and other

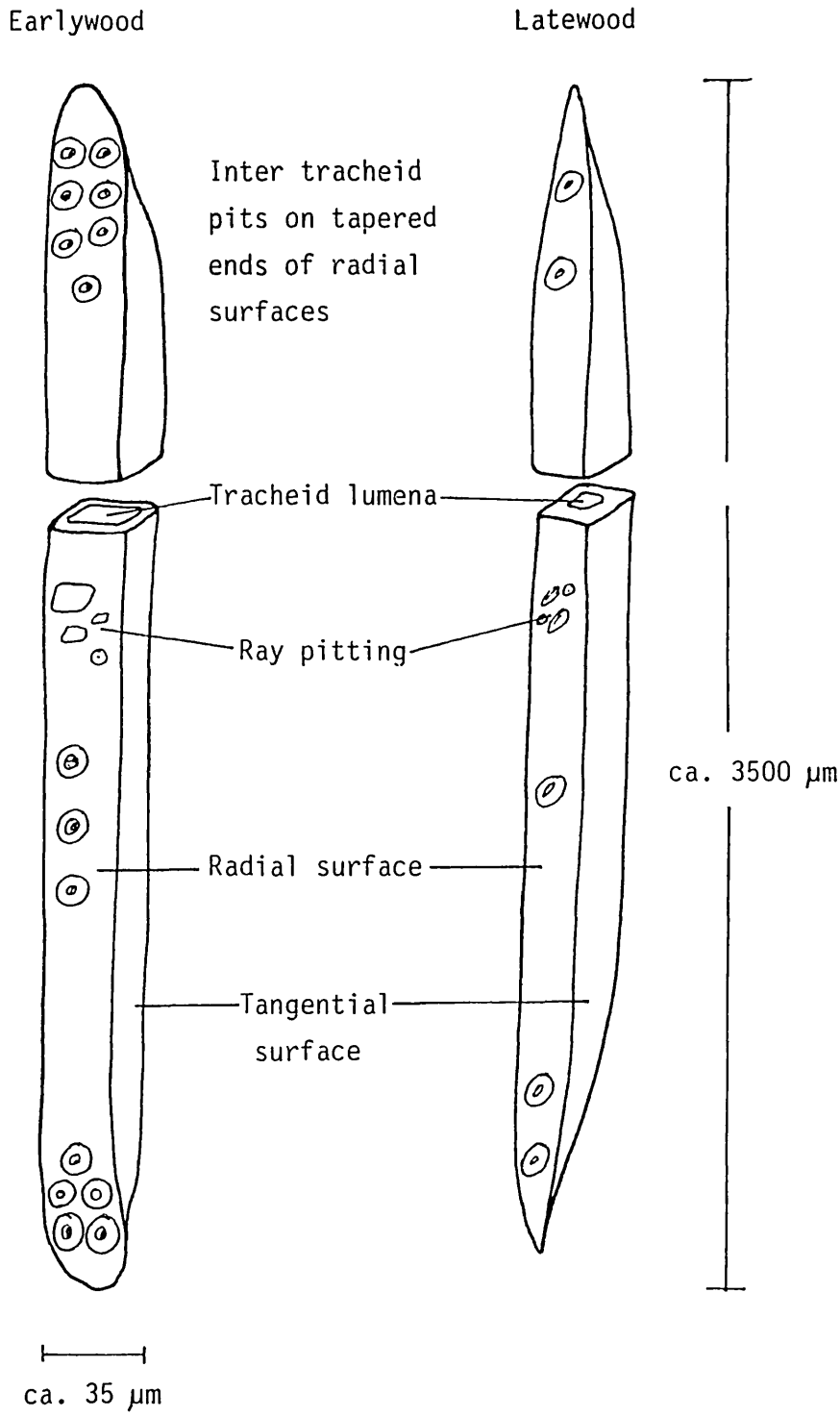
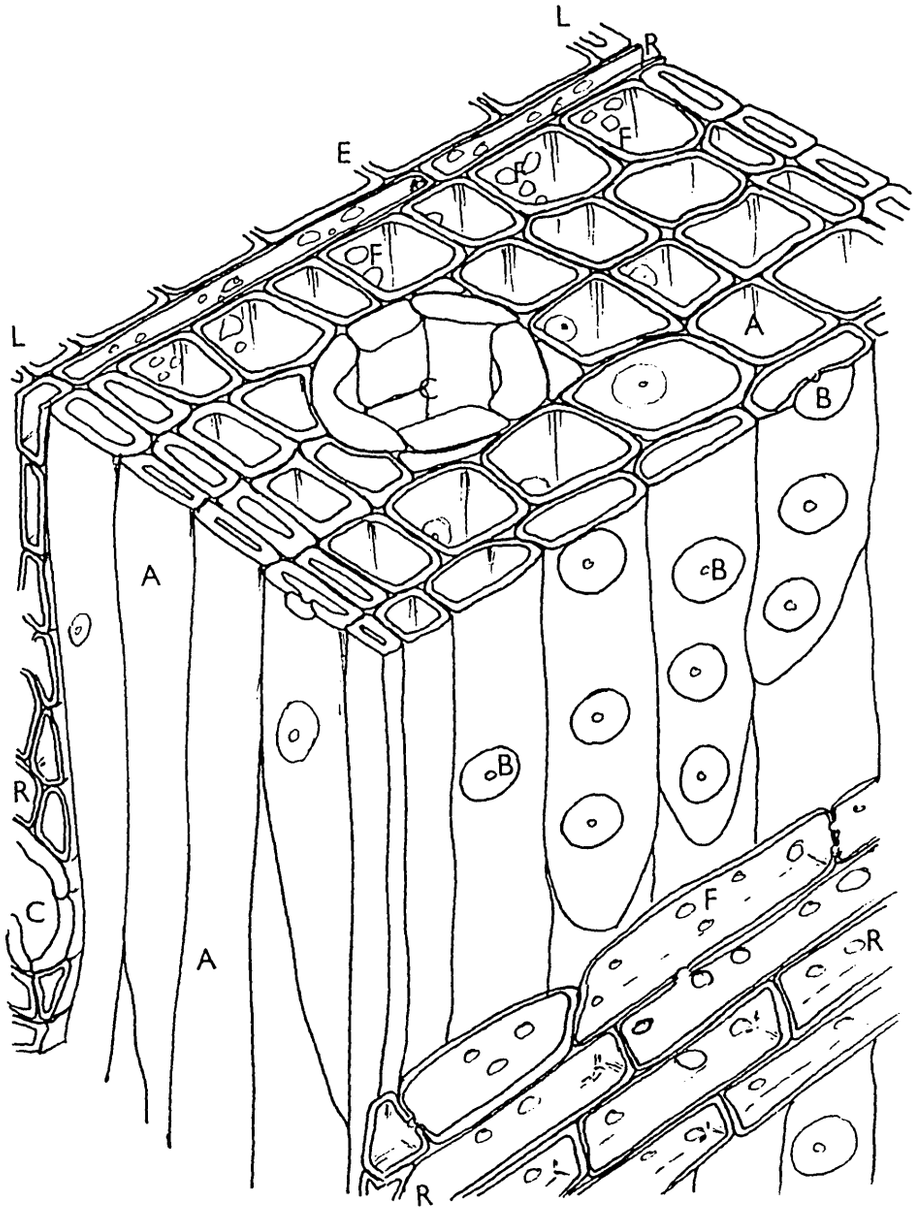


Figure 1.3 Generalised morphology of softwood tracheids (After Howard and Manwiller 1969)

Figure 1.4 Schematic diagram of generalised gross structure of a softwood - after Panshin and Zeeuw (1980)



Key A - Axial tracheid
B - Bordered pit
C - Resin canal
E - Earlywood
L - Latewood
R - Radial tissue

tissues, such as parenchyma, in a ray is a characteristic of the species of timber and is thus useful for identification.

Other features which are present in many commercially important softwoods are 'resin canals' (Fig 1.4). Typically, these are small axial and/or radial channels occurring at intervals between the tracheids. Each is lined with a layer of resin secreting epithelial cells. The functional significance of this resin in the living tree is somewhat obscure. In harvested timber, resin and like substances may markedly enhance the timber's natural resistance to decay and often decrease permeability.

1.2.2.1 Pits

From Figures 1.3 and 1.4 it is clear that a tracheid is a closed, roughly cylindrical structure with tapered ends. Tracheids have however as one of their two main functions 'in vivo' the transport of water up the tree. Thus water must be able to move from tracheid to tracheid.

It was at one time considered that this movement occurred by a diffusion mechanism (Tiemann 1910) but it was later shown by Bailey (1913) that this was not so. Instead, mass flow occurs through perforations in the secondary wall, descriptively termed 'pits'. Phillips (1933) estimated that, depending upon species, there are 50 to 100 pits in each tracheid. These are predominantly located on the tracheid's radial walls, particularly in the tapered region

where two tracheids overlap.

Morphologically, the simplest pits are located in the parenchyma (a tissue restricted to the rays in most softwoods). In this tissue the pits are usually roughly circular regions in which the primary wall is not overlayed by secondary wall. The exposed primary wall is termed the 'pit membrane' and two complementary pits in adjacent cells which share the same pit membrane are called a 'pit-pair'. (Most pits are paired.)

In inter-tracheid walls, the pits are not of this 'simple' kind. The secondary wall over-arches the pit membrane (Fig 1.5) forming a semi-enclosed pit cavity or chamber which is in communication with the tracheid lumen via the pit aperture. The whole is termed a 'bordered pit'.

In the Gnetales and Coniferales, a 'torus' or thickened disc forms on the central region of the pit membrane, its diameter being slightly in excess of that of the pit aperture. In some species, e.g. Thuja sp., the thickening is minimal while in Cedrus deodora for instance, a torus several microns thick develops.

Siau (1971) is among authors who have postulated that the torus may be slightly permeable to liquids. However, for all practical purposes tori of most commercially important species can probably be considered impermeable to liquids (Phillips 1933).

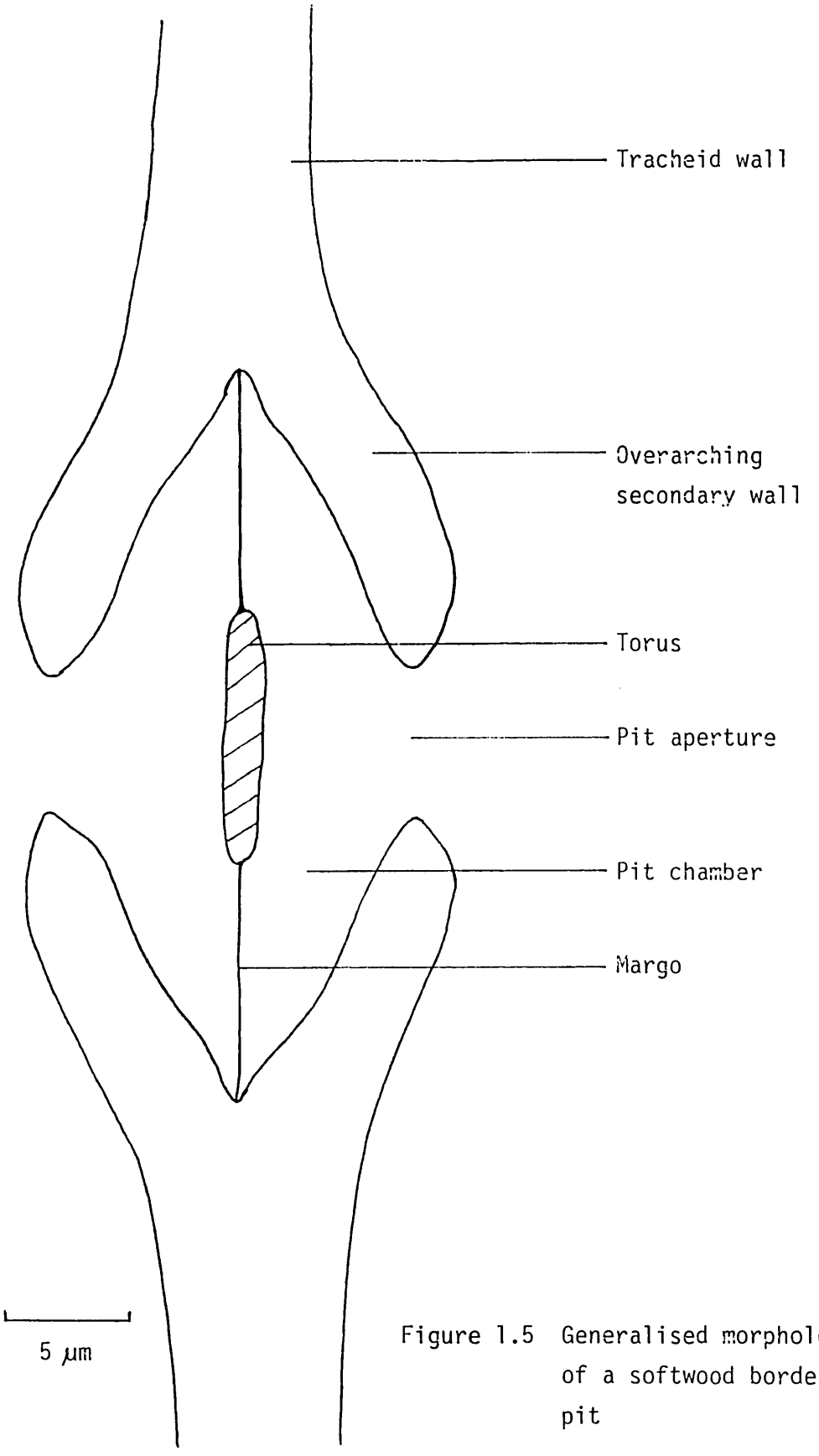


Figure 1.5 Generalised morphology of a softwood bordered pit

Around the torus, the remainder of the pit membrane (the margo) is unthickened. Instead it consists of a reticulum of thin strands. In species such as Abies grandis these are supplemented with thicker, often radially-arranged strands which run from the edge of the membrane to the torus, supporting the latter (Petty & Puritch 1970). The size of the pores in the membrane is variable and largely species-related. Typical values quoted include 0.2 μm dia. for Abies grandis and 0.28 μm dia. for Picea sitchensis (Petty & Puritch 1970 & Petty 1970 respectively). It is through these pores that liquid flow occurs.

1.2.2.2 Bordered Pit Aspiration

Figure 1.5, a diagramatic section of a bordered pit pair, shows the torus in the position it is assumed to occupy in water-conducting wood. Water is able to pass from one pit chamber to the next via the pores in the margo. The torus is suspended between the two pit borders and liquid flows around it. However, if through some cause, the torus becomes displaced and adpressed to either pit border, the pit pair is said to be 'aspirated' (Fig 1.6) and liquid flow is either severely restricted or totally prevented (Bailey & Preston 1970).

It is now generally accepted that the aspiration of pits is the main factor causing diminished permeability in softwoods (Hunt & Garratt 1953). Petty (1978c) has shown that if pit aspiration

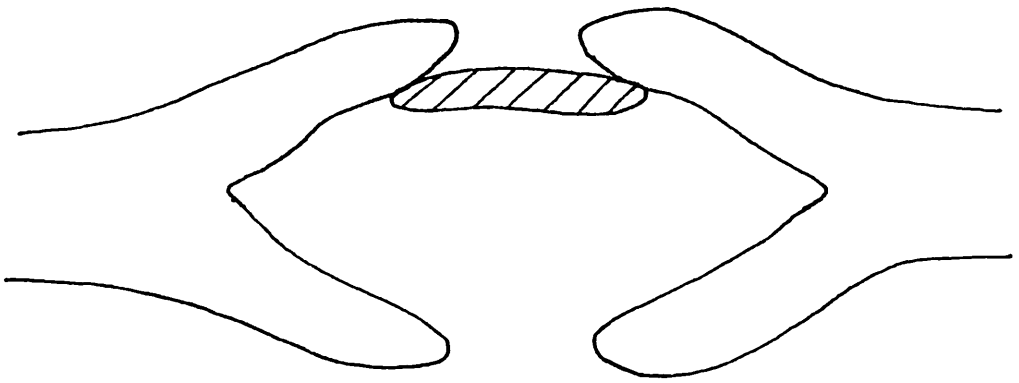


Figure 1.6 Diagramatic representation of aspirated bordered pit showing displaced margo occluding pit aperture

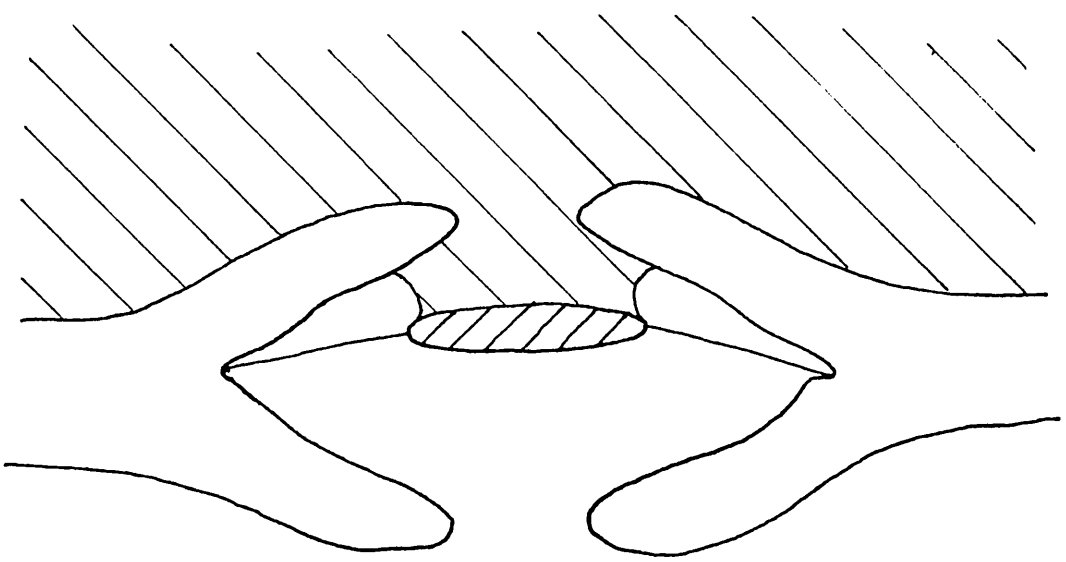


Figure 1.7 Diagramatic representation of bordered pit during final stages of drying towards fibre saturated point, showing torus being drawn towards the pit aperture by surface tension of the retreating liquid surface

during drying is prevented, the timber's permeability remains high. Conversely Bailey & Preston (1970) and Bolton & Petty (1978a) have suggested that if aspiration is induced mechanically such as by the application of a high hydrostatic pressure, permeability decreases markedly.

Hart & Thomas (1967) proposed a causal mechanism for natural pit aspiration which has since found general acceptance (Bailey & Preston 1970, Petty 1977). Their proposed mechanism arises from the observations such as those of Phillips (1933) that pit aspiration occurs during drying as fibre saturation point is reached i.e. at the point when, with all the cellulose fibres fully saturated, the last free water is lost. When this occurs a meniscus must pass through the pit (Fig 1.7). Hart & Thomas postulated that the interfacial tension between the water and the pit components is sufficient to pull the torus to one side of the chamber. Bailey & Preston (1970) have shown that the energy available from this source is adequate to overcome the mechanical forces in the radial strands of the margo of Douglas Fir. Petty (1972) and others have noted that pit aspiration may not always occur in latewood. These authors have postulated that the margo fibrils in latewood pit membranes are more rigid than those in the earlywood and thus the torus is less readily displaced. Petty (1972) further suggested that the occasional aspiration of latewood pits which may sometimes be observed is the result of the miniscus passing through the pit sufficiently slowly for 'creep' to occur in the stretched

cellulosic strands until finally they allow the torus and pit border to touch.

Comstock & Cote (1968) showed that high interfacial tension is not the only prerequisite characteristic of the liquid if pit aspiration is to occur. Other workers (Thomas & Kringstad 1971, Petty 1972) have confirmed Comstock & Cote's view that for permanent aspiration, the liquid must be a medium in which hydrogen-bonding of the membrane to the pit border can occur.

1.2.2.3 Cross field pits

In softwoods, each axial tracheid is in contact with at least one radial cell at some point along its length. At this point of contact, there occurs one or several 'cross field pits'. The size and morphology of these is quite variable (Phillips 1948). In the pines for instance (Plate 1.1) the pits are large and window-like. The spruces however have very much smaller cross field pits (Plate 1.2). All cross field pits are 'simple' and thus have neither thickened pit membrane, nor overarching secondary walls.

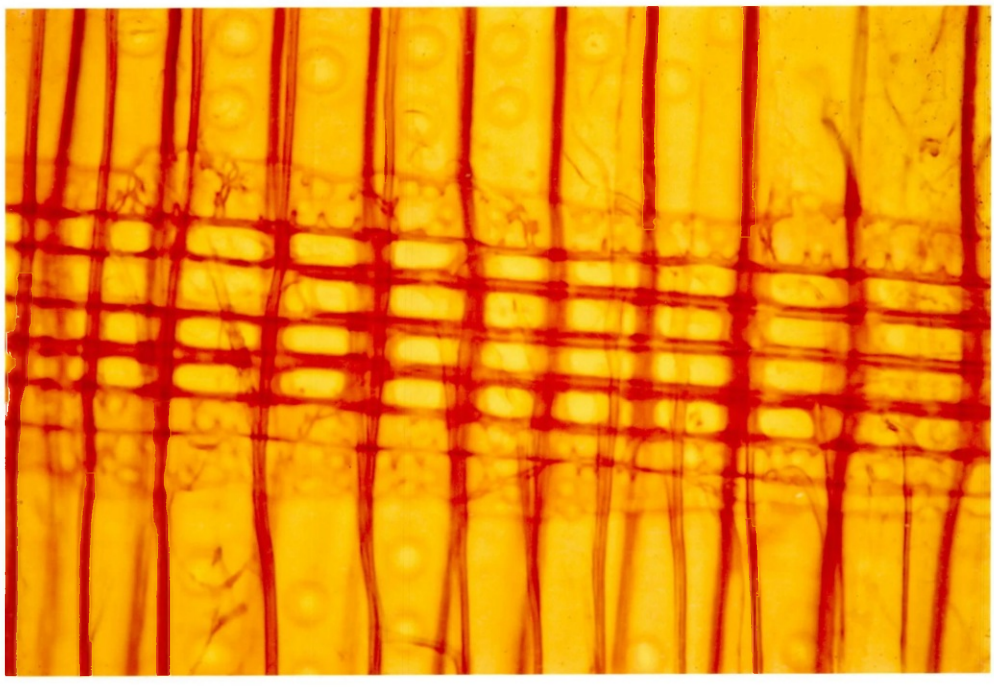


Plate 1.1 Cross field pits of Pine
Section 1.2.2.3 refers



Plate 1.2 Cross field pits of Spruce
Section 1.2.2.3 refers

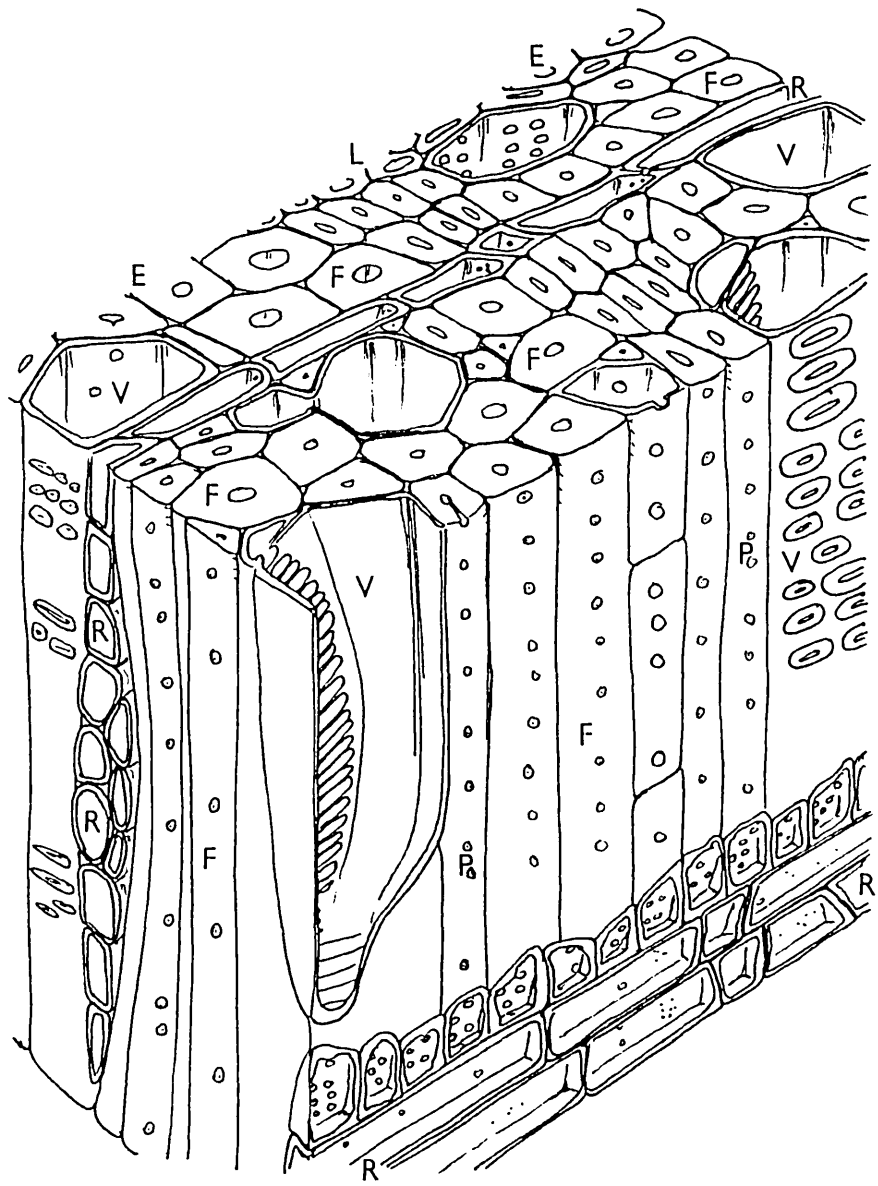
1.2.3 Hardwood structure

Hardwoods,i.e. those timbers derived from angiosperm trees, display a much greater variety of microscopic structure than do softwoods. Whereas in softwoods the axial tracheids comprise the main bulk of mature secondary xylem and serve for both mechanical support and water transport, in hardwoods these two functions are serviced by two distinctly different tissues.

The channels for axial and radial water flow are provided by wide and relatively thin-walled 'vessels' (Teesdale & Maclean 1918) which constitute seldom more than 60% (by volume) of the tree and frequently much less. A xylem vessel is a continuous tube derived from a column of cells. Each cell is shorter relative to its width than are tracheids. The upper and lower end walls are much less oblique than those of tracheids and may be close to transverse in many species. These end walls are always perforated - sometimes so much so that the vessel lumen is barely constricted. Vessel diameters vary widely, mostly in the range 15 to 500 μm.

Between these vessels are narrow, thick-walled fibres which do not significantly conduct water but instead provide mechanical strength (Fig 1.8). These fibres, strictly termed libriform fibres, are much longer and thinner than the component cells of the vessels. They often, though not invariably, have heavy secondary thickening and although tori do not form on the membranes of hardwood bordered pit pairs,the pit membranes in the bordered pits are apparently imper-

Figure 1.8 Schematic diagram of generalised gross structure of a hardwood - after Panshin and Zeeuw (1980)



Key E - Earlywood F - Fibre
L - Latewood P - Pits
R - Radial tissue V - Vessel

forate. Siau (1971) notes evidence that hardwood bordered pit pairs are permeable to fluids and thus suggests that tortuous flow paths may exist between microfibrillar strands. (Rudman (1965) noted fluid movement actually within the walls of dry Eucalyptus spp although this pathway appears only to be available to polar solvents[1]. The following year the same author published evidence of the existence of fine capillaries in the walls through which flow occurs (Rudman 1966).) However, in general, fibres and wall material do not contribute significantly to liquid flow pathways.

Other tissues found in relatively small quantities in hardwoods include parenchyma, which is rather more abundant than in softwoods, tracheids (similar to softwood tracheids) and fibre tracheids.

[1] The observation by Rudman that only polar solvents can flow through the cell wall is consistent with the earlier work of Stamm and Tarkow (1950) who also concluded that dry cellulose fibres can only be penetrated by solvents of this sort.

1.2.4 Softwoods and Hardwoods compared

In the context of preservative liquid impregnation, softwoods and hardwoods are fundamentally different materials.

Depending upon species, hardwoods are usually composed of up to about 60% by volume of fibres. These cells do not conduct water to any significant extent after reaching maturity and by their nature are inherently impermeable. Each fibre has a comparatively small lumen and even if preservative were to fill the lumen during impregnation, it might still be an inadequate loading to prevent biological degradation throughout the fibre wall. However, it is the fibres which contribute most to the strength characteristics of the timber and which therefore are most in need of effective preservative treatment.

Softwoods on the other hand are more uniformly porous and even latewood tracheids (q.v. 1.2.5) have a relatively large lumen relative to the thickness of their walls. Living softwood sapwood is quite permeable throughout and liquid movement occurs through all tracheids. Although maturation and post-harvesting changes such as resin deposition and pit aspiration may substantially diminish a softwood's permeability, it remains a highly porous material.

1.2.5 Growth rings

Longitudinal division of the cambium in both softwoods and hardwoods produces xylem initials which mature to produce secondary xylem. Although this division and maturation process occurs repeatedly, it does not usually do so at a continuous steady rate. Usually this activity is related to the rate of growth of the tree during growing seasons, the length and frequency of which depend on local climatic conditions.

At the beginning of a growing season the xylem initials produced are large in radial cross-section and during their maturation, comparatively little secondary thickening occurs. Thus the resultant tissue, termed 'earlywood'[1], is capable of rapidly conducting water to the newly developing foliage.

Towards the end of the season 'latewood' is formed. The xylem initials diminish in radial cross-section and in comparison with the earlywood, the latewood maturation phase involves more secondary thickening of each lumen wall. Hence a band of rather stronger tissue with a smaller lumen volume is produced. In hardwoods, vessels formed in latewood are often smaller than in earlywood and fibres predominate.

[1] The terms 'spring wood' and 'summer wood' are respectively synonymous with 'earlywood' and 'latewood'; however, the use of the former terms is to be deprecated owing to their inapplicability to tropical situations.

As a result of this cyclical, seasonal variation in cell morphology trees are composed of roughly concentric layers of alternately dense and less dense tissue and this can usually be clearly seen on a cross-cut stem where the narrow bands of dense latewood contrast with the usually rather wider bands of earlywood. In dried harvested timber, the earlywood and latewood may differ not only in porosity but also in permeability. Consequently, differences in the way liquid flows through these tissues are sometimes encountered. For example in hardwoods, the wider vessels of earlywood may be the most easily penetrated pathways for liquid flow. Conversely in Pinus radiata and fast grown Picea sitchensis, the latewood is often more readily penetrated than the earlywood because as has already been mentioned the pit membranes are more rigid in latewood and pit aspiration occurs less frequently (1.2.2.2).

1.2.6 Heartwood and Sapwood

When it is first produced, the role of xylem in providing a pathway for movement of water or 'sap' up the tree is of paramount importance. However, in most tree species, the water requirements of the foliage of a mature tree can adequately be serviced by a relatively few years' xylem. In consequence the oldest xylem (in the centre of the tree) falls into disuse so far as water transport is concerned. It is then termed 'heartwood' to distinguish it from the peripheral water conducting 'sapwood'.

At the time when sapwood becomes heartwood, a number of changes occur. The cells empty of water and for reasons already outlined this induces pit aspiration in many softwood tracheids, especially in the earlywood. Coupled with this, permeability of the pits is often still further reduced by the deposition of resin and other material. In many species (e.g. Thuja plicata) this resin deposition contributes to a clear colour distinction between heartwood and sapwood. In others, such as Pinus radiata, the colour change may be less marked and the use of reagents is sometimes required to show where the heartwood and sapwood meet (e.g. Stalker 1971). Species such as Picea spp on the other hand show so little resin deposition that, after drying, it is at present impossible to distinguish clearly between heartwood and sapwood on visual or chemical criteria.

Deposition of gums and other chemicals occurs during heartwood formation in hardwoods. Ebony, for example, has a white/grey sapwood but the deposition of extractives imparts to its heartwood the characteristic black colour.

The vessels of hardwoods usually present channels of easy passage for liquids in hardwood sapwood, but heartwood formation is often accompanied by the adjacent parenchyma cells growing into the vessels and forming 'tyloses' which occlude the pathways and often totally prevent flow of liquid. If the pits in the vessel wall are permeable, the tyloses may be by-passed. However, if the pits are covered by encrustations, such as are observed in black gum (Nyssa sylvatica), flow through these will be impaired also. In hickory (Caraya tomentosa), blockage of vessel lumena by tyloses, and encrustations around the pits so reduce flow that the fibres become the principle flow paths (Thomas 1976).

In the mature tree, the sapwood generally remains of roughly constant width, i.e. heartwood is formed at about the same rate as new sapwood is produced by the cambium. In some trees, e.g. Pinus sylvestris, the boundary between heartwood and sapwood is sharply defined and follows a single growth ring. In other species such as radiata pine the heartwood/sapwood transition is less distinct and the process of heartwood formation presumably therefore takes two or three seasons. In Larch, the distinction between heartwood and sapwood is usually fairly clear, especially in freshly cut wood but is often irregularly developed across four or five growth rings.

1.3 Fluid flow - with particular reference to pipes

From Section 1.2 it is clear that softwoods and, to a lesser extent, hardwoods are essentially composed of myriads of longitudinally aligned narrow tubes or pipes. In softwoods each pipe is only 2-3 mm long and has severely constricted ends. It is nonethe-less widely assumed that the flow of fluids through timber can largely be described in terms of flow through pipes. This section therefore sets out to describe the essential features of such flow.

1.3.1 Basic flow regimes

By their nature all fluids flow 'down' gradients of pressure. The regimes under which fluids may move are dealt with in standard texts (e.g. Daugherty & Franzini 1965) and have been clearly and succinctly summarised by Banks (1975). Detailed repetition is inappropriate here, owing to the nature of the experimentation described later in this thesis. The following summary is presented to establish background information.

1.3.1.1 Laminar flow

Both liquids and gases move at low speeds by laminar flow, in which regime the resistance to flow is mainly due to the shearing forces in the fluid, i.e. the gradient which is promoting flow is primarily resisted by forces resulting from the attraction between adjacent molecules.

Flow of liquids, through capillaries, in this regime was described mathematically in the mid 19th century by two independent workers, Hagen & Poiseuille, in the following relationship:

$$Q = \frac{r^4 \Delta P}{8 \mu l} \qquad \text{Equation 1.1}$$

where Q = Volume (V) of liquid flowing in unit time (commonly termed 'Volume rate of flow')

r = Capillary radius

ΔP = Pressure differential between the two ends of the capillary

l = Length of capillary

μ = Viscosity of fluid

When using this equation to describe flow of gases, appropriate corrections for the expansion of a gas as pressure drops must be applied (see Banks 1975).

1.3.1.2 Turbulent flow and Reynolds number

As the flow rate, and hence the molecular velocity, increases, the magnitude of inertia forces increases concomitantly. At the same time the viscous (inter-molecular attractive) forces remain essentially constant. Above a critical point the viscous forces become relatively too small to restrain flow within the laminar regime. The molecules flow past and around each other in a much less ordered manner and flow is said to have become 'turbulent'.

For predicting the behaviour of flowing fluids the relationship between viscous and inertia forces is obviously of great interest and is measured in terms of a dimensionless 'Reynolds number' (Re). For smooth straight pipes, this is given by the equations:

$$Re = \frac{D\ U\ d}{\mu} \qquad \text{Equation 1.2}$$

or

$$Re = \frac{U\ d}{\eta} \qquad \text{Equation 1.3}$$

where : Re = Reynolds number
D = Fluid density
U = Fluid velocity
d = Diameter of capillary
μ = Coefficient of viscosity
η = Kinematic viscosity (D/u)

High values of Re indicate conditions favourable to turbulent flow and in smooth straight pipes the laminar flow/turbulent flow transition usually occurs when Re ≈ 2000. In non-straight pipes, or when fluids flow around immersed objects, turbulent flow may occur at much lower Re values.

As flow through pipes becomes turbulent, the parabolic profile of velocity (increasing towards the centre), which occurs during laminar flow, is disturbed. The thickness of the less mobile peripheral 'boundary layer' adjacent to the pipe-wall is reduced, causing an apparent reduction in friction between the fluid and the pipe-wall.

Additionally eddies develop, particularly where bends and irregularities in the pipe-wall occur. Such, consequently, is the complexity of turbulent flow that precise mathematical description of it has not so far been achieved. However, experimental work has demonstrated the empirical relationship:

$$Q \propto (\Delta P)^{\frac{1}{2}} \qquad \text{Equation 1.4}$$

where Q = Volume rate of turbulent flow
ΔP = Pressure differential between opposite ends of pipe

1.3.1.3 Slip and Knudsen flow

The two regimes so far outlined relate to the flow of both liquids and gases. However, when gases flow through capillaries of very small diameter, or when they flow at very low pressures, additional factors must be considered.

At a given temperature and pressure it is possible to calculate the average distance each molecule of a gas travels between collisions with other molecules. This distance is termed the 'mean free path' (mfp) of the molecules and varies inversely with pressure and directly with temperature. (For equations see for example Siau (1971)). If flow occurs through a capillary only a few times larger than the mfp, the number of molecular collisions with the walls of the capillary becomes a significant fraction of the total number of collisions. The controlling influence of viscosity on

the rate of flow is diminished and molecules adjacent to the walls appear to slip past them instead of displaying the zero velocity assumed for laminar flow. Flow in this situation is described as 'slip flow' and is significantly faster than laminar flow. (For equations see Carman 1956.)

If the mfp of the gas exceeds the diameter of the flow channel, intermolecular collisions virtually cease to occur and thus the gas's viscosity ceases to be of relevance. The term 'Knudsen flow' refers to flow in this situation. (For equations see Banks 1975.)

1.3.2 Flow through pipes of varying diameter

The foregoing adumbration of the behaviour of fluids as they flow through pipes provides the background for a more detailed examination of their behaviour in situations in which the diameter of the flow channel varies along its length.

1.3.2.1 Bernoulli Equation

The total 'head' of a liquid in a stream-line (e.g. flowing along a pipe) may be resolved into three additive components, thus:

$$\text{Total head} = \underbrace{\frac{P}{D\,g}}_{\substack{\text{Applied}\\ \text{Pressure}\\ \text{Head}}} + \underbrace{\frac{U^2}{2\,g}}_{\substack{\text{Velocity}\\ \text{Head}}} + \underbrace{h}_{\substack{\text{Static}\\ \text{Head}}} \qquad \text{Equation 1.5}$$

where P = pressure
D = density
g = gravitational acceleration
U = velocity
h = height

In an inviscid fluid flowing without turbulence, the total head remains constant along a streamline and so:

$$\frac{P_1}{D\,g} + \frac{U_1^2}{2\,g} + h_1 = \frac{P_2}{D\,g} + \frac{U_2^2}{2\,g} + h_2 \qquad \text{Equation 1.6}$$

This is a simple form of the Bernoulli equation. For the following discussion, the important point arising from this is that when $h_1 = h_2$, pressure and velocity are inversely related in a stream-line. Despite the fact that the Bernoulli equation is strictly obeyed only by an inviscid fluid flowing without turbulence, the principles of the relationship have wide-spread applicability.

1.3.2.2 The Venturi effect

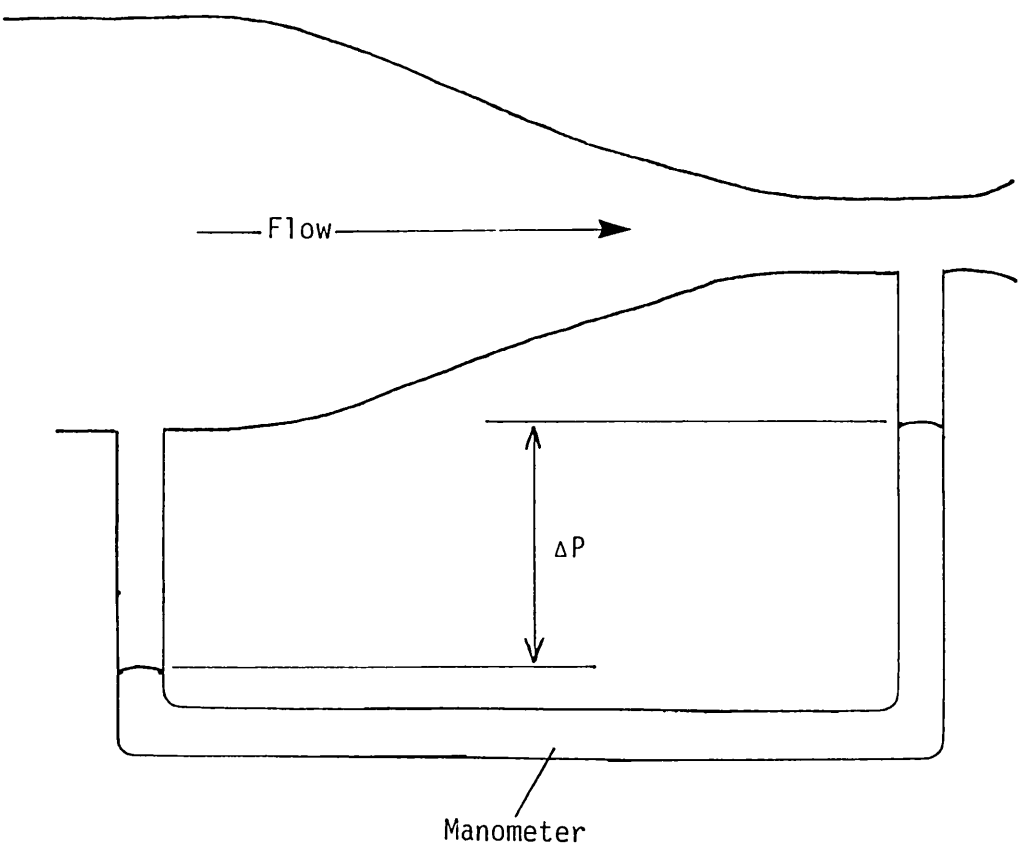
When an incompressible fluid flows in a restricted flow path, such as a pipe, it is clear that the volume rate of flow at each point must be constant along that stream-line. The volume rate of flow at each point is given by the product of the cross-sectional area and the velocity and thus:

$$A_1 \times U_1 = A_2 \times U_2 \qquad \text{Equation 1.7}$$

where A = Area of flow path cross-section
U = Velocity

From equation 1.6, the inverse relationship between pressure and velocity has already been noted. Combining this with the relationship explicit in equation 1.7, it becomes clear that as the cross-section of a flow path diminishes, so too does the pressure within it (Fig 1.9). This is termed the Venturi effect.

Figure 1.9 Diagram of manometer attached to pipe of non-uniform diameter, illustrating the Venturi effect whereby pressure in a flow path drops at constrictions



1.3.3 Interface phenomena

As a gas flows into a gas-filled pipe, the effects of interfacial interactions are so small that for all practical purposes they can be ignored. Not so with liquids.

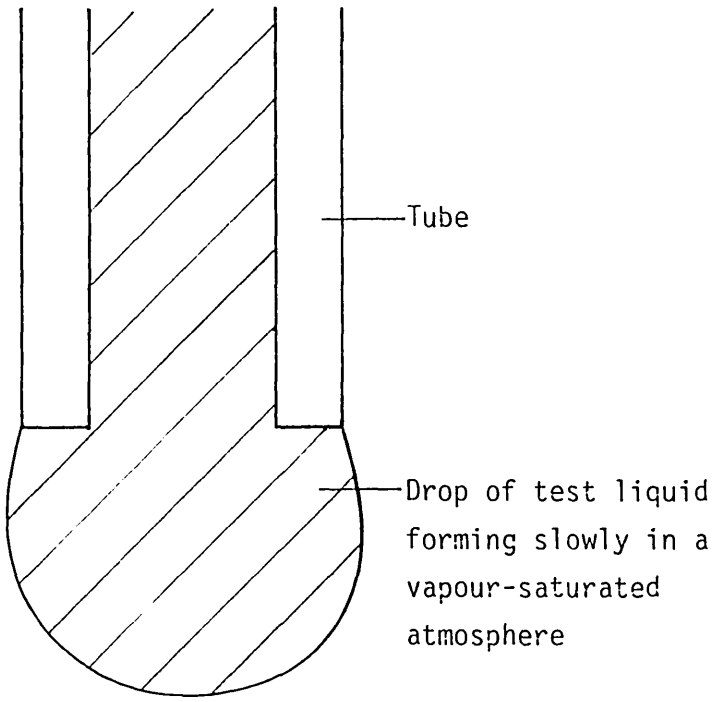
The surface characteristics of a liquid often considerably affect its flow. Indeed in very small capillary tubes these surface effects may be so appreciable as to dominate the liquid's behaviour.

1.3.3.1 Surface tension

As a consequence of the complex interaction of the forces of attraction between molecules, the surface of a liquid in contact with a gas acts as though it is bounded by a thin elastic 'skin'. The 'strength' of this skin, termed the 'surface tension', may be measured by several standard techniques (see for example Adam 1941).

Surface tension measurements made in connection with this work (see Section 2.2.2.2.4) were made using the 'drop-weight technique'. This technique is based on measuring the weight of a drop which falls slowly from the tip of a vertical tube (Fig 1.10).

Figure 1.10 Diagram illustrating the drop-weight method of surface tension determination



The weight of the drop may be related to the surface tension using the equation:

$$T_s = \frac{M\ g\ F}{r} \qquad \text{Equation 1.8}$$

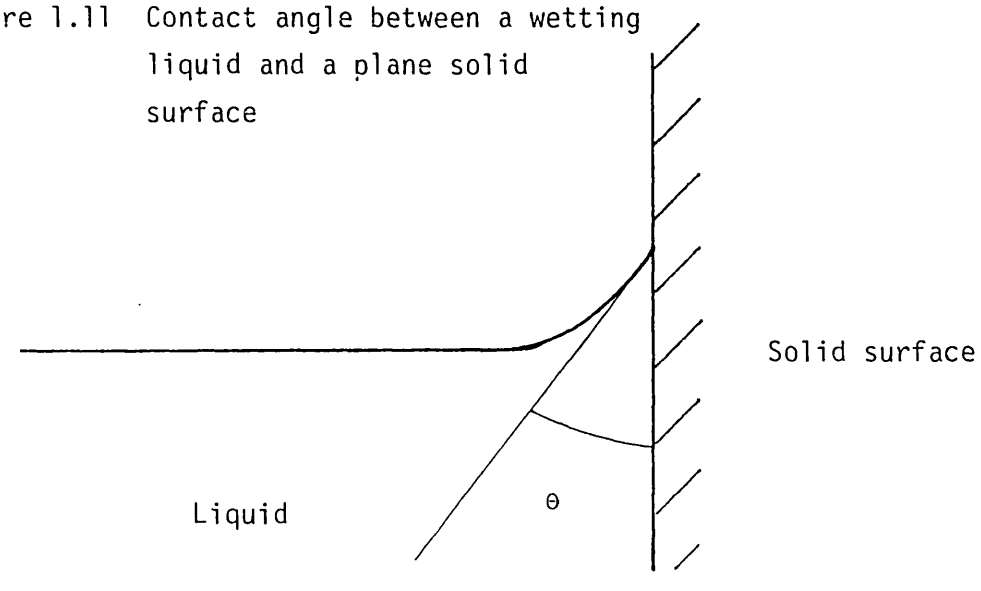
where T_s = Surface tension
M = Mass of drop
g = Gravitational acceleration
r = Radius of tube
F = A correction factor from tables (Adam 1941) - its value is related to the volume of the drop divided by the third power of its radius.

Surface tension which is measured in terms of force per unit length (Nm^{-1}), varies with the identity and purity of a liquid and also with the temperature. For example, at 20°C, water has a surface tension (T_s) of 72.75 x 10^{-3} Nm^{-1} which falls to 58.9 x 10^{-3} Nm^{-1} at 100°C. Methanol on the other hand has T_s = 22.6 x 10^{-3} Nm^{-1} at 20°C. The addition of electrolytes or surfactants may alter these values; 22% NaCl raises T_s for water at 20°C to c 80.9 x 10^{-3} Nm^{-1} while 34% ethyl alcohol reduces it to 33.24 x 10^{-3} Nm^{-1} at the same temperature.

1.3.3.2 Contact angle

The 'contact angle' (θ) between a liquid and a solid is the angle (measured through the liquid) which the surface of the liquid makes with the adjacent solid (Fig 1.11).

Figure 1.11 Contact angle between a wetting liquid and a plane solid surface



For most liquids in contact with many solids, the contact angle is less than 90° (as in Fig 1.11). In these situations the liquids are said to 'wet' the surface with which they are in contact. Indeed when the contact angle tends to 0° (a value it never actually attains) the liquid may spread over the surface of the solid. In convenient conceptual terms it can be considered here that the attraction between molecules of the liquid and those of the solid is greater than the liquid-liquid intermolecular attraction.

Conversely, when the liquid-liquid intermolecular attraction is greater than the liquid-solid attraction, the liquid is said to be 'non-wetting' with respect to that solid and a contact angle greater than 90° is observed. Mercury and glass interact in this way.

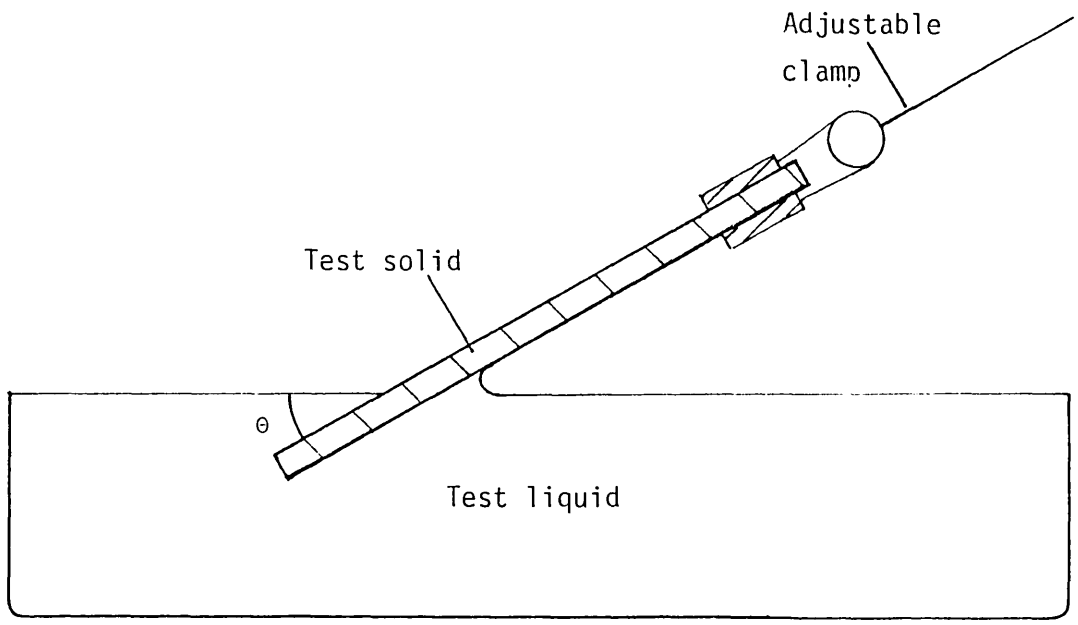
Some liquids such as acetone so frequently wet solids which they contact that they are loosely termed 'wetting liquids'. Others, such as mercury, so seldom wet solids that they are termed 'non-wetting'. However, the variable behaviour of water which is non-wetting to scrupulously clean, dry glass, but becomes wetting after a period of time, demonstrates the fact that it is the liquid/solid interaction and not the liquid alone which determines the contact angle.

A further point is illustrated by the water/glass example. As a liquid advances across a solid surface a transient, advancing contact angle (θ_A) is observed. If the liquid and solid are left undisturbed in contact with one another for a period, an equilib-

rium contact angle (θ) is observed. If the liquid is caused to recede across the solid surface a transient, receding contact angle (θ_R) is observed. For a given liquid the difference between θ_A and θ_R, is termed the hysteresis of wetting and varies from substrate to substrate.

Adam (1941) is among many authors to describe a simple technique for the measurement of both advancing and receding contact angles. The technique, commonly known as the 'plate' method, or 'tilting plate method' (Voulgaridis 1980), involves lowering the end of a sample of the solid material being examined into a container of the test liquid (Fig 1.12).

Figure 1.12 Tilting plate method of contact angle determination



The solid is slowly tilted until, at the upper air/liquid/solid interface, the liquid surface is completely level as it touches the solid's test surface. The solid is then immersed more deeply into the liquid and usually further fine adjustment is required until an angle is reached at which the water-surface remains smooth right up to the solid as the solid is lowered into the liquid. The angle (through the air) between the liquid surface and the solid's test surface is then the complement of the advancing contact angle.

Although the average advancing contact angle between water and timber is fairly readily measured, the present author found this angle much less easy to measure with organic solvents because of the rate at which it tended to an equilibrium contact angle of near zero.

1.3.3.3 Relationship between surface tension and contact angle

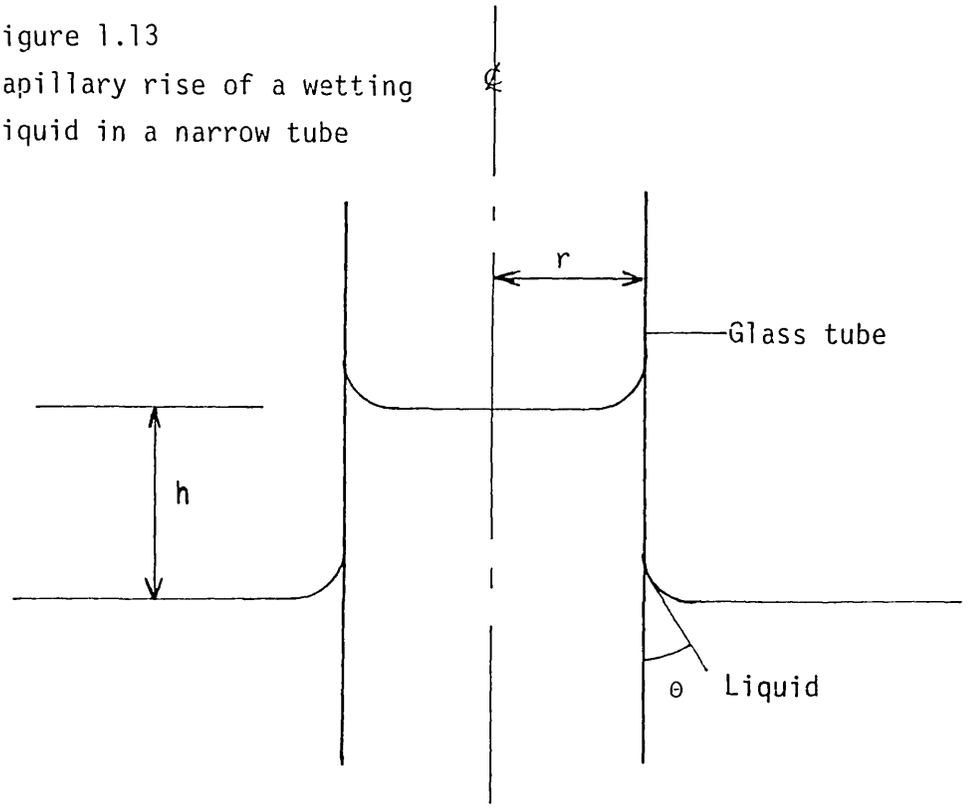
For reasons of the variability of both surface tension and contact angle there does not appear to be any clear or simple relationship between the two. Rafalski (1971) concluded that on timbers, 'surface tension values alone do not constitute a clear indication of wetting effect'. However, to a first approximation, in comparable test conditions, liquids exhibiting the lowest surface tensions also tend to exhibit the smallest contact angles.

1.3.3.4 Capillarity

In capillary-sized tubes the force exerted by surface tension is often sufficient to displace the liquid within the tube in the direction of the curvature of the meniscus, i.e. a liquid contacting the walls of the tube at an angle of less than 90° is pulled up the tube. This happens because the surface forces establish a natural balance between the static head of the liquid, the attraction between the liquid molecules and the walls of the tube, and the tendency to maintain minimum surface area and thus minimum surface energy.

Conversely, a liquid contacting the walls of the tube at an angle greater than 90° is pushed downwards.

Figure 1.13
Capillary rise of a wetting liquid in a narrow tube



The force inducing capillary rise depends on the circumference of the tube, the surface tension and the contact angle. Opposing this force is the product of the mass of liquid being moved and gravitational acceleration, i.e.

$$2\pi r\, T_s \cos\theta = \pi r^2\, h\, \Delta D\, g \qquad \text{Equation 1.9}$$

where
- r = Radius of capillary
- T_s = Surface tension
- θ = Contact angle
- ΔD = Density difference between liquid and gas
- g = Gravitational acceleration
- h = Height of capillary movement

Frequently, it is convenient to consider the capillary force in terms of pressure (force per unit area). At equilibrium:

$$P_o - P_l = \Delta D\, g\, h \qquad \text{Equation 1.10}$$

where
- P_o = Gas pressure
- P_l = Pressure immediately below liquid surface in capillary

therefore (from Equation 1.9)

$$P_o - P_l = \frac{2\, T_s \cos\theta}{r} \qquad \text{Equation 1.11}$$

For wetting liquids $P_o - P_l$ is positive and is termed the capillary pressure. For non-wetting liquids the converse applies and the negative value is termed the capillary tension.

1.3.3.5 Capillary flow

In the examples of vertical tubes discussed so far, capillary movement occurs until the forces inducing movement come into balance with the mass of the liquid being moved. However, if the tube in which these forces interact is horizontal, movement occurs over a very much longer distance at a rate described by the Rideal-Washburn equation:

$$L = \sqrt{\frac{T_s \, r \, t \, \mathrm{Cos}\, \theta}{2 \, \eta}} \qquad \text{Equation 1.12}$$

where L = Distance travelled in time
r = Radius
t = Time
θ = Contact angle
η = Viscosity
T_s = Surface tension

1.3.4 Bubble formation

It is well known that the solubility of a gas in a liquid varies directly with pressure and inversely with temperature. Thus if the pressure around a liquid containing dissolved gas is decreased, or its temperature is increased, gas dissolved in it will be released as bubbles.

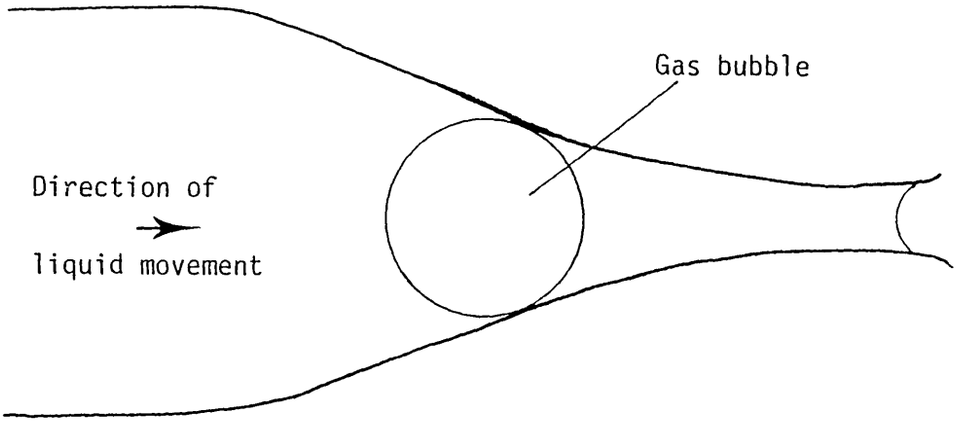
It has already been noted that according to the Bernoulli principle, the pressure in venturi tubes may locally drop below the pressure elsewhere in the system and thus the potential for bubble formation (cavitation) in such tubes is clear.

The pressure-drop required to induce bubble formation in a liquid may be reduced either by subjecting the liquid to mechanical shock, or by introducing nuclei around which bubbles can form. (This latter is the principle on which the use of anti-bumping granules rests.) Suitable nuclei need not necessarily be particulate. A foreign molecular species in a liquid may be associated with the formation of bubbles both of dissolved air, and of vapour from the lower boiling fractions of a mixed petroleum distillate (for instance).

It is widely held (Muskat 1946, Kelso et al 1963, Montigny & Maass 1935, Siau 1971) that once bubbles form in a fine flowpath of varying diameter they may occlude those pathways in the following way (Siau 1971).

When a bubble in a tube flows into a constriction smaller than itself, it has to be distorted in order that it may pass through (Fig 1.14).

Figure 1.14 An air bubble occluding a constriction in a flow path



The meniscus must be deformed from its initial r_o to a new radius r_1 equal to the radius of the constriction, assuming the contact angle is zero. The pressure differential required to induce this deformation is given by Equation 1.13, i.e.

$$P_o - P_1 = \frac{2\ T_s\ \text{Cos}\ \theta}{r} \qquad \text{Equation 1.13}$$

where P_o = Gas pressure inside the bubble
P_l = Pressure immediately beneath liquid surface
T_s = Surface tension
θ = Contact angle
r = Radius of constriction through which bubble is to pass

It may be noted that at $20^{\circ}C$, a pressure of 1.455×10^{6} Pa (14.5 atmospheres) is required to force a bubble in water (surface tension 72.75×10^{-3} Nm^{-1}) through a constriction 0.2 μm diameter (assuming $\theta = 0^{\circ}$).

Additionally, distortion of the bubble increases its surface energy and consequently it transmits less energy than it receives (the Jamin effect). Thus a succession of bubbles lodged in series at intervals along a capillary tube of varying diameter could substantially reduce the pressure differential available to dislodge the foremost bubbles of an advancing column.

1.3.5 Flow through porous materials

To describe and predict the flow characteristics through a sample of porous material, such as softwood for instance, on the basis of the dynamic behaviour of fluid in individual pipes would be an enormous undertaking, even supposing all the relevant dimensions were known or could be guessed. It is usual therefore to make recourse to more empirical equations to describe flow through permeable porous media. These are fully described in standard texts (e.g. Carman 1956) and are summarised by Banks (1975).

During the mid 19th century, Darcy framed a law describing 'steady-state' flow through porous media which is summarised in the equation:

$$K = \frac{V\,L}{t\,A\,\Delta P} \qquad \text{Equation 1.14}$$

where
- K = Permeability, constant
- V = Volume of liquid flowing through sample in time 't'
- L = Length of sample in direction of flow
- t = Time
- A = Cross-section area perpendicular to flow
- ΔP = Pressure difference between opposite ends of flow

(Steady-state flow is said to occur when the pressure gradient promoting flow remains unchanged with time and when the volume rate of flow is also consistent.)

As with the Hagan-Poiseuille equation, if Equation 1.14 is applied to gaseous flow, allowance must be made for the volume change of the gas due to pressure change (see for example Siau 1971).

Re-arranging Equation 1.14 for V/t gives:

$$\frac{V}{t} = \frac{K\ A\ \Delta P}{L} \qquad \text{Equation 1.15}$$

or

$$Q = \frac{K\ A\ \Delta P}{L} \qquad \text{Equation 1.16}$$

where Q = Volume rate of flow, Vt^{-1}
(other symbols as previously)

Darcy's equation is frequently used with a modification due to Carman (1938) who incorporated a factor to take account of viscosity, thus:

$$Q = \frac{K\ A\ \Delta P}{L\ \mu} \qquad \text{Equation 1.17}$$

where μ = Viscosity

Kozeny (1927) sought to employ the Darcy relationship to analyse the structure of a porous material by giving 'K' some physical meaning. This leads to the 'Kozeny Equation':

$$U = \frac{m^2 \Delta P}{k_o \eta Le} \qquad \text{Equation 1.18}$$

where U = Liquid velocity

m = Mean hydraulic radius

(i.e. $\frac{\text{pore cross-sectional area}}{\text{pore circumference}}$)

k_o = A shape factor

ΔP = Pressure drop along sample

η = Viscosity

Le = Effective length of flow paths (this might be in excess of the overall sample length)

The 'mean hydraulic radius' (m) must clearly be an approximation when the pores are other than circular - hence the need for the corrective shape factor 'k_o'.

The Kozeny equation does not take account of slip flow which may occur as gases flow through fine capillaries and an equation describing flow with this regime through an idealised model was developed by Adzumi (1937). Adzumi's model assumed straight capillaries of uniform circular cross-section. To take account of departures from this pattern, workers such as Carman & Arnell (1948) modified the Kozeny equation with the addition in slip flow and tortuosity factors.

1.4 Flow into and through timber - overview

It has already been noted that, at least 'in vivo', timbers are permeable materials. Softwoods and hardwoods can be considered conceptually as clusters of longitudinally aligned capillary pipes. These may sometimes be continuous for long distances (e.g. some hardwood vessels) or alternatively each pipe may be no more than a few millimetres long (as in softwood tracheids). In softwoods particularly, a permeating fluid flows through a succession of pipes and pores of varying diameter.

The flow of fluids through timber has interested many workers throughout this century from such workers as Ewart (1905) through to Siau, Petty, Bolton and others who are actively engaged in its study today.

Progress was at first rather slow. Rigorous analysis of gas-flow data was developed only after Adzumi's mathematical description of slip and Knudsen flow (q.v. Adzumi 1937) had been assimilated and developed (e.g. by Carmen and Arnell 1948). Examination of liquid flow struggled against technical problems until Kelso et al (1963) were able to demonstrate how to maintain a steady flow rate in laboratory experiments.

More rapid progress followed this breakthrough. Several studies which confirmed that fluid flow through timber could indeed be described by Darcy's law led Bramhall (1971) to publish an inte-

grated equation based on this law to describe unsteady-state flow. This approach has been developed by others (notably Petty 1975, 1978b). These studies have been facilitated by an improved appreciation of the structure of timber which has resulted both from steadily improving techniques for analysing gas-flow data, and also from direct observation with the electron microscope.

1.4.1 Gas-flow through timber

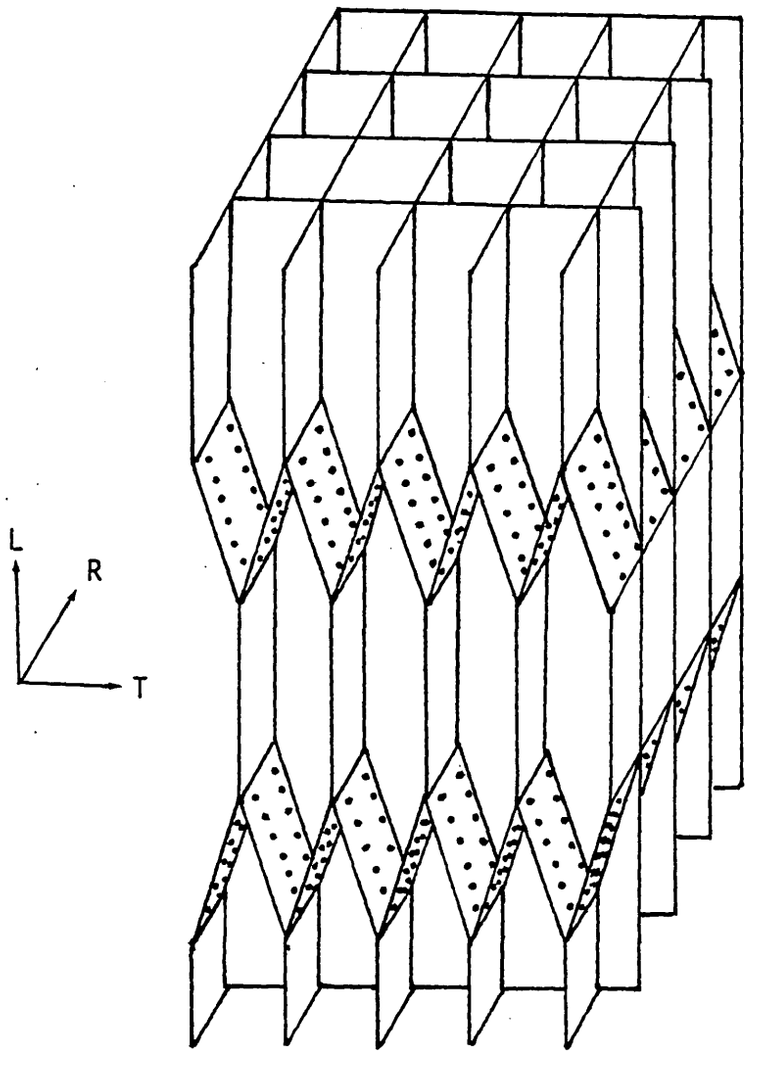
Darcy's law was published in 1856, and modified to include a term for viscosity by Carman in 1938. Somewhat surprisingly, therefore, the work of Smith and Lee (1958) appears to be one of the first significant studies of the permeability of timber to gas.

Smith and Lee (1958) measured the permeability to air of about 100 timber species and, by taking measurements at several pressure gradients, concluded from the results that 'flow obeyed Darcy's law'. These workers also attempted, with limited success, to correlate observed permeabilities with permeabilities calculated by inserting 'known' values for pore diameters etc. into the Poiseuille equation. However, other than this, their paper was mainly a record of experimental results. A somewhat similar paper to this was produced at about the same time by Buro & Buro (1959) who measured permeability of samples of pine taken at different locations and orientations within a tree.

This early work involving simply measuring permeability has been followed by several investigations in which permeability measurements have been used to investigate wood structure. For example Comstock (1970) used gas permeability data to investigate the structure of timber when he analysed data from four earlier papers (Choong & Fogg 1968, Osnach 1961, Resch & Ecklund 1964 and Smith 1963) and used them to compare two idealised models for the distribution of pits in softwood. The model which best fitted this

collected data assumed the pits to be restricted in distribution to the overlapping ends of axial tracheids.

Figure 1.15 Comstock's model



L = longitudinal
R = radial
T = tangential

After Comstock (1970)

Comstock conceded that "the models presented do not represent actual wood structure ....." and in his discussion he totally neglected radial flow. The inference of this would appear to be that there were no radial openings in the longitudinal tracheid walls and thus the variable level of radial permeability reported for different species is entirely due to the ray tissue.

The permeability of ray tissue appears very variable from species to species and is generally higher in the pines than in other softwoods (Maclean 1935). Erickson & Balatinecz (1964) have also reported ray penetration in Douglas fir (Pseudotsuga menzieseii).

Permeability measurements have been used to study not only the location of flow-paths, but also their geometry. Sebastian et al (1965) obtained gas permeability measurements from Picea glauca from which, using a modification of Adzumi's theory, they calculated the effective radii of the pores in the pit membrane. They concluded that their calculated values showed 'reasonable' agreement with electron microscope measurements of the large openings in the pit membranes. This approach to flow path measurements, also used by Resch & Ecklund (1964), Comstock (1967) and others, has been refined more recently. Whereas, following Stamm (1946), the above authors based their calculations on the assumption that all resistance to flow occurs at the pit membrane, Petty (1970), Petty & Puritch (1970) and Smith & Banks (1971) have all since demonstrated that two components (the pit membranes and the tracheid lumena) each contribute significantly to total resistance to

flow. In consequence Bolton & Petty (1975) point out that the work of earlier authors may contain inaccuracies due to the faulty assumption on which they were based. Additionally, these authors present data on the flow of gas through ponded Sitka spruce which indicate that the pit apertures also contribute to total flow resistance.

Siau (1976), using similar analysis on some gas-flow data in 'white ash', concluded that in this species there appear to be three parallel components to its gas permeability.

This type of analysis is of great practical value. For example Petty (1974) calculated both the size and abundance of pit membrane pores in two softwood species and such information is invaluable in interpreting the behaviour of liquids in these timbers.

Bramhall (1971) departed from the more commonly used techniques for measuring steady-state gas-flow in two significant respects. He examined longer samples than had been common previously and, more importantly, he varied sample length to examine the effect of length on permeability.

In his more permeable samples of Douglas fir, Bramhall observed permeability to vary with length as predicted by Darcy's law. However, in less permeable samples, significant deviations from this pattern were observed. Bramhall attributed this to the random termination of potential flow paths caused by the paths encounter-

ing tracheids with all pits aspirated. In consequence, fewer paths would be continuous from end to end in a long sample than in a shorter sample of the same cross-section. The following modification to Darcy's law takes account of this effect:

$$Q = \frac{K A e^{-bl} \Delta P}{\mu L} \qquad \text{Equation 1.19}$$

where e = Base of natural logarithm
b = A positive constant determined by experiment
l = Length of liquid in flow path
(other symbols as Equations 1.14 through 1.17)

Banks (1970) also noted this effect and, working independently, explained it on the basis of a similar mechanism. Noting that, above a certain length, rate of flow once again varies with length according to Darcy's theory, Banks (1975) suggested that a point is reached when all the pathways subject to occlusion are blocked and flow is totally controlled by residual flow paths such as latewood bordered pits.

Smith & Lee (1958), Buro & Buro (1959) and more recent workers such as Choong & Fogg (1972) and Sebastian et al (1965) made their permeability measurements on relatively small samples through which they established steady-state flow before taking measurements. Lin (1972) demonstrated that where a 'good estimate' of permeability adequately fulfilled requirements, measurements could be made with

rather more simple apparatus using a 'pseudo-steady-state' technique. His apparatus was similar in concept to that used by Petty (1967) to monitor gas removal from test specimens. The gas-flow measured is not steady-state but Lin held that it may be considered so during any given instant. The mathematical manipulation involved is thus rather more complex than for the analysis of true steady-state flow. Lin's results show good correlation (cr = 0.864) between permeability values determined by the two techniques.

The gas permeability of timber has thus been quite fully explored. Many variants on the design of gas permeability measuring apparatus have been described, using which, laminar flow has been shown to conform to behaviour predicted by Darcy's law. Departures from this behaviour pattern can be attributed to slip flow and changes in permeability. Analysis of flow rates at different pressure gradients yields fairly detailed information about the structure of the timber through which flow is occurring.

1.4.2 Liquid flow through timber

Ewart (1905) encountered difficulties in determining a precise liquid permeability for a timber because he was unable to establish flow at a steady rate. Instead he observed a decrease in flow rate with time. This was a phenomenon later to be reported by many workers including Buckmann et al (1935), Erickson et al (1937), Sutherland et al (1934) and Ohgoshi et al (1976). Anderson et al (1941) note that a diminution of flow rate with time is also seen as water passes through other porous media such as sand filter beds.

Explanations offered to account for this phenomenon included surface electrical effects or reduction in pore cross-section (Buckmann et al 1935), pivoting of the membrane fibrils into positions of maximum hydrodynamic resistance (Anderson et al 1941), lateral displacement of the torus (Huber & Mertz 1958), physical occlusion by particulate matter (Krier 1951), and the formation of air embolisms (Kelso et al 1963).

Of these possible mechanisms, surface electrical effects were discounted by Anderson et al (1941) and pivoting of membrane fibrils remains speculative because of a continuing lack of evidence that they are, in fact, free to move.

Movement of pit membranes, suggested by Huber & Mertz (1958) is strongly indicated by the work of Bailey & Preston (1970) on dry Douglas fir, of Lin et al (1973) on western hemlock and of

Bolton & Petty (1978a)on Pinus sylvestris. However, Anderson et al (op cit), applying logic which is not flawless, suggested that in view of the fact that diminution of flow rate is also seen in hardwoods, in which pit aspiration is obviously impossible, this mechanism is unlikely to account for the flow rate diminution in softwoods. Clearly this argument is invalid, but it does underline that pit aspiration may not necessarily be the only mechanism causing decreasing flow rates in softwoods. By the same token, reduction of pore cross-section by swelling of the walls, while probably being a contributory factor with polar solvents, is likely to be supplemented by another mechanism in view of the flow rate decrease also observed in inert filter beds.

The two remaining possibilities so far suggested are particulate blockage and air embolism formation. Kelso et al (1963) conclusively demonstrated the dominant role of air embolisms in the causation of flow rate reduction. Part of their work involved subjecting samples through which flow was occurring to mechanical shock. As was noted in Section 1.3.4 this treatment can induce cavitation (bubble formation). These workers showed that when cavitation was thus induced, a sudden and large drop in flow rate occurred.

The air embolism theory of permeability reduction has now become widely accepted and, using techniques (employed by Kelso et al (1963))for prior removal of dissolved gas from test liquids, several workers have been able to establish steady flow rates

through a range of species (e.g. Banks 1968, Petty 1978a).

The air embolism mechanism presents an explanation of another phenomenon, probably first reported by King in 1899 in quarried sandstone, and later reported in timber by several workers including Sutherland et al (1934) and Buckman et al (1935). These workers noted that in samples in which flow had decreased from its original rate, increasing the pressure gradient caused an increase in flow rate which was disproportionately higher than would be predicted by Darcy's law. Sutherland suggested that this was due to some sort of enlargement of the pores in the flow path, probably in consequence of stretching of the pit membrane. Buckman et al (1936) substantially agreed with this but Anderson Gortner & Schmitz (1941) held that the increase in pore diameter which they estimated to be possible due to this cause was inadequate to explain the results observed. Instead they suggested that the fibrils of the margo were shifting such that the larger pores increased in size at the expense of the surrounding smaller ones. However, using Sitka spruce heartwood and increasing pressure gradients in the range 0 - 300 kPa, Kelso et al (1963) showed that, with assiduous prior gas removal, the increase in flow rate caused by increasing pressure, in fact closely approximated to linearity as described by Darcy's law.

The disproportionate increase in flow rate, as the pressure gradient increased, thus appeared only to occur if the flow channels were partially occluded by air embolisms.

This observation may be explained by recalling that air bubbles lodged at constrictions in flow paths may be dislodged by application of a pressure above some critical threshold (Section 1.3.4). The value of this threshold pressure is related by Equation 1.13 (q.v.) to the diameter of the constriction in question.

For a porous material, having within it many pores encompassing a range of sizes, it can readily be seen that if a significant number of these pores become occluded by bubbles and pressure is increased over the range required to dislodge these bubbles, an increase in flow rate, quite unrelated to Darcy's law, will be observed.

Clearly, the problems of attaining a steady liquid flow rate were until quite recently a major impediment to precise liquid permeability determinations. In consequence, Comstock (1970) pointed out that the results of early investigators such as Sutherland et al (1934) & Erickson et al (1938) must be considered unreliable.

Once the problems of achieving a steady liquid flow rate had been overcome, the way was open to examine obedience to Darcy's laws during liquid flow. In the original 1963 paper, Kelso and his colleagues demonstrated satisfactory agreement between flow rate and pressure gradient in spruce, and in the same year one of those colleagues collaborated in work showing similar agreement between flow rate and viscosity, also through spruce (Chen & Hossfeld 1964).

Bailey & Preston (1970) agree that under ideal conditions Darcy's law describes the flow of liquid through wood, but note some of the many factors which may cause departures from this pattern.

In 1971 Bramhall developed Darcy's law to describe the unsteady-state flow of gases into timber (Section 1.4.1 refers). From the fact, demonstrated by the work discussed above, that Darcy's law describes the flow of liquids into timber, it follows that Bramhall's development of Darcy's law applies equally to liquid flow. Thus, in the absence of complicating factors such as bubble formation, unsteady-state flow of liquid into timber can be described by an integrated form of Darcy's law (Bramhall 1971).

$$\frac{dV}{dt} = \frac{K\ A\ \Delta P}{\eta\ L} \qquad \text{Equation 1.20}$$

where
V = Volume
K = Permeability constant
A = Cross-section area normal to flow
P = Pressure gradient along flow path
η = Viscosity
L = Length
t = Time

It will be recalled (Equation 1.19) that Bramhall included a factor (e^{-bl}) to take account of random path blockage in long specimens. Because, when b tends to zero, e^{-bl} tends to unity, other workers (Galvao et al 1974, Petty 1975 and 1978b) omit it, unless it is explicitly required (e.g. Petty 1975).

The distance (Le) a given volume (V) of liquid can penetrate into a sample of wood is obviously related to the fraction (f) of the sample cross-section (A) which is available as flow paths, i.e.

$$V = A\, f\, Le \qquad \text{Equation 1.21}$$

where Le = Length of liquid column in flow path
V = Volume (liquid)
f = Fraction of cross-section area available as flow paths
A = Cross-section of sample

From Equations 1.20 and 1.21, Petty (1975 q.v.) has shown that an expression for total liquid absorption (V) may take the form:

$$V = A\left(\frac{2\, K\, f\, \Delta P\, t}{\eta}\right)^{\frac{1}{2}} \qquad \text{Equation 1.22}$$

and thus during unsteady-state flow into fairly permeable timbers:

$$V \propto \sqrt{\Delta P} \qquad \text{Equation 1.23}$$

and $$V \propto \sqrt{t} \qquad \text{Equation 1.24}$$

and $$V \propto \sqrt{\frac{1}{\eta}} \qquad \text{Equation 1.25}$$

Petty (1975) showed that radial flow of 'petroleum distillate' into several softwood species can be described by Equation 1.22 as written, but inclusion of the e^{-bl} factor is needed adequately to

describe longitudinal flow into Douglas fir. Stamm (1973) also found agreement with Equation 1.22 for both water and toluene absorptions into a range of hardwoods.

Results reported by Siau (1970) indicate that Equation 1.22 does not describe the flow of liquids of different viscosities into refractory timber. However Petty (1978b) presents results for pine sapwood which are consistent with the viscosity/uptake relationship embodied in Equation 1.22

1.4.3 Relationship between gas and liquid flow

Industrially, there is an obvious need for a technique which may be used to give an *a priori* estimation of liquid uptake by a timber in a given set of conditions. The relative ease with which gas permeability measurements may be made makes the desirability of predicting liquid uptake from gas-flow measurements self-evident.

In one of the earliest studies of this subject, Smith & Lee (1958) compared the air and toluene permeability of four timbers and obtained good agreement in three. In the fourth, (*Abies grandis*) the air permeability was much higher, and they suggested that this may have been due to the occurrence of Knudsen flow. In retrospect it is also possible to speculate that air embolism formation retarded flow through this relatively impermeable species.

Following the work of Kelso et al (1963) it became evident that, under 'ideal' conditions, liquid - (at least non-swelling liquid) and gas-permeabilities should correlate well, although Bolton & Petty (1978b) felt that ideal correlation had still to be demonstrated. As Bailey & Preston (1970) observed, the requisite ideal conditions do not always exist, and certainly not during industrial timber impregnation. The question remained therefore: Could preservative uptakes be predicted from gas-flow measurements?

Tesoro et al (1966) cut 0.19 cm thick wafers from a range of timber species to measure their lateral permeability to air. They then

impregnated samples of the timber with creosote and observed good correlation between gas permeability and creosote retention. Slightly less good correlation between permeability and penetration was noted.

Conversely Arganbright & Wilcox (1969) did not observe a satisfactory correlation between transverse nitrogen permeability and uptake of PCP[1] in heavy oil in white fir (Abies conolor).

In 1972, Choong & Fogg compared gas permeabilities with creosote retentions in Pinus echinata and Liriodendron tulipifera. Their conclusions closely paralleled those of Tesoro et al (1966), with good correlation between gas permeability and uptake and slightly less close correlation occurring between uptake and penetration. They suggested this may be related to the problems of accurately estimating penetration. Tesoro & Choong (1976) also presented evidence that in a range of species, the uptakes of both water and creosote could be predicted from steady-state gas-flow measurements. They did, however, note that water uptakes could better be predicted from steady-state water permeability data. From their data it appeared that species with differential latewood/earlywood permeabilities were less satisfactory subjects for this sort of predictive testing. Furthermore, while very impermeable species normally showed correspondingly low uptakes, and the

[1] PCP = Pentachlorophenol - a common fungicide, also used for termite control.

converse, there was a good deal of overlap in the middle range.

As practical work-a-day tools for predicting fluid uptake during a preservative treatment, the techniques used in the foregoing investigations shared the drawback that they all involved samples of timber of a particular, quite critical, size, shape and condition for gas-flow measurements. The tests were destructive in that small samples had to be cut from larger timbers. Additionally, considerable skill was required for reproducible sample preparation.

Against this background, techniques such as those of Cooper, Bramhall & Ross (1974) are very attractive. These authors devised an apparatus with which they could make rapid estimates of unsteady-state gas-flow rates in a timber. (They used Douglas fir.) They observed good correlation between flow rates thus measured and creosote retentions in subsequent impregnation tests.

Cooper et al's apparatus was intrusive in that it involved the insertion of a short, ca. 12 mm diameter probe into the timber. Venturino and Arganbright (1979) produced similar apparatus but with a surface contact probe so that the testing left the timber's surface unmarked. Unfortunately, measurements with this apparatus correlated poorly with organic solvent liquid retentions during subsequent impregnations. The authors concluded that this poor correlation was due to air introduced into the timber escaping again through the surface just a few centimetres away and not flowing through the timber for any appreciable distance.

1.5 Industrial timber impregnation

Industrially, a range of timber impregnation techniques is available which facilitates the application of preservative to timber at any stage after felling. For example, immediately after felling, preservative can be introduced into logs by replacing the sap in them with preservative solution. Alternatively, after the timber has been converted and while it is still green, toxicant may be introduced by a diffusion process. Seasoned timber is best treated by total immersion and the entry of the preservative into the voids within the timber is usually accelerated either by partially evacuating the voids prior to immersion or by pressurising the treating liquid during immersion, or by a combination of the two.

A substantial quantity of industrial timber preservation is done after, often long after, the timber has been installed and here the preservative is applied by brushing and spraying. These techniques do not impregnate timber with preservative at all deeply, and thus they are not further discussed in this section.

1.5.1 Preservation of green timber

Several techniques are available for the preservation of freshly felled, unseasoned timber. The most complex of these is the Oscillating Pressure Method which is mostly used for the treatment of green radiata and Corsican pine and which employs sophisticated

apparatus (Henriksson 1958). Such sophistication is exceptional however and the two techniques most widely employed for the treatment of green timber require comparatively simple equipment. These techniques are the Boucherie process in which the sap in the tracheid or vessel lumena is replaced by a preservative liquid, and Diffusion Treatment in which a toxicant is introduced by diffusion into the sap which is in the timber, thus rendering the timber inhospitable to invading organisms.

1.5.1.1 The Boucherie Process

In the Boucherie process, the sap of freshly felled trees is replaced by preservative solution. To effect this, the butt end of each felled tree to be treated is fitted into a water-tight cap. This cap is connected via a flexible pipe to a reservoir of aqueous preservative solution which is supported about 8 m above the ground. This provides a hydrostatic head of ca. 100 kPa and this causes the preservative to flow longitudinally through the log, flushing sap out before it as flow progresses. The process is allowed to continue until the sap seeping from the top of the log is supplanted by preservative solution.

Historically, copper sulphate solution has been much used as the preservative solution in the Boucherie process. This chemical may be leached out of the timber in service however, and so mixed salt solutions such as copper, chromium and boron salt mixtures are now used. Treating solutions such as these impart to the

timber 'permanent' protection against degradation by basidiomycete fungi and insects.

The Boucherie process introduces preservative into sapwood before processes such as pit aspiration have occurred. In consequence it achieves excellent penetration into those regions occupied by sap at the time of felling. From these regions there may also be some subsequent migration of the preservative into the heartwood.

Timber treated in this way is suitable for use where the subsequent conversion required is zero or negligible such as in poles. It is not suitable for use where substantial conversion is required because conversion of treated timber not only wastes preservative but also generates toxic wood-waste which requires careful handling and disposal.

1.5.1.2 Diffusion Treatment

The aim of the diffusion treatment of freshly felled timber is to distribute a preservative chemical throughout those regions which have a sufficiently high moisture content. Freshly converted timber, having a moisture content above 50%, is suitable for diffusion treatment and currently all industrial diffusion treatment employs boron salts, such as disodium octaborate, as the toxicants.

To effect diffusion treatment, the freshly converted timber is first of all immersed in a concentrated solution of preservative, which is often heated somewhat above ambient temperature so that a sufficiently high concentration of boron salt can be maintained in solution. The length of time for which immersion is continued varies according to the moisture content of the timber. For example the French Centre Technique du Bois standard for diffusion treatment of spruce requires only five minutes immersion when the timber's moisture content is 80% but six to ten hours immersion if its moisture content is as low as 40%.

Immediately after immersion all the toxicant is located at the surface of the timber and opportunity must be given to allow diffusion throughout each piece of timber to occur before treatment can be considered complete. To keep the timber 'wet' and thus allow the diffusion phase to proceed, it is close-stacked under tarpaulins (or similar) for a period of weeks, the exact length of time being dictated by the moisture content of the timber at the time of treatment, the ambient temperature and the thickness of the pieces of timber.

At the end of the diffusion phase the timber is allowed to dry down to its end-use moisture content and is utilised in situations out-of-ground contact. Because boron salts do not chemically fix to the timber, diffusion treated timber is not suitable for use in situations where persistent leaching is liable to occur, such as in contact with the soil.

1.5.2 Preservatives for seasoned timber

To avoid the problems of wasting preservative, producing toxic wood-waste and exposing untreated timber during post-treatment conversion, the majority of preserved timber receives its preservative treatment only when it is seasoned and as near as possible to its final dimensions.

A very large number of preservative formulations are available (Cockcroft 1980) which, for the purpose of discussing their impregnation into seasoned timber, may be considered in three groups on the basis of the identity of their carrier solvents. These groups are described in publications such as BS 1282. Some of the salient features characteristically associated with each are set out in Table 1.1.

1.5.2.1 Tar oil preservatives

From Table 1.1 it can be seen that tar oil preservatives, more usually termed creosotes, are composed of mixtures of the oils derived from the distillation of coal tar. Creosotes have a wide spectrum of biological activity and are excellent wood preservatives. When properly applied they impart good protection against biological degradation to timbers used in even the highest hazard situations. There are certain constraints regarding their use in building-timbers however. These arise in consequence of the facts that creosotes have a strong odour and that it is difficult to

Table 1.1 Characteristics and uses of the principal preservatives for seasoned timbers

PRESERVATIVE	ACTIVE INGREDIENTS COMMONLY USED	CHARACTERISTICS OF TREATED TIMBER	SUITABLE FOR TREATMENT OF	METHOD OF APPLICATION
Tar Oil	Mixtures of Coal Tar Distillates	-Water repellent -Distinctive odour -Non corrosive to metals -Brown in colour -Cannot be satisfactorily painted -Protected against all wood destroying organisms -Preservative will not leach out of timber	Fencing Cladding Timber in Soil-Contact Farm Buildings	1. Brush/Spray/Deluge (superficial penetration only) 2. Hot & Cold soak for butt ends of fence posts 3. Rüping Process 4. Lowry Process
Water-borne	Tri-salt Mixtures such as Copper/Chrome/Arsenate Copper/Chrome/Borate	-Odourless -Light green in colour -Paintable when dry -Preservative chemically fixed to timber -May corrode metal fittings in some situations -Protected against all wood destroying organisms	As Above also Domestic Building Timbers	Bethell Process
Organic Solvent	Pentachlorophenol Metal Naphthenates Tributyltin Oxide Organochlorine Insecticides	-Protected against wood destroying organisms except soft rot -Moisture content and dimensions unchanged during treatment -No colour change -May be water repellent -Preservative non leachable -Active ingredients may be subject to superficial volatile depletion	Building Timbers out of Ground Contact	1. Brush/Spray/Deluge (for insitu application) 2. Immersion 3. Double Vacuum and Double Vacuum with Pressure Processes

paint over them successfully. Additionally, in recent years their use has been further curtailed by supply difficulties.

1.5.2.2 Water-borne preservatives

Being easy to handle and abundantly available, water has long been widely used as a carrier solvent for wood preservatives. It is very mobile (viscosity = ca. 1 x 10^{-3} Nsm^{-2}) and thus flows through timber easily. It also has a fairly high surface tension (ca. 73 x 10^{-3} Nm^{-1} at 20°C) and might therefore be expected to penetrate well into timber as a result of strong capillary forces (Richardson 1978). Water is not however particularly wetting to dry timber and thus flow due solely to capillary action is slow. Water-borne preservatives are therefore invariably applied by high pressure impregnation.

Mixed solutions of copper, chromium and arsenic salts or oxides are the most widely used formulations. They have wide ranging biological activity and are potentially suitable for the treatment of timber to be used in all situations.

Considerations which contra-indicate the use of water-borne preservatives do not usually relate to the preservative chemicals themselves but to the fact that their carrier solvent, water, swells the timber. Certainly this swelling is largely reversible, but gross distortion is sometimes encountered and raising of the grain is frequent. For these reasons water-borne preservatives

are not favoured for the treatment of finely-dimensioned timber such as window-joinery components.

1.5.2.3 Organic-solvent preservatives

The petroleum fractions used as carriers for 'organic-solvent' wood preservatives are typically those which have a flash-point above 32°C but which boil below 300°C and therefore evaporate fairly readily from timber after treatment. Solvents boiling in this range generally have a viscosity similar to that of water (ca. 1×10^{-3} Nsm^{-2}) while their surface tension (ca. 30×10^{-3} Nm^{-1}) is generally much lower. They are highly wetting to timber. Indeed, the advancing contact angle between organic-solvent and planed spruce, which is about 20 - 25°, is most difficult to determine accurately owing to the speed at which the equilibrium contact angle of near zero is reached. These solvents thus enter timber very readily.

The toxicants usually carried in organic-solvents are complex organic molecules such as pentachlorophenol and tributyltinoxide. These biocides are deposited on the surface of the tracheid lumena walls and are not particularly effective against soft rot fungi. Organic-solvent preservatives are not, therefore, suitable for use on timber which is to be used in contact with soil.

Recent concern about the permanence in the surface of the timber of the biocides used in organic-solvent preservatives has high-

lighted the importance of applying these preservatives in a way which ensures that deep penetration is achieved.

1.5.3 Impregnation of seasoned timber

The techniques available for the impregnation of seasoned timber have been reviewed in general terms by authors such as Richardson (1978). The more widely used of these techniques are mentioned in Table 1.1 and are more precisely defined in National Standards such as BS 144, which deals with impregnation with creosote, BS 4072 which deals with water-borne preservatives and BS 5707 part 3 which relates to the application of organic-solvent preservatives.

In essence, the feature common to nearly all industrial impregnation techniques is the immersion of the timber in liquid preservative for a period, during which there exists a pressure-gradient between the immersion liquid and the voids within the timber. The duration of the immersion phase, the magnitude of the pressure-gradient and the manner in which it is established are variables which are determined according to the type of preservative in question, the species of timber to which it is being applied and the end use of the treated timber.

1.5.3.1 Immersion Treatment

It has been noted in Section 1.3.3.4 that wetting liquids tend spontaneously to enter small tubes as a result of capillary forces. In consequence of this, it is possible to apply mobile, wetting preservatives, such as organic-solvent preservatives and low-viscosity creosote, to permeable timber, such as pine sapwood, by simply immersing it.

In industrial practice, treatment which relies solely on capillary forces has certain limitations. These limitations derive largely from the fact that flow motivated by capillary forces is often rather slow and this problem is exacerbated by the fact that, although capillary-force-induced penetration can continue until the pressure of any air trapped within the timber balances the 'capillary pressure' exerted at the interface of the liquid in each lumen, the rate at which liquid enters the timber by capillarity decreases exponentially with time.

Immersion treatments are thus really appropriate only for superficial treatment of permeable timbers such as pine sapwood with organic-solvent preservatives and low-viscosity creosote. Where deeper penetration of the timber is required, or where less permeable species such as European whitewood are to be treated, immersion treatments do not give adequately deep penetration unless they are continued for at least several days. Water-borne preservatives are, in practice, seldom applied by simple immersion

because, having an advancing contact angle against timber which is greater than 90°, their rate of flow into timber by capillarity is very slow indeed.

1.5.3.2 Double Vacuum treatment

For the application of organic-solvent preservatives to permeable timbers in which fairly deep penetration is required it is usual to speed-up natural capillary flow by the creation of a pressure-gradient between the liquid and the inside of the timber. The magnitude of the gradient used is small (usually ca. 35 kPa) and it is created, not by applying hydrostatic pressure to the preservative liquid, but by partially evacuating the timber prior to the immersion phase. The negative pressure so created within the timber draws liquid in, and results in, perhaps,5 - 10 mm lateral penetration into pine sapwood in three minutes. (This is a typical notional duration for the immersion phase of a double vacuum treatment for pine in the U.K.)

When impregnation has been effected in this way, the loading of preservative liquid in the penetrated zone adjacent to the surface of the timber is unnecessarily high. It is usual, therefore, to subject the timber to a post-immersion vacuum to withdraw the excess preservative and render the timber 'touch-dry' when it is removed from the treatment vessel. It is from the use of two vacuum phases, one before and one after immersion, that the name 'Double Vacuum' process is derived.

1.5.3.3 The Double Vacuum with Pressure (DVP) process

The Double Vacuum with Pressure (DVP) process is used to apply organic solvent preservatives in two situations where the double vacuum process is inadequate. These situations are encountered where very deep penetration of preservative into permeable timber is required and where less permeable timber such as whitewood (Picea spp.) is to be treated.

The essential difference between the double vacuum and DVP processes is that in the latter a hydrostatic pressure is applied to the treating liquid during the immersion phase. Thus, the pressure-gradient set up by the partial evacuation of the timber prior to immersion is supplemented by the application of a pressure of 100 - 200 kPa to the preservative liquid while the timber is immersed in it. Generally speaking the higher the applied pressure the faster the liquid flows into the timber and the shorter the period for which the pressure must be sustained.

1.5.3.4 The Rüping empty cell process

The empty cell or Rüping process is for the application of creosote and is designed to achieve deep impregnation into the timber and then, like the double vacuum and DVP processes, to withdraw excess preservative from the timber. Reclamation of rather viscous creosote at the end of the immersion phase proceeds slowly unless a large pressure-gradient is available to accelerate its flow out

of the timber. To provide for such a gradient the air in the cells of the timber is pressurised to 200 - 400 kPa for the 20 - 30 minutes immediately prior to the immersion phase. During the immersion phase a net pressure gradient of about 400 kPa is required to promote sufficiently rapid flow of creosote into the timber to effect treatment within three to four hours. Thus a total pressure of 600 - 800 kPa is applied to the creosote during the immersion phase. When the pressure is released and the vessel is drained the relatively high pressure within the timber, together with the vacuum which is drawn on the treating vessel, causes creosote to flow out of the timber. This empties the lumena of the cells leaving a protective layer of creosote around the cell walls.

1.5.3.5 The Bethell and Lowry full cell processes

When timber is to be treated with water-borne preservative, the Bethell process is normally used. This is designed to fill with preservative all the voids in the penetrated zone. The Lowry process is designed to achieve the same end, and is used for the application of creosote to timbers which are to be used in situations such as pier piles where the level of protection afforded by an empty cell treatment would not be adequate.

These techniques consist essentially of an initial vacuum which withdraws much of the air from the timber. Following this, the timber is immersed in preservative to which a high pressure is

applied in order to drive the liquid deeply into the timber. The level of initial vacuum used is usually at least -60 kPa and this is held for up to 60 minutes depending on the species being treated and the thickness of the pieces. When water-borne preservatives are being applied a pressure of about 1240 kPa is usually applied during the immersion phase and this is sustained for between 30 minutes and three hours, according to species and end use. The Lowry process traditionally involves rather lower immersion pressures which are sustained for slightly longer - usually one to four hours.

Although reclamation of excess preservative at the end of treatment does not form part of the concept of a full-cell treatment, a short post-immersion vacuum is frequently applied before the timber is removed from the treating vessel. This cleans up the timber's surface somewhat, thus making it more pleasant to handle, and is thought to help relieve any compression of the surface layers of the timber.

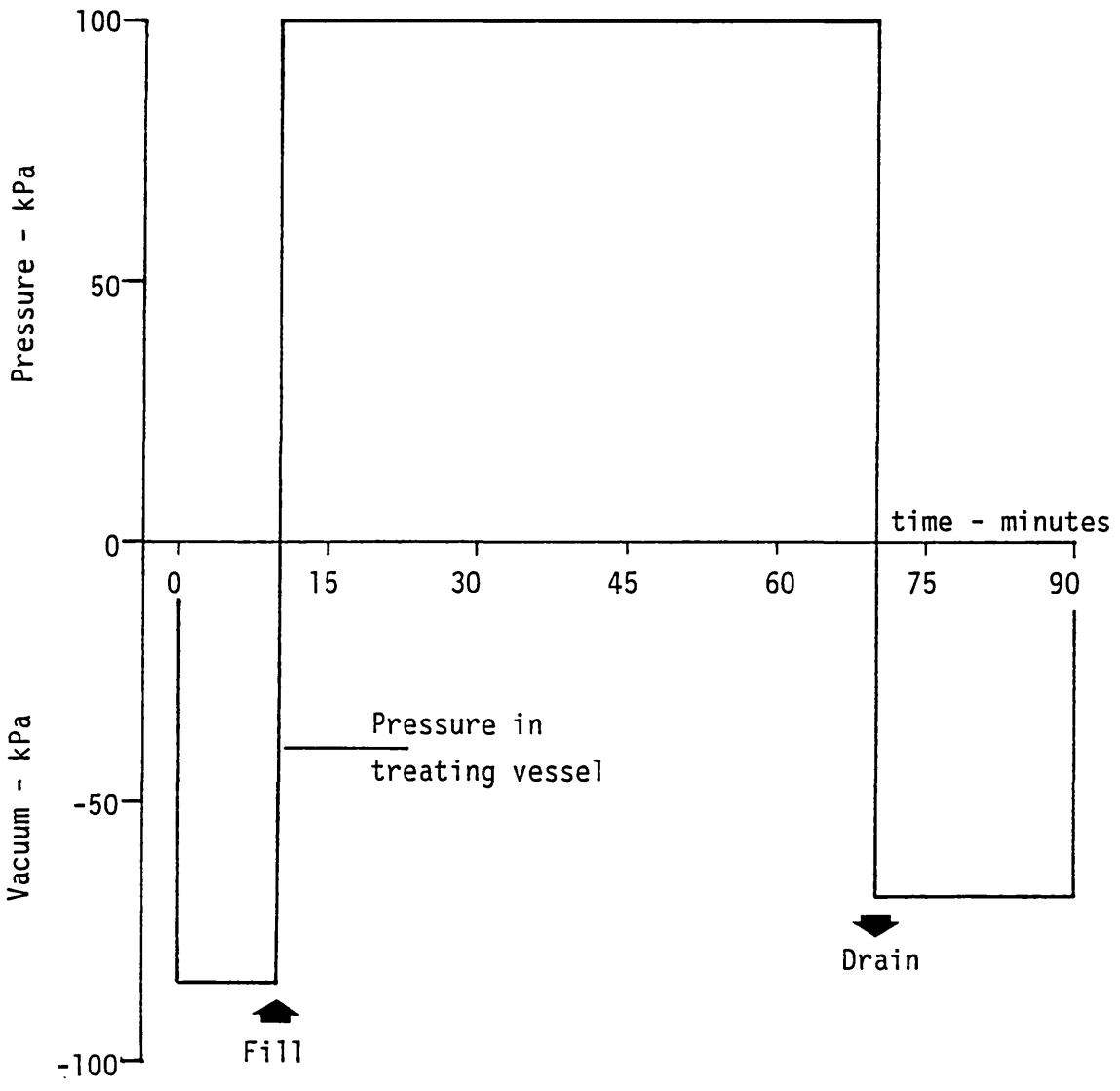
1.5.3.6 Impregnation of spruce by the DVP process

In Sections 1.5.3.1 through 1.5.3.5, the philosophy which underlies each of the main impregnation processes for seasoned timber has been outlined. A more detailed consideration of each process would reveal its shortcomings and limitations and, in the context of the present investigation, it is appropriate to focus on the problems encountered when the DVP process is used to apply organic-

solvent preservative to spruce.

Figure 1.16 represents the DVP schedule (colloquially termed the 'one-bar-schedule' because of the magnitude of the pressure applied during the immersion phase) for the treatment of spruce which was to be found in, for instance, British Wood Preserving Association specifications (e.g Leaflet No 4), when this present work was started (1977).

Figure 1.16 A DVP process for the impregnation of spruce



Following treatment by this schedule, spruce timber is not touch-dry but runs with surface liquid. This is presumably a consequence of the second vacuum being less severe than the first.

Depth of penetration of preservative following treatment by the above schedules is widely regarded as 'rather low' (BRE Technical Note 24 1975) although when this work was started there appeared to be little published information which sought to quantify this. It was considered (BRE Technical Note 24 1975, Purslow 1976) that uptake of preservative was correspondingly low but Kyte and Saunders (1978) reported liquid retentions twice to three times those quoted by Purslow (1976). This divergence in findings was assumed to be largely a reflection of the considerable variation displayed by different samples of spruce in their amenability to treatment by the DVP process.

The poor penetration of preservative noted above cannot apparently be significantly improved by using higher immersion pressures (Liese 1979) and it has sometimes been concluded that spruce cannot be preserved sufficiently well for it to be used in high-hazard situations where a 60 year service life is required (British Standard 5268 part 5). This conclusion has potentially far reaching implications because a significant proportion of North European forest resources are species of spruce. To be limited to using these species only in low-hazard and 'temporary' situations is not particularly satisfactory. There is a clear need, therefore, to develop improved techniques for the industrial preservation of timber from these species.

1.6 Scope and aims

Picea spp. comprise a significant proportion of North European forest resources. The utilisation of spruce timber (whitewood) is, however, being curtailed by limitations in the techniques available for its impregnation with preservative to ensure that it gives satisfactory service in situations where a decay risk exists.

When this investigation was started, the double vacuum with pressure schedules which were in use had been devised somewhat empirically by extrapolation from the technology devised for the double vacuum treatment of redwood sapwood. Many of these schedules are still in use but they cannot be considered to be entirely satisfactory in consequence of the following problems.

1. Irregular penetration. The schedules do not produce uniformly deep lateral penetration of preservative. Within a single piece of timber, lateral penetration may be barely measurable at some points and unnecessarily deep elsewhere.

2. High uptakes. Preservative uptakes are often much higher than is considered industrially acceptable. Uptakes in the region of 90 l/m^3 are sometimes recorded and these are three times the level usually considered economically viable.

3. Surface wetness. Following DVP treatment, whitewood often drips preservative from its surface. However, one of the advantages traditionally associated with double vacuum treatment of redwood is that its surface is touch dry at the end of the process.

4. Length of treatment. The duration of, for instance, the one bar DVP schedule (ca. 100 mins) is often regarded as excessive in comparison with the 25 - 30 minute schedules employed for the treatment of redwood sapwood.

The desirability of improving the techniques available for the preservative treatment of whitewood is thus clear. The present investigation deals primarily with organic-solvent preservatives and has the following aims.

1. To develop appropriate techniques to monitor the fluid flow phenomena which occur during the process of timber treatment.

2. To examine the existing schedule for the organic-solvent preservative treatment of spruce and to obtain more precise information about the performance of the standard DVP schedule, than has hitherto been published.

3. To investigate means whereby the techniques available for the industrial impregnation of spruce may be improved.

2 MONITORING FLUID FLOW IN TIMBER

2.1 Direct observation of liquid movement

In pursuance of the first aim of this investigation, consideration was given to devising a technique with which to investigate the route taken by white spirit flowing into spruce.

Although it is generally tacitly accepted that the flow of gases through timber can be measured only indirectly, by monitoring pressure and volume changes in and around a sample, several workers have devised techniques to observe liquid flow more directly.

Bailey (1913) passed suspensions of carbon-black in water through timber samples which he then sectioned and examined microscopically for carbon-black deposits on the walls of those channels through which flow had occurred. However, Bailey's results should perhaps be interpreted with caution because of the potential for blockage of small pores within the timber by the suspended carbon-black.

To overcome this blockage problem, Wardrop and Davis (1961) used a range of water soluble salts in the permeating liquid. After introduction into the timber these were precipitated from solution and subsequently located in thin sections by light microscopy.

As indicators of flow-paths, water soluble salts, and indeed water soluble dyes, have the drawback that they can diffuse away from

the primary flow channels during the interval between their introduction in solution and their precipitation onto the cell walls. To minimise this, Petty (1970) used basic fuchsin solution, wherein an oxidative reaction caused the fuchsin to become fixed to tracheid and vessel walls and thus gave a more accurate indication of the channels through which flow occurred.

All the above-mentioned work used water as the carrier solvent. In the context of this investigation of the flow of organic-solvent preservatives into timber, a question inevitably arises as to how far results from such work may be applied to non-polar solvents, since there are certain important differences between water and white spirit type solvents. The former is polar and interacts with the wood cell wall, penetrating and becoming chemically bound to the cellulose in the S2 and S1 layers causing them, and the piece of timber as a whole, to swell. It also has a comparatively high surface tension (73 Nm^{-1}) and often exhibits an advancing contact angle in excess of 90°. White spirit on the other hand is a non-polar solvent and shows no significant chemical interaction with cellulose. It can neither enter nor pass through wood cell wall material although, as its advancing contact angle against cellulose is much less than 90°, it readily flows along the surface of cell walls. It has a surface tension of about 23 Nm^{-1}.

These differences indicate that there is potential for water and white spirit to behave differently within timber and to travel via different routes and at different rates. Thus, the results of the

flow path investigations mentioned above cannot with confidence be applied to the flow of non-polar solvents, and additional investigation using non-polar solvents is clearly necessary.

An essential pre-requisite for monitoring flow paths within timber is a tracer which travels with the solvent and which can be located in thin sections. Three types of tracers were considered for this present work, namely:-

1. Suspended solid matter
2. Soluble colour tracers
3. Immobilised liquid

1. Suspended solid matter. Carbon-black was used by Bailey (1913) to follow the flow of water through timber. This type of approach was considered inappropriate for the present work for two reasons. Firstly, it is technically very difficult to prepare a stable suspension of sub-micron size particles in a solvent such as white spirit. Usually it is necessary to incorporate relatively high levels of resin in the solvent to achieve a stable dispersion. In recent years a colloidal iron oxide pigment dispersion, which may be infinitely diluted with white spirit, has become available. However, this is golden brown in colour and so is not readily visible at low concentrations in timber sections. Secondly, using suspended solid matter can cause occlusion of small flow channels. Spruce is a material in which the flow paths

are known to be very small in places and so any results obtained using suspended solids as the tracer would always have uncertainties associated with them.

2. Soluble colour tracers. For macroscopic assessment of the extent of liquid penetration into timber, the use of oil soluble dyes is well known (BRE Technical Note 24 1975). Exploratory work was therefore undertaken to assess their suitability for the present work. This work revealed three main reasons why oil soluble dyes are not suitable as tracers which are subsequently to be located in thin sections by light microscopy, namely:-

   a. their weak colour
   b. their low solubility in aliphatic solvents
   c. the fact that non-polar solvents do not enter the wood cell walls.

   As a consequence of c. the wood cell walls themselves, around a flow path, are not coloured. When thin sections are examined under the light microscope, a very thin deposition of dye on the surface of the S3 layer is the only indication of the location of flow paths. This deposit is only visible if its colour is very intense. Because oil soluble dyes do not have the vividness of water soluble dyes, such as malachite green for instance, the requisite intensity of colour is not present at low loadings of treating liquid.

Therefore, whereas oil soluble dyes are adequate as indicators of gross penetration, they are not suitable for examination of individual flow paths.

3. Immobilised treating liquid. The problem, mentioned above, of a tracer being present only as a thin deposit over the surface of the S3 layer may be overcome if all the treating solution is solidified 'in situ'. This approach has the advantage that it can substantially eliminate the risk of 'secondary penetration' occurring between impregnation of the timber sample with liquid, and examination of sections under the microscope. Using this technique it should be possible to map both the extent and pattern of the penetration which occurs in a given time.

A technique of this kind was used by Erickson and Balatinecz (1964) who studied flow paths by impregnating Douglas Fir with styrene monomer which they polymerised by heating it to 90° for two hours. However, for the purposes of the present work it was considered,for reasons already outlined, that the carrier solvent had to be an organic-solvent of the white spirit type, and neither the resin used by Erickson & Balatinecz (1964) nor that used for somewhat similar work by Thompset (1970) could be carried in this solvent. Consequently this approach was not pursued.

As an alternative approach to immobilising the treating liquid, the possibility of solidifying white spirit type solvents in situ by cooling them to below their wax point (ca. -50°C) was considered. However, as dry wood is a very good insulator, it was thought that even liquid nitrogen would not induce sufficiently rapid cooling in the centre of the sample for this technique to be effective.

Each of the three techniques considered for the purposes of tracing the flow paths of white spirit into spruce was thus rejected as being unsuitable in some way. It was concluded that a technique which would yield sufficiently accurate information to chart the route and speed of a white spirit type solvent flowing into spruce could not be devised.

2.2 Indirect observation of liquid movement

As noted in Section 2.1, because it is not possible to follow directly the flow of gas through timber, measurements of gas movement are made indirectly by monitoring, for example, pressure and volume changes around a timber sample. In a similar way, because it was concluded that it was not readily possible to devise a technique to follow directly the flow of white spirit-type solvents through spruce, the focus of this part of this investigation was directed towards measuring pressure and volume changes which could be assumed to be the result of liquid flow within the timber. Three techniques, briefly outlined below, were devised for this purpose and are reported in this section (2.2).

1. Monitoring liquid inflow during large scale impregnation (Section 2.2.1)

   By monitoring the entry of liquid into a sealed treatment chamber containing timber, the rate of liquid flow into wood during the immersion phase of an industrial timber impregnation schedule was monitored. In interpreting the results particular attention was paid to the relationship between rate of change of flow and elapsed time.

2. Monitoring liquid flow into timber on a small scale
(Section 2.2.2)

A piece of apparatus was designed in which the flow of liquid through a relatively small area (8.55 cm^2) into timber samples was measured. This apparatus was used to investigate, in particular, the effect of pressure gradient on flow rate.

3. Measuring pressure inside timber
(Section 2.2.3)

A novel technique was devised to monitor pressure variations within timber. Particular attention was paid to the pressure fluctuations which occurred during the period of an industrial impregnation schedule, and those changes which occurred during the immersion phase of such a schedule were interpreted on the assumption that they occurred in consequence of liquid movement.

2.2.1 Monitoring liquid inflow during large scale impregnation

2.2.1.1 Selection and basis of the technique

This section of work examined the rate of entry of organic-solvent preservative into spruce wood. The technique used consisted of enclosing the timber to be treated within a treatment vessel and then monitoring the flow of liquid into that vessel during the immersion phase of an industrial timber impregnation schedule.

At the commencement of this section of work, four techniques for following liquid inflow were considered, viz.

(i) A graduated sight glass
(ii) A conventional flow meter
(iii) Load cells
(iv) 'Constant volume' flow monitoring

For the reasons outlined below the first three were considered unsuitable and as a result the 'constant volume' flow monitoring technique (iv) was devised and used.

(i) Sight glass

A graduated sight glass to monitor liquid movement seemed at first to be an obvious technique to employ. It was envisaged that the treatment vessel should be equipped with a sight glass. This would

be positioned above the vessel and the vacuum line would connect with its top. At the beginning of the immersion phase liquid would be run into the vessel from the bottom until the sight glass was almost full. The liquid inlet line would then be sealed. Any desired pressure would be applied via compressed gas in the top of the sight glass and the level of the liquid surface within the sight glass would be monitored during the immersion phase.

A piece of apparatus embodying this arrangement was constructed, but in certain circumstances proved impossible to use effectively. It was found that when organic-solvent preservative was run through a constriction (such as a partly open valve) into a chamber in which a vacuum greater than ca. -60 kPa existed, the preservative 'erupted' into very large quantities of foam which filled the treating vessel and sight glass. Although evolution of foam may be minimised by enlarging the constriction on the inlet line and admitting liquid to the vessel very rapidly, rapid filling introduces difficulties in stopping the inflow of liquid at a suitable instant to keep the liquid surface within the graduated portion of the sight glass throughout the observation period. Conversely, very slow admission of liquid allows for degassing and collapse of the foam but causes unacceptable lengthening of the time taken to fill the treating vessel.

For these reasons, the use of a graduated sight glass was rejected, although it is acknowledged that a configuration of pipe-work other than the one outlined above may well have been more satisfactory.

(ii) Conventional flow meters

The flow meters which were commercially available were examined to ascertain if any was suitable for monitoring the entry of preservative liquid into a treating vessel during the immersion phase of an impregnation schedule.

The principal requirements for a meter to be considered suitable were that it should be accurate at the very low flow rates which were anticipated, and that it should be compatible with dyed organic-solvent preservatives. A flow meter fulfilling these two criteria could not be found and so the use of conventional flow meters was rejected.

(iii) Load cells

The absorption of preservative liquid by a stack of timber during impregnation may be monitored by monitoring changes in the buoyancy of that timber. Indeed, workers such as Arganbright & Resch (1970) have monitored buoyancy of samples of timber by attaching them to a load cell within an impregnation vessel and following changes in weight.

In many respects, buoyancy monitoring was considered to be the most satisfactory option, from a technical standpoint, for monitoring preservative uptake. Unfortunately, however, this technique could not be employed because a suitable load cell could not be obtained.

(iv) 'Constant volume' flow monitoring

This technique was partly developed by E.M. Pearce (personal communication) and had the following basis.

When a vessel containing partially evacuated timber is filled with liquid, is pressurised and then sealed, the pressure within the liquid in the vessel drops as liquid flows into the timber. To maintain the desired immersion pressure the vessel must, therefore, be repressurised by the introduction of further liquid. If the magnitude of the pressure-drop which is allowed to occur before repressurisation is undertaken is kept constant, the volume of liquid required to repressurise the vessel is also constant.

In the above circumstance, the frequency with which 'constant volume' aliquots of liquid need to be introduced to repressurise the vessel, is a measure of the speed with which liquid is entering the timber.

This technique is perhaps somewhat complex in its conceptual basis, but is quite straight forward to set up in practice and so was adopted for this section of the work.

The only significant shortcoming associated with this technique was that no satisfactory method was devised to quantify the volume of liquid entering the vessel during each repressurisation. Consequently this technique was not applied to compare absolute

rates of liquid flow into timber, treated in different loads. Rather, the technique was used repeatedly, purely to monitor the variation in rate of liquid absorption by timber during the period of the immersion phase of an industrial timber impregnation schedule.

2.2.1.2 'Constant volume' flow monitoring - experimental

2.2.1.2.1 Apparatus used

Impregnation apparatus suitable for conducting experimentation on the above basis is represented schematically in Figure 2.1, and the treating vessel of the apparatus used is shown in Plate 2.1. This apparatus was used in the following way to monitor variations in the rate of liquid absorption during the immersion phase of industrial timber impregnation schedules:

An initial vacuum was drawn on the treating vessel and at the end of a ten minute evacuation period, preservative liquid was pumped in. When the vessel was filled, the vacuum line was closed. The liquid pump continued to drive liquid into the vessel so that the pressure in the liquid in the vessel rose. At a preset pressure (Section 2.2.1.2.5), the pressure-sensitive switch indicated in Figure 2.1 closed the solenoid valve on the delivery line. Because this valve closure totally sealed the treating vessel, the pressure in the liquid within it dropped only in consequence of liquid entering the timber. As the pressure dropped through this cause, the pressure-sensitive switch, sensing the change in pressure, opened the solenoid valve to admit more liquid. As liquid entered the vessel, the pressure within it rose once again to the level at which the pressure sensitive switch reclosed the solenoid valve.

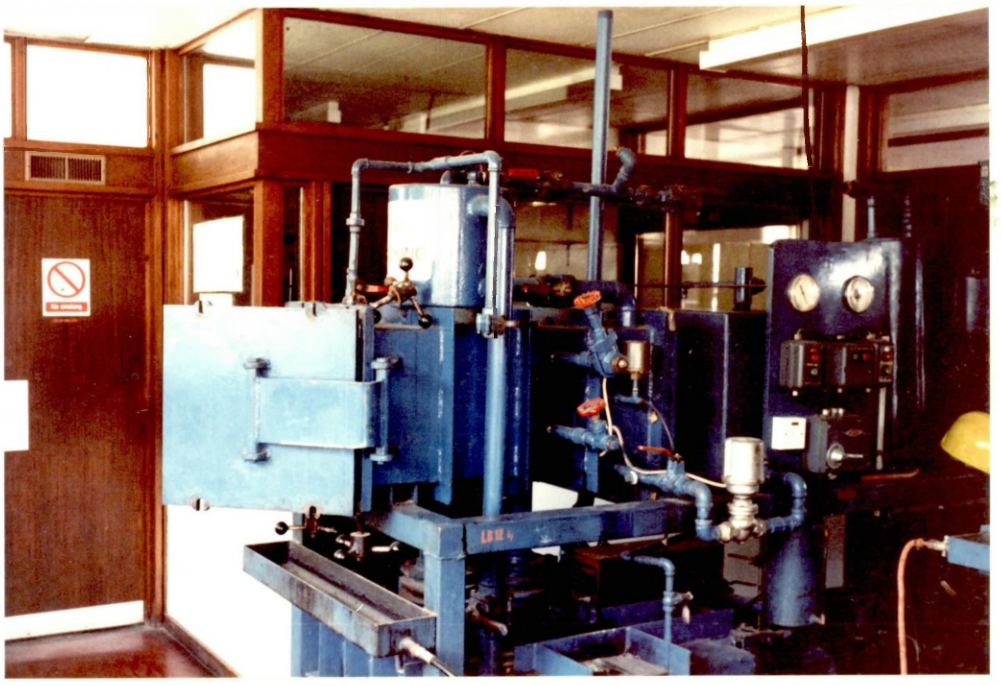
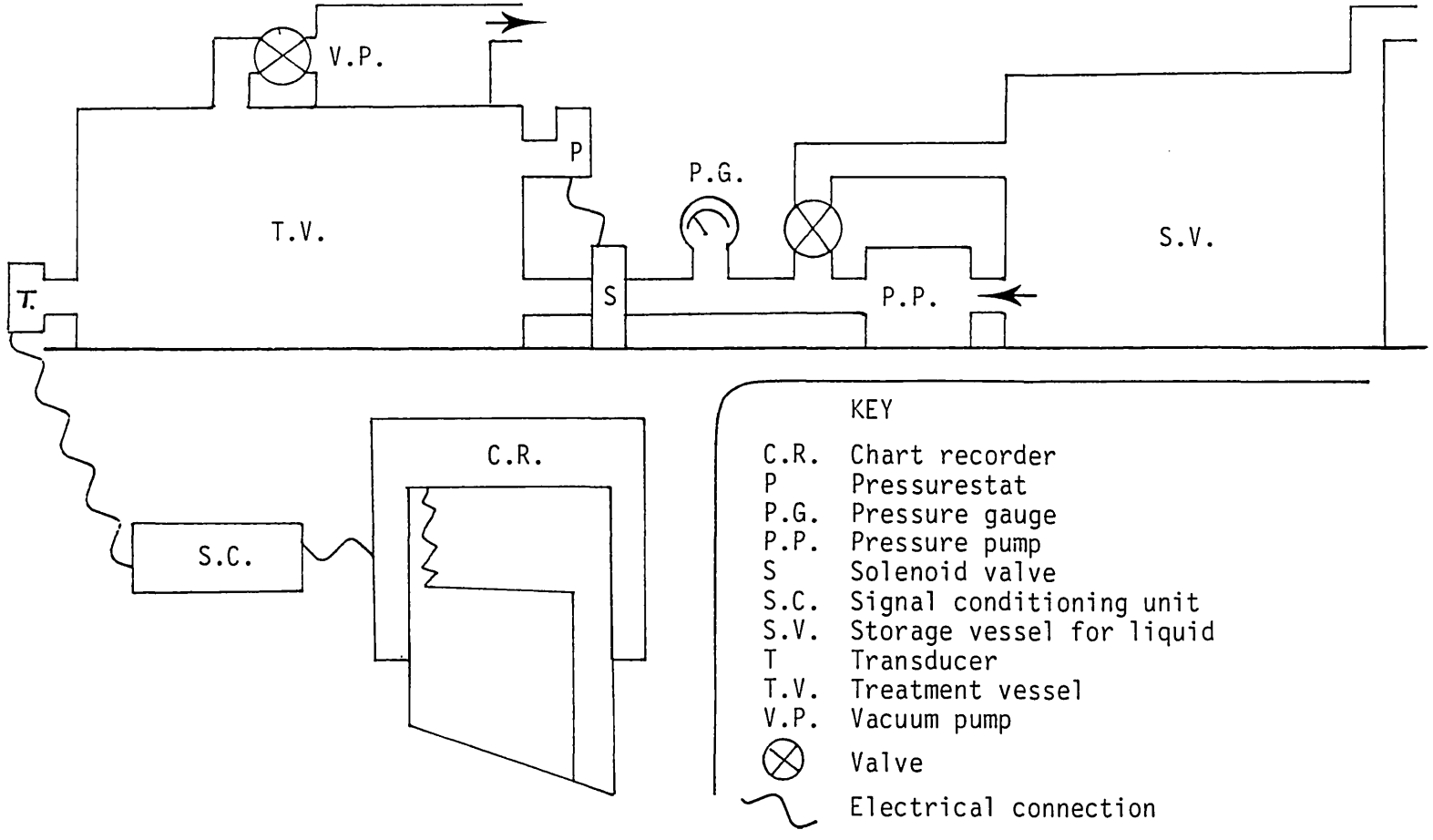


Plate 2.1A Plates 2.1A and B showing the impregnation apparatus used for work described in Section 2.2.1.2 and Section 3



Plate 2.1B

Figure 2.1 Schematic diagram of 'Constant-volume' flow measuring apparatus



The pressure in the liquid delivery line was held steady at 20 - 50 kPa above the working pressure in the vessel throughout each treatment. Thus, the volume of liquid required to raise the vessel's pressure from the level at which the solenoid valve was opened to that at which it reclosed was assumed also to be constant throughout each treatment. On this assumption the solenoid valve was regarded as admitting a constant (albeit unknown) quantity of liquid each time it opened and measurement of the rate at which liquid entered the vessel could be accomplished by recording the frequency with which the solenoid valve opened. This was conveniently done by using a pressure transducer and pen recorder to monitor fluctuations of the pressure in the liquid within the treatment vessel.

Pressure transducer and its output

In general, transducers convert mechanical stimuli into electrical signals. Many transducers, including the Pye Ether, type UP, strain gauge pressure transducer used for the present work, consist essentially of a Wheatstone bridge in which one or more of the elements is distortion sensitive (i.e. is a strain gauge). An excitation current is supplied to the resistor network of the Wheatstone bridge and this, on passing through the bridge, produces an electrical signal. After any appropriate attenuation and/or stabilisation, this signal may be fed into any convenient display or recording apparatus. In the present work, both bridge excitation and signal attenuation were effected using a Pye Ether signal

conditioning unit. The signal was recorded and displayed using a Perkin Elmer 56 pen recorder, and the system was calibrated such that deflections of the pen could be quantitatively interpreted in terms of pressure fluctuations within the vessel.

2.2.1.2.2 Timber samples

In order to investigate the flow of liquid laterally into seasoned spruce, most of the experimentation was undertaken using 5 cm square cross-section, 20 cm lengths of air dry sawn spruce, each of which was sealed across both of its transverse surfaces using epoxy resin (see Section 2.2.1.2.3). In addition, a small number of experiments was performed in which longer samples, having one or both ends unsealed, were used (Table 2.1). This was in order to note any substantial changes in pattern of rate of liquid uptake which might be observed with longitudinal penetration.

From Plate 2.1 it may be noted that, when the timber samples were loaded into the plant, the samples in each layer of timber were spaced from each other horizontally. In addition, successive layers were separated vertically by small sticks. Thus, the treating liquid had unimpeded access to almost the whole surface of each sample.

Table 2.1 Details of spruce samples used (Section 2.2.1.2.2) and treating schedules used (Section 2.2.1.2.5) during 'constant-volume' flow monitoring experimentation

Experiment Reference Number	Number of Samples	Size of Samples mm	End Sealing	Pressure During Immersion kPa	Duration of Immersion Minutes
1	26	50 x 50 x 900	One End	103	60
2	28	50 x 50 x 900	One End	103	60
3	17	mixed x 900	Both Ends	103	60
4	30	12 x 100 x 900	Both Ends	103	60
5	60	20 x 20 x 295	None	103	60
6	32	50 x 50 x 445	None	103	60
7	30	50 x 50 x 900	None	103	60
8	30	50 x 50 x 900	None	103	60
9	30	50 x 50 x 450	One End	103	60
10	75	50 x 50 x 200	Both Ends	103	60
11	75	50 x 50 x 200	Both Ends	103	60
12	75	50 x 50 x 200	Both Ends	103	60
13	75	50 x 50 x 200	Both Ends	103	60
14	75	50 x 50 x 200	Both Ends	103	60
15	75	50 x 50 x 200	Both Ends	103	60
16	75	50 x 50 x 200	Both Ends	103	60
17	75	50 x 50 x 200	Both Ends	103	60
18	75	50 x 50 x 900	None	172	15
19	75	50 x 50 x 200	Both Ends	207	30
20	75	50 x 50 x 200	Both Ends	172	30
21	75	50 x 50 x 200	Both Ends	207	15
22	75	50 x 50 x 200	Both Ends	207	15
23	75	50 x 50 x 200	Both Ends	207	15
24	75	50 x 50 x 200	Both Ends	172	15
25	49	50 x 50 x 200	Both Ends	103	>150

2.2.1.2.3 End-grain sealing

All the end (and other surfaces) sealing described was effected using an 'Araldite' epoxy resin mixture comprising two-parts (by weight) resin 'PZ 820' and one-part hardener 'HZ 820'. These two components were gently mixed to avoid entrainment of air-bubbles, and then applied to the surfaces to be sealed. The viscous mixture was occasionally applied by brush, in which case two coats, applied 24 hours apart, were required to produce an effective barrier coating ca. 0.5 - 1.0 mm thick. More usually, the resin and hardener mixture was allowed to cure slightly so that its viscosity rose to a level where it could be applied by palette knife to the horizontal transverse surface of samples standing on end. This latter procedure facilitated the application of a robust 1.0 - 1.5 mm layer of resin in a single coat. Cure time was about 24 hours, although it took several days for the coating to harden completely.

2.2.1.2.4 Treating liquid

The treating liquid used was an industrial organic-solvent preservative, Protim 80, which was coloured with 0.5% waxolene blue A dye.

2.2.1.2.5 Impregnation schedules

The impregnation schedule which was taken as the 'starting point' in this section of work was the one bar DVP schedule which has been described in Section 1.5.3.6. Thus, in all instances, an initial vacuum of -85 kPa was applied to the timber for ten minutes.

At the end of the initial vacuum, treating liquid was run into the vessel and pressurised, usually to 103 kPa. To ascertain whether the pattern of rate of liquid uptake differed at higher pressures, seven of the twenty-five tests undertaken employed an immersion pressure greater than 103 kPa. In three of these tests, an immersion pressure of 172 kPa was used while in the remaining four tests 207 kPa was applied (Table 2.1). The duration of the immersion phase is also indicated in Table 2.1 and from this it can be seen that a sixty minute immersion period was almost invariably used for the schedules in which the immersion pressure was 103 kPa. In certain instances, usually those involving higher immersion pressures, the immersion time had to be curtailed, in response to the requirements of other experimentation being pursued simultaneously. However, in Experiment 25, immersion at 103 kPa was continued for over two and a half hours.

In Experiments 1 through 24, a final vacuum of -85 kPa was applied for ten (exceptionally twenty) minutes after the vessel had been drained.

2.2.1.2.6 Uptake of treating liquid

The uptake of liquid by each sample in each charge was determined gravimetrically by weighing each sample to an accuracy of 0.2 g immediately prior to, and immediately following, treatment.

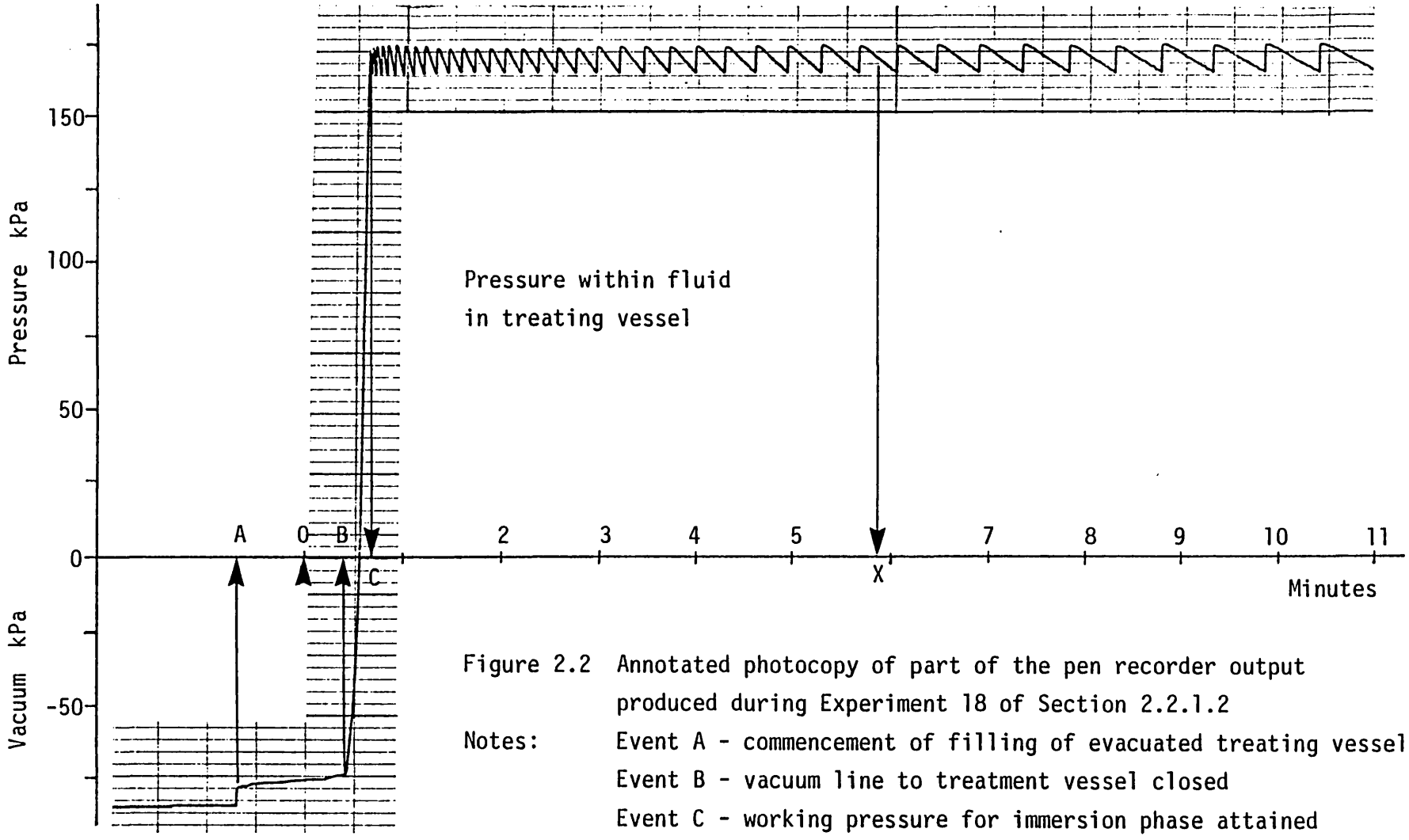


Figure 2.2 Annotated photocopy of part of the pen recorder output produced during Experiment 18 of Section 2.2.1.2

Notes: Event A - commencement of filling of evacuated treating vessel
Event B - vacuum line to treatment vessel closed
Event C - working pressure for immersion phase attained
O and X - see Sections 2.2.1.3.1 and 2.2.1.3.2

2.2.1.3 'Constant volume' flow monitoring - results

2.2.1.3.1 Designation of time 'zero'

Figure 2.2 is an annotated photocopy of part of a typical pen-recorder output produced during the experimentation described in Section 2.2.1.2.

In industrial practice, the instant at which the immersion pressure is reached (Event 'C' on Figure 2.2) is regarded as time 'zero' from which the duration of the immersion phase is measured. However, this convention was considered inappropriate to the interpretation of the results obtained from the present experimentation. This was because filling of the vessel (Event 'A' on Figure 2.2) commenced as much as 2.7 minutes prior to Event 'C', during which time substantial absorption of liquid by the timber lowest in the stack could have occurred. Conversely, Event 'A' is only an appropriate zero point for the timber lowest in the treatment vessel. As the best compromise available, time 'zero' was arbitrarily designated to be at a point midway between events A and C - i.e. at the point marked 'O' in Figure 2.2.

2.2.1.3.2 Interpretation of pen-recorder output

When this experimentation was undertaken it was anticipated that interpretation of the pen-recorder output would be based on a cumulative total of solenoid valve actuations against time.

However, designation of time zero in the manner described in Section 2.2.1.3.1 highlighted the fact that an unmeasured volume of liquid could enter the timber prior to Event 'C' on Figure 2.2. Consequently it was considered more appropriate to analyse the charts to obtain relative estimates of the rate of absorption of treating-liquid by the timber at intervals throughout the immersion period.

The rate at which liquid was absorbed by the timber was inversely proportional to the interval between successive solenoid valve actuations. Thus, to interpret each pen recorder chart, the intervals between successive valve openings were measured and these were tabulated (Table 2.2) against elapsed time. Elapsed time was measured as the period between time '0' and the instant midway between the two valve actuations in question. For example, the point 'X' on Figure 2.2 occurred 5.9 minutes after time '0' and is midway between two solenoid valve actuations which occurred 0.39 minutes apart. Thus in Table 2.2 a value for t_i of 0.39 minutes is recorded against an elapsed time (t) of 5.9 minutes in Experiment 18.

2.2.1.3.3 Net uptake of liquid

From the initial and final weights recorded for each sample, the net uptake of preservative was calculated. Average values for each load of timber are presented at the foot of each set of values for t_i and t in Table 2.2.

Table 2.2 Numerical analysis of pen recorder charts to show duration in minutes of time interval (t_i) between successive solenoid valve actuations, against elapsed time (t) in minutes, during the immersion phase of double vacuum treatment of spruce as detailed in Table 2.1. Figures at the foot of each set of data are average uptakes of preservative.

Experiment 1		1 continued		1 continued		Experiment 2		2 continued		2 continued			
t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t
0.4	5.5	0.7	15.7	1.15	33.7		1.17	0.89	10.35	2.61	41.35		
0.4	5.9	0.75	16.5	1.2	34.9	0.23	1.4	0.91	11.25	2.76	44.0		
0.4	6.3	0.8	17.2	1.2	36.1	0.25	1.65	1.0	12.2	3.04	46.9		
0.4	6.8	0.8	18.0	1.24	37.3	0.26	1.9	1.09	13.27	3.08	50.0		
0.4	7.1	0.8	18.8	1.25	38.5	0.29	2.17	1.15	14.4	3.16	53.25		
0.45	7.6	0.8	19.6	1.3	39.9	0.3	2.47	1.25	15.6	3.33	56.4		
0.4	8.0	0.85	20.4	1.35	41.1	0.34	2.77	1.26	16.85				
0.5	8.5	0.85	21.4	1.35	42.5	0.36	3.12	1.35	18.15				
0.45	9.0	0.9	22.2	1.4	43.9	0.4	3.5	1.44	19.57				
0.6	9.5	0.95	23.2	1.4	45.3	0.41	3.9	1.52	21.05				
0.55	10.1	0.96	24.1	1.45	46.9	0.48	4.42	1.6	22.6				
0.6	10.6	1.0	25.1	1.5	48.3	0.53	4.85	1.71	24.25				
0.6	11.2	1.0	26.1	1.5	49.8	0.54	5.4	1.79	26.0				
0.6	11.8	1.0	27.6	1.55	51.3	0.59	6.0	1.85	27.85				
0.6	12.4	1.0	28.1	1.6	52.9	0.61	6.6	2.0	29.8				
0.65	13.0	1.05	29.2	1.6	54.5	0.69	7.47	2.1	31.85				
0.65	13.6	1.1	30.3	1.6	56.1	0.73	7.92	2.26	34.0				
0.65	14.4	1.15	31.4	1.65	57.7	0.78	8.67	2.36	36.35				
0.65	15.0	1.15	32.5	1.7	59.5	0.84	9.5	2.49	38.75				
				112 l/m³						143 l/m³			

Table 2.2 continued

Experiment 3		Experiment 4		4 continued		Experiment 5		5 continued		5 continued		5 continued	
t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t
0.22	1.3	0.2	0.9	1.8	26.35	0.09	1.42	0.35	7.32	0.70	21.82	1.12	47.0
0.6	1.7	0.25	2.1	1.95	28.4	0.1	1.52	0.37	7.68	0.73	22.56	1.15	48.15
0.85	2.5	0.3	2.5	2.05	30.2	0.1	1.63	0.38	8.05	0.75	23.3	1.22	49.32
1.0	3.3	0.35	2.8	2.15	32.35	0.1	1.72	0.4	8.45	0.75	24.05	1.20	50.55
1.15	4.4	0.4	3.2	2.25	34.55	0.12	1.85	0.42	8.88	0.76	24.8	1.20	51.77
1.24	5.6	0.45	3.7	2.35	36.85	0.12	1.98	0.41	9.3	0.8	25.62	1.23	53.0
1.4	6.9	0.45	4.1	2.6	39.4	0.14	2.11	0.45	9.73	0.8	26.41	1.25	54.3
1.65	8.5	0.55	4.6	2.7	42.0	0.15	2.25	0.45	10.12	0.81	27.24	1.26	55.5
1.95	10.3	0.6	5.2	2.8	44.8	0.15	2.40	0.45	10.62	0.84	28.16	1.3	56.8
2.35	12.5	0.65	5.8	2.9	47.7	0.16	2.56	0.5	11.1	0.85	28.9	1.34	58.1
2.95	15.1	0.65	6.5	3.05	50.75	0.16	2.75	0.49	11.6	0.86	29.76	1.3	59.45
3.65	18.4	0.75	7.1	3.2	53.9	0.18	2.94	0.49	12.1	0.90	30.66		
4.3	22.4	0.8	7.95	3.4	57.3	0.2	3.14	0.5	12.6	0.92	31.58		
5.2	27.2	0.8	8.75			0.2	3.32	0.55	13.12	0.90	32.49		
5.8	32.7	0.85	9.6			0.21	3.55	0.53	13.9	0.90	33.39		
6.6	39.1	0.9	10.45			0.22	3.76	0.55	14.2	0.96	34.34		
7.05	45.8	1.0	11.4			0.25	4.0	0.55	14.76	1.0	35.32		
7.55	52.9	1.0	12.45			0.25	4.26	0.58	15.33	0.95	36.3		
		1.1	13.5			0.25	4.53	0.6	15.9	0.98	37.25		
		1.15	14.7			0.26	4.77	0.6	16.5	1.05	38.28		
		1.2	15.9			0.28	5.06	0.61	17.14	1.05	39.32		
		1.25	17.1			0.29	5.35	0.63	17.76	1.04	40.36		
		1.35	18.5			0.3	5.63	0.63	18.4	1.06	41.42		
		1.44	19.9			0.3	5.95	0.65	19.03	1.08	42.5		
		1.55	21.25			0.32	6.27	0.66	19.7	1.12	43.6		
		1.6	22.9			0.34	6.6	0.7	20.4	1.1	44.73		
		1.7	24.5			0.35	6.96	0.73	21.08	1.12	45.85		
27 l/m^3				93 l/m^3								121 l/m^3	

Table 2.2 continued

Experiment 6		6 continued		Experiment 7		Experiment 8		8 continued		8 continued			
t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t
0.1	1.3	1.5	21.1	0.26	0.94	0.2	4.1	0.9	19.3	1.75	54.4		
0.15	1.4	1.45	22.6	0.4	1.27	0.25	4.3	0.9	20.2	1.8	56.1		
0.2	1.6	1.61	24.2	0.58	1.77	0.25	4.7	0.95	21.1	1.8	57.9		
0.2	1.8	1.68	25.75	0.78	2.45	0.3	5.0	0.95	22.1				
0.28	2.02	1.71	27.45	1.0	3.34	0.35	5.3	1.0	23.1				
0.3	2.3	1.73	29.2	1.25	4.45	0.4	5.7	1.0	24.1				
0.38	2.65	1.8	30.95	1.5	5.85	0.4	6.0	1.05	25.2				
0.4	3.05	1.83	32.83	1.79	7.5	0.4	6.4	1.1	26.3				
0.48	3.52	2.0	34.73	2.11	9.45	0.4	6.8	1.15	27.4				
0.48	3.95	2.02	36.75	2.34	11.7	0.45	7.8	1.18	28.5				
0.53	4.45	2.05	38.8	2.69	14.2	0.45	7.7	1.2	29.7				
0.6	5.05	2.12	40.9	2.93	17.0	0.5	8.2	1.2	30.9				
0.6	5.68	2.1	43.0	3.25	20.12	0.5	8.7	1.2	32.1				
0.65	6.32	2.32	45.2	3.6	23.5	0.55	9.3	1.25	33.5				
0.7	7.0	2.32	47.55	3.84	27.25	0.55	9.8	1.3	34.7				
0.8	7.73	2.18	49.85	4.29	31.35	0.6	10.4	1.35	36.9				
0.8	8.53	2.3	52.1	4.71	35.85	0.6	11.0	1.4	37.3				
0.9	9.4	2.35	54.4	5.14	40.85	0.6	11.6	1.4	38.8				
0.9	10.28	2.5	56.8	5.5	46.2	0.65	12.2	1.4	40.1				
1.0	11.23	2.6	59.4	7.1	52.5	0.65	12.9	1.45	41.6				
1.05	12.3					0.7	13.6	1.5	43.1				
1.1	13.4					0.75	14.5	1.55	44.7				
1.18	14.5					0.8	15.1	1.6	46.2				
1.28	15.7					0.8	15.9	1.6	47.8				
1.28	17.0					0.8	16.7	1.6	49.4				
1.3	18.3					0.8	17.5	1.65	51.1				
1.4	19.7					0.9	18.4	1.65	52.7				
		89.1 l/m^3								39 l/m^3			

Table 2.2 continued

Experiment 9		9 continued		Experiment 10		10 continued		Experiment 11		11 continued			
t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t
0.1	1.16	2.6	35.2	0.1	1.15	1.1	16.0	0.25	1.16	4.3	56.7		
0.18	1.32	2.65	37.62	0.12	1.28	1.15	17.1	0.35	1.43				
0.25	1.53	2.8	40.57	0.15	1.43	1.2	18.3	0.45	1.81				
0.32	1.82	2.9	43.4	0.17	1.59	1.28	19.53	0.55	2.3				
0.4	2.2	3.05	46.4	0.2	1.80	1.3	20.85	0.65	2.93				
0.45	2.62	3.05	49.45	0.24	2.0	1.35	22.4	0.8	3.68				
0.53	3.12	3.2	52.55	0.26	2.26	1.45	23.57	0.9	4.55				
0.6	3.67	3.25	55.75	0.3	2.56	1.38	24.95	1.1	5.55				
0.7	4.35			0.33	2.86	1.58	26.45	1.1	6.6				
0.77	5.07			0.35	3.22	1.63	28.05	1.25	7.78				
0.85	5.86			0.4	3.62	1.68	29.7	1.4	9.05				
0.95	6.8			0.45	4.0	1.68	31.35	1.57	10.57				
1.05	7.8			0.45	4.45	1.8	33.1	1.72	12.22				
1.12	8.87			0.5	4.95	1.8	34.88	1.85	14.05				
1.2	10.03			0.55	5.5	1.86	36.7	1.95	15.9				
1.3	11.3			0.58	6.03	1.95	38.63	2.2	18.0				
1.45	12.65			0.63	6.63	2.0	40.6	2.35	20.26				
1.55	14.2			0.65	7.27	2.05	42.6	2.4	22.65				
1.7	15.85			0.7	7.93	2.11	44.7	2.7	25.2				
1.8	17.6			0.74	8.65	2.13	46.8	2.8	27.9				
1.9	19.43			0.79	9.45	2.22	48.95	3.05	30.9				
2.0	21.4			0.83	10.23	2.25	51.2	3.2	34.0				
2.15	23.47			0.85	11.07	2.3	53.5	3.35	37.3				
2.15	25.62			0.9	11.92	2.35	55.8	3.52	40.8				
2.3	27.85			0.97	12.85	2.43	58.2	3.8	44.4				
2.4	30.2			1.0	13.85			4.0	48.3				
2.5	32.63			1.08	14.9			4.25	52.45				
		69 l/m^3				63 l/m^3				72 l/m^3			

Table 2.2 continued

Experiment 12		Experiment 13		13 continued		Experiment 14		14 continued		Experiment 15		15 continued	
t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t
0.3	1.17	0.15	1.04	1.31	19.12	0.25	1.47	1.41	24.15	0.1		0.49	9.71
0.45	1.53	0.18	1.2	1.38	20.46	0.29	1.72	1.43	25.55	0.12		0.48	10.2
0.6	2.05	0.22	1.39	1.45	21.84	0.35	2.05	1.45	26.98	0.15		0.5	10.68
0.74	2.70	0.24	1.61	1.43	23.35	0.38	2.42	1.49	28.46	0.18		0.5	11.19
0.8	3.50	0.32	1.9	1.5	24.75	0.43	2.81	1.55	29.95	0.2	2.02	0.5	11.7
0.72	4.25	0.38	2.38	1.5	26.28	0.5	3.28	1.58	31.5	0.2	2.32	0.52	12.2
0.88	5.05	0.4	2.64	1.58	27.81	0.5	3.75	1.6	33.12	0.22	2.46	0.53	12.72
0.88	5.90	0.43	3.05	1.69	29.46	0.57	4.3	1.65	34.74	0.24	2.68	0.54	13.27
0.9	6.8	0.5	3.53	1.7	31.18	0.6	4.89	1.68	36.4	0.28	2.92	0.56	13.8
0.98	7.75	0.55	4.0	1.79	32.9	0.65	5.5	1.7	38.05	0.27	3.18	0.56	14.37
1.02	8.75	0.62	4.62	1.8	34.73	0.7	6.19	1.72	39.77	0.30	3.45	0.59	14.9
1.18	9.85	0.65	5.36	1.88	36.58	0.73	6.9	1.8	41.5	0.29	3.75	0.57	15.48
1.24	11.05	0.7	5.9	1.88	38.41	0.8	7.67	1.81	43.3	0.3	4.03	0.59	16.62
1.62	12.5	0.72	6.6	1.81	40.28	0.82	8.48	1.88	45.14	0.31	4.35	0.59	17.21
1.94	14.25	0.78	7.38	2.0	42.2	0.89	9.31	1.88	47.0	0.31	4.69	0.6	17.8
2.05	16.25	0.8	8.18	2.08	44.2	0.92	10.2	1.92	48.86	0.34	5.0	0.61	18.42
2.2	18.38	0.89	9.04	1.98	46.22	0.98	11.19	2.0	50.83	0.38	5.32	0.6	19.05
2.3	20.6	0.92	9.9	2.1	48.29	1.0	12.18	2.0	52.83	0.35	5.67	0.6	19.65
2.54	23.02	0.97	10.87	2.07	50.37	1.05	13.19	2.04	54.84	0.38	6.03	0.61	20.28
2.68	25.62	1.05	11.88	2.2	52.5	1.09	14.29	2.05	56.88	0.38	6.42	0.67	20.88
2.79	28.34	1.12	12.98	2.26	54.78	1.11	15.39	2.07	58.90	0.39	6.79	0.66	21.57
2.9	31.18	1.12	14.1	2.32	57.08	1.15	16.5			0.39	7.18	0.68	22.2
3.0	34.1	1.22	15.28	2.45	59.4	1.2	17.7			0.4	7.58	0.7	22.88
3.2	37.2	1.29	16.54			1.23	18.9			0.41	7.98	0.68	23.65
	chart					1.27	20.14			0.42	8.39	0.7	24.2
	feed					1.3	21.4			0.44	8.83	0.7	24.9
	fail					1.38	22.72			0.45	9.24	0.69	
82 l/m³				75 l/m³				83 l/m³					

Table 2.2 continued

Experiment 15		15 continued		Experiment 16		16 continued		16 continued		Experiment 17		17 continued	
t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t
0.71	26.29	0.93	48.73	0.1		0.6	11.2	1.05	33.72	0.2	1.4	2.95	40.22
0.72	27.0	0.94	49.69	0.13	1.38	0.61	11.8	1.05		0.3	1.67	3.1	43.22
0.71	27.71	0.95	50.6	0.16	1.52	0.62	12.43	1.08	35.87	0.4	2.05	3.22	46.38
0.71	28.42	0.98	51.6	0.18	1.68	0.68	13.1	1.11	36.94	0.46	2.5	3.3	49.6
0.73	29.15	1.0	52.59	0.2	1.9	0.70	13.75	1.1	38.04	0.58	3.0	3.5	53.02
0.75	29.89	1.0	53.59	0.21	2.1	0.70	14.46	1.1	39.11	0.64	3.6	3.6	56.6
0.79	30.66	0.98	54.57	0.23	2.32	0.71	15.17	1.1	40.22	0.76	4.29		
0.79	31.45	0.98	55.52	0.25	2.55	0.75	15.87	1.12	41.31	0.8	5.08		
0.8	32.22	1.0	56.52	0.28	2.82	0.78	16.62	1.13	42.42	0.89	5.91		
0.8	33.01	1.04	57.54	0.3	3.11	0.8	17.4	1.16	43.56	0.9	6.81		
0.82	33.82	1.0	58.58	0.3	3.41	0.8	18.15	1.18	44.71	1.0	7.78		
0.81	34.6	1.03	59.6	0.33	3.75	0.81	19.0	1.2	45.9	1.1	8.82		
0.81	35.48			0.38	4.08	0.82	19.8	1.2	47.08	1.2	9.99		
0.85	36.33			0.38	4.45	0.84	20.64	1.21	48.27	1.26	11.23		
0.8	37.16			0.40	4.8	0.86	21.47	1.21	49.45	1.35	12.51		
0.87	38.0			0.40	5.2	0.85	22.32	1.2	50.68	1.5	13.92		
0.9	38.85			0.41	5.65	0.9	23.13	1.23	51.88	1.6	15.5		
0.84	39.70			0.43	6.05	0.9	24.06	1.2	53.1	1.7	17.1		
0.89	40.55			0.49	6.51	0.91	24.94	1.26	54.3	1.85	18.88		
0.88	41.43			0.49	6.95	0.96	25.89	1.26	55.58	1.91	20.7		
0.91	42.32			0.49	7.45	0.98	26.8	1.3	56.8	2.07	22.71		
0.9	43.25			0.50	7.90	0.95	27.8	1.32	58.16	2.15	24.82		
0.91	44.18			0.50	8.41	0.98	28.76	1.3	59.42	2.34	27.01		
0.95	45.1			0.55	8.91	1.0	29.72			2.45	29.43		
0.88	46.02			0.58	9.5	1.0	30.72			2.6	31.92		
0.9	46.92			0.58	10.06	1.0	31.72			2.7			
0.9	47.82			0.58	10.64	1.01	32.71			2.8	37.32		
		86 l/m³						86 l/m³				53 l/m³	

Table 2.2 continued

Experiment 18		18 continued		Experiment 19		19 continued		Experiment 20		20 continued		20 continued	
t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t
0.9	1.00	0.48	7.6	0.2	1.03	0.83	15.24	0.16	2.09	0.42	10.03	0.68	24.97
0.1	1.08	0.48	8.1	0.2	1.22	0.87	16.15	0.19	2.25	0.44	10.49	0.7	
0.1	1.18	0.5	8.53	0.22	1.95	0.9	17.0	0.19	2.4	0.45	10.91	0.7	26.33
0.12	1.31	0.51	9.1	0.25	2.15	0.9	17.91	0.2	2.60	0.47	11.38	0.7	27.04
0.12	1.44	0.51	9.62	0.28	2.45	0.95	18.84	0.2	2.79	0.48	11.86	0.7	27.76
0.13	1.55	0.58	10.2	0.3	2.82	0.99	19.8	0.2	3.0	0.48	12.30	0.71	28.47
0.14	1.7	0.62	10.7	0.31	3.02	1.03	20.8	0.2	3.18	0.5	12.78	0.71	29.17
0.15	1.82	0.64	11.34	0.32	3.37	1.04	21.83	0.2	3.4	0.49	13.29	0.72	29.9
0.15	2.0	0.65	12.02	0.35	3.7	1.07	22.88	0.25	3.62	0.5	13.79	0.78	30.66
0.16	2.13	0.65		0.4	4.07	1.08	23.93	0.24	3.85	0.5	14.28		
0.17	2.33	0.65	13.3	0.42	4.58	1.1	25.0	0.28	4.08	0.51	14.78		
0.19	2.52	0.7	13.86	0.45	4.9	1.12	26.1	0.28	4.35	0.52	15.33		
0.2	2.72	0.71	14.7	0.48	5.38	1.15	27.23	0.3	4.65	0.53	15.89		
0.21	2.91	0.73	15.4	0.5	5.83	1.16	28.39	0.3	4.92	0.55	16.41		
0.24	3.13			0.53	6.34	1.18	29.58	0.3	5.22	0.56	16.97		
0.25	3.36			0.55	6.89	1.23	30.77	0.32	5.53	0.55	17.54		
0.25	3.62			0.59	7.47	1.31	32.02	0.33	5.85	0.58	18.10		
0.27	3.91			0.59	8.05			0.34	6.18	0.59	18.67		
0.3	4.19			0.63	8.67			0.34	6.52	0.6	19.29		
0.31	4.49			0.66	9.3			0.37	6.88	0.6	19.89		
0.33	4.78			0.68	9.95			0.37	7.35	0.62	20.5		
0.34	5.12			0.71	10.65			0.37	7.62	0.60	21.13		
0.38	5.58			0.72	11.37			0.39	7.99	0.61	21.71		
0.39	5.9			0.74	12.1			0.4	8.38	0.62	22.35		
0.41	6.27			0.78	12.86			0.4	8.79	0.63	22.97		
0.43	6.7			0.8	13.62			0.41	9.18	0.65	23.64		
0.45	7.15			0.8	14.42			0.41	9.62	0.68	24.31		
		46 l/m^3				73.5 l/m^3						66 l/m^3	

Table 2.2 continued

Experiment 21		21 continued		Experiment 22		22 continued		Experiment 23		23 continued			
t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t	t_i	t
0.13	1.28	0.9	15.03	0.17	1.94	0.53	11.01	0.15	1.39	0.87	14.95		
0.18	1.43	0.94	15.97	0.17	2.10	0.56	11.54	0.17	1.52				
0.21	1.60			0.18	2.36	0.58	12.12	0.2	1.7				
0.23	1.83			0.20	2.42	0.59	12.69	0.21	1.9				
0.25	2.06			0.21	2.61	0.59	13.26	0.21	2.15				
0.29	2.33			0.21	2.80	0.61	13.88	0.29	2.41				
0.3	2.62			0.21	3.04	0.63	14.49	0.29	2.72				
0.32	2.94			0.23	3.23	0.65	15.12	0.32	3.04				
0.37	3.28			0.25	3.50	0.64	15.77	0.33	3.44				
0.38	3.64			0.27	3.75			0.40	3.72				
0.4	4.04			0.29	4.02			0.41	4.12				
0.42	4.45			0.29	4.29			0.42	4.52				
0.48	4.9			0.3	4.60			0.48	4.96				
0.5	5.38			0.35	4.92			0.5	5.45				
0.5	5.77			0.35	5.27			0.51	5.93				
0.56	6.39			0.37	5.62			0.53	6.48				
0.58	6.94			0.39	5.99			0.58	7.0				
0.6	7.51			0.4	6.37			0.6	7.62				
0.63	8.15			0.4	6.78			0.6	8.2				
0.64	8.78			0.42	7.20			0.69	8.85				
0.69	9.46			0.44	7.62			0.7	9.54				
0.71	10.14			0.46	8.07			0.7	10.2				
0.73	10.88			0.47	8.52			0.75	10.94				
0.78	11.62			0.48	9.01			0.74	11.69				
0.81	12.41			0.5	9.46			0.78	12.46				
0.86	13.26			0.51	9.97			0.83	13.23				
0.89	14.13			0.52	10.50			0.87	14.08				
		66.5 l/m^3				68 l/m^3				47 l/m^3			

Table 2.2 continued

Experiment 24		24 continued		Experiment 25		25 continued		
t_i	t	t_i	t	t_i	t	t_i	t	
0.14	1.5	0.58	11.42	0.1	0.42	3.1	40.64	
0.18	1.65	0.58	12.0	0.9	0.55	3.35	43.87	
0.18	1.8	0.6	12.56	0.24	0.77	3.56	47.33	
0.20	2.0	0.6	13.2	0.29	1.03	4.0	51.1	
0.21	2.20	0.62	13.78	0.36	1.28	4.66	55.4	
0.25	2.45	0.62	14.40	0.43	1.75	4.8	60.17	
0.24	2.7	0.67	15.09	0.54	2.24	4.65	64.9	
0.27	2.96	0.68	15.78	0.6	2.81	5.1	69.77	
0.3	3.25			0.75	3.49	4.6	74.62	
0.29	3.55			0.85	4.29	4.5	79.16	
0.31	3.85			0.95	5.09	4.76	83.79	
0.35	4.18			1.1	6.11	4.75	88.55	
0.34	4.52			1.24	7.28	5.0	93.4	
0.4	4.88			1.36	8.58	5.0	98.4	
0.35	5.25			1.55	10.03	5.2	103.5	
0.39	5.65			1.65	11.63	5.4	108.8	
0.4	6.06			1.78	13.5	5.48	114.28	
0.4	6.45			1.85	15.16	5.46	119.75	
0.43	6.88			2.0	17.09	5.76	125.36	
0.48	7.31			2.15	19.16	5.8	131.14	
0.47	7.77			2.26	21.37	5.9	136.99	
0.49	8.25			2.4	23.70	6.0	142.94	
0.48	8.76			2.8	26.30	6.1	148.99	
0.5	9.25			2.8	29.10	6.46	155.27	
0.5	9.76			2.8	31.90			
0.56	10.29			2.8	34.70			
0.58	10.87			3.0	37.6			
			52 l/m³				Not available	

2.2.1.4 'Constant volume' flow monitoring - discussion

From inspection of the data in Table 2.2 the general pattern of results is clear. Flow of liquid into the timber proceeded most rapidly at first and thereafter slowed down considerably.

In Figures 2.3 through 2.7, approximately linear graphs are obtained with most sets of data when t_i is plotted against $\sqrt{t}$. As the following discussion seeks to show, this appears not to be totally consistent with established theory.

In Table 2.2 each interval (t_i) between successive solenoid valve actuations may be interpreted as an indirect indication of the overall rate (Q) at which liquid is flowing into the timber. Indeed, the length of the interval between successive solenoid valve actuations is inversely related to Q. Thus the values of t_i in Table 2.2 may be regarded as relative values of $\frac{1}{Q}$ and are plotted as such on Figures 2.3 through 2.7.

Figure 2.3 Data from Table 2.2 plotted to display relationship between elapsed time and rate of flow of liquid into spruce. See also Figures 2.4 through 2.7.

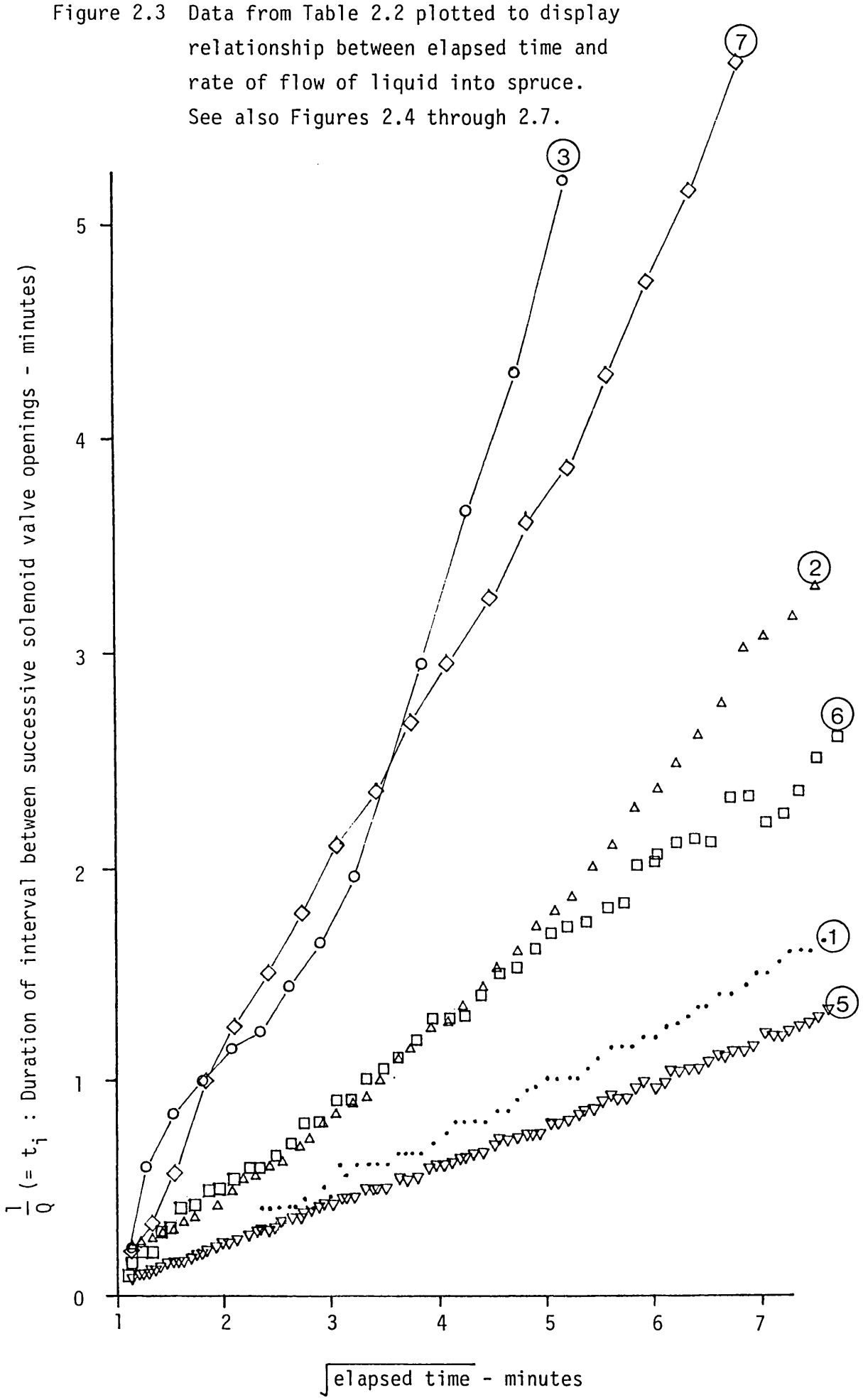


Figure 2.4 Data from Table 2.2 plotted to display relationship between elapsed time and rate of flow of liquid into spruce. See also Figures 2.3 and 2.5 through 2.7.

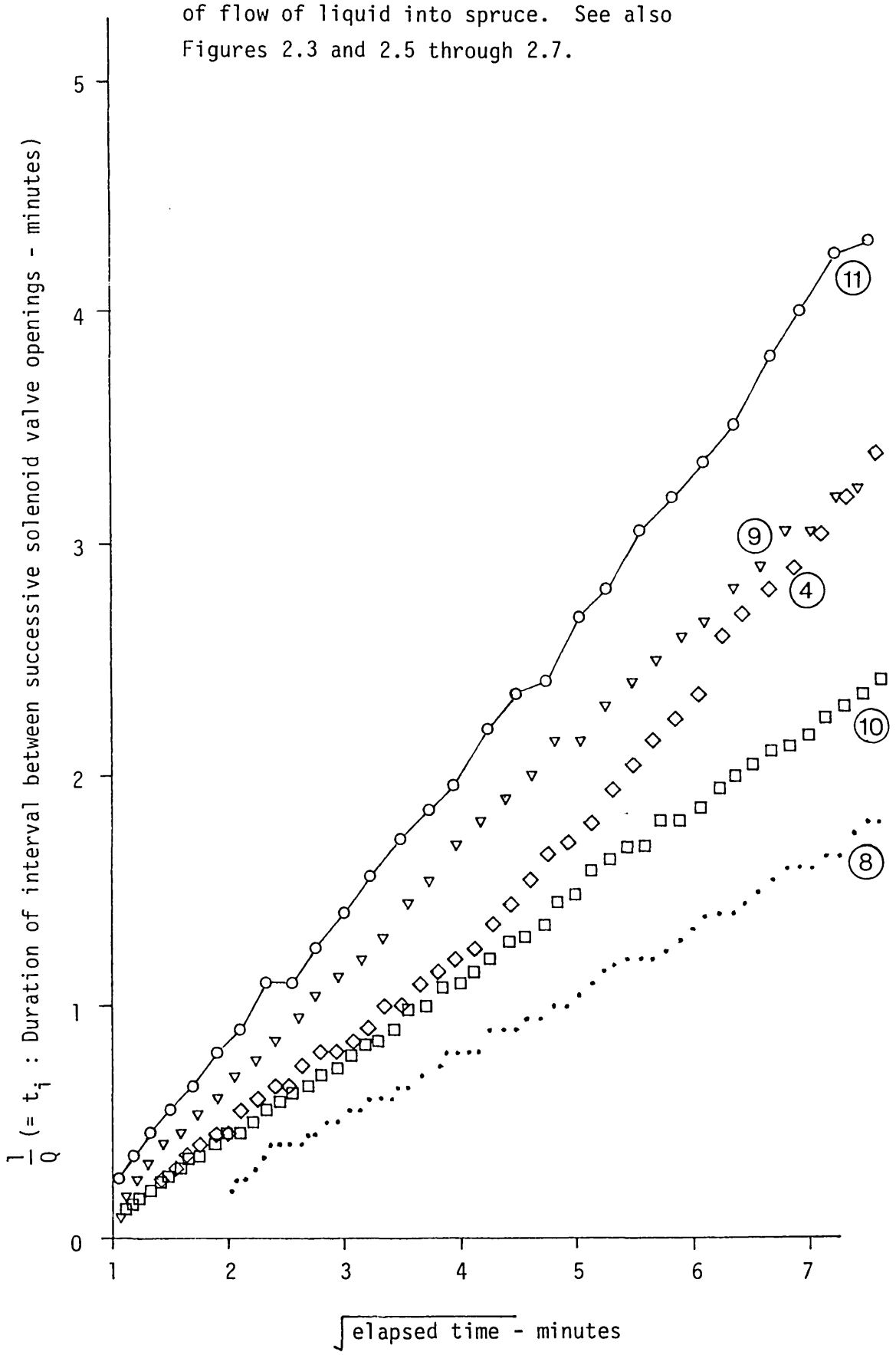


Figure 2.5 Data from Table 2.2 plotted to display relationship between elapsed time and rate of flow of liquid into spruce. See also Figures 2.3, 2.4, 2.6 and 2.7.

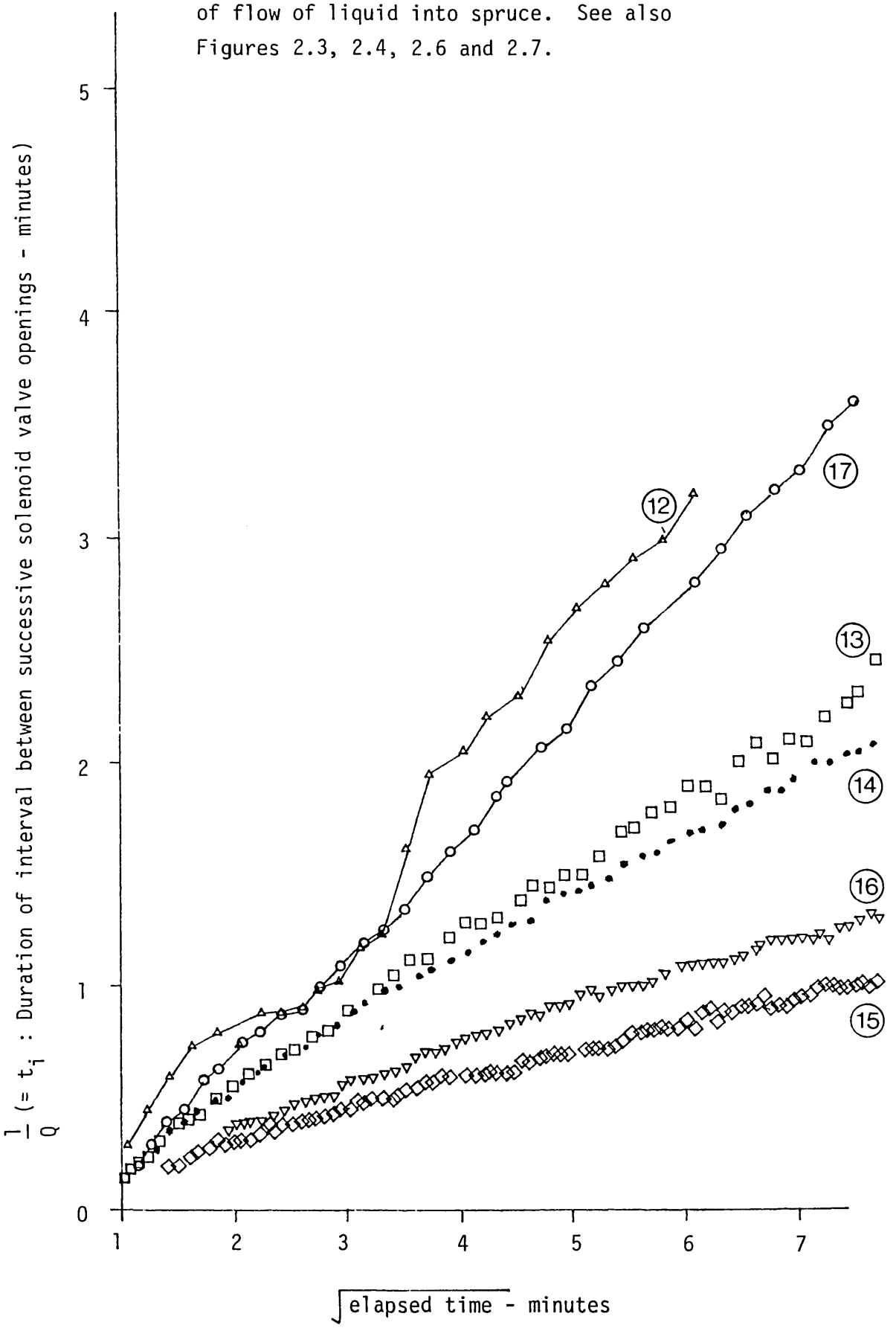


Figure 2.6 Data from Table 2.2 plotted to display relationship between elapsed time and rate of flow of liquid into spruce. See also Figures 2.4 through 2.5 and 2.7.

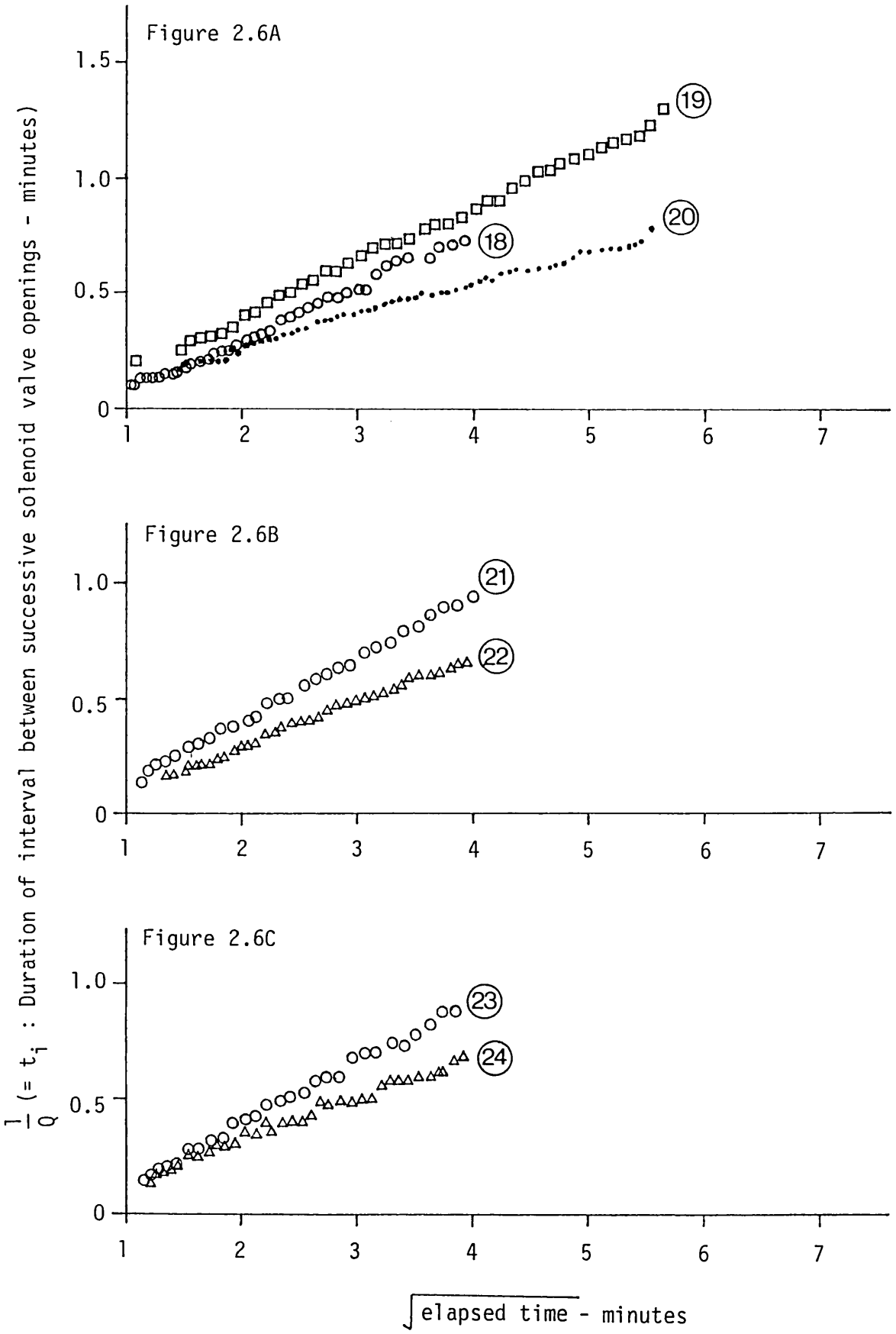
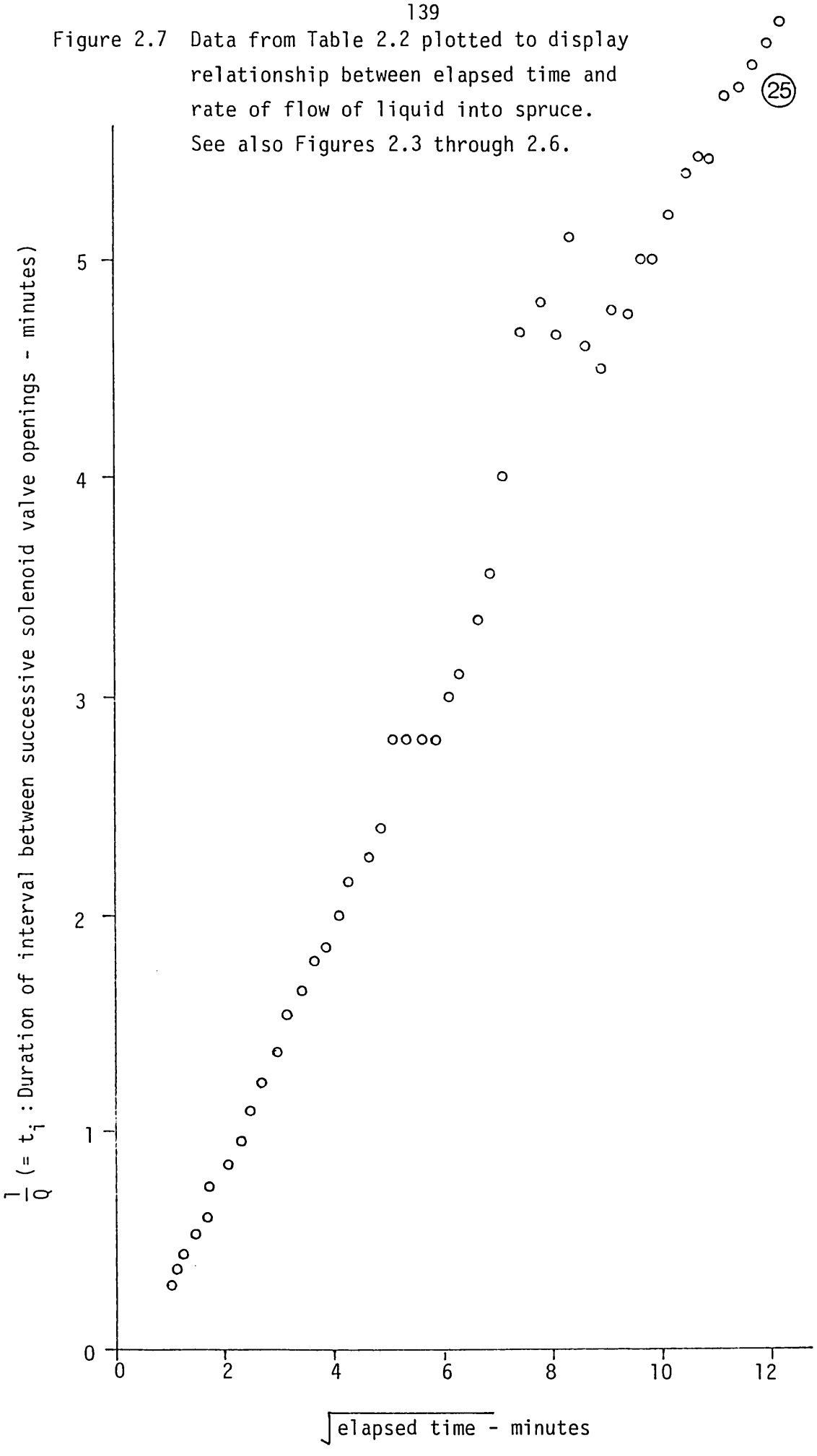


Figure 2.7 Data from Table 2.2 plotted to display relationship between elapsed time and rate of flow of liquid into spruce. See also Figures 2.3 through 2.6.

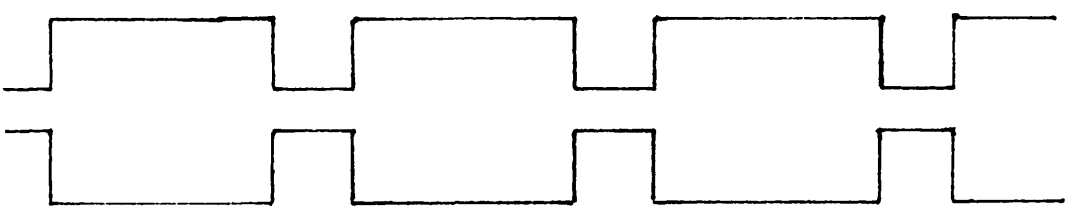


In Figures 2.3 through 2.7, minor deviations from the main trend of each curve and the stepped appearance of plots 1 and 8 for example are, on the whole, a reflection of the fact that it proved impossible to make very precise measurements during interpretation of the pen-recorder charts. At the time the experimentation was undertaken, it was anticipated that data analysis would consist of cumulative plots of solenoid valve openings against time, and the chart speed used (usually 10 mm/min) was selected accordingly. However, for the reasons discussed in Section 2.2.1.3.2, it was subsequently decided that a more appropriate analysis necessarily involved measuring the time interval between successive solenoid valve actuations. For this analysis a faster chart speed would have facilitated more accurate interpretation.

Where several successive points deviate similarly from the general trend of the curve of which they are a part, the deviations cannot be attributed to random mensuration error. Close examination of the original pen-recorder charts revealed that some of these deviations may have been contributed to by slight variations in the volume of liquid admitted by the solenoid valve. However, this cannot be invoked to account for all the deviations observed and it is concluded that, even in these comparatively large loads of timber, the rates of liquid-uptake displayed variation around a main trend.

The mechanism giving rise to these variations is unclear. As liquid flows into a parallel-sided pipe the volume rate of flow decreases as the length of the flow-path increases because, as the volume of liquid in the pipe increases, the viscous forces in that liquid increase concomitantly. Thus, volume rate of flow decreases with time. In uniform, parallel-sided flow-paths, this decrease will occur at a steady rate. Conversely, in flow-paths of the type indicated in Figure 2.8 (such as are encountered in softwood), the decrease in volume rate of flow into each individual flow-path will not be steady but will be dominated by stepped decreases as each successive constriction in the flow-path is encountered. However, during treatment of a charge composed of

Figure 2.8 Schematic representation of a single, simple softwood flow path



After Banks 1975

many pieces of timber, and thus of many thousand flow-paths through which flow will not be synchronised, the volume-rate of flow into the charge as a whole would be expected to exhibit gradual continuous changes and not to be affected by 'local' variations in individual flow-paths.

It was shown in the Introduction (Equation 1.20) that, according to Bramhall (1971), unsteady-state flow into a permeable porous material may fundamentally be described by an integrated form of Darcy's Law, although with less permeable materials a factor (e^{-bl}), must be included in the numerator to take account of random flow-path blockage. Petty (1975) further showed that an expression for total liquid absorption into permeable timbers may take the form:

$$V = A\left(\frac{2\,K\,f\,\,P\,t}{\eta}\right)^{\frac{1}{2}} \qquad \text{Equation 1.22}$$

and thus if all other factors remain constant:

$$V \propto \sqrt{t} \qquad \text{Equation 1.24}$$

(During unsteady-state flow of liquid into timber 'all other factors' do not 'remain constant' because the pressure gradient (ΔP) between the inside of the timber and the treating-liquid constantly changes as flow proceeds. However, in the absence of precise data about the changing pressure within the timber, the relationship in Equation 1.24 was assumed to apply, at least to a

first approximation.)

It follows from this relationship that:

$$\frac{1}{Q} \propto \sqrt{t} \qquad \text{Equation 2.1}$$

where Q = Volume rate of flow (V/t)

and therefore a plot of $\frac{1}{Q}$ against $\sqrt{t}$ would be linear. Accordingly, the data in Table 2.2 were examined for this relationship by plotting values for t_i against $\sqrt{t}$ in Figures 2.3 through 2.7.

Of the twenty-five sets of data represented in Figures 2.3 through 2.7, thirteen (Nos. 1, 5, 8, 10, 11, 17, 18, 19, 20, 21, 22, 23 and 24) appear to indicate direct linear relationships between $t_i (= \frac{1}{Q})$ and $\sqrt{t}$.

In addition, a further six (Nos. 3, 6, 7, 12, 13 and 25) fluctuate somewhat about what appear to be basically linear trends.

Thus a total of nineteen out of the twenty-five sets of data indicate inverse linear proportionality between Q and $\sqrt{t}$.

Air embolisms

The relationship $\frac{1}{Q} = \sqrt{t}$ (Equation 2.1) is expected when bubble-free liquid flows into a uniformly permeable porous material. However, the industrial-type double vacuum treatments monitored in this work involved 'severe' initial vacuums and thus involved conditions which would favour evolution of bubbles of dissolved gas from the treating solution. These bubbles could block the smaller flow-paths (Section 1.3.4) and would thus restrict flow. Additionally, spruce is clearly not a uniformly permeable material and so the component 'b' of the random path blockage factor 'e^{-bl}' (Equation 1.19) might have been expected to have a significant value and this would have led to a substantial upward deviation from linearity in each plot of t_i against $\sqrt{t}$. Despite this, substantial upward deviations were not observed in the nineteen sets of data identified above.

In two of the sets of data (Nos. 2 and 4) upward deviations, albeit small ones, were observed as expected. However, in the plot of the data from Experiment 14, and to a lesser extent in the plots of the data from Experiments 9, 15 and 16 also, a downward deviation from linearity is evident. This indicates that in these four experiments the rate of flow did not decrease in proportion to $\sqrt{t}$.

Although the downward deviations were slight, the possibility that they were artefacts indicative of a flaw in the experimental

system was particularly carefully examined in case the same flaw might have been distorting the results of the other experiments also.

The most obvious possible cause which could have given rise to an anomalously slow decrease in flow-rate was a leak from the treating vessel. However, no such leak could be identified, apart from which, a steady leak would have resulted in a progressively more marked deviation from linearity, which was not observed.

Alternatively, the observed downward deviations could, arguably, have resulted if one or several air bubbles became trapped in the treating vessel during filling. Any such bubbles would have compressed as pressure increased and would thus have led to an increased quantity of preservative having to enter the vessel in order to produce a given increase in pressure. For instance, in arbitrary numerical terms, if a vessel totally filled with liquid required two volumes of liquid to raise its pressure from solenoid-valve-opening-pressure to solenoid-valve-closing-pressure, that same vessel occupied partly by liquid (which is virtually incompressible) and partly by air (which is compressible) might require the introduction of six volumes of liquid to produce the same increase in pressure. If, as treatment proceeded, the bubbles dissolved into the liquid and thus decreased in size, the volume of liquid admitted by each solenoid valve actuation would decrease concomitantly. Thus, the basic assumption that the

solenoid valve would repeatedly admit a fixed quantity of liquid throughout treatment would be invalid and deviations from the 'expected' results would indicate no more than that the solenoid valve was admitting progressively less liquid in successive actuations.

Conceivably, the 'dissolving gas bubble' mechanism postulated above could have masked diminishing flow rates in these experiments. However, in reality it seems a very unlikely coincidence that in every experiment, despite differences in load size, end-grain sealing, immersion pressure and final preservative uptakes, the net results of the interacting effects should be so close to $\frac{1}{Q} = \sqrt{t}$. It seems wise, therefore, to be cautious of invoking speculative shortcomings in the experimental system to account for these data. Rather it seems more appropriate to conclude that the relationship $\frac{1}{Q} = \sqrt{t}$ appears to be a sufficiently accurate approximation for calculations and use in industrial spruce-impregnation practice. This relationship has previously been reported from somewhat similar work on two species of spruce by Petty (1975).

The above empirical conclusion raises many questions. For example, three factors, namely air embolism formation, random flow-path blockage and diminution of ΔP, might all be expected to cause decrease of the flow rate such that the relationship $\frac{1}{Q} \propto \sqrt{t}$ does not apply. The fact that they appear not to do so must call into question the extent to which classical 'pipe-flow' theory

can be used to describe the flow of light organic-solvent into spruce.

2.2.2 Monitoring liquid flow into timber on a small scale

In Section 2.2.1 a technique for monitoring the rate of liquid flow into a relatively large volume of timber has been described. From a practical point of view this technique has two serious drawbacks.

Firstly, the technique as described does not permit measurement of the actual volume of liquid flowing into the timber. Consequently the technique is not suitable for comparing absolute rates of liquid flow into timber during different experiments.

Secondly, the technique necessarily involves the use not only of substantial volumes of timber but also of a substantial volume of liquid. For example, over 150 litres of liquid are required to fill the timber treatment vessel pictured in Plate 2.1. Emptying, cleaning and refilling this apparatus is a major undertaking and, in consequence, apparatus of this size is completely impractical if it is desired to examine a range of test liquids to establish their suitability as preservative carrier solvents in terms of their penetration characteristics.

With this latter drawback particularly in mind an apparatus was devised with which to monitor quantitatively liquid flow into a timber sample through a restricted area of its surface.

2.2.2.1 Development and use of a flow meter

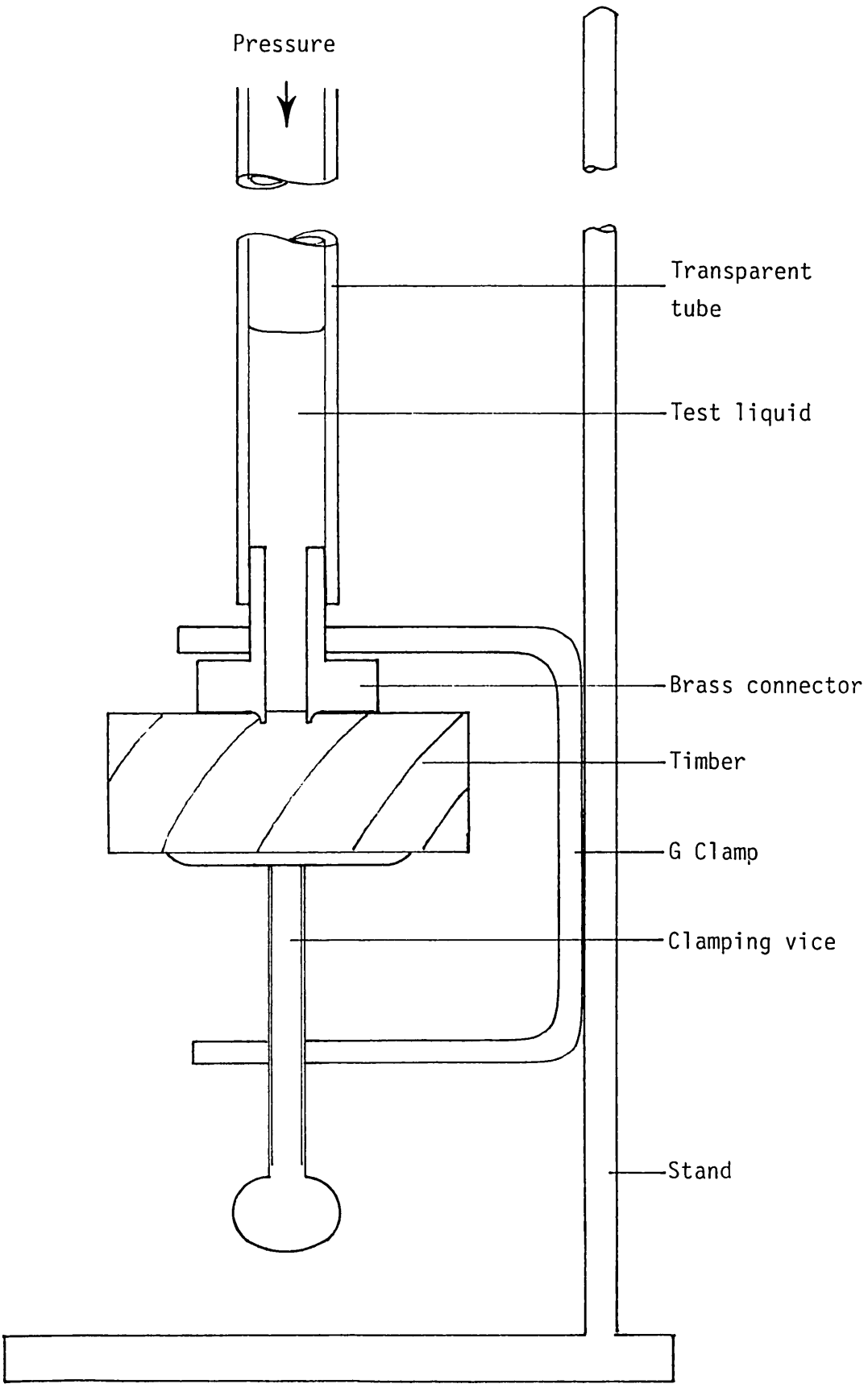
2.2.2.1.1 Concept

At the outset, a device functionally analagous to the intrusive probe used by Cooper et al (1974) was envisaged as a means through which liquid could be introduced into timber and with which the rate of liquid ingress could be monitored. During brief preliminary experimentation with the apparatus represented in Figure 2.9 it quickly became apparent that, because the volume rates of flow of liquids into timbers are orders of magnitude smaller than those of gases, a larger area through which to introduce liquid was required in order to observe and measure liquid movement.

Accordingly, a flow meter was constructed with an area through which liquid could be applied of 8.55 cm^2, i.e. a circle of 3.3 cm diameter. This dimension was selected as being an adequate size to allow liquid to enter the timber at a rate which could be conveniently measured, while not being so large as to require excessively large samples as subjects for experimentation.

One of the original (although in the event, unfulfilled) ways in which this piece of apparatus was to be used was for the purpose of predicting preservative-liquid uptake prior to industrial treatment. In this context it was important that use of the apparatus should not significantly damage the timber. This constraint clearly rendered the idea of an intrusive probe incom-

Figure 2.9 Schematic diagram of prototype flow meter



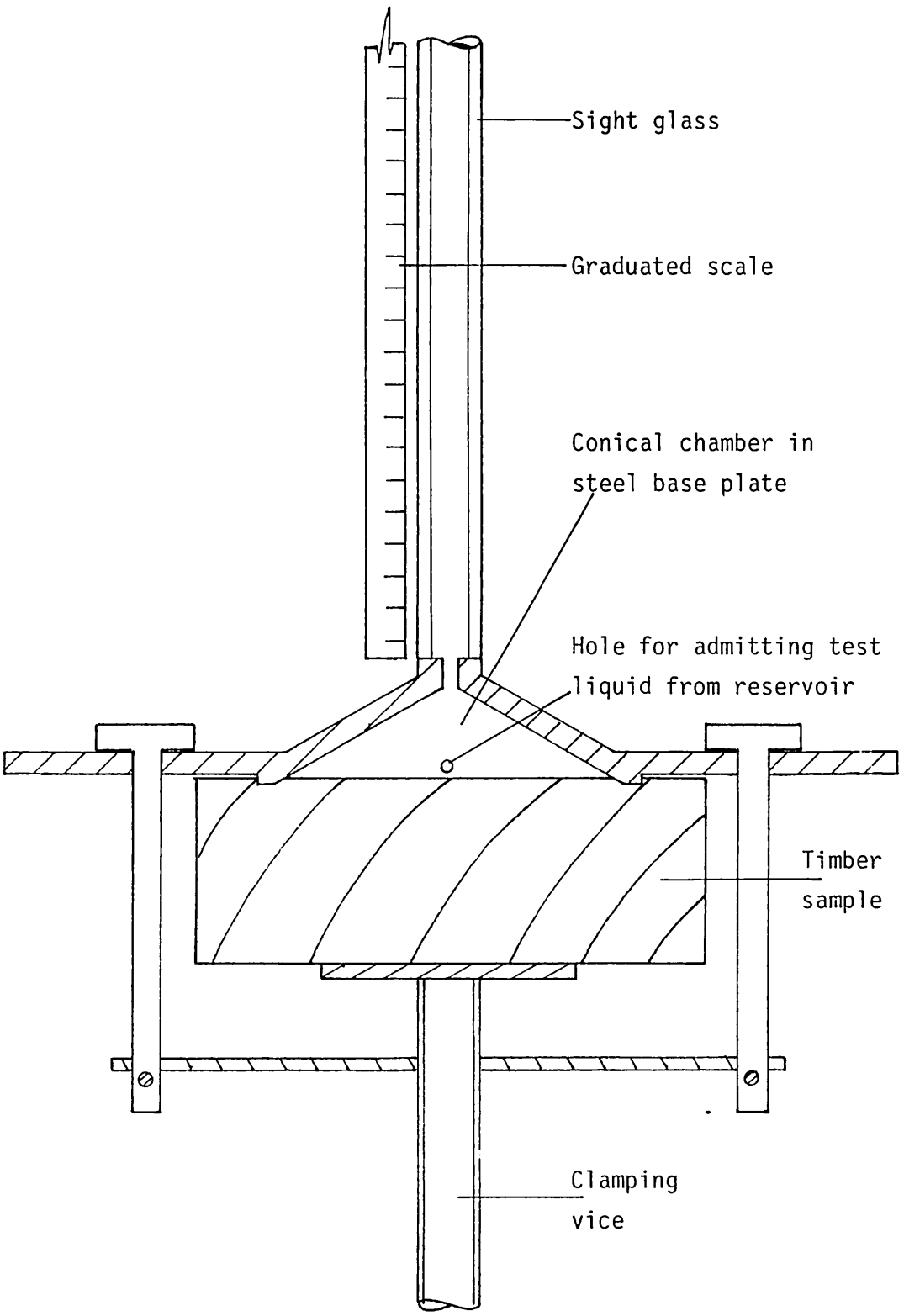
patible with applying liquid to an area 3.3 cm in diameter. For this reason a 'probe' which could be clamped to the surface of a sample was developed. This exhibited certain functional similarities to the one later described by Venturino and Arganbright (1979).

2.2.2.1.2 Basic design of the apparatus

In order to monitor the rate of liquid flow in through a known area of the timber surface, an arrangement similar to a plant potometer (see for example Bower 1947) was designed. In practical terms, however, there seemed only negligible advantage in monitoring the passage of a bubble in a horizontal tube as opposed to following the movement of an air/liquid interface down a vertical tube. An interface is easier to manipulate and so the 3.3 cm diameter opening in the flow-meter base plate was connected to a relatively tall vertical 'capillary' sight glass (Figure 2.10). This was equipped with a graduated scale so that the progress of the meniscus down the tube could be observed and measured. The apparatus was constructed with a range of interchangeable sight glasses, having internal diameters of between 1 and 5 mm, to accommodate examination of a fairly wide range of rates of flow of liquids into timbers.

Although liquid was applied to a circle of 3.3 cm diameter on the timber surface, the chamber in the flow-meter base plate was conical, reducing rapidly from 3.3 cm to 0.2 cm diameter at the

Figure 2.10 Schematic diagram of flow meter



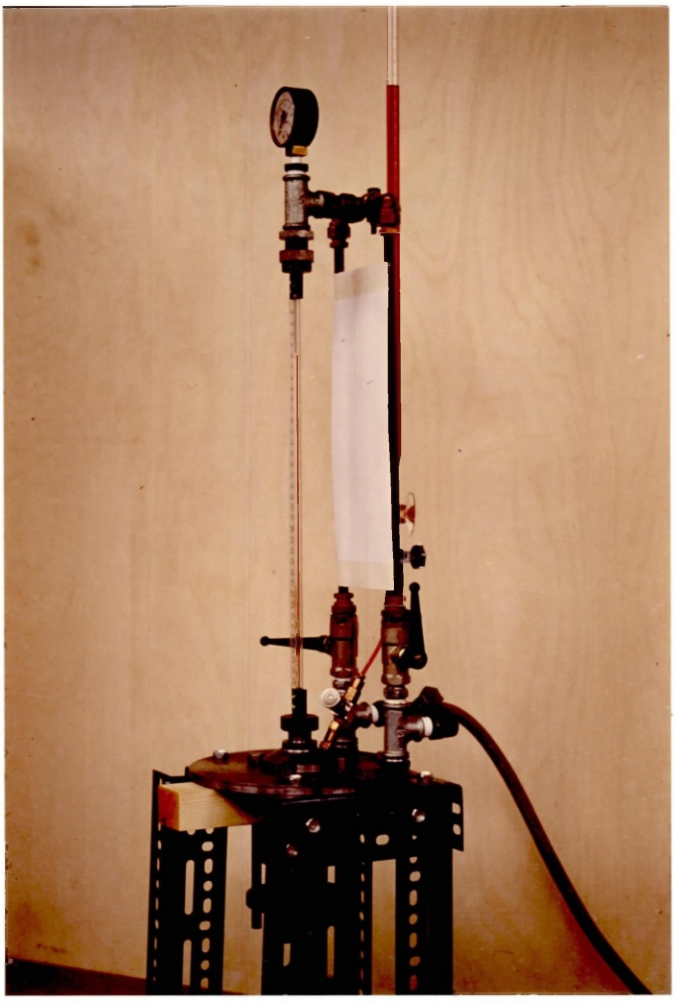


Plate 2.2 Flow meter (safety screen removed) used for experimentation described in Section 2.2.2. See also Figure 2.10.

point of union with the sight glass. At the beginning of each experiment, liquid was admitted to the conical chamber and sight glass through a 0.1 cm diameter hole which entered the chamber just inside its periphery. Thus, inflowing liquid filled the chamber from its bottom, sweeping air before it and minimising the risk of pockets of air being trapped and subsequently confusing the results.

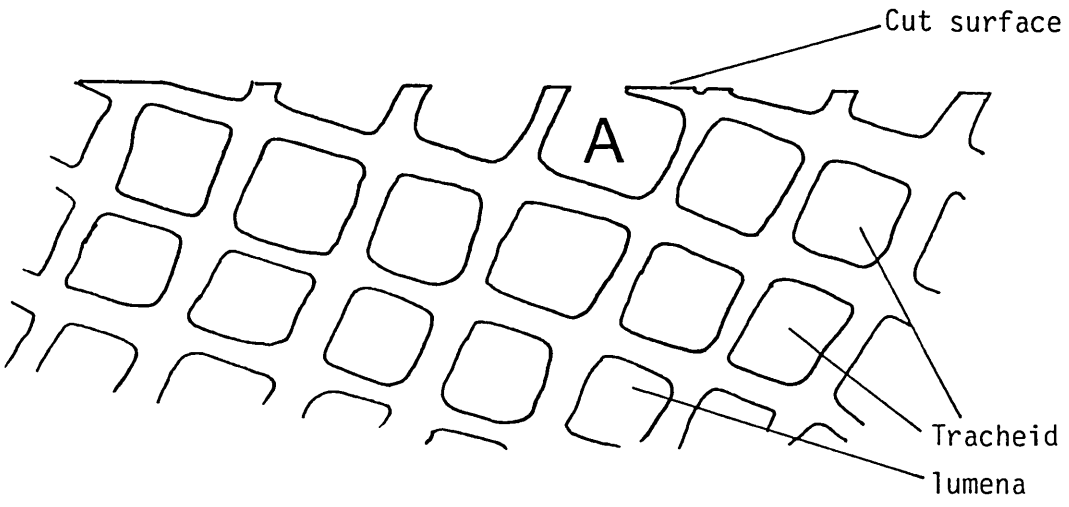
2.2.2.1.3 Clamping the flow meter to timber

In order to achieve a good seal between the timber sample being examined and the base plate, three alternative sealing systems were evaluated.

Soft 'O' ring seals

Rubber 'O' rings were an obvious choice of material to be used in order to make a cheap, easy to make-and-break seal between two hard surfaces. They seemed especially suited to the present application because it was anticipated that they would deform to accommodate even relatively large irregularities in the timber. They proved to be unsatisfactory, however, because to form seals on lateral surfaces of planed timber an exceptionally deformable material would be required to push into and seal the lumen of, for example, a tracheid cut as indicated at 'A' in Figure 2.11.

Figure 2.11 Diagramatic transverse section of a softwood to show possible shapes of micro-irregularities at a cut lateral surface of a timber sample



Even if such a deformable material could be identified it would almost certainly have a very short useful life and its mechanical resistance to applied pressures would be small.

In the absence of a suitably deformable material, it had to be accepted that a flexible seal would have to cover at least one tracheid-length, a distance typically of 0.35 cm. If it did not, liquid would be able to enter one end of a ruptured tracheid, flow under the 'seal' and out of the other end. Indeed a sealing ring

even 6 mm wide could allow liquid into the end of one tracheid, under the seal, passing through one pit pair only and out to the surface again. Rates of flow observed under such circumstances would reveal little about flow of liquid through the test sample, as Venturino and Arganbright (1979) subsequently found. A purely superficial seal must clearly therefore cover a wide area around the liquid application region if such problems are to be avoided.

Gasket seals

The failure of soft 'O' ring seals led to gasket seals being evaluated. Such seals restricted experimentation to planed timber because reusable gaskets will only accommodate small irregularities in the surfaces being mated.

Because of its stability in a wide range of solvents, a compressed asbestos gasket (Walkers 0.1 cm Unilion) was initially selected and for a period it yielded apparently satisfactory results. When abnormally impermeable softwood samples were used, however, the meniscus was observed to move very slowly but quite perceptibly up the sight glass even when non-swelling solvents were used.

After investigation it was concluded that this resulted from the force with which the timber had to be clamped against the base plate to effect a seal. This force was presumed to be such that it compressed the timber sample slightly. As experimentation progressed, the unrestrained region of the timber under the conical

chamber expanded upwards as it returned to its uncompressed state. (A uniform movement of less than 0.02 mm would account for the observations.) It was considered that this introduced unacceptable uncertainty as to the validity of the flow rates observed and so the use of hard gaskets was rejected.

A soft gasket material (Walkers 0.2 cm Synquesto) was tried and certainly an airtight seal could be achieved using a much lower clamping force. However, this material swelled badly in organic solvents and the inconvenience of repeatedly replacing the gasket was considered unacceptable.

Some satisfactory results were obtained by sealing the surface of the timber around the test-area with epoxy resin. Not only was this technique time consuming, however, it also failed to satisfy one of the main reasons for evaluating gaskets; namely that the test system should not significantly mark the timber.

To summarise, from experience with both soft 'O' rings and gaskets it was concluded that the seal had to be wider than the length of several tracheids (say > 2 cm). Soft gasket materials were insufficiently stable in white spirit type solvents. Hard gaskets failed to form a gas-tight seal with the timber except when so great a clamping force was required that the timber appeared to deform. An impasse was thus reached in which it was not readily possible to contrive a seal which allowed a surface-applied flow-meter to be used. To circumvent this impasse it was decided to

tolerate a minimal degree of intrusiveness, and thus to tolerate a degree of superficial damage to the timber samples during testing.

Hard 'O' rings

Having accepted the need for a degree of intrusiveness, a convenient means of accomplishing indentation of the surface was adjudged to be a very hard 'O' ring around the test area. To construct such a ring, and to avoid any problems with holding it in place, the underside of the base-plate of the flow-meter was skimmed on a lathe, reducing its thickness by 0.1 cm all-over, except for a 0.1 cm wide ring around the periphery of the conical chamber.

When a lateral surface of a sample of timber was clamped against this, the protruding ring of metal around the test area indented the timber sample, crushing the outermost tracheids as it did so. (The clamping force (2.4 Nm) used for spruce was selected as being near the minimum necessary reliably to form a gas-tight seal.) The resultant indentation of the sample would have resulted in 15 - 30 tracheids being crushed as the sample was clamped against the base-plate. Thus the shortest route by which liquid could by-pass the seal and re-emerge at the surface of the sample involved flow through twice this number of pit pairs. It was felt that this was sufficient for the volumes measured to have a useful relationship to the actual rate at which a liquid could flow into the whole timber sample.

2.2.2.1.4 Use of the flow-meter

Although the flow-meter could accommodate a wide range of sample sizes, almost all the experimentation reported here was performed using similarly-sized samples which were prepared from lengths of Picea abies measuring 4.3 x 1.9 cm in cross-section. These were numbered along one wide lateral surface and cross cut into 5.5 cm long pieces. Each of these was sealed across both end grains using epoxy resin (see Section 2.2.1.2.3).

One sample at a time was positioned with the side opposite its numbered face under the conical opening in the base-plate and the clamping vice was tightened to approximately 2.4 Nm on a $\frac{3}{8}$" (9.55 mm) BSF thread. This pressed the hard ring of metal around the opening in the base-plate into the timber.

When the sample had been clamped into place, the needle valve between the liquid reservoir and the conical chamber was opened to allow liquid to move by gravity from the reservoir to the conical chamber and from there to move up the sight glass. At the moment the liquid level in the reservoir started to drop, a stopwatch was started. When the liquid reached a convenient height near the top of the sight glass, the needle valve was closed and the appropriate test pressure was applied. Filling and pressurising the sight glass typically took less than 30 seconds, after which time an initial reading was taken.

The passage of the meniscus down the sight glass, and hence by inference the volume of test liquid entering the timber, was monitored at intervals and recorded on sheets such as the one in Figure 2.12. From this figure it may be noted that the position of the meniscus was recorded at one-minute intervals during the first 10 minutes, and at five-minute intervals thereafter. Usually, observation was discontinued after 20 minutes although in some of the experimentation reported a 30 minute period was used.

Figure 2.12 FLOW-METER - RESULTS SHEET NO.

Sample No.						
Species						
Sample size						
Sealing						
Treating solution						
Application pressure						
Application area						
I/D sight glass						
Scale						
Date						
Initials						
		R E A D I N G				
Time (min) 0.5						
1.0						
2.0						
3.0						
4.0						
5.0						
6.0						
7.0						
8.0						
9.0						
10.0						
15.0						
20.0						
1-20 min total						
1-20 min av. ml x $10^{-4}/cm^2/min$						

Notes:-

2.2.2.2 Flow-meter - experimentation undertaken

The experimentation undertaken using the flow-meter may conveniently be described in seven sections.

(i) Pattern of lateral flow and intersample variability (Section 2.2.2.2.1)
(ii) Effect of pressure gradient on flow (Section 2.2.2.2.2)
(iii) Correlation of flow-meter readings with uptakes-during-industrial impregnation (Section 2.2.2.2.3)
(iv) Flow of emulsions (Section 2.2.2.2.4)
(v) Flow of a range of liquids (Section 2.2.2.2.5)
(vi) Repeated treatment of samples (Section 2.2.2.2.6)
(vii) Longitudinal flow (Section 2.2.2.2.7)

2.2.2.2.1 Pattern of lateral flow and intersample variability

In order to examine both the characteristic pattern of lateral flow into spruce and also to estimate the variability in rates of flow into adjacent samples, a single length of 4.3 x 1.9 cm straight-grained spruce was used to prepare 46, 5.5 cm long, consecutively numbered samples of the type described in Section 2.2.2.1.4. For each sample the rate of entry of white spirit applied to the un-numbered broad face at 69 kPa above atmospheric pressure was observed for 20 minutes. Its progress was recorded at each of the time intervals indicated in Figure 2.12. The results obtained from this experimentation are presented and

discussed in Section 2.2.2.3.1.

2.2.2.2.2 Effect of pressure gradient on flow

Notwithstanding the theoretical prediction that total flow in unit-time varies in proportion to $\sqrt{\text{pressure gradient}}$ (Equation 1.23), there are indications in the literature (e.g. Petty 1978[b]) that for reasons such as the displacement of air-embolisms at constrictions, changes in flow rate may be disproportionately greater than changes in $\sqrt{\Delta P}$.

Further clarification of the relationship between pressure gradient and flow rate was clearly of considerable importance to this investigation. Thus, despite the intersample variability associated with the flow tester experimental system (Section 2.2.2.3.1), and the consequent poor sensitivity to small changes in flow rate, it was considered appropriate to attempt to use this apparatus to investigate the relationship between flow rate and pressure gradient.

The experimentation undertaken was designed such that, concurrently, observations were also made to establish whether or not a pressure gradient of 35 kPa (for instance), produced by applying pressure to liquid, resulted in a similar flow rate to that induced by a pressure gradient of 35 kPa, produced by partial evacuation of the sample prior to the introduction of liquid.

Experimentation in this section was undertaken in three sections, as follows:

(i) Flow at a range of pressure gradients (Section 2.2.2.2.2.1)
(ii) Flow at pressure gradients of 138 and 413 kPa (Section 2.2.2.2.2.2)
(iii) Flow at pressure gradients of 69 and 138 kPa (Section 2.2.2.2.2.3)

The results from these three sections are presented and discussed in Section 2.2.2.3.2 and its subdivisions.

2.2.2.2.2.1 Flow at a range of pressure gradients

In the first of these experiments, six beams of straight-grained spruce were each used to prepare sets of ten consecutive samples of the size and in the manner described in Section 2.2.2.1.4. (Where necessary the strict consecutive sequence was disrupted to avoid samples containing knots.) White spirit was introduced into these at the pressure gradients detailed in Table 2.3. From this table it may be seen that seven of the samples in each series had air at atmospheric pressure inside them at the start of the observation period. The remaining three in each series were partially evacuated prior to introduction of the liquid. To accomplish this, the sample, after being clamped under the base-plate in the normal way, was surrounded by a gas-tight cylinder which sealed against the underside of the base-plate (Figure 2.13).

Figure 2.13 Schematic diagram of chamber around sample clamped to the flow meter

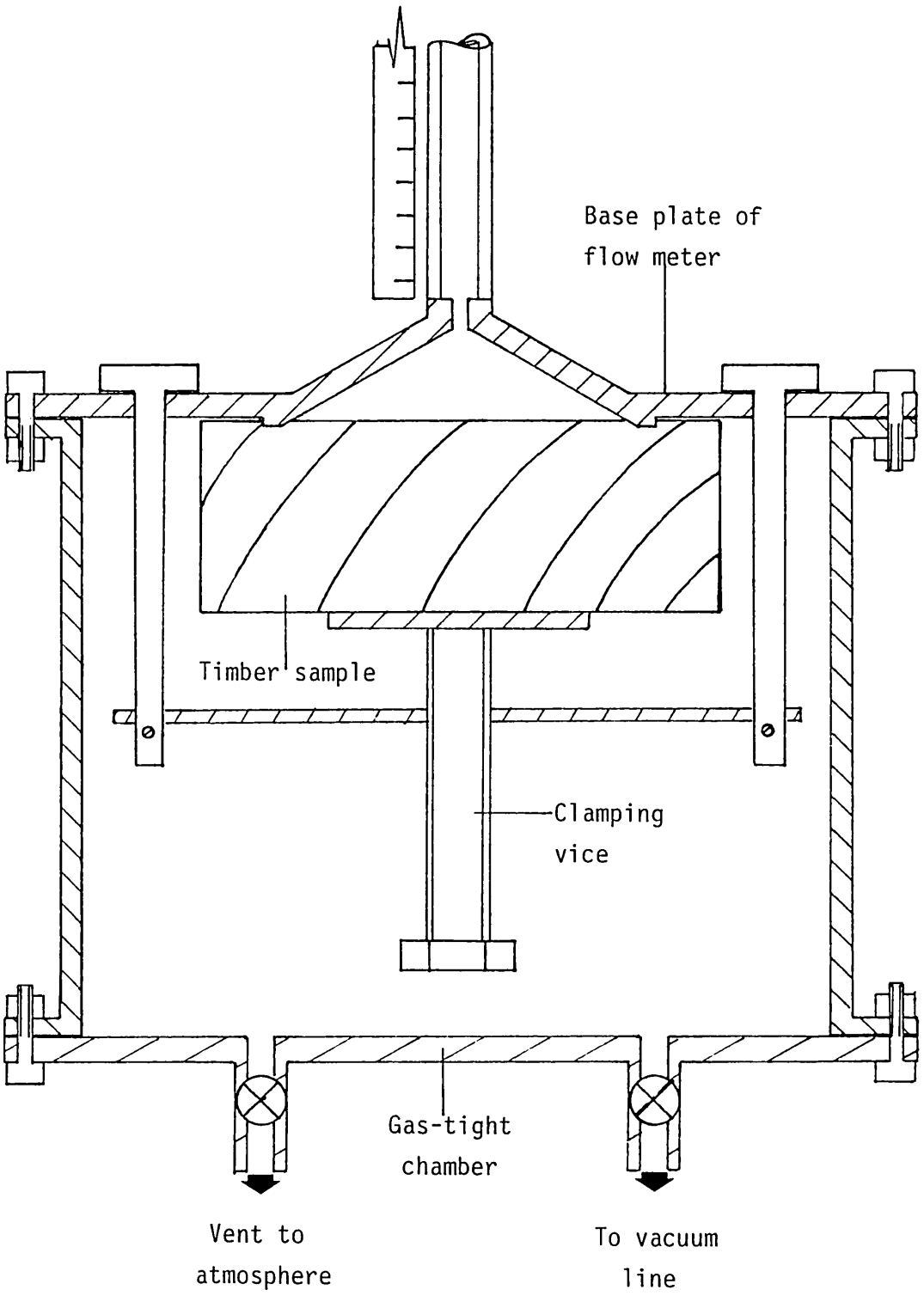


Table 2.3 Examination of the effects of pressure gradient on the lateral flow of white spirit into spruce - experimental pressures used

Example : Beam 1 } 69 = Pressure (kPa) on liquid in flow-meter
Sub-sample 9 } -69 = Pressure (kPa) in voids in timber sample

Beam sub-sample No.	Beam Number					
	1	2	3	4	5	6
1	0 / 0	35 / 0	69 / 0	69 / -69	276 / 0	0 / -35
2	35 / 0	69 / -69	35 / 0	-	0 / 0	276 / 0
3	0 / -69	207 / 0	413 / 0	0 / 0	0 / -35	35 / 0
4	138 / 0	276 / 0	207 / 0	207 / 0	207 / 0	138 / 0
5	413 / 0	413 / 0	276 / 0	138 / 0	0 / -69	0 / -69
6	69 / 0	69 / 0	-	35 / 0	69 / -69	69 / 0
7	276 / 0	0 / 0	69 / -69	276 / 0	413 / 0	413 / 0
8	207 / 0	0 / -69	0 / -69	69 / 0	138 / 0	69 / -69
9	69 / -69	0 / -35	0 / -35	413 / 0	69 / 0	0 / 0
10	0 / -35	138 / 0	138 / 0	0 / -69	35 / 0	207 / 0
11	-	-	0 / 0	0 / -35	-	-

Note : Allocation of samples to treatments was determined using tables of random numbers (Cochran & Cox 1957)

Then the sight glass and conical chamber above the sample were evacuated to the required level of vacuum for five minutes, following which the cylinder around the sample was evacuated to the same level. The vacuum was maintained both above and below the sample for a further 15 minutes. Following introduction of liquid into the conical chamber and sight glass, the vacuum in the sight glass was released, although the cylinder around the sample was maintained in its condition of partial evacuation throughout the observation period.

In two instances in each series, liquid at atmospheric pressure was introduced into the partially evacuated samples. In the remaining evacuated sample a pressure was applied to the liquid.

Where possible, the movement of the meniscus down the sight glass was monitored for 30 minutes, although flow sometimes occurred too quickly to allow observation to continue throughout this period.

The results of this first experiment were somewhat variable, and in particular were unsatisfactory with regard to the information they yielded concerning the relative effects of higher applied-pressures. To obtain more reliable results, closer sample matching and greater replication were clearly indicated. Accordingly, two further experiments were undertaken.

2.2.2.2.2.2 Flow at 138 and 413 kPa

In the first of these supplementary experiments, several sets, each of nine consecutive samples of the type already described, were prepared. The entry-rate of white spirit at atmospheric pressure into one sample from each set was measured. In three of the sets this flow rate was found to be zero or negligible and these sets were selected for further experimentation. The remaining eight samples in each set were allocated alternately to treatment at either 138 kPa (20 p.s.i.) or 413 kPa (60 p.s.i.). Flow rates were monitored for up to 30 minutes.

This experiment yielded more satisfactory information about the flow rates at these two applied pressures and so a similar pattern was adopted for a final experiment in this series.

2.2.2.2.2.3 Flow at 69 and 138 kPa

In this final experiment, preliminary checks were once again made to ensure that inflow rate at atmospheric pressure was near zero. Having selected three series of test samples in this way, each series of between nine and eleven consecutive samples of the type already described was divided by alternate selection and used to compare inflow rates at 69 kPa (10 p.s.i.) and 138 kPa (20 p.s.i.) for periods of up to 30 minutes.

2.2.2.2.3 Correlation of flow-meter readings with uptakes during industrial-impregnation

In 2.2.2.1.1 it was intimated that one of the original uses for which the flow-meter was designed was for the purpose of predicting preservative uptake during an industrial-impregnation. Three of the main reasons why this was considered desirable were:

(i) It was hoped that the cumbersome pilot scale evaluation of treatment schedules, more fully described in Section 3, could be supplanted by this technique.

(ii) Arising from (i) it was envisaged that the flow-meter might provide a suitable technique for predicting how an existing schedule should be modified to take account of changes in the characteristics of the treating liquid or in the permeability of the timber being impregnated.

(iii) Industrially, there is a need for an apparatus which can detect ponded or other 'over absorbent' timber prior to treatment, so that such timber can be treated by less severe treatment schedules. It was anticipated that the flow-meter might be just such an apparatus which, while clearly not suitable for routine industrial use in its presently described form, might demonstrate the principle upon which a modified design could be based.

In the light of this, an experiment was undertaken which was designed to examine the correlation between flow-meter results and uptakes resulting from an industrial scale impregnation.

Twelve samples of planed seasoned spruce, each measuring ca. 35 x 45 x 200 mm, were selected from 12 different beams of timber. Each was sealed across its end grain surfaces with epoxy resin (see Section 2.2.1.2.3). Two determinations of permeability to white spirit, applied at 69 kPa, were made on each sample - one on each broad longitudinal surface. Observation of rate of liquid entry was continued for a 10 minute period in each instance.

After testing with the flow-meter, the samples were open-stacked on the laboratory bench for ca. 28 days to allow the white spirit to evaporate. After this time, all 12 samples were together impregnated with an industrial organic-solvent preservative (Protim 80) which was applied using the standard industrial DVP schedule described in Section 1.5.3.6, modified to include a -85 kPa final vacuum. The uptake of preservative for each sample was determined gravimetrically from weighings made immediately before and immediately after treatment.

The results obtained from the above work are presented and discussed in Section 2.2.2.3.3.

2.2.2.2.4 Flow of emulsions

Although investigation of the flow of light organic-solvents into refractory softwood is the focus of interest and thus forms the central thrust of this investigation, this subject has not been viewed in isolation. During the execution of this work the cost of petroleum-derived solvents has steadily and inexorably risen and there has been a concomitant increase in interest within the industry in formulating organic-solvent-soluble wood preservatives as emulsions, or in water-dispersible emulsifiable concentrates. For instance, in the U.S.A., Hatcher (1980) has reported full commercial development of an emulsifiable pentachlorophenol solution, and in France similar developments are occurring.

Against this background the opportunity was taken to use the flow-meter to make an examination of the flow of emulsions laterally into spruce. Several pieces of work were undertaken, all of which yielded similar indications. One of these is described here.

The timber samples used were a consecutive series of 12, of the same type as were described in Section 2.2.2.1.4 and the flow-meter was manipulated in the manner which was also described in that section. Four test liquids, as detailed below, were applied at a pressure of ca. 103 kPa and their flows were monitored over a 20 minute period.

The four test liquids used were:

(i) Deionised water - surface tension 72.1 x 10^{-3} Nm^{-1}.

(ii) White spirit - surface tension 23.7 x 10^{-3} Nm^{-1}.

(iii) 2% Ethylan KEO in deionised water - surface tension 30.0 x 10^{-3} Nm^{-1}. Ethylan KEO is a nonylphenol ethoxylate emulsifier supplied by Diamond Shamrock and recommended by that Company for the preparation of white spirit in water emulsions.

(iv) An emulsion of 20% m/m white spirit in deionised water containing 2% m/m Ethylan KEO.

The surface tensions quoted above were determined by the drop-weight method already described (Section 1.3.3.1).

The above four liquids were allocated to the twelve consecutive samples in a repeating sequence. Thus, each was used to treat three samples which were equally spaced along the series of test samples.

The average uptakes during the twenty minute observation period are presented in Section 2.2.2.3.4 where their significance is also discussed.

2.2.2.2.5 Flow of a range of liquids

The work concerning the flow of emulsions which has been outlined in the previous section yielded results which were not anticipated and are not fully understood. However, in general terms they indicate that the chemical as well as the physical characteristics of a liquid profoundly affect its entry into timber. In a similar vein Barnacle (1977) also reported results of impregnation tests which could not be readily explained on the basis of the physical liquid characteristics of the solvents alone. Instead, they too were suggestive of influences derived from the chemical nature of the impregnant liquids.

Against this background, the flow-meter appeared to present a convenient means of rapidly examining the flow characteristics of a large number of solvents to look for patterns of characteristic behaviour. Accordingly, experimentation having this aim was initiated.

In the event, only four solvents were examined before the work was discontinued because, as the results presented in Section 2.2.2.3.5 show, it quickly became apparent that the accuracy of the technique was inadequate for the purpose in hand.

At the outset it was clear from preceding work that it would be pointless to try to establish some absolute flow rate for each liquid. Rather, experimentation was designed so that the flow

rate of each liquid could be compared with the flow rate, in closely matched samples, of some standard solvent. The selection of a standard solvent was a purely arbitrary decision and for convenience the solvent 'Presol 100' was chosen. Presol 100 is an 'industrial grade' white spirit-type solvent.

For each of the test liquids listed below, a consecutive series of between 6 and 13 (usually 7) samples of the type used previously (Section 2.2.2.1.4) was taken. Alternate samples from each series were treated at 69 kPa with either the standard solvent (Presol 100) or the test solvent being examined. Where possible the inflow of the solvent was monitored over a 20 minute period.

The liquids used in this section were:

(i) Xylene - This was selected as being a representative aromatic solvent. (White spirit and similar solvents are largely aliphatic.)

(ii) Acetone - In other work it had been reported that this solvent penetrates more evenly than white spirit-type solvents.

(iii) Industrial methylated spirits - It was anticipated that, being polar, this solvent would not penetrate well.

(iv) 10% tri iso butyl phosphate in Presol 100 - The suppliers of tri iso butyl phosphate (Tenneco Organics) claim, on the basis of uncited evidence, that this substance improves the penetration of aliphatic solvents into timber.

2.2.2.2.6 Repeated treatment of samples

The results of the work outlined in Section 2.2.2.2.5 which are reported and discussed in Section 2.2.2.3.5 indicated that the variability of spruce is such as to demand inconveniently high replication in order for the flow-meter to yield reliable results.

In response to this difficulty it was thought that the problem might be circumvented, or at least minimised, by treating each sample more than once. The initial treatment, or treatments, would establish a known flow rate for a reference-solvent into each sample. This solvent would then be allowed to evaporate-off and each sample could be reinserted into the flow tester and re-treated with the experimental solvent. The flow characteristics of the experimental solvent could then be quantified in terms relative to the flow of the reference solvent. Using such an experimental system it was hoped that a high level of accuracy could be achieved with comparatively little replication.

1,1,1, trichloroethane was selected as a solvent suitable for the 'pre-treatment' step because it is non-flammable and evaporates readily at room temperature.

As experimental samples, a consecutive series of six samples of spruce of the type previously described was used. The flow at 69 kPa of trichloroethane into each block was observed over a 20 minute period. Following this the blocks were open-stacked on the laboratory bench for almost 24 hours so that the solvent could evaporate before re-treatment with the reference-solvent was undertaken. Each block was treated in this way four times.

The total uptakes of liquid which occurred on each occasion are presented and discussed in Section 2.2.2.3.6.

2.2.2.2.7 Longitudinal flow

The results of the preceding six sections (2.2.2.2.1 through 2.2.2.2.6) all display considerable intersample variability. A technique to deal with this is suggested by the data in Section 2.2.2.3.6 but this involves treating each sample twice and so will probably always be accompanied by uncertainties about the effects which treatment by the first solvent will have on the behaviour of the second.

An alternative technique which may reduce intersample variability might be to use the flow-meter to examine longitudinal, rather than lateral, penetration. The advantages of this would be twofold. In the first instance, the rates of flow measured would be less affected by minor blemishes in the test sample. Secondly, the orientation of the main flow pathways (the axial tracheid lumena) would be such that lateral movement of liquid through the wood sample would be a less significant fraction of the total volume of liquid entering the sample.

Several attempts were made to compare the intersample variability in uptakes when introducing the liquid laterally and longitudinally. Lengths of spruce, usually ca. 45 x 45 mm in cross-section, were planed on one side and cross-cut to yield consecutive samples for experimentation. The flow of laterally-applied white spirit was first measured in the normal way. The samples were then allowed to dry before being clamped into the flow-meter with one of their transverse surfaces against the conical opening in the base-plate. An attempt was then made to monitor liquid inflow in the normal way.

The results of this experimentation are not in fact presented in detail because in only a minority of samples were satisfactory data obtained for longitudinal flow. This was because the rate of flow was often such that the narrow sight glass emptied too rapidly for sufficient readings to be taken to discern a pattern of rate of flow. Thus, whilst measurement of longitudinal flow

might, for the reasons outlined above, yield more reproducible results, this could not be demonstrated and it was concluded that the apparatus as presently constructed could not be used to monitor longitudinal flow.

2.2.2.3 Flow-meter - results and discussion - 1

2.2.2.3.1 Pattern of lateral flow and intersample variability

Resulting from the work described in Section 2.2.2.2.1, Table 2.4 presents values for the volumes of white spirit which had entered each of the 46 samples at each of the 12 monitoring intervals.

To interpret these data in their proper perspective it is apposite to note particularly the magnitude of volume movements being monitored. In Table 2.4 the average volume of liquid absorbed by each block during the 19.5 minute observation period is 0.377 cm^3. This indicates an overall average flow-rate of approximately 0.0023 $cm^3\ cm^{-2}\ min^{-1}$ (i.e. $2.3 \times 10^{-7}\ m^3\ cm^{-2}\ min^{-1}$). This flow-rate corresponds to the filling of about one layer of tracheids each minute. Clearly, tracheids will not in reality become filled in well-ordered sequential layers (Banks 1981). However, the approximation, 'one layer of tracheids per minute' is a useful indicator of what observed variations in flow-rate imply in physical terms.

Rates of flow

The data discussed in Section 2.2.1.4 indicate that, at least to a first approximation, unsteady-state flow of organic solvent into spruce may be described by the relationship $Q \propto \sqrt{t}$; the data in Table 2.4 were examined for this relationship. Figure 2.14

Table 2.4 Cumulative volumes (cm^3) of white spirit applied at 69 kPa which had entered 46 consecutive samples of seasoned spruce through 8.55 cm^2 of planed lateral surface at the times indicated. (Section 2.2.2.3.1 refers.)

Elapsed time (min)	Sample number							
	1	2	3	4	5	6	7	8
1	0.031	0.018	0.006	0.019	0.013	0.013	0.013	0.013
2	0.069	0.062	0.012	0.031	0.019	0.038	0.038	0.019
3	0.088	0.088	0.025	0.057	0.025	0.050	0.057	0.031
4	0.113	0.113	0.037	0.069	0.038	0.063	0.063	0.038
5	0.132	0.132	0.037	0.082	0.044	0.075	0.082	0.044
6	0.150	0.157	0.044	0.094	0.050	0.088	0.100	0.044
7	0.170	0.175	0.050	0.107	0.063	0.113	0.113	0.050
8	0.188	0.202	0.057	0.119	0.069	0.119	0.119	0.088
9	0.213	0.213	0.063	0.132	0.082	0.132	0.132	0.057
10	0.232	0.232	0.069	0.151	0.088	0.138	0.145	0.063
15	0.339	0.314	0.094	0.220	0.126	0.201	0.195	0.088
20	0.446	0.388	0.126	0.283	0.163	0.258	0.245	0.107

Elapsed time (min)	Sample number							
	9	10	11	12	13	14	15	16
1	0.019	0.013	0.006	0.013	0.006	0.031	0.006	0.019
2	0.025	0.019	0.038	0.031	0.025	0.031	0.013	0.057
3	0.038	0.025	0.063	0.038	0.038	0.119	0.025	0.082
4	0.050	0.031	0.075	0.050	0.050	0.145	0.031	0.113
5	0.063	0.038	0.101	0.057	0.063	0.189	0.038	0.145
6	0.069	0.044	0.107	0.069	0.069	0.220	0.044	0.157
7	0.082	0.050	0.126	0.082	0.075	0.251	0.057	0.189
8	0.088	0.057	0.146	0.088	0.088	0.276	0.063	0.214
9	0.101	0.063	0.163	0.094	0.101	0.314	0.069	0.245
10	0.107	0.069	0.170	0.101	0.113	0.352	0.075	0.258
15	0.151	0.088	0.245	0.145	0.163	0.478	0.101	0.364
20	0.195	0.119	0.308	0.189	0.201	0.584	0.132	0.459

Elapsed time (min)	Sample number							
	17	18	19	20	21	22	23	24
1	0.013	0.013	0.006	0.013	0.019	0.019	0.013	0.031
2	0.025	0.031	0.019	0.019	0.025	0.031	0.031	0.069
3	0.031	0.057	0.025	0.038	0.031	0.044	0.044	0.101
4	0.031	0.069	0.038	0.044	0.038	0.057	0.057	0.138
5	0.044	0.082	0.044	0.050	0.044	0.063	0.063	0.170
6	0.050	0.101	0.050	0.057	0.057	0.069	0.082	0.207
7	0.057	0.119	0.057	0.063	0.063	0.075	0.101	0.232
8	0.057	0.132	0.063	0.069	0.069	0.082	0.113	0.258
9	0.063	0.145	0.069	0.082	0.075	0.094	0.119	0.289
10	0.069	0.157	0.069	0.094	0.082	0.107	0.138	0.320
15	0.094	0.232	0.107	0.113	0.101	0.145	0.195	0.466
20	0.119	0.295	0.126	0.145	0.119	0.176	0.258	0.559

Table 2.4 continued

Elapsed time (min)	Sample number 25	26	27	28	29	30	31	32
1	0.013	0.013	0.019	0.013	0.050	0.019	0.038	0.031
2	0.038	0.031	0.038	0.025	0.107	0.050	0.069	0.069
3	0.050	0.038	0.050	0.031	0.157	0.063	0.107	0.101
4	0.069	0.044	0.063	0.038	0.214	0.094	0.132	0.126
5	0.075	0.057	0.088	0.044	0.258	0.119	0.157	0.151
6	0.088	0.063	0.101	0.050	0.295	0.138	0.195	0.170
7	0.101	0.082	0.113	0.057	0.333	0.151	0.214	0.189
8	0.113	0.094	0.126	0.063	0.352	0.170	0.245	0.220
9	0.132	0.101	0.138	0.075	0.371	0.182	0.270	0.239
10	0.145	0.107	0.151	0.082	0.390	0.201	0.295	0.264
15	0.207	0.151	0.207	0.113	0.528	0.264	0.402	0.352
20	0.264	0.182	0.264	0.145	0.566	0.327	0.509	0.440

Elapsed time (min)	Sample number 33	34	35	36	37	38	39	40
1	0.025	0.013	0.025	0.025	0.031	0.050	0.038	0.031
2	0.088	0.031	0.050	0.057	0.069	0.088	0.057	0.082
3	0.138	0.038	0.075	0.082	0.151	0.170	0.069	0.113
4	0.176	0.050	0.094	0.101	0.220	0.232	0.082	0.145
5	0.220	0.063	0.107	0.126	0.302	0.270	0.094	0.163
6	0.258	0.075	0.119	0.151	0.390	0.289	0.101	0.189
7	0.295	0.088	0.132	0.170	0.484	0.320	0.119	0.214
8	0.339	0.101	0.151	0.189	0.528	0.346	0.151	0.245
9	0.377	0.113	0.163	0.207	0.610	0.396	0.176	0.270
10	0.408	0.157	0.182	0.226	0.716	0.421	0.189	0.289
15	0.578	0.364	0.239	0.327	1.012	0.528	0.302	0.396
20	0.754	0.540	0.302	0.415	1.584	0.704	0.610	0.509

Elapsed time (min)	Sample number 41	42	43	44	45	46
1	0.019	0.025	0.013	0.013	0.013	0.013
2	0.050	0.069	0.044	0.038	0.050	0.038
3	0.069	0.145	0.069	0.050	0.075	0.050
4	0.088	0.207	0.088	0.063	0.113	0.069
5	0.107	0.276	0.101	0.075	0.138	0.088
6	0.119	0.339	0.119	0.088	0.163	0.101
7	0.138	0.421	0.138	0.094	0.182	0.113
8	0.157	0.478	0.151	0.107	0.207	0.126
9	0.176	0.515	0.170	0.113	0.226	0.138
10	0.189	0.578	0.182	0.119	0.251	0.157
15	0.440	0.779	0.270	0.176	0.352	0.214
20	0.842	0.974	0.352	0.264	0.540	0.276

Figure 2.14 A representative selection of data from Table 2.4 plotted to illustrate the relationships observed between liquid uptake and $\sqrt{\text{elapsed time}}$

Figure 2.14A

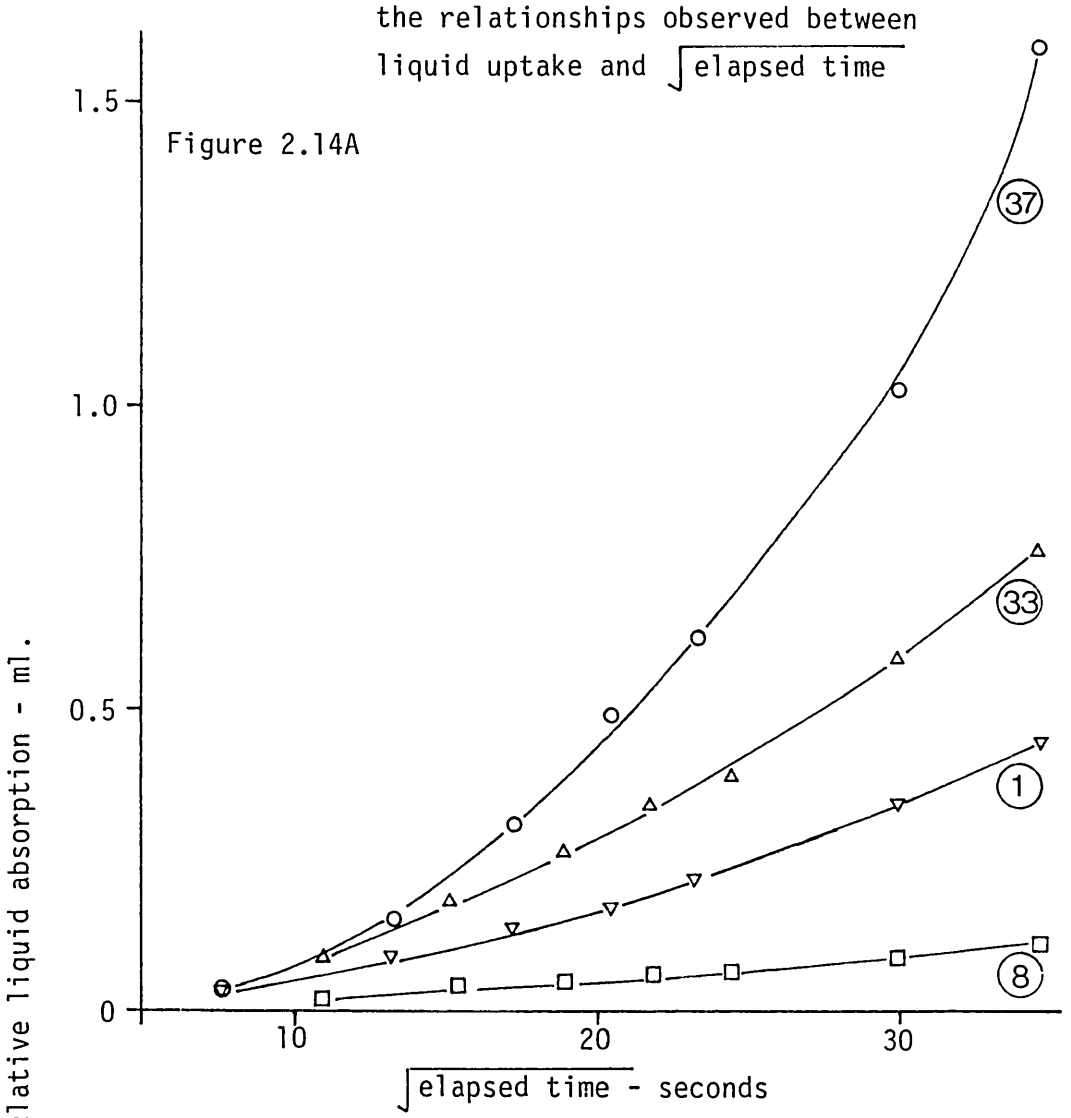
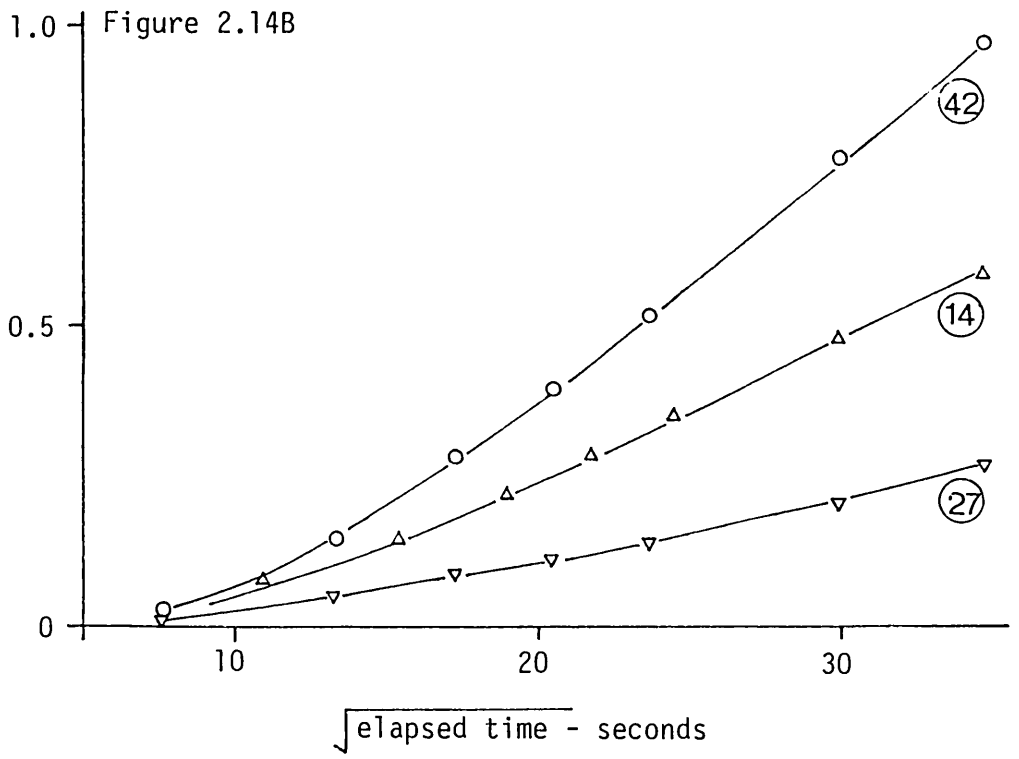


Figure 2.14B

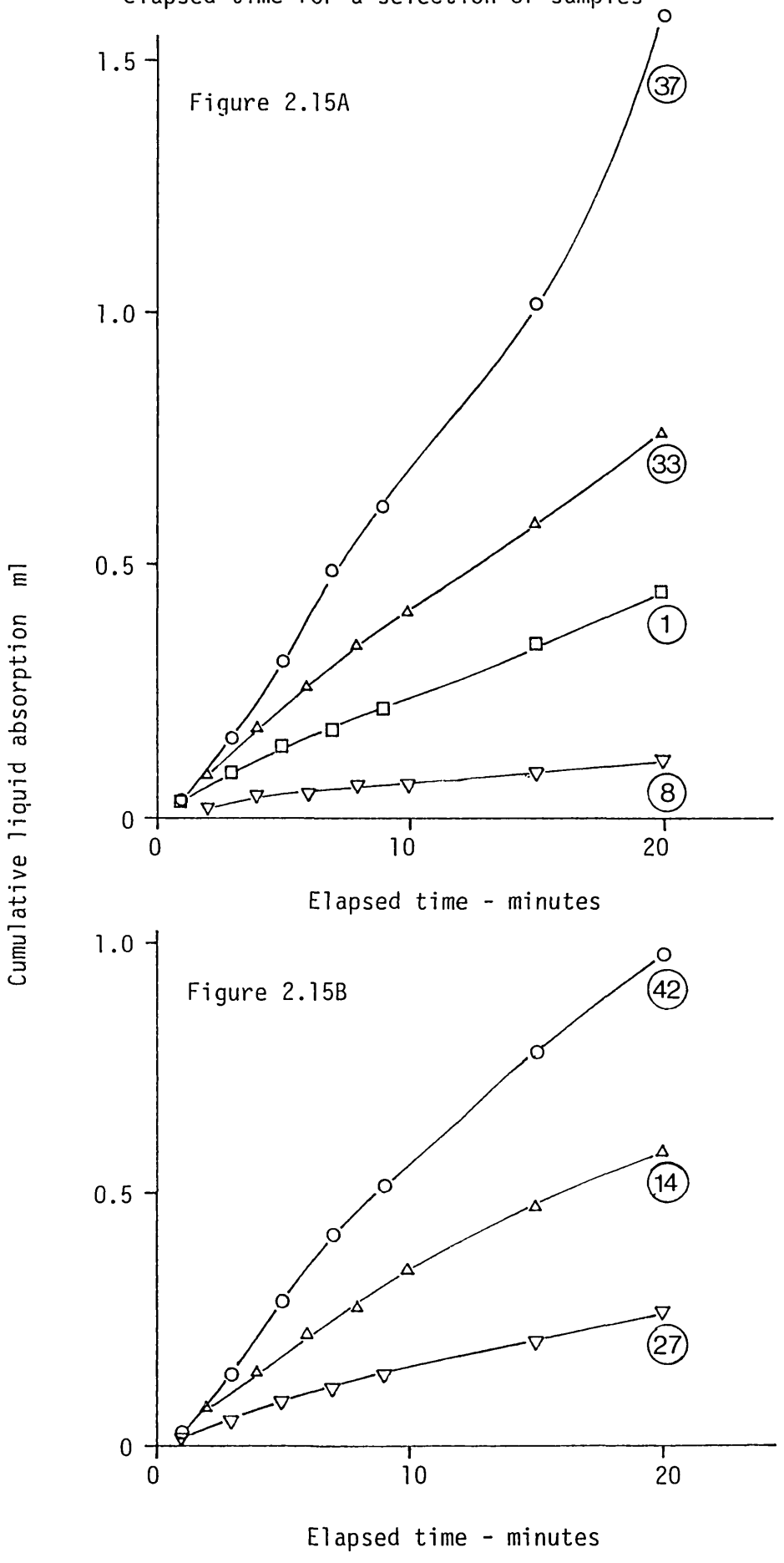


presents plots of liquid absorption against $\sqrt{\text{time}}$ for a representative selection of the samples tested. From this it is clear that, in all cases, the entry of white spirit into the samples proceeded faster than proportional to $\sqrt{\text{time}}$. Indeed, from Figure 2.15, it is apparent that in most cases the rate of liquid absorption dropped only slowly during the observation period and the gradient of log/log plots of these data is typically in the region of 0.75 - 0.8.

Of the several explanations which might be advanced to account for these observations, consideration should perhaps be given first to the possibility that these data do no more than indicate a defect in the seal around the test area of each sample. Clearly, a simple leak of liquid at the interface between the timber's surface and the base-plate of the flow-meter would introduce a steady-state component into the overall liquid movement observed. This, combined with the unsteady-state flow, which presumably would occur into the timber, could arguably lead to a gross rate of flow of liquid out of the sight glass which would display a pattern of behaviour similar to that seen in these samples.

However, there are several factors which do not support this explanation. The most compelling of these is consideration of the way in which the seal was formed. The planed surface of each sample was pressed against the steel ring around the test area until this ring was indented into the timber's surface to a depth of about 0.5 - 1.0 mm. The belief that this kind of seal would

Figure 2.15 Data from Table 2.4 plotted to display the relationship between cumulative absorption and elapsed time for a selection of samples



be fluid-tight is borne out by other work (e.g. Section 2.2.2.2.4) in which other liquids of similar viscosity to white spirit (e.g. water) sometimes showed zero flow-rates at similar test pressures.

In addition to the above, the volumes of liquids involved in these measurements are barely adequate to include liquid both moving into the timber and leaking away over the surface. It appears, therefore, that the observed flow-rates cannot simply be ascribed to deficiencies in the experimental technique.

A second possible explanation of these data is that they result from the same problem as that encountered by Venturino and Arganbright (1979) which has already been alluded to in Section 2.2.2.1.3. This is the problem of liquid entering the timber on one side of the seal around the test area, passing through a relatively small quantity of timber to by-pass the seal, and flowing away unimpeded across the timber's surface outside the test area. This sort of effect may well have contributed to the increase in flow-rate after about 15 minutes in Sample 37. However, 'leakage' of this sort is unlikely to account for all the observed flow patterns. Having regard for the approximation that one layer of tracheids filled each minute, there should have been several minutes during which flow-rate decreased with root-time before steady-state flow became established.

A third explanation of these data may be advanced, which accounts for the observed patterns fairly satisfactorily and which is consistent with the mechanism discussed in Section 2.2.1.4.

Attention has been drawn (Fig 2.8) to the fact that timber consists of a large number of relatively large voids (the tracheid lumena) communicating via comparatively small constrictions in the vicinity of the pit pairs. When liquid was introduced onto a planed lateral surface of each sample of spruce, the superficial tracheid lumena would have filled quite rapidly. From these the liquid would have flowed into lumena of underlying tracheids passing through the constrictions at the pits. If the pathways through these constrictions were sufficiently small they would have restricted flow to such an extent that the steady rise in viscous inertia in the accumulating liquid ahead of the constriction would have been masked by the tendency of capillary forces to move liquid further along the flow path. Thus the gross behaviour of the liquid would have been dominated by the rate-determining, essentially steady-state, flow which occurred through the constrictions at the pits.

In other words, in situations of slow liquid inflow, the effect of mounting viscous inertia in the accumulating bulk of liquid in the flow paths will be attenuated by the effects of capillary forces.

The net effect of these interacting factors will be that, as observed, the liquid inflow rate drops slightly with elapsed time, but not to the extent predicted by Equation 1.24.

It has already been noted that in larger-scale, industrial-type impregnations of spruce, the pattern of liquid uptake is more nearly described by $\frac{1}{Q} \propto \sqrt{t}$ (Section 2.2.1.3) than by $\frac{1}{Q} \propto t$. The different indications, on this point, yielded by that section of work and this one, are not easy to reconcile because there should have been a period of up to 15 minutes during which liquid was able to move only laterally (rather than longitudinally) away from the application-area. During this period, the rate of liquid ingress might have been expected to decrease in the same way as it had been found to do during an industrial treatment. The reason why this expectation was not borne out by the observations recorded has not yet been identified.

Intersample variability

A striking feature in the data presented in Table 2.4 is the variability in flow rates. The mean volume absorbed during the 19.5 minute observation period was 0.377 cm^3 or 4.4×10^{-2} cm^{-3} cm^{-2}. The overall standard deviation, however, was 3.26×10^{-2} cm^{-3} cm^{-2}, i.e. 74% of the mean.

Cursory examination of Table 2.4 suggested that consideration of the results in smaller groups might reduce the standard deviation within each group. Consequently the data were re-examined in groups, each of six consecutive samples, and the means and standard deviations of these groups are presented in Table 2.5.

Table 2.5 Mean uptakes of white spirit flowing laterally into Picea abies for 20 minutes at an applied pressure of 69 kPa

Samples	Mean uptake $cm^3\ cm^{-2} \times 10^{-2}$	Standard deviation	Standard deviation as % of mean
1 - 6	3.23	1.44	45
7 - 12	2.27	0.89	39
13 - 18	3.49	2.14	63
19 - 24	2.70	1.97	73
25 - 30	3.44	1.74	51
31 - 36	5.77	1.78	31
37 - 42	10.18	4.52	44
43 - 46	4.2	1.49	36

From Table 2.5 it can be seen that dividing the data into smaller groups does reduce the standard deviation of the data in most groups, but not in all. Even so in no group was the standard deviation less than 30% of the mean and in five of the eight groups it was over 40%.

The relationship between the inherent standard deviation of an experimental system and the amount of replication required for an experiment to have a specified accuracy is discussed in more detail in Section 3.1.3.1. Suffice it here to observe that where the standard deviation is as large as that quoted above, the experimental system is suitable only for studying effects which lead to very large changes in the parameter being monitored (in this instance, flow volume and flow rate). Increased accuracy can be achieved only by employing exponentially-increasing replication. Clearly, in apparatus such as this flow-meter which will accept only one sample at a time and which requires almost continuous attention throughout the monitoring period, use of a high level of replication is impractical.

Although the variability in the permeability of spruce is well known, the intersample variability between the straight-grained, closely-matched samples used in the flow-meter was a little unexpected. It is a little difficult to advance a satisfactory explanation to account for it, particularly in view of the shape of the $V/\sqrt{t}$ and V/t plots (Figures 2.14 and 2.15). Had flow rate been observed to diminish rapidly with elapsed time in consequence of random flow path termination, the observed intersample variability could have been interpreted on a similar basis. However, flow, although slow, proceeded in a broadly predictable pattern but at an unpredictable rate. The region which determined the rate of flow into the sample must therefore have been located at the surface.

Two speculative mechanisms which might lead to the formation of a relatively less permeable surface on timber samples may be proposed. Firstly, resawing, machining and handling may occlude the flow paths at the timber's surface. This could result in a slightly variable, rate-determining, less-permeable surface layer, beneath which flow could proceed more readily. Alternatively (or in addition), in view of the work of Koskelainen & Lindberg (1980) in which they note that solvent penetration proceeds better in wood having a high moisture content, it is possible that localised superficial drying at the surface of the samples could lead to a layer of relatively less permeable superficial tracheids.

2.2.2.3 Flow-meter - results and discussion continued - 2

2.2.2.3.2 Effect of pressure-gradient on flow

To recap briefly on Section 2.2.2.2.2, this sequence of experimentation was undertaken primarily to examine the extent to which the unsteady-state flow relationship, $V \propto \sqrt{\Delta P}$ (Equation 1.23), is modified by effects such as the displacement of air embolisms along a flow path.

2.2.2.3.2.1 Flow at a range of pressure gradients

Table 2.6 presents data concerning the cumulative volumes of liquid entering each of the samples at each of the observation intervals.

From this table it is immediately clear that some pieces, notably Beam 1, and to a lesser extent Beam 3, were more permeable than the remainder and flow-rate observation was curtailed because the sight glass emptied before the 30-minute observation period had elapsed. This sometimes occurred at the lower as well as the higher pressures because the internal diameter of the sight glass used at each test pressure was varied according to an a priori 'guesstimate' of the most suitable size.

Figure 2.16 presents graphically the data from Beam 2 (Table 2.6) and from this, two features, characteristic of all the data in Table 2.6 can be seen. Firstly, it is clear that at all the

Table 2.6 Sheet 1 of 6 Cumulative volumes (cm^3) of white spirit, applied at the pressure gradients indicated, which had entered samples of seasoned spruce through 8.55 cm^2 of planed lateral surface at the times indicated (Section 2.2.2.3.2 refers)

Beam no.	1	1	1	1	1	1	1	1	1	1
Sample no.	1	2	3	4	5	6	7	8	9	10
Pressure*	0/0	35/0	0/-69	138/0	413/0	69/0	276/0	207/0	69/-69	0/-35
Elapsed time (min)										
1.0	0.002	0.061	0.039	0.216	0.491	0.216	1.139	0.844	0.393	0.137
2.0	0.004	0.177	0.137	0.501	1.237	0.687	2.867	2.356	0.962	0.353
3.0	0.007	0.284	0.236	0.736	1.885	1.031	4.105	3.632	1.404	0.550
4.0	0.011	0.390	0.334	0.972	2.464	1.335	5.184	0.S.	1.767	0.736
5.0	0.014	0.492	0.432	1.208	3.024	1.600	0.S.		2.091	0.923
6.0	0.016	0.591	0.530	1.433	3.554	1.856			2.395	1.079
7.0	0.019	0.686	0.648	1.649	4.045	2.101			2.680	1.257
8.0	0.022	0.779	0.746	1.846	4.536	2.346			2.945	1.414
9.0	0.024	0.869	0.844	2.025	5.007	2.582			3.200	1.571
10.0	0.027	0.957	0.943	2.248	5.449	2.798			3.456	1.718
12.5	0.034	0.S.	1.168	2.729	0.S.	3.348			4.025	2.091
15.0	0.040		1.394	3.181		3.868			4.536	2.445
17.5	0.046		1.630	3.613		4.375			4.987	2.778
20.0	0.051		1.875	4.025		4.869			5.300	3.102
25.0	0.063		2.356	4.82		0.S.			0.S.	3.711
30.0	0.074		2.896	0.S.						4.300

Notes * For amplification see Table 2.3
0.S. = off scale

Table 2.6 continued Sheet 2 of 6

Beam no.	2	2	2	2	2	2	2	2	2	2
Sample no.	1	2	3	4	5	6	7	8	9	10
Pressure*	35 0	69 -69	207 0	276 0	413 0	69 0	0 0	0 -69	0 -35	138 0
Elapsed time (min)										
1.0	0.014	0.020	0.108	0.059	0.098	0.020	0	0.069	0.029	0.029
2.0	0.041	0.049	0.275	0.147	0.285	0.039	0	0.196	0.079	0.118
3.0	0.066	0.118	0.432	0.236	0.442	0.079	0.0004	0.314	0.118	0.177
4.0	0.091	0.157	0.589	0.314	0.599	0.118	0.0007	0.432	0.157	0.226
5.0	0.118	0.196	0.746	0.383	0.756	0.147	0.0016	0.550	0.206	0.275
6.0	0.143	0.226	0.884	0.452	0.893	0.187	0.0024	0.658	0.255	0.314
7.0	0.168	0.265	1.021	0.530	1.031	0.226	0.003	0.766	0.295	0.353
8.0	0.193	0.295	1.158	0.599	1.168	0.255	0.004	0.874	0.334	0.412
9.0	0.218	0.334	1.300	0.668	1.300	0.295	0.005	0.982	0.373	0.471
10.0	0.243	0.363	1.433	0.736	1.424	0.334	0.006	1.080	0.412	0.520
12.5	0.303	0.442	1.767	0.903	1.738	0.422	0.008	1.325	0.511	0.648
15.0	0.360	0.520	2.081	1.060	2.022	0.511	0.012	1.571	0.609	0.705
17.5	0.416	0.589	2.395	1.217	2.307	0.590	0.016	1.816	0.707	0.903
20.0	0.473	0.638	2.690	1.374	2.572	0.677	0.019	2.062	0.805	1.021
25.0	0.581	0.785	3.259	1.669	3.083	0.844	0.026	2.513	0.982	1.257
30.0	0.719	0.903	3.809	1.963	3.593	1.021	0.033	2.965	1.158	1.482

Notes * For amplification see Table 2.3
O.S. = off scale

Table 2.6 continued Sheet 3 of 6

Beam no.	3	3	3	3	3	3	3	3	3	3	3
Sample no.	1	2	3	4	5	6	7	8	9	10	11
Pressure*	69/0	35/0	413/0	207/0	276/0	-	69/-69	0/-69	0/-35	138/0	0/0
Elapsed time (min)											
1.0	0.187	0.044	0.491	0.344	0.471		1.374	0.020	0.039	0.098	0.003
2.0	0.471	0.112	1.168	0.884	1.139		2.081	0.167	0.128	0.255	0.013
3.0	0.717	0.176	1.787	1.355	1.649		2.631	0.314	0.216	0.393	0.020
4.0	0.942	0.242	2.356	1.777	2.111		3.122	0.452	0.295	0.511	0.027
5.0	1.149	0.305	2.916	2.170	2.513		3.554	0.589	0.373	0.638	0.034
6.0	1.345	0.364	3.436	2.543	2.886		3.996	0.717	0.452	0.746	0.040
7.0	1.532	0.424	3.947	2.886	3.230		O.S.	0.844	0.530	0.854	0.046
8.0	1.728	0.481	4.428	3.240	3.554			0.972	0.609	0.962	0.051
9.0	1.905	0.534	4.879	3.554	3.848			1.100	0.687	1.060	0.057
10.0	2.062	0.587	5.321	3.868	4.143			1.217	0.766	1.158	0.061
12.5	2.454	0.713	O.S.	4.595	4.830			1.502	0.952	1.414	0.073
15.0	2.808	0.826		5.282	5.439			1.767	1.149	1.649	0.084
17.5	3.151	0.936		5.910	6.008			2.013	1.335	1.885	0.095
20.0	3.495	O.S.		O.S.	O.S.			2.248	1.521	2.101	0.105
25.0	4.104							2.867	1.885	2.543	0.123
30.0	4.722							O.S.	2.229	2.955	0.139

Notes * For amplification see Table 2.3
O.S. = off scale

Table 2.6 continued Sheet 4 of 6

Beam no.	4	4	4	4	4	4	4	4	4	4	4
Sample no.	1	2	3	4	5	6	7	8	9	10	11
Pressure*	69 -69	-	0 0	207 0	138 0	35 0	276 0	69 0	413 0	0 -69	0 -35
Elapsed time (min)											
1.0	0.039		0	0.098	0.059	0.017	0.069	0.010	0.079	0.049	0.020
2.0	0.108		0	0.295	0.128	0.049	0.137	0.029	0.206	0.108	0.039
3.0	0.157		0	0.471	0.196	0.080	0.216	0.059	0.324	0.167	0.059
4.0	0.206		0	0.650	0.295	0.112	0.285	0.088	0.422	0.236	0.079
5.0	0.255		0.0008	0.805	0.353	0.143	0.344	0.108	0.520	0.285	0.098
6.0	0.295		0.0016	0.972	0.422	0.174	0.403	0.128	0.618	0.334	0.118
7.0	0.334		0.0024	1.129	0.481	0.206	0.452	0.147	0.717	0.383	0.147
8.0	0.383		0.0031	1.286	0.550	0.234	0.511	0.177	0.815	0.422	0.176
9.0	0.422		0.0039	1.433	0.609	0.262	0.560	0.196	0.913	0.461	0.196
10.0	0.461		0.0047	1.571	0.668	0.291	0.609	0.216	0.992	0.511	0.216
12.5	0.540		0.0051	1.924	0.805	0.361	0.726	0.265	1.188	0.628	0.255
15.0	0.628		0.0055	2.238	0.952	0.429	0.827	0.324	1.424	0.726	0.304
17.5	0.707		0.0063	2.553	1.090	0.495	0.923	0.373	1.620	0.825	0.353
20.0	0.776		0.0071	2.857	1.227	0.558	1.011	0.422	1.797	0.913	0.393
25.0	0.913		0.011	3.436	1.473	0.680	1.188	0.511	2.170	1.070	0.471
30.0	1.031		0.017	3.966	1.708	0.795	1.355	0.599	2.503	1.237	0.550

Notes * For amplification see Table 2.3
O.S. = off scale

Table 2.6 continued Sheet 5 of 6

Beam no.	5	5	5	5	5	5	5	5	5	5
Sample no.	1	2	3	4	5	6	7	8	9	10
Pressure*	276 0	0 0	0 -35	207 0	0 -69	69 -69	413 0	138 0	69 0	35 0
Elapsed time (min)										
1.0	0.236	0.002	0.039	0.226	0.088	0.079	0.295	0.177	0.098	0.028
2.0	0.369	0.004	0.098	0.511	0.245	0.196	0.707	0.530	0.236	0.069
3.0	0.844	0.006	0.157	0.766	0.383	0.295	1.041	0.864	0.353	0.104
4.0	1.100	0.008	0.196	1.021	0.511	0.393	1.374	1.080	0.452	0.134
5.0	1.355	0.009	0.215	1.247	0.628	0.471	1.669	1.335	0.560	0.163
6.0	1.590	0.011	0.226	1.463	0.746	0.540	1.963	1.571	0.648	0.192
7.0	1.816	0.012	0.236	1.679	0.854	0.609	2.199	1.787	0.746	0.220
8.0	2.042	0.013	0.255	1.895	0.962	0.697	2.435	2.003	0.834	0.248
9.0	2.258	0.015	0.275	2.101	1.070	0.776	2.690	2.219	0.923	0.275
10.0	2.474	0.016	0.295	2.297	1.178	0.844	2.935	2.435	1.011	0.302
12.5	3.014	0.019	0.334	2.778	1.433	1.001	3.515	2.955	1.217	0.369
15.0	3.515	0.022	0.373	3.240	1.669	1.158	4.045	3.446	1.414	0.434
17.5	4.005	0.026	0.412	3.691	1.905	1.296	4.555	3.927	1.610	0.498
20.0	4.496	0.028	0.452	4.123	2.140	1.433	5.027	4.388	1.787	0.564
25.0	O.S.	0.034	0.530	4.968	2.592	1.689	O.S.	5.301	2.140	0.691
30.0		0.040	0.609	5.773	3.030	1.944		O.S.	2.494	0.751

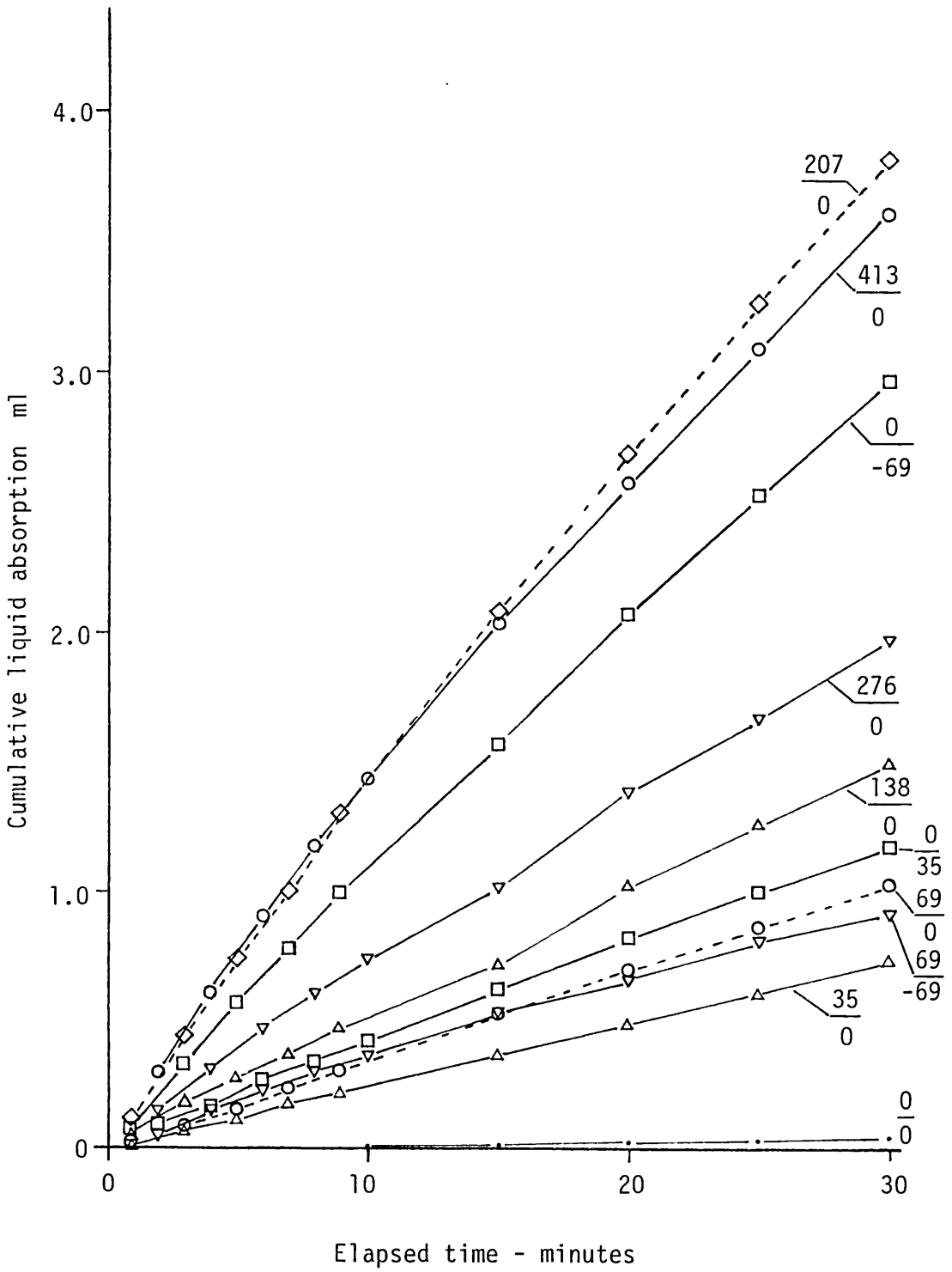
Notes * For amplification see Table 2.3
O.S. = off scale

Table 2.6 continued Sheet 6 of 6

Beam no.	6	6	6	6	6	6	6	6	6	6
Sample no.	1	2	3	4	5	6	7	8	9	10
Pressure*	0 / -35	276 / 0	35 / 0	138 / 0	0 / -69	69 / 0	413 / 0	69 / -69	0 / 0	207 / 0
Elapsed time (min)										
1.0	0.010	0.098	0.011	0.029	0.020	0.039	0.147	0.039	0.0004	0.098
2.0	0.029	0.236	0.028	0.079	0.059	0.088	0.393	0.098	0.002	0.236
3.0	0.049	0.363	0.044	0.118	0.098	0.137	0.619	0.157	0.004	0.344
4.0	0.069	0.491	0.060	0.157	0.128	0.187	0.834	0.206	0.006	0.452
5.0	0.088	0.599	0.075	0.196	0.157	0.236	1.031	0.255	0.008	0.569
6.0	0.108	0.707	0.091	0.226	0.196	0.275	1.217	0.295	0.010	0.668
7.0	0.128	0.805	0.105	0.265	0.236	0.314	1.394	0.334	0.012	0.766
8.0	0.137	0.903	0.119	0.295	0.265	0.353	1.571	0.373	0.015	0.864
9.0	0.147	1.001	0.135	0.334	0.304	0.393	1.728	0.403	0.017	0.962
10.0	0.167	1.100	0.149	0.373	0.334	0.432	1.885	0.442	0.019	1.050
12.5	0.206	1.335	0.187	0.461	0.422	0.550	2.258	0.530	0.025	1.247
15.0	0.245	1.561	0.223	0.550	0.511	0.658	2.611	0.609	0.030	1.443
17.5	0.285	1.787	0.258	0.628	0.589	0.756	2.926	0.677	0.036	1.640
20.0	0.314	1.993	0.292	0.707	0.668	0.854	3.240	0.746	0.041	1.826
25.0	0.393	2.395	0.361	0.854	0.825	1.041	3.819	0.884	0.051	2.179
30.0	0.481	2.769	0.427	1.001	0.982	1.217	4.379	1.011	0.062	2.313

Notes * For amplification see Table 2.3
O.S. = off scale

Figure 2.16 Data from Table 2.6 plotted to display variation in absorption against time at a range of pressure gradients. (See Table 2.3 for explanation of $\frac{207}{0}$ etc.)



pressure gradients monitored, regardless of whether the gradients were derived from a vacuum within the timber, from an applied pressure on the liquid, or from a combination of the two, the pattern of liquid ingress was broadly similar. Secondly, the rates of inflow did not increase in logical progression as the pressure gradient increased. Certainly, the zero pressure gradient was always associated with the lowest flow rate in each set of samples and, in five of the six beams, the largest pressure gradient of 413 kPa (60 p.s.i.) promoted one of the highest flow rates, but between these two extremes a good deal of overlap was observed.

When calculating average uptake values for all the beams at each of the pressures used (Table 2.7), only the first 10 minutes of flow was considered. This procedure was adopted so as to include as many samples as possible (only three had to be omitted due to insufficient data) and this analysis was considered justified in view of the fact, noted previously, that the pattern of liquid ingress was broadly similar in all samples and did not alter significantly with time.

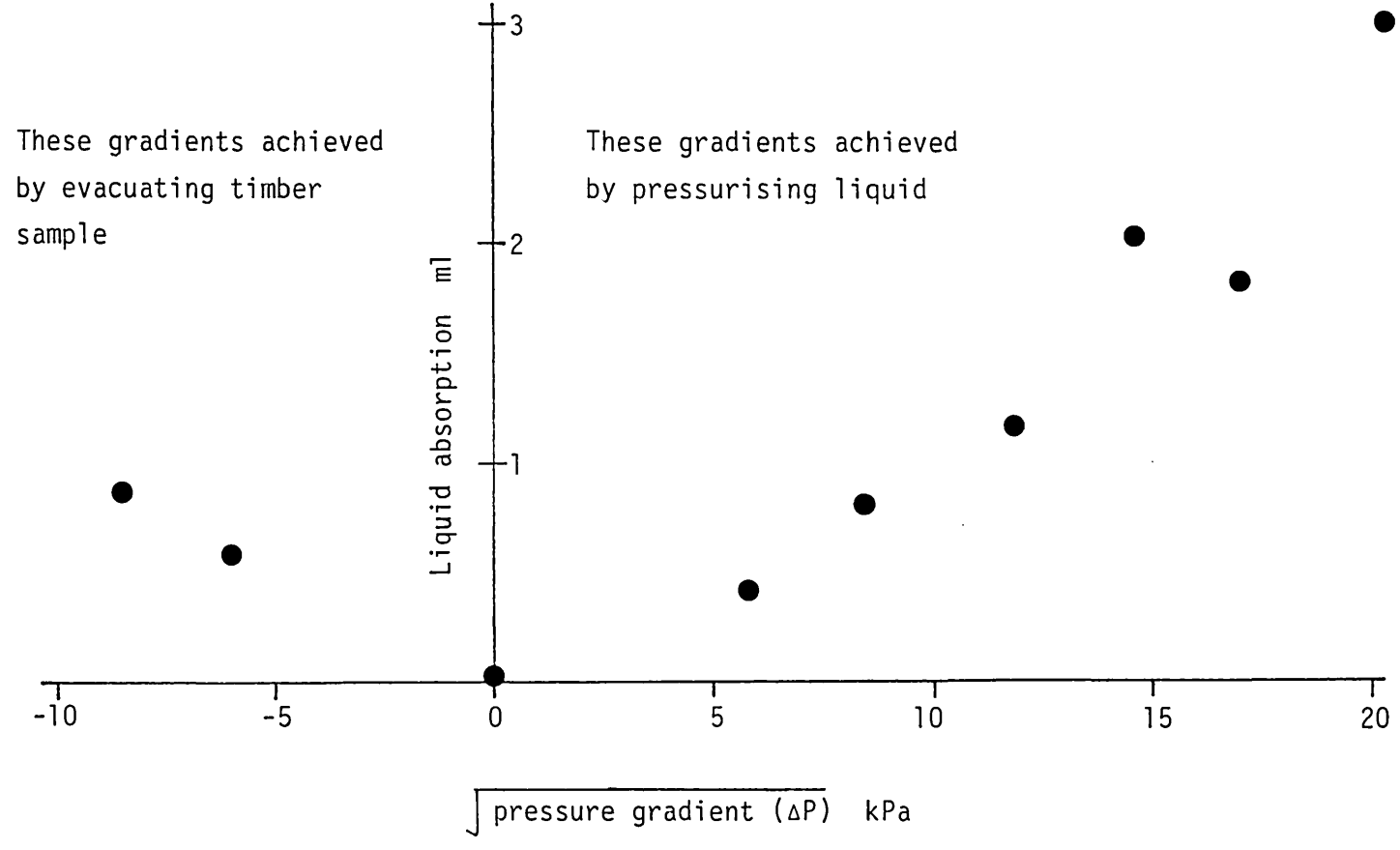
The average uptake data in Table 2.7 are plotted in Figure 2.17. This figure displays two particularly interesting features. One of these is the relative effects on flow rate of a pressure gradient set up by a vacuum within the timber vis-a-vis a similar gradient established by applying a pressure to the liquid. The other is the relationship between uptake and applied pressure.

Table 2.7 Volumes (cm^3), and average volumes (cm^3), of white spirit entering samples of seasoned spruce through 8.55 cm^2 of planed lateral surface during a 10 minute observation period. (Data abstracted from Table 2.6)

Pressure gradient*	Beam number 1	2	3	4	6	7	Average	Standard deviation
$\frac{0}{-69}$	0.943	1.08	1.217	0.511	1.178	0.334	0.877	0.37
$\frac{0}{-35}$	1.718	0.412	0.766	0.216	0.295	0.167	0.596	0.59
$\frac{0}{0}$	0.027	0.006	0.061	0.005	0.016	0.019	0.022	0.02
$\frac{35}{0}$	0.957	0.243	0.587	0.291	0.302	0.149	0.421	0.30
$\frac{69}{0}$	2.798	0.334	2.062	0.216	1.011	0.432	0.833	1.01
$\frac{138}{0}$	2.248	0.520	1.158	0.668	2.435	0.373	1.234	0.90
$\frac{207}{0}$	-	1.433	3.868	1.571	2.297	1.050	2.044	1.10
$\frac{276}{0}$	-	0.736	4.143	0.609	2.474	1.100	1.812	1.50
$\frac{413}{0}$	5.449	1.424	5.321	0.992	2.935	1.885	3.001	1.96
$\frac{69}{-69}$	3.456	0.363	-	0.461	0.844	0.442	1.113	1.32

* For amplification, refer to Table 2.3

Figure 2.17 Data from Table 2.7 plotted to display, against $\sqrt{\text{pressure gradient}}$, the average absorption of liquid by spruce samples during 10 minutes in the flow meter



Dealing firstly with the flow rates induced by vacuum and applied-pressure, it should be recalled that bubble formation is more likely to occur at low pressure than at high (especially when the low pressure is below ambient atmospheric pressure), and that bubbles in a flow path of varying diameter will interfere with flow. From these facts it follows that a partial vacuum is likely to be less effective at promoting and sustaining flow than is an applied-pressure because, in vacuum-induced flow, bubble formation and consequent path blockage are likely to be exacerbated whereas in a pressure-induced flow stream, bubble formation will be suppressed.

However, this theoretical prediction is not supported by the results, as presented in Figure 2.17. Instead, it appears that vacuum-induced pressure gradients were as effective in promoting flow into spruce as were applied-pressures of similar magnitude.

At this juncture it is apposite to note that in none of the three pieces of experimentation discussed so far (i.e. in Sections 2.2.1.4, 2.2.2.3.1 and 2.2.2.3.2 (thus far)) has it been possible to adduce any evidence that bubble formation impedes the flow of white spirit into spruce. Set against this observation, however, must be a consideration of the second interesting feature displayed in Figure 2.17.

It may be recalled that Petty (1978b) showed that a general equation to describe the effect of pressure on flow rate may take the form $Q \propto \sqrt{\Delta P}$, but in practice it is noted that flow rate (Q) increases disproportionally more than $\propto \sqrt{\Delta P}$ and this phenomenon is usually explained in terms of dislodging air embolisms. On this basis, the present experimentation was expected to reveal a non-linear relationship between V and $\sqrt{\Delta P}$ and, in so far as the rather variable results can be interpreted in this way, Figure 2.17 does indeed suggest this predicted pattern. Indeed, such is the departure from the $Q \propto \sqrt{\Delta P}$ relationship that a more nearly-linear relationship is perhaps suggested if V is plotted against ΔP (Figure 2.18).

The evident variability of these results is such as to make their further analysis somewhat speculative and it is for this reason that the two supplementary experiments described in Section 2.2.2.2.2.2 and 2.2.2.2.2.3 were undertaken. Their results, discussed below (2.2.2.3.2.2 and 2.2.2.3.2.3) supplement the foregoing and clarify the V/ΔP relationship.

2.2.2.3.2.2 Flow at 138 and 413 kPa

and

2.2.2.3.2.3 Flow at 69 and 138 kPa

These two groups of flow rate measurements yielded results which may be summarised in the two tables 2.8 and 2.9.

Figure 2.18 Data from Table 2.7 plotted to display the average absorption of liquid by spruce samples during 10 minutes in the flow meter at the pressure gradients indicated

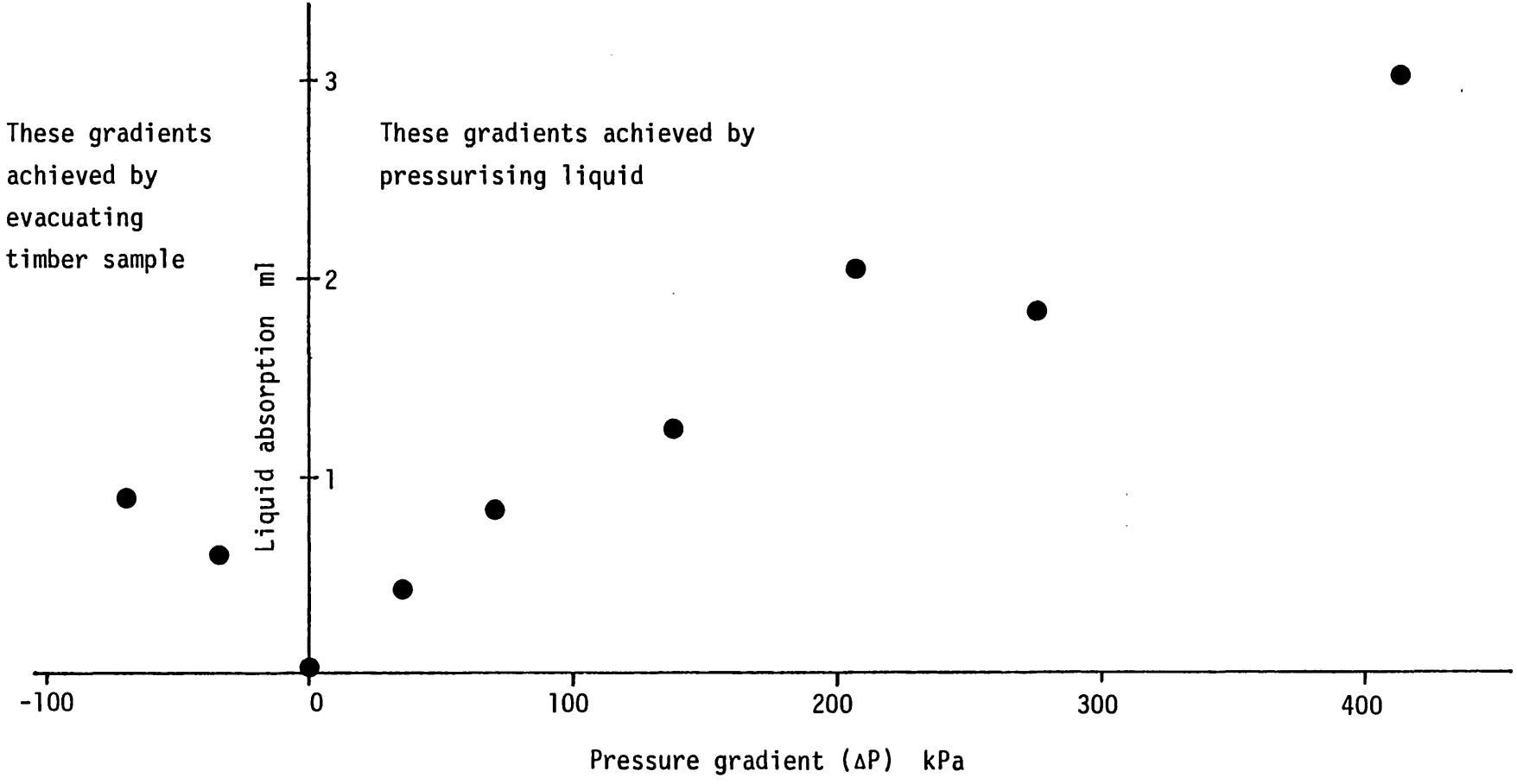


Table 2.8

Pressure gradient kPa	Average volume of white spirit absorbed during 10 minutes in flow-meter cm^3 (s. dev.)
0	0.03
138	1.02 (0.6)
413	2.78 (1.0)

Table 2.9

Pressure gradient kPa	Average volume of white spirit absorbed during 10 minutes in flow-meter cm^3 (s. dev.)
0	0.09
69	1.24 (0.8)
138	2.28 (1.7)

The high standard deviations which were recorded limit the reliance which should be placed on these data, although none of the figures is based on less than 11 replicates.

In order to examine the relationship between V and ΔP these data are plotted against $\sqrt{\Delta P}$ and ΔP (Figures 2.19 & 2.20). From these figures it is clear that the data are much more nearly indicative of a $V \propto \Delta P$ relationship.

Arguably these results simply demonstrate the apparent increase in permeability which occurs as increased applied pressure dislodges air-embolisms. Rather similar results quoted by Purslow (1976) have been interpreted in this way by Petty (1978b).

Certainly the observation '$V \propto \Delta P$' is not directly in conflict with the 'air-embolism' interpretation of Petty and others, but it is at the very least a coincidence that a random modification to one relationship ($V \propto \sqrt{\Delta P}$) should repeatedly indicate the relationship $V \propto \Delta P$, which is more familiar as a derivative of the steady-state flow equations.

The above line of discussion will be reintroduced and developed in Section 4. Suffice it here to conclude by observing that the relationship $V \propto \Delta P$, as well as being consistent with air-embolism clearance, is also consistent with the mechanism postulated in Section 2.2.2.3.1 in which the rate of liquid flow is controlled by the essentially steady-state flow through the constrictions encountered at the pits.

Figure 2.19 Data from Tables 2.8 (●) and 2.9 (■) plotted to display, against $\sqrt{\text{pressure gradient}}$, average absorption of liquid by spruce samples during 10 minutes in the flow meter

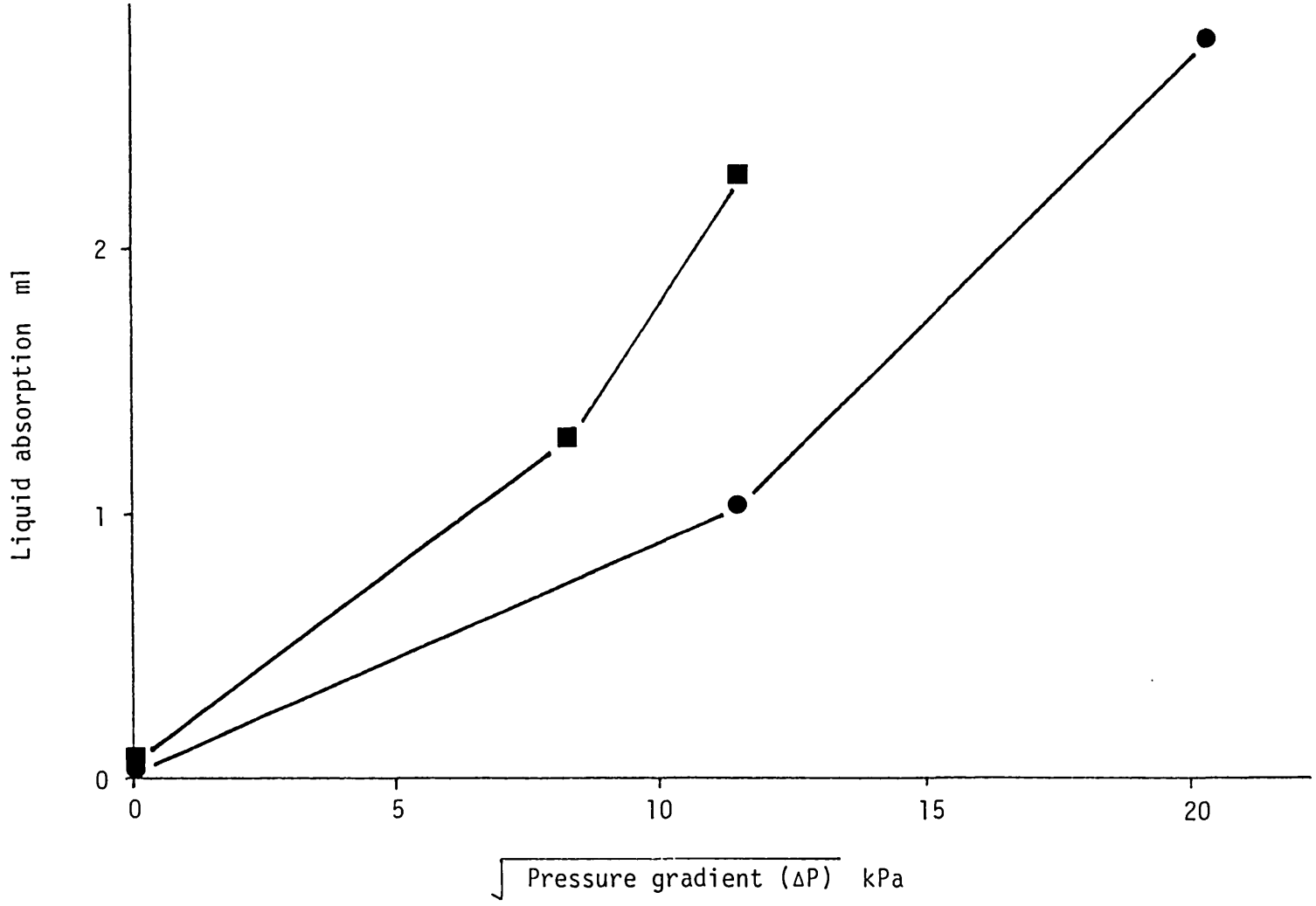
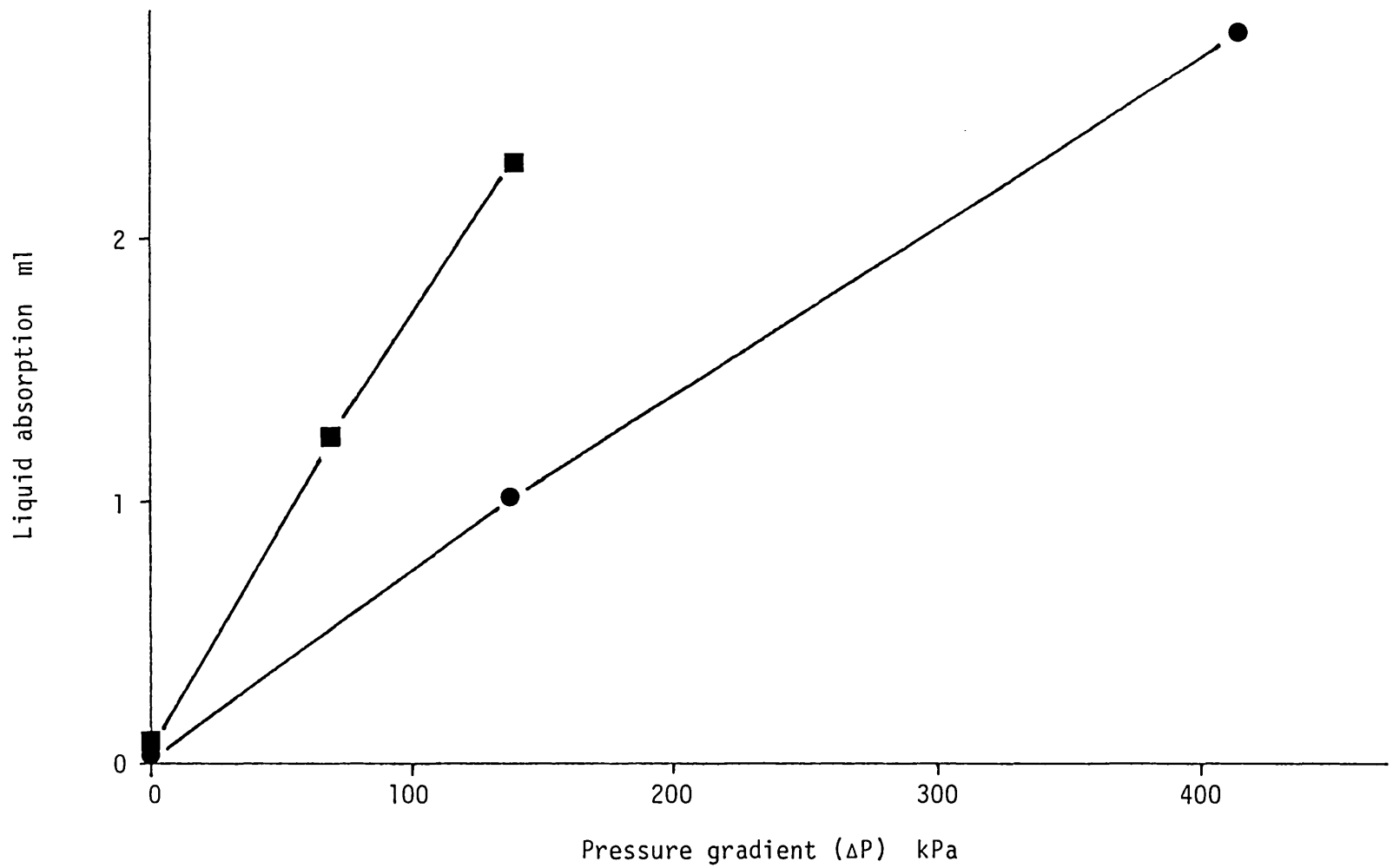


Figure 2.20 Data from Tables 2.8 (●) and 2.9 (■) plotted to display average absorption of liquid by spruce samples during 10 minutes in the flow meter at the pressure gradients indicated



2.2.2.3 Flow-meter - results and discussion continued - 3

2.2.2.3.3 Correlation of flow-meter readings with uptakes during industrial impregnation

The experimentation described in Section 2.2.2.2.3 yielded the results which are summarised in Table 2.10. Figures in the left hand column of data in this table are the uptakes of preservative which resulted from impregnation by the DVP process. Each of these figures compares with the corresponding figures in the two central columns of data which are the total volumes of liquid leaving the sight glass of the flow-meter during the final 9.5 minutes of each 10 minute observation period. These two central columns of data are headed respectively 'sap' and 'heart'. Those figures tabulated in the 'sap' column were obtained from testing the broad face of each 35 x 45 mm sample which was nearest the outside of the tree from which the sample was cut. Conversely, the 'heart' figures were obtained from the broad face which was nearest the inside. The column on the extreme right of Table 2.8 tabulates average values derived from the two central columns.

An immediately surprising feature of these data is that the average flow-meter result for the 'heart' side of the samples was higher than that for the 'sap' side. This is a slightly misleading observation because six of the twelve samples gave a higher reading on the 'sap' side, and the converse. Thus, in these samples there was no consistent difference in the permeability of their two sides.

Table 2.10 Comparison of sample permeability, as indicated by flow-meter results, with uptakes of an industrial organic solvent wood preservative applied by the 'one bar' cycle

Sample (Lab ref. no.)	Uptake of 'Protim 80' applied by one bar cycle gm/sample	Volume (ml) of white spirit at 69 kPa leaving sight glass of flow-meter during final 9.5 mins. of 10 min. observation period		
		Sap	Heart	Average
1043	41.53	0.481	1.532	1.007
1044	47.95	0.609	0.815	0.712
1045	47.57	0.471	0.324	0.398
1046	117.41	0.599	0.344	0.472
1047	29.82	0.412	0.785	0.599
1049	51.34	0.481	0.560	0.521
1051	140.44	1.158	1.365	1.262
1052	101.93	0.746	0.501	0.624
1053	52.10	0.216	2.022	1.119
1055	194.43	1.355	1.080	1.218
1058	95.89	0.550	0.314	0.432
1059	100.42	2.081	0.421	1.247
Means:	85.07	0.763	0.838	0.801
Correlation with uptake:		0.62	-0.05	0.43

In interpreting this finding, two factors must be borne in mind. Firstly, these samples were not 'orientated' laterally. That is to say, no attempt was made to select samples in which the growth rings ran parallel, or nearly so, to the broader lateral face, for example. Indeed, such an orientation of growth rings tended to be the exception rather than the rule in randomly selected samples, and rings running more or less diagonally across the sample's cross-section were the more common pattern. In consequence of this, the 'sap' side tended to be more nearly tangential than the 'heart' side which latter often presents a roughly radial face. Evidence is presented elsewhere (Section 3.2.2.2) that radial penetration is limited in many samples while tangential penetration, especially into and around the latewood rings, is a preferred pathway for liquid. Thus, penetration into a radial face which presents access to many growth rings is likely to proceed more rapidly than penetration into a nearly tangential face where the number of latewood rings available is more limited.

Secondly, not only were the samples not 'orientated' laterally, they did not necessarily include both 'heartwood' and 'sapwood'. Thus, there was no basis to expect that the 'sap' side of each sample would be materially more permeable than the 'heart' side.

With regard to the main purpose of this section of experimentation, i.e. investigation of the relationship between flow-meter readings and uptakes during industrial impregnation, the correlation between these two was poor. In fact, taking the 'heart' figures

alone there was effectively no correlation at all, although both the 'sap' and the 'average' values do show some correlation with the uptake figures.

This poor correlation was not unexpected in view of the intersample variability noted in Section 2.2.2.3.1. The total area through which liquid entered each of these samples during impregnation was 320 cm^2, whereas the area which was tested in the flow-meter was ca. 17 cm^2. This was only ca. 5% of the total surface area of each sample. The clear conclusion has, therefore, to be that the intrinsic variability of spruce is such that the flow-meter described here, or indeed any other device which tests only a small sample from a large piece, cannot give a precise indication of likely uptake of preservative during industrial treatment. This conclusion is supported by the results of very different work by Fowlie (1981) who was unable to demonstrate satisfactory correlation between the permeability of samples of spruce poles and the subsequent uptake of creosote by those poles during an industrial impregnation process.

While concluding that the flow-meter has little predictive value in connection with spruce treatment, it should, however, be noted that results from this piece of apparatus do display a broad measure of agreement with a timber's treatability. For example, results obtained during the early development of this apparatus consistently indicated the higher permeability of Pinus sylvestris sapwood vis-a-vis the heartwood of that species and the very high permeability of Pinus radiata sapwood was also demonstrated.

2.2.2.3 Flow-meter - results and discussion continued - 4

2.2.2.3.4 Flow of emulsions

This section of the investigation (described in Section 2.2.2.2.4), in which four test liquids were applied at ca. 103 kPa to a planed lateral surface of 19 x 43 mm cross-section samples of spruce, was undertaken as part of an exploratory examination of the flow of emulsions.

The results obtained are summarised in Table 2.11.

Table 2.11 Uptake during a 20 minute period of 4 liquids applied laterally to spruce over an 8.55 cm^2 area at a pressure of ca. 103 kPa

Liquid	Surface Tension	Contact angle	Average* uptake cm^3 (s. dev.)
Deionised water	72.1 x 10^{-3} Nm^{-1}	≤ca. 110°	0.033 (0.006)
White spirit	23.7 x 10^{-3} Nm^{-1}	≤ca. 25°	1.272 (0.412)
2% Ethylan KEO in water	30.0 x 10^{-3} Nm^{-1}	ca. 25°	0.003 (-)
20% white spirit in water containing 2% Ethylan KEO	30.0 x 10^{-3} Nm^{-1}	-	0.010 (0.00) *Average of 3 samples

The first two results presented in Table 2.11 were not unexpected. It is common experience that water flows into timber less readily than does organic solvent and thus the observed, ca. 40 fold, difference in flow rates is in keeping with this. However, although the phenomenon of differing flow rates is well known, the mechanism which causes this difference is perhaps less widely understood. The viscosities of water and white spirit are closely similar but their surface tensions, noted in Section 2.2.2.2.4, vary significantly. Richardson (1978) argued that the higher the surface tension of a liquid, the faster it should flow into timber (c.f. the Rideal Washburn equation - Equation 1.12). Clearly, Richardson's view is not supported by the present experimentation. This perhaps indicates that the liquid's advancing contact angle is the more critical factor. White spirit shows an advancing contact angle of about 25° tending rapidly to approach zero, whereas water is often non-wetting to timber on initial contact and the contact angle tends to zero very much more slowly. In addition to this, the greater the surface tension of a liquid, the greater the impediment to flow which is presented by bubbles which may develop in the flow path (c.f. Section 1.3.4).

Regarding the flow characteristics of emulsions of white spirit in water, it seemed reasonable to anticipate that the best which could be anticipated was that the emulsion would flow into timber at a similar rate to its continuous phase (water) alone. When it was observed (Table 2.11) that emulsions in fact flow into timber rather less well than their continuous phase alone, this

observation was interpreted on a basis analagous to the oft-cited air embolism problem, i.e. it was reasoned that discrete droplets of the discontinuous phase will behave in rather the same way as gas bubbles.

The results presented in this section suggest that the above interpretation is at best incomplete because a simple 2% solution of the surfactant 'Ethylan KEO' in water also exhibited a negligibly low inflow rate, similar to that of the emulsion. Thus, reducing both the surface tension and the contact angle of water by the addition of surfactant actually impaired its inflow characteristics. It seems, therefore, that not simply the physical but also the chemical characteristics of the wood/liquid interface must be considered in relation to the impregnation of timber with preservative liquids. More detailed consideration of this was judged to fall outside the scope of the present investigation, but this has since formed the basis of a separate piece of work (Saunders and Blow 1985).

2.2.2.3 Flow-meter - Results and discussion continued - 5

2.2.2.3.5 Flow of a range of liquids

In the light of the indications arising in the results presented in the previous section, that the chemical composition of a solvent may have a profound influence on its unsteady-state flow characteristics, this section of work was undertaken to examine the flow characteristics of a range of solvents.

The results obtained may be summarised as follows.

Table 2.12 Uptakes during 20 minutes of a range of liquids applied laterally at 69 kPa to 8.55 cm^2 planed seasoned spruce

Test liquid	Uptake of test liquid cm^3 (s. dev.)	Uptake of Presol 100 cm^3 (s. dev.)	Ratio of uptakes
Xylene	0.260(0.109)	0.138(0.050)	1.9 : 1
Acetone	0.287(0.064)	0.174(0.030)	1.6 : 1
I.M.S.[1]	0.093(0.036)	0.217(0.078)	1 : 2.3
10% m/m t IBP[2] in Presol 100 - A	0.284(0.06)	0.084(0.014)	3.4 : 1
10% m/m t IBP in Presol 100 - B	0.094(0.027)	0.068(0.022)	1.4 : 1

[1] I.M.S. = Industrial methylated spirits
[2] t IBP = tri iso butyl phosphate

Of the four figures in the upper portion of Table 2.12, three indicate that the test liquids flowed into spruce faster than Presol 100 while the fourth, I.M.S., which was the most polar of the liquids tested, flowed more slowly. However, before firm conclusions are drawn, these data should be considered in the light of the different indications from the two series of samples which were tested in order to examine the flow characteristics of t-IBP solutions. In the first of these - Series A - there appears to be a clear indication that this additive does indeed substantially improve the flow of Presol 100 through timber. This observation had clear practical implications and stimulated further investigation, including treatment of a second series of test samples - Series B. The results of this second series are included at the foot of Table 2.12. This second test series (and indeed the other additional work) failed to confirm the preliminary indications derived from Series A.

It is concluded that the discrepancy between the results of the two pieces of work was due to one or both of the experiments involving too few samples. This problem can obviously be overcome by greater replication. However, experimentation involving such replication would be very time-consuming to execute.

Having regard to the limited time available and to the facts that the subject of liquid/timber interactions is clearly a vast area of study to enter, and is somewhat aside from the main focus of this investigation, examination of the unsteady-state flow characteristicsof a range of solvents was not further investigated.

2.2.2.3 Flow-meter - results and discussion continued - 6

2.2.2.3.6 Repeated treatment of samples

As was indicated in Section 2.2.2.2.6, this section of work was undertaken to assess the feasibility of establishing the flow pattern of a reference solvent (1,1,1 trichloroethane) into spruce as a preparatory step to examining the flow characteristics of other solvents.

The results obtained are summarised in Table 2.13.

Table 2.13 Volumes (cm^3) of 1,1,1 trichloroethane applied at 69 kPa flowing into 6, 19 x 35 x 43 mm samples of planed seasoned spruce through 8.55 cm^2 during 4 successive 20-minute periods

Sample No.	Treatment No.				Coefficient of variance
	1	2	3	4	
1	0.547	0.484	0.484	0.760	0.23
2	1.269	0.880	1.257	0.917	0.20
3	1.081	1.269	1.477	1.467	0.14
4	0.980	1.301	1.382	1.602	0.20
5	1.188	1.351	1.514	1.451	0.10
6	0.905	0.936	1.144	0.999	0.11
Mean	0.995	1.037	1.210	1.199	$\overline{cv}$ 0.163
Coefficient of variance	0.26	0.32	0.32	0.29	$\overline{cv}$ 0.298

Examination of the coefficients of variance presented in Table 2.13 (coefficient of variance (C.V.) = standard deviation divided by the mean) reveals that there was consistently less variation between the uptakes noted during successive treatments of the same sample than between the uptakes of closely adjacent samples cut from the same beam. In fact the average C.V. between uptakes from successive treatments is only about 55% of the C.V. between samples.

The rather complex relationship between C.V. and the degree of replication required for an experiment to have a specified accuracy is discussed in Section 3.1.3.1. From this discussion it may be seen that there is a fundamentally exponential relationship between the C.V. and the level of replication. Thus a reduction in C.V. of 45% permits a very substantial reduction in the replication required for a given purpose - certainly more than enough to compensate for the need to treat each sample twice.

It appears, therefore, that when the flow-meter is used to continue the work introduced briefly in Section 2.2.2.3.5, the technique of establishing for each sample a flow rate for 1,1,1 trichloroethane may be a more efficient experimental system than that used in Section 2.2.2.2.5.

A facet of these data which requires further comment is the fact that successive treatments of each sample did not yield identical results. The first three successive treatments yielded steadily rising uptakes and, between treatments 3 and 4, in which the

average uptakes were virtually the same, individual samples showed quite marked variations. The systematic trends can probably be ascribed to loss or redistribution of extractives on a microscopic, if not macroscopic, scale. The random variations are less easy to explain. Perhaps the most satisfactory hypothesis is that in view of the very small volumes of liquid which are involved, this technique may be susceptible to interference from relatively small quantities of surface debris. Indeed, the tracheid lumena themselves may not be free of matter which may on one occasion partially occlude a flow path while on another occasion may lodge in a less critical location or orientation.

Unsteady-state flow of light organic solvent wood preservatives into refractory softwood

by

Laurence David Andrew Saunders B.Sc., M.Sc.

continues in

Volume 2

Unsteady-state flow of light organic solvent wood preservatives into refractory softwood

A thesis presented in part
fulfilment of the requirements for the
degree of Doctor of Philosophy
of the
University of London

by

Laurence David Andrew Saunders B.Sc., M.Sc.

Volume 2, of 2

May 1987

Errata

Throughout this thesis, the expressions 'gradient', pressure gradient' and 'pressure gradients' should respectively read 'differential', 'pressure differential' and 'pressure differentials'.

Unsteady-state flow of light organic solvent wood preservatives into refractory softwood.

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2.2.3 Measuring pressure inside timber

It will be recalled that in Section 2.1 it was concluded that it was not possible to devise a technique to follow directly the flow of white spirit-type solvents through timber.

As a result of the above conclusion the focus of this part (i.e. Section 2) of this investigation was directed towards measuring changes which could be assumed to be the result of liquid flow within timber. Three techniques were devised for this purpose. Two of these have been described in Sections 2.2.1 and 2.2.2 respectively and the third, namely - measuring pressure inside timber, is the subject of this section.

2.2.3.1 Measuring pressure inside timber - introduction

The theoretical basis on which this technique rests may be derived from Boyle's Law. This law, which deals with the behaviour of gases, states that, at constant temperature, the volume of a fixed mass of gas is inversely proportional to its pressure. Furthermore, the product of pressure times volume is constant for a given mass of gas. It follows, therefore, that reduction in pressure in a given volume of gas implies a reduction in the mass of gas present in that volume.

In the context of timber impregnation, the above relationships may be used in two ways. During the initial vacuum phase of a double vacuum schedule, a reduction in the pressure within the lumena in the timber may be interpreted as being indicative of a reduction in the mass of gas therein. During the immersion phase, on the other hand, when the mass of gas in the timber is substantially fixed, changes in the pressure of that gas indicate changes in its volume. These changes may usually be taken to imply movement of liquid.

From the literature it appears there have not previously been any extensive studies of the pressure fluctuations which occur within timber during impregnation, although in 1970, Arganbright & Resch reported a technique for measuring such fluctuations. Resch (1967) had used this technique to measure the movement of gas but not of liquid. Stamm (1973) assumed the continuing existence of a vacuum in white oak heartwood for up to 17 days after impregnation but accepts that his assumption was based on indirect evidence. (A.J. Stamm - personal communication.) Similarly, although trade literature circulated by timber preservative supplying companies contains generalisations about pressure regimes within timber (e.g. Protim undated), there is little direct basis for such statements.

In the face of the absence of information about the pressure changes which occur within timber as impregnation proceeds, the potential value of obtaining such information is considerable. For example, measurement of the time taken for the pressure in the central

lumen of a timber sample to equilibrate with the pressure in the treatment-vessel during the initial vacuum phase may not only yield empirical indications as to the optimum length of that phase, but it may also substantiate or repudiate such conclusions as that of Petty (1967) that air removal from spruce proceeds fairly rapidly. In addition, pressure changes within a timber sample which occur during the immersion phase of an impregnation cycle can be interpreted as indications of liquid movement. Once again, such results may be used both to estimate the optimum length of the immersion phase and also to examine whether differences in the flow rates of air and white spirit correspond to the differences in their viscosities, for example.

A method was therefore devised in order to monitor the pressure fluctuations within timber, such as might occur during the period of an industrial impregnation schedule.

2.2.3.2 Measuring pressure inside timber - development and use of the technique

2.2.3.2.1 Initial trials

Initially, trials were conducted using the pressure transducer already described in Section 2.2.1.2.1. This was connected to a hole bored into a sample of timber. The connection was made using a 0.375" (outside diameter) flexible nylon tube which passed through the side of the sealed treating-vessel. This sort of

arrangement had reportedly been used successfully by Vermaas (1978) but it quickly became apparent that the volume of air which was in the flexible tube attenuated to an unacceptable extent the pressure fluctuations which occurred within the timber. It became clear, therefore, that the volume of air between the diaphragm of the pressure sensor and the lumena within the timber, had to be kept to a minimum. A single attempt was made to effect this reduction in air-volume by filling the flexible tube with mercury. This procedure was, however, so comprehensively fraught with problems that it was not pursued.

Experience with these initial trials led to the conclusion that the measurement of pressure variations within timber could best be accomplished using a small pressure sensor embedded in the timber. Accordingly, this approach was adopted.

2.2.3.2.2 Selection and development of a suitable pressure sensor

On the basis of the experience gained during the initial trials described above, it was concluded that the overriding requirement for a sensor with which to measure pressure changes within timber samples was that it should be small enough to implant into the timber. It followed from this that the sensor must also be able to continue to function during submersion in white spirit-borne wood preservatives. An investigation to locate a suitable pressure sensor was thus undertaken. From this investigation it became clear that although the requirement for a small, implantable,

pressure sensor could fairly readily be satisfied, the design of all small pressure sensors, except one, was inappropriate for submersion in liquid for the following reason.

All the pressure sensors which were found were strain-gauge transducers which functioned on the same principle as that already outlined in Section 2.2.1.2.1. In order to minimise their temperature sensitivity, these units are constructed such that the air 'behind' the pressure-sensitive diaphragm does not have a fixed mass but is continuous with the surrounding atmosphere. Thus the diaphragm registers differences between the pressure in front of it and atmospheric pressure behind it; transducers designed in this way are termed 'differential'. To ensure that satisfactory equilibration of pressure occurs between the inside of the transducer body and the surrounding atmosphere, a so-called 'pressure-reference-tube' protrudes from the back of most small transducers. During impregnation tests this pressure reference tube would obviously have allowed liquid into the inside of the transducer during the immersion phase and this would often have led to malfunction.

One manufacturer, Gaeltec Limited, had apparently overcome the problem of liquid entering the pressure reference tubes of submerged transducers. They had done this by replacing the conventional rigid pressure reference tube with a much longer flexible one which could be passed out of the treating vessel. To obviate the need for two entry points in the wall of the

treating vessel the electrical connections to the Wheatstone Bridge were routed through the centre of the pressure reference tube. This arrangement appeared quite suitable for the purpose in hand and so Gaeltec transducers were selected for use for this section of work.

The particular Gaeltec transducers used were based on their '3EA/a' range which were built within stainless steel tubes which had an external diameter of 3.3 mm, and could have virtually any convenient length. The pressure-sensitive diaphragm was mounted across one end of the tube and had a silicon monoxide strain-gauge vacuum-deposited onto its 'back' surface. This comprised one 'side' of a standard Wheatstone Bridge.

2.2.3.2.3 Modification to the transducer

The transducer design outlined above had to be modified somewhat in order to overcome unforeseen problems in achieving a gas and liquid-tight seal between the pressure reference tube and the body of the transducer. These arose because, in response to the requirement that the system should function in white spirit, and as many other solvents as possible, PTFE was chosen as the material from which the pressure reference tube was made. It thus became necessary to effect a gas-tight and solvent-resistant seal between the stainless-steel transducer body and the PTFE pressure reference tube. Although this problem did not, at the outset, appear insurmountable, after about 18 months the attempt to construct a white

spirit-submersible, differential transducer was abandoned.

Being unsuccessful in constructing a satisfactory differential transducer, it was decided instead to dispense with the pressure reference tube and seal the back of the transducer with a fused metal/glass seal. This of course resulted in the mass of gas behind the pressure-sensitive diaphragm (i.e. within the transducer body) being fixed but in practice the resultant loss of temperature stability was not significant.

Electrical connection with the Wheatstone Bridge within these sealed transducers was effected by means of electrodes which passed through and were fused with the glass sealing bead. To the protruding ends of these electrodes were soldered PVC sheathed cables which connected, ultimately, to signal-conditioning and recording apparatus, similar to that already described in Section 2.2.1.2.1. Several pen recorders, other than the Perkin Elmer 56, were used from time to time. These included Servoscribe, Varian hot-pen and Phillips recorders, all of which accepted input signals in continuously variable overlapping ranges up to 100 mv and had chart speeds of 0.5 to 50 cm/minute.

2.2.3.2.4 Implanting the transducers in timber samples

General considerations

When this work was first planned it was envisaged that the transducers would be totally implanted into timber samples. However, this introduced several problems in connection with reliably retrieving the transducer undamaged after each experiment. Overcoming these problems introduced an unacceptable degree of complexity into the transducer's design. Consequently, the approach adopted involved a 'semi-implantable' transducer which was 2 - 4 cm long. The pressure-sensitive end of such a transducer could be inserted into a hole pre-drilled in the timber, leaving a portion protruding from the timber so that it could be gripped to effect removal.

In mounting the transducer it was essential to ensure a gas-tight seal between the timber into which the transducer was inserted and the sides of the transducer's stainless-steel casing. At the same time the mounting system had to allow for easy removal of the transducer after use. A variety of fairly robust 'housings', into which the rather less robust transducer could be inserted, were considered. All of these tended to increase the volume of the pocket of air at the sensitive tip of the transducer and, largely because of this, it was considered preferable to dispense with housings altogether and simply seal the transducer directly into the timber. This introduced the need to grip the protruding portion of the transducer very firmly in order to effect removal

from the timber. To eliminate the risk of damage occurring during this operation, the 'back' end of the transducer was reinforced with a metal collar.

Cyanoacrylate adhesive was selected for the purpose of sealing the transducer into the timber. The principal reasons for this choice were, the stability of this adhesive in a wide range of solvents, its fast cure time, its compatibility with both stainless-steel and timber, and its relatively low shear strength. This last property was considered important because, as a result of it, a simple twist action disrupted the seal and facilitated removal of the transducer.

For much of the experimentation described the cyanoacrylate adhesive 'Loctite I.S. 415' was used. For later experiments 'superglue', now more readily available, has been used with satisfactory results.

Implantation procedure

Of the several implantation procedures used from time to time, the following proved the most satisfactory. A 3.0 mm hole was drilled to the required depth using a standard 'Dormer' high speed twist drill or its equivalent. The hole was then expanded to 3.3 mm diameter using a length of stainless-steel tube, similar to that from which the transducer body was constructed.

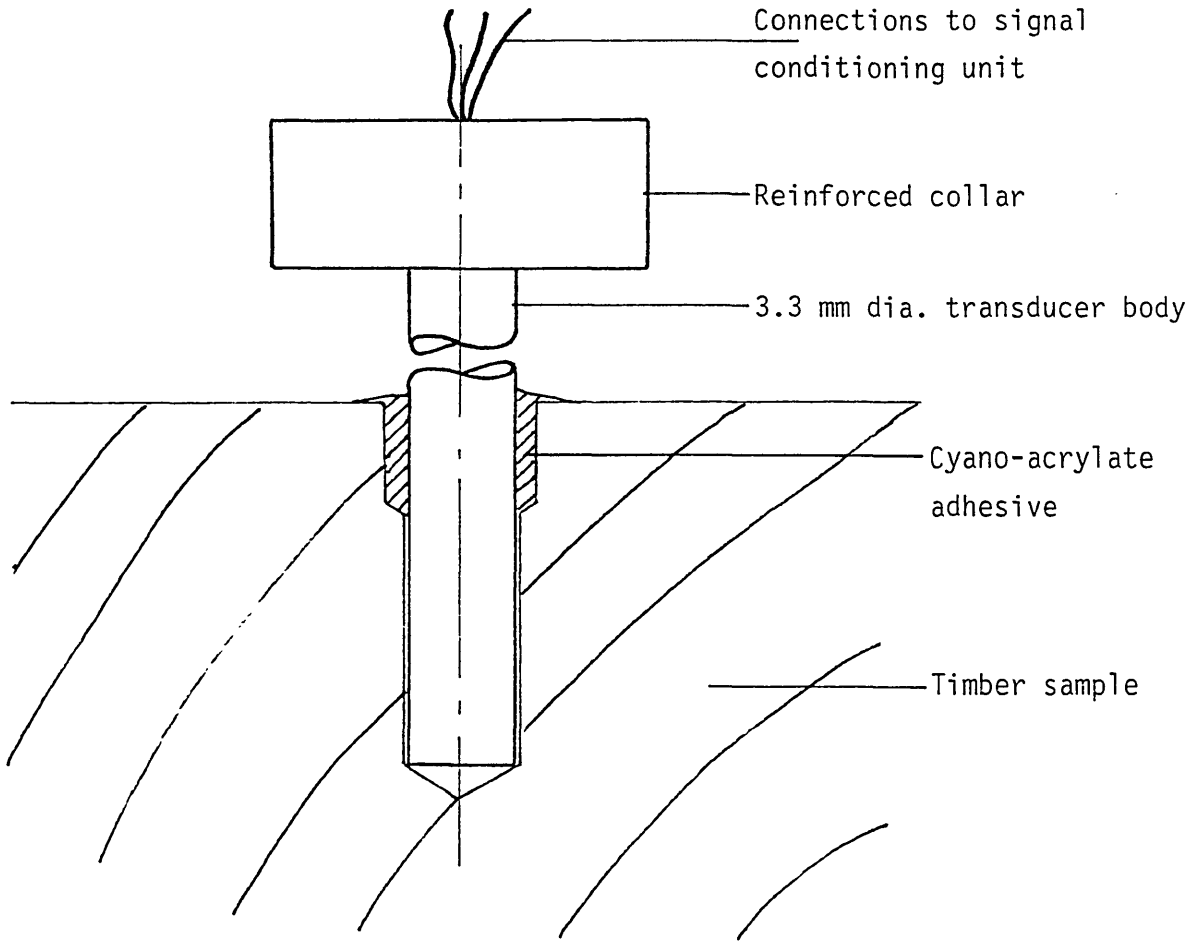
The outer few millimetres of the hole were then further expanded to ca. 5 mm diameter, taking care to remove wood shavings from the blind end of the hole.

After the mounting hole had been prepared in this way, the transducer was gently pushed into place. It was found useful to have the transducer 'on' during this operation in order to gain the earliest possible indication of the delicate transducer diaphragm meeting any potentially damaging obstruction. (As a further precaution against damage, the diaphragm was recessed, during manufacture, 0.5 mm into the transducer casing.)

During transducer insertion the pocket of air between the transducer diaphragm and the blind end of the hole became compressed and thus pressurised. In seasoned timber this pressure could be relieved by slight lateral pressure on the transducer body. During work with green timber the pressure could not be relieved in this way and it was concluded that the mounting system described here was not suitable for use with unseasoned timber.

When the transducer was inserted to the required depth, its protruding portion and the timber surface in its near vicinity were sprayed with an activator (Loctite Activator AC) to make them more compatible with the cyanoacrylate adhesive which was then applied to the region of the timber surface/transducer body junction (Fig 2.21).

Figure 2.21 Schematic sectional diagram to show a transducer mounted in a sample of timber, sealed into place with cyano-acrylate adhesive



2.2.3.3 Measuring pressure inside timber - experimentation undertaken

During the execution of this work, several attempts were made to conduct co-ordinated logical series of experimentation on groups of matched samples. In most instances these attempts proved abortive, either by reason of technical and instrumentation problems which beset this section of the investigation, or else in consequence of unsound experimental design which became apparent as initial results became available.

In consequence of this, the experimentation described in this section comprises several comparatively independent items of work, the results of which are illustrative of the phenomena which were observed. This work may conveniently be described under the five headings which are enumerated below. The first two of these five concern the movement of gas (i.e. air) through timber. In the remaining three sections the movement of liquid is the aspect of principal interest.

(i) Movement of air laterally through seasoned spruce (Section 2.2.3.3.1). As a starting-point, the movement of air laterally through a small sample of end-sealed seasoned spruce was examined.

(ii) Movement of air longitudinally through seasoned spruce (Section 2.2.3.3.2). This piece of work was undertaken in order to examine the flow of air longitudinally in seasoned spruce. This was done with a view to examining the pressure gradient which occurs during unsteady-state flow, and from this to gain, by implication, information about the structure of timber through which flow was occurring.

(iii) Movement of air and white spirit laterally through seasoned spruce (Section 2.2.3.3.3). As a means of obtaining a comparison of the rates of unsteady-state flow of air and white spirit through seasoned softwood, these two fluids were caused to flow through a small end-sealed sample of seasoned spruce.

(iv) Pressure changes in seasoned spruce during a series of DVP schedules (Section 2.2.3.3.4). Section 3.2.1.2 (q.v.) involved impregnating samples of air-dry spruce using a range of experimental DVP schedules, several of which involved variations in the pressure applied during the immersion phase. A series of measurements of the pressures within the samples was made in conjunction with this work.

(v) Post-immersion pressure changes (Section 2.2.3.3.5). Two experiments were undertaken in which monitoring of internal pressure continued after the immersion phase was completed.

2.2.3.3.1 Movement of air laterally through seasoned spruce

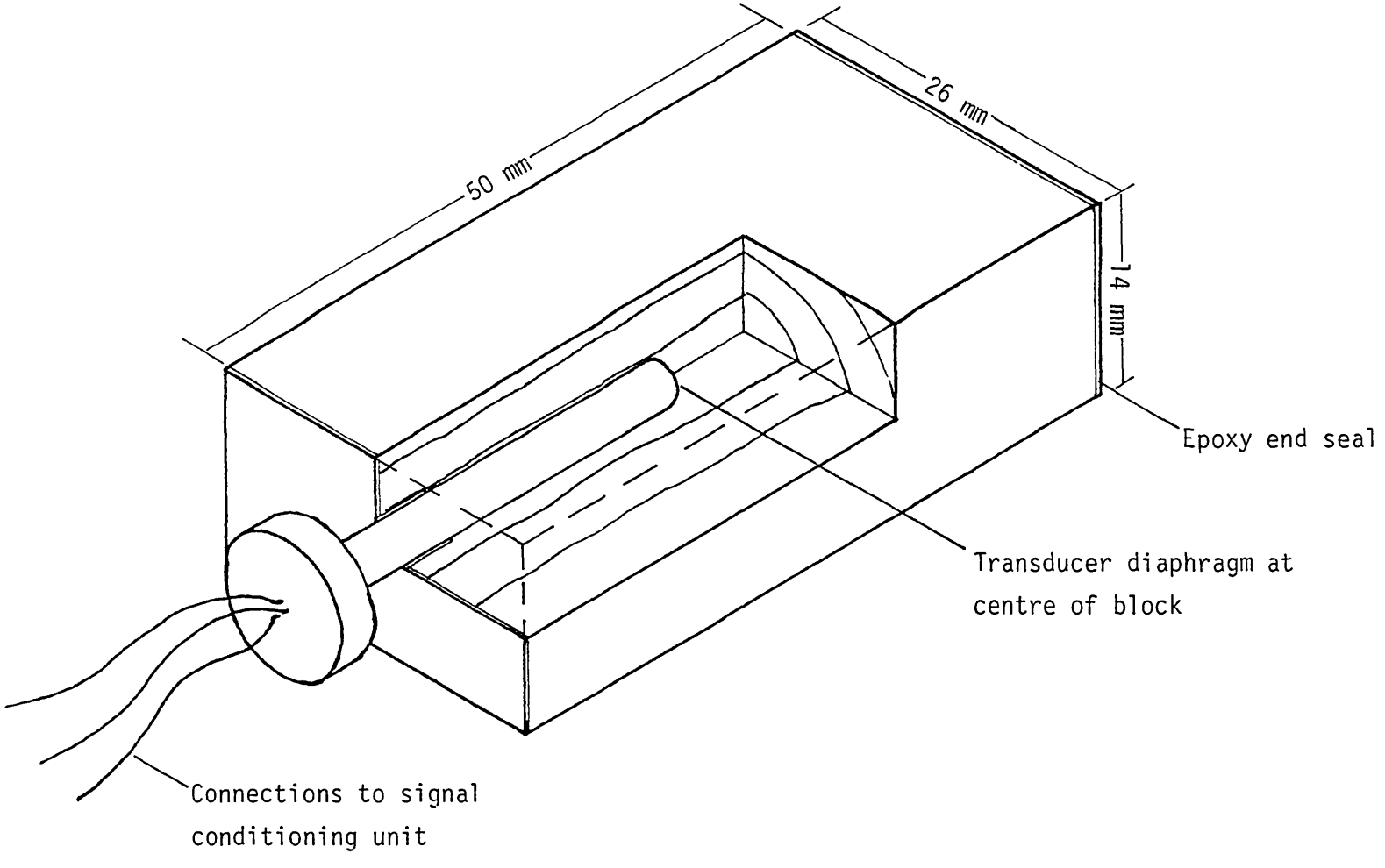
The aim of this experiment was to examine the lateral movement of air out-of and back into a small sample of seasoned spruce.

Timber sample

A length of straight grained, knot free seasoned Finnish spruce which had a moisture content of ca. 12% m/m was used to prepare a test sample measuring 50 x 26 x 14 mm (Fig 2.22). The 26 x 14 mm transverse surfaces of this sample were then sealed with epoxy resin (see Section 2.2.1.2.3) to eliminate longitudinal flow which, because of its speed, would be rather difficult to analyse in such a small sample.

To prepare this sample to receive the transducer, a 3.0 mm diameter hole was drilled through the cured epoxy resin, into the centre of one of the transverse faces. This hole was drilled parallel with the longitudinal axes of the axial tracheids, and penetrated 25 mm into the block so that it terminated at the block's geometric centre. This hole was then enlarged slightly to 3.3 mm diameter

Figure 2.22 Cut-away diagram to show transducer mounted in spruce block for experimentation described in Sections 2.2.3.3.1 and 2.2.3.3.3



all along its length and finally counter-drilled in the manner already described (Section 2.2.3.2.4).

Procedure

Linearity and calibration checks were performed on the transducer which was then inserted and sealed with cyanoacrylate adhesive into the block described above. The whole assembly was enclosed within a small chamber (Plate 2.3) which was then evacuated to -93 kPa. This level of evacuation was maintained until the pressure registered by the transducer had stabilised, following which the chamber was vented to atmosphere. Pressure monitoring was continued until atmospheric pressure was re-established at the transducer's tip. Following this, the chamber was re-evacuated to -93 kPa and the whole sequence of operations was repeated.

The results yielded by this work are presented and discussed in Section 2.2.3.4.1.

2.2.3.3.2 Movement of air longitudinally through seasoned spruce

The results of the experimentation described in Section 2.2.3.3.1 presented a rather complex pattern to analyse because air could flow towards and away from the transducer in several directions. In addition, pressure readings taken at only one point within a sample yield information about the pressure changes which occur at that point but do not yield information about the shape of the

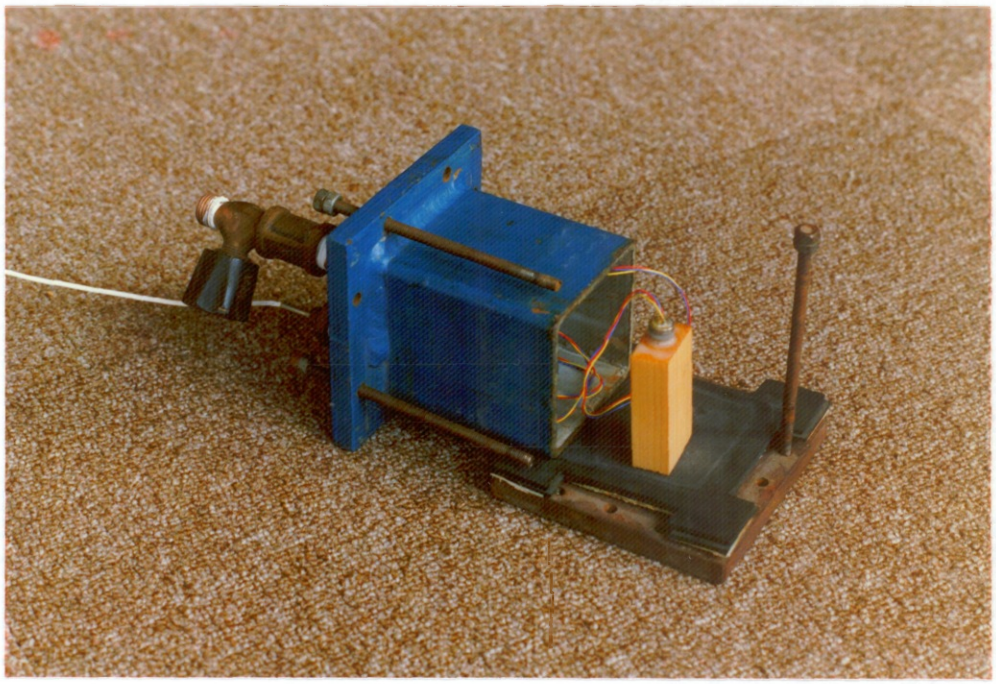


Plate 2.3 Transducer mounted in timber sample represented in Figure 2.22, together with impregnation chamber used for the work reported in Section 2.2.3.3.1

pressure gradients within the rest of the sample. Information on this latter point might enhance understanding of the structure and continuity of flow pathways.

In an attempt to overcome both these difficulties, an experiment was devised in which pressure was monitored at intervals along a 20 cm, longitudinally-orientated sample of spruce.

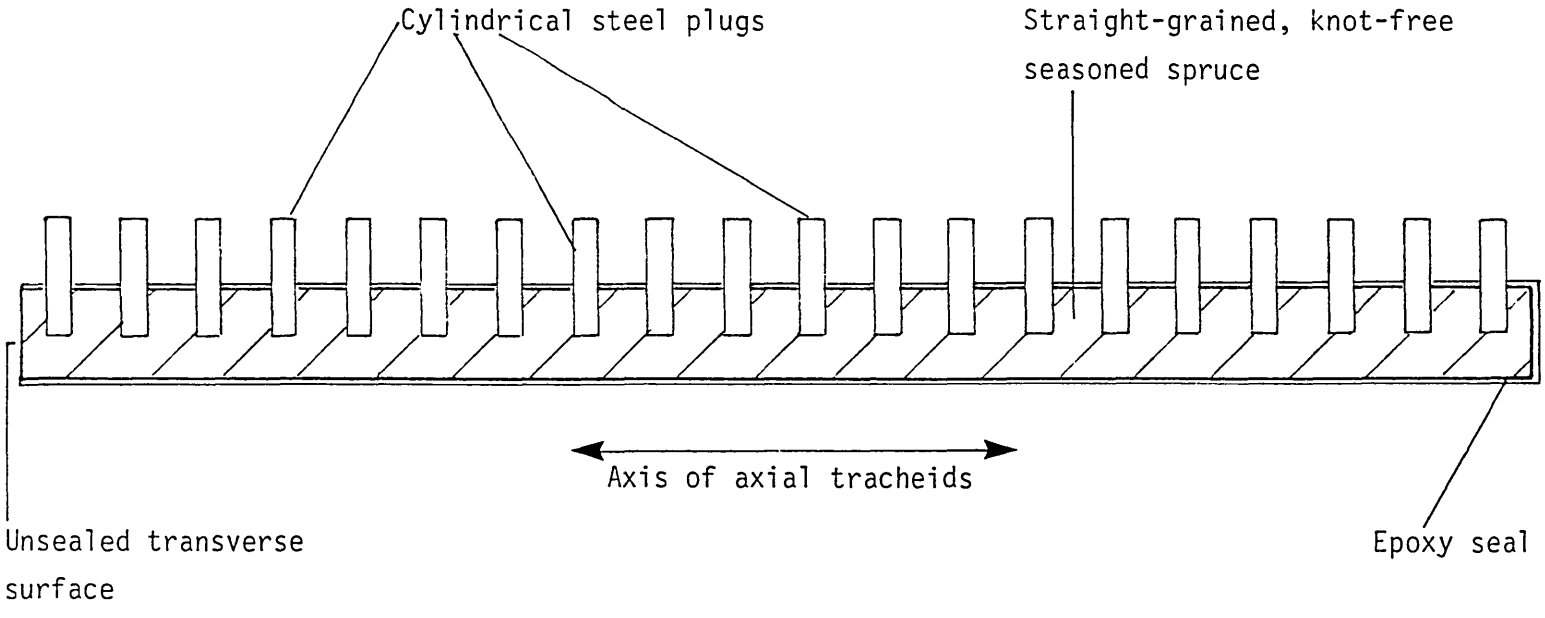
Timber sample

From a straight-grained, knot free length of seasoned spruce was cut a test sample which measured 1.2 cm x 1.2 cm in cross-section and 20 cm in length. This was sealed with epoxy resin on all four of its 1.2 cm x 20 cm lateral surfaces and across one of its 1.2 cm x 1.2 cm transverse surfaces (i.e. on five of its six faces in all). After the epoxy resin had cured, twenty, 3.0 mm diameter holes were drilled into one tangential surface of the sample. These were positioned 0.5 cm from each transverse surface, and at 1.0 cm centres. Each hole was drilled ca. 0.6 cm deep. Into each hole a ca. 1.5 cm long, 3.3 mm diameter metal plug was inserted and sealed (Fig 2.23).

Procedure

Pressure inside the timber sample described above was in fact measured at just four distances from the open end, namely 5.5, 10.5, 18.5 and 19.5 cm from the unsealed transverse surface.

Figure 2.23 Sectional diagram illustrating the arrangement of twenty 3.3 mm diameter steel plugs mounted in a 20 cm long, 1.2 cm section piece of seasoned European spruce used in the experimentation described in Section 2.2.3.3.2. One plug at a time was removed and replaced by a pressure transducer.



One plug at a time was removed and a calibration check was performed on the transducer which was then inserted and sealed into the hole previously vacated by the removal of its metal plug. The whole sample - and - transducer assembly was then enclosed within a chamber which was evacuated to ca. -81 kPa. This level of evacuation was maintained for a period, during which the pressure change within the sample was recorded. At the end of the observation period the chamber was vented to atmosphere. After measurement at each location had been completed the transducer was removed from the sample and the hole from which it had been removed was re-sealed with a metal plug.

The results yielded by this work are presented and discussed in Section 2.2.3.4.2.

2.2.3.3.3 Movement of air and white spirit laterally through seasoned spruce

The results of the work described in both Sections 2.2.3.3.1 and 2.2.3.3.2 indicated that the pressure within samples of spruce could fairly readily be reduced by enclosing those samples within an evacuated chamber. The inference drawn from this observation is that air can fairly readily be moved through spruce. It is common experience however that, during industrial timber impregnation, white spirit cannot always be made to penetrate spruce at all deeply, i.e. white spirit cannot always readily be moved through spruce.

From the above, many questions arose concerning the relationship between a timber sample's permeability to air and its permeability to white spirit. Accordingly the present experiment was undertaken to investigate this aspect.

Timber sample

The sample of spruce used was the same sample as was used for the work outlined in Section 2.2.3.3.1, and therefore it has already been described in that section. The transducer was not withdrawn from the block between the completion of the work described in Section 2.2.3.3.1, and the present experiment.

Procedure

With the calibrated transducer sealed into the timber sample, the sample was enclosed within a chamber which was partially evacuated to -93 kPa for ten minutes. The selection of a ten minute evacuation period was a fairly arbitrary decision based largely on the fact that this is the evacuation period of the standard 'one-bar' DVP schedule (Section 1.5.3.6).

At the end of the ten minute evacuation period, white spirit was admitted to the chamber around the timber sample. When the chamber had filled, a pressure of 103 kPa was applied to the treating liquid for a period slightly in excess of sixty minutes. As with the initial vacuum, the magnitude and duration of the

pressure applied during the immersion phase were selected on the basis of the industrial DVP schedule most often used for the impregnation of seasoned spruce with organic solvent preservative. After the treating-chamber had been drained at the end of the immersion phase, a 20 minute final vacuum of -93 kPa was applied. Following this the sample was removed from the treating-chamber and stood on the laboratory bench and monitoring of the pressure within the timber was continued for a period of 150 hours after the end of the final vacuum phase.

The results obtained from this section of work are presented and discussed in Section 2.2.3.4.3.

2.2.3.3.4 Pressure changes in seasoned spruce during a series of DVP schedules

From the results obtained from the work described in the last section (2.2.3.3.3) it became clear that even if a sample of spruce is sufficiently permeable for the air within it to be withdrawn fairly easily, the volume of white spirit which subsequently moves into the sample when evacuation is followed by immersion at 103 kPa, may be quite small. However, it may be recalled that results presented in Section 2.2.2.3.2.1 suggested that an increase in the pressure applied during the immersion phase may cause a directly proportional increase in the volume of liquid taken in by the timber. Therefore, the present experimentation was undertaken to examine the effect on pressure within the sample caused by altering the

pressure of the surrounding liquid during the immersion phase.

The experimentation described in this section was performed using samples which were withdrawn from an impregnation-experiment (Section 3.2.1.2). This impregnation-experiment was undertaken in order to measure the gross effects of changes in immersion-pressure on the uptake and penetration of an organic solvent wood preservative. The reason for using samples withdrawn from the impregnation-experiment for the present work was in order to allow comparison of the data generated during the two experiments.

Timber samples

The preparation of samples for the impregnation-experiment is described in detail in Section 3.2.1.2 and it is sufficient here to note the following features.

Samples were prepared by resawing lengths of air-dry Finnish white-wood, approximately 100 x 35 mm in cross-section, into four 22 x 35 mm sticks. Each stick was cut into consecutively-numbered, 66 mm long samples which were each sealed across their end grain surfaces with epoxy resin. From a single one of these sticks were selected all the samples for the pressure measurement experiment reported in this section. In reporting this experiment, the samples' numbers have been retained in order to indicate the positional relationship of the samples in the original beam.

Procedure

Into each test block a hole to receive the transducer was drilled to a depth of 11 mm at the centre of one of the 66 x 35 mm surfaces. The calibrated transducer was sealed into the hole in each block in turn, and the block with the transducer in it was enclosed within a chamber and impregnated with preservative. The preservative used was the commercial organic solvent formulation 'Protim 80' to which 0.5% m/m Waxolene Blue A had been added as a tracer dye. Each treatment consisted of an initial vacuum of -85 kPa for 10 minutes, followed by immersion-under-pressure in preservative for a period of 60 minutes. Following the immersion period the preservative was drained from the treatment vessel and a final vacuum of -85 kPa was applied for 20 minutes. The different pressures used during the immersion phases are presented in Table 2.14.

Table 2.14 Pressures used to apply the organic solvent preservative 'Protim 80' to 66 x 35 x 22 mm end-sealed blocks of Finnish spruce. Duration of immersion-under-pressure phase was 60 minutes in all cases. Initial and final vacuums were always -85 kPa and lasted 10 and 20 minutes respectively.

Sample No.	Immersion Pressure - kPa
2	103
3	827
9	103
10	413
16	620
17	103
27	1240
28	103
31	1033
32	103

The data derived from this section of the work are presented and discussed in Section 2.2.3.4.4.

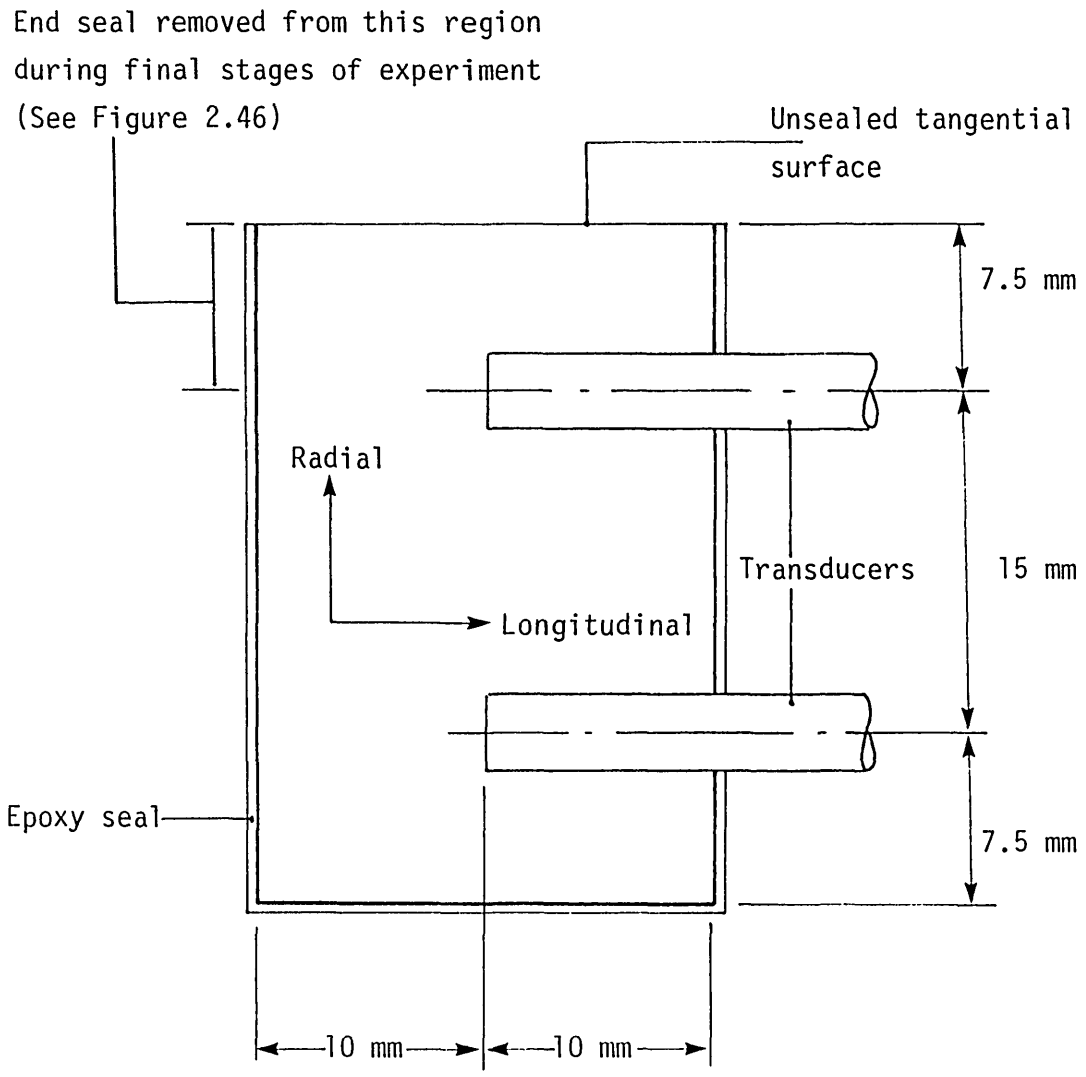
2.2.3.3.5 Post-immersion pressure changes

The results obtained from the work described in Sections 2.2.3.3.3 and 2.2.3.3.4 included limited information about the pressure changes which occurred within the timber samples after the immersion phase was completed. In order to extend this information, two further experiments were set up, each of which was designed principally to measure post-immersion pressure changes.

The first of these two experiments involved a sample (Sample I) of air-dry spruce which measured 15 x 25 x 50 mm and which was sealed across both its end-grain surfaces with epoxy resin (Section 2.2.1.2.3). A transducer was mounted in this sample in order to measure pressure changes which occurred at its geometric centre. The sample-and-transducer assembly was sealed into a treating vessel which was evacuated to -81 kPa for 9 minutes. After $6\frac{1}{2}$ minutes the vessel was flooded with white spirit and after 9 minutes it was vented to atmosphere. After 20 minutes immersion the vessel was drained of liquid and the pressure within the sample was continuously monitored for a further 60 minutes.

The second of the two experiments which was designed to examine post-immersion pressure changes also involved a sample of air-dry spruce. This sample (Sample II) measured 10 x 20 x 30 mm and was sealed on five of its six surfaces (Fig 2.24) with epoxy resin (Section 2.2.1.2.3). Egress of air and ingress of liquid and subsequently of air were thus restricted to the outer tangential

Figure 2.24 Sectional diagram illustrating the arrangement of two transducers in a sample of air dry European spruce - Sample II of Section 2.2.3.3.5



face. Consequently, pressure changes within the sample could occur only as a result of radial flow. It may also be seen from Figure 2.24 that in this second experiment pressure was measured at not one but two locations within the sample.

Sample II, with its two transducers mounted in it, was sealed within a treating vessel which was evacuated to -93 kPa and this level of evacuation was maintained until the pressures at both locations within the sample had restabilised. The treating vessel was then flooded with liquid. During most of the ensuing 122 hour immersion period the vessel was vented to atmosphere. However, for the short periods indicated in Figure 2.46 , Section 2.2.3.4.5, the vessel was sealed and pressures of up to 207 kPa were applied. After ca. 122 hours the vessel was drained of liquid and thereafter the pressure was monitored at intervals over an 11 week period. At the end of this time, 1 mm increments of epoxy seal were removed at intervals from one transverse surface, adjacent to the unsealed tangential surface (Fig 2.24) until a pressure change within the sample was observed. Pressure monitoring was discontinued ca. $7\frac{1}{2}$ months after the start of the experiment.

2.2.3.4 Measuring pressure inside timber - results and discussion 1

This section (2.2.3.4) presents and discusses the results obtained from the experimentation described in Section 2.2.3.3. Like Section 2.2.3.3, this section is subdivided into five parts, each of which is concerned with one of the items of experimentation described in Section 2.2.3.3.

2.2.3.4.1 Movement of air laterally through seasoned spruce

The experimentation described in Section 2.2.3.3.1 yielded results which are presented in Figures 2.25 and 2.26. In the first of these (Fig 2.25) the pen recorder traces, for the four periods of pressure change within the timber, are represented. In Figure 2.26 the same traces are presented in a form which facilitates comparison of the patterns of pressure change observed during evacuation, and the patterns observed during the periods when the evacuated samples were allowed to re-equilibrate with atmospheric pressure.

Graph A of Figure 2.25 exemplifies the pattern which was observed during all the four periods of pressure change. Initially, when the pressure in the chamber within which the block was sealed was changed, there was a period of 5 - 10 seconds during which the pressure in the centre of the test block remained unchanged. Following this the pressure within the sample came into equilibrium with the pressure surrounding it. This equalisation proceeded most rapidly at first when the pressure gradient between the centre

Figure 2.25 Pressure changes recorded in spruce sample represented in Figure 2.22 (Section 2.2.3.3.1)
(0 kPa = Atmospheric pressure)

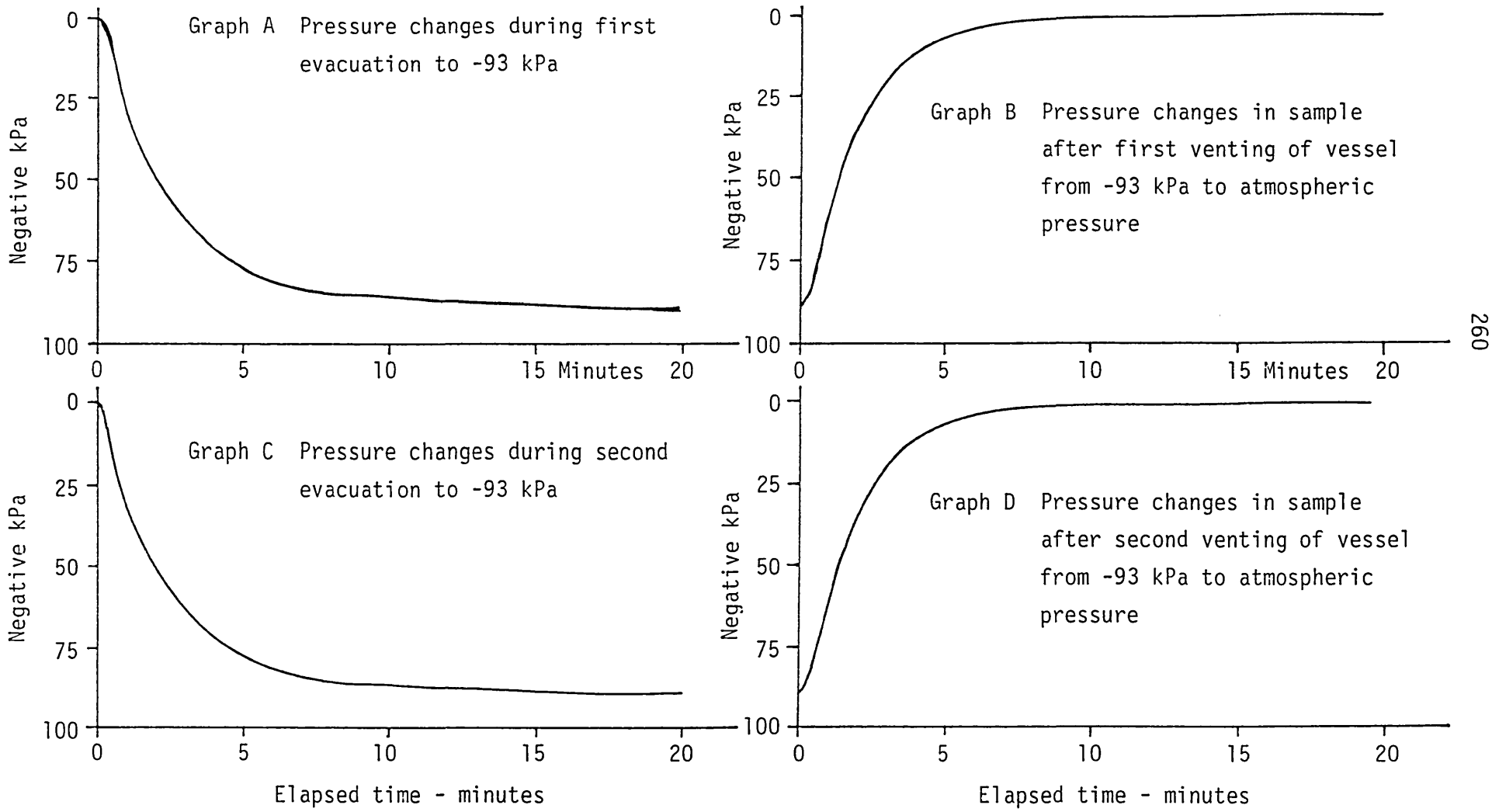
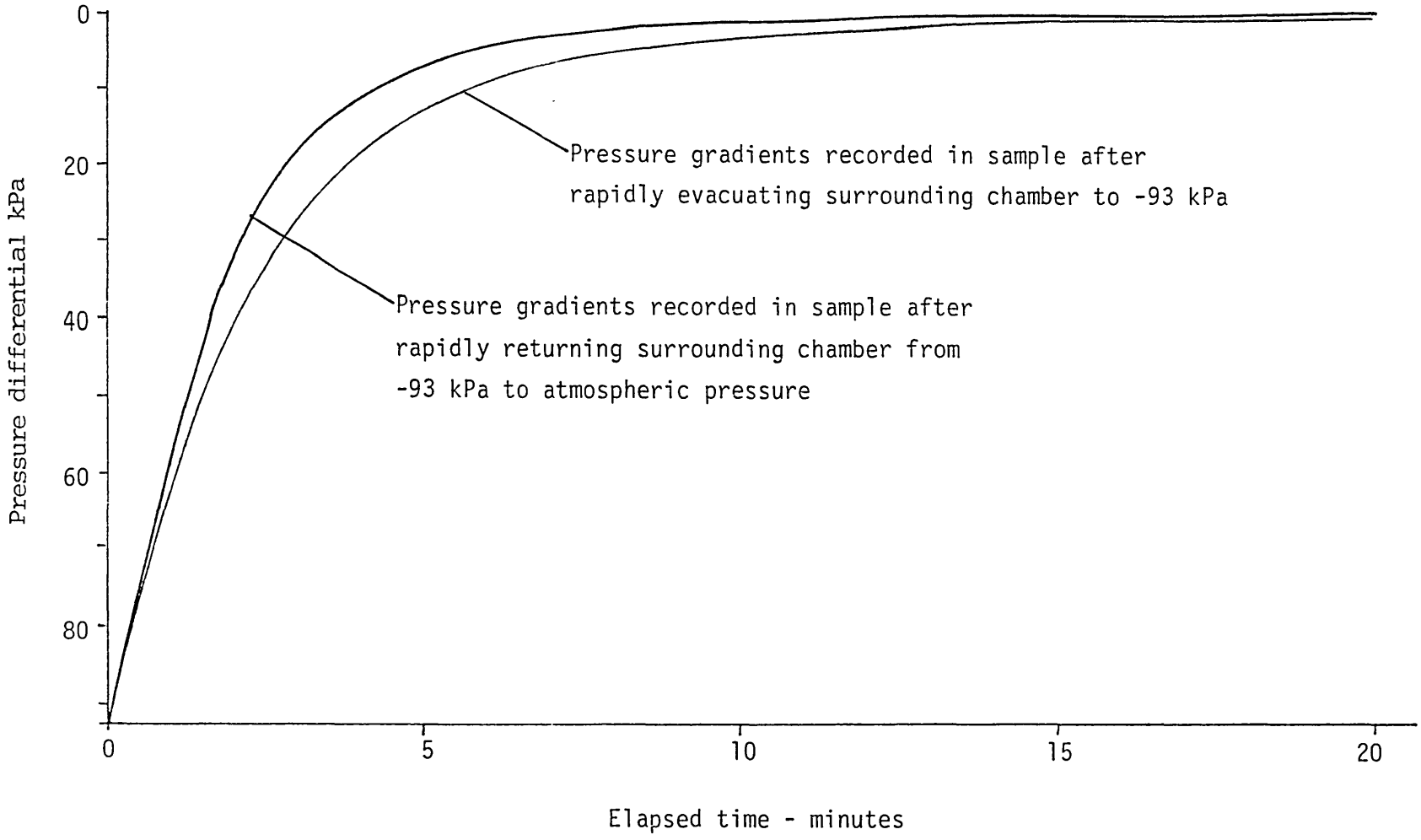


Figure 2.26 Data used to prepare Figure 2.25 replotted to facilitate comparison of pressure gradients between inside of spruce block (Figure 2.22) and its surroundings during evacuation to -93 kPa, and subsequent return to atmospheric pressure

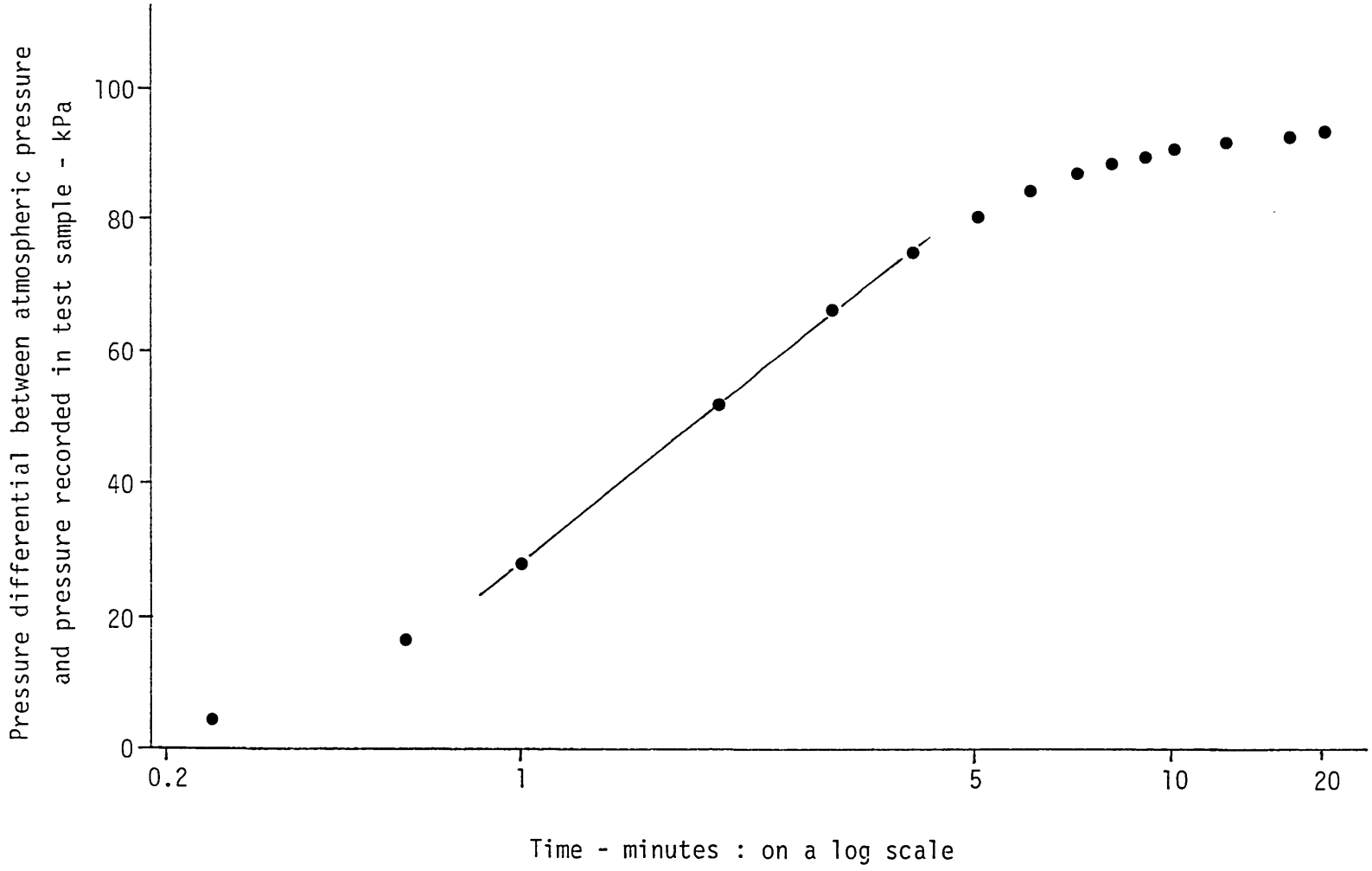


of the sample and the chamber was greatest. As this gradient diminished with time, so also did the rate of change of the pressure at the centre of the sample.

This general pattern of pressure change is much as might have been predicted by extrapolation from established knowledge of the behaviour of gases in permeable materials. It is known (Equation 1.16) that when a gas flows through a permeable porous material, the volume rate of flow (Q) is directly proportional to the pressure gradient (ΔP). At the outset, (time zero) the pressure gradient between the centre of the timber sample and its surroundings was ca. 93 kPa. After, for example, two minutes, this gradient had dropped to ca. 40 kPa. It is thus consistent with this to note that the rate of pressure change had also dropped.

Over the mid-part of the pressure range, a linear relationship may be derived from the results in Figure 2.25 if pressure in the centre of the block is plotted against log. time (Fig 2.27). This linearity continued until the gradient between the vessel and the inside of the sample was about 18 kPa. In other work on air removal from spruce, Beardsell (1981) measured the amount of air removed from spruce as evacuation proceeded and he noted a linear relationship between log. time and log. pressure over the mid-part of the experimental period, with deviation from this linearity occurring as total evacuation of the block was approached.

Figure 2.27 Data used to prepare Graph A of Figure 2.25 plotted to display relationship between pressure change and time on a log scale



The observed deviation from linearity may perhaps indicate that after the bulk of the air has been removed from a sample, there remain, within that sample, pockets of less-completely evacuated timber from which air flows more slowly.

Two further features of the results presented in Figures 2.25 and 2.26 merit comment at this juncture.

Firstly, it will be recalled that the level of evacuation to which the chamber containing the timber was reduced was -93 kPa, whereas the equilibrium pressure measured inside the timber in fact appeared to be ca. -90.6 kPa. In processing and discussing the results the assumption has been made that lateral deformation of the transducer's diaphragm during insertion into the timber, which almost invariably resulted in a zero-shift, also resulted in a slight calibration change in the transducer's output. In preparing Figures 2.26 and 2.27, appropriate adjustments to the results have been made on this basis. It must be said however that, when asked, the transducer's manufacturers did not necessarily agree that deformation of the kind described would result in this calibration change.

The above uncertainty, therefore, introduces the possibility that the equilibrium pressure within a permeable timber sample enclosed within a partially evacuated chamber, may not in fact be equal to the pressure of its surroundings. This seems unlikely and, therefore, for the present investigation the discrepancy is

ascribed to 'experimental error'. It is noted, however, that Petty and Beardsell (1981) reported that a small proportion of the air in spruce cannot be removed during evacuation. Petty postulated that this air was trapped in pockets. However, if the difference in pressure noted above is anything other than an artefact, the implication must be that air may be retained within permeable materials elsewhere than in isolated sealed pockets.

A further feature of the results which are presented in Figure 2.26 is the hysteresis between the plots of pressure against time recorded during evacuation and permeation of the timber sample. The full-scale instrument-response time for the transducer was well below one second and so it can be confidently stated that the observed hysteresis was not an artefact caused by a delay in the instrumentation registering a change in pressure.

The reason for this hysteresis is unclear. It is known that when a pressure gradient is established across a pit membrane, the torus may be displaced towards one of the pit aperture borders (Petty 1972, Gregory & Petty 1973, Bolton & Petty 1978a). However, the pressures required to promote such a movement in seasoned timber are fairly high and it appears that the pressure gradients which may be set up by movements of gas during evacuation are insufficient to induce any movement (J.A. Petty - personal communication).

On the assumption that seasoned timber is not physically altered by gas flowing through it, it was anticipated that in the present experiment the patterns of pressure change during evacuation and permeation would be mirror images of one another. Clearly this was not so. A clear hysteresis between evacuation and permeation occurred on both occasions. Interestingly, an analagous hysteresis was noted by Sebastian et al (1973). Observations such as these may indicate a hitherto undescribed asymmetry in the structure of timber. However, the present status of the knowledge of unsteady-state gas flow is not adequate to permit speculation as to the cause of the observed results.

In summary, the results obtained from the work described in Section 2.2.3.3.1 indicate that the rate of pressure-drop in the centre of a sample of seasoned spruce which is placed in a partially evacuated vessel decreases linearly with log. time during the early stages of evacuation, but deviates from linearity as evacuation nears completion. Although the results obtained were interpreted on the basis that the equilibrium pressure within a sample of timber which is enclosed in an evacuated vessel is equal to the pressure of its surroundings, an indication was noted that there may in fact be a small differential in these pressures. The mechanism which might lead to such a differential is however unclear, as indeed is the mechanism which caused the observed hysteresis between rates of pressure-change at the centre of the sample during evacuation and permeation.

2.2.3.4 Measuring pressure inside timber - results and discussion continued - 2

2.2.3.4.2 Movement of air longitudinally through seasoned spruce

The experimentation described in Section 2.2.3.3.2 yielded results which are presented in Figure 2.28. In view of the linear relationship revealed by plotting pressure against log. time (c.f. Section 2.2.3.4.1) these data were also plotted using a logarithmic time axis and the resultant graph is presented in Figure 2.29.

In interpreting these data, attention has been paid to general trends only. This particular piece of work was undertaken at an early stage of this investigation and certain technical details, particularly those relating to the measurement and regulation of the vacuum in the chamber around the timber, were not well developed.

Considering firstly the three recordings of pressure made respectively 5.5, 10.5 and 19.5 cm from the open end-grain of the sample, these results follow a pattern which was broadly predictable, particularly in the light of the results already discussed in Section 2.2.3.4.1. Pressure change at each monitoring point proceeded most rapidly at first and slowed as the magnitude of the gradient which was promoting flow gradually diminished. As previously, the plots of pressure against log. time (Fig 2.29)

Figure 2.28 Pressure changes monitored at four locations within a 20 x 1.2 x 1.2 cm length of spruce, which was sealed on all surfaces except one transverse end, during evacuation at ca. -81 kPa

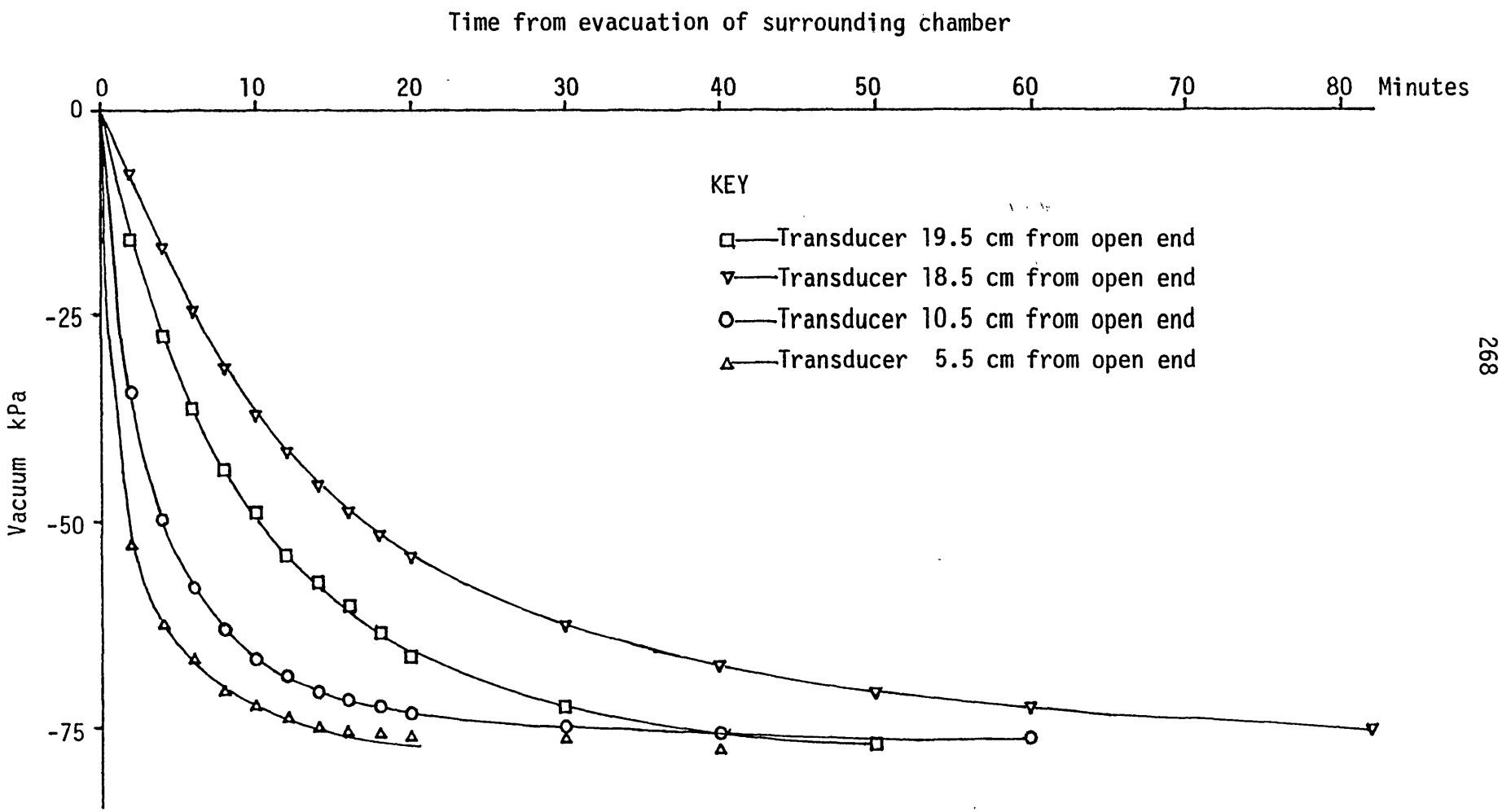
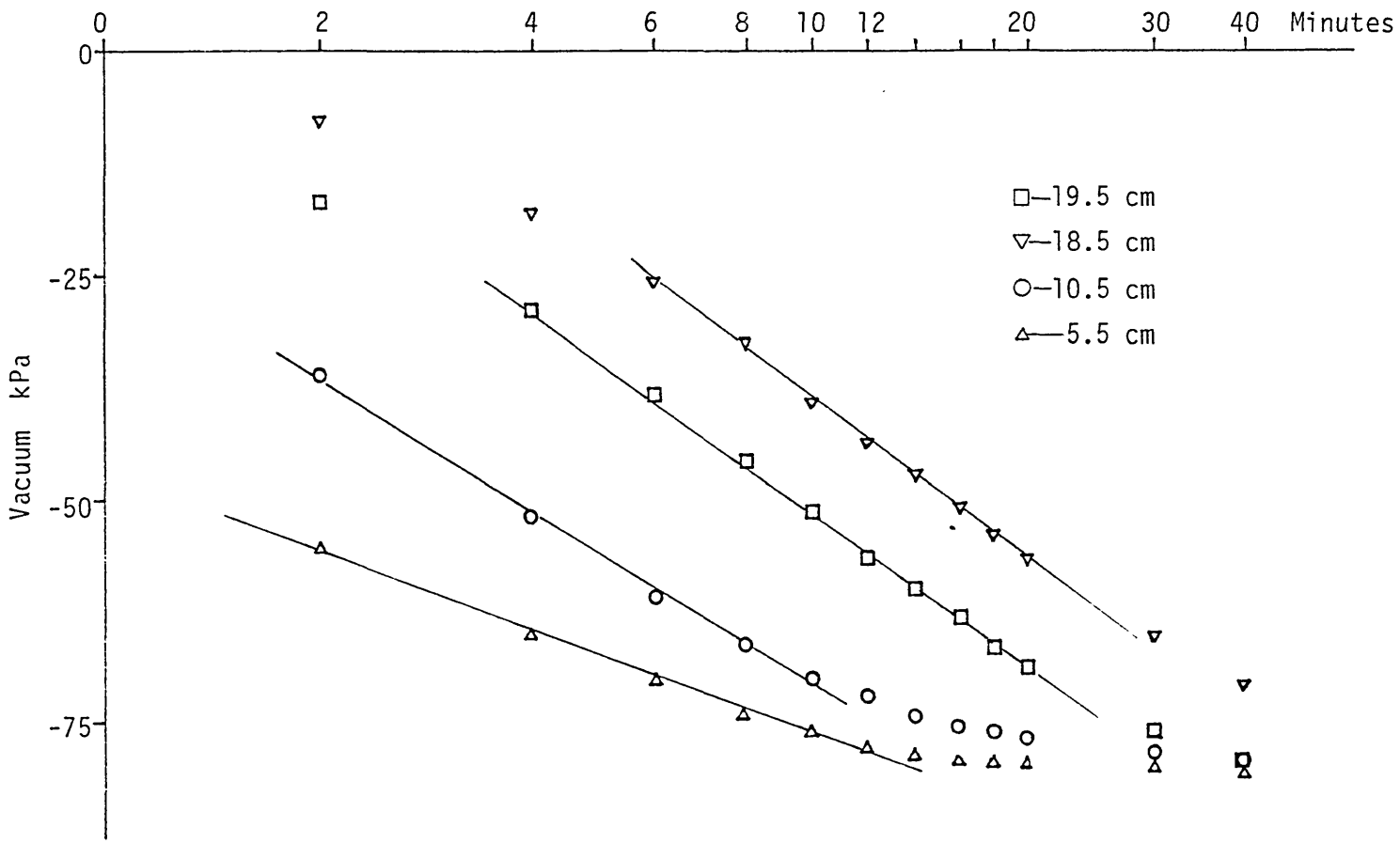


Figure 2.29 Data from Figure 2.28 plotted against time on a log scale

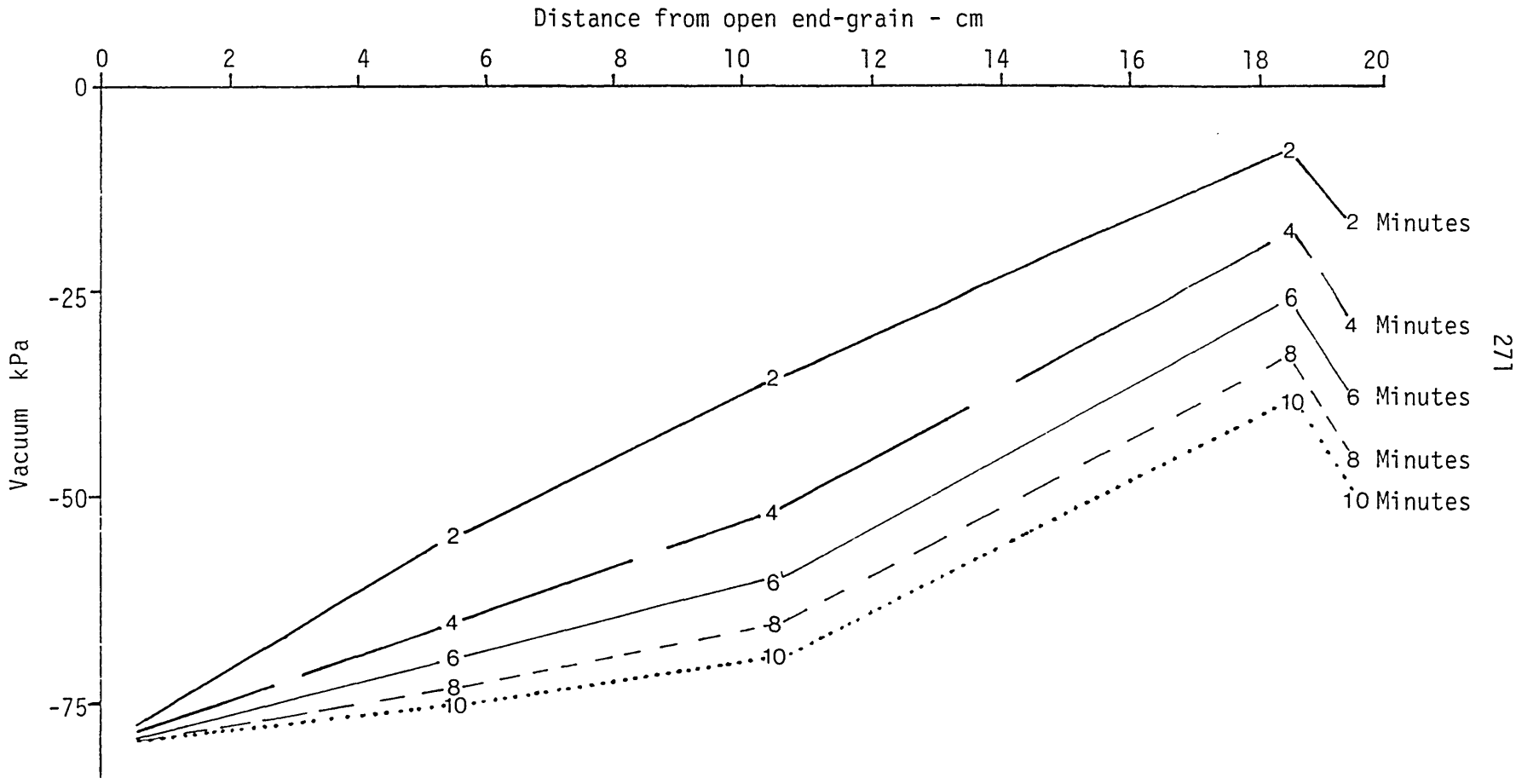


are, to a first approximation at least, linear over much of their length.

The most interesting feature of these results becomes apparent in Figure 2.30 in which the data are plotted to display the shape of the pressure profile within the sample at several intervals during the first ten minutes of evacuation. In preparing this Figure, the assumption was made that on each of the four occasions that the sample was evacuated, the sequence of pressure changes within it followed the same pattern. On this basis it is assumed that the results in Figure 2.30 are those which would have been generated had it been possible to take readings simultaneously from four transducers implanted into the sample at the positions indicated.

The particularly interesting and unexpected feature of these results is that Figure 2.30 does not portray a pattern in which the pressure differential between the inside of the sample and its surroundings increased steadily with increasing distance from the open end-grain surface. Instead, the pressure measured 18.5 cm from the open end-grain approached the pressure in the chamber more slowly than the pressure monitored 19.5 cm from the same end. Clearly, therefore, the 18.5 cm pressure monitoring point cannot have been in the same continuous flow path as the 19.5 cm point.

Figure 2.30 Pressure profiles within a 1.2 x 1.2 x 20 cm sample of seasoned spruce at intervals after evacuation of surrounding chamber to ca. -81 kPa



A satisfactory explanation for these data may be derived by recalling the fact that timber is not a homogeneously permeable material. Phillips (1933) reported that pit aspiration occurs less readily in latewood than in earlywood and observations are reported elsewhere in this thesis (Section 3.2.2.2.1), on the basis of which the comparatively greater permeability of latewood vis-a-vis earlywood in many pieces of spruce is beyond reasonable debate. It is suggested that in this experiment, the 18.5 cm pressure measurements were taken in a band of earlywood, whilst the 19.5 cm measurements were made in or near a band of latewood. The air, by moving through latewood tissue, could be removed from the 19.5 cm monitoring point more rapidly than from the monitoring point 18.5 cm from the open end.

From the above it appears that, during evacuation, the pressure gradient which is established is not necessarily uniform along or across a sample, in consequence of air being removed more rapidly from some tissues than others. Indeed, the pattern of air movement within a sample can be quite complex. Gas may be removed from the earlywood, for example, not only longitudinally but also laterally by passing into the adjacent, more readily evacuated latewood. Arising from this it is possible further to speculate that a 'pocket' of impermeable earlywood may be by-passed and enable air to be removed from the earlywood further away from the open end. This speculation highlights the possibility, therefore, that from the point of view of fluid flow, seasoned spruce may well be composed of a reticulum of anastamosing pathways, between which may be pockets of impermeable timber.

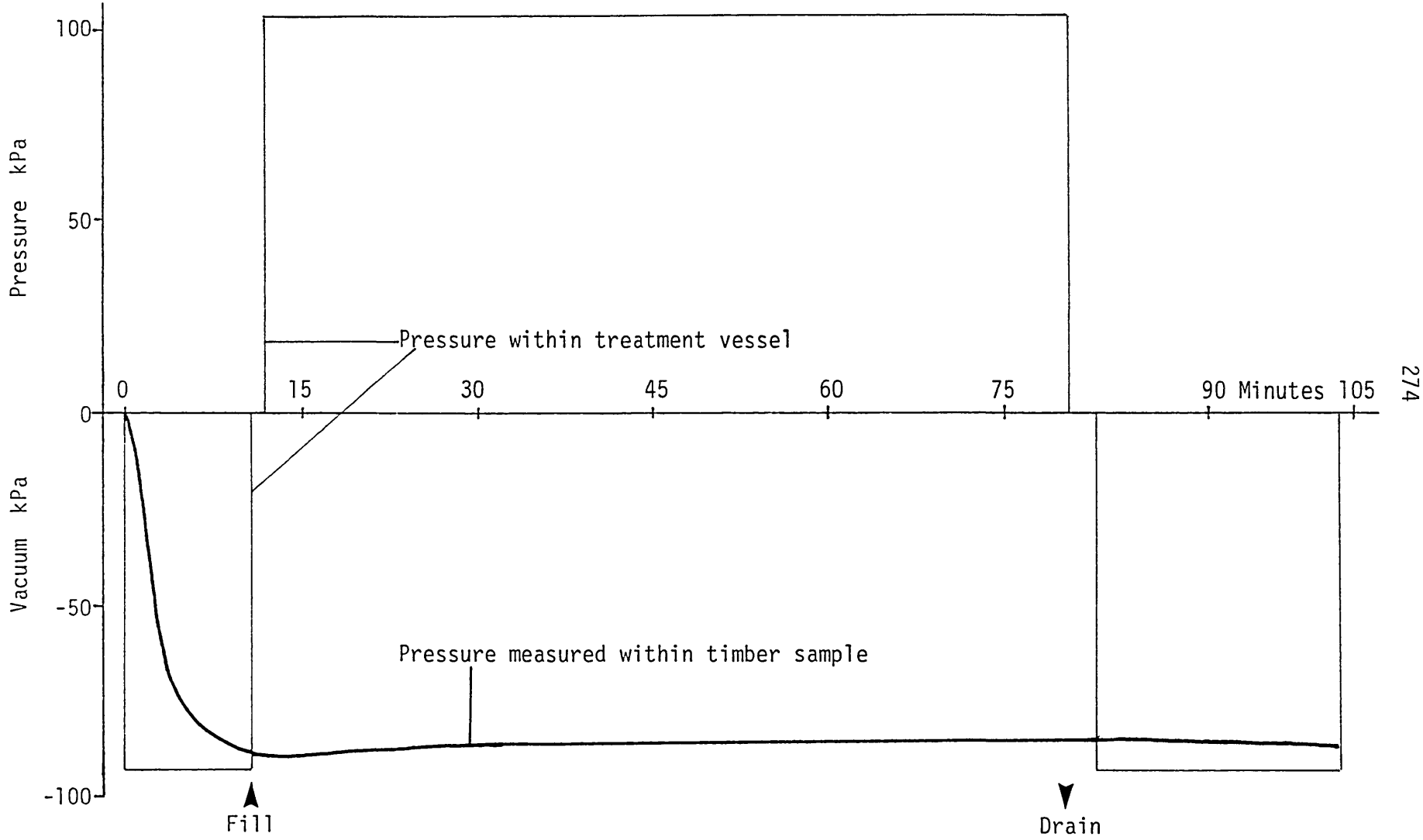
2.2.3.4 Measuring pressure inside timber - results and discussion continued - 3

2.2.3.4.3 Movement of air and white spirit laterally through seasoned spruce

The first part of the experimentation described in Section 2.2.3.3.3 yielded results which are presented in Figure 2.31. Figure 2.32 presents the results of pressure-monitoring within the spruce sample for the 150 hours after the end of the final vacuum.

It may be recalled from Section 2.2.3.1 that, in setting up this experimentation, the anticipated basis of interpretation of the results was derived from Boyle's Law. This law states that, for a given mass of gas, the product of 'pressure-times-volume' is constant at constant temperature. It follows from this that, if gas is removed from a piece of timber such that the diminished residual mass of gas occupies the same volume, the pressure of the residual gas falls. Conversely, if a substantially fixed mass of gas within a piece of timber is driven towards the centre of that timber by the ingress of liquid, its volume decreases and its pressure rises. Thus, when a change is measured in the pressure of the gas within a timber sample during the immersion phase of a DVP impregnation schedule, for example, the change may be considered to be indicative of the occurrence of liquid movement.

Figure 2.31 Pressure changes recorded within a sample of spruce during a double vacuum with pressure, organic solvent impregnation process



Referring firstly to the results presented in Figure 2.31, the pressure changes recorded during the evacuation phase followed an almost identical pattern to that already recorded using the same sample in earlier experimentation (Section 2.2.3.4.1). Thus, the pressure drop was linear with log. time during much of the 10 minute evacuation period, and the differential between vessel pressure and the pressure within the sample at the end of the evacuation period was in the order of 1 - 2 kPa. (The slight uncertainty about the transducer's precise calibration which was discussed in Section 2.2.3.4.1 applies equally here.)

From Figure 2.31 it can be seen that at the end of the evacuation period, the chamber around the sample was flooded with liquid to which ca. 103 kPa pressure was applied. The rate at which pressure change occurred within the spruce sample, after admission of white spirit to the chamber, was clearly very slow in comparison with the evacuation phase and indeed is considered too slow to permit valid analysis of patterns of change in rate etc.

The following discussion seeks to analyse the observed disparity in rates of pressure change in order to establish whether they do other than reflect the difference in viscosity between air and white spirit.

This analysis is, by its nature, somewhat involved and therefore, by way of introduction, the following paragraph adumbrates the principal steps in the analysis.

Reference is first made to Equations 1.23 through 1.25 and the justification for basing the analysis on these equations is presented. Following this, a combined equation (Equation 3.1), relating values for 'V' to both pressure gradient and viscosity, is derived from the above equations. This is used to estimate the relative volumes of air and white spirit which can move through the spruce sample in periods of equal duration. Next, the effect on pressure within the sample caused by flow of white spirit into the partially evacuated sample is discussed quantitatively on the basis of Boyle's Law (Equation 3.2). Equations 3.1 and 3.2 provide the basis on which the pressure within the sample at two selected time intervals is calculated. The calculated pressures are compared with the observed pressures and, on the basis of the relationship between these figures, a general conclusion about the flow of white spirit through the sample is derived.

To reiterate, the following paragraphs seek to analyse the observed disparity in rates of pressure change (as presented in Fig 2.31) to establish whether it does other than reflect the difference which will inevitably result from the difference in viscosity between air and white spirit.

Clearly, in this context it is necessary to seek to relate the observed pattern of pressure change to some sort of assessment of rate of flow of fluid. However, at the present time there does not appear to be any established mathematical interpretation of flow in spruce which has unequivocal validity in this situation.

Some of the reasons for this have already been discussed in Section 2.2.2.3.2.1. The best approximations which appear to be available have been introduced in Section 1.4.2, and are largely due to Petty (1975) who showed that generalised equations for unsteady-state flow may take the forms:

$$V \propto \sqrt{\Delta P} \qquad \text{Equation 1.23}$$

$$V \propto \sqrt{t} \qquad \text{Equation 1.24}$$

and

$$V \propto \sqrt{1/\eta} \qquad \text{Equation 1.25}$$

Apart from the uncertainties outlined in Section 2.2.2.3.2.1, the principal reasons why the applicability of these equations to the present situation is not unequivocal are that, strictly, they hold-good only whilst the pressure within the timber remains unchanged (J.A. Petty, personal communication) and also that they have previously been developed and applied only in the context of the flow of liquid into timber, and not in the context of the outward flow of gas.

Notwithstanding the above uncertainties, Equations 1.23 to 1.25 are used below on the assumption that, by substituting into them numerical values for the conditions which prevailed during the initial evacuation phase, they can be used to calculate a unitless value for the volume of tracheid-lumena 'emptied' of air during this phase. This volume corresponds to the volume of liquid which, when introduced into the block, would return its centre to

atmospheric pressure. Consideration of the conditions which prevailed during the immersion phase will indicate whether the return towards atmospheric pressure happened at a rate consistent with Equations 1.23 to 1.25.

In analysing the results of the present work on the above basis, in the experimentation under discussion, simultaneous consideration must be given to the fact that not only was there a difference between the viscosities (η) of air and white spirit, but the pressure gradients (ΔP) during the evacuation and immersion phases differed also. Thus, to analyse the results, Equations 1.23 and 1.25 need to be considered simultaneously, i.e.

$$V \propto \sqrt{\Delta P^{1}/\eta} \qquad \text{Equation 2.1}$$

To estimate a unitless value, V_1, for the volume of tracheid lumena 'emptied' during the initial evacuation, a value for the initial* pressure gradient (from Fig 2.31), and also a value for the viscosity of air (1.83×10^{-5}) must be substituted into Equation 2.1.

* Arguably, a more accurate value for V_1 would be obtained by using a value for 'average pressure gradient' rather than 'initial pressure gradient'. This procedure would result in a lower value for V_1 and it will be seen later in the discussion that rather than making any difference to the conclusions drawn, this would in fact strengthen them.

Equation 2.1 thus becomes:

$$V_1 = \sqrt{93 \times \frac{1}{1.83 \times 10^{-5}}}$$

and so $V_1 = 2.25 \times 10^3$

A similar unitless value (V_2) for the amount of liquid which might enter the sample during the first ten minutes of the immersion phase (i.e. during a period equal in duration to the initial evacuation phase) can be calculated by substituting into Equation 2.1 the relevant pressure gradient and viscosity figures, i.e.

$$V_2 = \sqrt{194 \times \frac{1}{1 \times 10^{-3}}}$$

and thus $V_2 = 4.40 \times 10^2$

The calculated value for V_1 is therefore 5.1 times larger than that calculated for V_2. This ratio may be used in the following way to estimate the pressure which, on theoretical grounds, might have been expected to occur within the timber sample after the first 10 minutes of the immersion-under-pressure phase.

At the end of the initial vacuum phase, the gas pressure in the sample was ca. 91 kPa below ambient atmospheric pressure; i.e.,

it was ca. 10 kPa absolute pressure.

It has been shown above that, in similar periods of time, the volume of tracheids 'emptied' of air by an initial pressure gradient of 93 kPa will be approximately 5.1 times the volume which may be refilled with white spirit applied at an initial pressure gradient of 194 kPa. From this it follows that, of the air removed during the 10 minute initial vacuum of the present experiment, $\frac{1}{5.1}$ (ca. 20%) may be replaced by white spirit during the first 10 minutes of the immersion phase. Thus the volume of the air in the sample may be diminished to not less than 81%, of its original volume, during the first 10 minutes of the immersion phase. The effect this diminution in volume will have on the pressure may be computed from the Boyle's Law equality:

$$P_1 \; V_1 \; = \; P_2 \; V_2 \qquad \text{Equation 2.2}$$

P_1 is observed to be 10 kPa. Assigning V_1 the arbitrary value 1, V_2 takes the value 0.80. Thus, on substitution, Equation 2.2 becomes:

$$10 \times 1 \; = \; P_2 \times 0.80$$

and thus $P_2 \; = \; 12.5$ kPa

Summarising therefore, the effect of replacing, with white spirit, 20% of the air removed during the initial vacuum, would be to

increase the 10 kPa pressure inside the sample by approximately 2.5 kPa to ca. 12.5 kPa.

In Figure 2.31, a pressure rise of 2 kPa during the first 10 minutes is not recorded. Instead, the pressure changed during this period by only about 0.8 kPa. It appears therefore that the flow of white spirit into spruce is regulated by some factor other than simply the liquid's viscosity. The above analysis may be extended to estimate the pressure changes which, solely on the basis of viscosity and pressure gradient, might be expected at intervals throughout the immersion phase. For example, the pressure within the sample which might be anticipated after 60 minutes may be estimated using the $V \propto \sqrt{t}$ relationship (Equation 1.24).

It has been estimated above that, theoretically, 20% of the removed-air could be replaced by white spirit during the first 10 minutes ($\sqrt{t} = 3.16$) of the immersion phase. By simple proportionation, the volume of removed-air which could be replaced in 60 minutes ($\sqrt{t} = 7.7$) is ca. 48.7%. Applying the equality, $P_1V_1 = P_2V_2$, diminution of the volume of air in the sample to ca. 51% of its original volume would cause a pressure increase of ca. 9.6 kPa to 19.6 kPa. However, from Figure 2.31 it may be seen that, during the first 60 minutes of the immersion phase, pressure in the sample increased by barely 2.5 kPa. This observation strengthens the conclusion, already expressed, that the difference in the flow rates through spruce of air and white

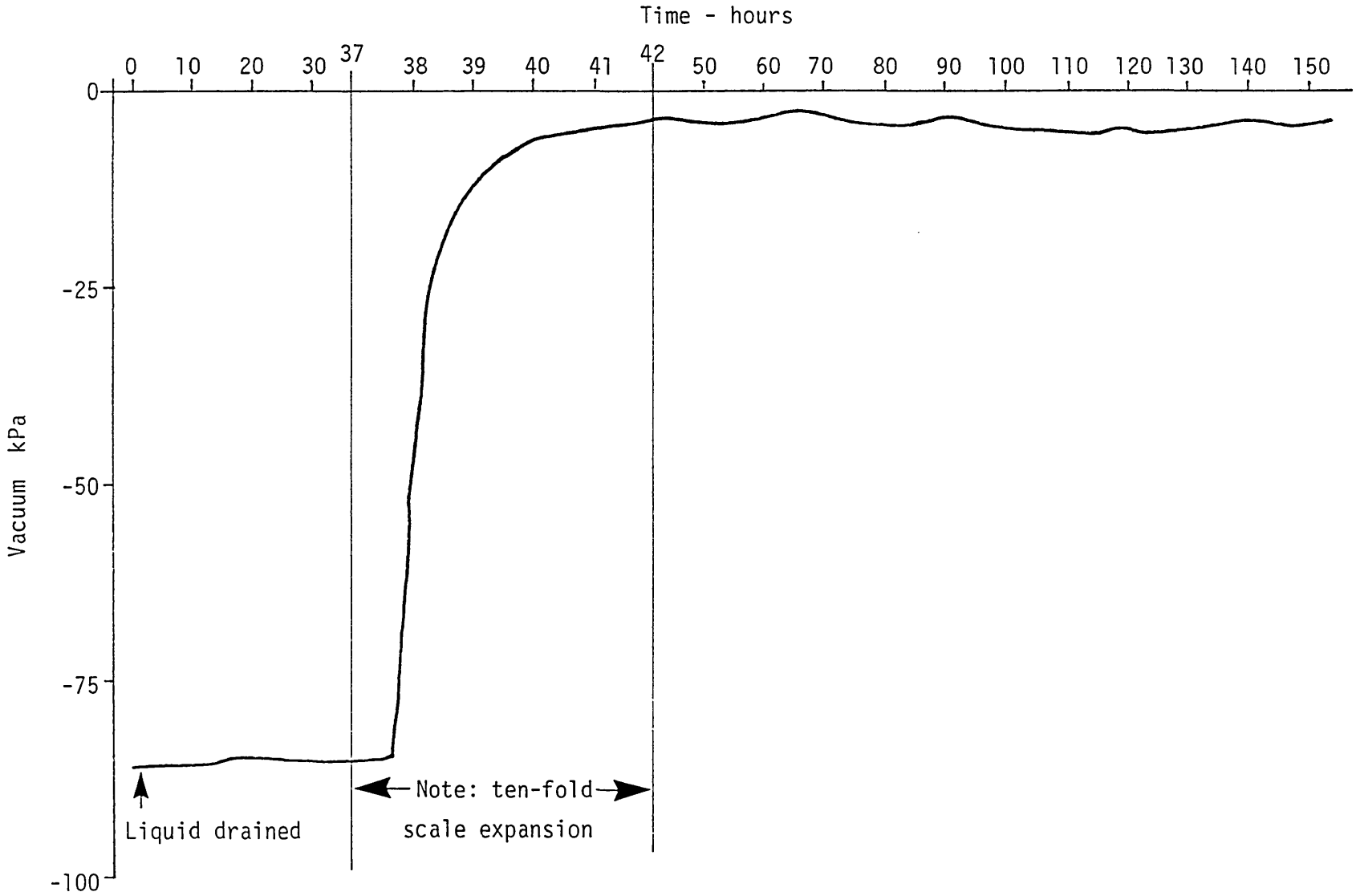
spirit cannot be ascribed to differences in viscosities alone; an additional factor has to be invoked to account for these data. The identity of this factor is discussed in Section 4.

Pressure changes within sample after removal from liquid

In Section 2.2.3.3.3 it was recorded that, at the end of the immersion phase, the spruce sample was removed from the white spirit and stood on the laboratory bench. The pressure within the sample continued to be monitored for a further 150 hours. The pressure changes observed during this period are presented in Figure 2.32 and they exhibit three principal phases as follows.

During the first phase, which ended approximately 37 hours after the beginning of the immersion phase, the pressure slowly rose towards atmospheric pressure. The total pressure change was small however, being no more than 4 or 5 kPa. It is not considered possible to decide whether the changes proceeded, for instance, linearly with log time, because it is uncertain whether the accuracy and stability of the instrumentation was adequate to record satisfactorily such small changes occurring over such a long period. Close examination of the original pen recorder output reveals at least one period when the signal drifted slightly (less than 0.5 kPa) away from atmospheric pressure and it remains unresolved whether this was a real effect or instability in the instrumentation. In either event the conclusion must be drawn that, because the pressure within the mass of gas remained substantially unchanged during the 40 hour period, its volume

Figure 2.32 Pressure within a sample of spruce during the period following the start of the immersion phase represented in Figure 2.31



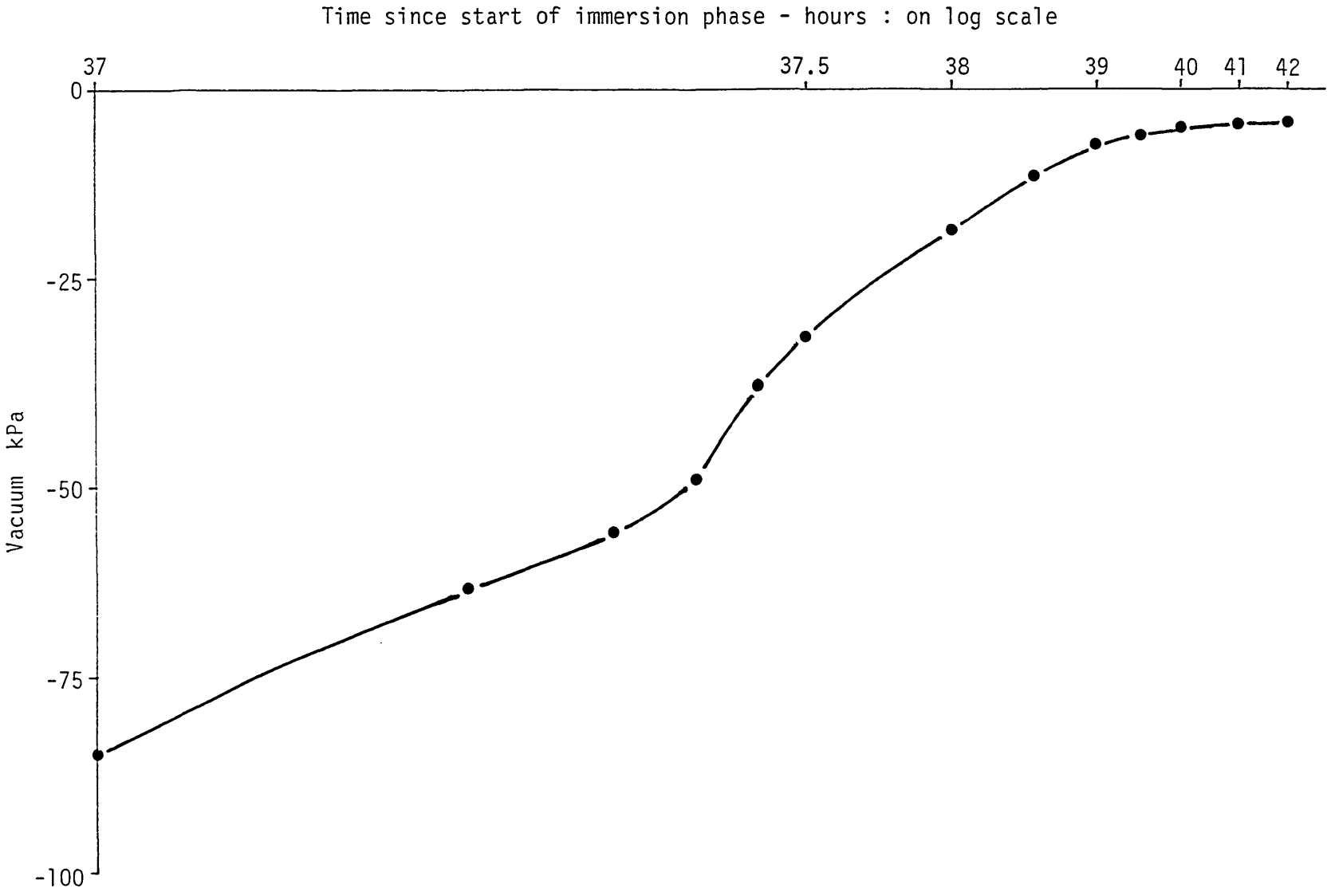
remained substantially unchanged also. Therefore there can have been only very little movement of liquid during this period.

The second phase, which lasted about 4½ hours, was characterised by much greater and more rapid pressure change. Starting approximately 37.7 hours after the start of the immersion phase, the pressure within the sample moved towards atmospheric pressure comparatively rapidly, such that after 60 minutes a pressure change of 68 kPa had occurred. During the succeeding 3 hours, the pressure changed progressively more slowly but continued to move towards ambient atmospheric pressure.

The pattern of variation in rate of pressure change did not follow any readily identifiable pattern. Figure 2.33 presents the data for this phase replotted against log time and from this it is clear that a linear plot is not obtained. Thus no simple exponential relationship can be derived from these data.

In seeking to postulate a physical basis to account for the overall pattern of pressure change observed during the second phase, it is necessary firstly to consider what could have led to the observed increase in pressure within the sample during the second phase. Given that the temperature remained essentially constant throughout the three phases, the increase in pressure can have resulted only from one of two causes. Either the volume of the gas within the sample diminished, or the mass of gas within the essentially fixed volume increased.

Figure 2.33 Pressure within a sample of spruce during the 37 - 42 hour period represented in Figure 2.32, plotted against time on a log scale



For the volume of gas in the sample to have decreased, the liquid in the sample, which for 37.7 hours barely moved, would have had 'suddenly' to move towards the centre of the sample. Certainly the pressure gradient across the sample favoured movement in this direction, but there appears to have been no event which might have 'triggered' the necessary, relatively sudden, widespread movement of liquid. Therefore the first possibility, namely that the volume of the fixed mass of gas decreased, is not apparently plausible. The second possibility identified above was that the mass of gas in the centre of the sample increased. For this to have occurred, air from around the sample would have had to 'by-pass' the liquid in the sample's outer layers of tracheids and enter the central lumena of the sample. Here, not only was the pressure gradient favourable to such a movement, but it is also possible to postulate a physical mechanism whereby this movement may 'suddenly' have occurred.

Throughout the 37.7 hour duration of 'phase one', the pressure differential between the inside and outside of the sample was being maintained in consequence of the presence of a continuous, near stationary 'layer' of liquid in the lumena of the sample's superficial tracheids. This liquid was white spirit which is a volatile aliphatic solvent. As such, it will have evaporated as time passed. Inevitably, therefore, a point must have been reached at which sufficient solvent had evaporated to interrupt the continuity of the layer of liquid-occluded tracheid lumena around the sample. At this point a continuous 'chain' of tracheid

lumena through which gas-flow could occur would be re-established. This would allow for air to flow into the tracheid lumena in the centre of the sample and the pressure within the sample would increase.

The rate of pressure change which occurred during the second phase was much slower than that which was observed during initial evacuation. This difference is consistent with the fact that, whereas during initial evacuation the air was flowing out of the sample through an unknown, but probably large, number of pathways, during the period of pressure change being considered here, liquid probably only flowed back into the sample through a single pathway, all the others being occluded by liquid. It would, in fact, be a considerable coincidence if, by evaporation of liquid, two gas-flow pathways became available simultaneously. However, it may be postulated that the inflexion in the line in Figure 2.33 may have been caused by a second pathway becoming available approximately twenty minutes after the first.

The third of the three phases into which the data in Figure 2.32 have been divided can be considered to have started about 42 hours after the beginning of the immersion phase. This third phase continued for the remainder of the time during which pressure was continuously monitored, i.e. for approximately 100 hours.

At face-value, the pen recorder output indicates that the pressure within the sample did not return to atmospheric pressure at the

end of the second phase. Instead the pressure increased to ca. 4 kPa below atmospheric pressure and thereafter relatively little change occurred. The signal from the instrumentation was not totally steady however and, in addition to being 'noisy', indicated a cyclic fluctuation in pressure in the range 2.5 - 5 kPa below atmospheric pressure. There appeared to be a 24 hour periodicity to these fluctuations, the cause of which is unclear.

When continuous monitoring of the pressure was discontinued after 100 hours, it was still uncertain whether the apparent indication that the pressure was 2.5 - 5 kPa below atmospheric pressure was reliable or due to inaccuracy in the experimental system. In an attempt to clarify this point, the apparatus was not dismantled. Instead it was turned on at intervals on the assumption that, as more solvent evaporated, there was progressively less chance of there continuing to be a pressure gradient within the sample. Four months later, the instrumentation continued to indicate a pressure within the sample which was fluctuating in the range 2.5 - 5 kPa below atmospheric pressure.

Whilst it remains impossible to be certain, it seems most likely that, at the end of the second phase of pressure change, the pressure within the sample did in fact return to atmospheric pressure. Indications to the contrary which came from the pen recorder output are considered to reflect the limitations of the instrumentation. It is not therefore considered appropriate to seek to advance a physical explanation to account for the continued existence of a small pressure gradient within the sample.

2.2.3.4 Measuring pressure inside timber - results and discussion continued - 4

2.2.3.4.4 Pressure changes in seasoned spruce during a series of DVP schedules

The experimentation described in Section 2.2.3.3.4 yielded results which are presented in Figs 2.34 - 41.

Two of the experiments, namely those involving samples 9 and 17, did not yield satisfactory results for technical reasons. Additionally, the results from block 31 (Fig 2.40) are incomplete. This arose because the signal went off scale 17 minutes into the immersion phase. When this occurred an attempt was made to continue monitoring the pressure within the sample by changing the excitation voltage setting on the pen recorder in order to attenuate the signal. It was anticipated that the apparatus could be recalibrated subsequently. This manipulation proved unsuccessful however and thus the remainder of the pressure recording was lost.

Initial vacuum phase

Discussion of the eight sets of results which were obtained is subdivided into four successive sections. Namely:

Figure 2.34 Pressure changes within spruce Sample 2 during a double vacuum with pressure organic solvent impregnation process

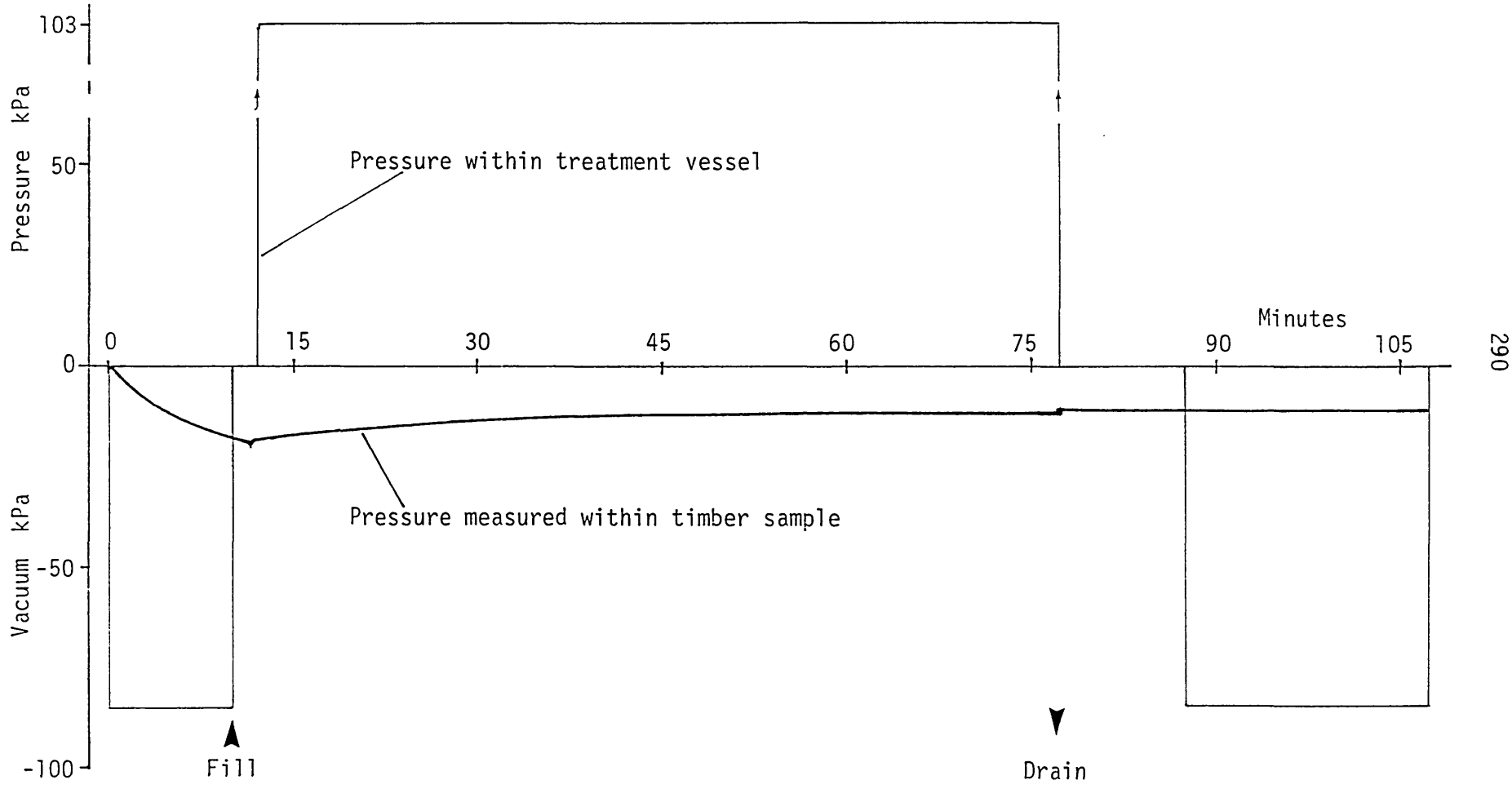


Figure 2.35 Pressure changes recorded within spruce Sample 28 during a double vacuum with pressure organic solvent impregnation process

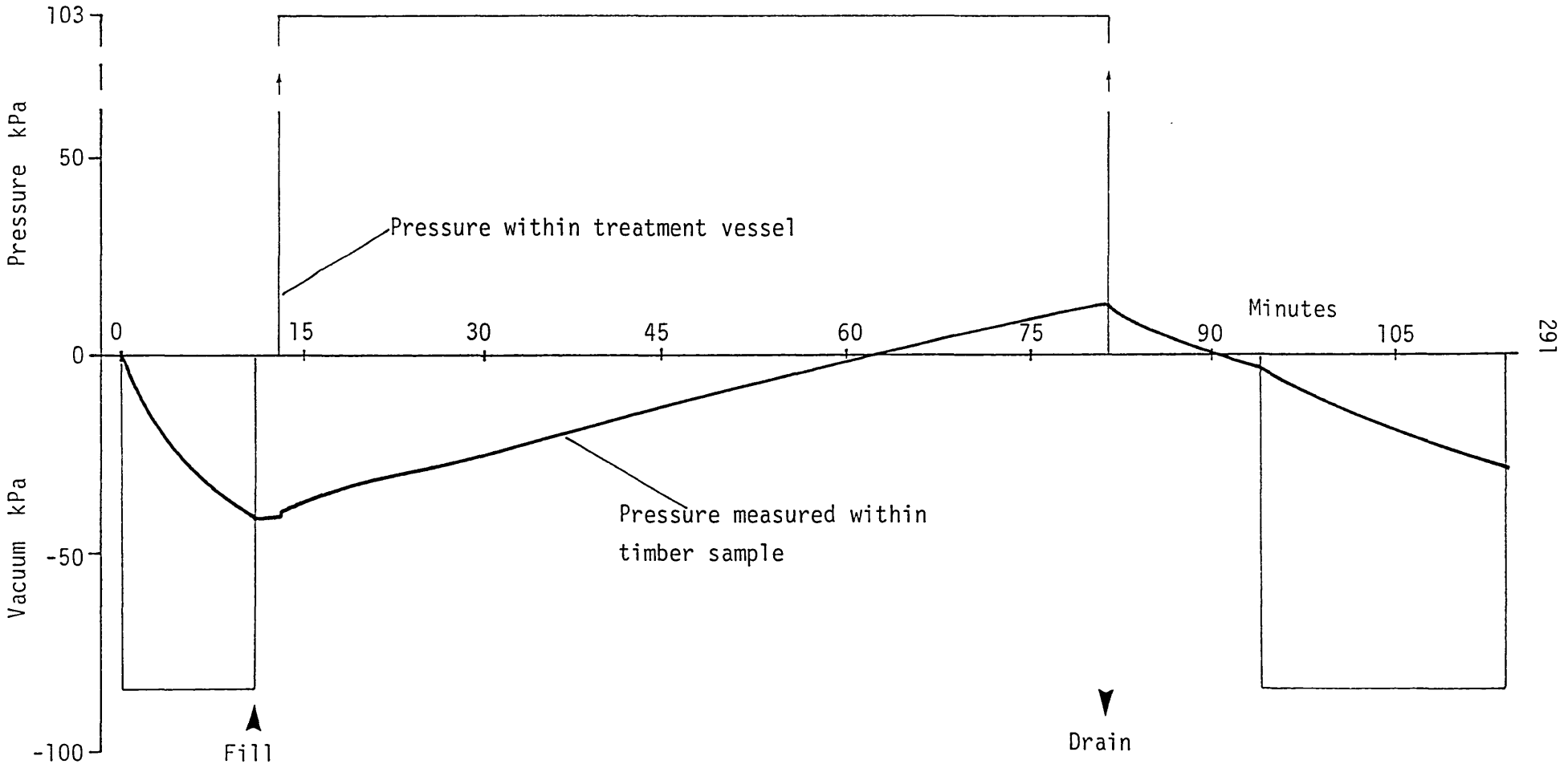


Figure 2.36 Pressure changes recorded within spruce Sample 32 during a double vacuum with pressure organic solvent impregnation process

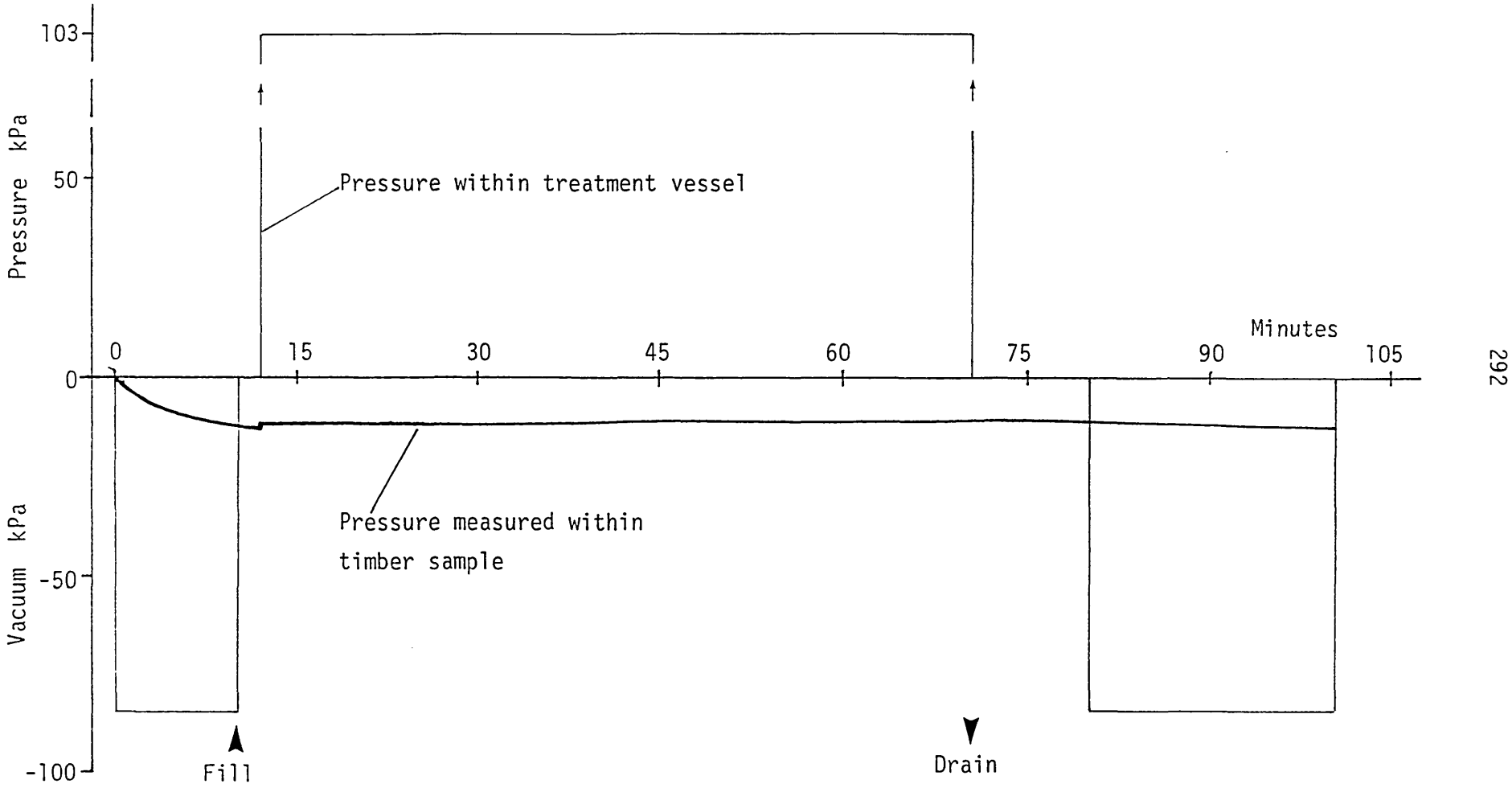


Figure 2.37 Pressure changes recorded within spruce Sample 10 during a double vacuum with pressure organic solvent impregnation

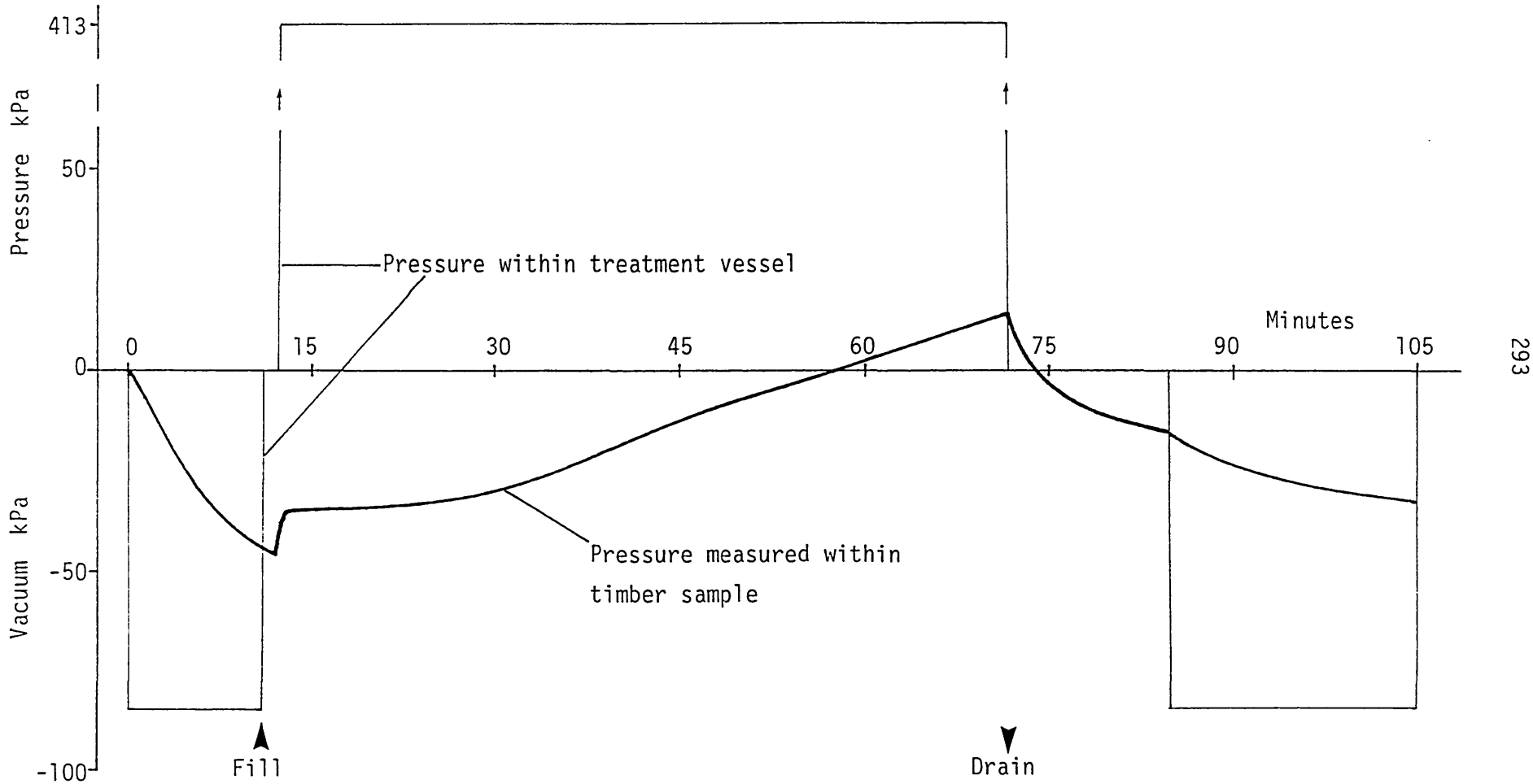


Figure 2.38 Pressure changes recorded within spruce Sample 16 during a double vacuum with pressure organic solvent impregnation process

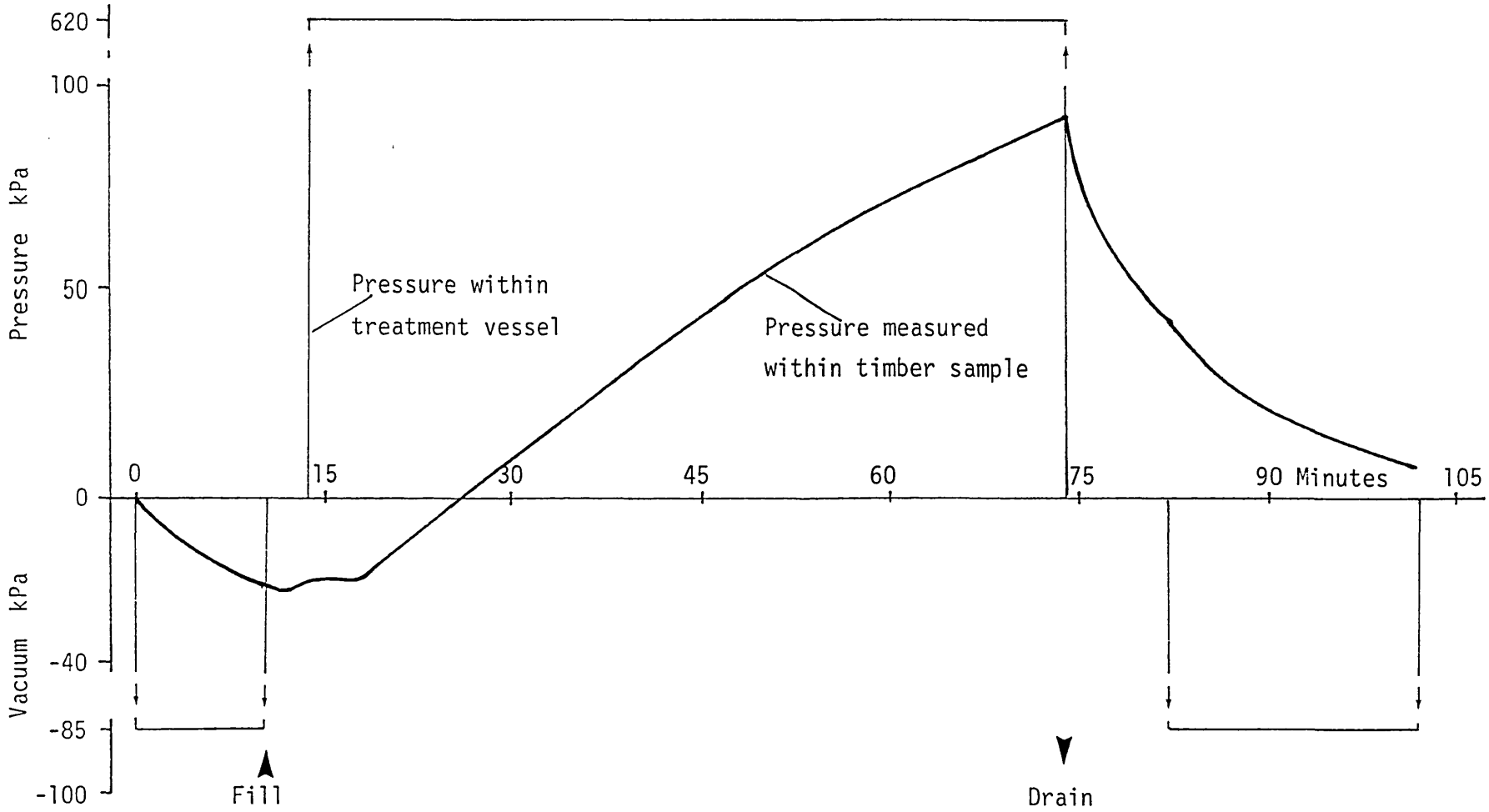


Figure 2.39 Pressure changes recorded within spruce Sample 3 during a double vacuum with pressure organic solvent impregnation process

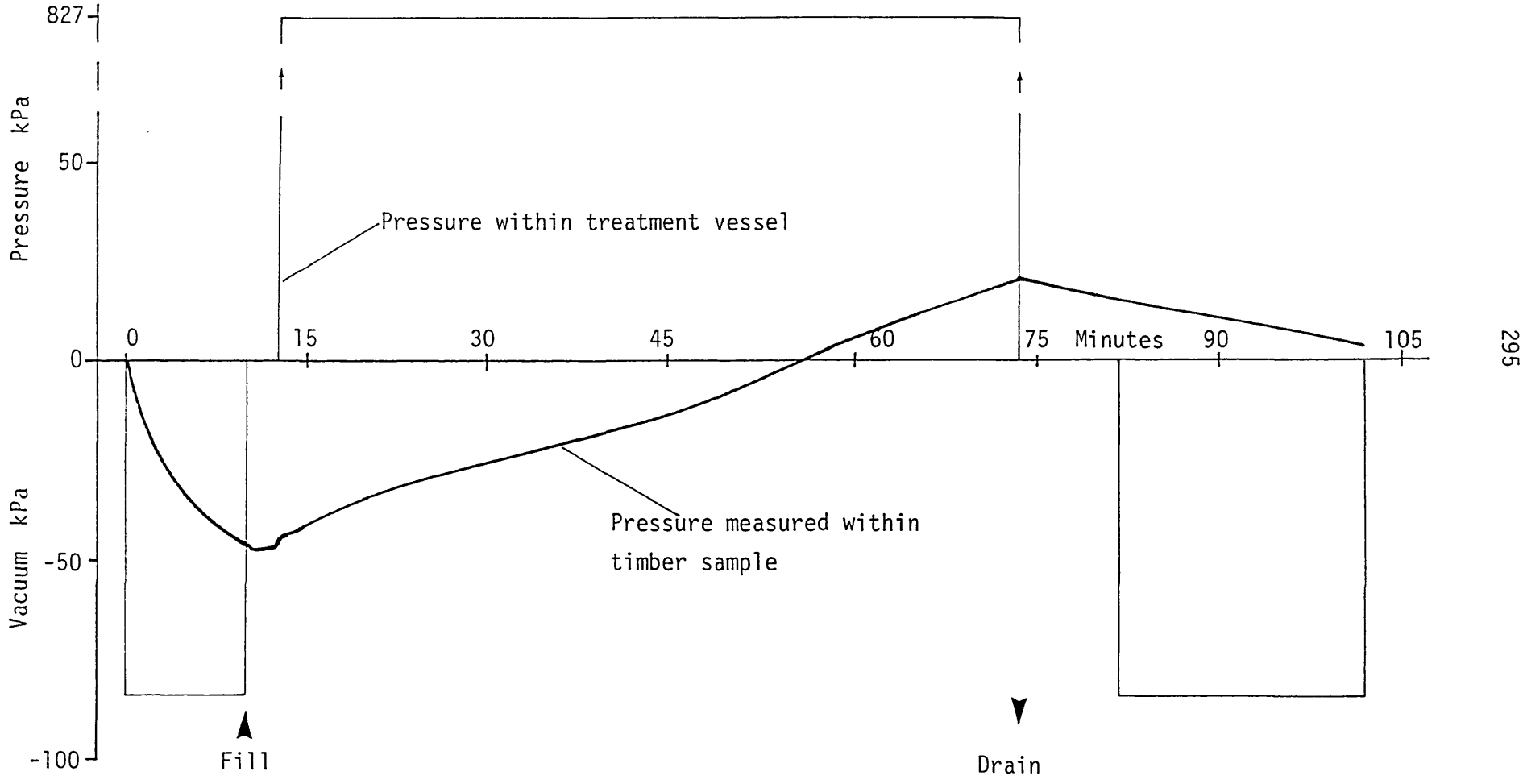


Figure 2.40 Pressure changes recorded within spruce Sample 31 during part of a double vacuum with pressure organic solvent impregnation

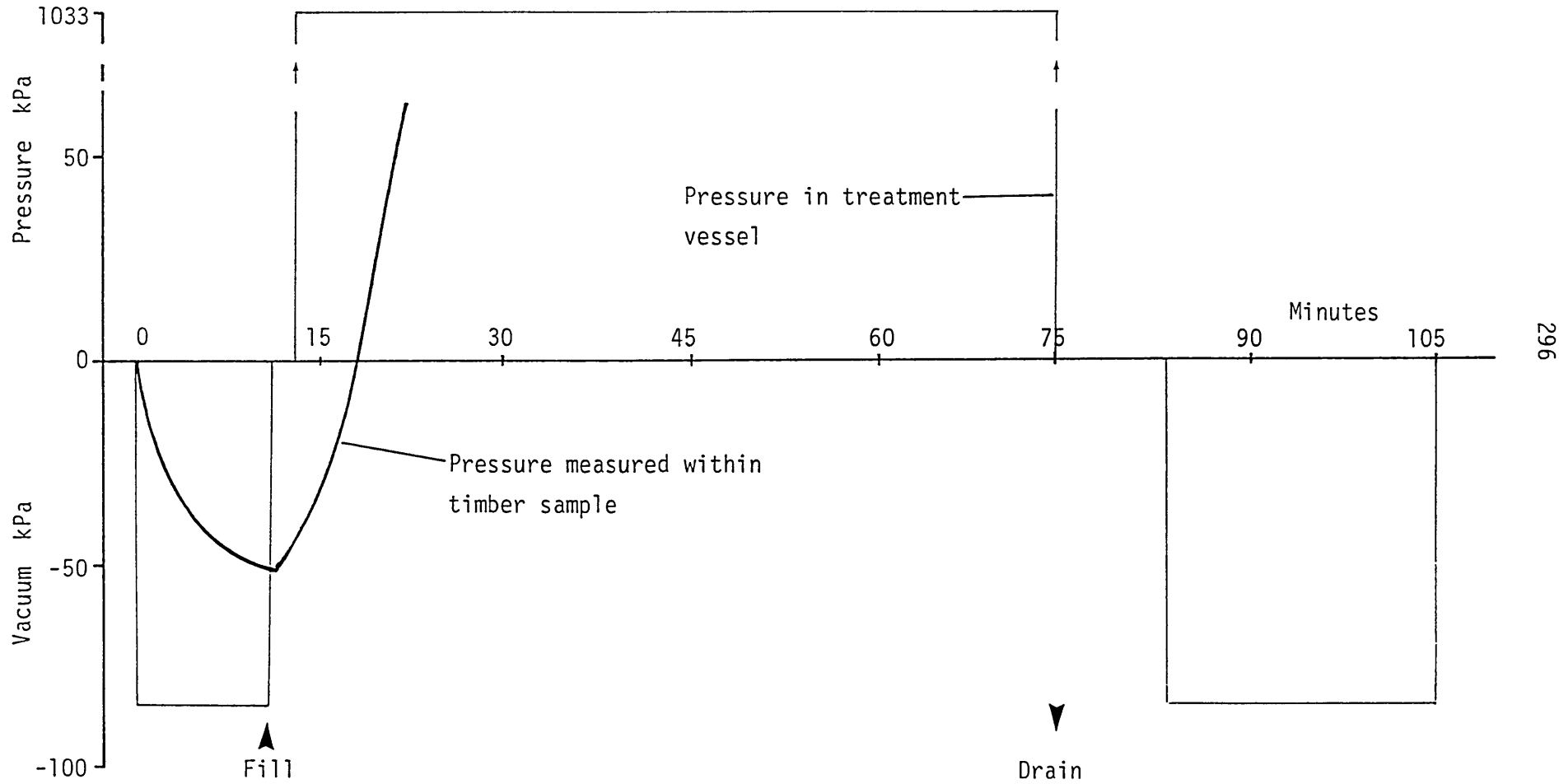
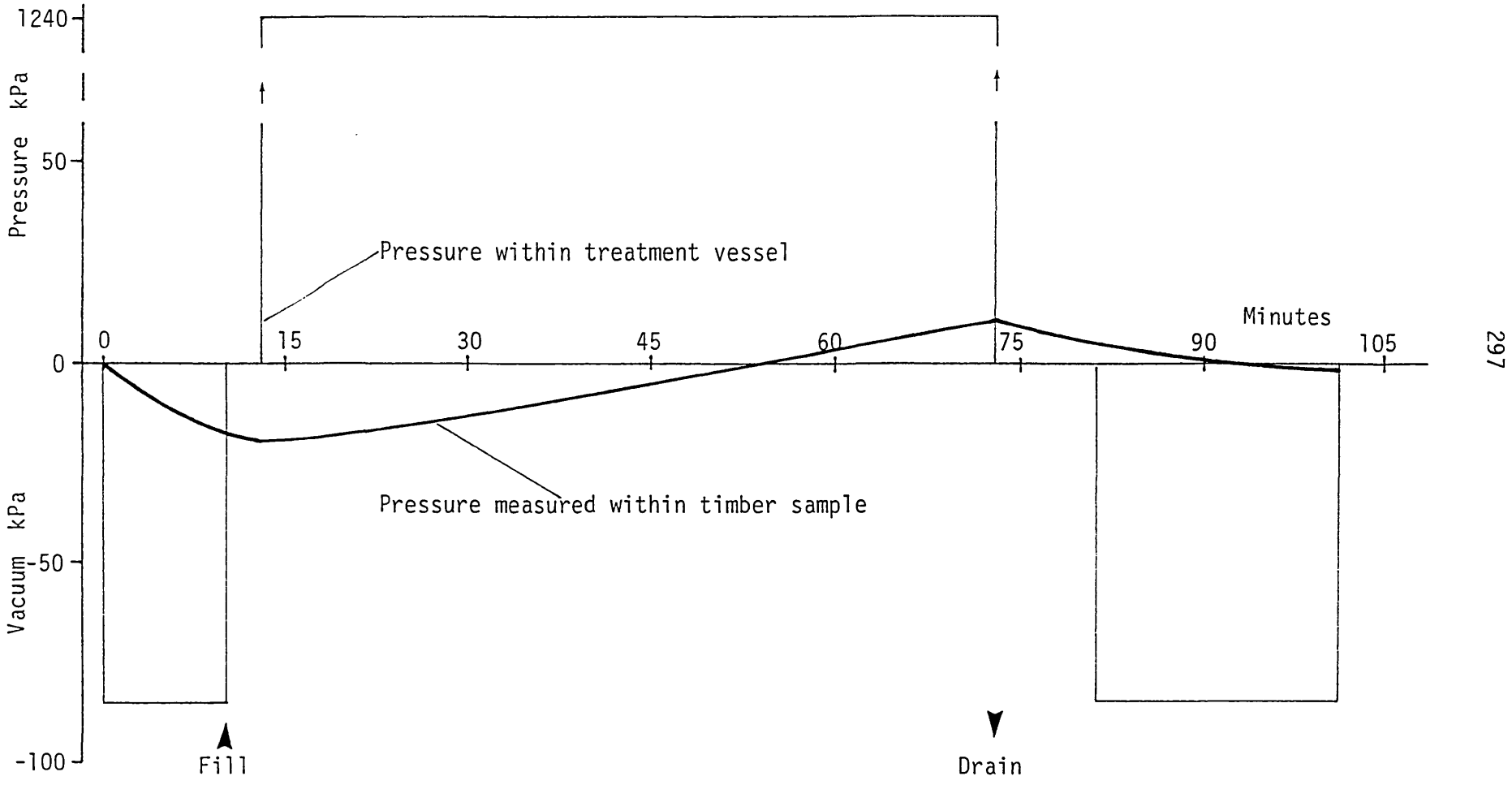


Figure 2.41 Pressure changes recorded within spruce Sample 27 during a double vacuum with pressure organic solvent impregnation process



Initial vacuum phase

Immersion-under-pressure phase

Final vacuum phase

Post-treatment phase

The recordings of pressure change made during the 10 minute initial vacuum phases show that reduction of pressure within the samples varied between ca. 11 and ca. 53 kPa. Thus, in no case was evacuation of the sample as complete as was seen in the spruce sample discussed in Section 2.2.3.4.3. The data for pressure change during the initial vacuum phases are replotted against log. time in Figure 2.42. From this figure it can be seen that in most cases pressure change was, as a first approximation, linear with log. time over some of the period recorded. Overall, all samples showed a positive deviation from linearity. More precise analysis of the pressure-time relationship is not possible because the length of time taken to achieve evacuation of the treating cylinder introduced uncertainties about the designation of time 'zero' analagous to those discussed in Section 2.2.1.3.1.

Whilst precise analysis of the results is not possible, a further feature which is clearly evident is the variability between samples. It may be recalled that all the samples were prepared from the same length of timber, and indeed samples 2 and 3, 27 and 28, and 31 and 32 were not only longitudinally aligned but were immediately adjacent to one another in the standing tree.

Figure 2.42 Pressure changes within spruce samples during initial vacuum phases of impregnation schedules (Figs 2.34 through 2.41), replotted against log time

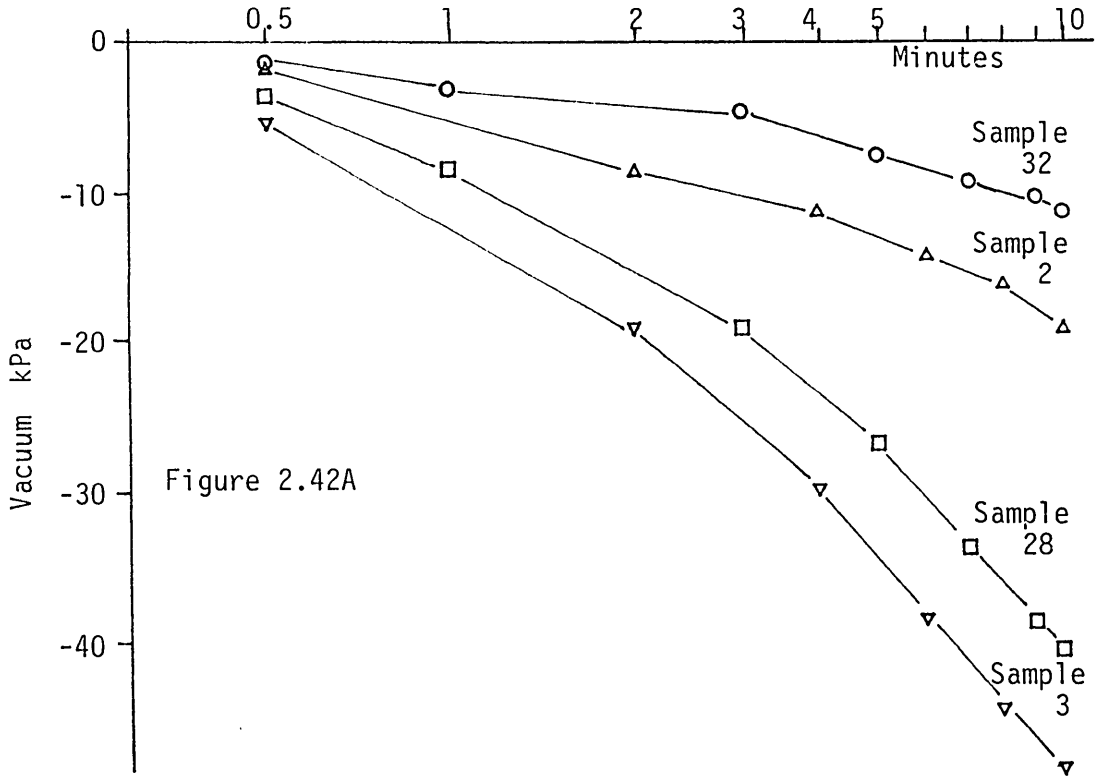


Figure 2.42A

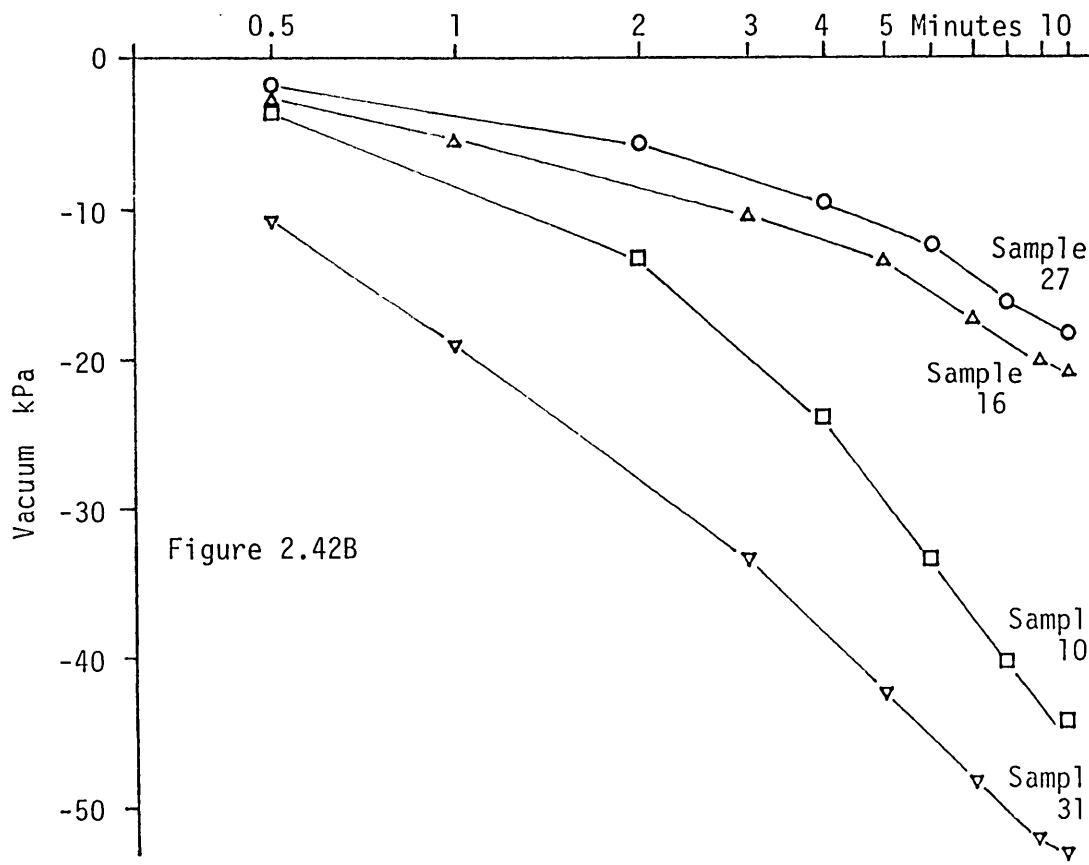


Figure 2.42B

However, in no case did the pairs of samples show closely similar permeabilities, and samples 31 and 32 displayed respectively the highest and the lowest permeabilities to air observed in this section of the investigation. This observation highlights the heterogeneity of seasoned spruce - a phenomenon which has been mentioned already and which is further discussed in both Section 3 and Section 4.

Immersion-under-pressure phase

Considering next the pressure changes which occurred during the immersion-under-pressure phases, three of the samples (nos. 2, 28 and 32, Figs 2.34 - 2.36) yielded results during immersion at 103 kPa. In these three samples the magnitudes of the pressure changes observed during the immersion phases were in logical proportion to the size of the pressure changes which occurred during the initial vacuum. That is to say, sample 32, which displayed the smallest change in its internal pressure during the initial vacuum phase, also showed the smallest change during the immersion phase. With sample 28, the converse of this was observed, and sample 2 obviously therefore showed intermediate results.

The results from sample 28 (Fig 2.35) are particularly interesting when they are compared with the results which have already been discussed in Section 2.2.3.4.3.1. Within sample 28, the pressure rose steadily throughout the immersion phase, and by the end of this phase it had risen to ca. 10 kPa above atmospheric pressure.

Conversely, the pressure within the sample discussed in Section 2.2.3.4.3.1 rose much less quickly, despite the fact that the pressure change during the initial vacuum was greater in this latter sample than in sample 28. Thus, from these results, it appears that the relationship between the rate of outflow of air during the initial vacuum and the rate of inflow of liquid during the immersion phase is not constant, but differs between different samples of spruce.

It is possible to postulate a physical basis to account for variable relationships between the rate of gas-flow through a sample and the rate of liquid-flow through the same sample. A sample in which there are a large number of flow paths, the constrictions of which are very small, may allow gas-flow through the sample to occur fairly quickly whereas liquid-flow is impeded at the constrictions by such causes as air embolism formation (q.v. Section 1.3.4). Conversely, a sample in which there are a comparatively smaller number of slightly larger pathways may not permit rapid gas-flow, but liquid is subsequently able to flow through more of the pathways through which gas-flow formerly occurred. Thus, variations in the ratio of pressure-change-during-initial-vacuum to pressure-change-during-immersion may indicate differences in the size, shape and abundance of flow paths.

It must be noted that there were slight differences in the liquids used in the work which yielded the results reported in the previous section and in this one. These differences may have contributed to the observed differences in behaviour although this seems somewhat unlikely.

Turning now to the remaining five sets of pressure changes which were recorded during the immersion-under-pressure phases, each of these was obtained using a different pressure on the liquid. Consideration of these data highlights the apparent absence of a constant relationship between the permeability of spruce to gas and to liquid.

The lowest of the remaining pressures was ca. 413 kPa (Sample 10, Fig 2.37) and here, although the pressure change during initial vacuum was slightly greater than in sample 28, the pressure change during immersion was substantially similar (despite the immersion pressure being four times greater).

Sample 16 (Fig 2.38) was immersed at 620 kPa and here, despite a fairly small pressure change during the initial vacuum (ca. 21 kPa), by the end of the immersion phase the pressure had risen to ca. 92 kPa above atmospheric pressure.

The sample 3 (Fig 2.39) results contrast with those of sample 16. The pressure within sample 3 changed by ca. 49 kPa during the initial vacuum, which was only slightly less than the largest of

the pressure changes observed during the initial vacuum phases of this series of experiments. However, despite the application of a pressure of 827 kPa during the immersion phase, the pressure change in the sample's centre was smaller than the sample 16 change, totalling only ca. 63 kPa by the end of the immersion phase.

The next highest pressure (1033 kPa) was applied to sample 31 - Figure 2.40 and here there is a great contrast between the results of sample 31 and sample 3. The pressure change during the initial vacuum was the largest recorded in this section, being ca. 53 kPa. Subsequent to this, after the vessel had been flooded and a pressure of 1033 kPa applied, the pressure within the sample rose rapidly and, 17 minutes after the pressure had been applied, the signal went off scale at 316 kPa. If air embolisms were indeed blocking flow paths in these samples, the pattern of pressure changes observed in sample 31 might be interpreted as evidence that 1033 kPa is the pressure necessary to overcome the resistance of air embolisms. However, supportive evidence for this conclusion is not forthcoming from the data yielded by the sample immersed at 1240 kPa.

The highest immersion pressure used, 1240 kPa, was applied to sample 27 (Fig 2.41). The pressure within this sample changed by only ca. 18 kPa during the initial vacuum phase, and then changed by ca. 29 kPa during 60 minutes immersion at 1240 kPa. The pressure change during the initial vacuum phase indicated that

this sample had fairly low permeability. Liese (1979) reported that, at immersion pressures of 1500 kPa and above, the cells of air-dried spruce may collapse. Collapsed cells around the surface of a sample could well lead to the sample being 'sealed' from ingress of liquid and thus, above some threshold immersion pressure, the pressure within the sample would be prevented from rising. Such an effect might also be expected to be accompanied by a reduction in uptake of preservative at very high immersion pressures. This aspect is investigated elsewhere (Section 3.2.1.2)

Final vacuum phase

The third of the four successive sections in which these data are being considered is the final vacuum phase. (In fact, for the purposes of this discussion, the pressure changes which occurred whilst the treating vessel was being drained of liquid will also be considered in this section.) Data are available from seven of the eight samples examined, the results from sample 31 (Fig 2.40) being incomplete as already stated.

In two of the samples (nos. 2 & 32), the size of the pressure change which was observed during the final vacuum phase was negligible. These two were the only two samples in which the pressure inside the sample did not rise above atmospheric pressure by the end of the immersion phase, and so were the two samples in which there was least potential for substantial pressure change

during the final vacuum period.

The remaining five samples all showed pressure changes of at least 10 kPa. Of these, two (nos. 3 and 27 - Figs 2.39 and 2.41) displayed more-or-less steady pressure change throughout both the draining and final vacuum phases. In the remaining three samples (nos. 10, 16 and 28 - Figs 2.37, 2.38 and 2.35 respectively), the pattern of pressure change was rather different. In all three the pressure dropped most quickly when the pressure on the vessel was first released and then progressively more slowly during the 10 minutes or so which it took to drain the treatment vessel. The rate at which pressure decrease occurred gained fresh impetus as the final vacuum was applied, and then again became progressively more slow. In none of the samples was the pressure as low at the end of the final vacuum as it was during the initial vacuum.

An unexpected feature of several of the patterns of pressure change during the final vacuum phase was that the rates of pressure change were sometimes as fast as, or faster than, were observed during the immersion phase. (For example, sample 16 - Fig 2.38.) This occurred despite the fact that, during the immersion phase, the pressure gradients promoting liquid flow (and thus promoting pressure change within the sample) were much greater than during the final vacuum. In sample 16 a pressure gradient of initially ca. 641 kPa, and after 60 minutes of ca. 523 kPa, promoted flow of liquid which increased pressure at the centre of the sample in

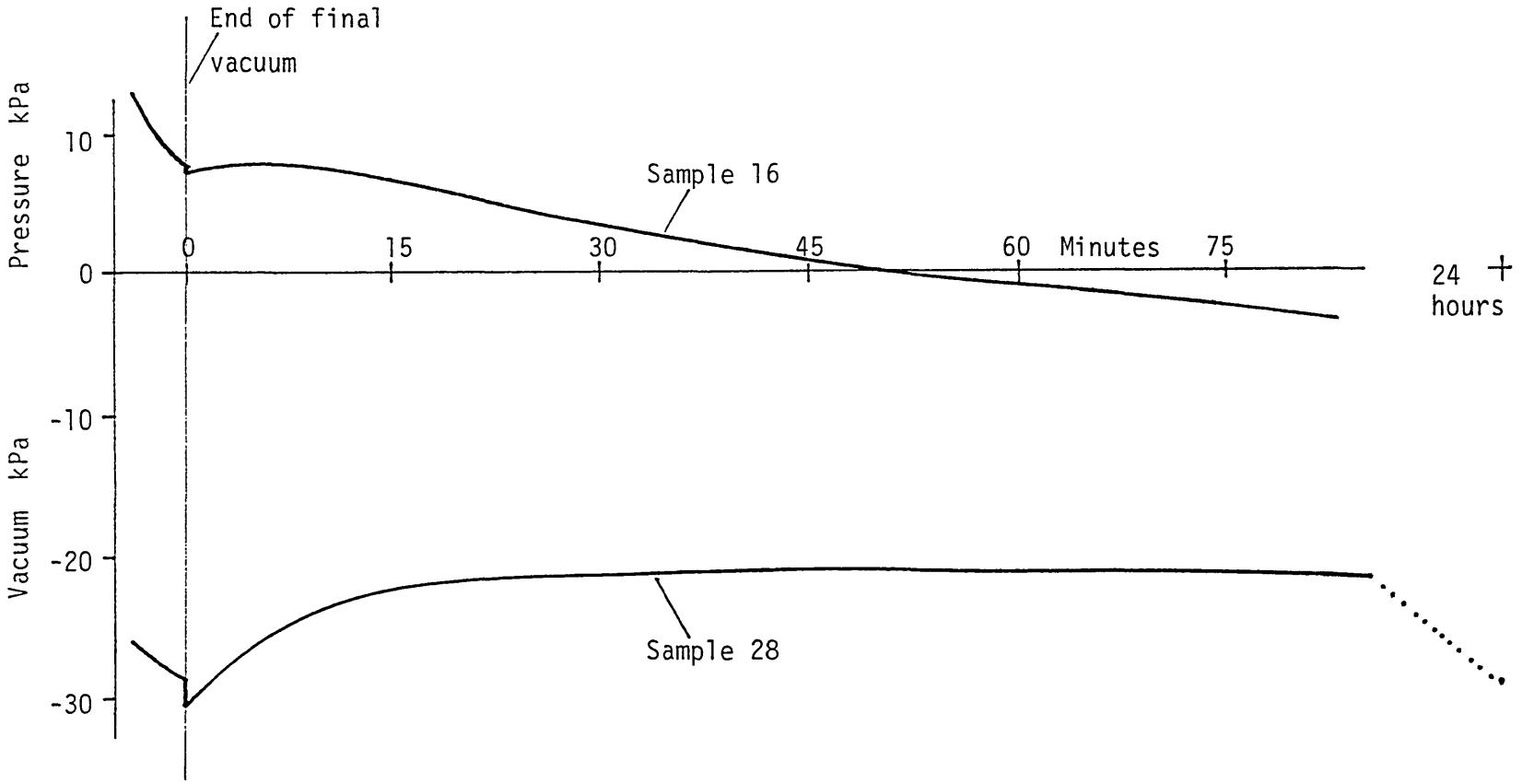
the manner indicated in Figure 2.38. When the immersion pressure was released, the direction of the pressure gradient was reversed and an internal pressure of ca. 95 kPa above atmospheric tended to expel liquid from the sample. The rate at which the sample's internal pressure decayed indicates that an initial internal pressure of 95 kPa expelled liquid from the sample faster than a pressure gradient at least six times larger was able to force liquid in. It does not appear to be possible to postulate a mechanism, based on existing knowledge or theories of flow of liquid in timber, to account adequately for these observations.

Post-treatment phase

The fourth and final section in which these data are being considered is the 'post-treatment' phase. The transducer was left inserted in two of the samples (nos. 16 and 28) for at least one hour after the end of the final vacuum phase.

The pressure changes monitored during this post-treatment phase are presented in Figure 2.43 and from this figure similarities in the patterns of pressure change can be seen. When the final vacuum was released, the pressures within the samples dropped a little and then rose by between ca. 0.6 kPa and ca. 10 kPa. Following this the pressure declined steadily.

Figure 2.43 Pressure changes measured inside two samples of spruce after the end of an industrial-type impregnation with organic solvent preservative



In sample 16, the slow pressure decrease was not unexpected because, at the end of the immersion phase, the pressure within the sample was still slightly higher than ambient atmospheric pressure. Arguably, therefore, the pressure within the sample did no more than equilibrate with its surroundings. Fifty minutes after the end of the final vacuum the pressure within the sample had returned to atmospheric pressure. The pressure continued to drop after atmospheric pressure had been reached and after a further 35 minutes the pressure was apparently ca. 4 kPa below atmospheric pressure. However, when the transducer was removed from the sample it continued to give a signal ca. 4 kPa below atmospheric pressure and so a zero-shift may have occurred at some time during the preceding experimentation.

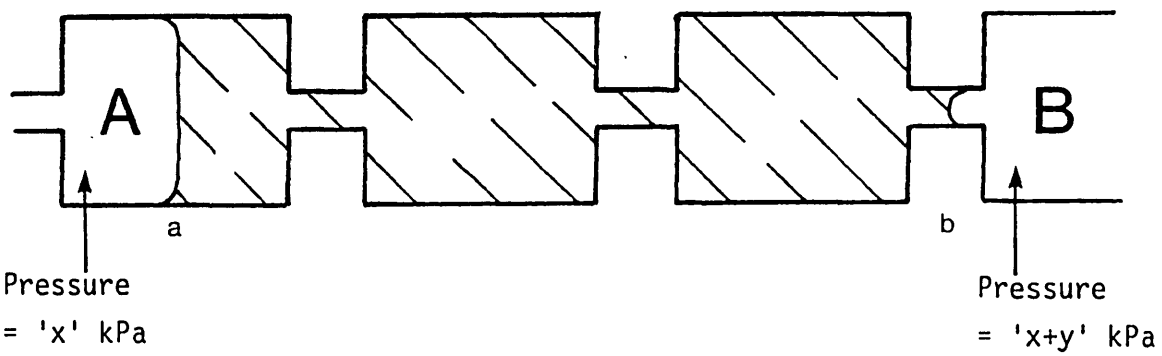
In sample 28, the pressure rose to -21 kPa approximately 55 minutes after the end of the initial vacuum, and thereafter dropped to ca. -29 kPa over the course of the following 23 hours.

Thus, from the above, it is clear that in sample 28, after the end of the final vacuum, the pressure within the sample did not return to atmospheric pressure. Instead the differential between the pressure within the sample, and ambient atmospheric pressure, increased. The pattern of pressure change which had been observed during the immersion phase (Fig 2.35) had demonstrated that there existed within the sample pathways through which liquid flow could occur. A question arose, therefore, concerning how a pressure gradient between the inside and outside of the sample

could be perpetuated, and even increased, even though movement of liquid along the flow paths had already occurred earlier in the experimentation. The following paragraphs seek to suggest, firstly, how a pressure gradient can be perpetuated and then, how it might increase.

An explanation of how pressure gradients may be perpetuated may be readily derived on the basis of the air embolism theory. However, an alternative explanation can also be advanced. This also has its roots in the fact that the flow paths in spruce, (and indeed in any softwood) do not have parallel sides but are composed of alternately wide portions (the tracheid lumena) and narrow portions (in the pits - possibly through the margos). To represent this situation, the schematic representation of a single, simple, softwood flow path, already used in Section 2.2.1.4 (Fig 2.8) is reproduced in Figure 2.44, together with a hypothetical body of liquid within it.

Figure 2.44 Schematic diagram of liquid in a single, simple softwood flow path



In the situation depicted in Figure 2.44, the pressure in the bodies of gas at either end of the body of liquid are such as to favour movement of liquid from B to A. However, at each of the two liquid/air interfaces there are forces deriving from the capillary attraction of the liquid to the walls of the flow path, and these may be considered in terms of the effective pressure they can exert (Section 1.3.3.4). The capillary pressure is inversely proportional to the diameter of the flow path. Thus the pressure at interface 'a' will be less than the pressure at interface 'b'. In consequence of this, if the liquid is to move from B to A, a pressure gradient which is greater than the differential between 'a' and 'b' must exist. It follows therefore that if, in the situation depicted in Figure 2.44, the pressure excess 'y' at B is less than the pressure differential between 'a' and 'b', the liquid will not move. On the basis of this explanation a pressure gradient between A and B could continue to exist.

Having outlined above a mechanism whereby a pressure gradient might be perpetuated, it is appropriate now to make specific reference to spruce and to estimate whether the pressure gradients which are implied in Figure 2.43 are within the range of pressure gradients which the structure of spruce would permit to exist. The first step in this process is to estimate what the magnitude of the pressures at 'a' and 'b' could be.

It may be recalled from Section 1.3.3.4 that capillary force $(P_0 - P_1)$ is given by:

$$P_0 - P_1 = \frac{2\ T_S\ \text{Cos}\ \theta}{r} \qquad \text{Equation 1.11}$$

where P_0 = Gas pressure (Pa)
P_1 = Pressure immediately below liquid surface (Pa)
T_S = Surface tension (Nm^{-1})
$\text{Cos}\ \theta$ = Cosine of the contact angle between liquid and flow-channel wall
r = Radius of flow channel (m)

The liquid in the present experiment was a preservative solution with physical properties closely similar to those of white spirit. Thus T_S in Equation 1.11 takes the value ca. $24 \times 10^{-3} Nm^{-1}$ and $\text{Cos}\ \theta$ may be assigned the value 1, in consequence of the white spirit/timber equilibrium contact angle being close to zero. Using these values, adjusted by a factor to express the results in kPa, Equation 1.11 becomes:

$$P_0 - P_1 = \frac{0.048}{r}\ \text{kPa} \qquad \text{Equation 2.3}$$

where r = Radius of flow channel in mm

If 'a' is located in the lumen of a tracheid, a typical value for 'r' might be 12 μm. A typical value for 'r' if 'b' is a pore in the margo might be 0.05 μm. Assuming these dimensions, values

for the capillary forces at 'a' and 'b' are 4 kPa and 960 kPa respectively. Thus, in the hypothetical situation depicted in Figure 2.44, a pressure of b - a kPa (956 kPa) would be required to initiate movement of the column of liquid from B to A.

Reverting to the central theme of the present discussion it is clear from the above that it is possible for a column of liquid to 'maintain station' within a flow path in a sample of spruce, between the ends of which is a pressure gradient of merely ca. 30 kPa, as seen in sample 28. Indeed, it follows from the above that as liquid is lost by evaporation, interfaces within the timber at locations analagous to 'b', in Figure 2.44, would be unlikely to move. Instead interfaces in positions analagous to 'a' would move towards interface 'b' in response to decrease in liquid volume. Were this to happen in circumstances where B were the atmosphere around a treated sample and A were the pocket of air within that sample, the volume of A would increase and hence its pressure would decrease. This mechanism would account for the decrease in pressure (i.e. increase in pressure gradient) which was observed with sample 28.

Summary of discussion (2.2.3.4.4)

To summarise the principal features noted as a result of measuring the pressure changes within eight samples of seasoned spruce during treatment with organic solvent applied by a range of DVP schedules:

(i) In all cases the rate of pressure change during the initial vacuum phase was approximately linear against log time during most of the evacuation period.

(ii) The differences between the rates of pressure change during evacuation, and the rates of pressure change during immersion under pressure could not be related to differences in the prevailing pressure gradient or the viscosities of the fluids. It is suggested that other factors which may have affected the observed results included the size, shape and abundance of flow paths.

(iii) Unexpectedly rapid pressure-changes were observed during some of the final vacuum phases, and it has not been possible to propose a mechanism to account for these changes.

(iv) After treatment, a pressure gradient between the inside of the sample and its surroundings may persist, even when flow paths apparently exist to enable liquid to move in response to this gradient. A physical basis to account for this is postulated.

2.2.3.4 Measuring pressure inside timber - results and discussion continued - 5

2.2.3.4.5 Post immersion pressure changes

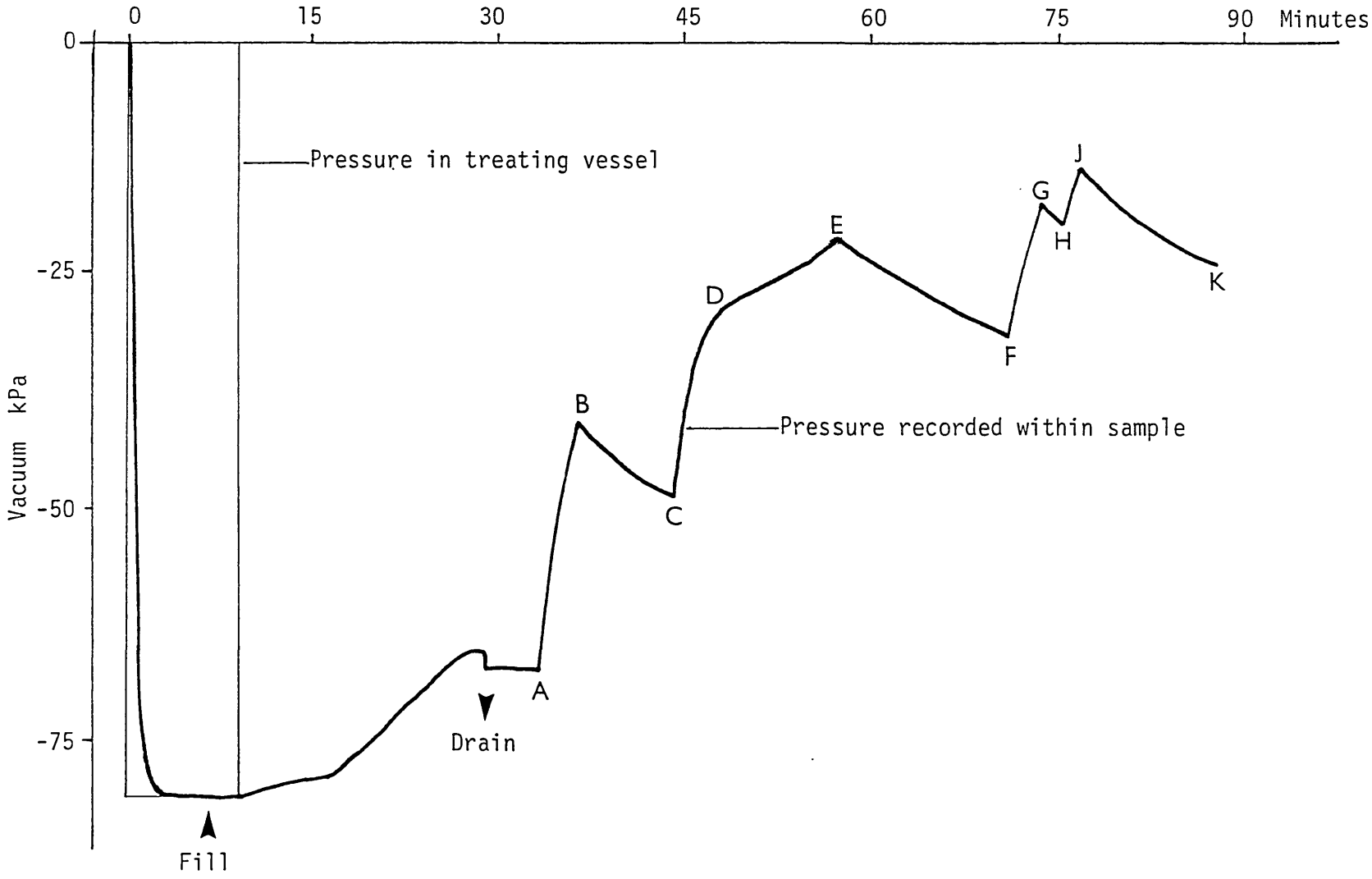
The experimentation described in Section 2.2.3.3.5 yielded results which are presented in Figure 2.45 (Sample I) and Figure 2.46 (Sample II). These two sets of results will be discussed separately.

Sample I

Dealing firstly with the results obtained from Sample I (Fig 2.45) and working through them chronologically, it is clear from the pressure change which took place inside the sample during the initial vacuum period that this was the most permeable of all the spruce samples in which pressure was measured during this investigation. Within three minutes the pressure in the lumena at the centre of the sample had come into equilibrium with the pressure within the treating vessel.

Despite this high permeability of Sample I, when the treating vessel was filled with treating liquid and vented to atmosphere, the rate at which the pressure within the sample returned to atmospheric pressure was initially quite slow. The pressure in the sample increased only ca. 2 kPa during the seven minutes immediately after venting to atmosphere. During the next twelve

Figure 2.45 Pressure changes recorded within spruce Sample 1 : Section 2.2.3.4.5



minutes, pressure in the block increased approximately four times more rapidly such that, nineteen minutes after venting to atmosphere, a total pressure change of ca. 16 kPa had occurred.

The reason why the rate of pressure change inside the block increased with elapsed-time is unclear. Presumably a pathway (or pathways) which was initially blocked, perhaps by an air embolism, subsequently cleared, perhaps by dissolution of air from the embolism into the surrounding white spirit. However, there appears to be insufficient information in these data on which to base more detailed speculation.

During the period 19 to 20 minutes after the vessel was vented to atmosphere, the white spirit was drained from the treating vessel. This caused a drop of 2 to 3 kPa of the pressure within Sample I, following which, for ca. 4.5 minutes, the pressure remained totally unchanged.

This short period of unchanging pressure at the tip of the transducer ended abruptly after 4.5 minutes when the pressure moved rapidly towards atmospheric pressure, and during the ensuing 3.3 minutes the pressure changed ca. 29 kPa. This return to atmospheric pressure also ended abruptly and 7.7 minutes after the vessel was drained, the pressure within the sample began to drop. This drop continued for ca. 7.6 minutes during which the pressure changed ca. 8 kPa.

From Figure 2.45, it can be seen that, in Sample I, pressure fluctuations of the type described above continued throughout the observation period. Unfortunately, for technical reasons, it became necessary to terminate this experiment 54.5 minutes after the vessel was drained.

Before a causal basis for the results presented in Figure 2.45 is postulated, their reliability needs to be examined. The possibility which requires particularly close examination is that they are artefacts of some failure of the experimental system. However, this possibility does not appear to be strong because neither of the plausible faults (discussed below), which might have occurred, is likely to have caused the observed signal fluctuations. A simple leak of air through one of the seals would almost certainly have led to a steady return to atmospheric pressure, and thus the possibility of this having occurred can almost certainly be dismissed. Instrument malfunction also seems unlikely to have caused the observed signal fluctuations. Electrical interference which has been encountered in other experimentation has been observed only to cause sporadic 'spiking' of the pen recorder signal. Slight zero shifting has been noted on occasions (Section 2.2.3.4.4) but has been orders of magnitude less pronounced than the observations under consideration here. It is therefore concluded that the results in Figure 2.45 are real effects arising from the movement of fluids within Sample I.

The conclusion that the fluctuations in pressure which are represented in Figure 2.45 are not due to instrument malfunction is further supported by the fact that the fluctuations appear to follow a pattern. They occur in eight phases of apparently random duration. During four of these the pressure rises (i.e. tends towards atmospheric pressure), and alternating with these are four phases during which the pressure drops (i.e. tends away from atmospheric pressure). In all four phases during which pressure drops (i.e. phases B-C, E-F, G-H and J-K), the average rate at which the pressure drops is similar (average rate 1.1 kPa/min : range 0.9 to 1.3). The pattern discernable during the phases of pressure-rise is less clear cut, but with the exception of the period D-E, the pressure rises during the four phases also occur at similar rates (average rate 5.6 kPa/min : range 4.6 to 6.7). Whilst the physical mechanism underlying these gradients is so far unknown, the similarity of the gradients is interpreted as being a confirmatory indication that the results in Figure 2.45 are real effects arising from the movement of fluids within Sample I.

Although it is not possible to be specific as to the liquid-flow phenomena which might have given rise to the results represented in Figure 2.45, certain general statements can be made.

In defining the basis on which measurements of pressure within timber can be interpreted (Section 2.2.3.1), it has already been established that pressure within a submerged sample varies

inversely with the volume of the substantially fixed mass of gas in the centre of the sample. i.e. pressure increase within a submerged sample results from decrease in the volume of gas in its centre, and the converse. In a sample totally surrounded by liquid, this volume decrease can occur only as a result of inflow of liquid.

In the case of a sample such as the one currently being considered where the immersion liquid has been drained from the vessel, there exist two ways in which pressure increase can occur. One of these is by movement of liquid towards the centre of the sample and the other is by leakage of air through discontinuities in the liquid layer. The speed and abruptness of the pressure increases tend to suggest that periodic leaks of air occurred.

Whereas the above two mechanisms can be suggested to account for increases in pressure within the sample, it is more difficult to postulate a mechanism to account for the observed decreases in pressure, which increased the pressure-gradient between the centre of the sample and its surroundings. Neither of the mechanisms suggested to account for pressure increase can satisfactorily be invoked to explain pressure decrease. For instance, it is quite impossible that air could have leaked out through discontinuities in the liquid, against a pressure gradient. Similarly, the possibility that for rather more than half the observation period the net liquid movement was away from the centre of the sample, against the pressure gradient, is equally difficult to support.

Certainly in Section 2.2.3.4.4 capillary forces and loss of solvent by evaporation were together suggested as the basis of the mechanism underlying small decreases in pressure within samples at the end of the final vacuum phase. However, it seems improbable that this mechanism could give rise to pressure decreases of the magnitude and speed of those currently being considered.

In conclusion, the post immersion pressure fluctuations observed in Sample I, in particular the periodic pressure decreases, are most difficult to explain on the basis of existing assumptions about the behaviour of liquid in timber. In order to come anywhere near devising a satisfactory explanation of these data an alternative basis for their interpretation is required, and this is further explored in the final discussion of this thesis (Section 4).

Sample II

Considering next the results obtained from Sample II (Fig 2.46) and, as with Sample I, working through them chronologically, perhaps the most immediately striking feature to emerge from them is the very low level of permeability displayed by this sample. For instance, during the initial evacuation phase it took ca. 70 hours for the sample to come into equilibrium with its surroundings. To an extent this was not wholly unexpected. The sample was sealed on five surfaces so as to restrict flow to the

radial direction, and previous authors (e.g. Maclean 1935) have noted that the existence of radial flow paths in seasoned softwoods other than pines cannot be relied upon.

In view of Sample II's low permeability, an interesting feature of the results represented in Figure 2.46 is the fact that the pressures registered by the two transducers followed each other quite closely during the evacuation phase. This feature, coupled with the fact that after 24 hours evacuation the pressure registered by transducer Y, 22 mm below the unsealed surface, was actually lower than the pressure registered by transducer X, 7 mm below the surface, might suggest a reticulum of unconnected pathways existed within the sample. Such a configuration would be similar to the one already suggested for the longitudinal flow sample (Section 2.2.3.4.2). However, the discrepancy between the pressures sensed by the two transducers was for the most part less than 5 kPa, and this may have been within the additive limits of linearity and hysteresis error. The alternative possibility that the timber between transducer X and the surface had very low permeability, whilst the permeability of the timber between transducers X and Y was adequate for these two to be in relatively free communication cannot therefore be ignored. Indeed, in some respects this latter possibility presents a more plausible basis for the interpretation of the data.

After 79 hours of evacuation, the treating vessel was flooded with liquid and it was then returned to atmospheric pressure. Following this the pressure at both monitoring points within the sample rose very slowly indeed, such that after 90 hours of immersion it had risen an average of ca. 4 kPa. Over the next 32 hours the vessel was resealed and pressures of up to 207 kPa were applied at intervals as indicated on Figure 2.46. However, these appeared to have negligible effect on the rate of pressure increase and because the treating vessel being used was unsuitable for operating at pressures in excess of 200 kPa it was vented to atmosphere and drained of liquid after a total of 121 hours of immersion.

After the vessel had been drained, the pressures within Sample II were monitored at intervals over 75 days. Overall, the pressure at both monitoring points rose ca. 30 kPa during this time. Within this overall rise there occurred fluctuations such that at some times the higher pressure was registered by transducer X whilst at other times the converse applied. Fluctuations of this kind are difficult to account for and are reminiscent in pattern, though not in magnitude and speed, of the fluctuations which were observed during the post-immersion phase in Sample I.

As the 75 day post-immersion period progressed, and the pressures within the sample changed so slowly, it became clear that the liquid which had been absorbed by Sample II during the 121 hour immersion phase must be forming a near stationary seal at or near

the open tangential surface of the sample.

It followed from the above that removal of the layers of timber in which the liquid was lodged would lead to a relatively rapid return to atmospheric pressure throughout the remainder of the sample. To examine this supposition and to try to gauge the depth to which liquid had penetrated, increments of the epoxy seal were removed, at the time-intervals indicated on Figure 2.46, from one transverse face adjacent to the unsealed tangential face (Fig 2.46). It was considered that providing longitudinal access for the air in this way would have the same effect as totally removing the surface layers. The first increment of 2 mm of end seal was removed 75 days after immersion had started and over the next 8 days a further 5 mm were cut away. Immediately following removal of the 6-7 mm increment, the monitoring of pressure within the sample continued for 3 hours and no change in pressure was observed during this time at either location within the sample. At the end of this 3 hour period the recording apparatus had to be switched off. By the time it was switched on again, 39 hours later, the pressure registered by transducer X had risen to just 3 kPa below atmospheric pressure. (It is assumed that this in fact represented a return to atmospheric pressure, because it is considered that base-line drift of 3 kPa over the 4 months since the start of the experiment was quite possible.)

The pressure registered by the transducer 17 mm below the open surface continued to be ca. 50 kPa below atmospheric pressure for at least 14 more days. Sixty days after the removal of the 7 mm of end seal, the pressure registered by this transducer had risen to ca. 30 kPa below atmospheric pressure and a further 52 days later it had returned to, at or near, atmospheric pressure.

From the pressure changes observed after the removal of end seal several conclusions can be drawn.

Penetration of liquid had clearly extended at least 7 mm because only after 7 mm of end seal had been removed was significant pressure change registered by transducer X. Indeed, tracheids longitudinally aligned with this transducer had open end-grains for at least 7½ hours prior to pressure change taking place. It follows therefore that sufficient liquid was lodged in these tracheids to prevent ingress of air throughout this period. Furthermore, by application of Equation 2.3 it may be calculated that because the liquid in the capillaries preserved a pressure gradient of 50 kPa, the smaller pores in these capillaries cannot have been larger than ca. 1.9 μm in diameter.

The fact that the pressure registered by transducer Y did not change for a substantial period after 7 mm of seal had been removed suggests that the liquid penetration had continued beyond 7 mm and this liquid impeded passage of air to the deeper parts of the sample. Penetration to this depth, and beyond, is quite

consistent with the pressure increase from ca. 91 kPa below atmospheric pressure (ca. 10 kPa absolute pressure) to 82 kPa below atmospheric pressure (ca. 19 kPa absolute pressure) which occurred during the immersion phase. Applying the argument which was developed in Section 2.2.3.4.3.1, it can be calculated that this pressure increase implies that the air within the sample decreased in volume by ca. 48%. Thus, penetration of liquid equivalent to uniform penetration to a depth of 12 mm, may have occurred during the immersion phase. This liquid presumably moved more deeply into the sample during the post-immersion phase.

If true, the above conclusion, that penetration during the immersion phase probably extended 12 mm into the sample, has implications concerning the continuity of the permeating liquid. Because a liquid is incompressible, a pressure gradient cannot exist within a continuous, effectively static body of liquid. Instead, in the event of a pressure being applied to a liquid, that pressure is quickly transferred throughout the body of liquid in question, so that it exhibits the same pressure throughout. Thus, if liquid penetrated 12 mm into Sample II, filling every void as it did so, the pressure registered by transducer X would have risen to the pressure of the liquid surrounding the sample as soon as, or very shortly after, the void at the transducer's tip became filled with liquid. Clearly this did not happen. It would appear therefore that the voids in the zone 0 to 12 mm from the unsealed edge did not all completely fill with liquid. Rather, certain pathways filled with liquid and in

so doing partially sealed off pockets of air into which liquid subsequently flowed.

In conclusion; the results obtained with Sample II indicate that this sample of spruce exhibited very low radial permeability. Certain features of the results are indicative of the sample containing a reticulum of flow paths, some of which contained constrictions which narrowed its diameter to not more than ca. 1.9 μm.

Aside, a final point which the results from Sample II highlights is a more general one relating to experimental design. It has been noted above that a comparatively small increase in pressure during the immersion phase was probably indicative of one third of the whole sample becoming filled with liquid. This increase in pressure was so small because in the 90%-evacuated sample, the 9 kPa pressure increase in question corresponded to a 90% pressure increase in the sample, which resulted in turn from a 48% volume decrease. In other words, when the absolute pressure within the sample is low at the start of the immersion phase, even quite large influxes of liquid result in only rather small increases in pressure. This, coupled with the uncertainties mentioned in Section 2.2.3.4.1 concerning the precise effect on calibration of slight lateral deformations of the transducer, leads to this technique being unsuitable for precise interpretation and therefore rather insensitive. Thus, whilst the measurement of pressure inside timber is a valuable technique for examining

gross trends, (e.g. Kyte & Saunders 1978) it has restricted value with regard to the more precise investigation of the flow of liquid into evacuated timber.

Errata

Throughout this thesis, the expressions 'gradient', pressure gradient' and 'pressure gradients' should respectively read 'differential', 'pressure differential' and 'pressure differentials'.

3. Evaluation of the performance of timber preservative impregnation schedules

In Section 1.6 the three principal aims of this investigation were set down.

The first of these aims was "To develop techniques to monitor the fluid flow phenomena which occur during the process of timber treatment", and this has been the focus of the whole of Section 2 in which the development and use of three techniques have been described.

The second and third aims, as detailed in Section 1.6 (q.v.), were to a large extent pursued simultaneously and the present section (Section 3) describes the work done and results obtained in connection with these aims.

Section 3.1 describes firstly the techniques used to quantify uptake of preservative (Section 3.1.1) and its penetration into spruce (Section 3.1.2). This section then describes the techniques employed for the comparative evaluation of impregnation schedules (Sections 3.1.3).

Section 3.2 has two parts. Section 3.2.1 describes the experimentation which was undertaken using the techniques described in Section 3.1. The results of this work are then presented and discussed in Section 3.2.2.

3.1 Techniques for the evaluation and comparison of the performance of industrial timber preservative impregnation schedules

3.1.1 Assessment of uptake

In this section of this investigation, preservative uptake was assessed gravimetrically. Each sample was weighed immediately before impregnation, and again immediately afterwards, and uptake was calculated by subtraction.

The untreated weight of the samples used (Section 3.2.1) was typically in the range 150 - 200 g. Uptakes varied widely, most often in the range 10 - 30 g. The accuracy with which each sample was weighed was $\pm$ 0.2 g.

It will be seen later (in Section 3.1.3) that most of the experimentation undertaken involved batches of 75 samples. Weighing each batch of samples after treatment usually took not more than 20 minutes.

3.1.2 Assessment of penetration

Information concerning the distribution of preservative within a treated sample is of at least as much practical importance as information about the total quantity of preservative absorbed by a sample during treatment. Gathering and recording such information is, however, a much more complex and much less precise procedure than measuring uptake. Choices must be made concerning whether distribution and penetration are to be assessed visually or by chemical analysis, whether examination is made of lateral or transverse surfaces (or both), and whether penetration is to be measured in terms of, for instance, 'average depth of penetration' or whether some other procedure is more appropriate.

In this present investigation the penetration assessment technique was developed as the work progressed. The technique which was finally adopted and which was used for most of the work described in Section 3.2.1 is set out in Section 3.1.2.2. The development and basis of this technique is outlined in Section 3.1.2.1.

3.1.2.1 Development of a technique for penetration assessment

Lateral penetration of preservative into spruce is frequently irregular (Plate 3.1). In such instances 'depth-of-penetration' measurements which are made at a relatively few locations around the sample do not adequately describe the pattern of penetration.

Plate 3.1 Whitewood, cross cut after impregnation with blue-dyed organic solvent preservatives, exhibiting irregular lateral penetration

Plate 3.2 Whitewood, cross cut after impregnation with blue-dyed organic solvent preservative, exhibiting more extensive penetration into the 'sapwood corner'than into the 'heartwood corner'. Note isolated island of 'broken' penetration, further discussed in text (Section 3.1.2.1).

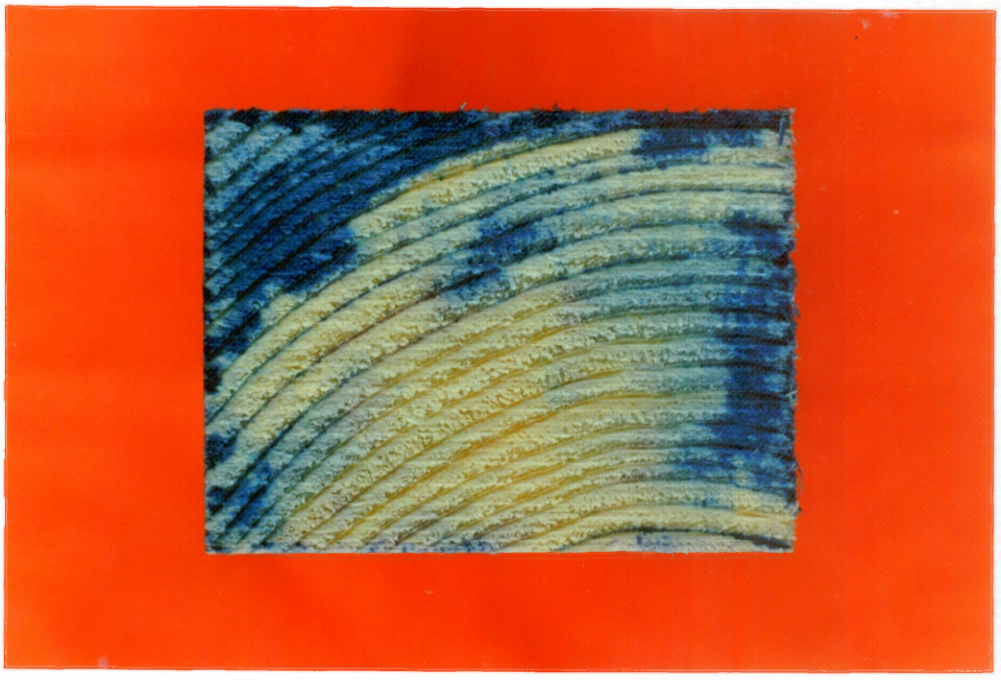


Plate 3.1

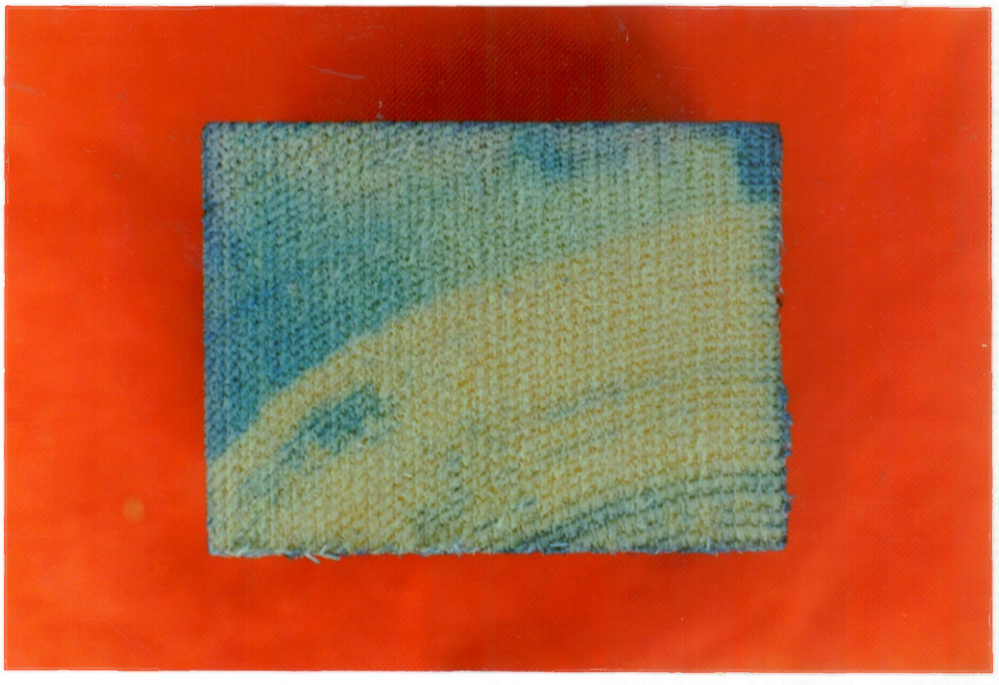


Plate 3.2

For example, the depth of the lateral penetration in Plate 3.2 could be assigned values between zero and 16 mm, depending upon where measurement is made. Moreover, for the sample shown in Plate 3.2, no value for 'average depth of penetration', however calculated, can convey much real information.

Attention was therefore focussed on developing a technique with which to make a quantitative description of penetration. The principal aspects which were considered during the course of developing this technique were those which are outlined in the remainder of this section (3.1.2.1).

Location of preservative within treated timber

In order to address the task of quantifying the extent of preservative penetration, it is obviously necessary to have a procedure for locating preservative within treated timber. The techniques which were considered for this purpose were quantitative and qualitative chemical analysis, bioassay and the use of tracer dyes.

Quantitative chemical analysis was rejected both because it would have been prohibitively time-consuming and also because, given the analytical facilities available (wet chemistry, Atomic Spectrophotometry and Gas Liquid Chromatography) this technique would have involved the extraction of the preservative's active ingredients from a relatively large mass of timber. For this

latter reason this technique would have been rather a 'blunt instrument' for the precise and detailed mapping of the pattern and extent of penetration.

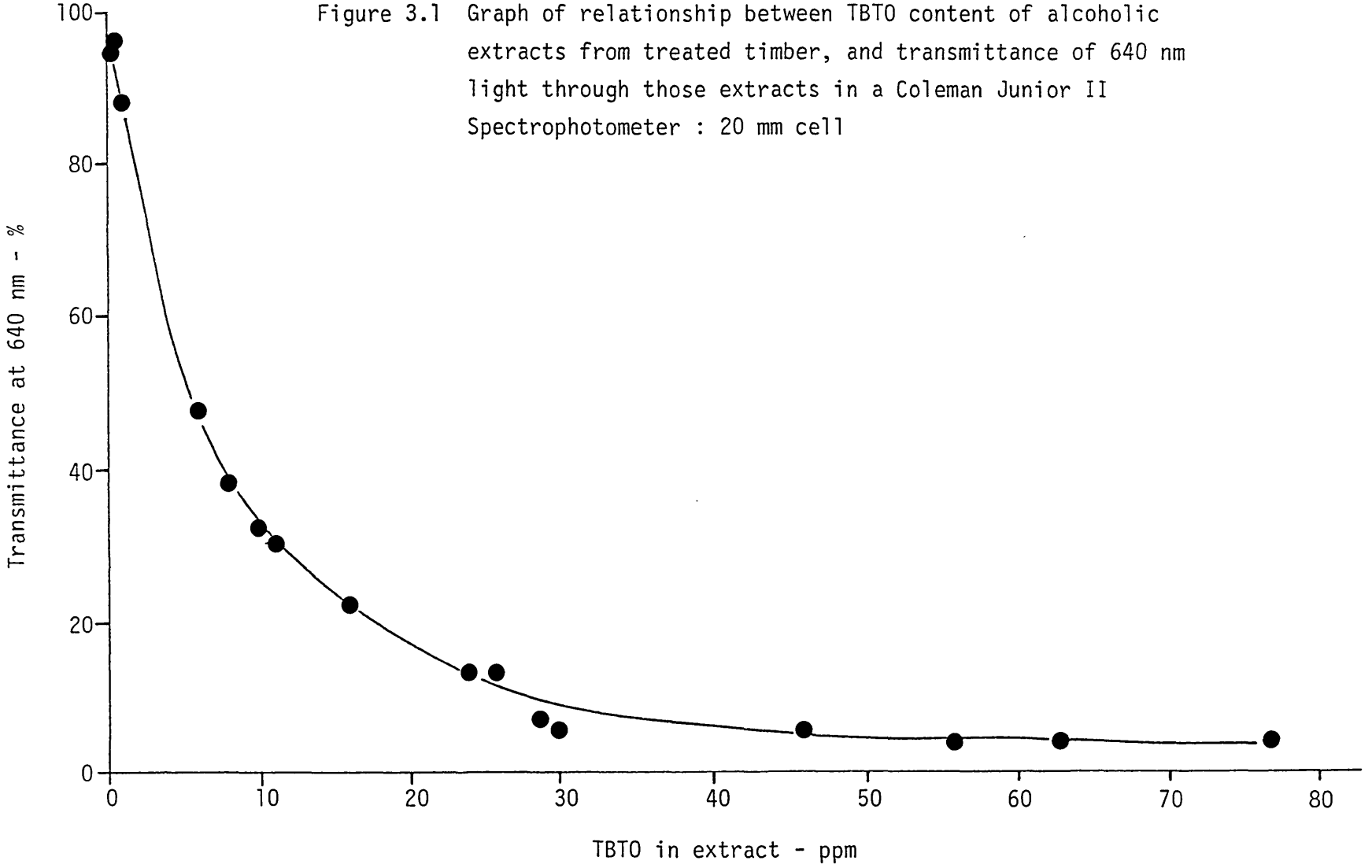
Qualitative chemical analysis, in the form of the application of reagents which produce coloured reaction-products on contact with certain of the components of the preservative, was evaluated but, like quantitative analysis, was rejected. This was because of uncertainties regarding the sensitivity of the available reagents to low concentrations of preservative.

Bioassay techniques such as that described by Sutter (1978) were rejected for the present work because, like qualitative analysis they were judged to be prohibitively time-consuming, particularly as a substantial amount of method-development was clearly required.

After the consideration of the alternatives outlined above, oil-soluble dyes were selected as providing the most satisfactory technique for locating preservative in treated timber. It may be recalled that in Section 2.1, oil-soluble dyes were rejected for the purpose of locating individual microscopic flow pathways. However, it was apparent the technical requirements of a dye to facilitate macroscopic examination of penetration were very different. In addition it was noted that the oil-soluble dye 'Waxolene Blue A' had been selected by other workers (BS 5707 1979) for following longitudinal penetration in pine sapwood.

The possibility that, due to chromatographic effects, Waxolene Blue A might not give a complete indication of the extent of penetration was considered. In preliminary tests, no evidence of chromatographic separation could be found in model systems using chromatography and filter papers. Further preliminary experimentation was conducted in which seasoned European whitewood was treated with an organic solvent preservative to which 0.5% m/m Waxolene Blue A dye had been added. After drying, alcoholic extracts of portions of treated timber were analysed both for tin (Williams 1973), by atomic absorption spectrophotometry, and for blue dye content by measuring the transmittance of light at 640 nm through the extracts. Figure 3.1 shows the correlation which was obtained between tin content and transmittance. On this basis, 0.5% m/m Waxolene Blue A was selected for addition to treating liquids to facilitate the location of preservative in treated timber.

Figure 3.1 Graph of relationship between TBTO content of alcoholic extracts from treated timber, and transmittance of 640 nm light through those extracts in a Coleman Junior II Spectrophotometer : 20 mm cell



Drying time

In the literature there exists a divergence of opinions concerning the minimum period of drying-time which needs to elapse between treatment and penetration assessment. Tesoro & Choong (1976) and Choong & Fogg (1972) opened up timber samples and assessed penetration of preservative fairly shortly after impregnation. However, when a similar practice was adopted in the present work it was found that blue-dyed preservative from the penetrated regions 'bled' across the transverse cut surface at a rate great enough to interfere with penetration assessment. In the light of this it was considered desirable to allow the samples to dry in the laboratory so that all the residual solvent evaporated prior to penetration assessment being undertaken. Accordingly a preliminary experiment was conducted in which the loss of solvent from treated samples stacked 'in-stick' in the laboratory was monitored. The results of this experiment (Fig 3.2) indicated that the time required for all the solvent to be lost was rather long. As a practical compromise it was therefore decided to allow an interval of at least 28, and preferably 42, days between treatment, and cutting the samples for penetration assessment. Additionally, it was decided to cut at any one time only as many samples as could be assessed within the following 15 - 20 minutes.

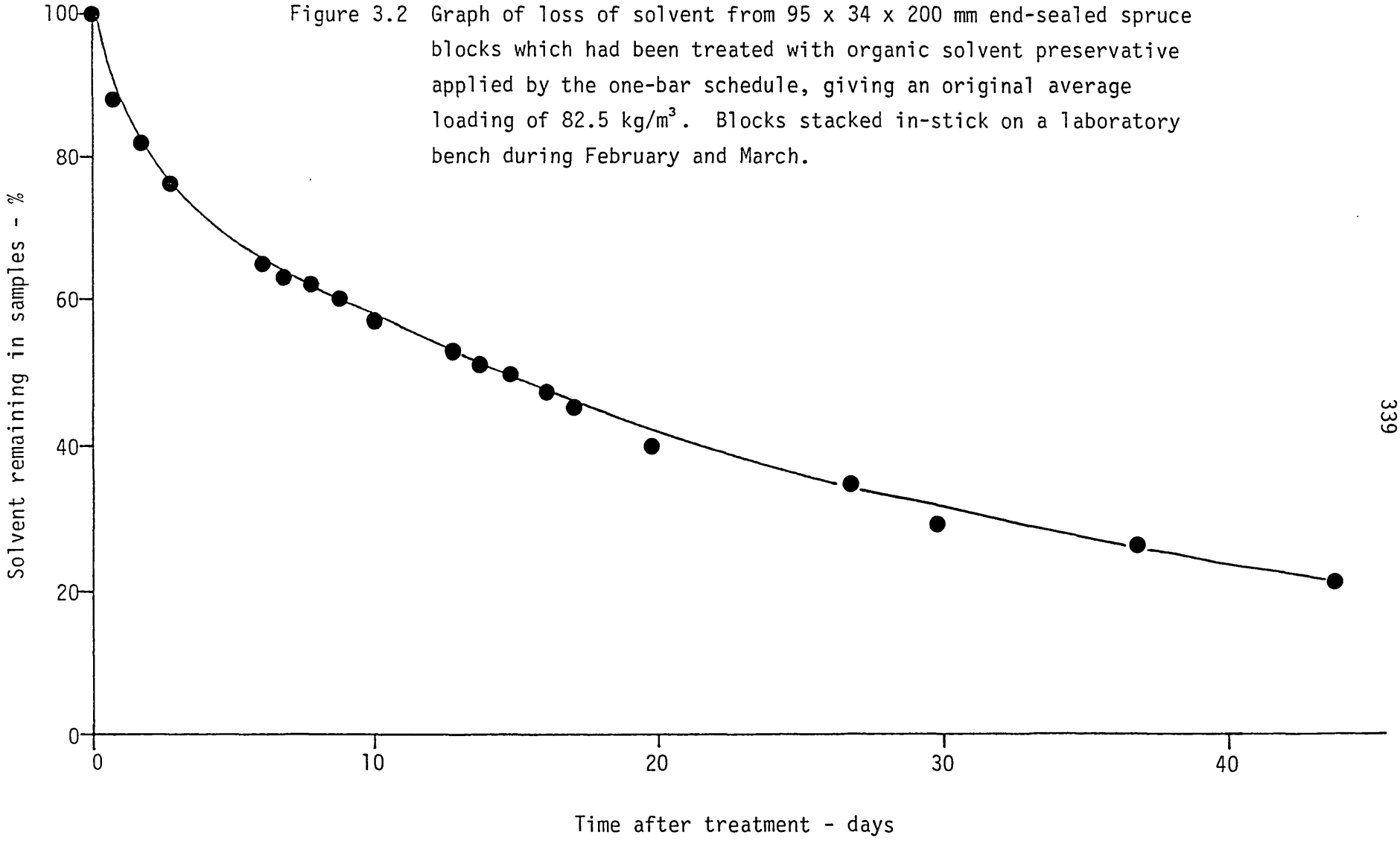


Figure 3.2 Graph of loss of solvent from 95 x 34 x 200 mm end-sealed spruce blocks which had been treated with organic solvent preservative applied by the one-bar schedule, giving an original average loading of 82.5 kg/m^3. Blocks stacked in-stick on a laboratory bench during February and March.

Measurement of penetration

Surfaces examined

Longitudinal penetration can be satisfactorily examined on cut or split longitudinal surfaces (see for example Choong & Fogg (1972) and Tesoro & Choong (1976)). However, in the literature there appears to be no concensus as to whether lateral penetration is best measured on longitudinal or transverse surfaces. Workers such as Tesoro et al (1966) examined longitudinal surfaces while Behr (1975) examined transverse surfaces. To assess these two approaches several samples from the matched triplet test (q.v. 3.1.3.3) were cross-cut and examined.

From the samples examined it was clear that when lateral penetration is to be assessed, as in the present work, examination of a relatively few longitudinal, split surfaces is satisfactory only when penetration is fairly uniform. If preferential penetration into either the earlywood or the latewood is encountered, the results from longitudinal surfaces can only be interpreted in conjunction with a transverse surface. Indeed, this applies to all samples in which penetration is irregular.

Plate 3.3 illustrates that the profile of lateral penetration tends not to change dramatically along a short, straight-grained sample. Variation is caused by major anatomical irregularities in the wood such as large knots or major deviations between the

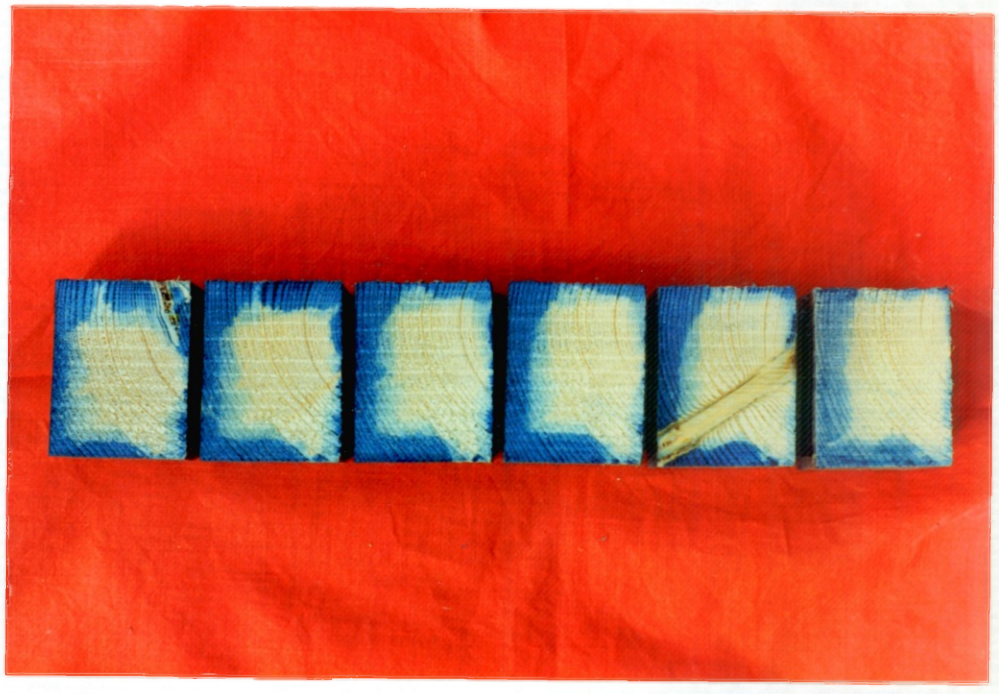


Plate 3.3 Seasoned whitewood impregnated with blue-dyed organic solvent preservative and cross cut at 20 mm intervals, demonstrating the comparative consistency of pattern of lateral penetration, except where disrupted by major anatomical irregularities. Sample also shows 'two-tone' penetration - see text (Section 3.1.2.1).

axes of the axial tracheids and the longitudinal axis of the timber sample. However, timber exhibiting features of this kind is largely eliminated during grading.

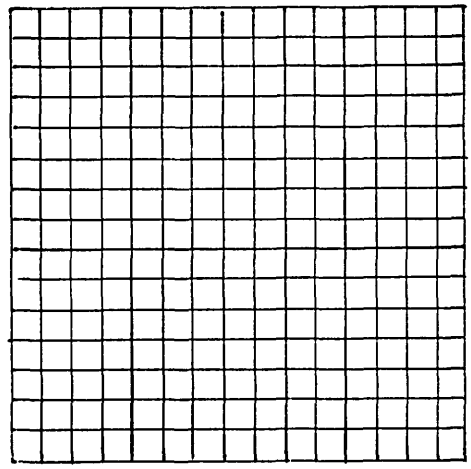
In consequence of the above it was decided that an adequate map of preservative distribution in a short sample, such as that used for the work described in Section 3.1.3.3, could be obtained by examining two transverse faces of each sample.

Quantitative assessment of penetration

After concluding that penetration was best measured by incorporating a blue dye into the treating solution and examining transverse surfaces at least 28 days after the timber samples were treated, the next logical step was to develop a method by which to make some quantitative expression of the observed penetration. The pattern of penetration into spruce is such that the method used had to be suitable for use where penetration was highly irregular.

An obvious technique for measuring an irregular area is to subdivide it into squares of known area and count these. This technique was evaluated. To effect conveniently the subdivision into squares, a grid was drawn onto a clear colourless acetate sheet which was then lain over the cut end of the sample being examined (Specimen 1).

Specimen 1



While square counting is theoretically an excellent technique, approximately two years' experience with its use, for this particular purpose, revealed several practical problems.

To achieve maximum theoretical accuracy each square should be as small as possible. However, examining large numbers of small squares is prohibitively tiring to the eyes. Additionally, because the lines delineating the squares must be thick enough to be seen readily against a dark background they obscure a significant proportion of the area of interest when small squares are used. Thus the practical accuracy of estimations of area

obtained by counting large numbers of small squares will almost certainly be poorer than the theoretical accuracy.

To minimise the problems associated with counting large numbers of small squares, it was decided to use 4.0 mm sided squares to subdivide the field of view. However, inaccuracy can arise if such comparatively large squares are scored on a 'go/no-go' basis. For example the cut surface under a squared overlay may score 30 totally penetrated squares, 30 totally unpenetrated squares and 30 partly penetrated squares. In this example, counting totally penetrated squares would indicate 33.3% of the cross-section penetrated. Conversely, counting only completely unpenetrated squares would indicate 66.6% of the cross-section penetrated. It therefore became necessary to estimate the percentage penetration in each partly penetrated square and this of course introduced a substantial degree of subjectivity into the technique.

When assessing samples such as that in Plate 3.4, estimating percentages in part squares presents no particular problems and should introduce no grave error. However, when assessing samples such as the one shown in Plate 3.5, part square estimation becomes far less accurate. This is because not only is it necessary to estimate how many blue flecks equal one square, it is also difficult to decide whether an area is blue or not. In many specimens there is no clear demarcation between penetrated and unpenetrated regions. The blue dye simply becomes decreasingly clear. In such instances the decision as to where precisely the dye becomes too

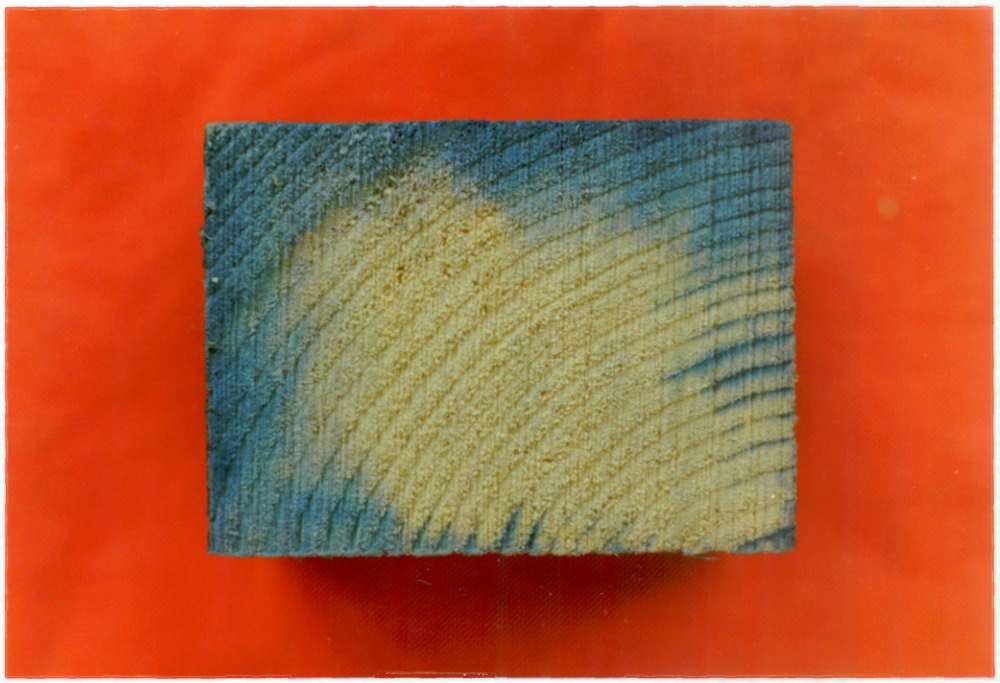


Plate 3.4 Whitewood impregnated with blue-dyed organic solvent preservative. For narration see text (Section 3.1.2.1).

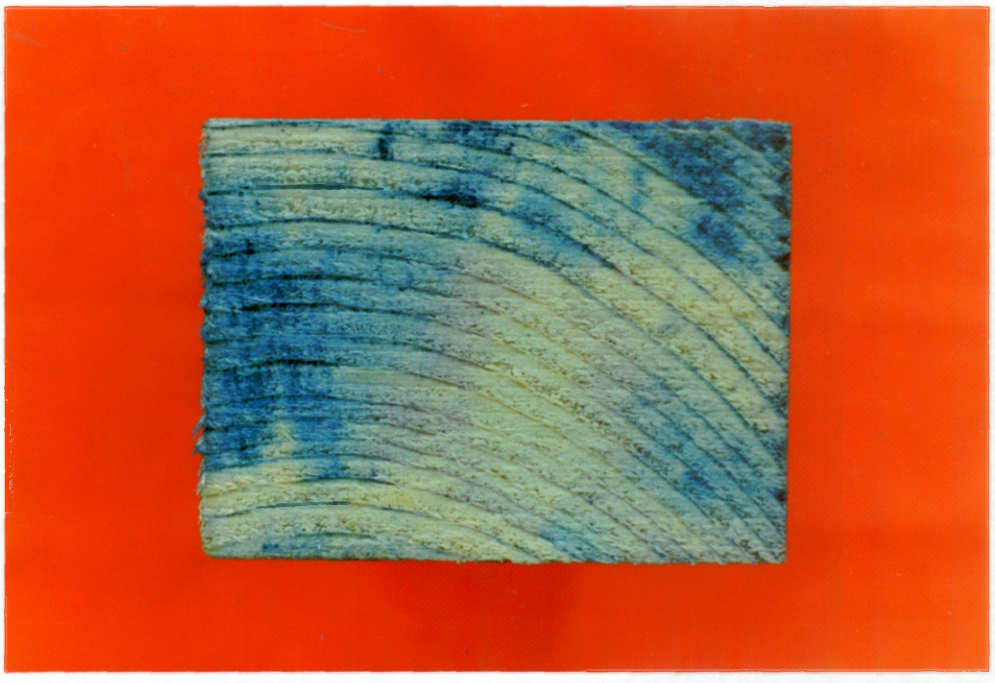


Plate 3.5 Whitewood impregnated with blue-dyed organic solvent preservative. For narration see text (Section 3.1.2.1).

faint to count may be biased by factors such as the sharpness of the saw used to cut the sample, the general level of colour in the specimen and the cleanliness of the acetate overlay. (The overlays discolour in use and are difficult to clean satisfactorily without removing the lines.)

The most serious problem with this technique is encountered with samples such as that in Plate 3.5. If the area coloured blue is assessed, and then the area not so coloured is also determined, the sum of the two areas may not reliably approximate to the total area of the sample. i.e. When penetration is particularly irregular or diffuse the part-square estimation procedure may be the source of significant inaccuracy. When this inaccuracy came to light, use of the square-counting technique for estimating penetration was discontinued.

To summarise:

In practice the area estimation technique outlined above was found to have three principal drawbacks.

1. Counting large numbers of small squares was prohibitively tedious and difficult for more than short periods of time.

2. When larger squares were used, part-square estimation may be a source of significant error on samples where penetration is particularly diffuse or irregular.

3. The decision as to when the blue dye is too faint to count is subjective and as such is a potential source of inconsistency in assessment.

Several ways of minimising or circumventing these problems were examined and evaluated including the use of a digitiser to facilitate area estimation, and the use of an electronic image analyser to eliminate subjectivity in the definition of penetrated regions. All the techniques assessed were deficient in some respect and had to be rejected.

It was eventually concluded that, despite its limitations on some samples, visual assessment of blue dye distribution was the best option available. Indeed, its drawbacks became much less significant when square counting was replaced by an alternative technique in which discrete points were examined instead.

Measurement by examination of sample points

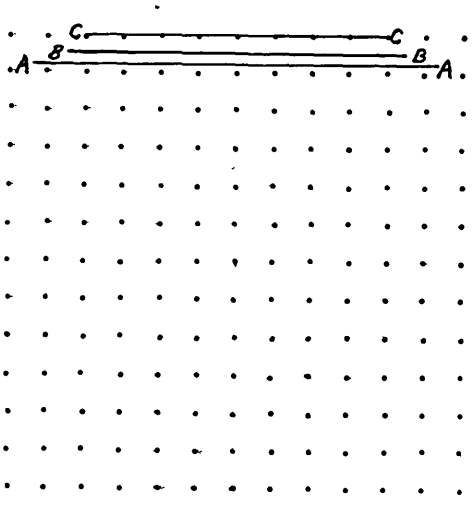
This technique was based on the widely used principle of random sampling and it consisted of examining a number of points selected at intervals on the cross-cut end-grain of each sample. Taking advantage of the irregularity of the penetration patterns, the points examined were not in fact distributed randomly but were evenly spaced (usually at 5.0 mm centres)

To select the points for examination a transparent acetate overlay (Specimen 2) was placed across the cut end of the sample such that:

(i) The maximum number of points was over the sample.

(ii) The distance between the edges of the sample and the outermost line of dots over the sample was roughly equal for opposite sides (Plate 3.6).

Specimen 2



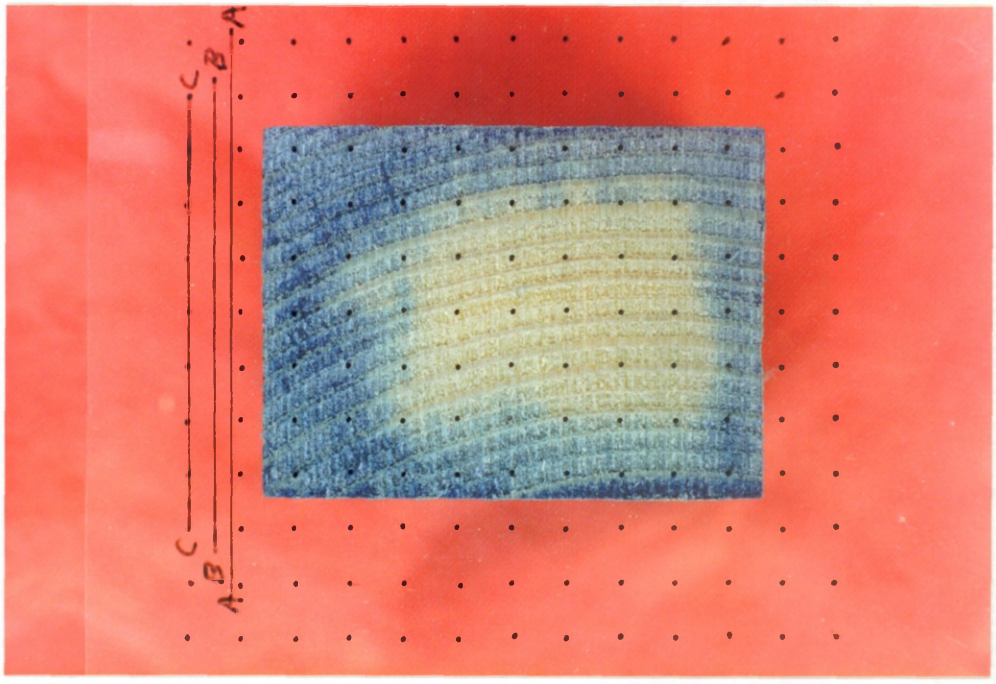


Plate 3.6 Transparent acetate sheet marked with dots at 5 mm centres positioned over a sample of whitewood, impregnated with blue-dyed organic solvent preservative, ready to determine 'Penetration Index'. Note lines A-A, B-B and C-C for measuring penetration at different depths. Note also whitewood sample shows a 'continuous envelope' of penetration - see text (Section 3.1.2.1).

Each dot was scored according to whether it overlay a penetrated or unpenetrated region of the cross-cut surface. The resultant scores were used to calculate an 'Index of Penetration' in the following way.

$$\frac{D\ p}{D\ t} \times 100 = P\ I \qquad \text{Equation 3.1}$$

where : D p = Number of dots overlaying penetrated regions
D t = Total number of dots examined on surface of sample end
P I = Index of Penetration

As an example of the use of this technique, in Plate 3.6.

D p = 41
D t = 63

and therefore

P I = 65

The 'Penetration Index' thus has values between zero (unpenetrated) and 100 (totally penetrated) and, as a first approximation, it may be considered roughly equivalent to an estimate of percentage of cross-section area penetrated. It is not an accurate percentage however because the technique consistently does not include the outer millimetre or so of each sample. Thus a 5.0 x 3.5 cm sample having a uniform 1 mm deep border or 'envelope' of penetration

would score PI = 0 whereas in fact 9.6% of the cross-cut surface is penetrated.

Measurement of depth of penetration

As this work progressed it became clear that while a measure of the overall degree of penetration is an important one, it would not satisfactorily stand alone. For example, PI = 50 could indicate (on a 5 x 3.5 cm sample) a uniform envelope of penetration between 3 and 7 mm deep, or equally it could indicate one half of the sample totally penetrated to 2.5 cm, and the other half not penetrated at all. It was clearly necessary therefore to supplement measurement of the Index of Penetration with information about depth of penetration.

A convenient way to assess depth of penetration was found to be a line drawn (line A-A on Specimen 2), 1 mm from a row of dots. The acetate overlay was positioned such that this line coincided with each edge of the sample in turn and the dots 1 mm away were scored according to whether they coincided with blue-coloured timber. The two distal dots of the line were ignored on each occasion to eliminate the possibility of duplicating assessments at the corners of the samples

This procedure was duplicated with lines 2.5 and 5 mm away from a convenient row of dots (lines B-B and C-C on Specimen 2). From these observations an estimate of the percentage of the surface of the sample under which penetration occurred at 1 mm, 2.5 mm and 5 mm was calculated.

Patterns of penetration

There appears to be little information in the literature concerning the pathways followed by preservative liquids flowing into spruce. Indeed, after briefly reviewing the techniques available (Section 2.1), it was concluded that it was not practically possible to devise experimentation in order to investigate these pathways individually. As an alternative, the penetration assessment procedure already described was therefore supplemented when possible by qualitative observation of certain gross features of the patterns of penetration observed. Individually, each group of qualitative observations was recognised as being rather a 'blunt-instrument' as a means of elucidating the routes taken by liquids flowing into spruce. However, large numbers of such groups of observations taken together provided a useful insight into the behaviour of preservative liquid within spruce.

The following are the features which were noted and scored on a form similar to that shown in Figure 3.3.

Figure 3.3 Design of results sheet used for recording penetration assessment data.
For explanation see text (Section 3.1.2).

RESULTS SHEET NO: CYCLE:

Sample No.	Size Dots	Dots Pen'td	% Gross Pen'tn	mm - Depth of Lateral Penetration - mm												Cont. Env.	Dis. Env.	Banded	Broken	Zero	E.W. Preferred	Zero	L.W. Preferred	'Sapwood'	Radial	No. of rings in 10 mm	Two Tone
				Side 1			Side 2			Side 3			Side 4														
				1.0	2.5	5.0	1.0	2.5	5.0	1.0	2.5	5.0	1.0	2.5	5.0												
Average																											

Continuous or discontinuous envelope ('Cont. Env.' & 'Dis. Env.' respectively in Figure 3.3)

On each sample examined, it was noted whether the outer few millimetres of the sample were penetrated with preservative, which therefore surrounded the central portion of the timber by a continuous envelope of preserved timber (Plate 3.6). The alternative to this was that the envelope of preserved timber was 'discontinuous'. i.e. There were portions of the sample where penetration of preservative was so shallow as to be negligible (Plate 3.4).

Banded penetration

During preliminary work it was noticed that a sample might be quite deeply penetrated, save for a band of a few adjacent annual rings into which preservative had not gained access. The converse was also observed. The frequency of this sort of penetration pattern (exemplified in Plate 3.7) was noted.

Broken penetration

Although the pattern of penetration most frequently encountered was the one in which a more or less continuous envelope of preservative treated timber surrounded an untreated central portion in each sample, a pattern which was also noted was one in which apparently isolated evidences of penetration occurred at irregular intervals over the sample's cross-section (Plate 3.2). The frequency of this was noted.

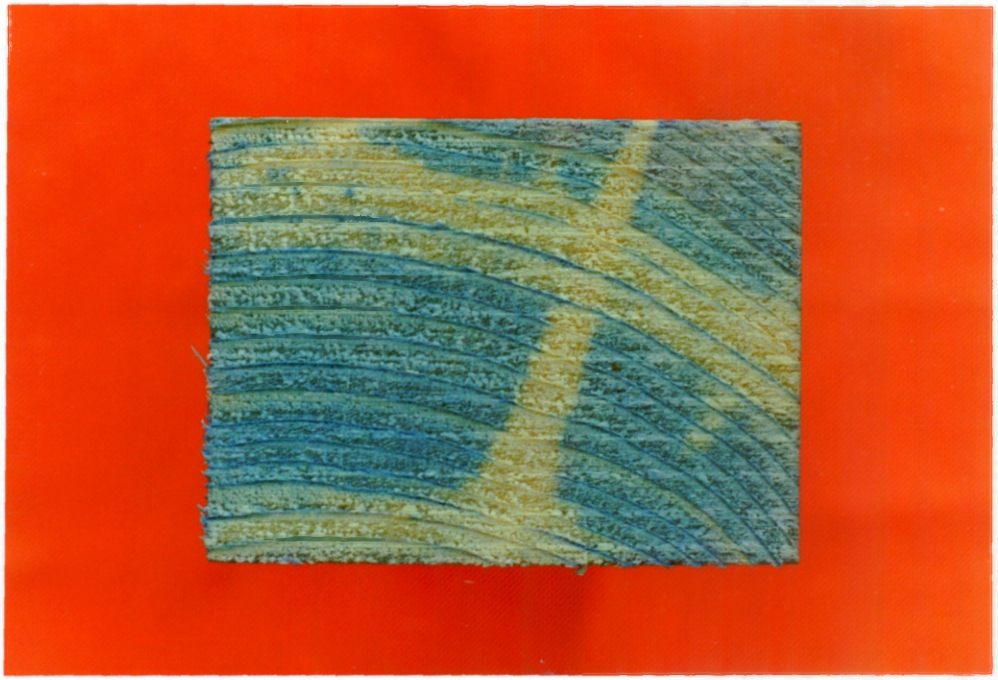


Plate 3.7 Seasoned whitewood showing 'banded penetration' of blue-dyed preservative. The radial discontinuity in penetration is assumed to be related to lateral branch formation. Note also 'Zero' penetration into both earlywood and latewood.

'Zero' and 'preferential' penetration into the earlywood (E.W. on Figure 3.3)

Each sample was examined to establish whether at any point the penetration into the earlywood was so low as to be immeasurably small, or 'zero' (Plate 3.7). The samples were also each examined in order to look for evidence whether, anywhere on the sample, penetration of preservative liquid had proceeded preferentially along the earlywood.

'Zero' and 'preferential' penetration into the latewood (L.W. on Figure 3.3)

A set of observations corresponding to those described above were made with respect to the latewood.

'Sapwood' penetration

Samples were examined to establish whether penetration was deepest in the corner of the sample which had been nearest the periphery of the log from which the sample was originally cut (Plate 3.2).

Radial penetration

In order to try and gain indications concerning the role of the ray tissue in the movement of preservative into spruce, the frequency of evidence of liquid movement in ray tissue was noted (Plate 3.8).

Plate 3.8 Whitewood, cross cut after impregnation with blue-dyed organic solvent preservative showing evidence of the ray tissue having been a pathway for liquid flow

Plate 3.9 Whitewood, cross cut after impregnation with blue-dyed organic solvent preservative showing abrupt 'two-tone' penetration. This effect is evident to a less marked degree in, for example, Plates 3.1 and 3.8.

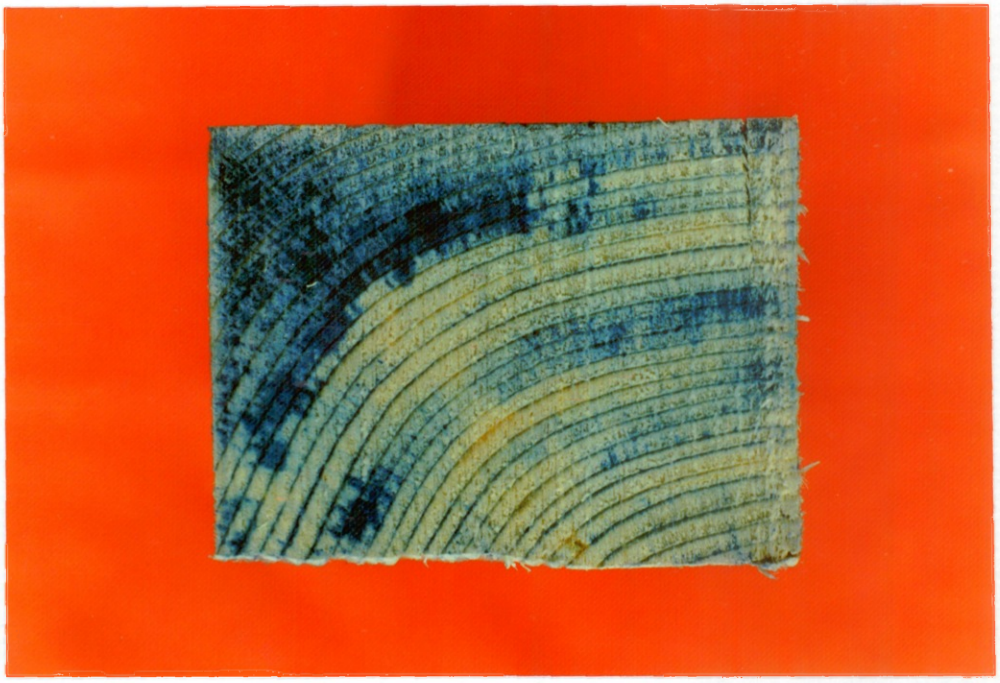


Plate 3.8

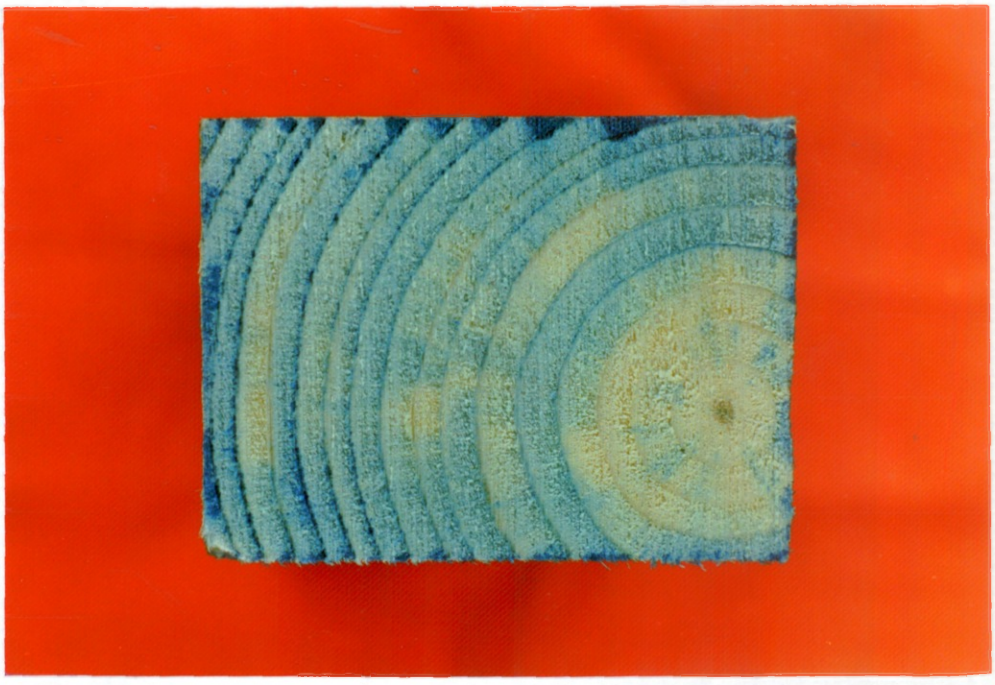


Plate 3.9

Width of growth rings

The number of growth rings in 10 mm was noted.

Two-tone penetration

In many samples the intensity of colour of blue dye was darkest nearest the outside of the sample and diminished towards the centre of the sample. In some instances this diminution of intensity was not gradual, but occurred abruptly (Plate 3.9). The frequency of this phenomenon was scored.

3.1.2.2 Synopsis of penetration assessment technique

After allowing adequate time for the solvent to evaporate, each sample in turn was cross-cut in two places at 5 cm from each end, avoiding large knots (Fig 3.4). As an arbitrary convention, with the sample number reading from left to right, the two cut surfaces on the left of the sawcuts were selected for examination.

Each sample was then rotated into a position where the corner which was nearest the heart of the original tree occupied the bottom left position (Fig 3.5). The longer face adjacent to this corner was designated 1,the shorter one 2, the long face opposite 1 was designated 3 and the remaining short face was designated 4 (Fig 3.5).

Within a maximum of 20 minutes of cutting, an acetate overlay (Specimen 2) was positioned over the cut end as indicated in Plate 3.6. The index of penetration was then determined in the manner outlined above and recorded on a form similar to Figure 3.4.

The percentage of each lateral face penetrated with preservative at 1.0, 2.5 and 5.0 mm was also determined using the procedure outlined above.

Finally, the presence or absence of certain gross features of the pattern of penetration was noted.

Figure 3.4 Cutting-pattern for penetration assessment

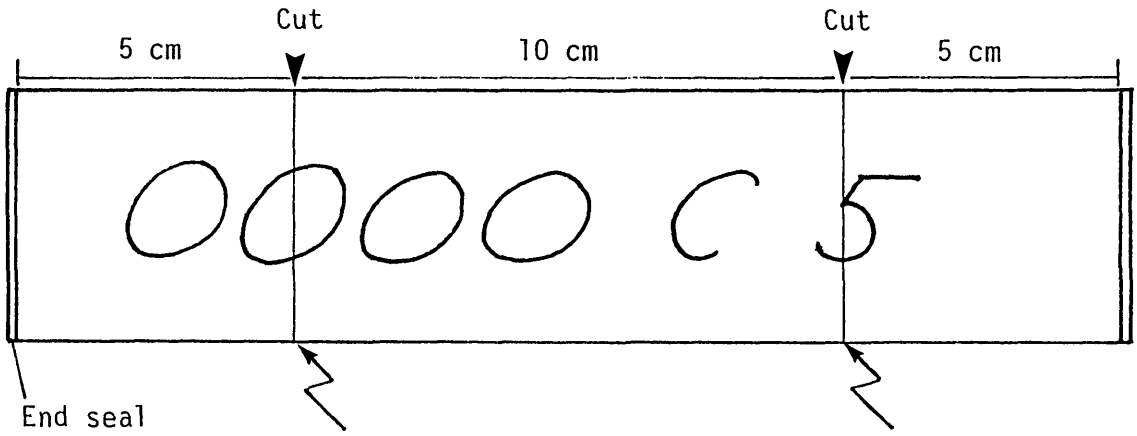
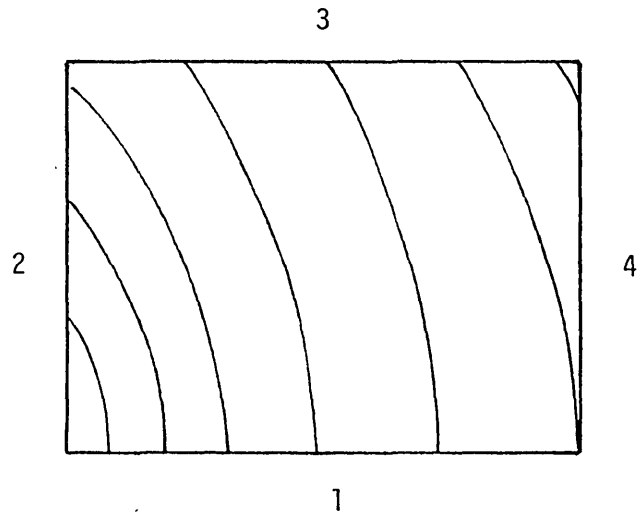


Figure 3.5 Convention used for numbering sides of samples during penetration assessment



3.1.3 Comparison of timber impregnation schedules

The third of the three aims of this investigation which were stated in Section 1.6 was "To investigate means whereby the techniques available for the industrial impregnation of spruce may be improved".

Industrial impregnation schedules are complex processes involving several phases and a number of independently variable parameters (see Section 1.5.3). From investigations such as those described in Sections 2.2.1, 2.2.2 and 2.2.3, it is possible to predict in broad terms the ways in which modification of the parameters of an existing impregnation schedule may change the performance of that schedule. However, the possible interactions of the variables in an industrial impregnation schedule are such that evaluation of modifications must, at some stage, involve their incorporation into the schedule in question. A comparative evaluation of the performance of the modified schedule, vis-a-vis the performance of an unmodified standard schedule must then be undertaken. The development and description of a technique for performing such comparative evaluations are the subjects of this section.

As a prerequisite for designing an experimental system for comparing timber impregnation schedules, criteria had to be set concerning the sensitivity required for the technique. Somewhat arbitrarily, it was decided that the technique should provide a means of being 90% sure of detecting differences of 10% or more in the net

preservative uptake resulting from different schedules. Having set these performance criteria, investigation of a number of experimental designs was undertaken in order to establish which would most efficiently and economically give the desired performance.

3.1.3.1 Variability and its implications for experimental design

The particular difficulties encountered when seeking to conduct precise impregnation experimentation using spruce, arise from the very variable degrees to which different samples of timber absorb preservative. The extent of the variability in preservative uptake is indicated by data presented in Figure 3.6, and similar within-species variability has also been encountered by other workers (Becker & Starfinger (1970), Behr (1975), Purslow (1958)).

In order to design an experimental system of a specified sensitivity, quantitative information about the variability of the test substrate is required, and standard statistical techniques exist for deriving this. The estimate of variability which is most frequently used is the 'standard deviation'. To obtain a value for standard deviation, any convenient number of samples are subjected to similar treatment and numerical measures of their reaction to treatment are processed using the equation:

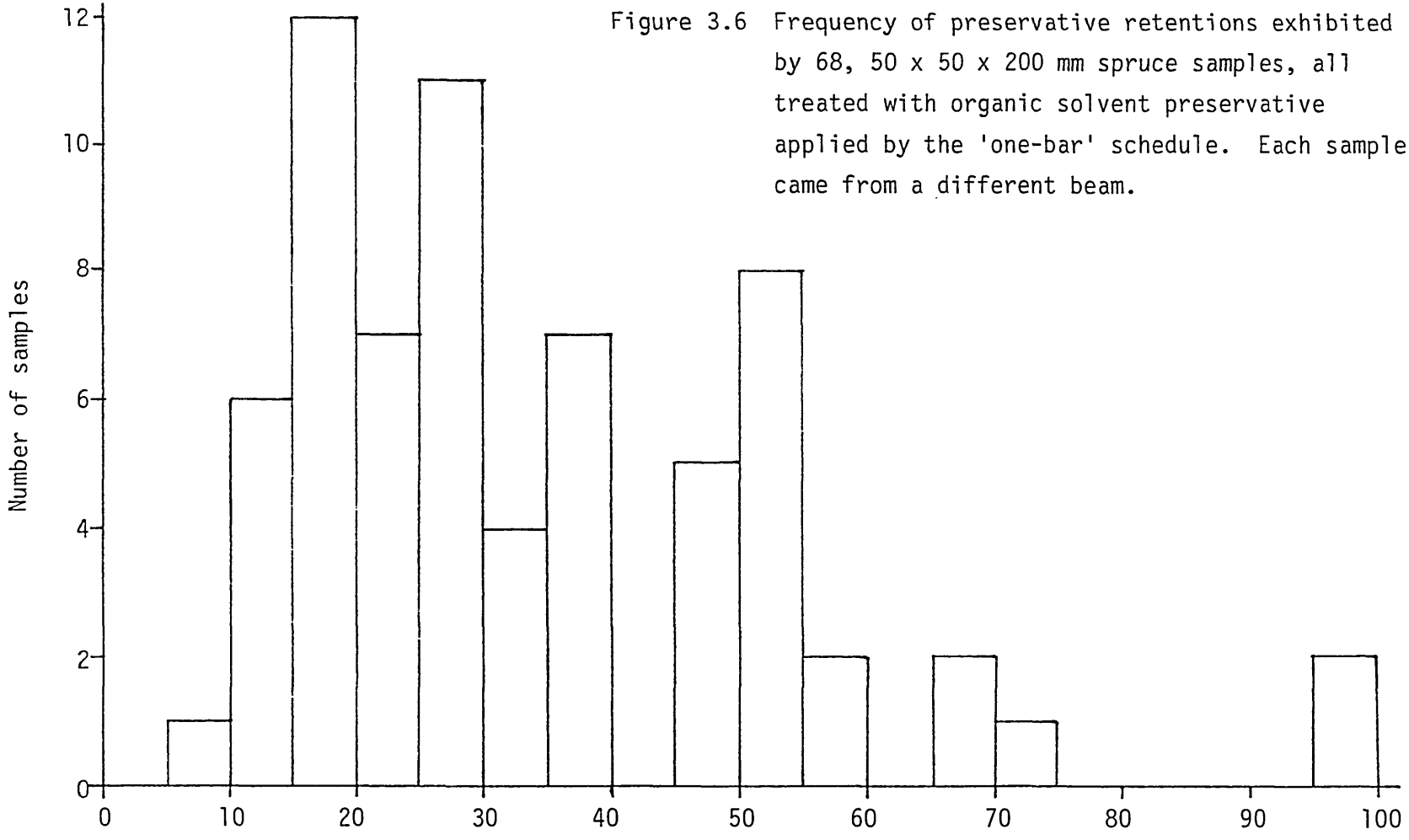


Figure 3.6 Frequency of preservative retentions exhibited by 68, 50 x 50 x 200 mm spruce samples, all treated with organic solvent preservative applied by the 'one-bar' schedule. Each sample came from a different beam.

$$sd = \left(\frac{\Sigma x^2 - \frac{(\Sigma x)^2}{n}}{n - 1} \right)^{\frac{1}{2}} \qquad \text{Equation 3.2}$$

where sd = standard deviation
x = experimental value
n = number of experimental values
(Σ denotes summation)

Equation 3.2 was used to process several sets of data generated during preliminary experimentation, of which the data in Table 3.1 are typical, and it was observed that the standard deviation was often as large as 40% of the mean value of the results.

The practical implication of this high variability of the test substrate is that the amount of replication required to detect relatively small differences between sets of data must also be large. There is a complex inverse relationship between the replication required and the magnitude of difference between two sets of data which it is possible to detect and this relationship is discussed in statistical texts such as Cochran & Cox (1967). The number of replicate samples required to detect a difference of a specified size using a test substrate of known variability may be calculated using the equation:

$$r \geq 2 \left(\frac{sd}{td} \right)^2 (t_1 + t_2)^2 \qquad \text{Equation 3.3}$$

where r = number of replicates required

sd = standard deviation per unit (must be estimated from preliminary experimentation)

td = true difference it is desired to detect

t_1 = value abstracted from a 't-table' (Fisher & Yates 1943) corresponding to the desired level of significance

t_2 = value abstracted from a 't-table' corresponding to 2(1-P), when P = probability of detecting a difference (has values between zero and unity)

The desired level of significance and the required probability of obtaining a significant result are set before experimentation is undertaken. The actual values of t_1 and t_2 are, however, also dependent on the number of degrees of freedom, or (r - 1). Thus iterative trial and error are required to determine the smallest value for r satisfying Equation 3.2.

3.1.3.2 Effects of patterns of sample matching on variability

Random samples

For impregnation trials, the use of randomly selected timber samples has obvious practical advantages. However analysis, using Equation 3.3, of data from previous work (Kyte & Saunders 1978 - Table iv) indicated that nearly four hundred such samples would need to be treated by each schedule to be 90% sure of detecting

a difference in mean uptake of 10% or more at the 95% significance level. Limiting experimentation to timber from a single provenance, did not, on the basis of the Kyte & Saunders data, appear to present a reliable means of reducing sample variability. Because of this, it was decided to match samples with one another before experimentation, and to evaluate the performance of a trial schedule relative to the performance of a standard schedule.

In view of the fact that it is well documented that within a tree permeability can vary laterally with distance from the pith (Buro and Buro 1959), lateral matching of large samples was not considered. Instead, two methods for matching samples which are cut from a beam in longitudinal linear sequence were examined.

Matched pairs

In the context of timber impregnation it seemed reasonable to suppose that a pair of samples cut consecutively from the same beam should exhibit similar treatability. To explore this assumption, an experiment was undertaken in which samples were prepared by cross-cutting 1.8 m long, end-grain-sealed, beams of 50 x 50 mm cross-section, rough-sawn whitewood, into two pieces of equal length. One of these was designated 'A' and the other 'B'. Twenty such pairs of samples were prepared and all were treated simultaneously with a commercial organic-solvent wood preservative formulation (Protim 80) applied by the one-bar schedule described in Section 1.5. The uptakes, calculated from weighing each sample before and after treatment, are presented in Table 3.1.

Table 3.1 Uptakes of organic-solvent preservative (Protim 80) by paired samples of European whitewood. Samples measured 50 x 50 x 900 mm and were sealed across their unmatched ends. Preservative applied by one-bar schedule (q.v. Section 1.5)

Beam Number	Uptake by part 'A' lm^{-3}	Uptake by part 'B' lm^{-3}	Difference in uptakes A - B
1	178.3	89.9	88.4
2	69.9	89.7	-19.8
3	53.1	51.5	1.6
4	21.4	20.2	1.2
5	111.9	66.4	45.5
6	96.8	90.2	6.6
7	69.2	63.1	6.1
8	73.6	78.3	-4.7
9	78.2	51.6	26.6
10	101.4	69.9	31.5
11	59.3	60.3	-1.0
12	88.3	113.8	-25.5
13	70.5	65.6	4.9
14	115.7	87.7	28.0
15	96.2	96.8	-0.6
16	91.3	101.3	-10.0
17	92.4	91.1	1.3
18	69.6	71.2	-1.6
19	115.5	137.2	-21.7
20	204.7	182.3	22.4
Mean	92.9	83.9	9.0
Standard deviation	40.9	34.2	26.2

From Table 3.1 it is clear that the assumption that members of a matched-pair will take up a similar amount of liquid when similarly treated must be used cautiously. At the two extremes are pair 15, in which the uptakes differed by only 1.4% of the lower value, and pair 1 in which piece 'A' took up 98.3% more than piece 'B'.

Overall, the standard deviation of the differences in uptakes between members of each pair was 26.2, which was a substantial reduction compared with the standard deviations of each individual series. Taking samples 'A' as the 'reference samples' the standard deviation is 28.2% of the mean. Thus to determine the level of replication required to be 90% sure of detecting differences of 10% or more at the 95% level of significance, Equation 3.3 takes the values

$$r \geq \left(\frac{28.2}{10}\right)^2 (1.960 + 1.282)^2$$

where 28.2 = standard deviation per unit of the experimental system (as % of mean)

10 = % difference it is desired to detect (as % of mean)

1.960 = value from t-table (2-tail test) corresponding to $n = \infty$, $p = 0.05$ (0.05 = 95% significance level)

1.282 = value from t-table (2-tail test) corresponding to $n = \infty$, $p = 0.2$ (ie 2(1 - 0.9) when 0.9 (or 90%) is desired probability of getting a significant result

so $r \geq 168$ pairs of samples

This result clearly demonstrated that, compared with the replication required with random samples, 'r' can be reduced by matching samples in pairs. The underlying logic of this approach was therefore extended and reversal matching was also evaluated.

Reversal matching

Reversal matching is peculiarly suited to materials along which there is a gradient of variability, and the results presented in Table 3.1 suggest that timber may be just such a material. To effect reversal matching, a group of three consecutive samples is selected. The two outermost samples are treated by the same test procedure and the assumption is made that an average of their two results will closely correspond to the result likely to be obtained from the central member of the triplet if it were similarly treated.

To evaluate this assumption, twenty, 50 x 50 x 885 mm lengths of timber were each cut into three equal samples (designated sequentially A, B and C) and these were all treated simultaneously by the one-bar schedule. The uptakes recorded are presented in Table 3.2, together with an analysis which assumed samples A and C to be the standard schedule samples against which samples B were compared.

Table 3.2 Uptakes of organic solvent preservative (Protim 80) by matched samples of European whitewood. Samples were matched in threes (see text) and each measured 50 x 50 x 295 mm. Preservative applied by one-bar schedule (q.v. Section 1.5)

Beam Number	Uptake by part 'A' lm^{-3}	Uptake by part 'C' lm^{-3}	Av. A & C $\frac{(A+C)}{2}$	Uptake by part 'B' lm^{-3}	Difference in uptakes $\frac{(A+C)}{2} - B$
1	101.7	98.1	99.9	100.9	-1.0
2	164.4	123.4	143.9	140.0	3.9
3	85.8	110.9	98.4	110.2	-11.9
4	138.0	140.9	139.5	132.2	7.3
5	100.2	108.1	104.2	100.7	3.5
6	152.0	103.1	127.6	139.3	-11.8
7	87.0	196.8	141.9	148.3	-6.4
8	90.3	103.6	97.0	116.1	-19.1
9	103.7	131.7	117.7	109.3	8.4
10	125.4	117.8	121.6	134.1	-12.3
11	54.2	68.4	61.3	56.8	4.5
12	110.0	144.8	127.4	124.6	2.8
13	55.9	54.9	55.4	51.0	4.4
14	77.0	196.4	136.7	124.2	12.5
15	130.9	264.2	197.6	171.5	26.1
16	107.5	151.4	129.5	124.6	4.9
17	141.9	147.5	144.7	159.0	-14.3
18	108.3	100.3	104.3	97.5	6.8
19	48.0	67.0	57.5	54.4	3.1
20	206.1	259.0	232.6	197.3	35.3
Mean			121.9		
Standard deviation					13.2

The standard deviation of this matching system indicated by these results was 10.8%. From this it was calculated that twenty-seven sets of 'triplets' would be required to satisfy the parameters already outlined.

Data from other 'ad-hoc' experiments suggested that in fact this level of replication would be somewhat too low and so a further trial was set up in which fifty sets of 'matched triplets' were prepared from eight 5 cm x 5 cm beams. Each of the three samples in a triplet was 20 cm long and was sealed at both ends to eliminate end-grain penetration which would be an unacceptably high proportion of the whole in such short samples. The method of treatment was the same as previously described except that the samples were impregnated in two groups, each of seventy-five samples. For the purposes of processing the results obtained, one group was regarded as having been treated by the 'Standard' schedule whilst the other (identically treated) group was regarded as having been treated by an 'Experimental' schedule. In half the triplets the outer two members of the triplet were treated by the standard schedule and the central members were treated by the test schedule. The converse arrangement was applied to the other triplets, and the treatment each sample received is indicated in Table 3.3.

Tables 3.3 Uptakes of organic solvent preservative (Protim 80) by matched samples of European whitewood. Samples were matched in threes (see text) and each measured 50 x 50 x 200 mm. Both ends of each sample were sealed with epoxy resin. Preservative applied by one-bar schedule (Section 1.5). Underlined results denote samples treated together and regarded as 'Standard Schedule'.

Triplet Number	Uptake by part 'A' lm^{-3}	Uptake by part 'C' lm^{-3}	Av. A & C $\frac{(A+C)}{2}$	Uptake by part 'B' lm^{-3}	Difference in uptakes*
1	206.3	198.3	202.3	201.3	1.0
2	203.5	216.8	210.2	209.5	-0.7
3	233.3	155.0	194.2	184.3	9.9
4	160.3	157.8	159.1	137.3	-21.8
5	141.0	206.8	173.9	142.0	31.9
6	216.5	150.0	183.3	206.3	23.0
7	98.5	80.5	89.5	90.5	-1.0
8	100.0	65.5	82.8	78.0	-4.8
9	90.5	86.5	88.5	25.3	-36.8
10	111.0	80.5	95.8	73.5	-22.3
11	87.5	113.8	100.7	112.0	-11.3
12	110.5	161.8	136.2	137.8	1.6
13	167.8	171.8	169.8	157.0	12.8
14	77.8	76.8	77.3	73.3	-4.0
15	88.5	50.3	69.4	56.0	13.4
16	65.5	52.0	58.8	46.8	-12.0
17	45.3	62.8	54.1	47.3	6.8
18	67.8	34.8	51.3	51.8	0.5
19	43.3	36.0	39.7	36.8	2.9
20	59.0	33.3	46.2	39.0	-7.2
21	39.5	43.5	41.5	60.5	-19.0

Table 3.3 continued

Triplet Number	Uptake by part 'A' lm^{-3}	Uptake by part 'C' lm^{-3}	Av. A & C $\frac{(A+C)}{2}$	Uptake by part 'B' lm^{-3}	Difference in uptakes*
22	46.8	40.5	43.7	58.5	14.8
23	52.8	53.0	52.9	39.8	13.1
24	57.3	69.5	63.4	65.5	2.1
25	53.8	60.8	57.3	56.3	1.0
26	71.3	38.8	55.1	50.0	-5.1
27	50.0	54.0	52.0	38.5	13.5
28	85.5	74.0	79.8	96.0	16.2
29	66.0	100.8	83.4	115.5	-32.1
30	65.8	82.8	74.3	60.0	-14.3
31	74.5	85.5	80.0	84.0	-4.0
32	103.8	95.8	99.8	110.0	10.2
33	112.5	65.5	89.0	85.3	3.7
34	74.0	57.3	65.7	48.5	-17.2
35	85.0	51.5	68.3	75.3	-7.0
36	70.0	90.3	80.2	68.3	-11.9
37	92.5	79.0	85.8	81.5	4.3
38	69.3	75.0	72.2	91.0	18.8
39	96.0	69.5	82.8	85.0	-2.2
40	74.8	43.5	59.2	58.0	-1.2
41	57.0	63.8	60.4	40.8	19.6
42	66.3	76.5	71.4	64.0	-7.4
43	73.8	54.3	64.1	66.0	-1.9
44	98.8	109.0	103.9	100.3	-3.6
45	69.0	62.0	65.5	75.8	-10.3
46	112.5	62.0	87.3	56.0	-31.3
47	91.3	66.0	78.7	56.5	22.2
48	68.8	53.0	60.9	53.5	-7.4

Table 3.3 continued

Triplet Number	Uptake by part 'A' lm^{-3}	Uptake by part 'C' lm^{-3}	Av. A & C $\frac{(A+C)}{2}$	Uptake by part 'B' lm^{-3}	Difference in uptakes*
49	62.0	40.3	51.2	58.3	-7.1
50	52.0	48.5	50.3	50.0	-0.3
Mean			86.2		
Stand'd Dev'n					14.5

* The figures which appear under this heading are, for each triplet, the result (or average result) of the 'Experimental' schedule, subtracted from the result (or average result) of the 'Standard' schedule. Thus for each odd numbered triplet the figure in the right hand column is the result of the calculation '$\frac{A+C}{2}$ - B' whereas, for each even numbered triplet the figure in the right hand column is the result of the calculation 'B - $\frac{A+C}{2}$'.

From Table 3.3 it can be seen that the standard deviation of the differences between the values '$\frac{A + C}{2}$' and 'B' for each triplet was 16.8% of the mean, which indicated that 60 triplets should be used in each comparison to satisfy the conditions already outlined.

Double Reversal Matching

Double reversal matching, in which four consecutive samples are allocated to treatments alternately, and the results of uptakes 'A' and 'C' are compared with those of 'B' and 'D', was examined. However, the indications were that this pattern of sample matching did not reduce the level of replication sufficiently to justify the concomitant increase in data manipulation and analysis. Consequently, this technique was not further considered.

3.1.3.3 The Matched-Triplet Test

3.1.3.3.1 Original experimental system

In Section 3.1.3.2, the two experiments aimed at evaluating the use of reversal matching indicated respectively that between 27 and 60 'triplets' of samples of spruce were required to be 90% sure of detecting a difference of 10% or more in the mean uptake resulting from two treating schedules. In the light of these results, the possibility of conducting all further experimentation with 60 sets of matched samples was considered. However, 50 sets of samples was the maximum number of 5 x 5 cm samples which could conveniently be treated in the available equipment on two charges (Plate 2.1). This level of replication was therefore adopted as the optimum balance between the sensitivity of the technique and its practicability in use.

On this basis the 'matched-triplet' test was designed. For each test, 50 'triplet' sets of wood samples were prepared from 5 x 5 cm beams of 'European Whitewood'. The overwhelming majority of timber used was of Finnish origin. To avoid introducing an element of subjectivity, triplets were prepared irrespective of defects such as knots etc. as the level of replication to be used had been determined using 'unselected' samples. The single exception to this procedure occurred when a sample was so deeply split that on cutting to length it fell into two (or more) pieces. Such samples were discarded.

At the commencement of the test, beams were marked and numbered in the manner indicated in Figure 3.7. Each beam was then cross-cut and the two ends of the resultant samples were sealed (see Section 2.2.1.2.3). Although samples were not conditioned prior to treatment, except to ensure that they were in a 'treatable-condition' (moisture content <20%), care was taken to keep all samples from each triplet together until immediately before treatment. Both treatments of a test were performed within a short time and usually on the same day so that it could reasonably be assumed that all samples in any one comparison were in the same condition at the beginning of treatment.

To reiterate, the matched triplet test was designed as a means of comparing two impregnation schedules. The principal limitation of such a technique is that if schedules 'x' and 'y' are compared in one test and schedules 'v' and 'w' are compared in another, it is not then possible to use the results obtained to compare 'x' and 'w'. To avoid this problem, all the work reported here utilises the same reference or standard schedule. Certainly in practice there are instances when it is desirable directly to compare two non-standard schedules. However, the results of such comparisons tend to stand alone and it is most difficult to establish patterns and trends from large numbers of experiments all involving non-standard schedules.

At the time of the inception of this work the one-bar schedule described in Section 1.5 was the only whitewood impregnation schedule appearing in British Standards and so this was taken as the standard schedule, except that a final vacuum of -0.85 bar was used in this work. Other experimental schedules were thus evaluated relative to this standard.

Samples were allocated to treatments so that 75 samples (i.e. one half of the total) were treated by the standard schedule and the remainder were treated by the experimental schedule. This allocation was effected by selecting alternate samples along a beam (Figure 3.7). Thus in 25 triplets the two outermost samples were treated by the standard schedule and the central sample was treated by the experimental schedule. In the remaining 25 triplets the converse applied.

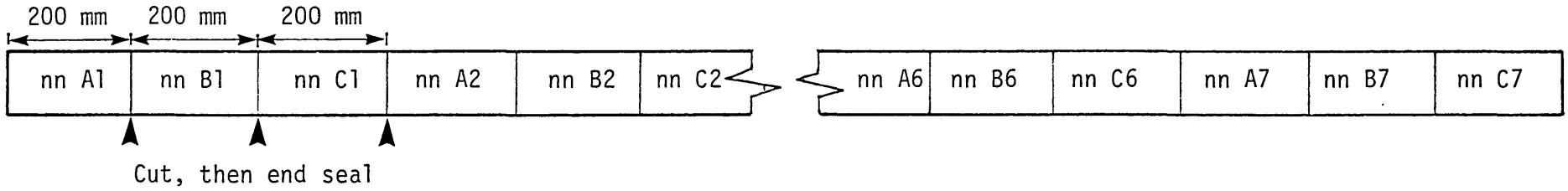
In all experiments the impregnation solution used was a commercial organic solvent wood preservative (Protim 80, Fosroc formulation reference FC 464/2) which had a specific gravity of 0.8 at 20°C. To facilitate visual examination of penetration this was dyed blue with 0.5% Waxolene blue A dye.

Uptake of this solution by each sample was determined by weighing the samples individually on a top-pan balance (Section 3.1.1).

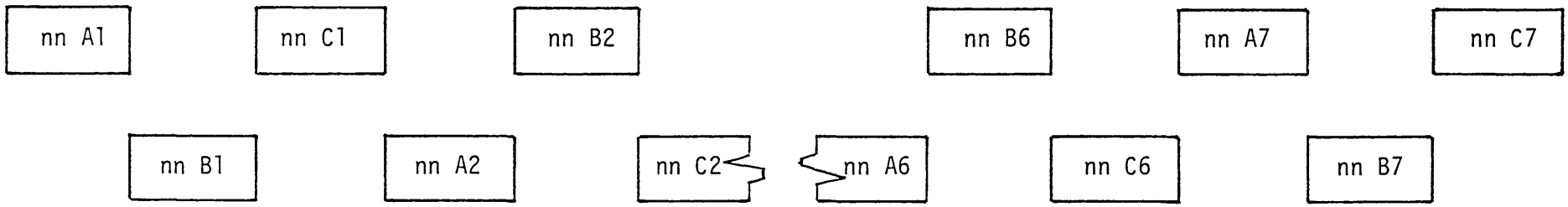
Penetration of preservative was assessed using the techniques described in Section 3.1.2.

Figure 3.7 Preparation of samples for matched triplet test

Step 1 - Beam marked and numbered as shown and then cut, and the pieces end sealed



Step 2 - Samples divided into groups for treatment, samples in the top row being treated by the standard schedule and those in the lower row by an experimental schedule - or the converse



3.1.3.3.2 Modifications to the original experimental system

Sample cross section

Part way through the experimentation, supply difficulties were encountered with respect to 5 x 5 cm rough-sawn whitewood. The timber merchant became unwilling to continue sorting whitewood from mixed spruce/pine/fir packs. In consequence the sample size was changed slightly to 5 x 3.4 cm cross section material. This could readily be prepared from 10 x 3.4 cm cross section truss rafter material which is imported in segregated whitewood bundles.

Pattern of sample selection

As has been indicated already, at the outset six or seven triplets were prepared from each of eight or nine beams of whitewood in the manner indicated in Figure 3.7. Indeed this method of sample preparation was used for much of the work reported in Section 3.2.1. A surprising, and to date unexplained, observation led to this practice being modified.

During a programme of schedule comparisons, which was not directly related to this work, a schedule involving ultrasonic vibration of the treating liquid during the immersion stage was being examined. Analysis of the data revealed that whereas in six of the seven beams from which samples had been prepared the non-

ultrasonic cycle consistently and clearly led to higher uptakes, on the seventh beam, the converse was equally clearly true.

The above observation highlighted the benefit of using triplets of samples from many different beams. In the experimental procedure outlined in Section 3.1.3.3.1, approximately 14% of the total experimental data was based on each beam of timber. If one beam was atypical in its behaviour, 14% of the data was correspondingly atypical and any conclusions drawn from the experiment may have limited applicability.

In response to the above, the pattern of sample selection was changed so that each triplet in a test came from a different beam, i.e. 50 beams were sampled to obtain one triplet from each. Thus only 2% of the data in each experiment was based on results from any one beam.

3.1.3.3.3 Analysis of data

Each triplet test yielded data relating to the uptake and to the penetration of preservative. The penetration data were analysed simply by calculating arithmetic mean values for each feature being examined. The uptake data however were subjected to more rigorous statistical analysis which is outlined below.

To reiterate Section 3.1.3.3.1, a 'triplet' of samples comprised three samples cut consecutively from a beam of timber. The central one of these was treated by one of the two impregnation schedules being compared, whilst the outer two were both treated by the second of the two schedules. The gravimetrically determined uptakes for the outer two samples were averaged and compared with the uptake occurring with the central sample. With each triplet, the test-schedule uptake was subtracted from the standard-schedule uptake in order to determine a value, designated 'x_i', which was used in further statistical analysis. From each impregnation schedule comparison, fifty values for x_i had to be calculated and processed and so a computer programme was written to facilitate interpretation of data. A copy of this programme is presented in Appendix 2.

Part of the output from the above programme is reproduced in Figure 3.8. From this it can be seen that the arithmetic mean uptake resulting from each impregnation schedule was calculated and expressed not only in grams per sample, but also as kilograms of preservative per cubic metre of timber. In addition, the mean uptake resulting from the experimental (or test) schedule was expressed as a percentage of the mean uptake resulting from treatment by the standard one-bar schedule.

Figure 3.8 Photocopy of results summary produced during computer-assisted analysis of uptake data

COMPARISON OF UPTAKES BY WHITEWOOD RESULTING FROM TREATMENT

BY CYCLES 25.10/15.60/25.20. AND 25.10/15.60/25.20.

(TEST SAMPLES NUMBERS 147 TO 154)

TREATING SOLUTION FC464/2

STANDARD CYCLE 25.10/15.60/25.20.

MEAN	34.69GN
	69.38KG/M
VARIANCE	315.43
ST.DEV.	17.76
COEF OF VAR	51.20
ST.ERROR	2.05
MEDIAN VALUE(S)	29.9

TEST CYCLE 25.10/15.60/25.20.

MEAN	34.51GN
	69.02KG/M
VARIANCE	356.09
ST.DEV.	18.87
COEF OF VAR	54.68
ST.ERROR	2.18
MEDIAN VALUE(S)	27.3
MEDIAN VALUE(S)	27.6

MEAN UPTAKE OF TEST CYCLE SAMPLES AS A PERCENTAGE OF THE MEAN UPTAKE OF STANDARD CYCLE SAMPLES

99.48 PERCENT

F (VARIANCE RATIO) = 1.13

75 TEST SAMPLES TREATED 75 STANDARD SAMPLES TREATED

RESULT OF PARAMETRIC STUDENTS T TEST T = .60 N = 50

USE PUBLISHED TABLES TO ASSESS PROBABILITY

22 TRIPLETS HAD A HIGHER UPTAKE AS A RESULT OF TREATMENT BY THE TEST CYCLE

20.0 TRIPLETS HAD AN UPTAKE LOWER AS A RESULT OF TREATMENT BY THE TEST CYCLE

7 AND 8 SAMPLES RESPECTIVELY (CA. 10 PERCENT OF THE STANDARD CYCLE SAMPLES) HAD UPTAKES LESS THAN OF EQUAL TO 16.2 AND 16.3 GN.
4 AND 4 TEST CYCLE SAMPLES RESPECTIVELY HAD UPTAKES BELOW THESE LEVELS

The distribution of uptake of preservative by samples of white-wood was not statistically 'normal', but 'skewed' with a tail to the right. In consequence the mean and median values did not coincide. For this reason the median value for each group of samples was also determined.

The variance, which is a measure of the variability of each batch of data was also calculated, and from this the 'variance ratio' (F) was calculated. When the variance of the standard schedule data, and that of the experimental schedule were sufficiently similar ($F < 1.5$) it could be assumed that the values of x_i were statistically normally distributed, and the statistical significance of the differences in the data were therefore assessed using 'Student's t-test'. In this test the values for x_i were processed using Equation 3.4.

$$t = \frac{\frac{\Sigma x_i}{n}}{\sqrt{\frac{\Sigma x_i^2 - \frac{(\Sigma x_i)^2}{n}}{n(n-1)}}} \qquad \text{Equation 3.4}$$

When comparison of the variances of two sets of data yielded a value for 'F' in excess of 1.5, it could not be assumed that the values for x_i were normally distributed, and so Student's t-test could not be used. Instead a non-parametric statistical test, namely the 'Wilcoxon Signed Rank Test' was applied. This test is

described in statistical texts such as Siegel (1956) and the computer programme was written to apply this test whenever a value for 'F' greater than 1.5 was encountered.

Like all non-parametric tests, the Wilcoxon Signed Rank Test does not need data which are normally distributed. However, it requires a greater quantity of data than Student's t-test in order to assign a given level of significance to a result, and for this reason it is less statistically powerful.

From the specimen computer output (Fig 3.8) it can be seen that, in addition to the standard statistical analysis outlined above, two further features of the data were also examined.

In the first of these additional features, the values for x_i were examined and the number of positive values and the number of negative values were recorded. A positive value for x_i indicated that, in that triplet of samples, the standard impregnation schedule had resulted in an uptake greater than that which resulted from treatment by the experimental schedule. A negative value for x_i indicated the converse. This feature of the results provided an immediately tangible and readily assimilated indication of whether a difference in overall mean uptake indicated a consistent difference in uptake in most of the triplets.

The second additional feature which was examined reflected a primarily industrial preoccupation. Industrially, the possibility

of inadequate treatment being applied to the least permeable pieces of timber in a charge is a constant concern. Thus, when evaluating modifications to industrial treatment schedules it is important to be aware of whether a modification has a disproportionate effect on the least permeable samples. For this reason, the standard schedule data were examined to identify the uptake value which was exceeded by 90% of the standard schedule samples. The uptakes from the experimental schedule samples were then compared against this value to determine the proportion of the data which fell below it.

3.1.3.3.4 Chequerboard matching for small samples

Certain of the experimentation undertaken during this investigation involved the use of applied pressures in excess of 200 kPa. For this work, the size of the impregnation apparatus which was available was such that a series of matched triplet samples could not all be accommodated in the same charge. It was therefore necessary to use an alternative experimental design which is outlined below.

On the basis of experience gained during the development and use of the matched-triplet technique it was clear that intimate matching of samples was the principal means of minimising the between-sample variability. In consequence alternate selection of samples cut longitudinally from small beams was used as the basic sample matching system. In addition, advantage was taken

of the fact that the small beams used were conveniently prepared by resawing a 3.4 x 10 cm whitewood beam into four, 3.5 x 2.5 cm beams. It was thus possible to allocate samples to treatments by alternate selection in two dimensions, producing a chequerboard effect (Fig 3.9).

The number of small samples prepared in this way which were used for a test corresponded to the number of samples which could be fitted into the treating vessel (Plate 3.10). Analysis of uptake data consisted of computing an arithmetic mean uptake resulting from each of the schedules being compared.

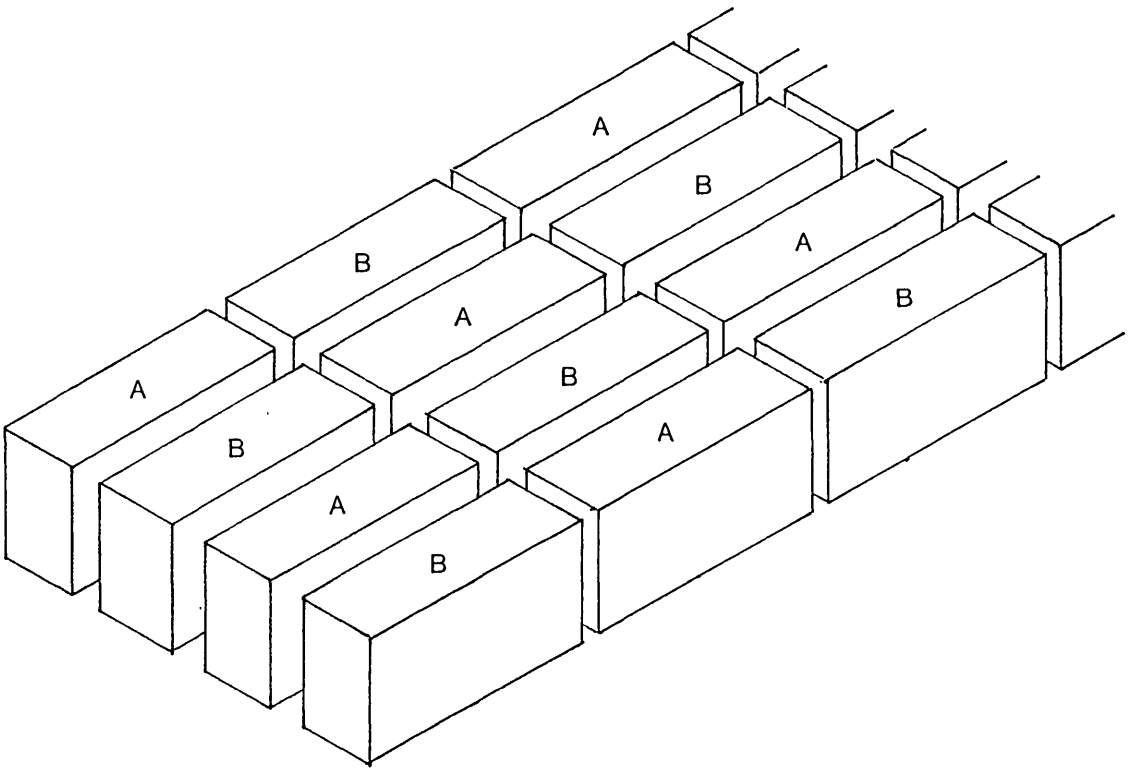


Figure 3.9 Chequerboard matching of small samples

Plate 3.10 Apparatus used for impregnation schedules involving pressures greater than 200 kPa



3.2 Comparative evaluation of timber preservative impregnation schedules

This section deals with the way in which the techniques described in Section 3.1 (especially 3.1.3.3) were used to investigate the second and third aims of this work, as outlined in Section 1.6.

Precise description of the results achieved by the principal existing technique for the impregnation of spruce (Aim 2) clearly required as its basis a large quantity of appropriate data. This requirement was one of the factors underlying the selection of the 'one-bar schedule' as the standard for matched-triplet testing. By using this standard schedule, each matched-triplet test added to the mass of data relating to the results obtained with the standard schedule. An analysis of the relevant data accumulated from the work described in Section 3.2.1 is presented in Section 3.2.2.2.1.

The third and final aim of this piece of work was the investigation of means whereby the techniques available for the industrial impregnation of spruce might be improved. The experimentation described in Section 3.2.1, the results of which are presented and discussed in Section 3.2.2, was undertaken with this aim in view.

3.2.1 Experimentation undertaken

3.2.1.1 Incorporation of a period of pneumatic-pressure after the immersion phase

In Section 1.5.3.6, the schedule which was specified in the British Wood Preserving Association Specifications for the impregnation of spruce, when this work was started, was described. In Section 1.6, the principal, practical problems associated with this schedule were outlined (namely, irregular penetration, high preservative retention, excessive surface wetness after treatment and a long treatment time).

In Sections 2.2.3.4.3 and 2.2.3.4.4 results have been presented which show that, contrary to the generally accepted basis on which the Double Vacuum and Double Vacuum Pressure processes were designed, the vacuum established within pieces of spruce at the beginning of an industrial treatment schedule is still present at the end of the immersion-under-pressure phase. This observation calls into question the role of the final vacuum.

Conceptually, the purpose of the final vacuum of a Double Vacuum Pressure process is to remove 'excess' liquid preservative from the timber's surface. If however at the end of the immersion phase, there still exists within the timber a negative pressure, it should be possible to move the liquid preservative at the timber's surface further into the timber by the application of a

pneumatic pressure instead of a final vacuum.

Application of a pneumatic pressure at the end of the immersion phase might also have a further benefit. By moving the preservative deeper into the timber it may improve penetration without increasing what is already considered to be the excessive uptake of preservative liquid. Indeed, extending the logic of this approach, application of pneumatic pressure may permit the immersion phase to be curtailed, thereby reducing uptake, whilst maintaining or improving penetration.

In the light of the considerations outlined above and using the matched-triplet and related techniques outlined in Section 3.1, the experimentation outlined in Table 3.4 was undertaken to assess the effect of curtailing the immersion phase, and of replacing the final vacuum with a period of applied pneumatic pressure. The results obtained from this experimentation are presented and discussed in Section 3.2.2.1.

3.2.1.2 Variation of treatment schedule parameters

The work described in Section 3.2.1.1 yielded results (3.2.2.1) which indicated that, contrary to expectation, in a standard double vacuum type treatment, the uptake of preservative by spruce does not necessarily correlate closely with its penetration. This indication was entirely unexpected and particularly when taken together with the microtransducer results reported in

Table 3.4 Impregnation schedules evaluated, using the matched-triplet test technique, to investigate the effect of incorporating a period of air pressure after the immersion phase

Impregnation Schedule	Initial Vacuum		Immersion Phase		Post-Immersion Pressure		Final Vacuum	
	Level	Duration mins	Applied Pressure	Duration mins	Applied Air Pressure	Duration mins	Level	Duration mins
Standard Schedule	-85 kPa	10	103 kPa	60	-	-	-85 kPa	20
Experimental Schedules								
1	-85 kPa	10	103 kPa	10	-	-	-85 kPa	20
2	-85 kPa	10	103 kPa	10	103 kPa	50	-	-
3	-85 kPa	10	103 kPa	10	186 kPa	300	-	-

Section 2.2.3.4.3, which were also unexpected, further highlighted the fact that even the basic behaviour of preservative liquid during double vacuum impregnation of spruce is incompletely understood. In response to this, a programme of experimentation was designed in order to elucidate the effect of each parameter of a double vacuum pressure impregnation schedule on the uptake and penetration of preservative.

The basis of this programme was the selection of each variable parameter in turn, and the examination of the effects on uptake and penetration of variation of that parameter. For example, a series of experiments was undertaken to examine the effect of varying the level of initial vacuum whilst holding all the other parameters constant. The matrix of experimentation undertaken is presented in Table 3.5.

In essence, the technique used for these experiments was the Matched Triplet Test as described in Section 3.1.3.3, although three variants on this basic procedure were employed when necessary. The first of these has already been described in Section 3.1.3.3.4 and involved the use of smaller samples for tests involving impregnation pressures greater than 200 kPa.

The second variant related to penetration assessment. The penetration assessment procedure described in Section 3.1.2.2 was very time consuming to perform. Consequently this aspect of impregnation schedule evaluation had to be performed somewhat

Table 3.5 Impregnation schedules evaluated using Scandinavian whitewood and the matched-triplet test technique to investigate the effects of systematic variations to the impregnation schedule parameters

Schedule Ref. No.[2]	Initial Vacuum		Immersion Phase		Final Vacuum	
	Level kPa	Duration Min.	Level kPa	Duration Min.	Level kPa	Duration Min.
Standard Schedule	-85	10	103	60	-85	20
4	0[1]	10	103	60	-85	20
5	-17	10	103	60	-85	20
6	-34	10	103	60	-85	20
7	-51	10	103	60	-85	20
8	-68	10[1]	103	60	-85	20
9	-85	0	103	60	-85	20
10	-85	5	103	60	-85	20
11	-85	20	103	60	-85	20
12	-85	40	103	60	-85	20
13	-85	10	0	60	-85	20
14	-85	10	69	60	-85	20
15	-85	10	138	60	-85	20
16	-85	10	207	60	-85	20
17*	-85	10	413	60	-85	20
18*	-85	10	620	60	-85	20
19*	-85	10	827	60	-85	20
20*	-85	10	1033	60	-85	20
21*	-85	10	1240	60	-85	20
22	-85	10	103	10	-85	20
23	-85	10	103	20	-85	20
24	-85	10	103	40	-85	20
25	-85	10	103	90	-85	20
26+	-85	10	103	900	-85[1]	20
27	-85	10	103	60	0	20
28	-85	10	103	60	-17	20
29	-85	10	103	60	-34	20
30	-85	10	103	60	-51	20[1]
31	-85	10	103	60	-85	0
32	-85	10	103	60	-85	30
33	-85	10	103	60	-85	60

Notes

[1] Tests 4 and 9 are identical - involving no initial vacuum. Similarly Tests 27 and 31 are identical, involving no final vacuum.

[2] To avoid possible confusion, schedule reference numbers follow on from those in Table 3.4.

* Chequerboard matching of small samples used in tests 17 through 21.

\+ Test 26 was conducted using 32 triplets in a small treatment vessel.

selectively and, additionally, early assessments conducted using the square counting technique (q.v. Section 3.1.2.1) were not repeated.

The third, and final, variant was that in several tests, a supplementary piece of information was recorded - namely the approximate 'Gross Uptake' of preservative. The total weight of all the samples in a treatment, plus 'stickers' and the framing supporting the samples, was recorded both before treatment and between the immersion phase and the final vacuum. Using these two weights and the nominal total volume of all the samples, a value for the gross uptake of liquid was calculated.

The results obtained from this programme of work are presented in Appendix 1 and are summarised and discussed in Section 3.2.2.2. The discussion in this section does not in fact discuss all the results of each test, but instead focusses on what are considered to be key features which are indicative of the underlying trends.

Because one half of each matched-triplet test was treated with the standard one-bar schedule, the experimentation outlined in Tables 3.4 and 3.5 generated a great deal of data concerning the results achieved by this schedule. Thus, concurrently with the investigation outlined above, data were gathered with a view to quantifying the performance of the one-bar schedule, which was one of the aims of this investigation (Section 1.6 q.v.).

3.2.2 Comparative evaluation of timber preservative impregnation schedules - results and discussion

3.2.2.1 Incorporation of a period of pneumatic-pressure after the immersion phase

The experimentation described in Section 3.2.1.1 yielded the results presented in Appendix 1. A summary of these results is presented in Table 3.6.

Table 3.6 Performance of schedules 1 to 3 (as detailed in Table 3.4) expressed in percentages relative to the performances of the Standard spruce impregnation schedule

Experimental schedule	Performance as percentage of Standard schedule			
	Uptake	Overall penetration	Penetration to 2.5 mm	Penetration to 4.0 mm
1	44	82	97	95
2	57	78	100	97
3	78	72	94	75

Note: Samples treated by schedules 2 and 3 were drier than others at the end of treatment

In Section 2.1.4 it was concluded that, as a working approximation, the relationship between the volume of preservative absorbed (Q) and the duration of the immersion phase was '$Q \propto \sqrt{\text{time}}$'. On this basis, schedule 1 was expected to result in an uptake ca. 41% of that resulting from the Standard schedule, because $\sqrt{10}$ (minutes) is 40.8% of $\sqrt{60}$ (minutes). The actual uptake of preservative resulting from treatment by schedule 1 was 44% of that resulting from the Standard schedule, which closely accords with the theoretical expectation.

In view of the identical conditions which prevailed during the immersion phases of schedules 1, 2 and 3, it was assumed that the uptakes of preservative which occurred during the three phases were all closely similar. However, only schedule 1 involved a final vacuum to withdraw liquid at the end of the immersion phase and it is logical therefore that the net uptakes resulting from schedules 2 and 3 were both higher than that which resulted from schedule 1.

Unfortunately it does not appear to be possible to advance a logical basis to account for the fact that uptakes 2 and 3 are not closely similar to one another. Additional recent experimentation has strongly indicated that the 'schedule 2' uptake recorded in this work was unusually low, and schedules involving a 10 minute immersion phase without a final vacuum more usually result in uptakes in the region of 70% of those resulting from the Standard schedule.

Concerning the penetrations achieved, schedule 2 appeared to confirm the a-priori anticipation that application of a final pneumatic pressure would enhance the penetration which could be achieved with a given quantity of preservative. Whereas schedule 2 reduced the uptake to 56% of the standard-cycle uptake, the overall penetration dropped to ca. 78% and hardly fell at all in the critical outer 4 mm.

It was because of this that a further schedule (schedule 3) was evaluated. The final pneumatic pressure applied in this instance was somewhat higher (186 kPa) and it was sustained for a longer period (300 minutes rather than 50) to prolong the period during which a pressure gradient existed to promote the movement of liquid into the timber.

Surprisingly, the conditions outlined above did not enhance penetration. Indeed, at face value the data indicate that the opposite appeared to be true, although the variances in the figures are more logically interpreted as being due to experimental error.

When taken in conjunction with the results presented in the different parts of Section 2.2.3.4, the 'schedule 3' results appear to indicate that, notwithstanding the period for which it was applied, the level of applied pneumatic pressure (186 kPa) was insufficient to move the layer of liquid away from the surface and more deeply into the timber.

A further experiment was therefore conducted involving a final pneumatic pressure of ca. 1000 kPa. Formal quantitative results of this investigation are not available, although it was clear from preliminary examination of the blocks that no significant improvement in penetration had been achieved.

Overall, therefore, it appears that the potential for using a final pneumatic pressure to promote penetration beyond that which may be achieved by a standard 'one-bar schedule' is rather limited. The application of pneumatic pressure does, however, appear to present a means whereby substantially similar penetration may be achieved using a lower volume of preservative than that used in the one-bar schedule.

Although the effect on penetration of a final pneumatic pressure was the focus of this section of the investigation, the most interesting and unexpected of the penetration results obtained were those from the control schedule, schedule 1. Here the shortened schedule, as anticipated, reduced uptake to 44% of that resulting from the standard schedule. However penetration, most unexpectedly, did not drop concomitantly, and penetration in the superficial 4.0 mm barely fell at all. This observation cannot readily be explained, and highlighted the need more fully to investigate the effects of altering the parameters of the existing spruce treatment cycle. Accordingly, the investigation outlined in Section 3.2.1.2 was undertaken.

3.2.2 Results and discussion - continued

3.2.2.2 Systematic variation of treatment schedule parameters

The experimentation outlined in Section 3.2.1.2 yielded results, which are summarised in Appendix 1. These data are further summarised in Tables 3.7 to 3.9.

Discussion of these results is divided into three sections as outlined in the following three paragraphs.

One objective of this section of this investigation was to quantify the performance of the one-bar schedule in terms of uptake and penetration. With this objective in mind, the data presented in Tables 3.7 and 3.8 have been abstracted from the Appendix and these data, together with the insights they give into the nature of spruce, are discussed in Section 3.2.2.2.1 (below).

The principal objective of this section of this investigation was to examine the effects on uptake and penetration of systematically altering each of the variable parameters of a double vacuum schedule. The data gathered in this context are summarised in Table 3.9 which is discussed in Section 3.2.2.2.2.

The industrial significance of the conclusions drawn from the Table 3.9 results is briefly considered in Section 3.2.2.2.3.

3.2.2.2.1 Results achieved by one-bar schedule

A feature which has already been observed (Section 3.1.3.1) and which is also evident in the data in the Appendix is the variability in the results achieved by the Standard schedule. Excluding Experiments 17 to 21 which employed rather small samples with which high uptakes might have been expected, the range of average uptakes resulting from the Standard schedule was 40 to 155 l/m^3, with penetration indices ranging between 30 and 87.

The results in Tables 3.7 and 3.8 represent average values derived from the data in the Appendix.

The uptake data in Table 3.7 corroborate other reports (Kyte & Saunders 1978) that the one-bar schedule, used to treat spruce, results in uptakes which are rather high. (For comparison, 20 to 40 l/m^3 are 'typical' uptakes recorded during the industrial impregnation of European redwood.)

The 'index of penetration' which is also presented in Table 3.7 is unexpectedly high. To a first approximation, this index may be regarded as the percentage cross-section penetrated and the data presented indicate that, on average, two-thirds of the total cross-section of each 35 x 48 mm piece was penetrated by preservative. Despite this, spruce is regarded as an impermeable timber which cannot be treated adequately to give long life in

high-hazard situations. (See for example British Standard 5589, 1978.)

Table 3.7 Average performance of one-bar schedule

Uptake l/m^3	Penetration			
	Index	At 1.0 mm	At 2.5 mm	At 5.0 mm
88.7	65.2	93.4%	80.5%	65.4%

There are perhaps two reasons why spruce is regarded as being difficult to treat, whilst none-the-less exhibiting a high penetration index. The first of these reasons has already been alluded to, although it is not explicitly evident from the tables, and is that spruce is a highly variable material and in any charge there are likely to be pieces showing very low penetration.

The second reason, which is evident in both Tables 3.7 and 3.8, is that a high penetration index seldom implies a continuous envelope, several millimetres thick, of preservative-loaded timber around each piece. Table 3.7 indicates that less than 95% of the surface of samples shows penetration at a depth of one millimetre. Indeed, in approximately 70% of samples examined (Table 3.8) there was at least one point at which penetration was so shallow

Table 3.8

Gross penetration characteristic	Frequency of occurrence
Continuous envelope of penetration	31%
Discontinuities in envelope of penetration	69%
Banded penetration	18%
Broken penetration	13%
Points of negligible penetration into latewood	42%
Evidence of latewood being preferred pathway	46%
Points of negligible penetration into earlywood	69%
Evidence of earlywood being preferred pathway	<1%
Evidence of more permeable sapwood corner	99%
Evidence of radial pathways of penetration	1%
'2-tone' intensity of tracer dye	62%

as to be immeasurably small and the envelope of penetration was thus judged to be 'discontinuous'. Almost invariably where a discontinuous envelope was observed, the predominant discontinuities were associated with the earlywood although in nearly two-thirds of the samples showing a discontinuity in the envelope of penetration, negligibly deep penetration was also seen associated with the latewood.

Nearly half the samples examined showed evidence of penetration proceeding more readily into the latewood than the earlywood, whilst the converse pattern of penetration was evident in less than 1% of the samples.

Very occasionally (in 1% of samples) there was evidence of the rays being pathways through which liquid travelled. Usually it appeared that if the rays played any part in liquid movement at all it was to form channels through which liquid moved from the latewood into the earlywood of the same annual ring.

In most samples examined, penetration essentially took the form of an irregular envelope around the sample, although in 13% of samples, there were discrete patches of penetration remote from the peripheral envelope. Such patches were interpreted as being indicative of liquid entering the timber laterally at some point remote from the place where cross-cutting subsequently occurred, and then penetrating longitudinally within the sample.

In a significant proportion of samples (18%) two, three or more adjacent annual rings exhibited permeability characteristics different from those of the surrounding timber giving 'bands' of penetrated or unpenetrated timber extending all or part of the way across the sample.

Banded penetration was quite distinctly different from the 'sapwood-corner' effect which occurred in 99% of the samples examined and which was characterised by the corner of the sample nearest the periphery of the original tree being distinctly more extensively penetrated than the rest of the sample. The frequency of occurrence of the sapwood-corner effect constitutes consistent evidence that the 'sapwood' of spruce is more permeable than its 'heartwood'. This suggests that pit aspiration which results purely from post-harvesting drying is in some way different from that which occurs during heartwood formation. For instance it is perhaps possible that the slower speed at which heartwood formation occurs permits pit aspiration to be accompanied by hydrogen-bonding between the margos and the pit chamber walls, or perhaps by the deposition of extractives which help to seal the pit-apertures.

When examining samples of spruce which had been impregnated with dyed preservative applied by the one-bar schedule it was also noted that a majority (62%) of the samples exhibited evidence of a 'two-tone' appearance rather than a steady decrease in intensity of tracer-dye colour. The possible significance of this

observation is discussed in Section 4.

In summary, from the observed patterns of penetration it is clear that although spruce may be penetrated quite extensively, it cannot be said to treat satisfactorily. This is because almost invariably there was at least one point around each treated piece where penetration was so shallow as to be negligible. Most frequently such regions of negligible penetration were associated with the earlywood. However, in many instances negligible penetration was also associated with latewood, despite the fact that in about half the samples examined this tissue was clearly the more permeable.

In nearly all samples examined there was evidence of a 'sapwood corner' which was very well penetrated. Evidence of radial tissue forming a pathway through which liquid flow occurred was virtually absent.

3.2.2.2.2 Effects of varying treatment schedule parameters

In Section 3.2.2.2.1 the variability in the reaction of spruce to treatment by a standard schedule has again been highlighted.

It was in response to this variability that the matched triplet test was designed and used for this investigation. This technique facilitates comparison of the results achieved by each experimental schedule with those obtained by the standard schedule on

closely matched timber. The results of using this technique to evaluate the performance of the schedules detailed in Table 3.5 are presented in Appendix 1 and the salient data are summarised in Table 3.9.

To facilitate interpretation of the results, the data in Table 3.9 are presented in Figures 3.10 and 3.15 in such a way that the performance of each experimental schedule is expressed as a percentage relative to the performance of the standard schedule.

The patterns of variation evident in Figures 3.10 to 3.15 are particularly instructive concerning the nature of spruce as a permeable substrate, and also concerning the behaviour of the impregnating liquid.

To describe the patterns of variation, prior to proposing a model to account for them, Figures 3.10 to 3.15 may be considered in six pairs, each pair relating to one of the variable parameters of the double vacuum schedule.

Level of initial vacuum (Figure 3.10)

The severity of the initial vacuum clearly had a significant effect on uptake, with the more severe vacuum levels leading to higher uptakes. In general terms this result was anticipated. Indeed, the experimentation reported and discussed in Section 2.2.2.3.2 indicated that uptake varies in direct linear proportion

Table 3.9 Summary of uptake and penetration data obtained in comparison-tests to evaluate the schedules detailed in Table 3.5 (Section 3.2.1.2)

Ref No.	Modified Schedule						Standard Schedule						See Foot Notes
	Uptake l/m^3		Penetration				Uptake l/m^3		Penetration				
	Gross	Net	Index	% at 1 mm	% at 2.5 mm	% at 5 mm	Gross	Net	Index	% at 1 mm	% at 2.5 mm	% at 5 mm	
4	84	51	72	-	78	-	199	155	87	-	93	-	x o
5	69	40	61	84	73	59	98	75	64	92	80	59	x o
6	78	50	64	86	78	64	104	78	66	93	84	63	x o
7	-	42	-	-	-	-	-	64	-	-	-	-	
8	96	65	68	82	82	67	98	80	69	93	84	67	x o
9	ASSUMED EQUAL TO COMPARISON 4												
10	104	84	58	-	67	-	113	91	67	-	78	-	x o
11	-	97	55	-	74	-	-	102	56	-	75	-	x
12	99	75	-	-	-	-	98	73	-	-	-	-	
13	87	63	-	-	-	-	106	80	-	-	-	-	
14	120	103	71	92	82	71	123	101	74	94	86	74	x o
15	119	96	69	95	85	68	104	81	68	94	84	67	x o
16	-	159	87	99	96	85	-	100	82	97	91	78	
17	-	203	-	-	-	-	-	148	-	-	-	-	
18	231	220	90	100	98	91	153	126	83	99	93	82	
19	-	220	-	-	-	-	-	142	-	-	-	-	
20	-	251	-	-	-	-	-	131	-	-	-	-	
21	282	274	95	100	100	98	171	160	83	99	95	80	
22	-	45	60	-	92	86	-	103	73	-	94	91	
23	80	60	52	86	71	46	103	84	67	92	82	63	x o
24	106	79	68	93	84	67	-	81	70	95	86	71	x o
25	143	120	70	92	85	69	122	95	69	95	85	62	x
26	-	137	80	97	93	84	-	56	50	86	68	46	
27	105	98	62	93	83	63	104	86	63	94	81	63	x o
28	131	117	-	-	-	-	116	96	-	-	-	-	
29	103	87	-	-	-	-	106	83	-	-	-	-	
30	106	97	-	-	-	-	116	94	-	-	-	-	
31	ASSUMED EQUAL TO COMPARISON 27												
32	119	98	-	-	-	-	126	102	-	-	-	-	
33	52	35	30	-	47	-	57	41	30	-	48	-	x o

[x] These data used to calculate average uptake and penetration of one-bar schedule - Table 3.7

[o] Data from these tests used to prepare summary of penetration patterns observed - Table 3.8

Figure 3.10 Influence of level of initial vacuum on impregnation schedule performance

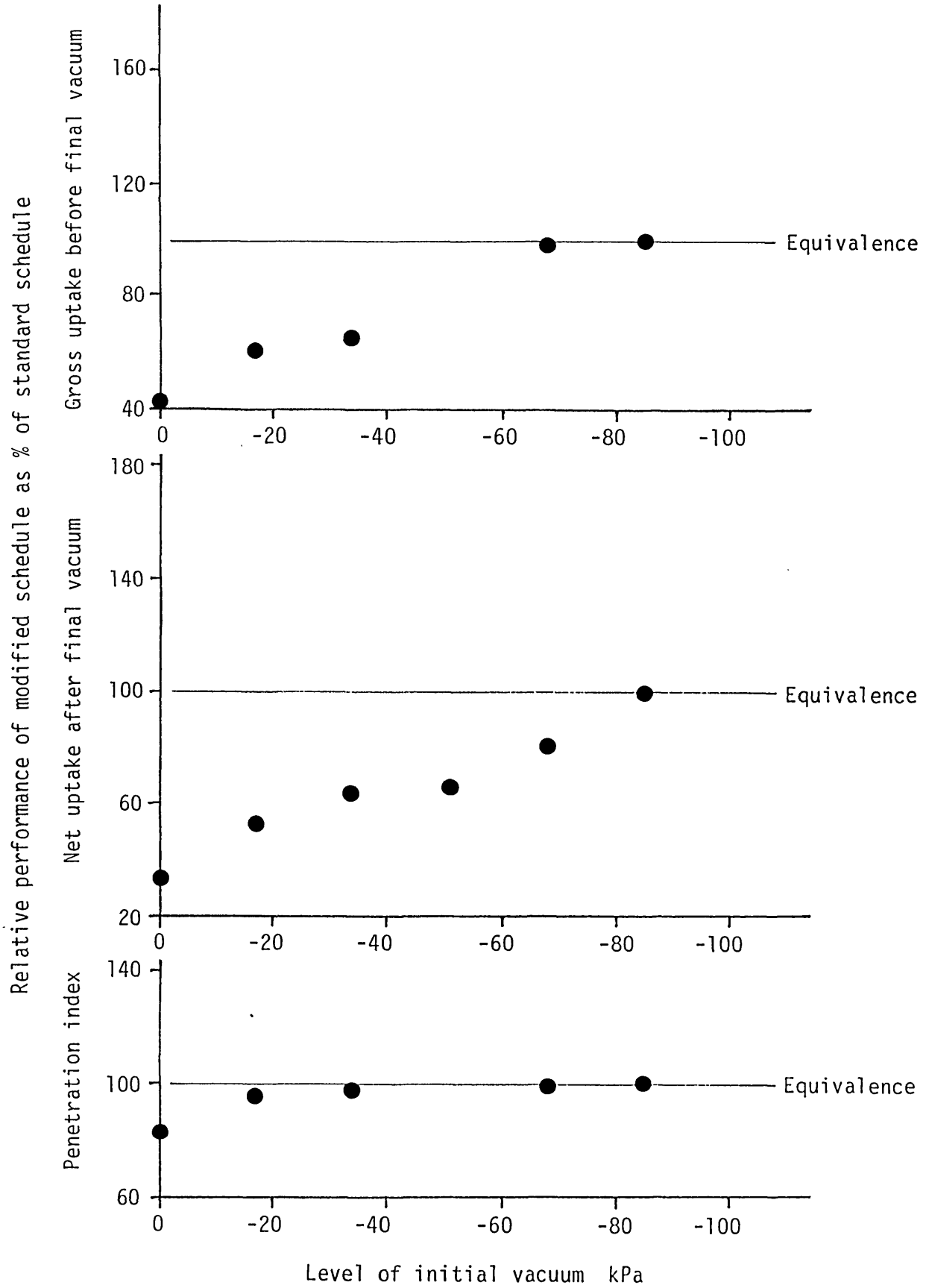


Figure 3.11 Influence of duration of initial vacuum on impregnation schedule performance

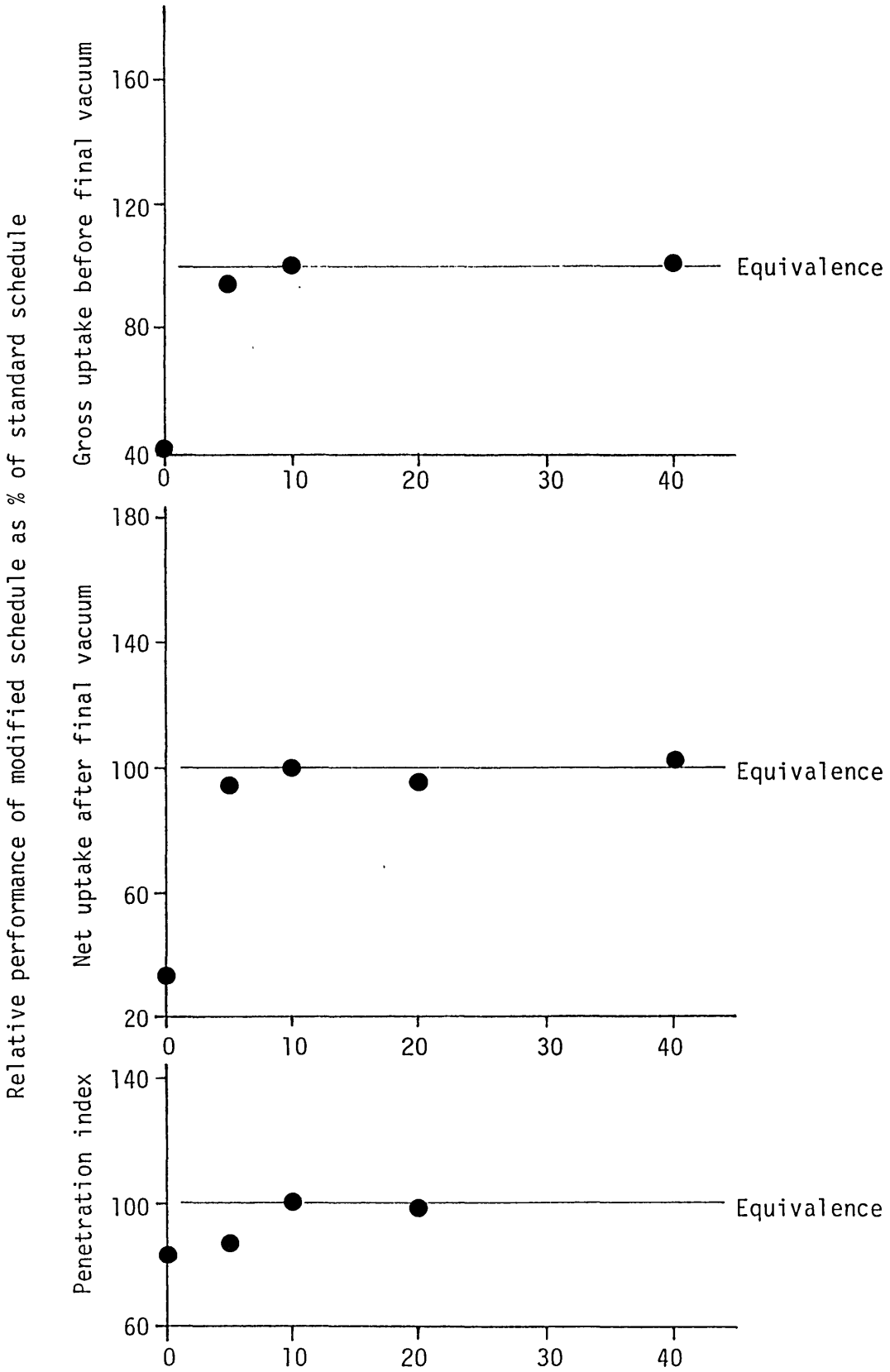


Figure 3.12 Influence of pressure during the immersion phase on impregnation schedule performance

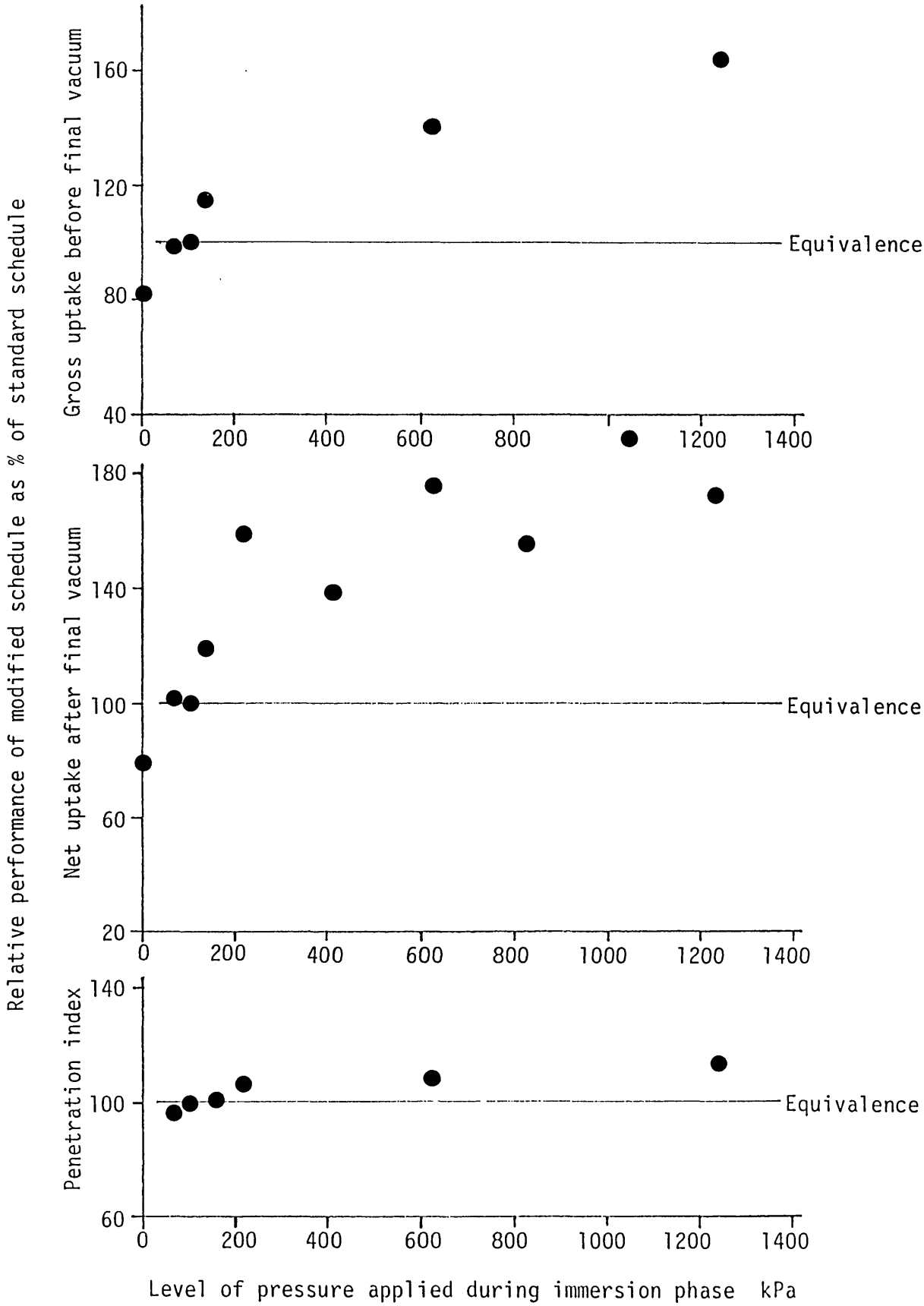


Figure 3.13 Influence of duration of immersion phase on impregnation schedule performance

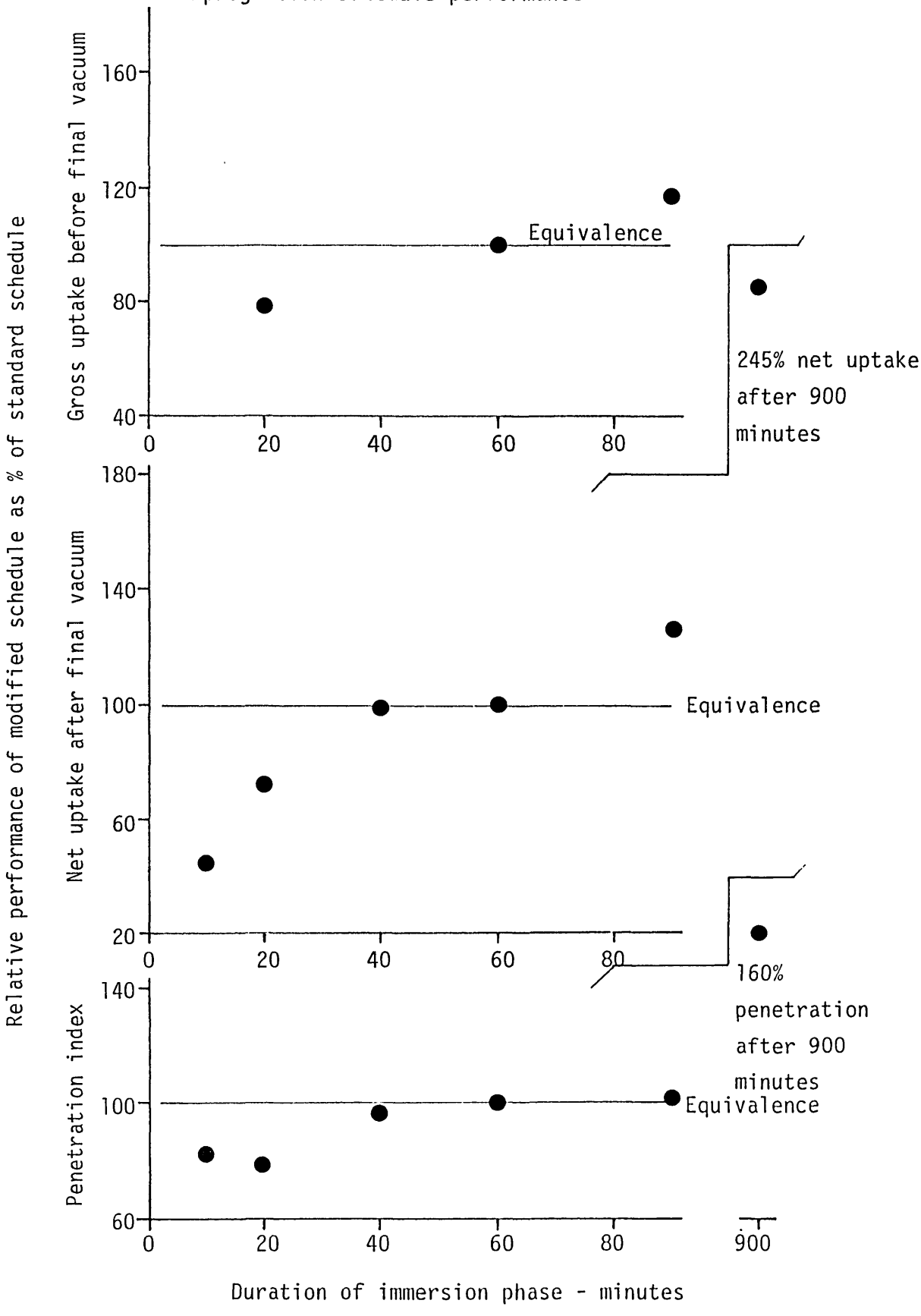


Figure 3.14 Influence of duration of final vacuum on impregnation schedule performance

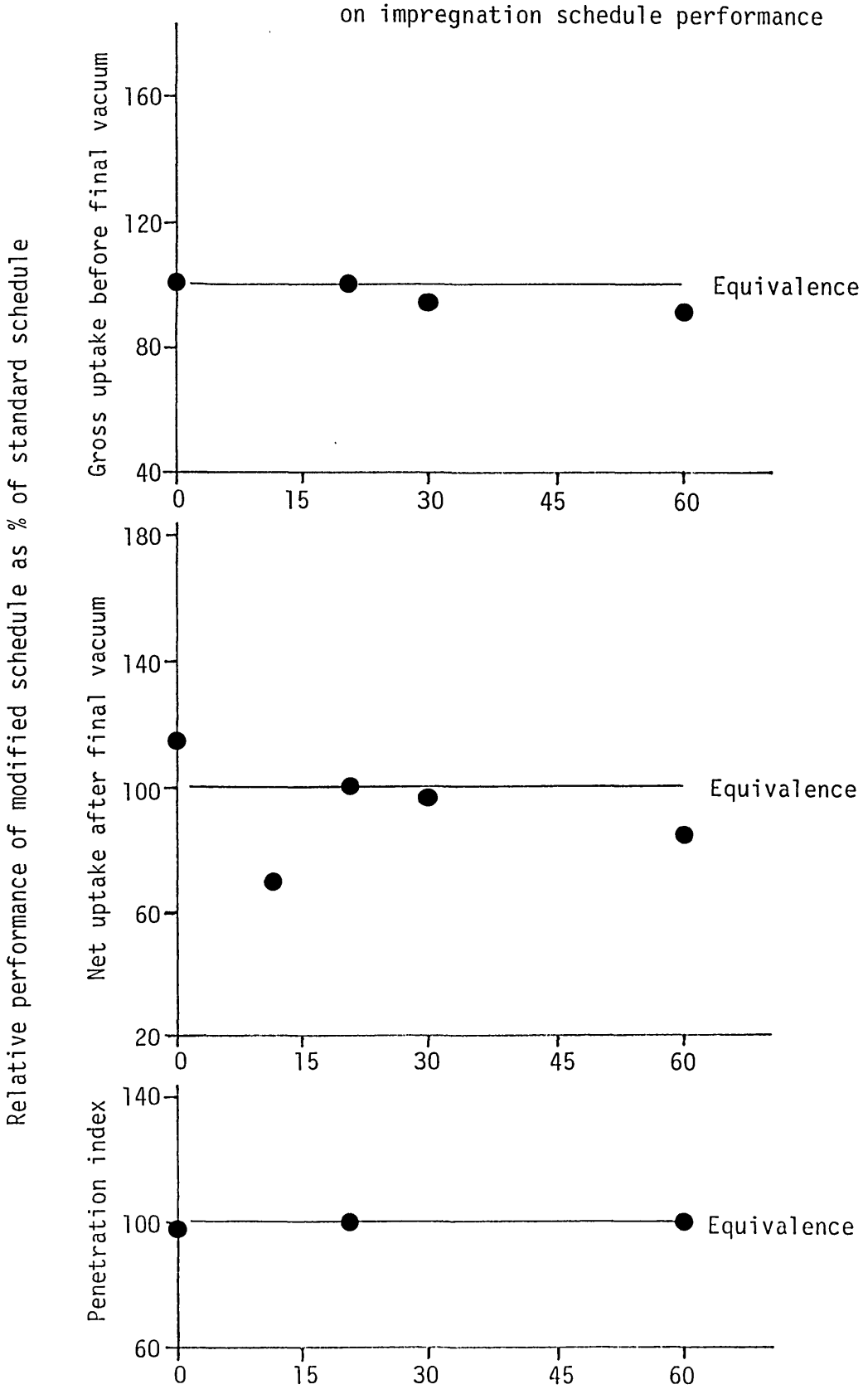
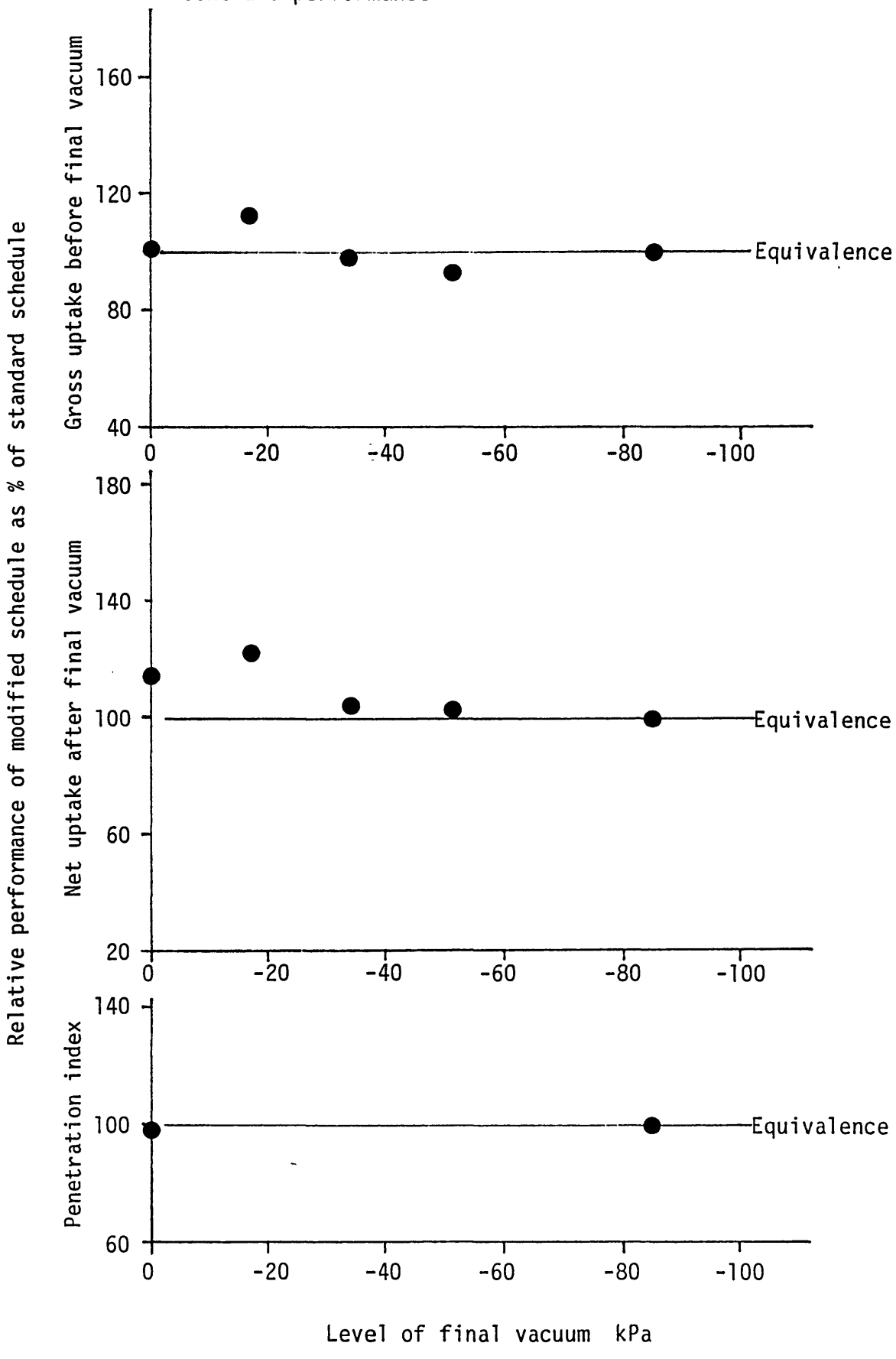


Figure 3.15 Influence of level of final vacuum on impregnation schedule performance



to pressure gradient, and the data in Table 3.9 and Figure 3.10 appear broadly to reflect such a relationship. It is also evident from Table 3.9 that the influence of initial vacuum on net uptake is due in part to lower gross uptake with low initial vacua, and in part to greater fluid recovery during the final vacuum phase, following a low initial vacuum.

Unexpectedly, above some threshold value between ambient pressure and -17 kPa, the severity of the initial vacuum had little or no effect on depth of penetration.

Duration of initial vacuum (Figure 3.11)

Use of no initial vacuum at all, or an initial vacuum (-85 kPa) of only five minutes duration resulted in both uptake and penetration being lower than that achieved by the standard schedule. However, prolongation of the initial vacuum beyond ten minutes did not further enhance either uptake or penetration. It would appear therefore that within ten minutes air was removed from all those tracheids to which liquid could subsequently gain access. The implication of the work reported in Section 2.2.3 is that continued evacuation removes air from deep within the sample but this does not appear further to assist the subsequent flow of liquid into the timber.

Level of applied pressure (Figure 3.12)

The level of pressure applied during the immersion phase clearly affected uptake. Again, from Section 2.2.2.3.2 it was anticipated that uptake and applied pressure would vary in direct linear proportion. However, such a relationship is clearly not evident throughout Figure 3.12, although the rather sparse data for the applied-pressure range 0 to 207 kPa may well be consistent with a linear relationship. Above 207 kPa, uptake did not rise consistently or appreciably with increased pressure, presumably indicating that 60 minutes immersion at 207 kPa effectively resulted in treatment which, although not total, was close to refusal. The uptakes recorded did not lend any support to the suggestion advanced in Section 2.2.3.4.4 that application of a particular pressure overcame blockages caused by air embolisms.

The effect of applied pressure on penetration was less marked than its effect on uptake. The ca. 70% increase in net uptake which resulted from treatment at 1240 kPa was accompanied by an increase in penetration of just ca. 14%.

A feature which is evident in several of Tests 4 to 33 is particularly clearly demonstrated in, for example, Test 21. In this test, the modified schedule's gross uptake was 282 l/m^3 and its penetration index was 95%. These two facts are not immediately easy to reconcile. 282 l/m^3 is sufficient preservative to fill no more than, at most, half the available void space and yet a

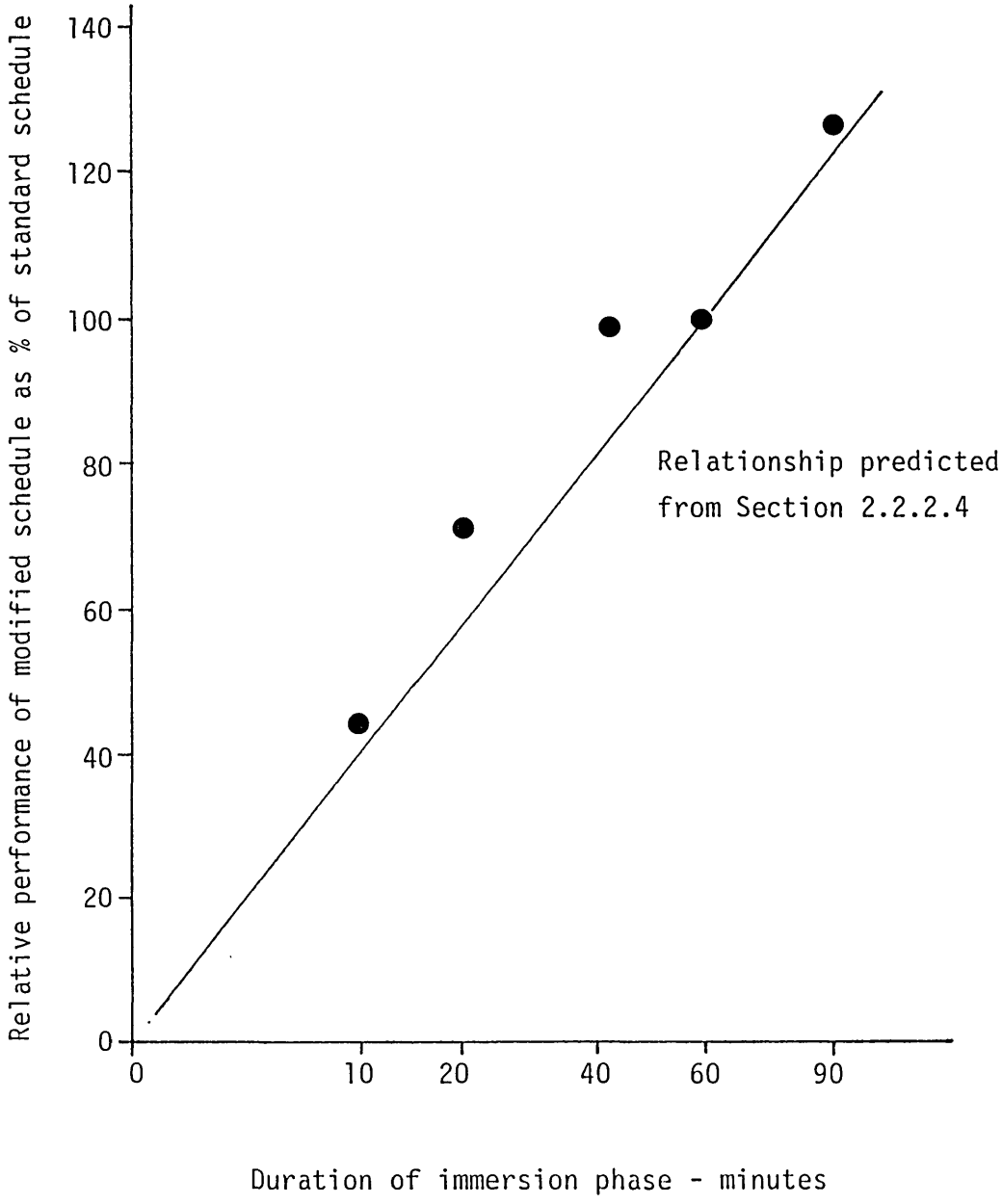
penetration index not of $\leq$ 50% but of 95% was recorded. Clearly, therefore, the liquid cannot have filled every void in the penetrated region. The ramifications of this are further discussed in Section 4.

An additional feature particularly clearly exemplified in the Test 21 modified schedule results is that, despite a penetration index of 95%, the pressure changes which were recorded within one of the samples did not imply substantial fluid movement (Section 2.2.3.4.4, Figure 2.41). Whilst it is not possible to assign too much significance to this observation alone (because the average penetration index does not necessarily relate to the particular sample in which pressure was measured), the fact that broadly similar indications came from several other tests does indicate that it appears to be possible for liquid to enter a sample quite extensively without causing as large a pressure increase as a-priori prediction would suggest. This too is further discussed in Section 4.

Duration of applied pressure (Figure 3.13)

From Figure 3.13 it is evident that uptake of liquid increases with the duration of the immersion phase. Other parts of this investigation (Section 2.2.2.4) have indicated that uptake varies with $\sqrt{\text{time}}$ and so the data in Figure 3.12 have been replotted on an exponential x axis (Fig 3.16). From this figure, there appears to be reasonably good general agreement with the expected uptake/

Figure 3.16 Net uptake data from Figure 3.1 replotted against time on a square-root scale



time relationship for immersion periods of 90 minutes or less. This relationship did not hold indefinitely, however, and the uptake which was recorded following an immersion phase of 900 minutes was considerably less than might have been predicted by linear extrapolation from the early part of the graph.

Although uptake varied quite widely in response to variations in the duration of the immersion-under-pressure phase (44 to 245% of standard) penetration was much less variable (78 to 160%). It should not be overlooked, however, that substantially prolonging the immersion phase was the only schedule modification which demonstrated materially-enhanced penetration.

Level and duration of final vacuum (Figures 3.14 and 3.15)

Prolonged and severe final vacua reduced net uptake of liquid but had negligible effect on penetration.

To summarise the discussion so far of Figures 3.10 to 3.15, the observed patterns of variation in uptake and penetration indicate that, by manipulating treatment schedule parameters, uptake may be adjusted within quite wide limits. In the present work, uptakes were reduced to as low as 33% of standard and increased to as much as 245%.

Penetration varied less widely. None of the variants tested reduced the penetration index by more than ca. 20%, and an increase in the penetration index of more than 14% was achieved only by a 15-fold increase in the duration of the immersion phase. Penetration thus appeared largely to be an innate characteristic of the timber, rather than a result of the way in which treatment was effected.

Although these results, particularly the poor correlation between uptake and penetration, initially appear somewhat anomolous, it is possible to propose two speculative models which together provide a logical framework with which these results are consistent. One of these models relates to the structure of European whitewood and the other describes the behaviour of the liquid, and both of these are discussed in Section 4.

3.2.2.2.3 Industrial implications of the results discussed in Section 3.2.2.2.2

In Section 1.5.3.6 it was noted that, although the one-bar schedule may result in excessively high uptakes of preservative, penetration is often not particularly deep. The results presented and discussed in Section 3.2.2.2.2 indicate that penetration of preservative may not readily be enhanced significantly. However, the results also indicate that, contrary to expectation (Kyte & Saunders 1978), uptake and penetration are not closely related and there is scope for modifying the one-bar schedule in order to reduce uptake of preservative without materially reducing penetration.

From the results already discussed (Section 3.2.2.2.2) it was concluded that the optimum balance between uptake and penetration would be achieved by use of a schedule involving a slight initial vacuum (-17 kPa) sustained for 10 minutes, followed by a 100 kPa (one-bar) pressure during a 40 minute immersion phase which should in turn be followed by a -85 kPa final vacuum sustained for 20 minutes.

Evaluation of this schedule which has been reported elsewhere (Saunders 1982) confirmed that this schedule did indeed reduce uptake (by ca. 60%) whilst barely reducing penetration, and this schedule has subsequently been incorporated into British Wood Preserving Association Standard 122 (1982) for the treatment of spruce in 'medium hazard' situations.

Errata

Throughout this thesis, the expressions 'gradient', pressure gradient' and 'pressure gradients' should respectively read 'differential', 'pressure differential' and 'pressure differentials'.

4 Final discussion

The aims of the present investigation have been set down in Section 1.6.

These aims provided the rationale for the experimentation which has been described in Sections 2 and 3. In these sections, four techniques for investigating aspects of the unsteady-state flow of non-polar liquid into European whitewood have been described, and fifteen groups of experimentation have been reported and discussed.

It is the purpose of this final discussion to draw together and develop the previous discussions of results, particularly highlighting those features which do not appear to be consistent with existing published information. To provide a framework within which the results obtained may be considered, 'models' for both the structure of spruce and for the behaviour of non-polar liquid within spruce, are proposed.

4.1 Review of results

To provide a basis for this final discussion, the salient features of the results obtained are now summarised:

The first piece of work described (Section 2.2.1) investigated the relationship between rate of liquid absorption by timber, and the

duration of immersion. This work yielded results which consistently indicated that, to a first approximation, '$\frac{1}{Q} \propto \sqrt{t}$' described the relationship between flow rate (Q) and elapsed time (t) for immersion periods of up to at least 150 minutes duration. This finding was unexpected in as much as it had been anticipated that any or all of three effects (decreasing pressure gradient, random path termination and air embolism formation) would distort this relationship by impeding liquid flow (Kelso et al, 1963, Bramhall 1971 and Banks 1970).

The second type of investigation (Section 2.2.2) involved the use of a purpose built flow meter to monitor the entry of liquid into a restricted area of timber surface. In this apparatus, liquid flow into spruce proceeded at a rate which was highly variable from sample to sample and which did not correlate closely with the uptake which resulted during conventional, industrial impregnation. However, the shapes of the flow/time graphs for all the samples were closely similar. This was interpreted as indicating that rate of flow into each sample was determined by the characteristics of that part of the timber which was closely adjacent to the surface.

Experimentation using the flow meter also indicated that the tendency of a pressure gradient of a given size to promote flow was the same irrespective of whether that gradient was created by partially evacuating the sample or by applying pressure to the liquid. This, like the results of the 'constant volume' flow monitoring work, was interpreted as indicating that air embolism

formation was not an impediment to the flow of non-polar liquid into spruce.

The flow meter was also used to investigate the effect of magnitude of pressure gradient on flow rate, and whereas a ' $V \propto \sqrt{\Delta P}$ ' relationship had been anticipated (Petty 1975), the relationship ' $V \propto \Delta P$ ' more nearly fitted the results obtained. This latter relationship is more indicative of steady-state flow than of the unsteady-state behaviour which had been anticipated.

Work with the flow meter yielded clear (although limited) indications that, although the standard flow equations consider solely a fluid's physical properties, its chemical characteristics also profoundly affect flow.

The experimentation which concerned the measurement of pressures within timber during impregnation (Section 2.2.3) revealed that the initial vacuum phase of an industrial-type impregnation schedule could establish vacua within samples of spruce which were not relieved during subsequent immersion-under-pressure. Mathematical analysis of the results was complex, involving several assumptions. This analysis did however indicate that the difference in permeability of spruce to air and to non-polar liquid was not merely a reflection of the different viscosities of these two fluids. Rather, it appeared that, relative to the flow of air, the flow of liquid was disproportionately slow. Additionally, it appeared that the relationship between permeability to liquid and to gas was not constant between samples.

Other features which emerged during the internal-pressure investigations suggested that spruce timber consists of a reticulum of pathways. During evacuation, gas may be removed from deep within timber, along pathways of high permeability, whilst,simultaneously, gas removal may be less complete from more superficial tissues which are less permeable.

Internal pressure measurement also revealed that liquid is able to 'maintain station' within spruce for many hours or days despite the existence of a pressure gradient across it. Moreover the liquid is also able to increase the magnitude of a pressure gradient by moving, presumably as a result of capillarity.

The fourth, and final, type of experimentation undertaken as part of this present investigation involved examination of the reaction of spruce to a range of preservative impregnation schedules (Section 3, especially 3.2.1 et seq.). This work revealed that, when treated by the standard industrial 'one-bar' schedule, most samples of spruce which were examined exhibited evidence of having a fairly permeable sapwood which could be impregnated deeply. Spruce heartwood however was much more variable and overall was much less permeable. About half the samples examined contained portions in which the latewood had been penetrated more deeply than the earlywood, but very few samples showed the converse. Almost no samples showed evidence of penetration proceeding through the rays.

Modifications to the industrial one-bar schedule resulted in quite substantial variation in uptake. Significantly, penetration varied much less than uptake, giving rise to the firm, although unexpected conclusion that uptake and extent of penetration are not necessarily directly proportional (c.f. Kyte & Saunders 1978).

4.2 Interpretation of results from Section 3

Considered separately, several of the results reviewed in Section 4.1 are not susceptible of conclusive interpretation. However, a framework may be derived from the results of the industrial-treatment investigations (Section 3.2.2) with which most of the results can be shown to be consistent. Therefore in the remainder of this discussion, this framework will be presented and used as a basis for reconsideration of the results obtained.

The results of Section 3.2.2.2 in particular, provoke two questions:

(i) Why was penetration so little affected by the treating schedule used?

(ii) How was it that quite substantial variations in final uptake could be associated with fairly insubstantial variations in extent of penetration?

In considering the first of these questions, the results presented in Section 3.2.2.2 can be interpreted as indicating that in each sample of spruce, certain regions are readily accessible to liquid and become penetrated almost regardless of the schedule used, whereas other regions are not accessible to liquid and almost no amount of schedule manipulation will make them so. The fact that only negligible evidence of radial penetration could be found was interpreted as being indicative that liquid flow occurred, if at all, through the bordered pits of the axial tracheids. Accordingly an SEM examination of the bordered pits of timber, taken from a sample which had been impermeable during treatment, was undertaken. This examination was unfortunately very limited in its extent, but it did not yield a conclusive indication of 100% pit aspiration in impermeable timber. This observation led to postulation of the following 'model' for the structure of European whitewood.

Model for European whitewood structure

It is known that during drying and heartwood formation in softwoods, bordered-pit aspiration occurs, and it is this which reduces permeability (Hunt and Garratt, 1953). It may reasonably be supposed, therefore, that dry spruce which retains a comparatively high permeability contains a higher than normal proportion of unaspirated or imperfectly aspirated bordered pits. Conversely, very impermeable dry spruce contains only few unaspirated pits. Most dried spruce heartwood is observed

to fall between the two extremes of total permeability and total impermeability. It follows, therefore, that most dried spruce heartwood contains a mixture, probably a fairly random mixture, of aspirated and unaspirated or incompletely aspirated pits. This must, almost inevitably, result in 'clusters' of tracheids which have intercommunicating flow paths between tracheids within each cluster, but which, by aspirated pits, are sealed off from the surrounding timber.

During conversion of dry spruce, many of the clusters of interconnecting tracheids will be cut 'open'. During subsequent impregnation, preservative liquid can gain relatively easy access to the lumena of the tracheids in the 'open' clusters near the timber's surface. However, penetration beyond these clusters is rather difficult to achieve. Thus, above certain impregnation schedule minima, the characteristically irregular penetration of liquid is similar in extent.

Relationship between uptake and penetration

The above model forms a basis of explanation for the observed similarity in the extent of penetration which occurred, irrespective of the impregnation schedule used. It does not however present an answer to the second question posed above, namely:

"How was it that quite substantial variations in final uptake could be associated with fairly insubstantial variations in the extent of penetration?"

To answer this question, attention must be turned from the timber to the liquid itself, and its behaviour within the timber, as indicated by the assessments of penetration which were made.

In part, some of the lower uptakes relative to the amount of penetration can be accounted for by proportionately greater liquid recovery during the final vacuum phase when low initial vacua were used. A partial series of measurements of gross uptake as well as net uptake was made and they did indicate that the proportion of gross liquid uptake which was recovered during the final vacuum phase was greater when lower levels of initial vacuum were used. These data also showed that, where low initial vacua were employed, the gross as well as the net uptake was decreased. Notwithstanding this, penetration did not decrease concomitantly.

During examination of samples and quantification of penetration, penetration was scored on a 'go/no-go' basis and no formal assessment was made of the intensity of colour at each point inspected. However, it may be stated as a general observation that, where the uptake of preservative by a sample was low relative to the extent of penetration, the penetrated regions were, for the most part, less intensely coloured by tracer dye (this effect is demonstrated

in Plates 4.1 and 4.2). Whilst this observation is, of itself, unsurprising, it is of fundamental importance to establish precisely what is indicated by differing levels of intensity of tracer dye. Direct investigation of this point is clearly an area where further work is required (Section 6) because intensity of colour may vary either with the number of 'liquid-filled' tracheids in the penetrated zone, or with the amount of 'liquid' in each tracheid.

If paler colour in fact indicated a lower proportion of liquid filled tracheid lumena within the apparently penetrated region, a possible explanation of why this should have occurred may be based on the phenomenon of air embolisms causing blockage of small capillaries (Section 1.3.4). These embolisms may be cleared by the application of pressure, higher pressures being required to clear narrower capillaries (Siau, 1971). If the narrow flow paths through pit membranes become blocked by air embolisms, application of moderate pressure would clear some of the wider paths. The greater the applied pressure, the more of these blockages would be cleared and hence the higher the proportion of tracheids which would be filled in the penetrated region.

This explanation does not by itself seem totally satisfactory however. In view of the fact that liquid has to move sequentially from tracheid to tracheid, air embolisms in its path would be expected to restrict the depth of penetration. However, from the results, it is clear that this was not observed. An alternative

Plates 4.1 and 4.2 show cross cut samples of seasoned spruce in which penetration of blue-dyed organic solvent preservative is broadly similar in gross extent, whilst the sample in Plate 4.1 is more heavily loaded.

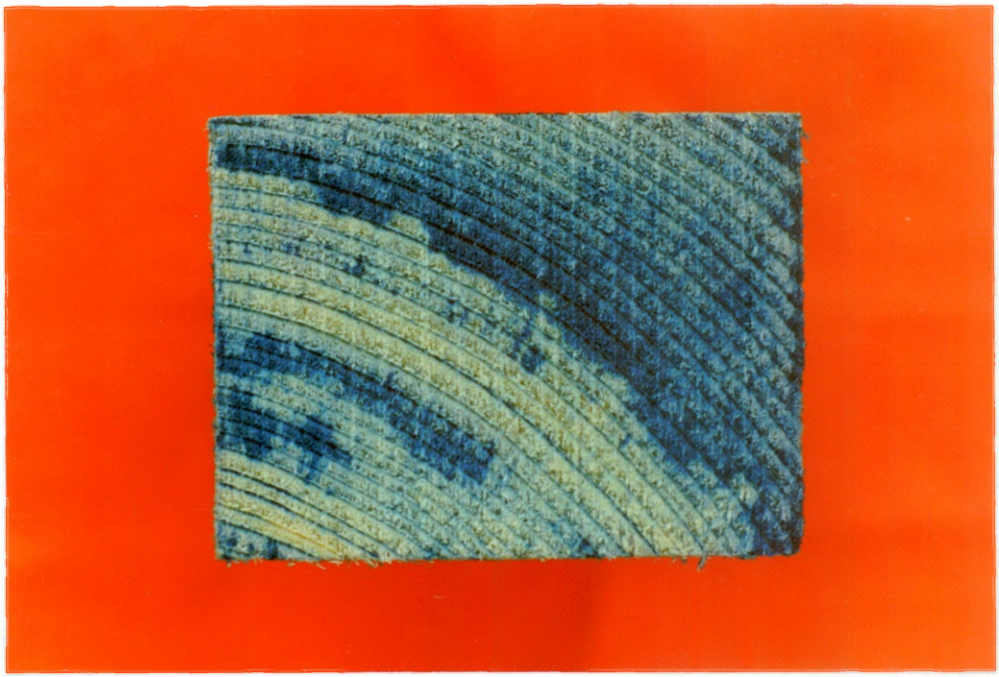


Plate 4.1 - For narration see facing page

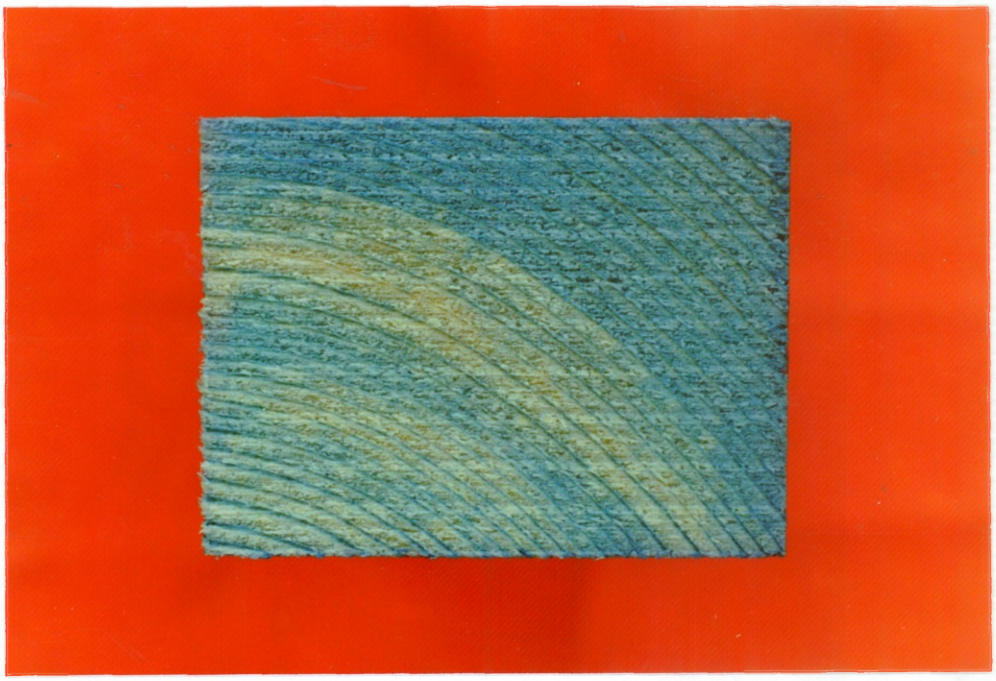


Plate 4.2 - For narration see facing page

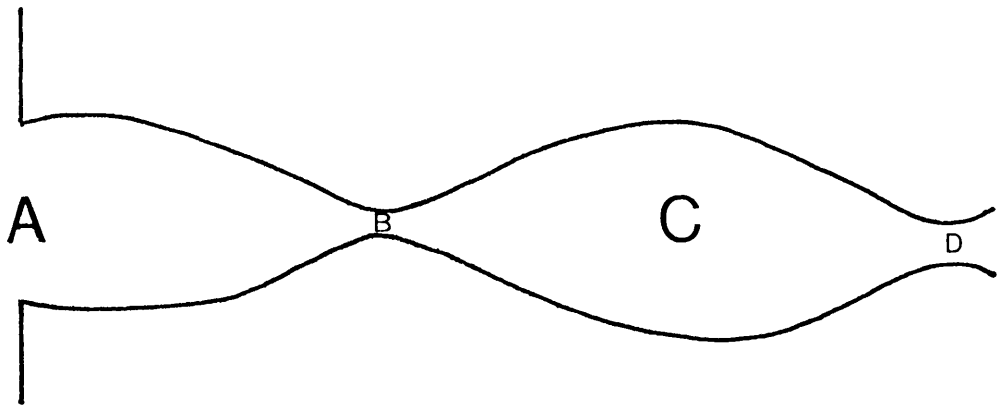
interpretation of the appearance of the samples is that pale tracer-dye-colour indicated tracheids penetrated, but with only a little liquid in each. In considering this mechanism it should be recalled that the white spirit-type solvents used in organic-solvent-borne wood preservatives are highly wetting to timber. They exhibit an advancing contact angle against Picea abies of approximately 25°, although accurate measurement of this is a little difficult. This is due to the speed at which this angle tends to zero as the solvent creeps along the timber surface. In this context it may also be noted that the need for 28 days drying time between preservative impregnation and penetration assessment (Section 3.1.2.1) arose because, if the samples were cross-cut while they still contained appreciable residual solvent, this crept across the freshly exposed transverse surface at a rate fast enough to interfere with accurate penetration measurement. A further factor which should be recalled from Section 3.2.2.2 is that, when samples were cross-cut for penetration assessment, there were frequently two quite clearly defined levels of intensity of the tracer dye (so called 'two tone' penetration), with the darker colour occurring around the periphery of the sample. (Chemical analysis has confirmed that the visually apparent intensity of dye colour shows good correlation with the concentration of preservative.)

These facts, coupled with the results already presented, may be interpreted according to the following model.

Speculative model for the behaviour of non-polar liquid in spruce

Figure 4.1 shows a schematic representation of a simple flow path through tracheids adjacent to a timber surface (A).

Figure 4.1 Schematic representation of a softwood flow path



For the purposes of this discussion, the wider portions (A and C) of the flow path depicted in Figure 4.1 represent the lumena of tracheids while the narrower portions (B and D) represent constrictions such as the pores in the margo, or, perhaps, spaces between the tori and pit aperture walls in imperfectly aspirated pits.

Liquid entering an open end of a tracheid ('A' in Figure 4.1) will flow in, either purely in response to capillarity at a rate described by the Rideal Washburn equation (Section 1.3.3.5), or at some higher rate if a pressure gradient exists. As the liquid advances towards 'C', its volume-rate of advance will be retarded as the diameter of the flow path diminishes. As the liquid flows through the narrow part of the flow path (B) and into another wide portion, the maximum rate of flow which the constriction will permit will be critical to determining the subsequent behaviour of the liquid.

If flow through constriction 'B' can occur sufficiently rapidly, void 'C' will become filled with liquid before constriction 'D' is encountered.

Conversely, if the flow through 'B' is sufficient only to satisfy the demand for liquid created by the tendency of the liquid to wet and creep along surfaces it touches, much of the liquid entering cavity 'C' may creep around the walls, leaving little liquid to fill the space within the void. Thus, by the time constriction 'D' fills with liquid, the walls of the tracheid may be coated with preservative whilst its lumen remains unfilled. Such a mechanism would result in tracheids full of liquid, and thus intensely coloured with tracerdye, around the periphery, and tracheids much less full of liquid, and hence much less deeply coloured, further in the sample. A stepped-change in colour intensity, as reported in Section 3.2.2.2, might also be produced.

This mechanism provides a basis of explanation for the fact that smaller pressure gradients (and thus smaller rates of flow through constrictions such as that at 'B') resulted in lower uptakes relative to the penetration achieved, because they would have produced fewer full tracheids.

The model outlined above appears to offer a satisfactory basis on which to interpret the results presented in Section 3.2.2.2, and is thus proposed as a basis for further experimentation (Section 6).

4.3 Reappraisal of results from Section 2

In Section 4.2, conceptual models have been derived from the results in Section 3, both for the structure of seasoned spruce, and for the behaviour of non-polar liquid within spruce. It is appropriate, therefore, to examine the extent to which these models may also be used to interpret the results presented in the various parts of Section 2.

The 'constant volume' flow monitoring work (Section 2.2.1) led to the overall conclusion that, at the pressures tested, for periods of up to 150 minutes, the relationship between uptake (V) and time (t) could be described by the relationship $V \propto \sqrt{t}$. It was noted, however, that this conclusion was not apparently consistent with established theory (Section 2.2.1.4) because the three phenomena of bubble formation, random path blockage and diminution of ΔP might all be expected to retard flow. However,

having in mind the models postulated in Section 4.2, the results reported in Section 2.2.1 may indicate that the overall rate of flow into samples of dried spruce is initially limited by the rate of flow which is possible through constrictions near their surfaces. Thus random flow path termination (i.e. saturation of the accessible voids) does not begin to restrict flow for some considerable period. Additionally, it has been shown by monitoring pressure within timber during impregnation (Section 2.2.3.4.3) that the pressure within a sample of timber may not rise appreciably during impregnation, wherefore decrease in ΔP would be small. Thus, of the three causes which might have decreased flow-rate, two can be discounted, and it is noted that the third, namely bubble formation, has been demonstrated in aqueous systems (Kelso et al 1963) but not elsewhere.

It thus appears that the results obtained from the 'constant volume' flow monitoring are consistent with the mechanisms outlined in Section 4.2.

The investigations involving the flow meter (Section 2.2.2) were divided into seven sections, of which two involved liquids other than white spirit. The remaining five sections yielded results which variously indicated the variability of spruce, and the fact that '$V \propto \Delta P$' appeared to describe the relationship between uptake (V) and pressure gradient (ΔP).

The mechanisms outlined in Section 4.2 go some way to explaining how it was possible for the flow rates which were recorded to differ so much from sample to sample whilst being very similar in pattern. As with other results discussed elsewhere (Section 2.2.1.4) it is possible to postulate that the rate of inflow was controlled by constrictions (analagous to 'B' in Figure 4.1) closely adjacent to the surface, so that path termination was not encountered during the observation period. In postulating this it is noted that an unsatisfactory feature of this proposition is that it requires that the rate determining apertures always occur close to the surface rather than more deeply within the timber. In Section 2.2.2.3.1 it has been suggested that aperture-occlusion by debris or damage during machining, and/or surface drying effects, may create the constrictions. This is pure speculation however. In an attempt to generate supplementary data on this point, a limited investigation was conducted in which twenty, 500 mm lengths of timber were docked from the ends of dry beams and were then immediately impregnated with organic solvent preservative. Longitudinal penetration was examined on sawn longitudinal surfaces to look for evidence of inferior penetration from the original end as compared with the fresh end. However, no evidence of this effect could be seen. It appears therefore that further work is required to derive a satisfactory understanding of whether the rate determining constrictions are for some reason confined to the surface layers or whether they are more widely distributed throughout each piece of timber.

Other information obtained from the flow meter work concerned the relationship between uptake and applied pressure. It was demonstrated that, whether a pressure gradient was produced by partial evacuation of the sample or pressurisation of the impregnating liquid, the uptake obtained was similar. This was interpreted as evidence that air embolism may not play a large part in the restriction of the flow of organic solvents into spruce and in this connection it is appropriate to observe that a role for air embolism formation has not been included in the model for liquid behaviour which has been outlined in Section 4.2.

Perhaps the most interesting result to emerge from the flow meter work was that the rate of inflow was proportional to ΔP and not to $\sqrt{\Delta P}$. The classical explanation of this, and the one which has been applied to other data by Petty (1978b) is that greater pressures dislodge air embolisms and thus enhance flow in a way unrelated to Darcy's Law. However, allusion has already been made (Section 2.2.2.3.2) to the coincidence involved in the relationship $V \propto \sqrt{\Delta P}$ being randomly modified to give the relationship $V \propto \Delta P$. In addition to this it may be noted that Petty (op cit) used data published by Purslow (1976). However, repetition of the work discussed by Purslow yielded slightly different results (Saunders 1977) which were, moreover, broadly consistent with the relationship $V \propto \Delta P$. For this to be consistent with an air embolism mechanism, it is necessary that the timber must contain a random spread of pore sizes through which embolisms have to be forced. However, recent work (Baines et al 1983) suggests this distribution

may not be present. It is therefore suggested that a more satisfactory explanation may be advanced based on the model outlined in Section 4.2. If, as suggested in the model, the speed of flow is governed by the rate-determining effect of short constrictions in the flow paths, the dynamics of liquid flow through these constrictions will be close to steady-state in as much as each constriction will contain a constant volume of liquid flowing in response to a virtually constant pressure gradient.

In summary, the mechanism outlined in Section 4.2 forms a fairly satisfactory basis for interpretation of the results obtained from the flow meter work. Additionally, if the proposed mechanism is accepted as a basis of interpretation, none of the results obtained lend any direct support to the proposition that air embolisms affect the flow of organic liquids through spruce (Petty 1978c).

The third type of investigation reported in Section 2 involved measurement of pressures inside timber (Section 2.2.3.3). The results obtained demonstrated that the permeability of spruce to air varied considerably. This may indicate that the integrity of the 'seal' around the clusters of tracheids postulated in Section 4.2 is variable. Alternatively it might be that the concept of 'sealed clusters' has limited applicability to gas flow. Instead it may be that even tightly aspirated pits have some residual permeability to air, although the apertures through which flow occurs are so small as to be substantially impermeable to liquids unless very high application pressures are used.

One of the conclusions of Section 2.2.3.3.3 was that the differences in the rates at which air can be removed from spruce compared with the rate at which white spirit may be introduced into it may be greater than can be accounted for purely by considering the different viscosities of the two fluids. Once again, the accepted way in which this sort of phenomenon is explained is on the basis of air embolisms blocking flow paths. However, here as elsewhere in this discussion, it is suggested that this is only one of a number of possible mechanisms. An alternative explanation which is consistent with the model for liquid flow proposed in Section 4.2 can be advanced.

During initial evacuation of spruce timber, air flows out of the timber, during which the whole cross-section of each open flow-path may be used. In addition, flow through constrictions such as openings in pit apertures may not necessarily be laminar or turbulent. Instead, more rapid Slip Flow may occur (q.v. Section 1.3.1.3). If Slip Flow does occur, subsequent inflow of liquid will be slower, out of proportion to differences in viscosity, because Slip Flow is a regime unavailable to liquids. In addition, if the liquid behaves as outlined in Section 4.2, in many of the flow paths the liquid will not utilise the entire cross-section potentially available. These two effects could together lead to the differences in flow rates which were discussed in Section 2.2.3.4.3.

Of the results obtained by measuring pressure inside timber, those which are most difficult to interpret on any known physical basis are the pressure fluctuations which were observed in Sample I of Section 2.2.3.4.5. However, as has been indicated in that section, there is no apparent basis for discounting the data and it is thus necessary to continue to seek an interpretation of them. In this connection, the model for liquid behaviour proposed in Section 4.2 merits consideration.

According to the traditional concept of liquid sequentially filling one lumen after another as it flows into a sample of softwood, it can be argued that there will be only two pressures within each flow path during impregnation. One of these will be the pressure in the surrounding liquid (pressure rapidly equilibrates in all directions in a continuous mass of liquid) and the other will be the pressure in the gas in the inner end of the flow path. However, with the model proposed in Section 4.2, there is clear potential for the groups of lumena between successive fluid-filled constrictions in the flow path to have different internal pressures.

After immersion it is probable that pressure fluctuations occur in response to continuing movement of liquid both around the complex reticulum of pathways within timber, and also possibly through small apertures into comparatively less permeable regions. This liquid movement might result in local decreases in pressure caused by liquid flowing in such a way as to increase the volume of gas it encompasses. Conversely, local increases in pressure

may occur if liquid flows from elsewhere in the sample into a cluster of tracheids and in so doing decreases the volume of gas therein. Additionally, both increases and decreases in pressure might result from the dispersion of a liquid plug separating two clusters of tracheids having dissimilar internal pressures.

The above interpretation of the pressure fluctuations reported in Section 2.2.3.4.5 is of course purely speculation. However, the model proposed in Section 4.2 does present a basis for such speculation which would not otherwise be possible.

4.4 General Review

Section 4.3 has demonstrated that most of the results obtained from the investigations described in Section 2 are broadly consistent with the behaviour predictable from the speculative mechanisms proposed in Section 4.2. It is therefore a conclusion of this whole investigation that the models proposed in Section 4.2 provide a conceptual framework for describing the behaviour of white spirit-based solutions in spruce.

However, in view of recent work (Saunders & Zuvcenko 1986, Morgan 1986) it is not yet clear whether the above models have validity for solutions other than those based on white spirit. It appears that other liquids with physical and chemical characteristics apparently similar to those of white spirit behave somewhat differently in timber, and this observation needs further

investigation. It is clear that consideration of a liquid's physical characteristics alone is not adequate to permit its flow characteristics in timber to be predicted (Section 2.2.2.3.5 and Saunders & Blow 1985).

5 Conclusions

The following conclusions have been drawn from the foregoing work.

5.1 Many of the results obtained are more consistent with the proposition that flow of light organic solvent into spruce involves creep of liquid along the walls of incompletely filled tracheids, rather than with the 'traditional' proposition that liquid inflow involves sequential filling of successive tracheids with occasional path occlusion by air embolisms.

5.2 The physical properties of a liquid are neither the sole, nor necessarily the dominant characteristics which affect the unsteady-state flow of liquid into timber.

5.3 During industrial double-vacuum-with-pressure impregnation of spruce, the relationship between uptake and elapsed time is, for all practical purposes, described by 'Volume absorbed $\propto \sqrt{\text{Immersion time}}$', for periods up to ca. 150 minutes.

5.4 During lateral flow of light organic solvent, in laboratory apparatus, into samples of spruce, the relationship between uptake and pressure differential was, for all practical purposes, 'Volume absorbed $\propto$ Pressure differential' for pressure differentials of up to ca.400 kPa.

5.5 During industrial double-vacuum-with-pressure impregnation of spruce, the quantity of liquid absorbed by the timber and the extent of that liquid's penetration are not necessarily directly related.

5.6 If a negative pressure (i.e. a pressure below atmospheric pressure) is created in spruce prior to its immersion in light organic solvent, that negative pressure may persist for a period stretching into weeks or even months.

5.7 On the basis of pressure measurements within samples, this investigation has not established a consistent relationship between the rate at which air can be withdrawn from spruce during evacuation, and the rate at which liquid can subsequently be made to enter that timber.

5.8 From the point of view of impregnation with liquid, spruce heartwood appears to consist of irregular, discrete clusters of interconnecting tracheids which are most difficult to access if they are not 'opened' during timber conversion.

6 Areas for further work

This investigation, and related work, have highlighted the following three areas in particular in which further investigation is required.

6.1 In Section 2.2.3 results were presented which, unexpectedly, indicated that there did not appear to be a consistent relationship between the rate at which air could be withdrawn from a sample, and the rate at which liquid could subsequently be made to enter that sample. However, in Section 4 a mechanism for liquid behaviour was proposed which involved creep along tracheid walls. This could lead to pockets of air within a liquid flow-stream, in which situation the relationship between pressure change in each pocket and the volume of liquid entering the whole sample would be very loose. Further experimentation aimed at looking at pressure profiles at several locations within orientated samples during evacuation and impregnation might clarify how close, at least in gross terms, is the relationship between permeability to air and unsteady-state permeability to liquid.

6.2 In Section 3, results were presented which indicated that high levels of penetration (95%) were achieved with quantities of liquid that could not possibly have filled all the void space in the samples. It was argued that the available information led to the conclusion that this could be explained

in terms of most tracheids being incompletely filled, rather than in terms of a minority of tracheids being completely filled with the remainder not being penetrated at all. On this conclusion rests the whole of the proposed mechanism for liquid behaviour which has been advanced in Section 4. Direct investigation of this conclusion is obviously, therefore, desirable. A suitable experimental approach to this investigation might be x-ray analysis of the internal walls of tracheid lumena under a scanning electron microscope, although when this approach was adopted in the present investigation, satisfactory results could not be obtained.

6.3 Since this investigation was undertaken, it has been found that light organic solvents other than white spirit, but having apparently similar physical characteristics, behave somewhat differently in timber (Saunders & Zuvcenko 1986, Morgan 1986). It has in fact become clear that even the longitudinal flow of organic liquids by capillarity into permeable softwoods is very imperfectly understood. Fundamental investigation of the effect of a wide range of liquid properties on the behaviour of liquid in timber is therefore required because until 'simple' situations, such as longitudinal flow by capillarity, are understood, there can be little prospect of arriving at a satisfactory fundamental understanding of more complex systems.

7 References

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Appendix 1 Sheet 1 of 3 Summary of results generated from experimentation described in Section 3.2.1

Schedule Comparison Reference Number [1]	Experimental Schedule (E) or Standard Schedule (S)	Uptake				Penetration - Quantitative				Penetration - Qualitative % of samples exhibiting features indicated										
		Gross l/m^3	Mean Nett l/m^3	Coefficient of variance (Net uptake)	Difference significant at 95% level	Index	at 1.0 mm	at 2.5 mm	at 5.0 mm	Cont. env.	Dis. env.	Banded	Broken	Zero L.W.	Preferred L.W.	Zero E.W.	Preferred E.W.	'Sapwood'	'Radial'	2-tone
1	E	-	45	50	YES	60	-	92	86	-	-	-	-	-	-	-	-	-	-	-
1	S	-	103	43	-	73	-	94	91	-	-	-	-	-	-	-	-	-	-	-
2	E	-	84	69	YES	57	-	91	-	-	-	-	-	-	-	-	-	-	-	-
2	S	-	148	65	-	73	-	91	-	-	-	-	-	-	-	-	-	-	-	-
3	E	-	51	31	YES	29	-	67	-	-	-	-	-	-	-	-	-	-	-	-
3	S	-	65	35	-	40	-	71	-	-	-	-	-	-	-	-	-	-	-	-
4	E	84	51	37	YES	72	-	78	-	1	99	0	0	29	100	99	1	100	0	-
4	S	199	155	37	-	87	-	93	-	10	90	0	0	7	100	93	0	100	0	-
5	E	69	40	35	YES	61	84	73	59	19	81	17	21	64	24	83	3	100	0	-
5	S	98	75	44	-	64	92	80	59	33	67	20	17	57	33	67	1	100	0	-
6	E	78	50	34	YES	64	86	78	64	17	83	21	17	73	26	83	0	99	1	-
6	S	104	78	43	-	66	93	84	63	39	61	15	24	52	28	69	0	100	3	-
7	E	-	42	58	YES	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
7	S	-	64	60	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
8	E	96	65	52	YES	68	82	82	67	37	63	29	15	59	19	63	1	100	4	69
8	S	98	80	45	-	69	93	84	67	39	61	22	18	58	30	61	0	100	2	77
9	E	Assumed equal to comparison 4																		
9	S																			
10	E	104	84	41	NO	58	-	67	-	4	96	17	6	17	100	97	1	96	0	-
10	S	113	91	43	-	67	-	78	-	6	93	16	7	13	100	95	0	99	0	-
11	E	-	97	64	NO	55	-	74	-	-	-	-	-	-	-	-	-	-	-	-
11	S	-	102	63	-	56	-	75	-	-	-	-	-	-	-	-	-	-	-	-

Appendix 1 Sheet 2 of 3 Summary of results generated from experimentation described in Section 3.2.1

Schedule Comparison Reference Number [1]	Experimental Schedule (E) or Standard Schedule (S)	Uptake				Penetration - Quantitative				Penetration - Qualitative % of samples exhibiting features indicated										
		Gross l/m³	Mean Nett l/m³	Coefficient of variance (Net uptake)	Difference significant at 95% level	Index	at 1.0 mm	at 2.5 mm	at 5.0 mm	Cont. env.	Dis. env.	Banded	Broken	Zero L.W.	Preferred L.W.	Zero E.W.	Preferred E.W.	'Sapwood'	'Radial'	2-tone
12	E	99	75	86	NO	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
12	S	98	73	77	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
13	E	87	63	43	YES	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
13	S	106	80	48	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
14	E	120	103	63	NO	71	92	82	71	45	55	37	15	52	23	55	0	98	1	51
14	S	123	101	69	-	74	94	86	74	51	49	41	15	45	41	49	0	100	1	46
15	E	119	96	48	YES	69	95	85	68	47	53	24	9	51	39	53	0	100	0	59
15	S	104	81	48	-	68	94	84	67	42	58	24	9	57	46	57	1	100	1	63
16	E	-	159	67	YES	87	99	96	85	88	13	44	12	7	41	10	0	100	0	44
16	S	-	100	69	-	82	97	91	78	70	30	36	15	22	44	29	0	99	0	74
17	E	-	203	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
17	S	-	148	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
18	E	231	220	-	-	90	100	98	91	99	1	62	21	1	18	3	0	100	0	24
18	S	153	126	-	-	83	99	93	82	90	10	57	7	4	35	10	0	71	0	57
19	E	-	220	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
19	S	-	142	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
20	E	-	251	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
20	S	-	131	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
21	E	282	274	-	-	95	100	180	98	100	0	54	27	0	25	0	0	100	0	71
21	S	171	160	-	-	83	99	95	80	84	16	44	6	16	47	15	0	100	0	57
22	E	-	45	50	YES	60	-	92	86		-	-	-	-	-	-	-	-	-	-
22	S	-	103	43	-	73	-	94	91		-	-	-	-	-	-	-	-	-	-

Appendix 1 Sheet 3 of 3 Summary of results generated from experimentation described in Section 3.2.1

Schedule Comparison Reference Number [1]	Experimental Schedule (E) or Standard Schedule (S)	Uptake				Penetration - Quantitative				Penetration - Qualitative % of samples exhibiting features indicated										
		Gross l/m^3	Mean Nett l/m^3	Coefficient of variance (Net uptake)	Difference significant at 95% level	Index	at 1.0 mm	at 2.5 mm	at 5.0 mm	Cont. env.	Dis. env.	Banded	Broken	Zero L.W.	Preferred L.W.	Zero E.W.	Preferred E.W.	'Sapwood'	'Radial'	2-tone
23	E	80	60	36	YES	52	86	71	46	12	88	7	12	73	33	82	0	98	3	74
23	S	103	84	35	-	67	92	82	63	33	67	17	20	57	40	65	1	100	2	55
24	E	106	79	43	NO	68	93	84	67	45	55	32	12	48	17	53	-	98	1	78
24	S	-	81	45	-	70	95	86	71	51	49	26	19	47	20	48	-	97	2	81
25	E	143	120	72	YES	70	92	85	69	-	-	-	-	-	-	-	-	-	-	-
25	S	122	95	71	-	69	95	85	62	-	-	-	-	-	-	-	-	-	-	-
26	E	-	137	-	YES	80	97	93	84	65	35	89	22	28	25	30	0	0	-	40
26	S	-	56	-	-	50	86	68	46	20	80	5	12	75	18	80	0	0	-	28
27	E	105	98	55	YES	62	93	83	63	36	64	19	9	57	43	64	0	0	-	59
27	S	104	86	53	-	63	94	81	63	42	58	21	12	55	45	57	0	0	-	47
28	E	113	117	91	YES	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
28	S	116	96	80	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
29	E	103	87	65	NO	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
29	S	106	83	56	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
30	E	106	97	53	NO	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
30	S	116	94	50	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
31	E	Assumed equal to comparison 27																		
31	S																			
32	E	119	98	53	NO	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
32	S	126	102	47	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
33	E	52	35	49	YES	30	-	47	-	-	100	18	15	51	49	66	0	100	0	47
33	S	57	41	59	-	30	-	48	-	-	100	19	20	43	35	59	0	100	0	48

[1] For details of schedules see Tables 3.4 and 3.5

Appendix 2 Computer Programme

```
C
C     MATCHED TRIPLET UPTAKE COMPARISON
C     THIS PROGRAMME IS FOR THE ANALYSIS OF GRAVIMETRIC UPTAKE DATA
C    2FROM A MATCHED TRIPLET TEST INVOLVING UP TO 180 SUB-SAMPLES
C
      DIMENSION ISAM(260),TS(260),ITN(260),ICYCL(260),UWT(260),
     2TWT(260),V(85,3,20),SX(85),TX(85),TTX(85),TRX(85),SRX(85),SSX(85),
     3TCYCL(18),SCYCL(18),XI(85),A(85),B(85),IRDIFF(85),SAJ(85),TAJ(85),
     4ITRX(85),ISRX(85),TRESOL(9),SPEC(9)
      DO 3 J = 1,85
      DO 3 K = 1,3
      DO 3 L= 1,20
      V(J,K,L) = 0.0
    3 CONTINUE
      DO 4 N = 1,85
      SSX(N)=SX(N)=TTX(N)=TX(N)=A(N)=B(N)=SAJ(N)=TAJ(N)=XI(N)=0.0
      ISRX(N)=ITRX(N)=IRDIFF(N)=0
    4 CONTINUE
      AMICOM = 0.0
      ANOCOM = 0.0
      BWPA = 0.0
      I = 0
      ISTOT = 0
      ITOT = 0
      ITTOT = 0
      LEND = 0
      LSN = 0
      M=0
      MINCOM = 0
      N=0
      NEG = 999
      NONCOM =0
      POS = 999.9
      SCUMUT = 0.0
      SSQ = 0.0
      TCUMUT = 0.0
      TRIO = 0.0
      TSQ = 0.0
      UPTK = 0.0
      SSQ=0.0
      SCUMUT=0.0
      TSQ=0.0
      TCUMUT = 0.0
      ITTOT = 0
      ISTOT = 0
      ITOT = 0
      READ(5,2)LSN,MSN,(TCYCL(I),I=1,18),(SCYCL(I),I=1,18)
    2 FORMAT(6X,I4,5X,I4,5X,18A1,5X,18A1)
      READ(5,6)MMLONG,MMWIDE,MMTHIK,(TRESOL(I),I=1,9),(SPEC(I),I=1,9),
     2BWPA
```

```
 6 FORMAT(6X,I3,5X,I3,5X,I3,5X,9A1,5X,9A1,5X,F3.0)
   NBEAM = MSN-(LSN-1)
   IF(NBEAM.LT.1) GO TO 1750
   IF(NBEAM.LE.12) NTRIP = NBEAM * 7
   IF(NBEAM.LE.12) NSAM = NBEAM * 21
   IF (NBEAM.GT.12.AND.NBEAM.LE.85) NTRIP = NBEAM
   IF(NBEAM.GT.12.AND.NBEAM.LE.85) NSAM = NBEAM * 3
   IF(NBEAM.GT.85) GO TO 1750
   DO 20 N=1,NSAM
   READ(5,01) ISAM(N),TS(N),ITN(N),ICYCL(N),UWT(N),TWT(N),LEND
01 FORMAT(5X,I4,1X,A1,1X,I1,5X,I1,10X,F5.1,11X,F5.1,21X,I1)
   IF(LEND.EQ.9)GO TO 114
   ITOT = ITOT + 1
   IF(NBEAM.LE.12)J = (ISAM(N)-LSN)*7 +ITN(N)
   IF(NBEAM.GT.12) J=ISAM(N) -(LSN-1)
   IF (J.LT.1) GO TO 20
   IF (J.GT.NTRIP) GO TO 20
   IF (TS(N).EQ.1HA) K=1
   IF(TS(N).EQ.1HB) K=2
   IF(TS(N).EQ.1HC) K=3
   IF(TS(N).NE.1HA.AND.TS(N).NE.1HB.AND.TS(N).NE.1HC) GO TO 20
   V(J,K,13) = FLOAT(ISAM(N))
   V(J,K,14) = TS(N)
   V(J,K,15) = FLOAT(ITN(N))
   V(J,K,1)=ICYCL(N)
   V(J,K,2)=UWT(N)
   V(J,K,3)=TWT(N)
   UPTK = TWT(N) - UWT(N)
   IF(UPTK.LT.0.0) GO TO 20
   IF(V(J,K,4).NE.0.0) GO TO 8
   V(J,K,4)=UPTK                                                    14
   GO TO 7
 8 WRITE(6,09)(V(J,K,L),L=13,15)
09 FORMAT(1X,"SAMPLE NO ",F5.0,1X,1A1,1X,F2.0," OCCURS TWICE OR NO OR
  2BEAMS GREATER THEN 12 BUT TWO TRIPLETS COME FROM ONE BEAM")
   GO TO 1902
 7 CONTINUE
   IF(ICYCL(N).NE.6.AND.ICYCL(N).NE.2.AND.ICYCL(N).NE.0) GO TO 1902
   IF(ICYCL(N).EQ.2) GO TO 10
   ISTOT = ISTOT + 1
   SCUMUT=SCUMUT+UPTK                                               17
   SSQ=SSQ+UPTK*UPTK                                                18
   GO TO 20
10 CONTINUE
   ITTOT = ITTOT + 1
   TCUMUT=TCUMUT+UPTK
   TSQ=TSQ+UPTK*UPTK
20 CONTINUE
114 CONTINUE
   TRIO = 0.0
```

```
      DO 120 J = 1,NTRIP
      IF(V(J,1,4).EQ.0 0.AND.V(J,3,4).EQ.0.0) GO TO 119
      IF(V(J,2,4).EQ.0.0) GO TO 119
      IF(V(J,1,1).EQ.V(J,2,1).OR.V(J,2,1).EQ.V(J,3,1)) GO TO 119
      TRIO = TRIO + 1.0
      IF(V(J,1,1).EQ.6.0.OR.V(J,3,1).EQ.6.0) GO TO 116
      IF(V(J,1,4).EQ.0.0.OR.V(J,3,4).EQ.0.0)V(J,1,11)=AMAX1(V(J,1,4),
     2V(J,3,4))
      IF(V(J,1,4).NE.0.0.AND.V(J,3,4).NE.0.0)V(J,1,11)=(V(J,1,4)+V(J,3,4
     2))/2
      V(J,2,11) = V(J,2,4)
      GO TO 119
  116 IF(V(J,1,4).EQ.0.0.OR.V(J,3,4).EQ.0.0)V(J,2,11)=AMAX1(V(J,1,4),
     2V(J,3,4))
      IF(V(J,1,4).NE.0.0.AND.V(J,3,4).NE.0.0)V(J,2,11)=(V(J,1,4)+V(J,3,4
     2))/2
      V(J,1,11) = V(J,2,4)
  117 CONTINUE
      V(J,3,11) = V(J,1,11)-V(J,2,11)
      WRITE(6,118)V(J,2,13),V(J,2,15),V(J,1,11),V(J,3,11)
  118 FORMAT(10X,F4.0,1X,F2.0,5X,F5.1,5X,F5.1)
  119 CONTINUE
  120 CONTINUE
      WRITE(6,171)(TCYCL(J),J=1,18),(SCYCL(J),J=1,18)
  171 FORMAT(1H1,10X,"TEST CYCLE (CODE 2)    ",18A1,15X,"STANDARD CYCLE
     2(CODE 6)    ",18A1)
      WRITE(6,21)
   21 FORMAT(////5X,"SAMPLE NUMBER",5X,"CYCLE CODE",5X,"INITIAL WEIGHT(G
     2M)"10X,"FINAL WEIGHT(GM)",10X,"UPTAKE(GM)",10X,"(TRIPLET AJUSTED)"
     3)
      WRITE(6,22)
   22 FORMAT(/67X,"TEST",6X,"STND",11X,"TEST",4X,"STND",10X,"TEST",7X,"S
     2TND"//)
      DO 29 J = 1,NTRIP
      DO 29 K = 1,3
      IF(K.EQ.1.AND.NBEAM.LE.12) WRITE(6,221)
  221 FORMAT(/)
      IF(V(J,K,13).EQ.0.0) GO TO  271
      IF(V(J,K,1).EQ.2.0) GO TO 26
      IF(K.EQ.2.0) GO TO 25
      WRITE(6,23)(V(J,K,L),L=13,15),(V(J,K,L),L=1,4)
   23 FORMAT(8X,F5.0,1X,1A1,1X,F2.0,11X,F3.1,13X,F5.1,27X,F5.1,18X,F5.1)
      GO TO 29
   25 CONTINUE
      WRITE(6,24)(V(J,K,L),L=13,15),(V(J,K,L),L=1,4),V(J,1,11),V(J,2,11)
   24 FORMAT(8X,F5.0,1X,1A1,1X,F2.0,11X,F3.1,13X,F5.1,27X,F5.1,18X,F5.1,
     28X,F5.1,6X,F5.1)
      GO TO 29
   26 IF(K.EQ.2.0) GO TO 281
  271 WRITE(6,27)(V(J,K,L),L=13,15),(V(J,K,L),L=1,4)
```

```
 27 FORMAT(8X,F5.0,1X,1A1,1X,F2.0,11X,F3.1,13X,F5.1,16X,F5.1,20X,F5.1)
    GO TO 29
281 WRITE(6,28)(V(J,K,L),L=13,15),(V(J,K,L),L=1,4),V(J,1,11),V(J,2,11)
 28 FORMAT(8X,F5.0,1X,1A1,1X,F2.0,11X,F3.1,13X,F5.1,16X,F5.1,20X,F5.1,
   217X,F5.1,6X,F5.1)
 29 CONTINUE
    I=M=0
    DO 30 J = 1,NTRIP
    DO 30 K=1,3
    IF(V(J,K,1).EQ.0.0) GO TO 30
    IF(V(J,K,1).EQ.6.0) GO TO 291
    I = I + 1
    TX(I) = V(J,K,4)
    GO TO 30
291 M = M+1
    SX(M) = V(J,K,4)
 30 CONTINUE
    WRITE(6,39)
 39 FORMAT(1H1)
    WRITE (6,601)(SPEC(J),J=1,9),(TCYCL(J),J=1,18),(SCYCL(J),J=1,18),
   2LSN,MSN,(TRESOL(I),I=1,9)
601 FORMAT(3X,"COMPARISON OF UPTAKES BY ",9A1," RESULTING FROM TREATME
   2NT",//7X,"BY CYCLES ",18A1," AND ",18A1,//15X,"(TEST SAMPLES NUMBE
   3RS ",I4," TO ",I4," )",//18X,"TREATING SOLUTION ".9A1)
    WRITE(6,605) (SCYCL(I),I=1,18)
605 FORMAT(/1X,"STANDARD CYCLE",2X,18A1,//)
    CALL STAT(SCUMUT,ISTOT,SSQ,MMLONG,MMWIDE,MMTHIK,SAV,SVAR)
    CALL RANK (SX,ISRX,M)
    CALL TIE (SSX,ISRX,M)
    CALL MEDIAN(SSX,ISTOT,SX)
    WRITE (6,610)(TCYCL(I),I=1,18)
610 FORMAT(/1X,"TEST CYCLE",2X,18A1,//)
    CALL STAT(TCUMUT,ITTOT,TSQ,MMLONG,MMWIDE,MMTHIK,TAV,TVAR)
    CALL RANK (TX,ITRX,I)
    CALL TIE (TTX,ITRX,I)
    CALL MEDIAN(TTX,ITTOT,TX)
    PERCEN = (TAV/SAV)*100
    WRITE(6,620)PERCEN
620 FORMAT(//1X,"MEAN UPTAKE OF TEST CYCLE SAMPLES AS A PERCENTAGE OF
   2THE MEAN",//1X,"UPTAKE OF STANDARD CYCLE SAMPLES",//30X,F6.2," PER
   3CENT")
    IF(TVAR.GT.SVAR) F= TVAR/SVAR
    IF(TVAR.LT.SVAR) F=SVAR/TVAR
    IF (TVAR.EQ.SVAR) F= 1.0
    WRITE(6,615) F
615 FORMAT(//20X,"F (VARIANCE RATIO) = ", F5.2)
    WRITE (6,616)I,M
616 FORMAT(/6X,I3," TEST SAMPLES TREATED",2X,I3," STANDARD SAMPLES TRE
   2ATED",/)
    IF(M.GE.63.AND.I.GE.60) GO TO 621
```

```
      WRITE(6,617)
  617 FORMAT("USE PUBLISHED TABLES TO DECIDE WHETHER PARAMETRIC TESTING
     2JUSTIFIED")
      GO TO 625
  621 CONTINUE
      IF(F.GT.1.5) GO TO 300
  625 CONTINUE
C     TTEST
      XICUM = 0.0
      XITOT = 0.0
      XISQ = 0.0
      POS = 0.0
      NEG = 0
      JOT = IFIX(TRIO)
      DO 210 N = 1,NTRIP
      SAJ(N) = V(N,2,11)
      TAJ(N) = V(N,1,11)
      XI(N) = SAJ(N) - TAJ(N)
      IF (XI(N) .EQ. 0.0) GO TO 205
      IF (XI(N) .GT. 0.0) POS = POS + 1.0
      IF(XI(N) .LT.0.0) NEG = NEG + 1
      XITOT = XITOT + 1.0
      XICUM = XICUM + XI(N)
      XISQ = XISQ + (XI(N)*XI(N))
  205 CONTINUE
  210 CONTINUE
      XIMEAN = XICUM/XITOT
      XICMSQ = XICUM*XICUM
      XCMDVN = XICMSQ/XITOT
      ANUMER = XISQ - XCMDVN
      DENOM = XITOT*(XITOT-1.0)
      RES = ANUMER/DENOM
      SQTRES = SQRT(RES)
      T=XIMEAN/SQTRES
      WRITE(6,213)T,JOT
  213 FORMAT(//1X,"RESULT OF PARAMETRIC STUDENTS T TEST",3X,"T =",F6.2,
     25X,"N = ",I3)
      WRITE(6,240)
  240 FORMAT(//10X, "USE PUBLISHED TABLES TO ASSESS PROBABILITY")
  280 IF(M.LT.60.AND.I.LT.60) GO TO 300
      IF (F.LT.1.5) GO TO 1850
  300 CONTINUE
C     WILCOX
      POS = 0.0
      NEG = 0
      DO 302 I = 1,NTRIP
      TAJ(I) = V(I,1,11)
      SAJ(I) = V(I,2,11)
      XI(I) = SAJ(I) - TAJ(I)
      IF (XI(I).GT.0.0) POS = POS + 1.0
```

```
      IF (XI(I) .LT.0.0) NEG = NEG + 1
      A(I) = SAJ(I) - TAJ(I)
  302 B(I) = ABS(A(I))
      CALL RANK(B,IRDIFF,NTRIP)
      CALL TIE(B,IRDIFF,NTRIP)
      DO 303 I = 1,NTRIP
      B(I) = SIGN(B(I),A(I))
  303 CONTINUE
C
      SPOS = 0.0
      SNEG = 0.0
      NN = TRIO
      DO 305 I = 1,NTRIP
      IF (A(I).EQ.0.0) NN = NN-1
      IF (B(I).LT.0.0) GO TO 304
      SPOS = SPOS + B(I)
      GO TO 305
  304 SNEG = SNEG + B(I)
  305 CONTINUE
      T=AMIN1(SPOS,ABS(SNEG))
C
C     COMPUTE NORMAL DEVIATE FOR T
      AN = NN
      Z = ABS((T-(AN*(AN+1.0))/4.0)/SQRT((AN*(AN+0.5)*(AN+1.0))/12.0))
C
C     OUTPUT RESULTS
      WRITE (6,310) NN,T,Z
  310 FORMAT(//"NUMBER OF COMPARISONS =",I3," TEST STATISTIC =",F6.2," N
     20RM. DEVIATE =",F5.2)
      WRITE(6,315)
  315 FORMAT(/"IF THE NUMBER OF COMPARISONS IS LESS THAN 25 USE",/1X,"TE
     2ST STATISTIC T AND WILCOXON TABLE TO DETERMINE PROBABILITY",/1X,"I
     3F N GREATER THAN 25, USE NORMAL DEVIATE AND PUBLISHED",/1X,"TABLES
     4OF THE Z DISTRIBUTION")
 1850 CONTINUE
      WRITE(6,214)NEG
  214 FORMAT(//1X,I3,  " TRIPLETS  HAD A HIGHER UPTAKE AS A RESULT OF ",
     2/1X,"TREATMENT BY THE TEST CYCLE")
      WRITE(6,212)POS
  212 FORMAT(//1X,F5.1," TRIPLETS HAD AN UPTAKE LOWER AS A RESULT OF ",/
     21X,"TREATMENT BY THE  TEST CYCLE")
  230 CONTINUE
      IF(BWPA.NE.99.0.OR.ITOT.LT.70) GO TO 1902
      DO 40 N = 1,ISTOT
  309 FORMAT(10X,F6.2)
      IF(SSX(N).GT.6.4.AND.SSX(N).LE.7.5) ANOCOM = SX(N)
      IF(SSX(N).GT.7.4.AND.SSX(N).LE.8.5) AMICOM = SX(N)
   40 CONTINUE
      DO 110 J = 1,ITTOT
      IF(TX(J).LT.ANOCOM.AND.TX(J).GT.0.0)NONCOM = NONCOM + 1
```

```
      IF(TX(J).LT.AMICOM.AND.TX(J).GT.0.0)MINCOM = MINCOM + 1
  110 CONTINUE
      WRITE(6,71)ANOCOM,AMICOM,NONCOM,MINCOM
   71 FORMAT(//" 7 AND 8 SAMPLES RESPECTIVELY (CA. 10 PERCENT  OF THE ST
     2ANDARD CYCLE",/1X,"SAMPLES) HAD UPTAKES LESS THAN OF EQUAL TO ",
     3F4.1," AND ",F4.1," GM.",/1X,I2" AND "I2," TEST CYCLE SAMPLES RESP
     4ECTIVELY HAD UPTAKES BELOW THESE LEVELS")
      GO TO 1902
 1750 CONTINUE
 1902 CONTINUE
      WRITE(6,1950)
 1950 FORMAT(////////)
      STOP
      END
      SUBROUTINE STAT(SUM,NUM,SSQ,MMLONG,MMWIDE,MMTHIK,CMEAN,VAR)
      DIMENSION CYCLE(18)
C
C     THIS SUBROUTINE COMPUTES MEAN STANDARD DEVIATION VARIANCE
C    2COEFFICIENT OF VARIATION AND STANDARD ERROR FOR A SET OF DATA
C    3WHERE THE FOLLOWING HAVE ALREADY BEEN DETERMINED..NUMBER OF
C    4OBSERVATIONS(NUM) SUM OF OBSERVATIONS(SUM) AND SUM OF SQUARES (SSQ)
C
      CMEAN = SUM/NUM
      VAR = (SSQ - SUM*CMEAN)/(NUM - 1)
      SDEV = SQRT(VAR)
      SERR=SDEV/SQRT(FLOAT(NUM))
      COEFF = SDEV/CMEAN*100
      RETN = (CMEAN * 1000000)/(MMLONG*MMWIDE*MMTHIK)
      WRITE(6,01)CMEAN,RETN,VAR,SDEV,COEFF,SERR
   01 FORMAT(34X,"MEAN",11X,F6.2,"GM",/48X,F7.2,"KG/M",/34X,"VARIANCE",7
     2X,F6.2,/34X,"ST.DEV."8X,F6.2,/34X,"COEF OF VAR",4X,F6.2,/34X,"ST.E
     3RROR",7X,F6.2)
      RETURN
      END
      SUBROUTINE RANK (X,IRX,N)
C
C     THIS SUBROUTINE RANKS THE N ELEMENTS OF A VECTOR X AND PLACES THE
C    2RANKS IN A VECTOR IRX
C
      DIMENSION X(N),IRX(N)
      DO 2 J = 1,N
      NX=1
      DO 1 I=1,N
      IF(X(I).LT.X(J).AND.X(I).GT.0.0001) NX = NX + 1
    1 CONTINUE
    2 IF(X(J).GT.0.0001) IRX(J) = NX
      RETURN
      END
      SUBROUTINE TIE(X,IRX,N)
C
```

```
C     THIS SUBROUTINE AJUSTS THE RANKINGS IN VECTOR IRX FOR TIES AND
C    2PLACES THE AJUSTED RANKS IN VECTOR X
C
C
      DIMENSION X(N), IRX(N)
      NP1 = N+1
      DO 4 J=1,N
      IF(IRX(J).EQ.NP1) GO TO 4
      NX = IRX(J)
      NTIE=-1
      DO 2 I= 1,N
      IF (IRX(I).NE.NX) GO TO 2
      NTIE=NTIE + 1
      X(I) =IRX(I)
      IRX(I) = 0
    2 CONTINUE
      DO 3 I =1,N
      IF (IRX(I).NE.0) GO TO 3
      X(I) = X(I) + FLOAT(NTIE)*0.5
      IRX(I) = NP1
    3 CONTINUE
    4 CONTINUE
      RETURN
      END
      SUBROUTINE MEDIAN(X,ISUM,XY)
      DIMENSION X(ISUM),XY(ISUM)
      AMED=ISUM/2.0
      DO 3 I = 1,ISUM
      IF(X(I).LT.AMED-0.55.OR.X(I).GT.AMED + 1.0) GO TO 3
      WRITE(6,01)XY(I)
   01 FORMAT(34X,"MEDIAN VALUE(S)", F5.1)
    3 CONTINUE
      RETURN
      END
/
```

Figure 2.46 Pressure changes recorded within spruce Sample II : Section 2.2.3.4.5

