LABORATORY SCREENING TRIALS WITH CHEMICALS FOR THE PROTECTION OF GREEN TIMBER AGAINST FUNGI

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ABSTRACT

Thirty-two chemicals were tested for effectiveness against stain, decay, and mould fungi on freshly sawn sapwood of **Pinus radiata** D. Don. Eighteen of these were quaternary ammonium compounds. The performance of some quaternary ammonium compounds was improved when used in combination with other fungicides or under highly alkaline conditions. None of the chemicals or mixtures which controlled sapstain are currently cost-effective for the New Zealand market when compared with the standard treatments of 0.2% a.i. captafol or 0.5% sodium pentachlorophenoxide plus 1.5% borax pentahydrate.

INTRODUCTION

All freshly sawn green timber is susceptible to infection by sapstain and decay fungi during seasoning and needs to be protected by a chemical treatment. Also, protection is required for natural rounds which are prone to degrade during drying and for packaged green timber destined for export. Apart from degrade by sapstain and decay fungi, the superficial growth by mould fungi can be a problem marring the appearance of timber.

In New Zealand, sodium pentachlorophenoxide (NaPCP), was the only phophylactic chemical used commercially until the early 1970s. Since that time, various alternative chemicals have been introduced after successful laboratory and mill trials (Butcher 1973, 1980; Butcher & Drysdale 1978). These trials were undertaken because of concern regarding possible environmental and health risks associated with the use of sodium pentachlorophenoxide and its future availability. The antisapstain chemicals now used in New Zealand have been described by Butcher (1980). It is of interest to note that although less toxic chemicals are now available, other factors limit their widespread adoption by industry.

Captafol is less toxic than sodium pentachlorophenoxide but it has been known to cause a skin irritation or allergic reaction in some workers. Because captafol is insoluble in water it is unsuitable for some methods of application, in particular for

re-circulating spray systems and in large dip tanks without agitation. A concentration of 0.2% a.i. is recommended but in winter it can be dropped to 0.15% a.i. which makes the cost comparable to sodium pentachlorophenoxide. Captafol is also available formulated with chlorothalonil, which gives improved mould control.

Folpet is another water suspension which has been used from time to time. It is more expensive than captafol and is not as effective. However, Folpet has the advantage of not causing skin irritation problems often associated with the handling of captafol-dip-treated timber.

Commercial formulations of copper-8 quinolinolate at 0.025% a.i. have been shown to give good mould, stain, and decay control. Although this product is water soluble and can be applied by spraying or dipping, it is of low pH and therefore corrosive. Also, copper-8 quinolinolate is chemically unstable in the presence of iron which can discolour dipped timber if a simple inhibitor is not added. Existing dip and storage tanks need to be protected with epoxy resin coatings.

None of these alternative antisapstain chemicals fulfil all the requirements of the "ideal" treatment. This elusive chemical should be colourless, odourless, of high fungicidal activity, suitable for application by dipping or spraying, and persistent on the wood without being toxic to plant or animal life; it should not cause skin irritation or be harmful to the environment. It must also be freely available at a price comparable to existing treatments, or be likely to be available in the future at a comparable price. Because of these constraints on any alternative chemical, sodium pentachlorophenoxide remains the most widely used antisapstain chemical – accounting for approximately 70% of the market.

The inadequacies of existing treatments have necessitated the continuance of an active screening programme. Although some of the chemicals have been tested previously overseas, the effectiveness of chemicals can vary between timber species and between countries. For example, captafol has performed satisfactorily in Australasia and the Pacific but has provided inadequate protection under North American and European conditions. By contrast, methylene bisthiocyanate has performed well under European conditions but poorly in New Zealand. Laboratory tests of all potential prophylactic treatments are therefore necessary so that performance against local fungal species on local timber can be defined. This paper records the results of laboratory screening trials carried out over the past 4 years on *P. radiata.*

MATERIALS AND METHODS

The laboratory screening method has been described previously by Butcher (1973) and Butcher & Drysdale (1978). One half of a 300 × 100 × 25-mm piece of freshly sawn *P. radiata* sapwood was dipped into the test chemical. The entire length was then sprayed with a fungal suspension of *Penicillium* sp., *Trichoderma viride* Pers. ex Fr., *Alternaria tenuis* Nees, *Ceratocystis* sp. *Diplodia pinea* (Desm.) Kickx, *Phlebia gigantea* (Fr. ex Fr.) Donk, *Fibroporia vaillantii* (D.C. ex Fr.) Parm., *Schizophyllum commune* Fr., and *Trametes versicolor* (L. ex Fr.) Pilat. Ten replicate pieces for each solution concentration were randomly selected to account for any between-tree variability. After inoculation the 10 replicate pieces were fillet-stacked in two piles of five pieces each.

Pile were randomly stacked to eliminate the effect of any fumigant action. High humidity conditions were maintained by wrapping the complete test in polythene. The test was incubated at room temperature and chemical effectiveness was assessed after 6 weeks.

On completion of the test each piece of wood was examined visually and the mean percentage surface area affected by sapstain and/or decay was assessed. Growth by mould fungi affected the grading of a treatment only where more than 20% of the surface area was covered.

At least two commercially accepted treatments were included in each test. These were usually 0.5% NaPCP plus 1.5% borax, or 0.2% a.i. captafol. Any treatment which performed as well as these "standards" was considered successful.

The chemicals tested are listed in Table 1 with their toxicity where known; known chemical structures are given in Fig. 1. Eighteen were quaternary ammonium compounds and four were copper-8 quinolinolate formulations. Sodium carbonate and various other chemicals (products 32–39) were tested as mixtures with quaternary ammonium compounds.

RESULTS AND DISCUSSION

Sample boards dipped in the standard treatment 0.5% NaPCP plus 1.5% borax developed slight sapstain; up to 10% of the surface area was infected after 6 weeks.

Captafol, the second standard treatment always included, was tested as Haipen 50 WP, a 50% a.i. wettable powder formulation. At 0.2% a.i. this treatment performed better than 0.5% NaPCP plus 1.5% borax. After 6 weeks test boards were completely free of stain and decay. Occasionally the mould *Trichoderma viride* grew on some test boards but less than 20% of the surface area was infected.

Haipen C, another wettable powder formulation tested, was as effective against sapstain at 0.2% a.i. as captafol (0.2%). The chlorothalonil additive improved mould control.

Two water-soluble copper-8 quinolinolate formulations (PQ57 and PQ375) protected the test boards from sapstain, decay, and mould at 0.025% a.i. Plackett (1982) recommended a 0.11% a.i. solution for protection of hemlock and fir species in Canada. A water-dispersive formulation, Copakote (50% a.i.), did not control sapstain at any concentration up to 0.1% a.i. Another formulation, Copakote C, performed no better, complete control being achieved only at 0.17% a.i. It was concluded that although water-dispersive formulations had the advantage of being non-corrosive, they were less satisfactory as fungicides than the water-soluble formulations.

Velcide tested as a 0.5% a.i. solution was ineffective. Plackett (1981) found a 2.6% solution (equivalent to 0.95% a.i.) to be effective on hemfir* and Douglas fir (*Pseudotsuga menziesii* (Mirb.) Franco). We found the addition of 1.5% borax pentahydrate to Velcide improved performance but it was still less effective than the

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* Hemfir is a collective term for three species - western hemlock (*Tsuga heterophylla* (Raf.) Sarg.), red fir (*Abies amabilis* (Dougl.) Forb.), and grand fir (*A. grandis* (Dougl.) Lindl.).

TABLE 1—List of chemicals tested

Product	Active ingredient	Toxicity LD_{50} (acute, oral) to rats
1.* Santabrite technical grade granules	sodium pentachlorophenoxide	50–140 mg/kg
2.* Haipen 50 WP (50% active) (Chevron)	cis-N-[(1,1,2,2-tetrachloro ethyl) thio]-4-cyclohexene-1,2-dicarboximide; captafol	6200 mg/kg
3.* Haipen C (Chevron)	50% captafol (as above) plus 16.7% tetrachloro-isophthalonitrile (chlorothalonil)	chlorothalonil 10 000 mg/kg
4.* (a) PQ57 (5% active) (b) PQ357 (3.5% active) (Chemical Cleaning Co.) (c) Copakote (NZ Farmers Fertiliser Co.)	copper-8 quinolinolate	>10 000 mg/kg
5.* Copakote C (NZ Farmers Fertiliser Co.)	37.5% copper-8 quinolinolate plus 25% chlorothalonil	>10 000 mg/kg
6.* Velcide (40% active) (Velsicol Int.)	sodium tribromophenoxide (2,4,6-tribromophenol, Na salt)	1905 mg/kg
7. R23979/R42611	confidential	
8. R28644/10EC (Janssen Pharmaceutical Products. NZ agent — NZ Farmers Fertiliser Co.)	confidential	
9.* Busan 30 (30% active) (Buckman Laboratories)	2-(thiocyanomethylthio) benzothiazole	1590 mg/kg
10.* Kilstain (10% active) (R. H. Gurney Chemicals Ltd (UK))	methylene bisthiocyanate	150–300 mg/kg
11.* Rovral (50% active) (May & Baker New Zealand Ltd)	3-(3,5-dichlorophenyl)-N-(1, methylethyl)-2, 4-dioxo-1-imidazolidine carboxamide. Also 3-(3,5-dichlorophenyl)-1-isopropylcarbamoyl hydantoin and IPRODIONE	4400 mg/kg
12. Kabicut K2 (Mitsui Kumaia)	"organo nitrogen compound"	>100 000 mg/kg
13. Kabicut K400 (Mitsui Kumaia)	"organo chlorine compound"	11 000–15 000 mg/kg
14. Prochloraz (40% active) (NZ Farmers Fertiliser Co.)	1-N-propyl-N-(2-(2,4,6-(trichlorophenoxyl)ethyl) carbamoyl)imidazole	
15.* Arquad C50 (50% active) (Akzo Chemie UK Ltd)	alkyl (8% C_8, 9% C_{10}, 47% C_{12}, 18% C_{14}, 8% C_{16}, 5% C_{18} octadecyl, 5% C_{18} octa decenyl) trimethyl ammonium chloride	
16.* Arquad 2C (75% active) (Akzo Chemie UK Ltd)	dialkyl (8% C_8, 9% C_{10}, 47% C_{12}, 18% C_{14}, 8% C_{16}, 10% C_{18}) dimethyl ammonium chloride	

TABLE 1—List of chemicals tested—continued

Product	Active ingredient	Toxicity LD_{50} (acute, oral) to rats
17.* Arquad T50 (Akzo Chemie UK Ltd)	Tallow trimethyl ammonium chloride	
18.* Ethoquad C_{12} (75% active) (Akzo Chemie UK Ltd)	coco di(hydroxyethyl) methyl ammonium chloride	>2500 mg/kg
19. ES229 (75% a.i.) (Akzo Chemie UK Ltd)	confidential	
20.* Bardac 20 (50% a.i.) (Lonza)	dialkyl (50% C_8 C_{10}, 25% C_8 C_8, 25% C_{10} C_{10}) dimethyl ammonium chloride	520 mg/kg
21.* Bardac 22 (50% a.i.) (Lonza)	didecyl dimethyl ammonium chloride	600–700 mg/kg (mice)
22.* Gloquat C (50% active) (ABM Chemicals)	alkyl aryl trimethyl ammonium chloride	
23. IWD3491 (75% a.i.)	quaternary ammonium compounds	
24. IWD3492 (75% a.i.)		
25. IWD3493 (75% a.i.)		
26. IWD3494 (50% a.i.)		
27. IWD3495 (40% a.i.)		
28. IWD3496 (20% a.i.) (Ivon Watkins Dow)		
29.* Protek Q (50% active) (Ivon Watkins Dow)	alkyl (64% C_{12}, 30% C_{14}, 6% C_{16}) benzyl dimethyl ammonium chloride	975 mg/kg
30.* CABQ100 (80% active) (Buckman Laboratories)	n-alkyl (50% C_{14}, 40% C_{12}, 10% C_{16}) benzyl dimethyl ammonium chloride	
31.* Bradophen (100% active) (CIBA-Geigy)	benzyl-dodecyl-bis-(2-hydroethyl)-ammonium chloride	1000 mg/ka
32.* O-phenyl phenol (100% active)	o-phenyl phenol	2480 mg/kg
33. RH948 (96.6% active) (Rohm and Haas)	N-cyclohexyl-4,5-dichloro-isothiazolin-3-one	
34.* Skane M8 (45% active) (Rohm and Haas)	2-*n*-octyl-4-isothiazolin-3-one	1470 mg/kg
35.* C9211 (85% active)	4,5-dichloro-*n*-octyl-4-isothiazolin-3-one	
36.* Sisthane 242 (25% a.i.) (RH2161-fenapanil from Rohm and Haas)	α-n-butyl-α-phenyl-1H-imidazole -1-propanenitrile	
37. Sodium DMG (100% a.i.)	sodium dimethylglyoxine	
38.* Sodium omadine (40% active) (Olin Corp NZ)	sodium 1-hydroxy-2-pyridimethione	
39.* Thiabendazole (45% active)	1,2-(thiazol-4-yl) benzimidazole	

* Chemical structure given in Fig. 1

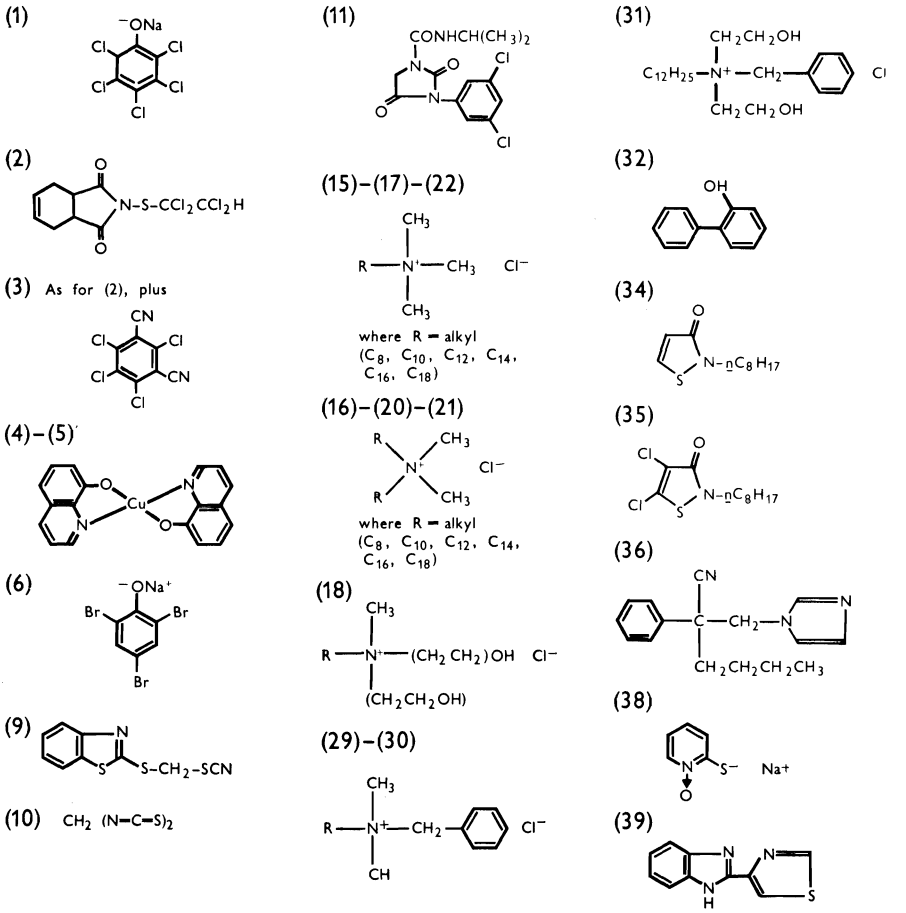

FIG. 1—Chemical structures of some of the products tested

standard sodium pentachlorophenoxide treatment, confirming the earlier results of Butcher & Drysdale (1978). Although of lower mammalian toxicity than sodium pentachlorophenoxide, Velcide is unlikely to be a cost-effective treatment.

Two experimental solutions, R23979/R42611 and R28644/10EC, were tested. The R23979/R42611 formulation at 0.3% product was ineffective but sapstain control was achieved at 0.5% product. The R28644/10EC at 0.5% product was less satisfactory, Neither formulation is cost-effective at the 0.5% concentration.

Busan 30 is an emulsifiable microbiocide already used in the pulp and paper industry in New Zealand. This product was tested over a range of concentrations from 0.15 to 0.3% product, but at 0.3% Busan 30 the test boards were still being decayed. This confirmed the results of Butcher & Drysdale (1978). A 1.5% a.i. solution was recommended by Milano (1981) for the protection of *Pinus elliottii* Engelm. in Brazil and Plackett (1982) has suggested a 2.0% a.i. level for hemlock and some fir species in Canada, but cost-effectiveness limits the adoption of these high concentrations of

Busan 30 in New Zealand. Control of fungal infection could be improved by the addition of borax pentahydrate. Lower borax levels (×2 to ×6 the Busan concentration tested) had an adverse effect but the addition of ×8 or ×10 borax was beneficial. Whereas surface infection was 91% with 0.2% Busan 30, it was less than 20% with the higher levels of borax. The ratio of borax to Busan 30 appeared critical and would probably be difficult to control on a commercial scale; therefore work on this product with borax has been limited.

Kilstain (methylene bisthiocyanate) was tested at 0.5, 0.1, 0.2, and 0.4% active ingredient. After 3 weeks' incubation, 5–20% of the surface area was affected by mould although free of stain and decay. At 6 weeks every test board was completely covered by the mould *Trichoderma viride.* Similar excessive growth of *Trichoderma viride* was previously noted by Butcher & Drysdale (1978) on another formulation of methylene bisthiocyanate. In the United Kingdom, Dickinson (1977) found the performance of methylene bisthiocyanate at 0.2% a.i. to be equivalent to 1.0% NaPCP. He observed slight mould infection on *Pinus sylvestris* L. after 2 months' storage but this was considered acceptable. Plackett (1981) tested this product at 0.2 and 0.5% a.i. on hemfir and Douglas fir. He recommended the higher concentration as an acceptable alternative treatment and no mention was made of a mould problem under Canadian conditions.

Rovral was tested at 0.1 and 0.2% a.i. but even at the higher concentration all the test boards were 50% sapstained.

An organo nitrogen compound, Kabicut K 2, and an organo chlorine compound, Kabicut K400, were both tested at 1.0 and 2.0% product. Kabicut K 2 was the superior formulation but at the 2.0% concentration test boards were still 17% sapstained.

Prochloraz was screened because this chemical has specific activity against Ascomycetes and Fungi Imperfecti to which the mould and sapstain fungi belong. Tested at 0.1 and 0.2% a.i., test boards were 40 to 45% sapstained.

Eighteen quaternary ammonium compounds were screened in these laboratory tests and complete sapstain control was achieved with 1.0% a.i. solutions. This supports the findings of Butcher & Drysdale (1978) who tested three quaternary ammonium compounds (an alkyldimethylbenzyl ammonium chloride and two dialkyldimethyl ammonium chloride compounds). However, Richardson (1972) and Milano (1981) found even double this concentration to be inadequate for other timber species.

No significant differences in the level of sapstain control on *P. radiata* were found between the various quaternary ammonium types (products 15–31). Richardson (1972) found alkyldimethylbenzyl, alkyltrimethyl, or alkylbenzyltrimethyl formulations controlled stain of *P. sylvestris* at half the concentrations required for the dialkyldimethyl and dialkylmethylbenzyl formulations. Hulme & Thomas (1979) also found quaternary ammoniums varied in effectiveness in controlling fungal stain of *Pinus strobus* L. in Canada. An alkyltrimethyl compound, where the alkyl chain was predominantly dodecyl and tetradecyl, was best. Although the present tests indicated that a far lower solution concentration was required than recommended by Richardson (1972), quaternary ammoniums would not at present be a cost-effective treatment when used at 1.0% a.i.

Richardson (1972) found the performance of Gloquat C was improved by the addition of Timbor (sodium octaborate) or boric acid. Since then Hulme & Thomas

(1979) in Canada have tested nine quaternary ammonium compounds at 0.5% a.i. with sodium carbonate (Na_2CO_3) in laboratory tests and found the addition of sodium carbonate improved effectiveness against sapstain. In a field test, 0.25 and 0.5% a.i. trimethylcocoammonium chloride plus 5.0% sodium carbonate treatments performed better than the 0.5% NaPCP plus 1.5% borax standard treatment. Consequently, in the present study a limited number of quaternary ammoniums were compared at 0.25% a.i. (which is approaching cost-effectiveness) to which different levels of sodium carbonate were added.

The addition of 5% Na_2CO_3 to the quaternary ammoniums improved their performance against sapstain, decay, and mould (Table 2) confirming the results of Hulme & Thomas (1979). They thought the higher alkalinity of the solution caused the quaternary ammonium to rapidly fix to the surface of the test board. The chemical mechanism by which this occurs has been explained by Rosen (1975). Arquad C50 showed the best response of the four quaternary ammoniums amended with sodium carbonate. Results with other quaternary ammoniums were affected by the development of an even light-brown stain caused by the high alkalinity of the solutions. Bardac 20 plus carbonate was the worst affected.

TABLE 2—Effect on sapstain, mould, and decay control of adding sodium carbonate to quaternary ammonium compounds

Quaternary ammonium compound (0.25% a.i.)	Surface area infection (%)			
	0	1.0% Na_2CO_3	2.5% Na_2CO_3	5.0% Na_2CO_3
Arquad C50	100	85	65	36
Bardac 20	100	100	65	82*
Protek Q	100	100	95	87
Gloquat C	93	100	98	73

* Brown alkaline stain

As Arquad C50 showed the best response with sodium carbonate, it was tested in a further trial in combination with a number of other fungicides (Table 3). The two standard treatments (0.5% NaPCP plus 1.5% borax, and 0.2% a.i. captafol) almost completely protected the test boards from any infection by sapstain, decay, or mould fungi. The unamended Arquad C50 0.25% a.i. boards were completely sapstained. Arquad C50 plus Busan 30, Prochloraz, 0-phenylphenol, Skane M8, sodium DMG, thiabendazole, or sodium omadine were also ineffective treatments. Test boards dipped in Arquad C50 plus RH948 0.04% a.i. were lightly sapstained. Arquad C50 plus 5.0% Na_2CO_3, or Sisthane 242 0.05% a.i., or C9211 0.025% a.i., were all combinations giving sapstain control equivalent to the standard treatments. The Arquad C50 plus 5.0% Na_2CO_3 appeared to give an improved performance in this later trial as the earlier result (Table 2) was influenced by excessive mould growth.

Skane M8 is added to paint as a fungicide (Post *et al.* 1976) and has previously been tested as an antisapstain treatment (Butcher & Drysdale 1978), and so Arquad C50

TABLE 3—Control of sapstain, mould, and decay with Arquad C50 modified solutions

Formulation	Surface infection after 6 weeks (%)
NaPCP 0.5% + 1.5% borax	10
Haipen 50WP 0.2% a.i. captafol	4
Arquad C50 0.25% a.i.	100
Arquad C50 0.25% a.i. + 5.0% Na_2CO_3	8*
Arquad C50 0.25% a.i. + Busan 30 0.75% product	100
Arquad C50 0.25% a.i. + Busan 30 1.5% product	100
Arquad C50 0.25% a.i. + Prochloraz 0.05% a.i.	52
Arquad C50 0.25% a.i. + O-phenylphenol 0.025% a.i.	100
Arquad C50 0.25% a.i. + O-phenylphenol 0.05% a.i.	70
Arquad C50 0.25% a.i. + Skane M8 0.025% a.i.	100
Arquad C50 0.25% a.i. + Skane M8 0.05% a.i.	44
Arquad C50 0.25% a.i. + C9211 0.025% a.i.	11
Arquad C50 0.25% a.i. + C9211 0.05% a.i.	10
Arquad C50 0.25% a.i. + Sisthane 242 0.025% a.i.	100
Arquad C50 0.25% a.i. + Sisthane 242 0.05% a.i.	15
Arquad C50 0.25% a.i. + sodium DMG 0.025% a.i.	100
Arquad C50 0.25% a.i. + sodium DMG 0.05% a.i.	100
Arquad C50 0.25% a.i. + thiabendazole 0.025% a.i.	100
Arquad C50 0.25% a.i. + sodium omadine 0.025% a.i.	100
Arquad C50 0.25% a.i. + sodium omadine 0.05% a.i.	100
Arquad C50 0.25% a.i. + RH948 0.025% a.i.	100
Arquad C50 0.25% a.i. + RH948 0.04% a.i.	30

* Some light-brown alkaline stain

and five other quaternary compounds were screened in combination with it (Table 4). All six quaternary ammonium treatments at 0.25% a.i. without Skane M8 were 100% sapstained. The addition of 0.04% Skane M8 was sufficient to improve the performance of all the quaternary ammoniums except Arquad 2C. Protek Q performed best of all the quaternary ammonium compounds with 20% of the surface area of the test boards sapstained.

CONCLUSIONS

None of the chemicals or mixtures tested controlled sapstain, decay, or mould of *P. radiata* at a concentration that is presently cost-effective for the New Zealand market in comparison with 0.5% sodium pentachlorophenoxide plus 1.5% borax pentahydrate, 0.025% a.i. copper-8 quinolinolate, or 0.2% a.i. captafol.

Quaternary ammonium compounds are biologically effective at a concentration four times higher than that which could currently be considered approaching cost-effective-

TABLE 4—Sapstain, mould, and decay control with Skane M8 added to various quaternary ammonium compounds

Formulation	Surface infection after 6 weeks (%)
Arquad C50 0.25% a.i. + Skane M8 0.04% a.i.	35
Arquad 2C 0.25% a.i. + Skane M8 0.04% a.i.	100
Protek Q 0.25% a.i. + Skane M8 0.04% a.i.	20
Bardac 22 0.25% a.i. + Skane M8 0.04% a.i.	33
Gloquat C 0.25% a.i. + Skane M8 0.04% a.i.	31
Ethoquad C12 0.25% a.i. + Skane M8 0.04% a.i.	50

ness. Amendment of quaternary ammoniums by the alteration of pH or addition of other fungicides is either not practicable or too costly to be commercially acceptable.

Treatments considered as acceptable alternatives by overseas research workers have been found to be either less effective on *P. radiata* against local fungi (e.g., methylene bisthiocyanate) or too expensive (e.g., Busan 30).

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