# **Evaluation of New Creosote Formulations**

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### **Abstract**

This study compared two new formulations of creosote and one pigment-emulsified creosote (PEC) with a formulation of creosote that met requirements of the AWPA standard P1/P13 for creosote. Two softwood and two hardwood species were treated to four retention levels with each formulation. The four creosote formulations were evaluated by (1) soil-block test, (2) fungal cellar test, and (3) field test. This paper presents results from the soil-block tests and preliminary findings from the fungal cellar tests after 72 weeks. The field stakes have been exposed for only 1 year, thus these data are not available. Data from the fungal cellar tests show that softwoods are protected much better than hardwoods for all four formulations of creosote tested. The soil-block tests show comparable performance in softwood and hardwoods. No major difference between formulations was detected in the two laboratory tests.

### Introduction

Creosote, or coal-tar creosote, has persisted since the earliest days of treating wood with preservatives. In the United States, creosote was first used for treating marine pilings in 1889. By the 1920s, it became the treatment of choice for the railroad industry and continues to be so today, During the late 1960s, high temperature creosote largely replaced low temperature

creosote for timber preservation as a result of the decreasing gasification of bituminous coals, In 1982, low temperature creosote, conforming to Australian standard specification K-55 (AS 1965, Watkins and others 1983), ceased production in Australia.

One problem associated with the use of oil-type preservatives is the tendency to exude or "bleed" from some treated commodities, producing an oily or tarcovered surface that can cause handling problems. To minimize this exuding problem, laboratories, such as Koppers Industries Inc., USA and Commonwealth Scientific and Industrial Research Organization (CSIRO), Division of Chemical and Wood Technology, Melbourne, Australia, have developed changes in processing of coal tar, which produces distillates with fewer contaminants. This "clean distillate" is then used to formulate "clean creosote" as a preservative utilized in the treatment of utility poles.

Soil-block and fungal cellar test methods (accelerated field simulator) (Johnson and others 1982) are two laboratory procedures used to characterize the effectiveness of wood preservatives. The soil-block tests determine the minimum threshold level of the preservative that is necessary to inhibit decay by pure cultures of decay fungi. The fungal cellar test exposes treated wood to accelerated attack by mixtures of soil-borne fungi. The field stake tests are used to verify

service life of the new creosote formulations *in vivo*. Results from accelerated tests are indicative of field performance, but the correlation between laboratory and field results is still being investigated. Field stake testing is regarded as a critical, long-term evaluation that provides results most directly related to the performance of treated products in service.

The objective of this study was to compare two new formulations of creosote and one pigment-emulsified creosote (PEC) with a formulation of creosote that met requirements of AWPA standard (1995a) P1/P13 for creosote. Two softwood and two hardwood samples were treated to four retention levels with each formulation. The four creosote formulations were evaluated by (1) soil-block test (ASTM 1986), (2) fungal cellar test (AWPA 1995d), and (3) field test (AWPA 1995c). Results from the soil-block test and preliminary findings from the fungal cellar tests after 72 weeks are presented in this paper. However, stakes in the field test have been exposed for only 1 year, thus data are not available.

## **Material and Methods**

# **Wood Species**

The following wood species were used in this study:

Douglas-fir (Pseudotsuga menziesii)	Mixture of heartwood and sapwood from second-growth trees in the Pacific states of Oregon and Washington.
Pine (Pinus sp.)	Sapwood with 5-15 rings/ 25.4 mm (rings/in.) Wood was kiln dried without the use of antistain chemicals.
Red oak (Quercus rubra)	Red oak heartwood was predominantly selected to represent a dense, ring-porous hardwood. Wood was used as supplied.
Red maple (Acer rubrum)	Both heartwood and sapwood of northern red maple were used to represent a diffuse porous

hardwood. Wood was visually

selected for clear material.

### **Test Specimens**

Evaluations of preservative efficacy were made using the following wood specimen sizes:

- Soil block: 19.05 mm (0.75 in.) cubes
- Fungal cellar: 3 by 19 by 150 mm (0.118 by 0.748 by 5.91 in)
- Field stakes, two sizes:
  19.05 by 19.05 by 457.2 mm (0.75 by 0.75 by 18 in) and
  25 by 50 by 457 mm (1 by 2 by 18 in.)

#### **Preservatives**

The following preservatives were used, which were either creosote or modifications thereof. These preservatives were selected because of the recognized contribution of creosote treatments to resist physical abrasion in addition to their resistance to colonization by biological organisms.

• Creosote	(P1/P13)	Creosote meeting AWPA Standard P1/P13 was used as the reference preservative treatment
• Creosote	A	New formulation being developed by Koppers Industries Inc., USA
• Creosote	В	New formulation being developed by Koppers Industries Inc., USA
• P E C		Pigment Emulsified Creosote—A formulation developed and used in Australia (not available in USA)

## Soil-Block Test

Two brown-rot fungi, *Postia placenta* (Fr.) M. Lars. et. Lomb. [MAD-698] and *Neolentinus lepideus* (Fr.:Fr:) Redhead and Ginns [MAD-534], and two white-rot fungi, *Trametes versicolor* L. ex Fr: Pilate [MAD-697] and *Irpex lacteus* (Fr.:Fr.) Fr. [HHB-7328 sp.], were used as test fungi. Test fungi were maintained on 2% malt agar. Both untreated and treated pine (*Pinus* spp.) and red maple (*Acer rubrum* L.) blocks were exposed to brown- and white-rot decay fungi in soil-block tests according to ASTM D1413-76 (1986). Five replications of treated blocks and 10 replications of untreated controls were exposed to decay fungi for 12 weeks. Blocks were not weathered prior to exposure to test fungi.

Four concentrations of each preservative were evaluated. To maintain some consistency among species in this study, treating solutions were prepared in a series of concentrations of active ingredients that was used with all wood species. This produces different actual retention levels in the various species, but this pattern provides a good overall evaluation in multiple species.

Several formulations of PEC have been developed in Australia. We utilized PEC 30W. This is an anionic emulsion that consists of 30% water and 3.5% finely dispersed micronized titanium dioxide pigment. The preservative is manufactured as an oil-in-water emulsion, which is inverted to a highly stable water-in-oil emulsion before use. The same four treatment levels were used.

Four concentrations of treating solution using toluene as the diluent were used for each U.S. creosote formulation to obtain four different active ingredient levels: 65%, 30%, 15%, 7.5%. Immediately after treatment, the surface of each specimen was wiped clean to removed excess chemical and weighed. Retention levels after treatment of each specimen were calculated on the basis of weight gained and were confirmed through AWPA Standard A2-94 (1996).

# **Fungal Cellar**

The fungal cellar evaluation is currently in progress at the USDA Forest Service, Forest Products Laboratory, in Madison, Wisconsin. Fifteen replicate stakes of each wood species were treated with each of the four retention levels of each creosote formulation. The stakes are 3 mm (thick, transverse) by 19 mm (radial direction) by 150 mm (parallel to the grain) (0.118 by 0.748 by 5.91 in.). Stakes are exposed in soil that is maintained at a moisture content of 50% to 70% of water holding capacity to promote growth of soft-rot fungi (Nicholas and others 1991). Soil beds are maintained in a controlled environment at 26°C and a relative humidity of 86% to 90%.

Prior to exposure in the fungal cellar, all treated and control specimens were vacuum impregnated with water. The stakes were grouped by treatment, subjected to vacuum (about 100 mmHg) for 30 minutes before being soaked for 2 hours in distilled water. The test specimens were then inserted vertically into the fungal cellar until the top end was level with the soil.

At 3-month intervals, for 18 months, wood specimens were removed from the fungal cellar (soil bed), cleaned with a brush to remove excess soil, and placed in watertight plastic bags until evaluation for strength loss using the bending strength apparatus shown in Figure 1, Strength loss was determined as described by

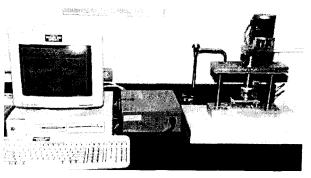


Figure I—Bending apparatus with computer interface

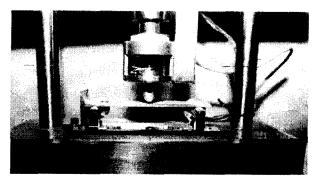


Figure 2—Orientation of specimen on bending apparatus

Crawford (1994). Care was taken to ensure that the test specimen was oriented in the same way during subsequent strength evaluations (Fig. 2).

Initial load measurements were made, and modulus of elasticity (MOE) was calculated using the following:

$$MOE = \frac{L^3}{4} \frac{P}{bh^3D} \tag{1}$$

where

MOE = modulus of elasticity (kPa),

L = constant span of 126 mm,

b = constant specimen width of 19 mm,

h = constant specimen thickness of 3 mm,

D = constant specimen deflection of 2.50 mm, and

P = variable force to maintain constant deflection (g).

$$MOE = 389.94(P)$$
 (2)

A stake that demonstrated MOE losses greater than 60% of its original MOE was considered failed.

# Results and Discussion Soil-Block Test

# Retention

The actual retention levels for individual formulations at their targeted concentrations (as calculated from

Table 1—Decay resistance of pine soil-blocks treated with PEC and three creosote formulations (12-week exposure).

				Weight loss (%)					
	Concentration	Rete	ention	Brow	n rot	White	rot		
Formulation	(% Al*)	(pcf)	(kg/m³)	N. lepideus	G. trabeum	T. versicolor	L. lacteus		
Control		0	0	40.10	62.50	21.20	17.60		
PEC <sup>b</sup>	7.5	3.09	49.52	6.56	2.35	0.34	0.95		
	15	6.26	100.32	1.01	0.96	0.81	1.29		
	30	13.44	215.38	1.23	1.06	1.50	1.38		
	65	27.04	433.33	2.17	2.29	3.38	2.53		
Control		0	0	40.10	62.50	21.20	17.60		
P1/P13	7.5	2.81	45.03	15.00	7.10	0	0		
1 171 10	15	4.95	79.33	4.30	1.20	0	0		
	30	7.34	117.63	0	0	0	0		
	65	19.60	314.10	1.60	1.80	1.70	1.60		
Control		0	0	40.10	62.50	21.20	17.60		
Creosote A	7.5	2.10	33.65	19.60	14.10	0	0		
01000010 71	15	4.54	72.76	12.80	5.00	0	0		
	30	9.29	148.88	0	0	0	0		
	65	23.19	371.63	Ō	0	0	0		
Control		0	0	40.10	62.50	21.20	17.60		
Creosote B	7.5	2.09	33.49	15.50	11.90	0	0		
	15	4.18	66.99	6.20	4.70	0	0		
	30	9.07	145.35	0	0	0	0		
	65	22.39	358.81	1.00	0	0	1.00		

<sup>&</sup>lt;sup>a</sup>Al is active ingredient.

weight gain during treatment) differed slightly (Tables 1,2, Fig. 3) for both the pine and the red maple.

In both species, the PEC was more readily absorbed at the higher targeted concentrations than were the other formulations. In the pine at a targeted concentration of 7.5% active ingredient (AI), the retention for all four formulations was between about 33 to 50 kg/m³ (2.06 to 3.12 pcf (pound per cubic foot)). At a targeted retention of 30% AI, the PEC had a retention level of 215 kg/m³ (13.42 pcf), and the other three formulations had retention levels about half that of PEC.

In the red maple at a targeted concentration of 7.5% AI, the PEC and P1/Pl3 formulations absorbed twice as much as did the other creosote formulations, with retention levels of about 50 kg/m $^3$ (3.12 pcf) for PEC and P1/Pl3 and 25 kg/m $^3$ (1.56 pcf) for the creosotes. At the higher targeted concentration of 30 kg/m $^3$ (1.872 pcf), the PEC had a retention of about 240 kg/m $^3$ (14.98 pcf), and the three other formulations were about 110 to 120 kg/m $^3$ (6.86 to 7.48 pcf).

There does not appear to be any species differences in absorption of the individual formulations. The red

maple retention levels were similar to the pine retention levels for any given formulation. The only exception appears to be with creosote B, where the pine had slightly higher retention levels of creosote B than did the red maple.

### Weight Loss

Although some general trends were evident, the soil-block test did not distinguish major differences among the formulations in protecting pine sapwood (Figs. 4, 5). In a comparison of creosote A and B with creosote P1/P13, creosotes A and B were slightly less effective than P1/P13 against *G. trabeum* at the lowest retention level (Figs. 4, 5). At subthreshold levels, creosote A appears to be less effective than P1/P13 against *N. lepideus*.

The protection provided by PEC at low retention was better against all fungi than the reference preservative P1/P13 creosote and the other two creosote formulations (A and B). However, the increase in weight loss observed in blocks of red maple treated at increased retention levels of P1/P13 creosote is difficult to interpret.

<sup>&</sup>lt;sup>b</sup>PEC is pigment emulsified creosote.

Table 2—Decay resistance of red maple soil-blocks treated with PEC and three creosote formulations (12-week exposure).

					Weight loss (%)					
Formulation	Concentration	Retention		Brow	wn rot	White rot				
	(% Al <sup>a</sup> )	(pcf)	(kg/m³)	N. lepideus	G. trabeum	T. versicolor	L. lacteus			
Control		0	0	29.20	64.80	57.50	44.30			
PEC <sup>b</sup>	7.5	3.25	52.08	0.10	0.06	0	0			
	15	6.40	102.56	0.63	0.29	0.66	0.39			
	30	15.26	244.55	1.30	1.33	1.90	1.33			
	65	30.98	496.47	7.76	7.56	6.07	4.56			
Control		0	0	29.20	64.80	57.50	44.30			
P1/P13	7.5	2.78	44.55	1.40	3.80	6.00	0			
	15	4.98	79.81	0	0	0	0			
	30	7.50	120.19	0	0	0	Ō			
	65	21.49	344.39	1.70	2.00	2.30	2.30			
Control		0	0	29.20	64.80	57.50	44.30			
Creosote A	7.5	1.67	26.76	10.70	9.30	20.08	0			
	15	3.68	58.97	1.30	2.90	0	0			
	30	8.40	134.62	0	0	0	0			
	65	20.88	334.62	0	0	1.30	1.10			
Control		0	0	29.20	64.80	57.50	44.30			
Creosote B	7.5	1.46	23.40	11.20	7.30	27.30	10.90			
	15	2.91	46.63	1.20	2.20	1.90	0			
	30	6.96	111.54	0	0	0	0			
	65	18.33	293.75	0	0	Ō	1.00			

<sup>&</sup>lt;sup>a</sup>Al is active ingredient.

# **Fungal Cellar Test**

Modulus of elasticity (MOE) results are reported after 72 weeks of exposure (Table 3). None of the creosote formulations appeared to prevent attack by soft-rot fungi in red oak or red maple as determined by reduced MOE. Decrease in MOE approached or exceeded 60% at all retention levels for all creosote formulations in those species. At all creosote retention levels, softwoods were protected better than hardwoods. As retention levels increased, the relative difference between hardwoods and softwoods increased.

After 72 weeks of exposure in the fungal cellar, there was no loss in MOE in the Douglas-fir stakes and a minimal loss in the pine stakes treated with 65% PEC (23 pcf). Both hardwood species showed approximately 50% loss in MOE at 65% PEC (19 to 23 pcf) after 72 weeks of exposure in the fungal cellar. Loss in MOE for other formulations in Douglas-fir ranged from about 30% to 60% at AI concentration of 7.5% (1.8 to 2.0 pcf) to 30% (7.6 to 9.7 pcf). In pine, all formulations had equivalent performance.

# **Retention Analysis**

The treatability of all four wood species at a given concentration of active ingredient of the different formulations provided a range in retention levels of active ingredient in the treated wood that ranged from slightly more than 16 kg/m³ to approximately 320 kg/m³ (20 pcf) (Table 4). This range in retention of active ingredient spanned the targeted retention of 160 kg/m³ (10 pcf), which is specified for oak ties (AWPA 1995b).

At higher concentrations, retention levels for creosote P1/P13 and A were approximately 20% less than that achieved with PEC and creosote B. Still, these retention levels were greater than 160 kg/m³ (10 pcf).

Red oak had the lowest retention at all solution concentrations for all formulations. The PEC yielded the highest retentions as calculated by weight gain. Retention levels of the other three formulations tended to be relatively comparable for each concentration of treating solution, but with an occasional spurious result for a given wood species. As with the soil-block test, general trends indicate relative little difference in effectiveness between the four formulations.

<sup>&</sup>lt;sup>b</sup>PEC is pigment emulsified creosote.

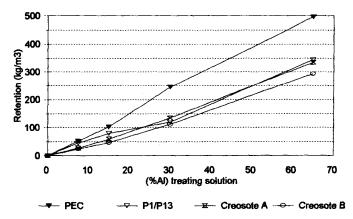


Figure 3—Retention levels of four formulations of creosote in red maple soil blocks that were treated with solutions that had comparable concentrations of active ingredients.

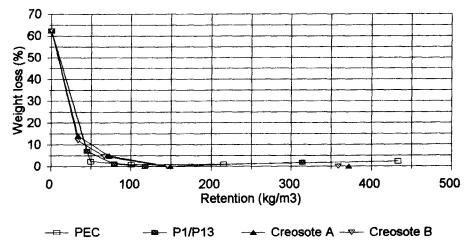


Figure 4—Weight toss of pine soil blocks treated with four formulations of creosote and exposed to *G. traveum*.

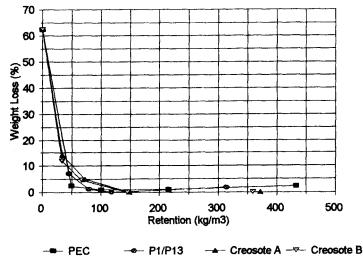


Figure 5-Weight loss of red maple soil blocks treated with four formulations of creosote exposed to *G. trabeum*.

Table 3—Average modulus of elasticity loss at four active ingredient (AI) concentrations.

	Modulus of elasticity loss (%)							
	Concentration (% Al <sup>a</sup> )	Creosote (P1/P13)	Creosote A	Creosote B	PECª	Control		
Pine	0					96.54		
	7.5	59.99	60.46	64.04	65.97			
	15	44.21	56.04	54.25	57.62			
	30	39.07	43.68	38.33	40.01			
	65	9.40	22.73	12.89	8.05			
Douglas-fir	0					81.02		
•	7.5	55.55	57.74	60.71	69.48			
	15	38.88	42.40	42.77	69.43			
	30	29.98	26.74	28.56	49.88			
	65	7.90	13.17	11.94	0			
Red oak	0					89.37		
	7.5	85.16	86.07	91.74	83.07			
	15	78.43	82.47	79.89	79.10			
	30	70.68	73.92	76.06	73.02			
	65	52.58	62.67	59.43	53.91			
Red maple	0					97.70		
•	7.5	86.65	89.22	84.47	87.91	3		
	15	80.66	84.18	80.43	83.29			
	30	62.69	75.45	69.34	79.63			
	65	46.70	51.08	49.23	49.95			

<sup>\*</sup>PEC is pigment emulsified creosote.

Table 4—Calculated retention at different concentrations of four creosote formulations in fungal cellar test.

	Average	F	Pine		Douglas-fir		Red oak		Red maple	
Formulation	(% Al²)	(pcf)	(kg/m³)	(pcf)	(kg/m³)	(pcf)	(kg/m³)	(pcf)	(kg/m³)	
PEC⁵	7.5	2.99	47.92	3.33	53.37	2.94	47.12	3.12	50.00	
	15	5.76	92.31	5.76	92.31	5.55	88.94	5.36	85.90	
	30	12.98	208.01	12.99	208.17	12.68	203.21	12.64	202.56	
	65	27.75	444.71	23.11	370.35	19.06	305.45	23.35	374.20	
P1/P13 Creosote	7.5 15 30 65	2.07 4.15 11.15 20.60	33.17 66.51 178.69 330.13	1.99 4.09 9.72 20.55	31.89 65.54 155.77 329.33	1.20 2.45 10.68 14.24	19.23 39.26 171.15 228.21	1.65 3.52 9.52 18.12	26.44 56.41 152.56 290.38	
Creosote A	7.5	1.98	31.73	1.79	28.69	1.17	18.75	1.63	26.12	
	15	4.16	66.67	3.67	58.81	2.51	40.22	3.66	58.65	
	30	8.90	142.63	8.50	136.22	5.47	87.66	7.96	127.56	
	65	20.15	322.92	20.07	321.63	20.60	330.13	13.19	211.38	
Creosote B	7.5	1.96	31.41	2.02	32.27	1.24	19.87	1.87	29.97	
	15	4.04	64.74	1.94	31.09	2.43	38.94	1.89	30.29	
	30	8.89	142.47	7.63	122.28	5.56	89.10	8.23	131.89	
	65	21.35	342.15	20.52	328.85	13.28	212.82	20.24	324.36	

<sup>&</sup>lt;sup>a</sup>Al is active ingredient.
<sup>b</sup>PEC is pigment emulsified creosote.

# **Concluding Remarks**

Results from the soil-block test indicate that products treated with any formulation in the test should have comparable durability. The tendency for formulations A and B to be slightly less effective than the P1/P13 formulation at the lowest retention level places more importance on quality control during treatment. With these two modified creosotes, low retention levels may not perform as well as low retention levels of P1/P13. Generally, there was little difference in the ability of the four creosote formulations to prevent decay at the three highest retention levels.

Results to date from the fungal cellar tests indicate the potential for poorer performance of treated hardwoods than has been observed in practice. The historical success of P1/P13-type creosotes in U.S. hardwoods at retention levels less than those tested in this study begs for a fundamental explanation of the cause of these results. The relative low retention of red oak in comparison with retention levels of other formulations per concentration of treating solution may somehow be related to the relatively poor performance of that wood species in the fungal cellar. Still, a calculated retention in excess of 160 kg/m3 (10 pcf) was obtained with all formulations at the highest treatment concentration. Furthermore, red maple, which usually had higher retention levels than red oak for each treatment, also performed poorly in the fungal cellar.

The field stake test will be used to verify service life of the new creosote formulations. As previously noted, few data are available from the field stake test. Thus, we are unable to correlate field stake data with the fungal cellar data at this time.

# **Acknowledgment\***

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