EVALUATING POTENTIAL DECAY CONTROL AGENTS WITH A SMALL BLOCK TEST¹

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ABSTRACT

A small-scale test was developed to evaluate the ability of fungicides to control decay fungi established in wood. The test, which uses blocks $2.5 \times 2.5 \times 10$ cm, tests the ability of a chemical to migrate from the middle of the block to control a previously established decay fungus, *Poria carbonica*. The effect of block size, degree of fungal development, chemical exposure period, and aeration during exposure were evaluated on chemicals previously shown to be effective for remedial control of decay. The method was then used to evaluate potential decay control chemicals. Highly volatile chemicals proved to be most effective, while water-soluble and oil-borne chemicals produced much poorer control. The small block tests appear to provide a simple, rapid, and accurate method for predicting how chemicals will control established decay fungi in wood.

Keywords: Fumigants, Douglas-fir, Poria carbonica, wood decay, internal decay, remedial treatment.

INTRODUCTION

The use of volatile chemicals to prevent and arrest decay of wood in service has become a widely accepted practice (Morrell and Corden 1986). A recent survey revealed that 85% of the utilities contacted used fumigants as a part of their routine wood maintenance program (Goodell and Graham 1983). At present, three fumigants—sodium n-methyldithiocarbamate as an aqueous 32.7% solution (Vapam®), Vorlex® (20% methylisothiocyanate, 80% chlorinated C-3 hydrocarbons), and chloropicrin (trichloronitromethane)—are registered with the United States Environmental Protection Agency for application to wood.² However, these formulations are all liquids that present considerable handling problems. There is much interest in identifying safer fumigant formulations.

The development of effective decay control chemicals generally involves a series of extensive field tests. These tests usually are conducted over at least 5 years, since effective chemicals must be able to remain in the wood for long periods after treatment. Thus, field-testing of these chemicals is a time-consuming and

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expensive process that may not be warranted except for those formulations most likely to perform well under actual service conditions. Small-scale accelerated tests can identify these most promising formulations before extensive field testing occurs.

Accelerated laboratory block tests to identify promising fumigants should set parameters that reflect conditions under which the chemical is expected to perform in the field. These conditions include variations in wood moisture content, aeration, temperature, incubation period, and the presence of wood defects that may affect chemical movement and fungitoxicity. This paper presents a laboratory method that duplicates these conditions well enough to predict the field performance of potential agents for control of decay.

MATERIALS AND METHODS

Potential fumigant formulations were tested with a small block assay in which freshly cut, defect-free Douglas-fir heartwood (Pseudotsuga menziesii (Mirb.) Franco) blocks $2.5 \times 2.5 \times 10.0$ cm were sterilized for 25 min at 121 C before being sealed on each cross section with masking tape. Douglas-fir was chosen because this species is susceptible to internal deterioration in service and because its heartwood is difficult to penetrate with liquids. The sealed blocks were briefly dipped in hot paraffin (60 to 70 C) to seal the longitudinal wood surfaces. The tape was then removed and the blocks were soaked in sterile distilled water to raise the wood moisture content to above 50%. The moistened blocks were inoculated by placing on each cross section a 1-cm agar square cut from a 1.5% potato dextrose agar medium containing actively growing mycelium of the test fungus, Poria carbonica Overh. The agar squares were pressed against the block ends with water-soaked Douglas-fir heartwood blocks $2.5 \times 2.5 \times 1.25$ cm, held in place with tight-fitting rubber bands. The assembled blocks were placed into plastic boxes on plastic supports. One hundred ml of a 100-ppm benomyl solution was added to the bottom of each chamber to retard growth of contaminants. The blocks were incubated at room temperature (25 C) until the fungus had thoroughly colonized the wood. At selected time intervals, blocks were removed and the wood from the center of each block was cultured to determine the degree of fungal colonization. These tests indicated that the fungus had colonized the whole of each block after 2 weeks of incubation. At this point the wood moisture content had declined to between 30 and 40%-sufficient for fungal growth but not high enough to inhibit chemical movement.

After incubating, a hole 2.2 cm deep \times 1.25 cm in diameter was drilled in the center of each block and sealed with a tight-fitting rubber serum cap. The candidate chemicals were applied through the cap in 1.5-ml dosages with a 5-ml syringe. Their concentration was varied by diluting the active chemical with acetone or water to the required dosage. Solid chemicals were applied before the rubber serum caps were added.

At the end of each incubation period, the blocks were taken from the chambers. The small blocks were removed, and two sections 0.5 cm thick were cut from each block end. The outer section of each was discarded and the next section was cut into 16 equal-sized cubes. The outer cubes were discarded. The inner four cubes were tested for fungal viability. They were placed on the surface of 1.0% potato dextrose agar plates amended with 10 ppm benomyl. A drop of the same

Fumigant	Concentration _ (mg/block)	Inhibition of P. carbonica after exposure for		
		1 Wk	2 Wk	4 Wi
Chloropicrin	5.0	27	98	85
Vorlex [®]	17.2	37	80	100
NH ₄ HF ₂	25	62	97	100
MIT	53	31	100	100
Vapam [®]	100	13	52	96
Shell DD®	450	77	97	95

 TABLE 1. Effects of three exposures after treatment on the toxicity of six fumigants to Poria carbonica in Douglas-fir heartwood.

benomyl solution was applied to each cube. The plates were incubated for 1 month at room temperature. The cubes were observed 4 weeks later for evidence of fungal growth, which was used as a measure of chemical performance.

This method was used to evaluate test variables and chemical performance in the following procedures:

Evaluation of known fumigants

The ability of the small block test to select effective fumigant formulations was evaluated on five formulations that had previously been field-tested. Chloropicrin, Vorlex[®], Vapam[®], MIT, and Shell DD[®] (chlorinated C-3 hydrocarbons) were individually applied in dosages ranging from 3 to 1,000 mg of active ingredient per block. The blocks were sampled 1 week after treatment to determine the percentage of fungal survival. These values were plotted to produce a dosage response curve.

Evaluation of treatment variables

A small-scale test should approximate actual field conditions but be flexible enough to permit evaluation of formulations with variable properties. Four variables that can affect test performance are block size, length of incubation of the fungus, duration of exposure to the chemical, and degree of aeration of the treated block. Block size makes a difference because the chemical must spread farther in larger blocks to effect fungal control. Blocks 10, 20, and 30 cm long were treated with either Vapam[®] or crystalline NaMDC. The Vapam[®] contained 32.7% active ingredient (NaMDC) in water, while the pure NaMDC was applied dry.

Because the fungus colonizing the wood must be as sensitive in the small block test as it would be in the field, the effect of the length of fungal incubation time prior to chemical treatment was investigated. Blocks were incubated for 2, 3, 4, and 20 weeks prior to treatment with Vapam[®] or chloropicrin at concentrations ranging from 0 to 150 mg per block. The blocks were then harvested as described above.

The effect of the duration of chemical exposure was investigated with both the 1-week evaluation of known fumigants and a longer-term investigation. In the short-term tests, chloropicrin, Vorlex[®], MIT, Vapam[®], Shell DD[®], and ammonium bifluoride (ABF) were applied and incubated for 1, 2, and 4 weeks before sampling (Table 1). These chemicals either are volatile or produce volatile compounds that can move rapidly through the wood to control the test fungus. They

	Concentration (mg/block)	Inhibition of P. carbonica after exposure fo		exposure for
Fungicide		1 Mo	2 Mo	6 Mo
Hollow Heart		100	100	100
OPC×906		100	100	100
OPC×968		97	100	100
Mylone [®] (Dazomet [®])	316	87	100	100
FCAP	476	58	100	100
ZnMDC	302	0	100	100
ACA		50	72	75
Penta (40%)		0	95	95
Penta (5%)	1,110	0	7	35
Captan [®]	1,000	0	0	0
Creosote	1,653	0	0	0
Chlorothalonil®	1,000	0	0	0
Maneb	933	0	0	0
CCA		0	0	0

 TABLE 2. Effects of three exposures after treatment on the toxicity of 14 fungicides to Poria carbonica in Douglas-fir heartwood.

were evaluated at levels that produced about 40% fungal inhibition after 1 week of exposure. The effect of longer exposure on the performance of less volatile chemicals was investigated over a 6-month period with a total of 14 chemicals (Table 2).

The final test variable investigated was the necessary degree of aeration of the treated blocks during chemical exposure. Highly volatile chemicals move rapidly through and out of the wood. If tests are conducted in closed containers, they run the risk of yielding falsely high readings because accumulated vapors of fungitoxic chemicals may enhance performance beyond that actually found in the field. The effect of aeration was examined in blocks treated with chloropicrin, Vorlex[®], allyl alcohol, and Vapam[®] by incubating blocks in sealed and unsealed chambers.

Once the test parameters had been determined, the small block test was evaluated on a wide variety of potential agents for control of wood decay, 28 chemicals in all. They included chemicals currently used as initial wood treatments and fumigants currently used for remedial decay control. The chemicals were compared on the basis of the estimated dosage of each required to eliminate at least 90% of the test fungus from the wood (ED-90).

RESULTS AND DISCUSSION

Effectiveness of the assay for known fumigants

The previously tested soil fumigants all reduced fungal survival in the small block tests, although the levels of control varied (Fig. 1). Chloropicrin produced the most effective control, followed by MIT, Vorlex[®], Vapam[®], and Shell DD[®]. With the exception of Shell DD[®], which has not been field-tested in wood, the results closely reflect the field performance of these chemicals (Helsing et al. 1984) and indicate that the small block test can be an effective predictor of field performance of fumigants for wood.



FIG. 1. Ability of selected dosages of chloropicrin, methylisothiocyanate (MIT), Vorlex[®], Vapam[®], and Shell DD[®] to inhibit *Poria carbonica* in a small block test on Douglas-fir heartwood.

Effect of treatment variables on test performance

Block size was found to strongly influence the performance of Vapam[®] and pure NaMDC. Vapam[®] produced 92%, 82%, and 17% inhibition of *Poria carbonica* in blocks 10, 20, and 30 cm long, respectively. NaMDC produced 75%, 0%, and 0% fungal control in blocks of the same size. Using blocks 10 cm long and of a uniform size appears to yield the most reliable prediction of a chemical's field performance in short-term tests.

It is not surprising that Vapam[®], which contains 67.3% water, performed better than the dry NaMDC, since the movement of the latter's decomposition products depends heavily on the presence of moisture in the wood. Furthermore, there is evidence that the moisture in Vapam[®] is necessary for decomposition of this chemical into more fungitoxic compounds.

Length of fungal incubation influenced sensitivity of the fungus to the chemical. Blocks incubated in the presence of the test fungus for 2, 3, 4, and 20 weeks prior to treatment with Vapam[®] or chloropicrin indicated that chemical sensitivity, as measured by the estimated dosage (in mg per block) required to kill 50% of the test fungus (ED-50), decreased between 2 and 4 weeks of incubation but appeared to change little between 4 and 20 weeks of incubation (Fig. 2). As the test fungus grows through the wood, older hyphae begin to form thick-walled chlamydospores that can resist desiccation and may thus be more tolerant of toxicants (Przybylowicz 1985). Apparently the fungi in the blocks that were incubated for 2 or 3 weeks had not formed enough chlamydospores to permit fungal survival at lower



FIG. 2. Susceptibility of *Poria carbonica* in Douglas-fir heartwood to Vapam[®] and chloropicrin at various times after inoculation of the wood with the decay fungus.

chemical dosages. Blocks should be incubated for a minimum of 4 weeks before chemical application to insure that the results accurately reflect chemical performance against mature fungal hyphae.

Duration of chemical exposure was strongly correlated with chemical performance in small block tests (Table 1). Two weeks after the chemicals were applied, all of them except Vapam[®] had inhibited the test fungus at levels exceeding 80%. Exposure for 4 weeks resulted in nearly complete inhibition by all six of the test chemicals. Although these results indicate that longer periods of exposure will provide a more complete characterization of a particular formulation, shorter periods can be used to predict trends when evaluating variables, such as dosage, temperature, and moisture content, of modified formulations of previously tested chemicals.

The effect of chemical exposure time on the performance of less volatile fungicides varied (Table 2). Several water-soluble compounds, including Hollow Heart, $OPC \times 906$, $OPC \times 968$, and FCAP, produced complete fungal inhibition 2 or 6 months after treatment; however, control 1 month after treatment ranged between 58% and 100%. These formulations have one or more water-soluble components that must have moisture in order to migrate through the wood. Since fungal decay substantially reduces the strength of the affected wood, rapid control is necessary. Consequently, the relatively slow rate at which these less volatile chemicals migrate makes them less desirable for control of internal decay. They may be useful for providing longer-term protection against reinvasion. Two other water-soluble compounds, ammoniacal copper arsenate (ACA) and chromated copper arsenate (CCA), which are both used as initial treatments for wood products intended for use in adverse environments, produced less effective control than the four mentioned above. ACA, which is strongly bound to wood, produced

Fumigant	Concentration (mg/block)	Inhibition of P. carbonica	
		Sealed	Unsealed
Chloropicrin	7	100	85
Vorlex [®]	25	90	47
Allyl alcohol	31.4	90	47
MIT	50.8	100	30
Vapam [®]	150	94	44

TABLE 3. Fumigant effectiveness in Douglas-fir heartwood blocks exposed in sealed and unsealed chambers after treatment.

some fungal inhibition 1, 2, and 6 months after application, but never eliminated the fungus. The ammonia used to solubilize this formulation may have provided some of the control. CCA, which is also strongly bound to wood, produced no fungal inhibition even 6 months after treatment. Neither of these formulations appears to control established decay fungi.

None of the oil-borne compounds except pentachlorophenol had any effect on fungal survival even 6 months after treatment. These compounds are apparently unable to migrate through the wood and thus would not perform well as remedial decay control agents. The performance of pentachlorophenol in this test is puzzling, since this compound has very low water solubility and should not have been able to migrate. Penta is a highly effective fungicide, and it is possible that minute amounts volatilized over the test period. However, it could not be counted on for decay control at long distances from the application point.

Two potentially useful, slow-acting fumigants, Mylone[®] (Dazomet[®]) and ZnMDC, produced more effective fungal control after longer exposure periods. These chemicals are crystalline solids that slowly decompose to produce MIT and other fungitoxic compounds. The rate of decomposition is so slow that these chemicals would be ineffective in short-term tests, but they perform well in tests of longer duration. Mylone[®] has performed well in one field test (Eslyn and Highley 1985). These results indicate the need to include longer period of evaluation when testing new formulations.

Finally, the degree of aeration also exerted an influence in the small block tests. Of the five fumigants tested, all produced markedly greater fungal inhibition when the treated blocks were exposed to the chemicals in sealed chambers (Table 3). While most of the currently registered fumigants have reasonably strong wood interactions that bind significant quantities of chemical (Goodell 1983; Zahora and Corden 1985), minute amounts continuously escape (S. Lebow and J. J. Morrell unpublished). Fumigant vapors accumulate in closed containers, retarding further loss from the wood. This accumulation markedly enhances fungal control; however, a closed container is not typical of normal wood conditions. Thus, blocks should be treated with sufficient aeration to prevent chemical build-up. Conversely, care must be taken to insure that excessive aeration does not make the blocks drier during treatment than they would be under conditions of normal use.

On the basis of its simplicity and apparent reproducibility, the small block test was used to evaluate the ability of 28 wood-preserving chemicals to eliminate the test fungus (Table 4). Fifteen of the test chemicals were reasonably toxic to the fungus over the 1-week treatment period (Table 5). While several other chemicals

Common or trade name	Source	Active ingredients	
Allyl alcohol	Eastman Kodak	allyl alcohol	
Allyl isothiocyanate	Eastman Kodak	allyl isothiocyanate	
Bunema [®]	Buckman Lab., Inc.	potassium n-hydroxymethyl-n-methyl dithio carbamate	
Captan®	Chevron Chem. Co.	N-trichloromethylmercapto 4-cyclo-hexene- 1,2-dicarboximid (75%)	
ABF	Osmose Wood Pres.	ammonium bifluoride	
ACA	J. H. Baxter & Co.	ammoniacal copper arsenate	
Chloropicrin	Dow Chemical Co.	trichloronitromethane	
Chlorothalonil®	Diamond Shamrock	tetrachloroisophtalonitrate	
Creosote	Koppers Co.	coal tar distillate	
CCA	Osmose Wood Pres.	chromated copper arsenate	
FCAP	Osmose	Na bichromate (34.3%), Na fluoride (29%), di- sodium arsenate (23.8%), 2,4 dinitrophenol (8%)	
Formaldehyde	Eastman Kodak	formaldehyde (37%)	
Hollow Heart®	Osmose	Na borate, ABF, arsenic acid, sodium bichro- mate	
Maneb	Rohm & Hass	manganese ethylene-bis dithiocarbamate (70%)	
MIT	Nor-Am Chem. Co.	methylisothiocyanate (95%)	
Mylone [®] (Dazomet [®])	Union Carbide	3,5-dimethyltetrahydro-1,3,5,2H-thiadiazine- 2-thione	
NaMDC		Na-\eta-methyldithiocarbamate	
NH₄OH	J. T. Baker Co.	ammonium hydroxide	
$(NH_4)_2S_3$		ammonium polysulfide	
OPC×906	Osmose	40% creosote with 5% penta, 46% Na fluo- ride + arsenic trioxide, 14% Na bichromate + dinitrophenol	
OPC×968	Osmose	same as OPC×906 without aresnic	
Penta	Chapman Chem.	pentachlorophenol (40%)	
Penta	Osmose	pentachlorophenol (5%)	
Shell DD®	Shell Chem.	chlorinated C-3 hydrocarbons	
Vapam [®]	Stauffer Chem.	32.7% NaMDC	
Vorlex [®]	Nor-Am Chem.	20% MIT, 80% Shell DD®	
Vorlex 201®	Nor-Am Chem.	17% MIT, 68% Shell DD [®] , 15% chloropicrin	
ZnMDC	Nor-Am Chem.	Zn-η-methyldithiocarbamate	

TABLE 4. Fungicides tested against Poria carbonica in Douglas-fir heartwood.

also produced some fungal control, they required far more time to perform and thus do not appear to be practical for remedial control of decay.

The fifteen most effective chemicals tested had ED-90 concentrations ranging from 28 mg per block for chloropicrin to over 1,000 mg per block for Mylone[®]. These results provide relative estimates of fumigant effectiveness that, with a few exceptions, have paralleled field performance. In field tests, chloropicrin has proved to be the most effective fumigant, followed closely by MIT and Vorlex[®] (Helsing et al. 1984). These tests showed that Vapam[®], which had the highest ED-90 of the four chemicals evaluated, protected wood for the shortest time. In other tests, ABF has eliminated established decay fungi and continued to protect Douglas-fir piling from decay for 6 years (Helsing et al. 1986). ABF differs from the other compounds tested in that it is water-soluble and has a relatively simple fungitoxic component. As moisture comes into contact with ABF, toxic hydrogen fluoride

Fumigant	Active ingredient	Diluent	ED-90 concentration (mg/block)
Chloropicrin	99	acetone	28
Vorlex 201®	100	acetone	79
Allyl alcohol	98	acetone	105
MIT	95	acetone	120
ABF	96	dry packed	125
Allyl isothiocyanate	99	acetone	130
NH₄OH	29	water	175
Volrex [®]	100	acetone	355
Vapam [®]	32.7	water	435
$(NH_4)_2S_3$	65	ethanol	565
Shell DD®	100	acetone	925
Formaldehyde	37	water	>600
Bunema®	40	water	>750
ZnMDC	99.3	water	>750
Mylone [®]	95	water	>1,000

 TABLE 5. Relative toxicity of fumigants to Poria carbonica in Douglas-fir heartwood after 1 week of exposure.

is released and moves through the wood, eliminating any decay fungi present. Fluoride is highly mobile and may not remain in the wood for as long as the other chemicals tested.

One formulation, allyl alcohol, provided excellent protection in laboratory tests but had little or no effect on decay fungi in field tests (Helsing et al. 1984). Its failure to perform in the field is perplexing, but may have occurred because an allyl alcohol formulation different from that used in the laboratory was substituted in the field tests.

CONCLUSIONS

The small block test provides a simple, rapid, and accurate method for assessing the effectiveness of volatile chemicals that show potential for controlling decay in wood. The procedures are sufficiently flexible to achieve satisfactory early indications of the performance of chemicals with a wide variety of properties. The procedures can also be applied to other wood or fungal species not tested here (Morrell et al. 1986). Furthermore, additional samples can be removed from the test blocks for solvent extraction and chemical analysis, usually with gas chromatography, to determine the levels of fungitoxic compounds present. This procedure can determine the quantity of chemical required to effect a particular level of fungal control, which information can then be used to develop systems for delivering the chemical to the wood in the most effective fashion. Thus, the small block test can be used to develop new or improved formulations while eliminating the need to field-test a wide array of less effective chemicals.

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ERRATUM

Between the galley stage and the final printing of the article *The Effects of CCA Preservative Treatment* and *Redrying on the Bending Properties of 2 \times 6 Southern Pine Lumber*, by J. E. Winandy and R. S. Boone in Volume 20, Number 3, several words were dropped from the abstract. The correct abstract should read as follows:

ABSTRACT

Southern pine dimension lumber (commercially graded No. 2 loblolly pine $2 \times 6s$) was treated with chromated copper arsenate (CCA) preservative (0.4 or 0.6 pcf) and then air-dried or kiln-dried (160, 190, or 240 F). CCA treatment significantly reduced average bending strength, but no discernible differences were found between controls and CCA-treated groups in the extreme lower portions (<10th percentile) of the bending-strength distributions. When these same specimens were then considered solely on the basis of strength-reducing characteristics, there were obvious differences in how the CCA treatments and subsequent redrying affected these various strength-ratio grades of 2×6 lumber; higher grades appeared to be less affected than lower grades. Similar to the trend shown when commercially graded, the middle and upper portion of each strength-ratio grade bending-strength distribution was affected more than the lower portion. In kiln drying the lumber after treatment, drying at 240 F affected a broader range of the bending-strength distribution than did drying at 160 F. The broadened range of significant effects noted after high-temperature redrying indicates that posttreatment kiln-drying temperatures higher than 190 F should be avoided.

The effects of CCA treatment and redrying were highly interactive with strength-ratio grade and the presence or absence of pith. CCA treatment reduced the strength of lumber containing pith and having a strength ratio of <0.65 to a greater extent than pith-free lumber of any strength-ratio grade. Lumber having a strength ratio of ≥ 0.65 and containing pith was not affected by CCA treatment. The magnitude of this pith-related interaction demands recognition.

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