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# Residual Pentachlorophenol Still Limits Decay In Woodwork 22 Years After Dip-Treating

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IN 1952, experiments were started in western Oregon, southern Wisconsin, and southern Mississippi to determine how effectively dip treatments in wood preservatives would protect exterior woodwork against

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## Abstract

Experimental panels of conventional tongue-and-groove flooring lumber of 5 different wood species were dip-treated for 3 minutes in a 5 percent, light-oil solution of pentachlorophenol, then exposed off the ground in 3 diverse climates. Limited amounts of decay 22 to 23 years later evidenced continuing protection on all sites. Bioassay and x-ray spectrometric analysis of panels in western Oregon, where decay control was greatest, indicated that penta was still present in small amounts.

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decay. Five species of wood were treated by 3-minute soaking ("dipping") then used to construct two types of testy units - a post with diagonally attached rail and a flooring panel of conventional tongue-and-groove flooring boards. For comparison, the study included untreated units of western redcedar heartwood. The units were placed off the ground on the three exposure sites, and the results were reported after 7 and 15 years (2, 3). During the 15-year examination the post-rail units were dismantled, but the flooring panels were left intact.

At the end of 22 years, many of the flooring panels treated with pentachlorophenol (penta) were still in excellent condition, some entirely free from decay. This remarkable service life of wood given only a superficial treatment and exposed in climates moderately to highly conducive to decay prompted this report; contained here is a quantitative appraisal of the condition of the variously treated panels and the results of assays for residual penta.

## Experimental<sup>1</sup>

### Preparation and Maintenance of Flooring Panels

The flooring panels were conventional kiln-dried and surfaced boards as marketed for flooring. After the 3-minute dipping, the boards were nailed to creosoted frames of 2- by 4-inch lumber, forming panels approximately 21 by 23 inches (Fig. 1). Shortly thereafter, the panels were set on bricks so that they would be off the ground during the exposure.

Three treating solutions were prepared in mineral spirits using 5 percent penta, 5 percent penta plus 13 percent water repellent ingredients, and copper naphthenate (1% copper). Each solution was applied to boards going into 5 panels. However, this report deals chiefly with results with the penta solutions as they were superior to those with copper naphthenate.

To evaluate whether or not paint provided additional protection, the top surface of some panels was painted every 4 or 5 years with an exterior-type varnish-base gray enamel. However, for the last 10

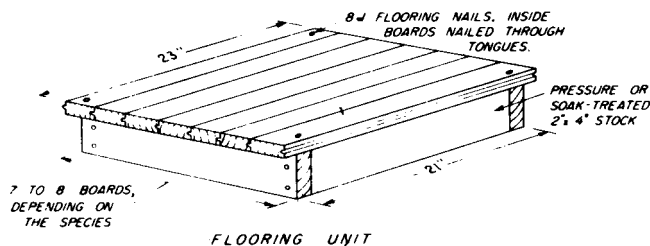


Figure 1. — Design of flooring panels.

<sup>1</sup>The Oregon field exposures were established cooperatively with the Oregon State Univ. School of Forestry (through arrangements by Robert Graham), the USDA Forest Service Pacific Northwest Forest and Range Expt. Sta., and the Western Wood Products Assoc.

years, painting was discontinued so that the panels received no attention except occasional cutting of surrounding vegetation.

### Rating the Panels for Condition

The following criteria were used to provide numerical ratings of the condition of individual boards in each panel:

- 0, board in good condition, no visible decay;
- 20, board in good condition but with slight, generally spotty decay;
- 40, board "serviceable" but with moderate, mostly localized decay;
- 60, board approaching "unserviceability," decay extensively distributed;
- 80, board intact but so weakened by decay as to be no longer "serviceable";
- 100, board essentially destroyed, easily broken by a sharp blow.

The condition of each set of 5 panels treated with a particular preservative was evaluated by averaging the ratings for each board in the set. In Oregon the condition of panels was further assessed according to the percentages of boards receiving a high or a low rating.

### Assaying for Residual Penta

To detect any residual penta in panels, the outer 5 mm of wood was assayed in two ways - by bioassaying for indications of fungus-inhibiting chemical and by using an x-ray spectrometer to measure penta content.

Bioassay samples were collected with an increment borer and placed in 90-mm petri dishes on a nutrient medium<sup>2</sup> seeded an hour or two earlier with spores of *Aspergillus niger* van Tiegh, a common mold fungus. To get the spores, a test tube containing the sporulating fungus was washed with 100 ml of water containing a mild detergent to keep the spores from clumping. After flooding the medium with the spore suspension, the excess was poured off. The dishes were incubated for 2 weeks at 27°C, then examined for any indication that toxic material in the sample wood had affected the assay fungus.

For x-ray spectrometric analysis of penta content, plugs 25 mm in diameter were cut from the boards with an electrical plug cutter. These were trimmed to eliminate all but the outer 5 mm of wood, then reduced in a Wiley mill so that all particles would pass through a 1-mm screen. The particles were compacted and analyzed for penta by the Koppers Company.

### Fungi Associated With Decay

Where decay was evident in flooring units from Oregon and Wisconsin, a representative board was removed to isolate and identify associated fungi. Where evident, basidiomycetous fruit bodies were also removed for identification. Decayed flooring units exposed in Mississippi were discarded before fungi could be isolated.

<sup>2</sup>Potato-dextrose agar: hot water extract of 200 g of potatoes plus 20 g of dextrose and 15 g of agar per liter.

Table 1. — AVERAGE RATINGS OF CONDITION OF DIP-TREATED AND UNTREATED FLOORING BOARDS AFTER 22 TO 23 YEARS ON THREE EXPOSURE SITES.<sup>b</sup>

Site and wood	Pentachloro-phenol	Pentachloro-phenol plus water repellent	Copper naphthenate	Untreated <sup>c</sup>
<b>WESTERN OREGON</b>				
Douglas-fir, mill run	0 (4)	0 (3)	13	24
Ponderosa pine, heartwood	0	—	—	21
Ponderosa pine, sapwood	26 (25)	24 (24)	74	98
Southern pine, sapwood	17	—	—	100
Western hemlock, mill run	0	0 (6)	46	81
Western redcedar, heartwood	—	—	—	16
White fir, mill run	7	—	—	100
<b>SOUTHERN WISCONSIN</b>				
Douglas-fir, mill run	8 (0)	16 (11)	4	54
Ponderosa pine, heartwood	44 <sup>d</sup>	— <sup>d</sup>	—	52
Ponderosa pine, sapwood	—	—	88	100
Southern pine, sapwood	—	—	—	—
Western hemlock, mill run	18	11 (44)	23	84
Western redcedar, heartwood	—	—	—	19
White fir, mill run	49	—	—	100
<b>SOUTHERN MISSISSIPPI</b>				
Douglas-fir, mill run	32 (35)	24 (29)	31	83
Ponderosa pine, heartwood	—	—	—	—
Ponderosa pine, sapwood	62	—	—	100
Southern pine, sapwood	—	99	—	100
Western hemlock, mill run	42	34 (62)	56	100
Western redcedar, heartwood	—	—	—	73
White fir, mill run	65	—	—	100

<sup>a</sup>Ratings are averages for 35 or 40 boards contained in five flooring panels.

Rating scheme:

- 0 - no visible decay
- 20 - definite decay but slight and localized
- 40 - decay moderate and mostly localized
- 60 - decay general but board still "serviceable"
- 80 - board intact but "unserviceable"
- 100 - board essentially destroyed

<sup>b</sup>Values in parentheses are for unpainted units; others are for painted units.

<sup>c</sup>A rating of 100 denotes units decayed to virtual destruction in prior years.

<sup>d</sup>Units lost.

Table 2. — PROPORTIONS OF DIP-TREATED FLOORING BOARDS IN GOOD OR FAILED CONDITION AFTER 22 TO 23 YEARS ON THE WESTERN OREGON EXPOSURE SITE.<sup>a</sup>

Wood	Good boards (%) <sup>b</sup>				Failed boards (%) <sup>c</sup>			
	Penta-chloro-phenol	Penta plus water repellent	Copper naphthen-ate	Untreated	Penta-chloro-phenol	Penta plus water repellent	Copper naphthen-ate	Untreated
Douglas-fir, mill run								
painted	100	100	77	65	0	0	8	10
unpainted	95	98	—	—	2	2	—	—
Ponderosa pine, heartwood								
painted	97	—	—	71	0	—	—	14
Ponderosa pine, sapwood								
painted	57	60	17	0	6	3	68	94
unpainted	62	60	—	—	11	6	—	—
Southern pine, sapwood								
painted	71	—	—	0	6	—	—	100
Western hemlock, mill run								
painted	100	100	45	12	0	0	30	77
unpainted	—	92	—	—	—	5	—	—
Western redcedar, heartwood								
painted	—	—	—	75	—	—	—	10
White fir, mill run								
painted	86	—	—	0	0	—	—	100

<sup>a</sup>Each value represents 35 to 40 boards.

<sup>b</sup>"Good" board; had a condition rating of 0 or 20, thus had no more than slight, localized decay.

<sup>c</sup>"Failed" boards had ratings of 80 or 100, thus were either "unserviceable" or essentially destroyed.

## Results

### Condition of Panels

On the whole, all boards in all three regions – especially those treated with penta – exhibited continuing benefit from the dip-treating (Table 1). The degree of benefit is especially evident when average decay ratings of all penta-treated Douglas-fir, hemlock, and white fir boards are compared with those of the untreated controls:

	W. Oreg.	So. Wis.	So. Miss.
Rating for treated boards	3	23	40
Rating for untreated boards	68	79	94

The ratings also reflect the regional aboveground decay hazards. According to a climate index devised for estimating decay potential in untreated, decay-susceptible wood exposed off the ground (4), indexes for decay hazards at the exposure sites were: 44 (moderate) in western Oregon; 40 (moderate) in southern Wisconsin; and 99 (severe) in subtropical southern Mississippi. The level of decay control was highest in Oregon; there the Douglas-fir, hemlock, and white fir panels either had no decay or had average board ratings no greater than 7.

The outstanding protective influence of the dip treatment is further indicated by comparing frequencies of “good” and “failed” boards of these species in western Oregon (Table 2). From 86 to 100 percent of the penta-treated boards were good (rating: 0-20), and 5 percent or less of the boards had failed (rating: 80-100). Especially remarkable was the good condition of the hemlock and white fir boards, woods notably subject to decay. For no apparent reason, boards of ponderosa and southern pine sapwood had less, though still

substantial, protection; possibly the greater permeability of the pine sapwood favored loss of chemical.

Painting the top surface of the panels did not materially affect decay development during the first 15 years (3). But, by the end of the 22d year, painting had considerably benefited 3 of 11 paired sets of painted and unpainted panels (Table 1), presumably by reducing surface checking that allows decay organisms to infect the wood. Surprisingly, checking in other panels was not accompanied by significantly greater decay, suggesting movement of preservative into the checks.

For the first time in these experiments, it was noticed that combining a water repellent with the penta solutions was beneficial. In 5 of 11 possible comparisons, panels treated with penta plus water repellent had moderately less decay than did those without repellent protection.

### Evidence of Residual Pentachlorophenol

The excellent condition of many penta-treated panels on the Oregon site and the better condition, relative to the controls, of corresponding panels on the other sites suggested that residual penta was still present in decay-inhibiting amounts. The bioassays of Oregon increment-core samples on nutrient agar seeded with *Aspergillus niger* confirmed the presence of residual penta, especially in hemlock panels (Table 3). Although cores from treated boards did not noticeably inhibit fungal growth, nearly all cores prevented the fungus from maturing sufficiently to produce its typical black spores (Fig. 2). In contrast, cores from untreated boards prevented development of black spores in only 1 of 94 assays. Cores from the top surface of the boards generally inhibited black-spore

Table 3. – BIOASSAY FOR EVIDENCE OF CONTINUED PROTECTION BY RESIDUAL PENTA-CHLOROPHENOL IN DIP-TREATED FLOORING PANELS AFTER 23 YEARS OF ABOVEGROUND EXPOSURE IN WESTERN OREGON.

Wood and treatment	No. of boards sampled at each location <sup>a</sup>			Boards with indicated residual protection <sup>b</sup> at each location		
	Top	Bottom	Edge	Top(%)	Bottom(%)	Edge(%)
Douglas-fir, mill run						
Penta, painted	18	18	18	11	17	33
Penta, unpainted	18	23	18	17	35	11
Penta, WR, <sup>c</sup> painted	24	18	24	8	6	54
Penta, W R, unpainted	18	18	18	0	28	22
Avg.				9	21	30
Not treated, painted	18	18	—	6	0	—
Western hemlock, mill run						
Penta, painted	24	23	23	37	74	65
Penta, WR, painted	18	18	18	22	55	67
Penta, WR, unpainted	15	17	17	27	65	65
Avg.				29	65	66
Not treated, painted	12	12	—	0	0	—
Ponderosa pine, heartwood						
Penta, painted	24	16	18	0	44	33
Not treated, painted	12	12	10	0	0	0

<sup>a</sup>The samples were increment core segments from the outer 5 mm of wood.

<sup>b</sup>Protection denoted where samples on nutrient agar seeded with *Aspergillus niger* prevented darkening of the fungus spores.

<sup>c</sup>WR = with repellent.

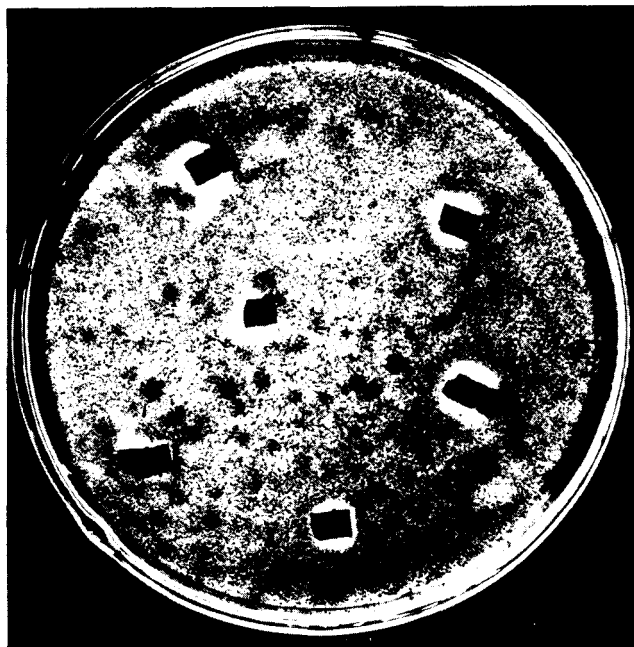


Figure 2. — Bioassay of Increment-core samples for evidence of residual pentachlorophenol, using *Aspergillus niger* seeded on nutrient agar. Penta fungicide is denoted by a light zone around the samples, where the fungus was unable to produce its typical black spores.

production less than did cores from the bottom, presumably because weathering removed much penta from the top.

Spectrometric analyses also indicated the presence of very small amounts of residual penta (Table 4). The readings would seem inconclusive except for the marked prevalence of positive findings and for their quantitative correlation with bioassay results. Separate analyses of tops and bottoms of 19 panels provided 38 readings, of which 31 were positive for penta (not shown in table). Higher levels of penta were indicated for panel bottoms than for tops (7 zero readings, all for top surfaces). This preponderance of positive readings for penta, particularly for bottom

surfaces, seems extremely unlikely to have occurred by chance.

Additional x-ray spectrometric indication of penta may be seen by comparing results of samples from Douglas-fir and hemlock boards (Table 4). In six instances where the two woods were treated alike, the greater amount of residual penta occurs in the hemlock. The chance probability of getting this consistent difference is less than 5 percent. The record of average solution absorptions further supports the likelihood that the x-ray spectrometric readings truly reflected penta in the wood; the Douglas-fir panels received 9 grams of solution per square foot of board surface, and the hemlock panels received 12 grams.

### Fungal Identifications

Basidiomycetes isolated from or found fruiting on test units exposed outdoors in Wisconsin were identified as:

- A. Western hemlock (mill run)
  - Untreated (painted)
    - Hyphodontia* sp.
  - Penta WR (unpainted), treated
    - Hyphodontia* sp., *Lentinus tigrinus* Bull. ex Fr., *Shizophyllum commune* Fr.
  - Copper naphthenate (painted), treated
    - Poria medula-panis* (Jacq. per Fr.) Cke., unknown white-rot fungus A
- B. Ponderosa pine
  - Untreated heartwood (painted)
    - Hyphoderma pratermissum* (Karst.) J. Erikss. & Strid
  - Penta (painted), treated heartwood
    - Poria medula-panis*
  - Copper naphthenate (painted), treated sapwood
    - Phanerochaete* sp.
- C. Douglas-fir (mill run)
  - Penta (painted), treated
    - Hyphodontia brevista* (Karst.) J. Erikss.
  - Penta WR (painted), treated
    - Hyphodontia* sp.
  - Penta WR (unpainted), treated
    - Poria rivulosa* (Berk. & Curt.) Cke.

Table 4. — RESIDUAL PENTACHLOROPHENOL<sup>a</sup> IN THE TOP AND BOTTOM 5 mm OF DOUGLAS-FIR AND HEMLOCK FLOORING BOARDS AFTER 23 YEARS EXPOSURE ON THE WESTERN OREGON SITE.

Board treatment	Percent of penta (w/w) in individual units <sup>b</sup>							
	Douglas-fir				Western hemlock			
	Top		Bottom		Top		Bottom	
	Range	Avg.	Range	Avg.	Range	Avg.	Range	Avg.
Pentachlorophenol painted	0.00-0.04	(0.017)	0.03-0.04	(0.033)	0.01-0.03	(0.02)	0.03-0.04	(0.037)
unpainted	0	(0)	.03	(.03)	—	—	—	—
Pentachlorophenol + water repellent painted	0.00-0.01	(0.003)	0.03	(0.03)	0.00-0.03	(0.017)	0.03-0.04	(0.037)
unpainted	.01-0.02	(0.015)	.03	(.03)	.01-.03	(.02)	.04	(.04)
Mean avg.		0.009		0.033		0.019		0.038

<sup>a</sup>Determined by x-ray spectrometric analysis.

<sup>b</sup>Average percentages represent analyses of 3 flooring panels except for unpainted Douglas-fir and hemlock treated with water-repellent penta, both represented by 2 panels. Analysis of each panel was made on a composite sample of 6 boards.

- D. White fir (mill run)  
 Penta (painted), treated  
*Hyphoderma praetermissum*  
*Hyphodontia* sp.

Basidiomycetes obtained from test units exposed in Oregon were:

- A. Western hemlock (mill run)  
 Copper naphthenate, treated  
 Unknown B., Unknown brown-rot fungus, C,  
*Gloeophyllum saepiarium* (Wulf.) Karst.
- B. Ponderosa pine  
 Untreated heartwood (painted)  
 Unknown B., *Chaetoderma luna* (Rom) Parm.  
 Penta- and copper naphthenate-treated  
 sapwood  
 Unknown white rot fungus D (3 isolates)
- C. Douglas-fir (mill run)  
 Untreated and copper naphthenate-treated  
 Unknown B
- D. Western redcedar  
 Untreated (painted) heartwood  
 Unknown B<sup>3</sup>

Regional differences between the attacking fungi are indicated by the significantly larger number of identifiable species found in the Wisconsin units.

### Summary and Comments

Experimental panels of commercial flooring boards that had been dip-treated in a mineral-spirits solution of pentachlorophenol, alone or with a water repellent, exhibited surprisingly limited amounts of decay after open exposure off the ground for 22 to 23 years in the diverse climates of western Oregon, southern Wisconsin, and southern Mississippi. In fact, many Oregon panels were free of decay. Similarly, a superficial treatment with penta solution effectively protected pine millwork over a 13 to 14 year period in eastern Canada (5).

Painting the top surface of treated panels reduced weather checking, which in turn restricted decay in some panels. In many unpainted panels, the checking did not significantly worsen decay control; presumably the wood at the checks was protected by the initial penta penetration or by penta that leached into the checks as they developed. A water repellent added to the penta moderately improved protection during the later years.

<sup>3</sup>The isolates designated as Unknown B were characterized by consistent negative phenol oxidase reactions on acid medium and positive reactions on tannic acid medium. *Schizophyllum commune* is another species that consistently gives this unusual oxidase reaction.

A bioassay depending on failure of *Aspergillus niger* growing on nutrient agar to produce black spores in the presence of increment-core samples containing penta indicated the presence of penta in a large proportion of treated boards. More penta was evident in wood from the bottom surface than from the top surface of the boards, apparently reflecting the effects of the greater weathering of the exposed surface. Analysis of the wood by x-ray spectrometry showed penta in very small amounts up to 0.04 percent (w/w). Although these quantities are within the range of experimental error, we believe that they confirm the presence of penta because variations substantially correlated with the bioassay results.

These observations point to a greater than previously recognized potential for dip-treating as a way to extend the service life of millwork and lumber used above ground. Penta residues seem rather tenaciously held in surface wood and in weather checks so that the penta, after many years, remains potent enough to suppress infection by spores of decay fungi. Penta residue sufficient to saturate water may be all that is needed because even an extremely weak concentration of penta in a water-saturated solution is fungistatic (1).

More information is needed about the long term capacity of the principal preservative chemicals to protect wood off the ground, when applied by dipping or copious brushing. A major requirement in some situations would be the ability of the preservative to protect weather checks by initial penetration sufficiently deep into the wood or by migration of residue near the surface into checks as they develop. Additional factors to consider include strength of treating solution, kind of wood, wood dimensions, climate, and need for protecting end grain or face grain. In experiments to gather such information, a bioassay similar to ours might be useful for early assessment of protection.

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