# A LABORATORY SOIL-CONTACT DECAY TEST: AN ACCELERATED METHOD TO DETERMINE DURABILITY OF TREATED WOOD SHAKES

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ABSTRACT

In this study, cross sections of preservative-treated shakes were evaluated in soil-contact decay tests using *Gleophyllum trabeum.* The number of preservatives by species combinations used allowed evaluation of procedural aspects of this method. We conclude that the soil-contact decay test is a good tool for an accelerated evaluation of the durability of treated shakes. Experimental designs need to be structured so that each wood species can be analyzed separately. Interpretation of results requires consideration of both the distribution and median or mean of weight loss data for each treatment. Levels of retention should be well dispersed so that the effects of retention upon distribution of weight loss data for individual treatments can be determined. Selection of a sufficient sample size to represent the variation within the population of each wood species by treatment combination is judged to be more important than duration of the incubation period in excess of 12 weeks. Stated differently, a rigorous experimental design permits discrimination of major differences without a need to achieve a weight loss of 50% in untreated materials. Wood species utilized in this study were western hemlock (*Tsuga heterophylla* (Raf.) Sarg.), Pacific silver fir (*Abies amabilis* Dougl. ex Forbes), grand fir (*Abies grandis* Dougl. ex D. Don Lindl.), western white pine (*Pinus monticola* Dougl. ex D. Don), southern pine spp., and red alder (*Alnus rubra* Bong.).

*Keywords:* preservative, method, shake, roofing, hemlock, pine, alder, wood decay, durability

## INTRODUCTION

New composites and alternative species of solid woods are under consideration as source materials for wood roofing. When preservative treatment of these new materials is required, the estimation of long-term durability of the resultant product poses a challenge. We are exploring the potential of the following as source material for the manufacture of shakes and shingles: western hemlock (*Tsuga heterophylla* (Ref. Sarg), Pacific silver fir (*Abies amabilis* Dougl. ex Forbes), grand fir (*Abies grandis* (Dougl. ex D. Don) Lindl., western white pine (*Pinus monticola* Dougl. ex D. Don), and red alder (*Alnus rubra* Bong.). All these woods lack natural durability; therefore, treatment with preservatives is required to achieve an acceptable service life.

A soil-contact decay test with entire cross

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sections of treated shakes is one evaluation method that we have used. Entire cross sections of treated shakes more truly represent the variation in wood quality and preservative distribution within the shake and the proportionate mix of heartwood and sapwood that occurs in the actual product than do small, uniform units of either sapwood or heartwood, which are used in standard methodologies (ASTM 1986a, b—D1413, ASTM D2017) for estimating decay resistance.

A laboratory soil-contact test with cross sections of shakes is more challenging than a 5-year exposure in an open field (De Groot 1994a). Thus, evidence of resistance to decay in this laboratory test should be indicative of long-term potential in adverse environments. However, questions remain as to whether: (1) the severity of the challenge is too great for the test either to have predictive value or to discriminate among treatments; (2) the length of the challenge (incubation period) should be referenced to a minimum weight loss in untreated controls or defined as a fixed time period; and (3) different incubation periods will yield different relative results.

In preparing numerous wood species by preservative treatment combinations for outdoor exposure (De Groot 1994c), we included additional shakes in the charges for use in laboratory decay experiments. To present results from those decay experiments, we address the utility of this methodology as an accelerated means to predict long-term durability of new roofing materials.

## MATERIALS AND METHODS

Laboratory-scale, soil-contact decay tests were used. The test methodology was a modification of the standard ASTM 1413 (ASTM 1986a) soil-block procedure in that the bottles were incubated on the side to accommodate the length of the cross sections placed in the decay jars. This approach was previously used by Wilcox (1980); but in this work, entire cross sections of shakes, rather than subunits, were assayed. Incubation times also varied and are described for each respective experiment. Other procedural steps were those described in the ASTM standard method. The amount of wood lost to decay in each specimen during the course of the test is expressed as a percentage of original weight.

Two decay experiments were conducted with cross sections of treated shakes to gain a preliminary estimate of likely long-term durability of representative treatments and species. The first experiment was continued until an average weight loss of 50% occurred in untreated reference blocks of at least one species in the test; this required 16 weeks of incubation. The second experiment was conducted within 12 weeks without regard to the amount of weight lost in untreated controls or references. Additional shakes were treated with chromated copper arsenate (CCA) and didecyldimethylammonium chloride plus 3-iodo-2-propynyl butyl carbamate (DDAC/IPBC) to investigate a possible relationship between concentration of active ingredient in the treating solution and variability of resistance to decay within the population of treated shakes.

Analysis of data revealed interactive effects between wood species and chemical treatments within individual experiments. Also, weight loss data were not normally distributed for some wood/treatment combinations. Furthermore, data sets for the different treatments within a given species had significantly different variances. Therefore, within each experiment, comparisons of chemical treatments were made by wood species, individually. Nonparametric statistical analyses were employed using commercial software (SAS 1994).

Some data sets are displayed using box plots (Velleman 1989), as illustrated in Fig. 1. The shaded regions for each plot enable comparison of medians at a 5% level of significance with a modified 95% confidence interval for the median. If the shaded regions do not overlap, the corresponding medians are significantly different at the 5% level. Box plots can also be used to assess the possible normality of the data set by looking for the symmetry and equality of mean and median that characterize a normal distribution.

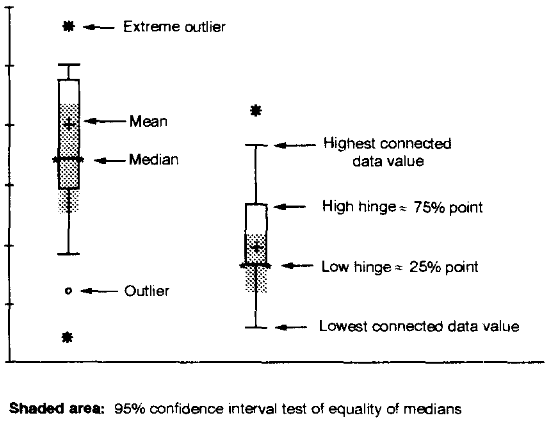


FIG. 1. Components of a boxplot. Boxplots are used to present graphic summarizations of results at each observation.

### *Shakes*

Tapersawn shakes 11/16 to 13/16 in. (17 to 21 mm) thick and either 18 or 24 in. (457 or 610 mm) were used with western hemlock, Pacific silver fir, western white pine, and grand fir. Western hemlock and Pacific silver fir shakes were cut from butt logs of old-growth trees that were harvested in several locations on the Puget Sound Peninsula in the state of Washington. Western white pine shakes were cut from insect-killed trees in Idaho (Govett et al. 1991). The grand fir shakes also came from Idaho, but live trees were harvested as source materials for those shakes. Red alder shakes, 18 in. (457 mm) long and 3/4 in. (19 mm) thick at the butt end, were cut from logs harvested from the Puget Sound Peninsula. Tapersawn, No. 1 grade southern pine shakes, 0.5 in. (13 mm) thick at the butt and 24 in. (610 mm) long, were obtained from Louisiana.

### *Preservatives*

Chromated copper arsenate (CCA) was used as a reference preservative. Two preservatives that contained a quaternary ammonium compound were included. Several different emulsions of copper and zinc naphthenate were also investigated (Table 1). Petroleum solutions containing 1% metal, then diluted in toluene, were used in preliminary and experimental treatments with oil-borne formulations of copper and zinc naphthenate. Paraffinic and naphthenic carriers with viscosities of approximately 40 SUS (100°F/37.8°C), a paraffinic petroleum distillate with a viscosity of about 65 SUS (100°F/37.8°C), and a naphthenic distillate with a viscosity of about 108 SUS (100°F/37.8°C) were used. The flashpoints for these solvents are probably greater than that for most AWPA P9 Type A solvents currently being used commercially (S. Grove, personal communication, 1993).

### *Treatments*

The full-cell process with an initial vacuum of 30 or 60 min and a press of 120 min at 125 lb/in$^2$. (861.8 kPa) was used with all but three charges. With these charges (239, 241, 243), an alternating pressure cycle (Bergervoet 1984; Rudman et al. 1963) was used to treat with CCA. Shakes that were treated by the full-cell process were dried to 12% moisture content (MC) or less prior to treatment.

At the time of treating, preservative uptake was determined on a weight gain basis. Actual retention levels were subsequently determined by chemical analysis. When the shakes had dried, samples from the entire cross section of the shakes, 6.0 to 7.0 in. (152 to 178 mm) from the butt end, were removed, ground in a Wiley mill to pass a 30-mesh screen, and analyzed using either atomic absorption spectroscopy for the metals or liquid chromatography for the organics.

Retention levels determined by chemical analysis were reported as w/w% and on a weight/volumetric basis. Wood density was accepted as published in AWPA Standard A12 (AWPA 1989) or computed using specific gravity as published in Table 4-2 of the *Wood Handbook* (Forest Products Laboratory 1987).

For metallic-naphthenate and-octoate preservatives containing copper or zinc, computations of active ingredient in the treating solutions and within wood were made on the basis of percentage (w/w) of metal ion in either

TABLE 1. *Preservatives evaluated in decay tests with cross sections of treated shakes.*

| Preservative | Code[a] | Description |
|---|---|---|
| An ammoniacal copper/quaternary ammonium compound. (CuO/ODAC) | 2 | An ammoniacal, waterborne preservative. The quaternary ammonium component is octyldecyldimethylammonium chloride. The CuO:Quat ratio is 1:1. |
| Chromated copper arsenate, Type C (CCA) | 1 | An industrial reference. An acidic waterborne preservative. |
| Copper naphthenate (CuN) | 6 | A 40/60 w/w mixture of copper naphthenate (8% copper) and polyoxethylen sorbitol tetraborate, diluted with water to achieve desired concentration of copper in treating solution. |
| Copper naphthenate (CuN) | 8 | Proprietary waterborne emulsion, with water repellent |
| Copper naphthenate (CuN) | 17 | Solution in petroleum carrier B (naphthenic oil) |
| Copper naphthenate (CuN) | 23 | Solution in petroleum carrier D (naphthenic oil) |
| Copper octoate (CuOct) | 13 | Solution in petroleum carrier A (paraffinic oil) |
| Copper octoate (CuOct) | 25 | Solution in petroleum carrier D (naphthenic oil) |
| Didecyldimethylammonium chloride plus 3-iodo-2-propynyl butyl carbamate. (DDAC/IPBC) | 3 | A patented aqueous mixture of DDAC and IPBC in a ration of 8.5 to 1.[b] |
| Zinc-copper mixture | 12 | Proprietary combination of waterborne emulsions of zinc and copper naphthenates, with water repellent. |
| Zinc naphthenate (ZnN) | 10 | 75/25 w/w mixture of zinc naphthenate (8% zinc) and polyoxethylen sorbitol tetraborate, diluted with water to achieve desired concentration of zinc in the treating solution. |
| Zinc naphthenate (ZnN) | 11 | Proprietary waterborne emulsion, with water repellent |
| Zinc naphthenate (ZnN) | 18 | Solution in petroleum carrier A (paraffinic oil) |
| Zinc naphthenate (ZnN) | 20 | Solution in petroleum carrier E (paraffinic oil) |

[a] The same code number is used for each preservative in a series of experiments. Code numbers are included here, out of sequence, to aid the reader in cross referencing and comparing results from this investigation with results reported for other studies. Detailed description of petroleum carriers are given in DeGroot (1994b).

[b] Ward 1990.

the solution or treated product. For CCA, computations of treating solution strength or retention levels in treated wood were on an "oxide" basis. Computed concentrations or retention levels of other preservatives reflect w/w% of the active ingredient either in the solution or treated wood product.

### *Decay experiments*

Experiment 1: In this experiment (Tables 2, 3), 10 shakes/charge were randomly selected. These shakes were of various widths. The entire cross section, 6.0 to 6.5 in. (152 to 165 mm) from the butt end, was cut from each of these 10 shakes. These entire cross sections were exposed to attack by *Gleophyllum trabeum* (Pers. ex. Fr.) Murr. [Madison 617, ATCC 11539], following ASTM D1413 standard method of testing wood preservatives by laboratory soil-block cultures (ASTM 1986a). Instead of 8-oz (236-cm$^3$) jars, 1-qt (0.95-L) jars were used; these jars were laid horizontally. Soil (150 g) was loaded into each jar. This was sufficient to cover the entire bottom side of the jar. Elongated feeder strips were placed on top of the soil, and water was added to bring the soil to 130% MC. Processing of the decay chambers (jars) was in accordance with ASTM D1413. Entire cross sections from one or two shakes, treated with the same preservative and retention, were incubated in each jar. Whenever the cross section was too long to fit into the jar, it was broken and both halves were inserted into one jar. Cross sections were exposed to decay fungi in the jars for 16 weeks.

For chemical analysis, three, 1/8-in.-thick (3.2-mm) slices were also cut from the entire

TABLE 2. *Percentage weight loss in cross sections of preservative-treated shakes in 16-week, soil-contact laboratory decay test with* G. trabeum *(Experiment 1).*

| Species and preservative | Code[b] | Charge | Retention[a] analyzed w/w% | Retention[a] analyzed lb/ft$^3$ | Median weight loss (%)[c] | Tukey's studentized range test[d] |
|---|---|---|---|---|---|---|
| Western hemlock | | | | | | |
| ZnN | 11 | 322 | 0.25 | 0.07 | 60.2 | A |
| ZnN | 10 | 385 | 0.16 | 0.04 | 56.9 | A |
| CuOct | 25 | 634 | 0.09 | 0.02 | 53.7 | A B |
| ZnN | 10 | 381 | 0.22 | 0.06 | 51.8 | A B |
| None | — | — | — | — | 46.4 | B |
| ZnCu | 12 | 193 | 0.13 | 0.03 | 29.4 | C |
| CuN | 6 | 397 | 0.15 | 0.04 | 22.9 | C D |
| CuN | 8 | 453 | 0.10 | 0.03 | 22.8 | C D |
| CCA | 1 | 445 | 1.27 | 0.33 | 16.2 | D E |
| CCA | 1 | 202 | 0.97 | 0.27 | 15.8 | D E |
| CuO/ODAC | 2 | 470 | 0.95 | 0.25 | 14.7 | D E |
| CuO/ODAC | 2 | 447 | 1.36 | 0.35 | 9.3 | E F |
| DDAC/IPBC | 3 | 461 | 0.98 | 0.26 | 2.1 | F |
| DDAC/IPBC | 3 | 359 | 0.94 | 0.24 | 1.6 | F |
| DDAC/IPBC | | 361 | 0.95 | 0.25 | 1.2 | F |
| Pacific silver fir | | | | | | |
| ZnN | 20 | 631 | 0.15 | 0.04 | 54.9 | A |
| None | — | — | — | — | 50.2 | A B |
| CuN | 23 | 628 | 0.13 | 0.03 | 42.2 | B C |
| CuOct | 13 | 585 | 0.11 | 0.05 | 39.0 | C |
| CuN | 17 | 622 | 0.16 | 0.04 | 25.6 | C |
| Western white pine | | | | | | |
| ZnN | 18 | 593 | 0.20 | 0.04 | 45.7 | A |
| None | — | — | — | — | 43.7 | A |
| CuOct | 13 | 585 | 0.11 | 0.03 | 43.7 | A B |
| CuN | 23 | 628 | 0.15 | 0.03 | 31.0 | B C |
| CuOct | 25 | 634 | 0.14 | 0.03 | 19.2 | |
| CCA | 1 | 574 | 1.04 | 0.23 | 8.8 | D |
| DDAC/IPBC | 3 | 564 | 2.30 | 0.51 | 1.8 | E |
| DDAC/IPBC | 3 | 563 | 1.74 | 0.40 | 1.7 | E |
| DDAC/IPBC | 3 | 569 | 3.82 | 0.83 | 1.6 | E |
| Red alder | | | | | | |
| None | — | — | — | — | | |
| DDAC/IPBC | 3 | 626 | 1.92 | 0.44 | 3.1 | B |
| CCA | 1 | 623 | 1.04 | 0.43 | 2.8 | B |

[a] For metallic-naphthenate and -octoate preservatives containing copper or zinc, computations of preservative retention were made on the basis of % (w/w) metal ion/wood in treated product. For CCA, computation of retention in treated wood was on an "oxide" basis. Computed retention levels of other preservatives reflect w/w% of the total active ingredient of the respective preservative in the treated wood. (1 lb/ft$^3$ = 16 kg/m$^3$)
[b] See Table 1 for explanation of code numbers.
[c] Median of 10 observations.
[d] Median percentage weight losses with the same letter were not significantly different.

cross section of each shake. These were cut in consecutive order, starting 6 in. (152 mm) below the butt and moving towards the butt end. All slices from each of the 10 shakes were combined in one lot for each treatment, retention, and species combination and processed for chemical analysis.

Experiment 2: The second decay experiment (Table 4) used shakes that had been cut to a constant width of 4.5 in. (114 mm) prior to

TABLE 3. *Performance of cross sections of preservative-treated shakes in 16-week laboratory decay tests (Experiment 1).*

| Species[b] | Preservative | Code[c] | Charge | Retention[a] Target lb/ft³ | Analyzed w/w% | Analyzed lb/ft³ | Weight loss (%)[d] |
|---|---|---|---|---|---|---|---|
| PSF | Control | — | — | 0.00 | — | — | 50.67 (13.11) |
| WH | Control | — | — | 0.00 | — | — | 44.29 (6.31) |
| WP | Control | — | — | 0.00 | — | — | 43.11 (1.11) |
| WP | CCA | 2 | 574 | 0.30 | 1.04 | 0.23 | 8.82 (4.84) |
| ALD | CCA | 1 | 623 | 0.60 | 1.85 | 0.43 | 2.80 (0.68) |
| WH | CCA | 1 | 202 | 0.28 | 0.97 | 0.27 | 15.71 (4.26) |
| WH | CCA | 1 | 445 | 0.33 | 1.27 | 0.33 | 16.79 (5.42) |
| PSF | CuN | 17 | 622 | 0.04 | 0.16 | 0.04 | 26.20 (7.12) |
| PSF | CuN | 23 | 628 | 0.04 | 0.13 | 0.03 | 39.59 (12.22) |
| WH | CuN | 6 | 397 | 0.04 | 0.15 | 0.04 | 24.23 (11.57) |
| WH | CuN | 8 | 453 | 0.04 | 0.10 | 0.03 | 21.81 (10.08) |
| WP | CuN | 23 | 628 | 0.04 | 0.15 | 0.03 | 30.15 (5.57) |
| PSF | CuOct | 13 | 585 | 0.04 | 0.11 | 0.05 | 34.34 (12.43) |
| WP | CuOct | 13 | 585 | 0.04 | 0.11 | 0.03 | 39.66 (10.03) |
| WH | CuOct | 25 | 634 | 0.04 | 0.09 | 0.02 | 53.24 (4.72) |
| WP | CuOct | 25 | 634 | 0.04 | 0.14 | 0.03 | 20.90 (12.17) |
| WH | CuO/ODAC | 2 | 470 | 0.30 | 0.95 | 0.25 | 14.56 (4.42) |
| WH | CuO/ODAC | 2 | 477 | 0.45 | 1.36 | 0.35 | 9.33 (2.41) |
| ALD | DDAC/IPBC | 3 | 626 | 0.66 | 1.92 | 0.44 | 3.09 (0.41) |
| WH | DDAC/IPBC | 3 | 359 | 0.33 | 0.94 | 0.24 | 1.71 (0.79) |
| WH | DDAc/IPBC | 3 | 461 | 0.33 | 0.98 | 0.26 | 2.65 (1.81) |
| WH | DDAC/IPBC | 3 | 361 | 0.49 | 0.95 | 0.25 | 1.33 (0.43) |
| WP | DDAC/IPBC | 3 | 564 | 0.33 | 2.30 | 0.51 | 1.88 (0.47) |
| WP | DDAC/IPBC | 3 | 569 | 0.49 | 3.82 | 0.83 | 1.60 (0.19) |
| WP | DDAC/IPBC | 3 | 563 | 0.66 | 1.74 | 0.40 | 1.77 (0.27) |
| WH | ZnCu | 12 | 193 | 0.04 | 0.13 | 0.03 | 27.92 (10.46) |
| PSF | ZnN | 20 | 631 | 0.06 | 0.15 | 0.04 | 55.35 (5.58) |
| WH | ZnN | 11 | 322 | 0.09 | 0.25 | 0.07 | 54.97 (5.91) |
| WH | ZnN | 10 | 381 | 0.12 | 0.22 | 0.06 | 50.91 (5.58) |
| WH | ZnN | 10 | 385 | 0.06 | 0.16 | 0.04 | 56.63 (3.40) |
| WP | ZnN | 18 | 593 | 0.06 | 0.20 | 0.04 | 48.16 (7.99) |

[a] For metallic-naphthenate and -octoate preservatives containing copper or zinc, computations of preservative retention are made on the basis of % (w/w) metal ion/wood in treated product. For CCA, computation of retention in treated wood is on an "oxide" basis. Computed retention levels of other preservatives reflect w/w% of the total active ingredient of the respective preservative in the treated wood. (1 lb/ft³ = 16 kg/m³)
[b] ADL = red alder; PSF = Pacific silver fir; WH = western hemlock; WP = western white pine.
[c] See Table 1 for explanation of code numbers.
[d] Average of 10 observations; standard deviation is in parentheses.

treatment. Entire cross sections were cut from treated shakes for either decay tests or chemical analysis as described in Experiment 1.

Cross sections were exposed to attack by *Gleophyllum trabeum* (Pers. ex. Fr.) curr. [Madison 617, /ATCC No. 11539], following the principles of the ASTM D1413 standard method of testing wood preservatives by laboratory soil-block cultures (ASTM 1986a), except that the soil-block jars were incubated horizontally. Eight-ounce (236 cc³) jars were used. Soil (65 g) at 25% MC (an amount of soil sufficient to cover the entire bottom side of the jar) was loaded into each jar, elongated feeder strips were placed on top of the soil, and water was added to bring the soil up to 130% MC. Processing of the decay chambers (jars) was in accordance with ASTM D 1413. One entire

TABLE 4. *Percentage weight loss in cross sections of preservative-treated shakes in 12-week laboratory soil-contact decay test with* G. trabeum *(Experiment 2).*

| Species and preservative | Code[b] | Charge | Retention[a] analyzed w/w% | Retention[a] analyzed lb/ft³ | Median weight loss (%)[c] | Tukey's studentized range test[d] |
|---|---|---|---|---|---|---|
| Western hemlock | | | | | | |
| ZnN | 12 | 321 | 0.13 | 0.03 | 45.9 | A |
| ZnN | 11 | 323 | 0.32 | 0.09 | 47.0 | A |
| ZnN | 10 | 386 | 0.02 | 0.06 | 45.3 | |
| ZnN | 10 | 385 | 0.16 | 0.04 | 35.8 | A B |
| None | — | — | — | — | 33.2 | A B |
| ZnCu | 12 | 480 | 0.25 | 0.06 | 33.5 | A B |
| CuN | 8 | 374 | 0.12 | 0.03 | 26.5 | A B C |
| ZnN | 10 | 381 | 0.35 | 0.09 | 30.5 | B C D |
| CuN | 8 | 369 | 0.20 | 0.05 | 18.3 | B C D E |
| CuN | 6 | 397 | 0.15 | 0.04 | 13.1 | B C D E F |
| ZnCu | 12 | 487 | 0.32 | 0.08 | 16.5 | C D E F G |
| ZnCu | 12 | 416 | 0.37 | 0.10 | 13.8 | C D E F G |
| CCA | 1 | 273 | 0.01 | 0.03 | 12.6 | D E F G H |
| CCA | 1 | 317 | 1.13 | 0.29 | 12.4 | E F G H |
| CuN | 6 | 399 | 0.17 | 0.04 | 9.3 | E F G H |
| CCA | 1 | 239 | 1.50 | 0.39 | 8.4 | E F G H |
| CCA | 1 | 445 | 1.27 | 0.33 | 8.1 | F G H I |
| CCA | 1 | 241 | 1.46 | 0.38 | 7.2 | G H I |
| CuO/ODAC | 2 | 477 | 1.36 | 0.35 | 6.4 | G H I J |
| CuN | 6 | 393 | 0.21 | 0.05 | 4.5 | H I J |
| CCA | 1 | 243 | 1.76 | 0.46 | 3.6 | I J |
| CuO/ODAC | 2 | 424 | 2.04 | 0.53 | 2.8 | J |
| CCA | 1 | 201 | 1.42 | 0.37 | 2.2 | J |
| Pacific silver fir | | | | | | |
| CuN | 15 | 590 | 0.12 | 0.03 | 43.7 | A |
| None | — | — | — | — | 41.2 | A |
| CuOct | 13 | 586 | 0.24 | 0.06 | 14.4 | B |
| CuOct | 13 | 583 | 0.31 | 0.08 | 12.0 | B |
| Grand fir | | | | | | |
| CuN | 15 | 628 | 0.13 | 0.03 | 18.2 | |

[a] For metallic-naphthenate and -octoate preservatives containing copper or zinc, computations of preservative retention were made on the basis of % (w/w) metal ion/wood in treated product. For CCA, computation of retention in treated wood was on an "oxide" basis. Computed retention levels of other preservatives reflect w/w% of the total active ingredient of the respective preservative in the treated wood. (1 lb/ft$^3$ = 16 kg/m$^3$)
[b] See Table 1 for explanation of code numbers.
[c] Median of 10 observations.
[d] Median percentage weight losses with the same letter were not significantly different.

cross section from one shake was incubated in each jar. Cross sections were exposed to decay fungi in the jars for 12 weeks.

Experiment 3: The third experiment (Table 5, Figs. 2, 3) was designed to explore whether the variation in protection provided by a preservative treatment to a population of shakes was influenced by strength of the treating solution.

The performance of preservative-treated western white pine shakes was of particular interest because of prior concerns about uniformity of treatment in shakes of that species (De Groot 1994b). In the previous two experiments reported here, we also observed that populations of treated shakes sometimes involved individual shakes with substantial weight loss even though the median weight loss for the lot was low. In Experiment 3, shakes manufactured from western hemlock, Pacific silver fir, western white pine, and southern pine were treated with 1.0% or 0.75% CCA or 1.1%,

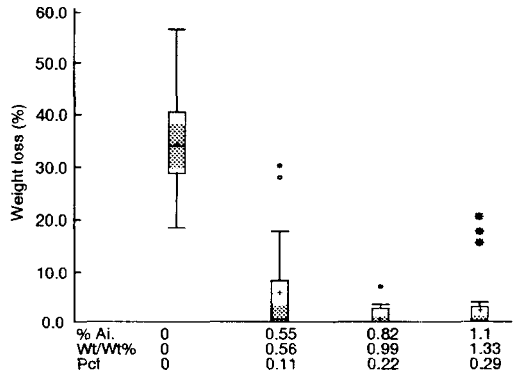


FIG. 2. Percentage weight loss in cross sections of preservative-treated western white pine shakes that were incubated 13 weeks in soil-contact tests with *G. trabeum.*

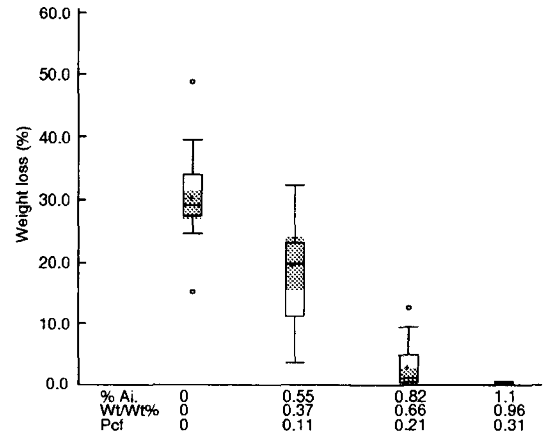


FIG. 3. Percentage weight loss in cross sections of preservative-treated southern pine shakes that were incubated 13 weeks in soil-contact tests with *G. trabeum.*

0.82%, or 0.55% DDAC/IPBC. Shakes from all species were cut to a width of 5 in. (127 mm) prior to treatment.

Twenty shakes of each species were treated simultaneously with each concentration of each preservative. Thus, processing conditions were comparable for all species within each retention by preservative combination.

After air-drying, cross sections were cut from treated shakes for decay tests and chemical analysis, as was done in Experiments 1 and 2. A cross section was taken 6 in. (152 mm) from the butt of each shake, and additional slices were taken adjacent to that position for chemical analysis. Untreated control shakes of each species were drawn from the same lot of shakes as were the treated shakes within each species and, except for southern pine, were cut to the same width. The southern pine control shakes were cut to a 5.25-in. (133-mm) width. Cross sections of shakes were incubated in soil-block decay jars for 13 weeks.

## RESULTS

A general pattern of relative performance of preservatives in the different wood species (Tables 2–5) remained the same, even though the incubation period and the volume of wood that was challenged with a decay fungus were different in the three experiments. The preservatives DDAC/IPBC and CuO/ODAC provided protection equivalent or somewhat better than comparable retention levels of the reference preservative CCA in the various wood species. With zinc naphthenate (ZnN) at a retention less than 0.09 lb/ft$^3$ (1.44 kg/m$^3$), the amount of decay that occurred was not significantly different or less than that of the untreated controls for the respective species. Results with the remaining preservatives were intermediate.

In Experiment 1, a surprising amount of weight loss was observed in western hemlock shakes that were treated with CCA to retention levels of 0.27 and 0.33 lb/ft$^3$ (4.32 and 5.28 kg/m$^3$) (Table 1). This could have represented a distribution-dependent phenomenon within the treated shakes or a reflection of the 16-week incubation. Chemical analysis of the treating solution revealed that the component balance of the concentrate was within the limits defined by AWPA Standard P5, as shown in Table 6.

For cross sections of shakes treated with either CCA or CuO/ODAC, the decayed tissue was not uniformly distributed across one surface or throughout the wood unit that was in

TABLE 5. *Percentage weight loss in cross sections of CCA- or DDAC/IPBC-treated shakes in 13-week laboratory soil-contact decay test with* G. trabeum *(Experiment 3).*

| Species and preservative | Code[b] | Active ingredient in treating solution (%) | Retention[a] analyzed w/w% | Retention[a] analyzed lb/ft³ | Median weight loss (%)[c] | Tukey's studentized range test[d] |
|---|---|---|---|---|---|---|
| Western hemlock | | | | | | |
| None | — | — | — | — | 32.1 | A |
| DDAC/IPBC | 3 | 0.55 | 0.47 | 0.12 | 2.6 | B |
| CCA | 1 | 0.75 | 1.05 | 0.27 | 2.8 | B |
| CCA | 1 | 1.00 | 1.30 | 0.34 | 2.5 | B |
| DDAC/IPBC | 3 | 0.82 | 0.88 | 0.23 | 0.8 | C |
| DDAC/IPBC | 3 | 1.10 | 1.03 | 0.27 | 0.9 | C |
| Pacific silver fir | | | | | | |
| None | — | — | — | — | 50.5 | A |
| CCA | 1 | 0.75 | 1.65 | 0.44 | 1.8 | B |
| CCA | 1 | 1.00 | 2.27 | 0.61 | 1.5 | B |
| Western white pine | | | | | | |
| None | — | — | — | — | 34.1 | A |
| CCA | 1 | 0.75 | 1.26 | 0.28 | 3.8 | B |
| CCA | 1 | 1.00 | 1.59 | 0.39 | 3.8 | B |
| DDAC/IPBC | 3 | 0.55 | 0.56 | 0.11 | 0.5 | B C |
| DDAC/IPBC | 3 | 1.10 | 1.33 | 0.29 | 0.2 | C |
| DDAC/IPBC | 3 | 0.82 | 0.99 | 0.22 | 0.1 | C |
| Southern pine | | | | | | |
| None | — | — | — | — | 29.3 | A |
| DDAC/IPBC | 3 | 0.55 | 0.37 | 0.11 | 19.8 | B |
| DDAC/IPBC | 3 | 0.82 | 0.66 | 0.21 | 0.9 | C |
| CCA | 1 | 0.75 | 0.89 | 0.28 | 1.4 | C |
| CCA | 1 | 1.00 | 1.19 | 0.39 | 0.7 | C |
| DDAC/IPBC | 3 | 1.10 | 0.96 | 0.31 | 0.1 | D |

[a] For CCA, computations of retention in treated wood are made on an "oxide" basis. Retention levels listed for DDAC/IPBC reflect total active ingredient. (1 lb/ft³ = 16 kg/m³)
[b] See Table 1 for explanation of code numbers.
[c] Median of 20 observations.
[d] Median percentage weight losses with the same letter are not significantly different.

the test. Rather, it appeared to be restricted to small pockets within the cross sections. For CCA, decay was evident on the surface of the section that contacted the feeder strip in the decay jar, but did not extend through the section except in the localized areas. With CuO/ODAC, less decay occurred on the surface of the cross sections that contacted the feeder strip than on the CCA-treated sections. Also, fewer pockets of decay penetrated the entire cross sections of CuO/ODAC-treated shakes. The bands of phenolic-saturated tissues in western hemlock shakes did not appear to be prone to decay.

TABLE 6. *Active ingredients in CCA, Type C.*

| Ingredient | Component balance (%) AWPA Standard | AWPA Minimum | AWPA Maximum | Our treating solution |
|---|---|---|---|---|
| Hexavalent chromium, as $CrO_3$ | 47.5 | 44.5 | 50.5 | 46.87 |
| Copper, as CuO | 18.5 | 17.0 | 21.0 | 18.05 |
| Arsenic, as $AS_2O_5$ | 34.0 | 30.0 | 38.0 | 35.07 |

In the shorter incubation periods of Experiments 2 and 3, less decay occurred in the western hemlock shakes treated at comparable retention levels of CCA. Still, there was a difference in amounts of decay observed in the two latter experiments in which the incubation period differed by only 1 week. Whether this represented source-dependent differences in the wood shakes or reflected some other variable was not determined. For each species in Experiment 3, results from the two retention levels of CCA were not significantly different.

After 12 weeks of incubation, the difference between the two formulations of waterborne copper naphthenate was most apparent at the higher retention levels. With both formulations, relatively less decay and less variation within the population of shakes evaluated at each retention were associated with the very minor increases in the weight percentage of active ingredient in the treated wood (Table 4, Fig. 4). The percent weight loss observed for each formulation at their respective highest retention levels (0.20 and 0.21 wt/wt%) was significantly different.

Alder shakes were well protected by CCA treatments at 0.43 lb/ft$^3$ and DDAC/IPBC at 0.44 lb/ft$^3$ (Tables 2, 3). Protection was uniform. There was no evidence of decay pockets within cross sections of the alder shakes. The efficacy of CCA treatments in preventing decay in alder in laboratory experiments was recently demonstrated by Mitchoff and Morrell (1988).

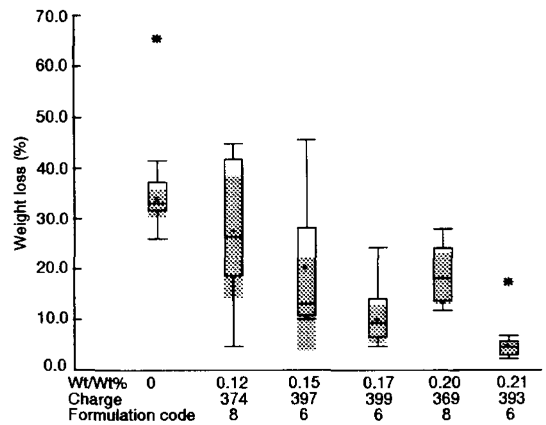


FIG. 4. Percentage weight loss in cross sections of western hemlock stakes that were treated with formulations of copper naphthenate and incubated 12 weeks in soil-contact decay tests with *G. trabeum*.

## DISCUSSION

The repetition of the same pattern of relative performance of preservative treatments in the different experiments indicates that duration of incubation beyond 12 weeks is not critical. Inclusion of an adequate number of replicates for each species by treatment combination to accurately reflect the natural variation of the product seems to be more important in the design of soil-contact tests involving cross sections of shakes than is incubation period.

Because of potential interactive effects between treatments and species, these tests should be designed so that each wood species can be analyzed separately. A treatment with an industrial reference and untreated control should be used with each species that is evaluated. Then, the data set for each species can be evaluated relative to performance of the control and the reference treatment or treatments. A lack of significant difference from the untreated control would not indicate good potential for protecting shakes exposed in adverse environments. Those treatments that show potential with one fungus could then be challenged with different wood-degrading organisms that would represent the spectrum of deteriogens that might be expected on the particular wood species. For example, white-rot decay fungi and wood-degrading ascomycetes should be included in an assay of hardwoods.

Interpretation of results requires consideration of both the distribution of results from each treatment and the mean or median of that treatment. A pattern of decreasing variation with increasing retention and efficacy of active ingredient was observed with several preservatives.

In Experiment 3 that focused on treatments with DDAC/IPBC (Table 5, Figs. 2, 3), the inverse relationship between retention and variation about median weight loss for both DDAC/IPBC and reference CCA treatments was most pronounced in western white pine (Fig. 2), less so in southern pine (Fig. 3), and minimal in western hemlock. For western white pine, southern pine, and western hemlock, the variation in observed weight losses decreased with increasing retention levels of both CCA and DDAC/IPBC, even though differences between CCA retention were not significant in any species used in that experiment. For western white pine, the highest retention of CCA (0.39 $lb/ft^3$) minimized variability in treatment. Minimization of the variation in weight losses was not accomplished in western white pine with the highest retention of DDAC/IPBC used (0.29 $lb/ft^3$), even though the median weight loss of the population of shakes with that treatment indicated excellent decay suppression (0.2% weight loss). For southern pine (Fig. 3), variation in weight loss was minimal at the highest retention levels of both preservatives.

This pattern of variation in laboratory decay tests may portend long-term potentials for onset of decay in shakes exposed in the field. After 6.5 years of exposure on experimental decks in southern Mississippi, small pockets of decay were observed on western white pine shakes that were treated with several preservatives. This pattern appears to be species-dependent as it was not observed in shakes of other wood species with comparable treatments.[2]

If there had been more replicate shakes within each treatment with CuN and a greater separation of retention levels in the CuN treatments, perhaps a more rigorous comparison of formulations and retention levels might have been possible in the 12-week experiment (Table 4). The distribution of weight loss data (Fig. 4) for the copper naphthenate treatments indicates that most retention levels were less than that which gave uniform protection. Reduction in variation of weight loss within the data set appears to begin at retention levels at or greater than w/w% of 0.20% copper. Data sets from the two treatments with a retention less than that level were characterized by substantial variation.

The results with copper naphthenate may, in part, reflect combined effects of formulation chemistries and the active ingredient. The efficacy of copper naphthenate against decay fungi is influenced by the type of petroleum carrier (solvent) in which it is dissolved (Duncan 1957). In the study reported here, formulations were either waterborne or were made with toluene-diluted petroleum carriers that had specifically been formulated to address current environmental requirements for volatile organic components. Thorough penetration of western hemlock shakes was achieved with both formulations of waterborne copper naphthenate reported here, but the response of the Chrome Azurol S test indicated some discontinuity in distribution of the preservative within the shakes (De Groot 1994b). The increased retention resulted from different concentrations of active ingredient in the treating solution. The increased composition of inert ingredients in those treating solutions may also have contributed to differences in the microdistribution of the active ingredient within the wood and that, in turn, contributed to slightly improved efficacy. In another study (Barnes et al. 1985), a water-dispersed copper naphthenate appeared to have promise after 28 months of field exposure.

Substantial decay occurred in shakes treated with zinc naphthenates. This was surprising because thorough penetration of the shakes was achieved with a variety of zinc-based formulations (De Groot 1994b). The analytical results indicate that retention was at a level where some protection would have been anticipated with these preservatives. A threshold level of 0.06 $lb/ft^3$ (1.0 $kg/m^3$) Zn had previously been determined for zinc naphthenate against *G. trabeum* in soil-block studies using red alder

[2] De Groot, R. C. [in press]. Performance of preservative-treated wood shingles and shakes. NIST symposium, Washington, D.C. (September 1995 meeting).

for the wood substrate (Mitchoff and Morrell 1988).

## CONCLUSIONS

The soil-contact decay test of cross sections of treated wood shakes is a good tool to evaluate likely performance of these products in adverse environments. The advantage of this method is that it provides a more accurate representation of the variability within the cross section of treated shakes compared with the small samples of uniform sapwood or heartwood used in standard decay tests (ASTM D1413, D2017). (In prior work (DeGroot 1994b), it was demonstrated that resin-filled annual rings precluded uniform treatment throughout cross sections of western hemlock shakes.) Interpretation of results requires consideration of both the distribution and median or mean of weight loss data for each treatment. Failure of treatments to suppress variation in amount of weight loss caused by decay fungi in candidate woods portends long-term potential for decay in service. The number of replicate shakes used per evaluation needs to be sufficiently large to fully represent the variation in wood quality and treatment as well as the mix of heartwood and sapwood that will be present in the actual product. Levels of retention should be well dispersed so that the effects of retention upon distribution of weight loss data for individual treatments can be determined.

As demonstrated here, relative performance and questionable potential of some treatments can be demonstrated with only one fungus. However, new, durable wood products should be challenged with a series of fungi that represent the complement of deteriogens to which the product would be exposed in nature. In a programatic evaluation, challenge with several fungi could be deferred until the more promising treatments were identified.

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