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Section 2 Test methodology and assessment

Patterns of Long-Term Performance—How Well are They Predicted From Accelerated Tests and Should Evaluations Consider Parameters Other Than Averages?

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ABSTRACT

This paper is a discussion of whether different service-life distribution patterns of products treated with unlike preservatives can be predicted, modeled, characterized, or even anticipated from accelerated laboratory tests. Graphic displays of data from Forest Products Laboratory field plots with preservative-treated and fire-retardant-treated stakes demonstrate the importance of local environment as a factor that affects field performance and exhibits differences in dose-response patterns among treatments. These distribution patterns are discussed with reference to early failures, first quartile and median failure times, and distribution about medians. Questions are then asked about the relevance of these parameters to practical applications, about the need to consider population characteristics other than average in evaluations on new preservatives, and about the capability of accelerated tests to estimate these parameters.

Key words: preservatives, testing, actuary, durability

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BACKGROUND

The minimum retention of a preservative (threshold value) that prevents growth of decay fungi is determined in standardized laboratory tests, such as the soil-block procedure (ASTM 1996; AWPA 1996c). This threshold value is an expression of toxicity. As such, it has relevance to an environment that remains static or relatively static over time.

The natural environment however, is dynamic. The natural selection of fungi tolerant or adaptation of fungi to an active ingredient may be one dynamic component in this environmental setting. Because of the dynamics of natural systems, field tests of treated-wood members, vertically inserted in the ground (stake or graveyard tests)(AWPA 1996b), are conducted to verify laboratory results. Uniformately, these field trials require years of exposure. This provides little opportunity for quick evaluation of new preservative systems.

In the United States, the traditional approach to evaluate data using experimental field plots has been to compare averages. The merits of recognizing the variability in the analysis of treated products have been set forth (Link and De Groot 1990). Characterizing durable products on the basis of percentiles of survivability may provide a more sensitive indicator of dose–response effects than does a comparison of averages. To wit, does more preservative have the same relative impact in delaying failure of the first 5th, 10th, or 25th percentile as it may have in delaying failure of the first half of the population? Do items treated with mobile and relatively immobile preservatives have unique service-life distribution patterns? Even more fundamental to the question of predicting service life of new systems is the question. Can the different failure distribution patterns of products treated with various preservatives be predicted, modeled, characterized, or even anticipated using accelerated laboratory tests? In the same manner, Can relative performance in different edaphic or climatic locations be predicted?

In the report herein, service-life distribution patterns of stakes treated with different types of preservative systems are presented and discussed with reference to the first quartile and median survival times. Where possible, patterns of field performance are related to outputs of current laborarory methodologies.

METHODS AND MATERIALS

Service-life data for preservative-treated or fire-retardant-treated stakes were plotted to allow comparison of quartiles (Link and De Groot 1989) and median service lives and to show distribution pattern of failures about the respective medians for different retention levels of individual preservatives at one location and among locations. Data from field plots maintained by the USDA Forest Service, Forest Products Laboratory (FPL), during the past 50 years were utilized (Gutzmer and Crawford 1995). Some plots were located in quite different environments (Table 1). A randomized block design had been used in all field plots. All stakes included in this report were southern pine sapwood, standard 50- by 100- by 450-mm (nominal 2- by 4- by 18-in) specimens. All preservative treatments referenced in this discussion were pressure treatments (Table 2).

Table 1. Environmental description of field plots

Location	Environmental description
USDA Forest Service Harrison Experimental Forest Southern Mississippi, USA 32 km north of Gulf of Mexico	Mean annual precipitation, 1580 mm Average annual temperature, 19.6°C Soil type, poarch sandy loam
Madison, Wisconsin USA	Mean annual precipitation, 780 mm Average annual temperature 7.4°C Soil type, clay loam soil
Bogalusa, Louisiana USA	Mean annual precipitation, 1536 mm Average annual temperature, 19.1°C Soil type, sandy loam
Jacksonville, Florida USA	Mean annual precipitation, 1347 mm Average annual temperature, 20.0°C soil type, sandy

We placed emphasis on plots in which all the stakes had failed or sufficient data were available to at least determine the quartiles of service life for individual treatments. This permitted examination of treatments with creosote, copper-based metal organics, and inorganic systems. The following briefly describes these preservatives.

Creosote that complies with U.S. Environmental Protection Agency (EPA) regulations today is slightly different from the coal-tar creosote used to treat the stakes that we documented. However, the principle that creosote is an organic system vis a vis an inorganic system remains valid. The threshold value for creosote in laboratory tests varies with type of creosote tested and tolerance of the assay fungus. Threshold values encompass a range from 48 to 192 kg/m^3 (3 to 12 lb/ft^3) (Cookson and Greaves 1986; Duncan and Richards 1951).

Metal organic systems are represented by copper formate and copper naphthenate. Sodium pentachlorophenate represents an organic salt. It is recognized that the efficacy of treatments, such as copper napthenate, which utilize an organic solvent, could reflect biocidal activity of either the metallic component, the organic component, or a combination of the two as well as the physical effect of the organic system. Therefore, data on stakes treated with petroleum solvents only were also reviewed to gain some indication of whether petroleum solvents influence patterns of failure in populations of stakes. The minimum retention levels required in the United States (AWPA 1996a) for copper naphthenate in southern pine lumber are 0.64 kg/m^3 (0.04 lb/ft^3) elemental copper in wood used above ground and 0.96 kg/m^3 (0.06 lb/ft^3) elemental copper in wood used in ground contact. Most reported threshold values for copper naphthenate were within 0.5 to 1.0 kg/m^3 (0.03 to 0.065 lb/ft^3) elemental copper (De Groot et al. 1988).

Copper formate is not included in AWPA standards for wood preservatives. In soil-block tests, no threshold value was established for copper formate. In soil-block tests with red pine, copper formate did not prevent decay by the copper-tolerant decay fungus, *Poria monticola*, even at retention levels of 4.0 kg/m^3 (0.25 lb/ft^3) elemental copper (McKnight and Merrall 1958). Results of an unpublished study by Dunan[1] showed that copper-tolerant decay fungi could decompose wood treated to retention levels of 2.9 kg/m^3 (0.18 lb/ft^3) metallic copper.

Inorganic treatments include zinc chloride, chromated zinc chloride, and flur-chrome-arsenate-phemnol. Zinc chloride is perhaps the most mobile system included in this review. Zinc chloride has been used as a wood preservative extensively in Europe and the United States (Drefahl 1930), but information on required minimum

[1] Duncan, C.G. Laboratory soil-block test of copper formate for decay control. Madison, WI: US. Department of Agriculture, Forest Service, Forest Products Laboratory. Unpublished preliminary report. 1957. 7 p.

Table 2. Summary of treating procedures

Chemical treatment	Exposure Site	Plot	Treating process
chromated zinc chloride in water	Mississippi, Wisconsin Louisiana Florida	2	Full cell -No additional information available
coal-tar creosote	Mississippi Wisconsin	4	Reuping process: -initial air: 310.2 x 10^{+3} Pa -pressure: 1034.1 x 10^{+3} Pa for 1.5 h -vacuum: 698.5 mm for 15 min -treating temperature: 93.3°C
copper formate in water	Mississippi	47	Full cell -pressure: 1034.1 x 10^{+3} Pa for 1 h -post treatment steaming at 103.4 x 10^{+3} Pa for 1.5 h [121°C]
copper naphthenate in No. 2 fuel oil	Mississippi Wisconsin	7	Reuping process: -initial air: 103.4 x 10^{+3} Pa -pressure: 1034.1 x 10^{+3} Pa for 1 h -vacuum: 698.5 mm for 1/2 h -treating temperature: 51.7°C -post treatment heating: 79.4°C
copper naphthenate in caralytic gas base oil	Missisippi	20	Reuping process: -initial air: 517.0 x 10^{+3} Pa -pressure: 1034.1 x 10^{+3} Pa for 2 h -vacuum: 698.5 mm for 1/2 h -treating temperature: 82.2°C -post treatment heating: none
fluor chrome arsenate phenol-type a in water	Mississippi Wisconsin Louisiana Florida	2	Full cell -No additional information available
sodium pentachlorophenate in water	Mississippi, Wisconsin Louisiana Florida	2	Full cell -No additional information available
zinc chloride in water	Mississippi Wisconsin Louisiana Florida	2	Full cell -No additional information available
zinc naphthenate in No. 2 fuel oil	Mississippi Wisconsin	7	Reuping process: -initial air: 103.4 x 10^{+3} Pa- -pressure 1034.1 x 10^{+3} Pa for 1 h -vacuum: 698.5 mm for 1/2 h -treating temperature: 51.7°C -post treatment heating: 79.4°C

retention levels that may have been required in the past was not available. Scheffer and Van Kleeck (1945) demonstrated in laboratory tests that wood treated to retention levels of 6.9 and 13.0 kg/m^3 (0.43 and 0.81 lb/ft^3) zinc chloride lost resistance to decay by *Trametes serialis* under conditions that permitted loss of the fire retardant through leaching. They used a decay test in which small blocks were incubated for two sequential 3.5 months over agar in an enclosed vessel. During the first incubation, the treated blocks absorbed so much water that much of the chemical was lost as water dripped from the blocks.

Chromated zinc chloride (CZC) and fluor chrome arsenate phenol (FCAP) were formerly accepted for treatment of southern pine lumber used above ground (AWPA 1982), but were not recommended for treatment of southern pine lumber exposed in ground contact. Minimum retention levels for CZC and FCAP in above ground exposure are 7.2. and 4.0 kg/m^3 (0.45 and 0.25 lb/ft^3), respectively.

Table 3. Time to failure of first 25 perceut (quartile) and first half (median) of stakes that were pressure treated with coal tar creosote and exposed in Mississippi or Wisconsin.

Field plot location	Average retention (kg/m^3) (lb/ft^3)	Total stakes in set of 10 replicates that failed	First quartile of failure (years)	Median of failure (years)
Mississippi	67(4.2)	10	11.5	16.5
(plot 4)	128 (8.0)	8	26.9	39.9
	189 (11.8)	5	43.9	53.4+
	264 (16.5)	0	—	—
Wisconsin	68 (4.3)	10	34.1	36.1
(plot 4)	128 (8.0)	2	48.7+	48.7+
	189 (11.8)	0	—	—
	261 (16.3)	1	52.1+	52.1+

Table 4. Time to failure of first 25 percent (quartile) and first half (median) of stakes that were pressure treated with copper naphthenate and exposed in Mississippi or Wisconsin.

Field plot location	Average retention (kg/m^3) (lb/ft^3) copper, as metal	Total stakes in set of 10 replicates that failed	First quartile of failure (years)	median of failure (years)
Mississippi	0.19 (0.012)	10	11.8	15.8
(plot 07)	0.46 (0.029)	10	21.8	21.8
	098 (0.061)	10	21.8	26.8
	1.31 (0.082)	8	24.8	30.8
Wisconsin	0.19 (0.012)	8	21.0	26.0
(plot 07)	0.43 (0.027)	4	34.0	49.0+
	0.98 (0.061)	5	26.3	37.0+
	1.34 (0.084)	3	41.6	42.6+

RESULTS

Data presented in Figures 1 to 4 and Tables 3 to 6 allow visual estimation of quartiles and median service life and distribution of failures about the respective medians.

A clear dose–response effect is shown with creosote, wherein increased retention levels resulted in increased years of survival. Increased retention yields increased in both first quartile and median failure times (Table 3). Similarly, evidence of a dose-response relationship is shown with sodium pentachlorophenate in three locations. although it is most obvious only in the Wisconsin and Mississippi plots (Figure 1).

With copper naphthenate, there is a marked increase associated with the presence of a small amount of preservative. Increases in preservative retention beyond that point often do not yield a proportionate gain in quartile or median values. Of these two population parameters, the first quartile values seem less influenced by additional preservative than is the median survival time. The latter sometimes benefits from the increased retention when the quartile does not (Tables 4,5). For those stakes treated with copper naphthenate and exposed in Mississippi (Table 4), there seems to be little gain in the first quartile with retention levels greater than 0.46 kg/m^3 (0.029 lb/ft^3) copper as metal. This retention is at the lower bounds of threshold values reported for copper naphthenate (De Groot et al. 1988). Quartile values for stakes treated above that retention with copper naphthenate and exposed in Wisconsin were quite variable, with a two-fold gain in service life occurring at a four-fold increase in retention to 1.34 kg/m^3 (1.3 lb/f?).

With the wood preservative copper formate (Table 6), the median survival time, but probably not the first quartile of stakes treated with 0.48 kg/m^3 (0.03 lb/ft^3) elemental copper, was significantly greater than that of the untreated controls. Doubling the retention of copper from 0.48 to 0.96 kg/m^3 (0.03 to 0.06 lb/ft^3) quintupled the first quartile, but only increased the median slightly more than three-fold. The increments in first quartile and median values from 0.96 to 1.92 kg/m^3 (0.06 and 0.12 lb/ft^3) copper as metal, however, were not proportionate to the two-fold increase in retention.

Exposure sites have a marked effect upon service-life patterns of stakes treated with inorganic waferborne systems, zinc chloride, CZC, FCAP, and sodium pentachlorophenate (Figures 1 to 4). With zinc chloride, CZC, FCAP, and sodium pentachlorophenate, the exposure site at Boglusa, Louisina, was most severe. There, survival times for stakes treated to the respective lowest retention levels were sometimes significantly less than those of comparable retention levels in other locations. With sodium pentachorophenate, the service life of stakes at the highest retention in Louisiana was also significantly less than that of stakes at the highest retention in Wisconsin, Mississippi, and Florida.

For stakes treated with sodium pentachlorophenate, the range of failure times at the lowest three retention levels was almost twice as great in Mississippi than in other plots. The range in failure times within their respective retention levels for stakes treated with ECAP and zinc chloride was also greater in Mississippi than at the other locations. For stakes treated with zinc chloride and exposed in Wisconsin, the distribution of failure about the median was different from that of stakes in other plots in that the range of failures was small (±1 to 2 years) for all retention. levels.

Among these four systems, the greatest absence of a dose-response relationship between retention and longevity of treated material was with zinc chloride. This was true for all locations.

For stakes treated with CZC, there was no real gain in median life span of the population of treated stakes greater than 12.3 kg/m^3 (0.76 lb/ft^3) At all locations, the median value for stakes treated to 16.3 kg/m^3 (1.02 lb/ft^2) was not greater than that of stakes treated to at least one of the lower retention levels. The range of stake failures about the median was somewhat reduced by increased retention. A marked in service life was observed in the Mississippi plot with the increase of retention from the lowest level, 7.8 kg/m^3 (0.49 m/ft^3), to the next retention level, 12.2 kg/m^3 (0.76 lb/ft^3), but this was not evident in the other plots. Overall, for each respective combination of retention by location, the median service was greater than or equal to the average service life.

For stakes treated with FCAP, a large dose–response relationship between retention and median service life was observed only in the Mississippi plots. The median of stakes treated at the lowest retention was less than that of the higher retention levels at each location. For plots in Wisconsin, Louisiana, and Florida, the dose-response relationship between retention levels of 3.2 and 4.8 kg/m^3 (0.2 and 0.3 lb/ft^3) was greater than that between stakes treated to 4.8 and 9.6 kg/m^3 (0.3 and 0.6 lb/ft^3). At those three plots, only a difference of one to several years separated both average and median population life times at the two higher retention levels.

A unique feature in the service-life distribution patterns for stakes treated with sodium pentachlorophenate is the reduced spread in failure times about the median value that occurs at the highest retention level. The time between first and third quartiles of stake failures was the least at the highest retention of 15.8 kg/m^3 (0.99 lb/ft^3) at all locations. This reduction in distribution of failures about the median seemed to be an abrupt change between the last two retention levels rather than a progressive reduction in distribution from lowest retention to highest. A dose–response relationship for the median life of stakes treated with sodium pentachlorophenate is shown in plots in Mississippi, Wisconsin, and Louisiana. Median values tended to approximate the mean or were greater than the mean.

Table 5. Time to failure of first 25 percent (quartile) and first half (median) of stakes that were pressure treated with catalytic gas-based oil plus copper naphthenate and exposed in the Harrison Experimental Forest in southern Mississppi (plot 20).

Treatment	Average retention (kg/m^3) (lb/ft^3) treating solution [kg/m^3 (lb/ft^3) copper as metal]	Total stakes in set of 10 replicates that failed	First quartile of failure (years)	Median of failure (years)
Untreated controls		10	1.7	1.7
No. 2 fuel oil		10	3.1	4.7
Catalylic gas-base oil	64 (4.0)	10	5.7	7.6
Catalytic gas-base oil with copper naphthenate (15:1, CGB:8.0% CuN) = treating solution of 0.5% copper metal treating solution	67 (4.2) [0.33 (0.02) copper as metal]	10	12.6	13.7
Catalytic gas-base oil with copper naphthenate(9.6:1. CGB:8.0% CuN) = treating solution of 0.75% copper metal	70 (4.4) [5.0(0.003) copper as metal]	8	13.6	19.7

Table 6. Time to failure of first 25 percent (quartile) and first half (median) of stakes that were pressure treated with copper formate and exposed in the Harrison Experimental Forest in southern Mississippi (plot 47)

Average retention (kg/m^3) (lb/ft^3) copper as metal	Total stakes in a set of 10 replicates that failed	First quartile of failure (years)	Median of failure (years)
Untreated controls	10	3	3.0
0.48 (0.03)	10	5	8.0
0.96 (0.06)	10	27.0	27.0+
1.44 (1.09)	5	22.5	37.0+
1.92 (0.12)	3	32.0	32.9+

DISCUSSION

Dose-response relationships in terms of increased longevity per unit increment of chemical treatment (retention) seem to be more directly linked in organic systems than in organo-metal or inorganic systems. This is especially true for creosote. It is this type of linkage that permitted the development of performance-based preservative specifications from test data (Huber et al. 1960).

The threshold values, as determined in laboratory soil-block tests, may have value in estimating for some metal-organic and inorganic systems the upper bounds of a proportionate dose–response effect in ultimate field durability. Retention levels above those values do not seem to yield proportionate gains in median survival time and have even less, if any, effect on increasing the longevity of the first quartile of treated members to fail.

If this ultimately proves to be an underlying principle of wood protection by these systems, then a key objective of accelerated laboratory investigations should be to define the upper limit of the dose–response curve for candidate systems. Then, verification of the laboratory-defined dose–response pattern should become a primary component in long-term evaluations in field trials.

The survival time of stakes treated with copper formate was surprising, given the absence of threshold values as determined in laboratory soil-block studies. Whether this reflects lack of contact of the treated stakes with copper-tolerant decay fungi, overemphasis of copper tolerant fungi in laboratory studies, inadequate design of either the laboratory or field evaluations, or a real effect was not determined.

Even though there are differences between locations for the four preservatives that were tested in three neotropical sites and one temperate site. there seems to be a pattern of very minimal response between increased dose (retention) and response (increased service life in years) of stakes treated with the more mobile systems, such as zinc chloride, CZC, and, FCAP.

The lack of a distinctive dose–response relationship with the more mobile treatments indicates that the governing factors are time or rate limited rather than dose limited. The absence of a marked difference between the temperate, drier site in Wisconsin and the results in warmer, wetter southern sites of Louisiana, Mississippi, and Florida and the fact that differences between locations in the southern United States are sometimes greater than those between the southern states and Wisconsin implies that site-specific, edaphic factors are perhaps more important than general climatic parameters. It is these factors that contribute to binding of the eluted material near the stake or govern rate of movement of leached chemical components away from the stake. Gross climatic differences would seemingly govern the rate reactions of microorganisms that degrade the wood.

If there is a relationship between performance in ground contact and performance of materials treated to comparable retention and exposed above ground, we may infer that those past minimum retention levels for CZC and FCAP did not provide comparable protection of treated-wood products in service. Materials treated with ECAP to a minimum mention of 4.0 kg/m^3 would likely perform in ground contact for at least 10 years, with only occasional failures at some locations. In contrast, southern pine materials treated with CZC to a minimum retention of 7.2 kg/m^3 and exposed in ground contact could be expected within 10 years to show some failures (some within 2 years) at all locations and loss of nearly half the population in environments equivalent to that in Louisiana.

This also questions whether or not field experimentation should be designed to test for differences among the initial 5 or 10 percent of the treated items that fail. similarly, criteria that are keyed to acceptance of a candidate system that performs equivalent to some level of a “reference” treatment when the “reference” is showing some percentile of failure assumes a direct relationship between increased retention (dose) and response (longevity of treated product). That relationship should be demonstrated in laboratory testing before the assumption of its existence is implemented. If that relationship does not exist, i.e., there is an upper bound to field efficacy regardless of retention, then accepting a candidate preservative on the hypothesis that increased retention ensures increased performance could be fallacious.

Regarding laboratory test procedures for preservative efficacy, the American-type soil-block tests (ASTM 1996; AWPA 1996c), in which the preservative-treated assay block is challenged with fungi growing in soil, are regarded as being more severe than the European agar-block tests in which the assay black is challenged with fungi growing on agar (Cockroft 1974; Richards and Addoms 1947). In the soil-block tests, the type of soil used can affect the amount of decay caused in untreated wood by either brown- or white-tot fungi (Amburgey 1978). However, Duncan (1953) concluded that there is little evidence that difference in soil nutrient levels will change threshold values in such tests. Protocols for leaching blocks prior to exposing them to decay fungi are included within these standard tests, but those protocols still operate within a fixed time domain. We anticipate that a series of tests conducted at different times in different laboratories or with different assay fungi would yield a range of threshold values. This has happened in soil-block tests using copper naphthenate (De Groot et al. 1988).

With some of these systems that were field-tested, the location effect seemed to have more importance than the dose–response consideration. The soil-block referenced in this paper and similar laboratory tests do not give clues about potential location-dependent differences in long-term viability of the treated product. Other approaches need to be taken to gain some predictive capacity for long-term performance in different environments.

In this study, we analyzed failure times. In evaluating the performance of treated products, you should also look at patterns of change in products prior to absolute failure. The question as to whether products treated with similar distributions have similar or different degradation patterns prior to failure should also be addressed.

CONCLUSIONS

Dose-response relationships in terms of increased longevity in the field per unit increment of chemical treatment (retention) seem to be more directly linked in organic systems, especially creosote, than in inorganic systems or in organo-metal systems. With creosote, increased preservative retention levels net increased product longevity in the field. With inorganic systems, retention levels above a certain upper bound apparently do not yield proportionate gains in increased durability.

Dose-response effects of metal-organic preservatives may reflect patterns seen with inorganic systems. With copper naphthenate, an upper bound of response that is little affected by increased dose seems apparent. The actual performance life of products treated with copper naphthenate is strongly influenced by location and the type of carrier used. With copper formate, demonstrated field durability was not anticipated on the basis of soil-block tests, which failed to determine a threshold value for that preservative. We conclude that the experimental designs of laboratory tests should be rigorous enough to describe dose–response effects of candidate preservatives if those tests are going to have relevance to the prediction of field performance of metal-organic preservatives.

We also submit that an additional emphasis area of accelerated testing should be in prediction of location effects. Location-dependent factors can have a major impact on resultant longevity of treated products. Singular, dose-response laboratory tests do not provide information on potential location effects.

we encourage others to examine their long-term data sets on field trials with treated-wood products to learn whether our observations reflect a general principle or are unique to the few data sets to which we had access.

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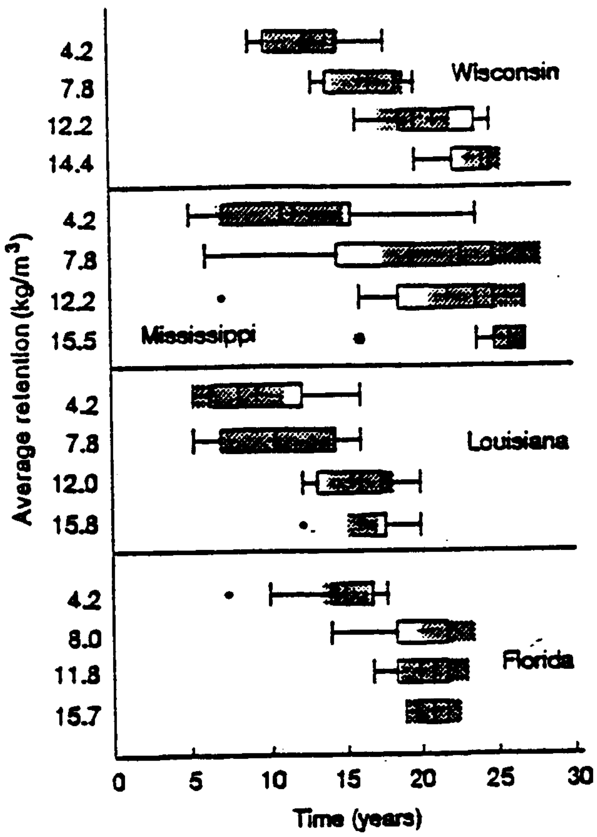

Figure 1. Service-life distributions of stakes treated with sodium pentachlorophenate.

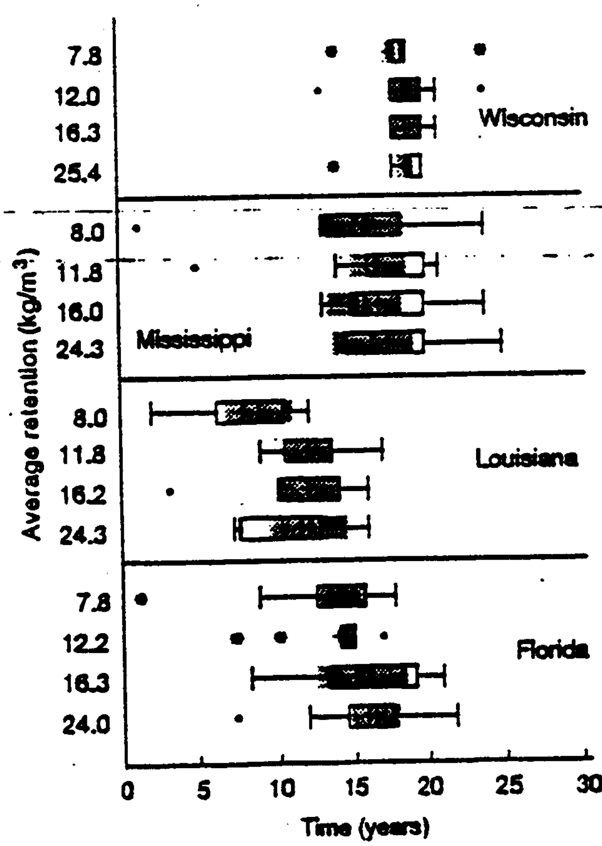

Figure 2. Service-life distributions of stakes treated with zinc chloride.

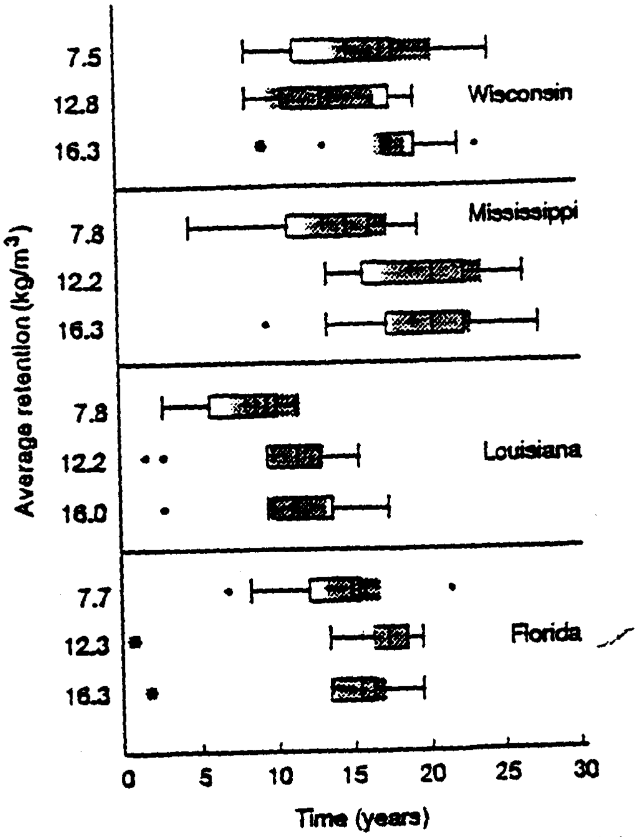

Figure 3. Service-life distributions of stakes treated with chromated zinc chloride (CZC).

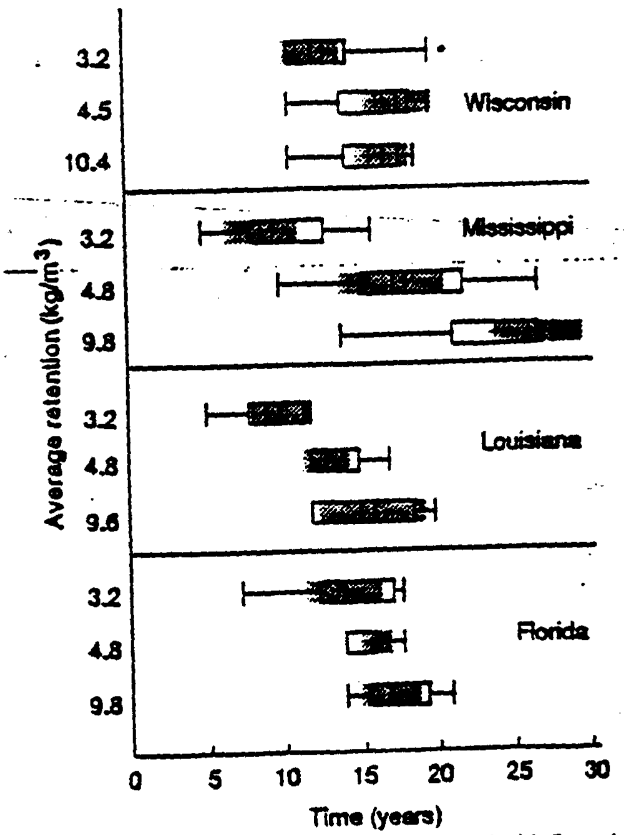

Figure 4. Service-life distributions of stakes treated with fluor chrome arsenate phenol-type A (FCAP-A).