RECOVERY OF STEAM-DISTILLED EXTRACTIVES FROM DOUGLAS FIR VENEER DRYING

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Abstract

A pilot study was conducted on the exhaust vapour which is expelled through the roof-ventilator stacks of a Moore cross circulation veneer dryer, in order to determine whether or not any by-products of commercial importance could be recovered from Douglas fir veneer while it is undergoing normal drying operations prior to the manufacture of plywood, Separate studies were made on the heartwood and sapwood veneer of this species.

Some steam-distilled volatile oil was recovered from the stack gases, but because of the small amount which was collected, this material was considered to be of no commercial value. Recommendations have been made for future work in this field.

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Acknowledgement

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Introduction

Research is the key to industrial progress. The main objective of industrial research is to apply scientific knowledge in order to create new products, enhance the value of old products, and increase the efficiency of production, by designing new machines, reducing and utilizing waste, and upgrading the average quality of the product.

In 1958 the Vancouver Plywood Division of MacMillan, Bloedel and Powell River Ltd., located in Vancouver, British Columbia, surveyed the possibility of obtaining commercial by-products from vapour driven off in veneer drying. This drying was done by means of mechanical veneer dryers into which the veneer was fed on rolls. As it moved through these drying ovens, the veneer was subjected to temperatures ranging from 174 deg. C to 204 deg. C which resulted in the moisture content of the wood being lowered to a level of between 2 per cent and 5 per cent. Moisture from the wood was driven off in gaseous

form and this vapour was either exhausted through roof ventilator stacks or was condensed on the inside of the dryer walls. Because of the high temperatures involved, it was felt that these exhaust gases might contain some products which would be of commercial importance and which would be capable of being recovered by a simple industrial distillation procedure. The large volume of vapour which was driven off each day from a veneer dryer added optimism to this belief and led to a recommendation, at that time, that this question merited further and more careful investigation to determine whether or not such a development would be commercially profitable.

An additional investigation was carried out by the author. The objectives were as follows:

(1) To determine the quantity of vapour which is exhausted through the roof ventilator stacks.

(2) To determine the proportion of chemical extractives in this vapour.

(3) To investigate the composition of this extractive material in an effort to determine the major components present and the relative quantities of these components.

(4) To make a commercial evaluation of the recovery process on the basis of these results.

Douglas fir (Pseudotsuga menziesii (Mirb.) Franco) has for many years been the leading species used by the

softwood plywood industry in the Pacific northwest. In order to relate this work as much as possible to the normal manufacturing conditions of the softwood plywood industry in British Columbia, it was decided that only Douglas fir veneer would be investigated.

No sharp line of demarcation exists between fundamental and applied research in forest products, or between applied research and wood products development. This project was not intended to be a fundamental research study into the elementary chemical composition of exhaust vapours. It was a utilization problem which sought a method of reducing physical wastage by retrieving material which is at present rejected and using this material for the manufacture of a variety of commercial products. In other words, it was an attempt towards a higher and more effective utilization of the wood material which is presently used for the manufacture of plywood, by determining whether or not chemical extractives, derivatives or products of commercial value could be profitably recovered from this material without adversely effecting normal veneer drying operations.

Some of the many benefits to be gained from the recovery of chemicals by this means are:

(a) There would be no competition for wood raw material, or for markets, between plywood and the chemical by-products.

(b) A greater economic return would be realised for the same wood resources. This higher conversion rate for available wood raw material, along with additional revenue from the sale of the by-products, would help to reduce the expense of veneer drying for the production of plywood.

(c) The recovery process would not require a large investment in additional equipment, nor would additional supplies of fuel for heating the veneer dryers be needed.

(d) Greater product diversification would lend stability to the plywood industry and make it less vulnerable to market fluctuation in price or demand.

(e) The future potential of chemical products derived from wood is very great and these products may eventually become as important and valuable as the primary wood products of today.

Review of Literature

Wood composition

Wood as it occurs in nature is made up of cellulose, hemicellulose, lignin and extraneous components. The extraneous components are not an integral part of the cell wall and can

usually be removed by neutral solvents such as ether, alcohol, water, etc., without affecting either the chemical composition of the wood substance, or the physical structure of the cell wall. They also have a non-uniform distribution throughout the bole of the tree.

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

Extractives are generally found in the cell cavities, but they may also be present in very fine capillaries of the cell wall thereby making their complete removal quite impossible. The heartwood of softwoods has a high content of water, ether, and alkali-soluble extractives (27).

Naval stores industry

General

Naval stores can be defined as "Chemically reactive oils, resins, tars and pitches derived from the oleoresin contained in, exuded by, or extracted from trees, chiefly of the pine species (Genus Pinus), or from the wood of such trees"(2).

Oleoresin is the non-aqueous secretion of resin acids dissolved in a terpene hydrocarbon oil which is;

(1) Produced or exuded from the intercellular resin ducts of a living tree.

(2) Accumulated, together with oxidation products, in the dead wood of weathered limbs and stumps (2).

The outmoded but unshed name of the naval stores industry was derived from the former uses of its products, namely, wood tar and pitch, for caulking wooden ships and for preservative treatment of their rigging. The chief products of the naval stores industry are now turpentine and rosin. Turpentine is a volatile oil which consists of a number of terpene hydrocarbons and is obtained by distilling the oleoresin exuded by, or contained in, the wood of certain species of pine trees (2). Rosin is a specific kind of natural resin obtained as a vitreous, water insoluble material from pine oleoresin by the removal of the volatile oils (2).

Naval stores are classified according to the method of obtaining the resinous raw material. "Gum" naval stores consist of products which are derived from the secretion or exudate of the living tree, this exudate having been collected by the process known as turpentining. "Wood" naval stores on the other hand, are products which have been manufactured from the wood substance after the tree has been cut. This wood usually consists of forest and industrial residues which have been specially selected because of their high resin content.

Processes

Wood naval stores can be produced in two ways:

(1) Destructive distillation. This consists of heating the wood in a closed retort equipped with an opening leading to a condenser where the vapours escape, condense and are collected. The wood is disintegrated as a result of this heat treatment.

A more recent development in this field has been the alkali solvent process which is the recovery of P.M.L. (pulp mill liquor) grade turpentine as a by-product from the spent cooking liquors of sulphate pulp mills. This is used for terpene-based chemicals (for example, synthetic pine oil) and its production has become a profitable venture in some areas of the U.S.A. where some companies have reported a net return of 29.6 per cent on P.M.L. grade turpentine recovery (4). Actual areas in which this profitable operation is being conducted were not reported, but it most probably refers to the southern pine region of the U.S.A.

Newmarch[1] reported that the recovery of turpentine from kraft pulp, consisting of 50 per cent Douglas fir mixed with hemlock and cedar, would only be profitable if allowance were made for heat recovery from the hot water coming from the turpentine condensers. Yields were recorded of 0.9 U.S. gallons of turpentine and 2.1 U.S. gallons of water at 180 deg.C,

1 Newmarch, O.H., Recovery of Turpentine from Douglas Fir Kraft Pulp. Unpublished report, MacMillan, Bloedel and Powell River Limited, Harmac Division, November 1959.

per ton of fir pulp. It was observed that direct steaming greatly increased the yield of turpentine, but that a heavy steam flow of up to 20,000 pounds per hour was required for a significant effect. A higher than normal rate of digester relief was also necessary in order to get a yield approaching that reported by others. Newmarch stated that as more volatiles were released early in the steaming, an improved product, with a lower boiling point range, would probably be obtained if later relief gases were neglected.

(2) Non-destructive distillation. In this process, the wood is not destroyed but is treated with a medium which extracts some of the resinous products from the wood, producing a chemical composition change, but not a marked physical change. In normal commercial practice the wood is ground or cut into particles prior to distillation.

A common medium used for this work is steam, which removes various products from wood, as illustrated in Figure 1.

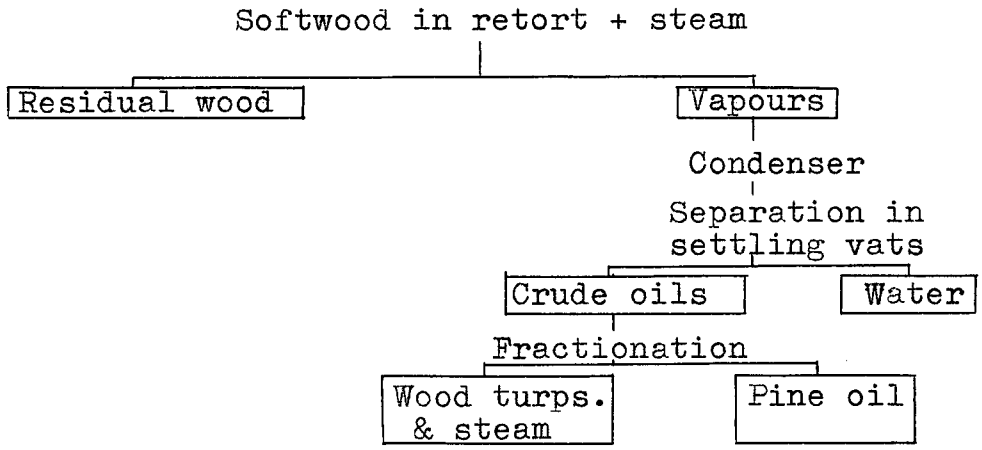

Fig. 1. Steam distillation of wood (7)

Pine oil is a colourless to amber coloured volatile oil with characteristic pinaceous odour, consisting principally of tertiary and secondary terpene alcohols, with variable quantities of terpene hydrocarbons, ethers, phenols, etc., the amount and character of which depend on the source and method of manufacture (2).

Some theoretical aspects of steam decomposition of wood

When two non-miscible (mutually insoluble) liquids, such as oil and water, are heated while kept in equilibrium (by stirring), the mixture will begin to boil when the sum of the vapour pressures of the constituents equals atmospheric pressure. The mixture will therefore boil at a temperature which will be lower than the boiling point of either oil or water. When considering the oil and water contained in wood, however, this concept becomes complicated because the wood substance itself prevents free contact between steam and the oils to be distilled, so that there is not a true equilibrium existing within the wood.

Steam distillation

Hawley (17) reported that in the presence of steam, some volatile oils were distilled below 100 deg. C, and if enough water was in the wood to furnish steam, these volatiles might distill completely at temperatures below 100 deg.C. It was noted further that most of the volatile oils would distill below the temperature (approximately 250 deg.C) at which wood

begins to decompose strongly, but that rosin also decomposed below this temperature, and some contamination of the turpentine by this rosin might occur. Such contamination, however, was felt to be very slight below 200 deg.C. Hawley observed further that not much water and turpentine distillate was received below 160 deg.C because a true equilibrium did not exist within the wood, and not until the resin became a thin fluid with the heat, and began to flow in the wood, did much distilling take place. It was stated that instead of getting an initial distillation of turpentine and some pine oil, followed by rosin, often all three would be distilled at the same time.

In steam distillation, the condensate, if allowed to settle, will separate into an upper oil layer and a lower aqueous layer. The oil layer is drawn off and fractionally distilled into a lower boiling fraction (temperature not exceeding 180 deg.C) consisting of steam and turpentine, and a higher boiling fraction of pine oil.

Steam and solvent extraction

The steam and solvent process consists of normal steam distillation, followed by treatment of the steam-extracted wood with a solvent, such as ammonium hydroxide, to remove the remainder of the rosin in the wood.

In describing this process, Hawley and Palmer (18) reported that the steaming produced a very good quality of

"crude turpentine" consisting essentially of turpentine and pine oil, but because this was the only product obtained, and because the yield of this product was generally lower than that of "crude turpentine" from other processes, steam and solvent process plants were only successful when especially favourable conditions existed.

Palmer's (24) account of this process stated that the bulk of the volatile oil present in wood was driven off by the steam. This primary distillate consisted essentially of turpentine and pine oil in varying proportions depending upon the conditions of steaming. Some terpene hydrocarbons (for example, limonene, dipentene, etc.), with a boiling range between that of pinene (the principal constituent of turpentine) and pine oil, might also be present but this fraction could be removed in fractional distillation.

In modern practice for the production of wood naval stores, steam distillation is eliminated, and all extractions of the wood raw material is done by solvents, such as benzene and naphtha.

Steam distillation variables and their effect (upon the products obtained)

Palmer (24) stated that the total yield of crude oil, the composition of this oil and the amount of steam required to remove the oil, would be affected by variables such as:

(1) Wood characteristics (for example, species, resin content, dimension of the wood material)

(2) Steam pressure

(3) Speed of distillation

(4) The end point at which distillation is stopped.

Hawley and Palmer (18) established that the yield of crude oil was improved by increasing steam pressure and decreasing the speed of distillation as well as by using highly resinous, small sized wood raw material. This point was reaffirmed by Newmarch[2] in his recent investigation into the recovery of turpentine from kraft pulp liquor.

Hawley (16) found that the proportion of dipentene in refined turpentine increased in direct relation to the maximum temperature of production. Hawley and Palmer (18) observed that increase in steam pressure had no effect on the proportion of pine oil contained in the distillate but that it did increase the amount of dipentene present. The light oil (boiling point below 175 deg. C) fraction had a higher specific gravity which indicated that substances of low boiling point and high specific gravity were produced at higher steam pressures.

Chemical treatment prior to fractional distillation

Hawley (16) declared that a chemical treatment was

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2 Op. cit.

required before fractional distillation of the crude wood oil would produce a good grade of refined turpentine because it would remove the light oils which had boiling points lower than that of turpentine, thereby improving the colour and odour of the refined turpentine. Palmer (24) referred to a chemical distillation with caustic soda prior to fractional distillation by which any wood acids which might be present would be neutralised, and contaminous material, such as light oils, which adversely effect grade, colour, and odour would be polymerised. The American Society for Testing Materials (3), however, suggested that chemical treatment prior to fractional distillation was not necessary in cases where distillation temperatures would be kept below 200 deg.C.

Hot air process

It is of interest to note that a non-destructive distillation process using hot air as a medium for the removal of volatile oils, along with a small amount of low grade rosin, has been attempted, but with no apparent commercial success (13).

Species characteristics

Although hardwoods, such as beech (Genus Fagus) and oak (Genus Quercus), are used to produce wood naval stores, this industry mainly relies upon softwoods for the manufacture of its products. Each species of wood yields a crude oil which is more or less different from those of the others. Only a few of the many species of conifers yield satisfactory

quantities of turpentine and wood oil. The commercial softwood species consist mainly of the pines although other species, among which is Douglas fir, have been used with a certain amount of success.

In the Unites States, which produces about 53 per cent of the world's naval stores requirements (4), the principal species used are the longleaf pine (Pinus palustris Mill.) and the slash pine (P. elliottii Engelm.). France, on the other hand, mainly uses the maritime pine (P. pinaster Aiton.) and the aleppo pine (P. halepensis Miller.) to produce 20 per cent of the world's supply (4).

The amount of resinous extractives in common Canadian woods is relatively low. Harkom and Colleary (13) pointed out that apart from the southern pines (P. palustris Mill., P. rigida Mill., P. elliottii Engelm., etc.), only three other species, Norway pine (P. resinosa Aiton.), western yellow pine (P. ponderosa Dougl.), and Douglas fir had been suggested as offering possibilities as a source of raw material. These were also the only Canadian species regarding which production yields from commercial practice or industrial research were available.

One of the measures of the resinous content of a wood is its ether solubility. This is the weight of dried ether-extracted residue calculated as a percentage of the weight of the oven-dry (105 deg. C) initial wood sample. While ether

solubility does not include all extractives in wood, it provides some indication of the amount of extractives present, exclusive of most water-soluble materials (12). Hawley (17) determined the ether solubility of several softwood species as shown in the following table:

Ether solubility of some softwood species

Species	Per cent O.D. Weight
Western yellow pine (Pinus ponderosa)	8.52
Longleaf pine (Pinus palustris)	6.32
Western white pine (Pinus monticola)	4.26
White spruce (Picea glauca)	1.36
Douglas fir (Pseudotsuga menziesii)	1.02

It is not known what type of wood (sapwood, heartwood, stumpwood, etc.) was used for these determinations, or the moisture content of the wood. Greaves and Schwartz (12) found the ether solubility of Douglas fir heartwood to be 1.14 per cent. It can be seen from these figures that the species of greatest commercial use in the United States naval stores industry, namely longleaf pine, has a considerably higher ether solubility factor than Douglas fir.

Resin is not evenly distributed in the tree. Sapwood contains very little of it, while heartwood of the stump and butt log may be very resinous. Heartwood of the main bole of the tree is intermediate in its resin content. In longleaf pine, sapwood may contain about 2 per cent resin, the average heartwood 7 to 10 per cent, heartwood of the butt log 15 per cent, and the heartwood of the stump 25 per cent or more (13).

It is important to note that the wood which is actually used as a raw material for the commercial production of naval stores is specially selected for its extra high resin content. The wood raw material which is most preferred is called "lightwood", in which the gum in the wood cell has hardened and become sealed into the cell and wood structure. Longleaf pine "lightwood" is available in commercial quantities in certain parts of the southern states region of the United States, where it is reported to have an average resin content of 20 to 25 per cent (13).

Schorger (29) examined the composition of oleoresin which was extracted from the base of living Douglas fir trees and his results were:

Composition of oleoresin of Douglas fir

Source of oleoresin	Volatile oils		Colophony	Water and losses
	proportion %	Specific gravity	%	%
Heartwood	30.40	0.862	68.82	0.78
Heartwood	28.64	0.863	69.77	1.59
Sapwood	22.70	0.863	Not given	Not given

The initial moisture contents of neither the heartwood nor the sapwood of these standing trees were determined by Schorger.

Past history using Douglas fir

There appears to be no doubt that certain trees contain a higher resin content than others, but it is generally conceded that the average yield from western conifers is low compared to that of trees in the southern pine region of the United States. Frequent reference has been made to Douglas fir as a probable source of both wood and gum turpentine, and rosin. Between 1910 and 1914, a few companies attempted to extract turpentine and rosin from Douglas fir by both steam distillation and destructive distillation, but these plants were small and apparently did not meet with sufficiently pronounced success to induce others to invest in similar enterprises. One such company was formed in Vancouver, British Columbia, around 1910. This plant employed a method of destructive distillation known as the Snyder electrical process for the removal of turpentine from Douglas fir mill residue (9, 13).

The effect of the 1914 to 1918 war in curtailing production of naval stores in the United States, and bringing an increase in prices for these products, revived an interest in Douglas fir. Attention became centred more on gum products rather than wood products, and a few companies were established in the Pacific northwest to manufacture the former.

The Douglas Fir Turpentine Co. of Vancouver, British Columbia, pioneered the production on a commercial scale of

gum turpentine and rosin from Douglas fir in British Columbia. This company secured sap rights to large blocks of privately-owned forest on Cortez Island, which is situated due east of Campbell River on Vancouver Island, and began turpentining operations in a small way (9). This enterprise, however, eventually failed.

No work was reported to have been done using either Douglas fir heartwood or sapwood in the bole of the tree as a wood raw material. The University of Washington Engineering Experimental Station (13), however, reported a certain amount of success from steam and solvent extraction of ordinary stumpwood of this species. The results of this distillation work were:

Total oil fraction	0.9 U.S. gallons from 3800 lb. wood
Turpentine	0.6 " " " " " "
Pine oil	0.3 " " " " " "
Rosin	75 pounds from 3800 lb. wood

Marketing

No wood or gum naval stores have been produced in Canada successfully on a commercial scale. The main reasons for this are firstly, because the average resin content of the trees grown in this country is considerably lower than that of the southern pines; secondly, the wood raw material would have to be specially selected to give commercially profitable yields, and finally, because the cost of obtaining the raw material

would be excessive (30). Harris (15) stated that any attempt to produce naval stores from the British Columbia forests would be doomed to failure because of severe competition from the southern pine region, but that the possibility of obtaining these naval stores as by-products of another wood products industry would be a very different story. It was pointed out that a company producing naval stores as a by-product would not be dependent upon the sale of these secondary products as a source of revenue. In addition to this, these chemical by-products would have no detrimental effect upon the market for the major product and their sale would further increase the revenue derived from the original wood raw material.

The naval stores industry today is still a million-dollar business, even though the market value of gum turpentine and rosin has been cut in half in the period from 1949 to 1959 (4). The increasing need for both turpentine and rosin as chemical raw materials has been met by supplies of wood naval stores from diversified sources. For example, P.M.L. grade turpentine derived from spent sulphate pulp liquor is combined with steam distilled turpentine in the manufacture of 30 per cent of the world's synthetic pine oil production (4).

Turpentine

Crude oil produced by steam distillation is almost entirely free from rosin spirits and rosin oil owing to the fact that rosin is not decomposed by the steaming and remains

in the wood after the turpentine has been distilled off. The normal products of distillation are turpentine, which has a boiling point range of 150 deg. C to 175-180 deg. C, and pine oil, whose boiling point range is 175-180 deg. C to 240-250 deg. C (16). The temperature of a mechanical veneer dryer seldom exceeds 205 deg. C, and usually fluctuates within the range of 175 deg. C and 197 deg. C. Because of these high temperatures, some crude wood oil will probably be steam distilled out of the veneer. It is to be expected that some of this crude oil will be driven out of the dryer through the roof ventilator stacks. When considering the temperature range involved in the drying operation, there is some justification in assuming that the major product to be derived from the crude wood oil will be turpentine.

It has been predicted by the chemical industry (25) that 16 per cent of chemical sales in 1960 will be in the form of new products. It is also estimated that due to research work in this field, about 10,000 new chemical products are made annually, 400 of which are of commercial significance. Two use patterns of turpentine are illustrated in the table below. Because these examples are derived from different sources, however, they are not directly comparable.

The uses of turpentine

	1941 (10)	1959 (4)
Chemicals and pharmacy	8.9 %	97.9 %
Paints, varnish, lacquer	11.1	1.3
Shoe polish, shoe material	2.7	0.5
Rubber	-	0.1
Over the counter sales	75.1	-
Miscellaneous	2.2	0.2

The most important modern uses of turpentine occur in the manufacture of synthetic pine oil, insecticides, synthetic resin and as a paint thinner and a petroleum additive.

The American Society for Testing Materials has suggested standard specifications to assist in the task of grading steam-distilled turpentine (1).

A.S.T.M. standards for steam-distilled turpentine

	Max.	Min.
Specific gravity at 20 deg. C	0.875	0.860
Refractive Index at 20 deg. C	1.478	1.465
Residue after Polymerization with 38 N sulphuric acid; volume per cent	2	
refractive index at 20 deg. C		1.500
Initial boiling point at 760 mm. pressure, °C	160	150
Distilling below 170°C at 760 mm. pressure per cent		90

Summary of salient points

(1) Douglas fir has a much lower resin content than the pine species upon which the naval stores industry is based. This difference is considerably

increased when comparison is made between Douglas fir and the selected highly resinous lightwood of southern pine, which is commonly used.

(2) Heartwood has a much higher resin content than sapwood, and this resin is not uniformly distributed throughout the wood of the tree.

(3) Steam produces crude wood oil in small quantity. A commercial enterprise employing only steam must have an abundant and readily available supply of highly resinous material in order to be profitable.

(4) Steam-distilled crude wood oil is mainly composed of turpentine and pine oil. The boiling point ranges of these fractions are separate and distinct, the turpentine being lower than that of pine oil. A small fraction which has an intermediate boiling range between the main constituent of turpentine (namely, pinene) and pine oil may also be present.

(5) Steam distillation has only been commercially successful with wood which has been broken down into small chips. No production of naval stores has been reported using wood material in the form of veneer.

(6) Veneer dryer operating temperatures slightly exceed the upper limit of the turpentine fraction's boiling point range.

(7) Increased speed of distillation and decreased steam pressure reduces total yield of crude oil.

(8) A good potential market exists for wood naval stores products of suitable quality. There is an increasing absorption by markets of wood naval stores which have been manufactured as by-products of other wood using industries, but the market value of gum naval stores is steadily declining.

(9) A company in Canada which produces only naval stores apparently cannot compete successfully with the corresponding industry in the United States because of a tremendous raw material advantage in the latter country.

Veneer Drying Equipment and Conditions of Drying

The investigation was conducted at the Vancouver Plywood Division of MacMillan, Bloedel and Powell River Ltd. in Vancouver, British Columbia. Actual sampling was performed on No. 1 and No. 2 dryers of "B" mill in this division. Both dryers were identical in design and a simplified plan of one of them can be found in Appendix A.

The dryer is a cross-circulation type constructed by the Moore Dry Kiln Company. The drying principle is to have the air come off the fans, circulate across the top duct, through the heating coils and into the plenum chamber at the side of the dryer, between the doors and the veneer rolls. This air then passes across the veneer before returning to the fan intake. The manner of circulating the air is similar in every zone of the dryer, but its direction of flow is reversed in

alternate zones.

The dryer had six decks and consisted of sixteen 63-inch drying, and two 63-inch cooling sections. Total dimensions of the dryer were as follows: length 117 ft. 8½ in., height 13 ft. 8 in., width 21 ft. 3 in. Electric power supply was 440 volts, 60 cycles and 3 phases. An eight-blade propeller-type fan, with an outside diameter of 42 in., and a rated capacity of 30,000 cu. ft. per minute, provided circulation. Each fan on the dryer sections was driven by a 10 hp General Electric induction motor at speeds of up to 1760 rpm. Each of the two fans in the cooling section was driven by a 15 hp General Electric induction motor at the same speed as used in the dryer. Steam was supplied through one 4-inch main and two 2½-inch sub-mains at a pressure of 260 pounds per square inch (psi). Pressure was recorded on an automatically controlled Moore-Kiln Pressure Recorder which had a range of zero to 300 psi. The dryer was equipped with 162 reheater coils of 1¼-inch fin pipe. The coil feeds decreased in diameter from 2-in. at the green end to 1¼-in. at the dry end. No steam traps were present, the condensate being drained through check valves and carried directly along a 6-inch main to the adjoining Canadian White Pine Division of the same company, where it was reused.

The dryer was equipped with automatic feeders, and speed rates could range from 4½ to 54 minutes. Normal drying speeds for Douglas fir, however, were as follows:

Dryer times for Douglas fir

1/10	in.	clear	7½ min.	1/10	in.	sap	17 min.
1/8	"	"	11 "	1/8	"	"	22 "
1/6	"	"	16½ "	1/6	"	"	29 "
3/16	"	"	18 "	3/16	"	"	36 "
1/7	"	"	14 "	1/7	"	"	25 "

In winter the time in the dryer for all thicknesses was one minute longer than the above times.

Six roof-ventilator stacks were located as shown on the plan in Appendix A. These stacks had a total height of 24 ft. and an inside diameter of 19 in. Because of the curvature of the factory roof, the ventilator stacks on No. 1 dryer projected 7½ ft. above the roof, while those on No. 2 dryer projected only 4 ft. above the roof. Each stack was equipped with a damper situated 6 ft. above the top of the dryer. Each damper was circular in cross section (18-inch diameter), and made of solid sheet metal. This was welded to a ½-inch diameter pipe through which ran a metal rod, this rod being held securely in position by set screws. For convenience of description, the stacks were numbered, starting at the green end of the dryer, as shown in Appendix B. Dampers were set as wide open as possible, provided suitable zone temperatures could be maintained. If the temperature in any zone could not be maintained within a range of 175 to 188 deg. C, then the damper was progressively closed to accomplish this condition. The most commonly used damper settings are shown in Appendix C.

Moore-Kiln Recording Thermometers, each with a range of 93 to 204 deg. C, measured the temperature of the air as it emerged from the reheaters. Thermometer bulbs were filled with pure, theophane-free benzene vapour. Two 3-pen recorders were used for sections one to six, and seven to twelve, the former having tube lengths of 30 ft., 70 ft., and 60 ft., and the latter 90 ft., 70 ft., and 110 ft. One 2-pen recorder, used on sections thirteen to sixteen, was equipped with tubes which were 55 ft. and 65 ft. long respectively. An examination of the twenty-four hour, automatically-recorded temperature charts for the period during which actual sampling took place, gave the figures for maximum, minimum, and average temperatures which are shown in Appendix B. The temperature of zone one was low because the circulated air in this zone was continually in contact with a mass of cold green veneer which had just entered the kiln, while the temperature of zone eight was lower because of its close proximity to the cooling sections of the dryer.

Prior to sampling the dryer vapours, a brief temperature study of No. 2 dryer was conducted. This was done under normal operating conditions of drying, in order to determine the temperature of these vapours both at the damper level and at the top of the stack. The temperatures were measured by a Brown Portable Potentiometer (Model 126 W3) using a copper-constantan thermocouple. These temperatures were compared with the maximum temperatures recorded on the Moore-Kiln Temperature Recorder charts at the exact times of measurement. Results of

this investigation are shown in Appendix B. From the results it is seen that there is no temperature gradient along the length of the ventilator stack. Any difference in temperature between the air in the stack and the air in the dryer would be caused by vapour which had been picked up by the exhausted air during drying operations.

The velocity of the air emerging from the stack varied through a fairly wide range. These velocities were measured by an Anemotherm Air Meter (Model 60) manufactured by the Anemostat Corporation of America, New York, and results obtained by this instrument are recorded in Appendix C. Air velocity will vary due to several factors, among which are damper setting; the position of the stack in relation to the fans; initial moisture content of the wood; temperature, humidity, and wind velocity of the atmosphere at the top of the stack; speed, dimensions, and type of fan used in the dryer, and vapour pressure of the dryer zones.

Collection of the Dryer Vapour

Sampling procedure

Only the vapour which emerged from the top of the stacks of the dryer was collected. Because No. 1 dryer handled only Douglas fir sapwood, whereas No. 2 dryer used only Douglas fir heartwood, each dryer was sampled separately so that a

comparison could be made between the amounts of extractive recovered from each type of wood. The stacks were sampled individually so that a comparison of the relative yields from different points on the dryer could be made, both along the length of each dryer as well as between corresponding locations on the two dryers. Each sample was gathered in one continuous operation which lasted over a period of several hours.

Method of collection

The apparatus was erected as illustrated in Figures 2 to 6 (inclusive).

The stack was capped with a circular (24-inch diameter) metal lid which had a circular hole, 4 in. in diameter, removed from its centre. This lid was held down by heavy rocks which gave a fairly tight seal around the perimeter of the lid. A 4-inch diameter stove pipe elbow, made of non-insulated, galvanized metal, was inserted into the centre of the lid before being connected to a 6-ft. length of stove pipe, made of the same material, and having the same diameter as the elbow. This pipe led down to a condenser (Figures 4,5,6) which was supported in a horizontal position by a cradle made of 3/4-inch plywood.

The condenser had three individual components namely, lid, water jacket, and condensing coils. The first two were made of cast iron (3/4-inch thick) whereas the coils were made of 1/2-inch (inside diameter) copper tubing. These three components

Fig. 2. Condensing apparatus in position

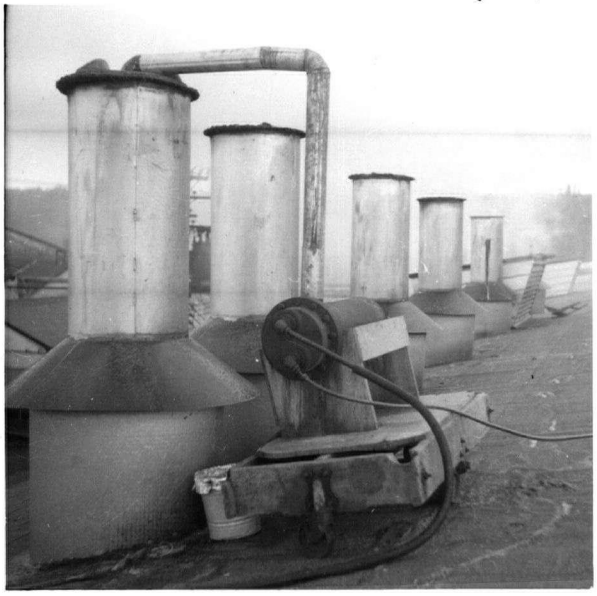

Fig. 3. Condensing apparatus in position

Fig. 4. Condenser

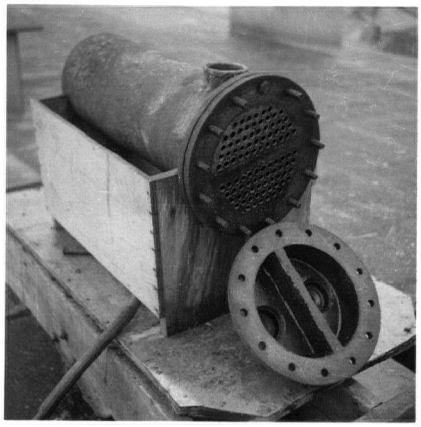

Fig. 5. Condenser (with lid removed)

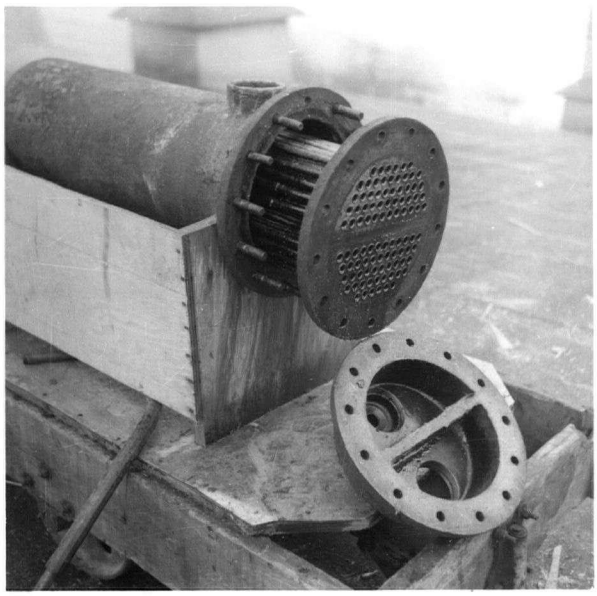

Fig. 6. Condenser (showing coils)

were held firmly together by thirteen 5/8-inch bolts. The condensing coils were arranged in five rows, each row having a different length. Measuring from the centre of the condenser towards the outside, the numbers of tubes and their lengths were:

Row	No. of Tubes	Length per Tube (in.)
Inner	12	39
2	11	40
3	10	42
4	9	44
Outer	6	45

The dimensions of the assembled condenser were as follows:

Total length	34 in.
Water jacket diameter (inside)	10½ in.
Upper rim diameter	14¾ in.
Diameter of outlets/inlets:	
Inside	3½ in.
Outside	4 in.

Water was fed to the condenser through a 50-ft. length of 3/4-inch plastic pipe connected to a fire hydrant which was located on the roof of the plant. Both the condenser and the cradle were mounted on a four-wheeled, flat-decked cart for ease of transportation between stacks. A 3½-ft. length of 1-inch rubber hosepipe led from the outlet valve at the base of the condenser to a five gallon drum in which the condensate was collected. This drum was fitted with a handle which facilitated handling when it was full. While collection of condensate was in progress, the top of the drum was completely sealed off from the atmosphere by means of a sheet of aluminum

wrapping foil. The period of time taken to collect each sample of condensate was recorded, as well as the actual date and times of sampling. This latter information was necessary in order to determine maximum temperatures during the period of sampling, these temperatures being read subsequently from the Moore-Kiln Temperature Recorder charts.

Instead of using the condenser in the conventional way with water going into the surrounding water jacket and the vapour being condensed within the coils, it proved more advantageous to reverse this procedure. This was done because of technical difficulties which became evident during trial runs with the equipment. The weight of the condenser, combined with the height of the stacks of No. 1 dryer above the roof of the plant, prevented the condenser from being used in a vertical position, directly above the stack opening. Further difficulty was caused by the high velocity of the emergent vapour. When the condenser was mounted horizontally in its cradle and was connected to the stack opening in the conventional manner, it was found that the high velocity of the emergent vapour created a back pressure at the entrance to the condenser coils. This negative pressure forced some of the vapour back up the lead-in pipe. As a result of this back flow, much of the vapour was forced out of the system at joints in the pipe. When connected in a reverse manner to that intended by the designer, and fitted with a regulatory valve attached to its outlet, the condenser was found to function with satisfactory

efficiency. Consequently, it was used in this manner throughout the investigation.

Quantitative Analysis of the Condensate

Description of the condensate

The condensate consisted of three general layers. The first of these was a layer of small, individual solid particles of wood. These were yellow ochre in colour and covered the bottom of the receiving drum. Above this was the main body of condensate consisting of a colourless, translucent aqueous mass. The surface of the condensate was covered by a thin film whose multi-coloured appearance was similar to that of gasoline floating on water. Also floating on the surface were small amounts of two different substances, both of which were tacky to the touch, and had a slightly higher viscosity than water. One of these substances was black in colour while the other was prussian green. Most of this material, however, was not floating on top of the condensate, but had become adhered to the metal wall of the container from which it could not be easily removed by the aqueous condensate.

Assumptions of the analysis

The following assumptions were made prior to the commencement of quantitative analysis work:

(1) The steam-distilled crude wood oil is composed of turpentine and pine oil.

(2) Components are all simple substances which are completely soluble in one another.

(3) None of the components is soluble in water.

These assumptions were based upon work done by Hawley and Palmer (18), and Palmer (24), and while they are not strictly true in all cases, none of them is sufficiently incorrect to have a serious effect upon the results and conclusions of the analysis.

Analytical procedure

A diagrammatical outline of the analysis of the condensate would be as follows:

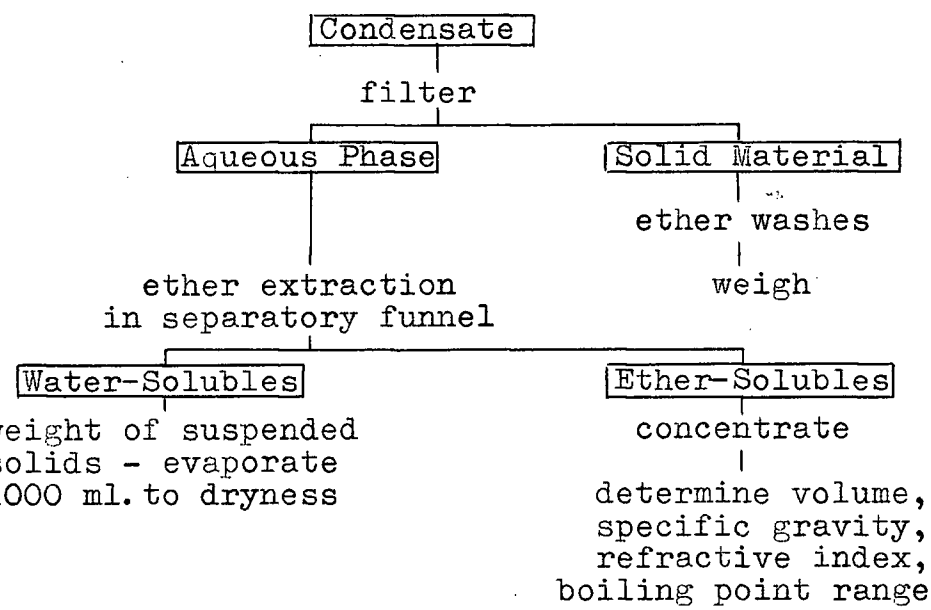

Fig. 7. Analysis of the condensate

Actual analysis was conducted in a series of stages:

Stage 1. Removal of solid material

This was done by means of vacuum filtration. The condensate was poured into Buchner-style porcelain filtering funnel.

The colourless, translucent filtrate was poured from the filtering flask into a 1000-ml. graduated cylinder which had a graduation interval of one milliliter. Each 1000-ml. quantity, or measured portion thereof, was then transferred for storage to a short-necked, narrow-mouthed, 2450-ml. round bottle equipped with a plastic screw cap.

When the rate of filtration declined appreciably, the Buchner funnel was transferred to another filtering flask of similar capacity and design to that already used, and the precipitate was washed with an ether solvent, using vacuum filtration. A minimum of three washes was applied, and washing was continued until the filtrate became colourless. The funnel was filled almost to the rim for each wash. The solvent used was reagent grade petroleum ether with a boiling range of 30 to 60 deg. C (ligroine). The condensates from stacks number 4 and 6 of No. 2 dryer were treated throughout with purified dry ethyl ether. For simplicity, however, only petroleum ether will be named as the solvent used in this investigation. The clear, amber-coloured solvent was was transferred from the filtering flask to a separate 2450-ml. bottle from that used for the initial filtrate.

Stage 2. Weight determination of solid material

All filter papers containing the entire solid portion from one sample of condensate were placed in a clean, dry, weighed, Petri dish. The dish was then weighed again before being placed, for a minimum of forty-eight hours, in an oven which was kept at a constant temperature of 105 deg. C. Periodic check weighings were made, and when loss of weight became negligible, the dish and contents were reweighed, the weight of the filter paper deducted, and the exact weight of the solid material calculated. All weighings were done using a Spoerhase Analytical Rapid Balance (Type No. 10).

Stage 3. Separation of ether-solubles from water-solubles

Two globe-shaped separatory funnels were used for this purpose. One had a capacity of 2000 ml. while the other had only a 250-ml. capacity. The filtrate from Stage 1. was poured through a short-stemmed filter funnel into the separatory funnel, followed by a substantial quantity of the same type of solvent which was used in Stage 1. The mixture in the funnel was then shaken vigorously before being allowed to settle into an upper solvent layer and a lower aqueous layer. The water-soluble layer was drawn off into 800-ml. beakers and the ether-soluble layer was run off into another 2450-ml. bottle. Removal of the water was done quite slowly in order to avoid a whirlpool effect in the funnel, which would have resulted in a

loss of some ether-soluble material. The water-soluble portion in the beakers was again placed in the separatory funnel and the procedure repeated before this water-soluble portion was discarded. The amber-coloured wash material from Stage 1. was also placed in the separatory funnel along with some of the initial filtrate and fresh solvent, and the separation process conducted in a similar manner to that described.

Stage 4. Recovery of solvent

Much of the solvent used in the analysis was recovered for re-use by the following method: Some of the ether-soluble solution was placed in a 1000-ml. long-necked, round-bottomed boiling flask. This was fitted to a flash-evaporator, and the ether recovered at a temperature not exceeding 37 deg. C and at a vacuum of 15 to 20 inches of mercury. The vandyke-brown-coloured mixture of crude oil and solvent was transferred to a 450-ml. short-necked, narrow-mouthed, round bottle equipped with a plastic screw cap, and the bottle labelled with the appropriate sample number.

Stage 5. Determination of the weight of suspended solids in the water-soluble portion

Exactly 1000 ml. of the water-soluble solution which was discarded from Stage 3. were boiled to dryness in a 250-ml. beaker whose initial clean dry weight was known. The beaker was weighed after evaporation had been completed, and the weight of the light red, solid material contained in it was

calculated in relation to 100 ml. of this water-soluble portion. Because of the small total volume collected from number 6 stack on No. 1 dryer, only 200 ml. of the water-soluble solution was boiled to dryness. Before heat was applied, the aqueous liquid was colourless and translucent, but shortly thereafter it turned a pale chrome-yellow colour. This colour became darker as more solution was evaporated from the beaker, and ended as a light red, solid material.

Stage 6. Determination of ether-soluble content

The ether-soluble material from Stage 4. was placed in a 250-ml. distilling flask. This flask was connected to a Liebig condenser which was made of heat resistant glass. A 100-ml. graduated cylinder with graduations at 0.1-ml. intervals was used to collect the ether solvent. A total-immersion, nitrogen-filled thermometer, graduated at 1 deg. C intervals, and with a range of zero to 200 deg. C, was inserted in the neck of the distilling flask so that the top of the mercury bulb was level with the bottom of the side tube. Heat was applied by a bunsen burner to the base of the distilling flask. Maximum temperature of evaporation was kept below 80 deg. C. Solvent was distilled off and the dark brown material left in the distilling flask was poured out into a 10-ml. graduated cylinder, which had 0.2-ml. graduation intervals, whereupon its volume was measured. Allowance had to be made at this stage for the very small amount of material which did not enter the graduated cylinder because of its surface tension.

After measurement, this material was transferred into a 120-ml. French square bottle equipped with a plastic screw cap. The distilling flask and graduated cylinder were washed out with petroleum ether solvent and this wash material was also poured into the bottle. This bottle was used to collect all other soluble material coming from one dryer so that at the conclusion of this stage of the analysis, the total amount of ether-soluble material collected from No. 1 dryer was in one bottle, while the corresponding material for No. 2 dryer was contained in a second bottle.

Stage 7. Determination of boiling point ranges

Micro-distillation equipment had to be used for this work because of the small quantity of ether-soluble material obtained in Stage 6. A connecting three-way tube with a 75-degree side tube was inserted in the top of a 25-ml. micro boiling flask which had a conical bottom. This flask was immersed in a bath of C.P. grade butyl phthalate (boiling point of 340 deg. C) so that the level of the bath was above that of the material inside the flask. A Liebig micro condenser was attached to the side tube. A 10-ml. graduated cylinder with 0.2-ml. graduation intervals was used as a receiving cylinder. A nitrogen-filled thermometer was fitted into the top of the connecting tube so that the top of the mercury bulb was level with the bottom of the side tube. This thermometer was graduated in 2 deg. C intervals with a range of zero to 250 deg. C.

The ether-soluble material was poured into the boiling flask and a small piece of pumice added to promote smooth distillation in case the sample contained any dissolved or suspended water. The flask was then attached to the apparatus. Heat from a bunsen flame was applied cautiously to the bath. The temperature was raised to 70 deg. C and all distillate collected up to this point was discarded because it mainly consisted of the ether solvent. The flask was allowed to cool slightly, after which it was removed and had more ether-soluble material added to it. This procedure was continued until all ether-soluble material had been poured into the boiling flask, and all distillate up to 70 deg. C was discarded.

The temperature was then raised cautiously, and the initial boiling point was taken as the thermometer reading at the instant when the first drop fell from the end of the condenser. When the temperature reached 150 deg. C, the flame was removed, and the volume of distillate in the graduated cylinder recorded. This material was returned to the original bottle which had contained the ether-soluble material. The flame was then replaced under the bath, and the temperature raised very slowly until 170 deg. C was reached, whereupon it was again removed. The volume of this fraction was recorded after which it was poured into a small glass capsule for further examination.

The flame was then replaced, and the volume of distillate was measured between 170 and 200 deg. C before

returning both this fraction, and the fraction above 200 deg. C which remained in the distilling flask, to the bottle which had contained the ether-soluble material. Distillation took place slowly and fairly uniformly throughout these three temperature ranges.

Stage 8. Determination of specific gravity and refractive index at 20 deg. C

Exactly 0.5 ml. of the clear, pale lemon-yellow fraction with the boiling point range of 150 and 170 deg. C was pipetted into a clean dry weighed bottle and its weight determined on an Analytical Rapid Balance. Specific gravity could be calculated from this information.

Refractive index of the same material was measured on an Abbe Refractometer. The temperature of the instrument room where both measurements were made was exactly 20 deg. C, so that no correction of the results was necessary because of temperature difference.

Calculation of Results

Total condensate - (theoretical loss of moisture in drying)

The results obtained from the analysis of the condensate are shown in Appendices D and E.

The initial moisture content of Douglas fir sapwood is approximately 116 per cent while that of the heartwood of the same species is approximately 40 per cent (26). All veneer is dried to a maximum moisture content of 5 per cent.

The average specific gravity of Douglas fir (green volume, oven-dry weight of wood) is 0.45. Total weight of wood at any moisture content can be calculated from the formula:

$W = W_o (1 + M/100)$ where W = weight of wood at moisture content M.

W_o = oven-dry weight of wood.

The oven-dry weight of one cubic foot of Douglas fir is 0.45 x 62.4 = 28.1 lb. Using the above formula, therefore, the moisture losses in drying the different types of Douglas fir wood to a 5 per cent moisture content will be as shown below:

	(Weights in lb. per cu. ft.) Wt. at initial M.C.	Wt. at final M.C.	Weight Loss
Sapwood	60.7	29.5	31.2
Heartwood	39.3	29.5	9.8

The average amounts of wood used in each dryer are as follows: No. 1 dryer = 25,000 square feet of 1/16-in. veneer per hour, or approximately 130 cubic feet per hour.

No. 2 dryer = 50,000 square feet of 1/16-in. veneer per hour, or approximately 260 cubic feet per hour.

The theoretical weight losses from these dryers are: (Conversion factor: 1 lb. = 453.6 gm.)

No. 1 dryer = 130 x 31.2 = 4056 lb. per hour

= 1,840 kg. per hour

No. 2 dryer = 260 x 9.8 = 2548 lb. per hour

= 1,156 kg. per hour

Table 1. Relationship between theoretical and experimental yields of total condensate

		(a)	(b)	(c)	(d)
Dryer No.	Stack	Tot. condensate collected per hour		Theoretical wt. loss per hr. on basis of (b)	% of (c) actually collected
		Amount	% of tot. amount		
		(gm.)		(kg.)	
1	1	197	7	129	0.15
	2	257	10	184	0.14
	3	743	28	515	0.14
	4	1221	46	846	0.14
	5	210	8	147	0.14
	6	14	1	19	0.07
	Total		100	1,840	Av. 0.14
2	1	318	18	208	0.15
	2	365	20	231	0.16
	3	242	14	161	0.15
	4	600	33	382	0.16
	5	189	11	127	0.15
	6	78	4	46	0.17
	Total		100	1,156	Av. 0.15

Explanation of Table 1.

Column (a): The period of time taken to collect the total condensate varied from stack to stack. Because different lengths of time were used for this work, it was necessary to calculate each yield of total condensate in terms of the same common time basis before any comparison of relative yields from the stacks could be made. Total condensate figures were therefore calculated on the basis of total quantity collected per hour. This was done by dividing column (c), Appendix E, by column (a) in the same Appendix. As the condensate consisted largely of water its weight was considered to be 1 gram per milliliter.

Column (b): This states the amount of total condensate collected per hour from each stack expressed as a percentage of the total amount of condensate collected per hour from the entire dryer.

Column (c): The theoretical weight loss for the wood in the dryer distributed between the dryer stacks on the basis of the percentages in column (b). This gives

an indication of the theoretical amount of moisture which should be collected from each stack of the dryer.

Column (d): The amount of total condensate actually collected (a) expressed as a percentage of the theoretical amount which could be collected (c).

Total volatile oil fraction of the condensate

Theoretical determination of the amount of volatile oil driven off in steaming can be made if the ether solubility and the composition of the resin are known. The ether solubility of Douglas fir was found by Hawley (17) to be 1.02 per cent of the intial oven-dry weight of wood. In his work on Douglas fir, Schorger (29) concluded that the volatile oil comprised only 29.5 per cent (average) of the heartwood oleoresin and 22.7 per cent of the sapwood oleoresin. Using Hawley's ether solubility factor as a measure of the average resin content of both the heartwood and sapwood of Douglas fir, it is seen that, on the basis of Schorger's results, the amount of volatile oil in heartwood will be approximately 0.30 per cent of the oven-dry weight of this type of wood. The corresponding value for sapwood will be 0.23 per cent of the oven-dry weight of sapwood.

The assumption that none of the non-volatile rosin material comes off is not strictly correct. As only a very small amount of this material might be present (16, 17, 23) it

is felt that its exclusion will not have a serious effect upon deductions and conclusions of the analysis.

No. 1 dryer:

The average oven-dry weight of Douglas fir = 28.1 lb. per cu. ft.
The yield of volatile oil = 28.1 x 0.23% = 0.065 lb. per cu. ft.
= 29.5 gm. per cu. ft.
Specific gravity of volatile oil = 0.863 (Table 6)
Weight of an equal volume of water = 29.5 ÷ 0.863 gm.
= 34.2 gm.
Yield of volatile oil, therefore, equals 34.2 ml. per cu. ft.
Volume of wood used in dryer = 130 cu. ft. per hour
Theoretical volume of volatile oil driven off from sapwood, therefore, will be 34.2 x 130 = 4446 ml. per hour

No. 2 dryer:

Yield of volatile oil = 28.1 x 0.30% = 0.084 lb. per cu. ft.
= 38.1 gm. per cu. ft.
Volume yield of volatile oils = 38.1 ÷ 0.863
= 44.1 ml. per cu. ft.of wood
Volume of wood used in the dryer = 260 cu. ft. per hour
Theoretical volume of volatile oil driven off from heartwood, therefore, will be 44.1 x 260 = 11,466 ml. per hour

The actual yields of ether-solubles which were collected from the dryers have been calculated from Appendix E to give the results shown in Table 2.

Table 2. Volatile oil recovered from the dryers

		(a)	(b)	(c)
Dryer No.	Stack No.	Volatile oil recovered per hr. (ml.)	% of total vol. of oil recovered	Volatile oil fraction as % of total condensate
1	1	0.04	4	0.01
	2	0.13	14	0.05
	3	0.28	29	0.04
	4	0.39	40	0.03
	5	0.07	7	0.03
	6	0.06	6	0.43
	Total	0.97	100	Av. 0.10
2	1	0.07	9	0.02
	2	0.09	11	0.03
	3	0.21	26	0.09
	4	0.33	41	0.06
	5	0.06	8	0.03
	6	0.04	5	0.05
	Total	0.80	100	Av. 0.05

Explanation of Table 2.

Column (a): The ether-soluble fraction of the total condensate recovered from each stack was calculated on the basis of quantity recovered per hour, for the same reason that was given for doing similar calculations to the figures for total condensate (Table 1). Results in this column were calculated from Appendix E

by dividing the figures in column (e) by those in column (a).

Column (b): This is the amount of volatile oil collected per hour from each stack expressed as a percentage of the total amount of these oils collected per hour from the entire dryer.

Column (c): The volume of volatile oils collected per hour (a) calculated as a percentage of the total volume of condensate collected per hour (column (a), Table 1).

Theoretical percentage of volatile oils recovered in the experiment.

No. 1 dryer:

Theoretical volume of volatile oils driven off was calculated to be 4446 ml. per hour.

Actual volume collected = 0.97 ml. per hour

Theoretical recovery = 0.97 ÷ 4446 = 0.022%

No. 2 dryer:

Theoretical recovery = 0.80 ÷ 11,466 = 0.007%

These are assessments of the percentage recoveries achieved in the experiment based upon the ether solubility calculations of Hawley (17), and the work done by Schorger (29) on the composition of Douglas fir oleoresin.

The most important limitations of this theoretical data have already been pointed out, but this method gives some indication of the proportion of volatile oil material which was probably recovered in the experiment.

An assessment of the proportion of volatile oil material which was recovered in the experiment can also be made by comparing the experimental results with yields from actual commercial distillation work which has been done in the past. For this purpose the yields from steam and solvent distillation of ordinary stumpwood of Douglas fir (13) will be used as a standard, as this wood raw material most closely approximates to the resin content of the wood from the bole of the tree which was used to manufacture veneer. The heartwood-to-sapwood ratio in this stumpwood is not known.

As the stumpwood is termed "ordinary" it is felt that its moisture content will be between the green condition and the air-dry (12 per cent) condition. Weight of wood at 12 per cent moisture content (air-dry) = 31.5 lb. per cu. ft. Weight of wood at 116 per cent moisture content (green sapwood) = 60.7 lb. per cu. ft. Weight of wood at 40 per cent moisture content (green heartwood) = 39.3 lb. per cu. ft.

The results of the steam and solvent extraction of ordinary Douglas fir stumpwood (13) are recorded on page 18. The total oil fraction recovered was 0.9 U.S. gal. = 7.2 U.S. pints = 3430 ml. (1000 ml. = 2.1 U.S. pints). This was

extracted from 3800 lb. of wood, therefore, 1 gram of wood should yield 0.002 ml. of volatile oil.

No. 1 dryer:

(1) Green wood: Total weight of wood dried per hour.
= 60.7 x 130 = 7891 lb. per hour
= 3,579,280 gm. per hour
Yield of stump volatile oil
= 3,579,280 x 0.002 = 7160 ml. per hour
Yield from the dryer = 0.97 ml. per hour
Recovery = 0.97 ÷ 7160 = 0.01 per cent.

(2) Air-dry wood: Recovery = 0.97 ÷ 3715 = 0.02 per cent.

No. 2 dryer:

(1) Green wood: Total weight of wood dried per hour
= 39.3 x 260 = 10,218 lb. per hour
= 4,634,780 gm. per hour
Yield of stump volatile oil
= 4,634,780 x 0.002 = 9,270 ml. per hour
Yield from the dryer = 0.80 ml. per hour
Recovery = 0.80 ÷ 9,270 = 0.0086 per cent.

(2) Air-dry wood: Recovery = 0.80 ÷ 7430 = 0.01 per cent.

The calculations of volatile oil from the veneer dryers based on the yields recorded from ordinary Douglas fir stumpwood show distinct similarity to the corresponding calculations using the data of Hawley and Schorger. Exactly similar recoveries occur if the sapwood of ordinary Douglas fir stumpwood was in an air-dry condition while the heartwood

was in a green state. One possible explanation for similarity in recoveries is that the ordinary stumpwood which was distilled in a commercial steam and solvent plant was fresh cut, green and consisted largely of heartwood, with only a narrow zone of sapwood being present. The yields from ordinary Douglas fir stumpwood will be used for further calculations because these are based on actual recoveries and are not merely theoretical amounts which might be recovered. It is also felt that results calculated on the basis of green wood will be more realistic of the actual condition of the wood which was distilled.

The theoretical yield of volatile oil from the dryers based on ordinary Douglas fir stumpwood recoveries are therefore as follows:

No. 1 dryer = 7160 ml. per hour

No. 2 dryer = 9270 ml. per hour

These yields can be subdivided into turpentine and pine oil fractions on the basis of the actual recoveries which were achieved from the extraction of this same stumpwood material.

Table 3. Relationship between theoretical and experimental yields of volatile oils.

	No. 1 Dryer	No. 2 Dryer
Total volatile oil (ml. per hour)	7,160	9,270
Recovery from dryer (per cent)	0.01	0.008
Turpentine fraction (ml. per hour)	4,770	6,180
Recovery from dryer (per cent)	0.002	0.0005
Pine oil fraction (ml. per hour)	2,390	3,090
Recovery from dryer (per cent)	Not known	Not known

Explanation of Table 3

Results of the work done on Douglas fir stumpwood (13) show the total volatile oil material to consist of 67 per cent turpentine and 33 per cent pine oil. The above volumes were calculated on the basis of these percentages, using values for theoretical volatile oil yields of 7160 ml. per hour for No. 1 dryer, and 9,270 ml. per hour for No. 2 dryer.

The total amount of volatile oil, and the volume of the turpentine fraction (boiling point range of 150 to 170 deg. C) of this volatile oil were determined from data in Appendix D and Table 2, and these amounts were calculated as a percentage of the theoretical volatile oil yield.

Summary of calculations

The following is a summary of the volume yields obtained by the investigation. Also shown are theoretical volumes which have been calculated in order to provide standards for comparison between actual yields and the volumes which theoretically could have been recovered.

Table 4. Summarized comparison between theoretical and actual yields

(Volumes expressed in U.S. gallons per 24-hour day)

Dryer No.		Theoretical yield	Actual yield	Percentage recovered
1	Total condensate	11,590	16.5	0.1
	Total volatile oils	45	0.006	0.01
	Turpentine	30	0.0006	0.002
	Pine oil	15	-	-
2	Total condensate	7280	11.4	0.1
	Total volatile oils	58	0.005	0.009
	Turpentine	38	0.0002	0.001
	Pine oil	20	-	-

Discussion of Results

Assumptions in the theoretical calculations

Several important assumptions were made when calculating and assessing the results of the investigation. Initial moisture contents of both sapwood and heartwood veneer were not measured. The moisture content figures which have been used in calculating the theoretical loss of moisture in drying, therefore, are averages which were obtained by Peck (26) over a large number of samples. The proximity of these values to the average moisture content of the veneer during the period in which the vapour was collected is not known. If Peck's averages are higher than the amount of moisture which was actually present in the veneer, then a larger proportion

of the vapour would have been collected than is indicated by Table 1.

The amounts of veneer passing through the dryers per hour were estimated, but these estimates were considered by the management of the plant to be very representative of the average amounts of sapwood and heartwood veneer which are handled each day in No. 1 and No. 2 dryers respectively.

The limitations of both Hawley's ether solubility value and Schorger's data on volatile oils have already been pointed out. One most important addition to these limitations, however, is the fact that these figures were derived from stumpwood and not from the ordinary wood in the bole of the tree from which veneer is made. Stumpwood has a higher resin content. In longleaf pine for example, ordinary sapwood contains 2 per cent resin, heartwood 7 to 10 per cent resin, while the heartwood of the stumpwood contains 25 per cent or more resin. There is, therefore, a substantial difference between the amount of resin which is present between the wood of the stump and the wood of the bole of the tree. In this investigation the recovery of volatile oil from wood which has the least resin content has had to be related to a corresponding theoretical recovery from the most resinous wood because of a lack of more applicable data from previous work done with Douglas fir in this field.

Another assumption which has been made is that the proportion of volatile oil in the ether-soluble extractive

material is fairly constant. In the case of maritime pine, however, it has been reported that the volatile oil content of the ether-soluble extractives may vary from 0.57 per cent to as low as 0.07 per cent (31), and no records are available to substantiate or deny the possibility of a variation of similar magnitude occurring in the ether-soluble extractives of Douglas fir.

The assumption that none of the non-volatile rosin material comes off was also mentioned above. Should any of this rosin be present in the ether soluble portion of the condensate, this will decrease the yield of volatile oils which was thought to have been collected. While steam distillation is known to produce the lowest volatile oil yield of all commercial distillation processes, the standards which were calculated for the assessment of these yields were based on data derived from more efficient extractive processes.

Total condensate

From Table 1 it can be seen that No. 1 dryer produced a higher total volume of condensate than No. 2 dryer. This is because the sapwood had a much higher initial moisture content than heartwood, and both were dried to a 5 per cent moisture content.

Moisture was driven off fairly uniformly along the length of No. 2 dryer although an abrupt increase in volume of condensate occurred at stack no. 4. Stack nos. 1,2, and 6

of this dryer produced higher yields than the corresponding stacks of the No. 1 dryer. The volume recovered from stack no. 3 is quite low compared to recoveries of condensate from the adjacent stacks but there is no apparent reason for this occurrence.

In the case of No. 1 dryer, moisture was driven off slowly at first, but the volume of moisture increased sharply at stack no. 3, and reached a maximum at stack no. 4, before decreasing markedly at the dry end. This trend is thought to be due to the high initial moisture of the wood. This would increase the time required for the wood substance to reach a temperature level at which the moisture would be readily removed.

The dryer is so designed that maximum volume of vapour should be released from stack nos. 2,3, and 4, and this is confirmed by the results of the investigation. These stacks are located in the centre of the zone and directly in the path of the main stream of air which comes off from the fans. Twin fans are used to circulate air in these zones, thereby forcing more of the moisture-laden air up the stacks. The first stack is located between zones one and two, as comparatively little moisture is released in this initial area because the wood is still relatively cold. The area of the dryer from zone 5 to zone 8 is equipped with only one stack between two zones, and only one fan per zone, because most of the moisture has been removed from the veneer before it reaches these zones.

The most important single factor which caused the total yield of condensate to be much lower than the amount anticipated prior to sampling, is that only one stack was capped at a time, while all of the five others remained open during sampling operations. There is no doubt that a substantial amount of vapour was diverted up adjacent stacks in the dryer, thereby being allowed to escape, because of the partial blockage of the stack which was being sampled. As the investigation was considered to be only a pilot study of the condensate and its constituents, a large investment in the proper equipment which would have been necessary to condense all of the vapour from all stacks of the dryer simultaneously was not felt to be economically justifiable. This work, therefore, had to be done in the best way possible using the limited amount of equipment which was available at that time. It was also understood that such a study should not adversely affect, or hinder, normal drying operations at the plant. Any measure which would restrict the free escape of dryer vapour by physically blocking off all stack exits except the one being sampled, therefore, was considered to be out of the question.

The position of the condenser in relation to the direction of escaping vapour was not ideal but was necessary for practical reasons such as the condenser's great weight, together with the fact that the stacks on No. 1 dryer were 7½ feet high. An additional, and important drawback to the

condensing apparatus, however, was the fact that vapour-laden air in the 19-inch diameter stack had to be fed into the condenser through a 4-inch diameter pipe. This meant that only a portion of the air which was forced up the stack actually entered the condenser. In the case of stack no. 4 on No. 2 dryer, air velocity was increased from 2000 feet per minute in the stack, to 3000 feet per minute in the lead-in pipe, but the actual volume of air which was transferred was reduced from 3940 to 260 cubic feet per minute. If the air was heavily moisture-laden, this factor would greatly reduce the amount of collectable condensate. Air velocities of adjacent stacks were not noticeably affected by the presence of the condensing apparatus.

Damper setting appears to have little or no effect upon the amount of vapour which was collected from each stack as evidenced by the fact that the largest yield from both dryers was obtained from stack no. 4 of No. 1 dryer, the damper of which was set in a "closed" position. The half-inch gap between the edge of the damper and the inside wall of the stack, together with the high velocities at which the air is circulated inside the dryer is thought to render the damper comparatively ineffective as a means of providing a physical barrier to the passage of moist air out of the dryer.

Volatile oil

The pattern of volatile oil recovery was very similar to that of total condensate recovery. The condensate from

almost every stack on No. 1 dryer produced a higher volume of volatile oil than the corresponding condensate from No. 2 dryer, thereby resulting in a higher total yield of volatile oil from the former. Volatile oil recovery also showed the same trend along the dryer as before, with the maximum yields occurring at stack no. 4 of each dryer. It is felt that the reasons for these yield patterns are similar to those which have already been suggested for the recovery of the total condensate.

There are several factors affecting the yields of volatile oil which throw some light on possible reasons both for the low total recovery of these volatile oils which was experienced, and for the difference in recovery between heartwood and sapwood.

The length of time taken for steam distillation is of major importance with regard to the amount of volatile oil recovered. In general, the longer the period of distillation, the better will be the yield of volatile oil. In commercial practice, wood is steam distilled for three to four hours before it gives what is considered to be a satisfactory yield of crude wood oil. Veneer which passed through the dryers, on the other hand, was only subjected to a steam distillation for a period of eight to eight and a half minutes in the case of No. 2 dryer, and seventeen minutes in the case of No. 1 dryer, thus the yields are bound to be considerably lower than those experienced in commercial naval stores practice using the same

species. The veneer in No. 1 dryer was steam treated for twice as long as that in No. 2 dryer and this probably accounts for the higher yields from the former in spite of the fact that the heartwood is known to have a larger resin content than sapwood.

Although maximum dryer temperatures did not greatly exceed the upper boiling range of the turpentine fraction, the vapour pressures of the volatile oil constituents had a beneficial effect by lowering the boiling point of the oil and water mixture. Examination of vapour pressures of the main volatile oil constituents leads to the conclusion that the temperature within the dryer is more than adequate to produce total distillation of the turpentine fraction, and that none of this fraction will condense inside the dryer. The same cannot be said for the pine oil fraction, which has a higher boiling range than turpentine. Tar-like deposits of a dark brown material of unknown composition were visible along the top of the dryer doors in zones 7 and 8, as well as on the inside wall of stacks 5 and 6 on both dryers. This material most probably consists of a mixture of pine oil and wood tar. One possible reason for this deposition of material would be the cooling influence of the air in the two cooling sections situated at the end of the dryer.

The turbulence of air within the dryer produces a good mixture of gases, and the dryer is designed to exhaust all gases through the roof ventilator stacks.

The amount of volatile oil recovered from the one-tenth inch thick veneer which was dried will be less than the amount calculated on the basis of yields from the steam and solvent process, because, in the latter case, volatile oils were extracted from wood which had been broken down into particle form. Wood particles allowed for more intimate contact with the wood substance by the steam, and the greater amount of end grain exposed by the particles would be beneficial to the amount of extractive recovered.

The method of sampling and the efficiency of the equipment which was used to collect the condensate have already been discussed in relation to total volume of condensate collected. The same points which were made in this case also apply to the amount of total volatile oil material which was recovered, as this volume is directly related to the amount of condensate collected.

A factor which would markedly affect the volatile oil yields which were recovered would be the presence of rosin in the ether-soluble material. The latter was assumed to consist entirely of turpentine and pine oil. Rosin lowers the vapour pressure and decreases the oil-to-water ratio in the distillate. The occurrence of rosin would mean a reduction in the amount of volatile oil which was actually recovered in the investigation. This rosin, however, would increase the proportion of the turpentine fraction which was actually collected in relation to the total volume of volatile oil which was present, thereby

enabling this fraction to approximate more close to the theoretical proportion of sixty-seven per cent which was found in the case of the volatile oil from Douglas fir stumpwood. The presence of non-volatile rosin in the ether-soluble material might have been made possible when the small solid wood particles which were driven out of the stacks along with the vapour were washed thoroughly with petroleum ether. It was noted during the quantitative analysis (stage 1) that the ether wash was amber coloured and that this colour became darker as the solution became more concentrated.

The boiling range study on the volatile oil material produced the results which are recorded in Appendix D. The clear, pale lemon-yellow coloured fraction with a boiling range of 150 to 170 deg. C comprised only 10 per cent and 4 per cent of the total volumes of volatile oil recovered from No. 1 and No. 2 dryers, respectively. This is the boiling range within which most of the turpentine will occur. Specific gravity and refractive index of this fraction were measured and gave results which were slightly higher than the maximum values for wood turpentine suggested by the A.S.T.M. standards (1). The volume of this material was extremely small compared to that of the total condensate collected. In view of the approximate selling price of turpentine of ninety-two cents per gallon, it is quite clear that the amounts, either separate or combined, of this material which were collected from the dryers were definitely not of commercial value.

Most of the volatile oil fraction consisted of a black, slightly viscous, oily-looking substance with a boiling point in excess of 200 deg. C.

Summary

Crude wood oil is steam distilled out of Douglas fir veneer as it passes through mechanical dryers. A small amount of this oil was collected from the moisture-laden dryer vapours which are forced out of the roof ventilator stacks. More volatile oil was collected from sapwood veneer than from heartwood veneer.

A boiling range determination on this oil showed that over 80 per cent of it had a boiling point in excess of 200 deg. C, while only 4 to 10 per cent of it occurred within a boiling range similar to that of wood turpentine (150 to 170 deg.C).

The quantity of volatile oil which was recovered in this investigation was too small to be considered of commercial value.

It is recommended that no further work of this type should be done on the dryers until proper standards are determined for evaluating both the efficiency of sampling and the quality of the ether-soluble material collected. This will require both a quantitative and a qualitative analysis of the

ordinary Douglas fir heartwood and sapwood which is used to manufacture veneer for plywood production. Both analyses should be performed by a qualified chemist and should be conducted under laboratory conditions so that ideal values may be determined. If the results of the analyses are favourable, an examination of the market potential of those volatile oil components which will be of commercial importance should then be undertaken. Only if this market analysis work also proves encouraging should further sampling work on the dryers be carried out. Better condensing equipment will be necessary if more efficient sampling is desired. With regard to sampling, it appears that any additional work on the dryers need only be done on stack numbers 2, 3, and 4, although some method must be found at the same time to prevent the escape of excessive amounts of vapour from the other stacks, without adversely affecting the normal conditions of drying within the veneer dryer.

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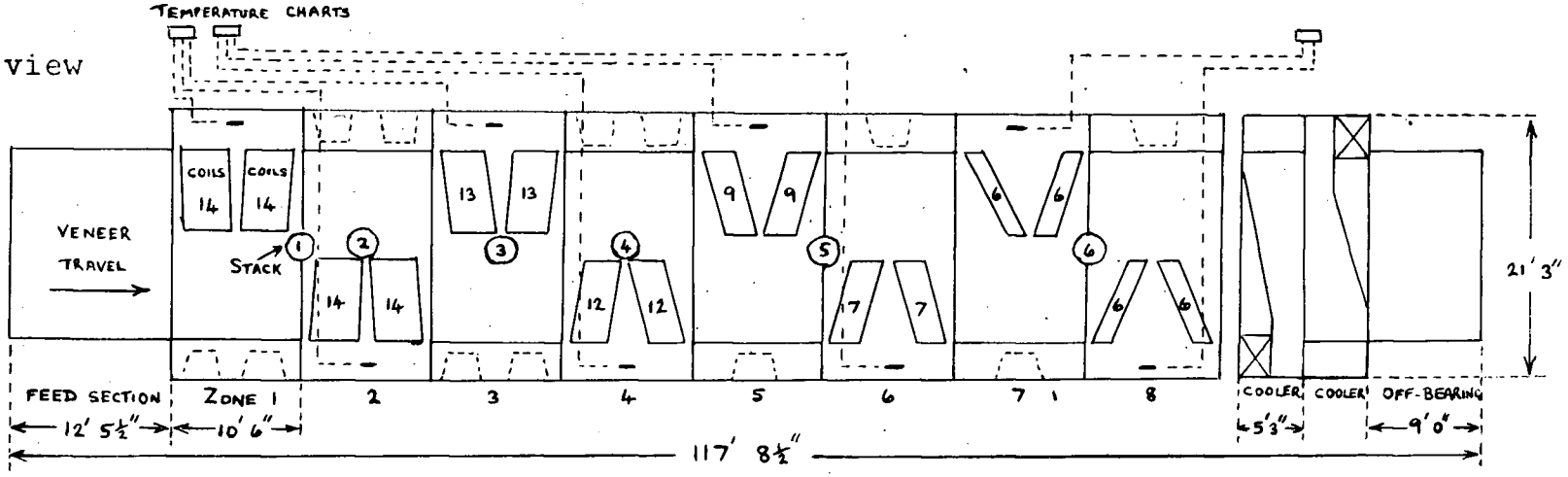
APPENDICES

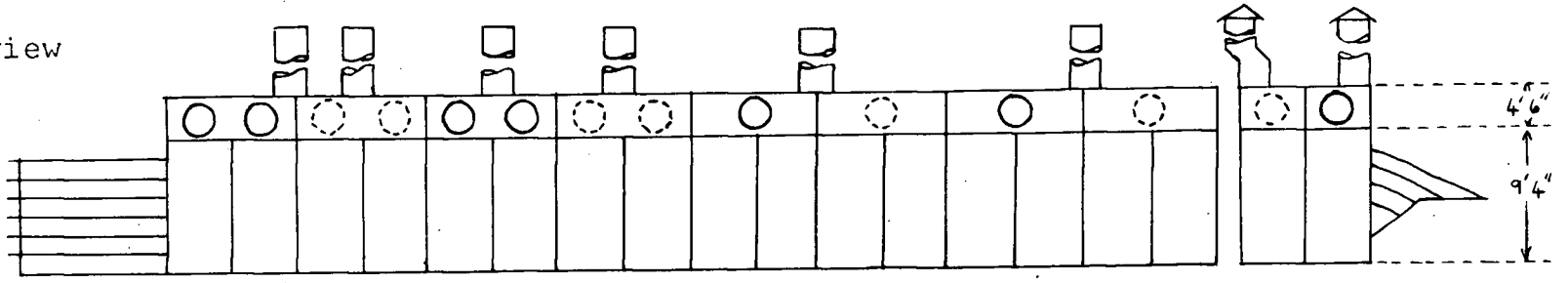
Appendix A

Simplified Plan of a Veneer Dryer (not to scale)

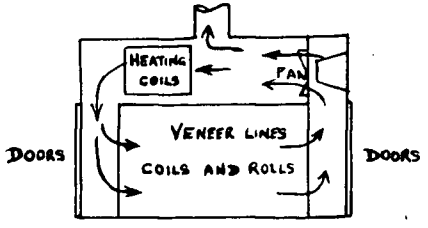
Plan view



Side view



End view



Appendix B

Dryer Temperatures

No. 1 Dryer		Temperature (°C)		
Stack no.	Zone no.	Min.	Max.	Average
1	1	174	182	175
1 and 2	2	187	195	192
3	3	189	200	194
4	4	189	195	192
5	5	195	204	199
5	6	197	204	201
6	7	196	202	200
6	8	183	202	185

No. 2 Dryer		Temperature (°C)		
Stack no.	Zone no.	Min.	Max.	Average
1	1	162	177	168
1 and 2	2	182	193	185
3	3	177	191	183
4	4	190	200	192
5	5	194	203	197
5	6	192	199	195
6	7	188	192	190
6	8	189	197	192

Temperature Reduction Due to Moisture Pick-up and Radiation Losses

Stack	Vapour Temperature (°C)		Dryer (°C) Temperature
	Top of Stack	Damper Level	
1	158	158	167
2	154	154	183
3	159	159	180
4	178	178	191
5	159	164	194
6	57	82	189

Appendix C

Stack Air Velocity

No. 1 Dryer

Stack	Zone	Damper Setting	Velocity (ft. per min.)
1	1 and 2	Quarter open	550
2	2	Quarter open	1400
3	3	Quarter open	700
4	4	Closed	1350
5	5 and 6	Closed	700
6	7 and 8	Closed	350

No. 2 Dryer

Stack	Zone	Damper Setting	Velocity (ft. per min.)
1	1 and 2	Quarter Open	450
2	2	Quarter open	550
3	3	Quarter open	1400
4	4	Half open	2000
5	5 and 6	Half open	400
6	7 and 8	Closed	300

Appendix D

Boiling Point Range Determination

Temperature (°C)	Percentage of Sample	
	No. 1 Dryer	No. 2 Dryer
75 to 150	2	11
150 to 170	10	4
170 to 200	4	4
above 200	84	81

Note: Results for No. 1 dryer are based on a sample of 17.8 ml.

Results for No. 2 dryer are based on a 0.9 ml. sample (initial B.P. of 78 deg. C).

Examination of 150 deg. C to 170 deg. C Fraction

Specific gravity at 20 deg. C = 0.877

Refractive index at 20 deg. C = 1.4788

Appendix E

Quantitative Analysis Results for Total Condensate

No. 1 Dryer

	(a)	(b)	(c)	(d)	(e)
Vent. stack	No. of hours collecting	Wt. of solids (gm.)	Tot.vol. collected (ml.)	Wt. of suspended solids in 100 ml. H_2O Solubles(gm.)	Volatile oils (Ether-solubles) vol. (ml.)
1	22	1.91	4330	0.26	0.8
2	23	1.10	5920	0.36	3.0
3	26	3.50	19310	0.30	7.2
4	16	5.44	19530	0.08	6.2
5	23	1.46	4840	0.13	1.5
6	23	0.37	330	0.13	1.4
Total	133	13.79	54260		20.1

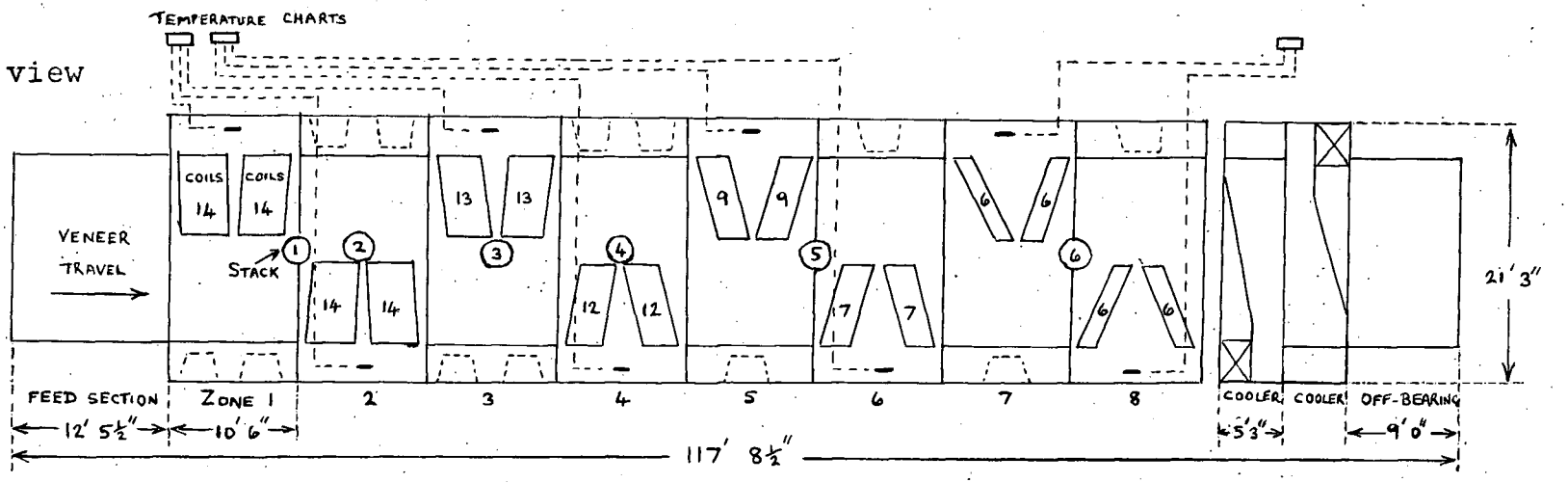
No. 2 Dryer

	(a)	(b)	(c)	(d)	(e)
Vent. stack	No. of hours collecting	Wt. of solids (gm.)	Tot.vol. collected (ml.)	Wt. of suspended solids in 100 ml. H_2O sol-ubles(gm.)	Volatile oils (Ether-solubles) vol. (ml.)
1	24	7.87	7640	0.09	1.7
2	49	5.63	17870	0.06	4.6
3	24	5.17	5810	0.13	5.0
4	18	3.78	10810	0.07	6.0
5	23	3.22	4340	0.10	1.4
6	64	0.35	4960	0.10	2.7
Total	202	26.02	51430		21.4

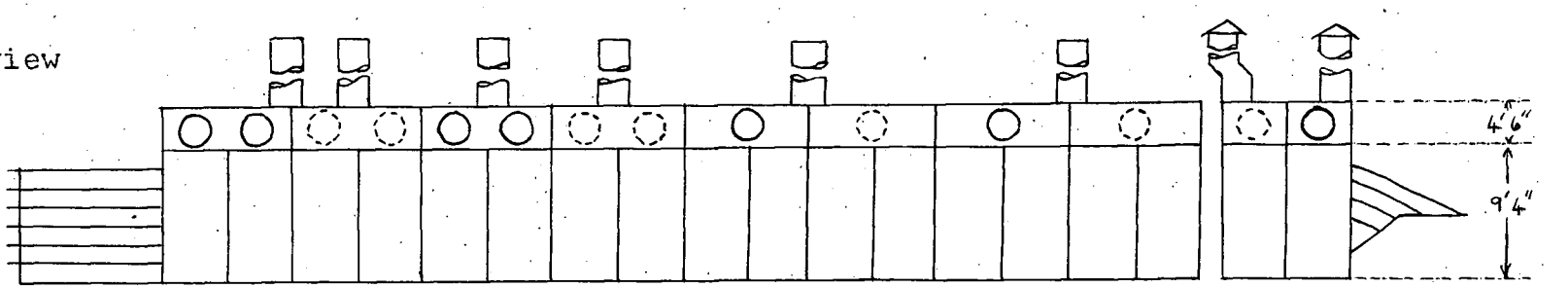
Appendix A

Simplified Plan of a Veneer Dryer (not to scale)

Plan view



Side view



End view