

The Evaluation of Wood Preservatives

Part II

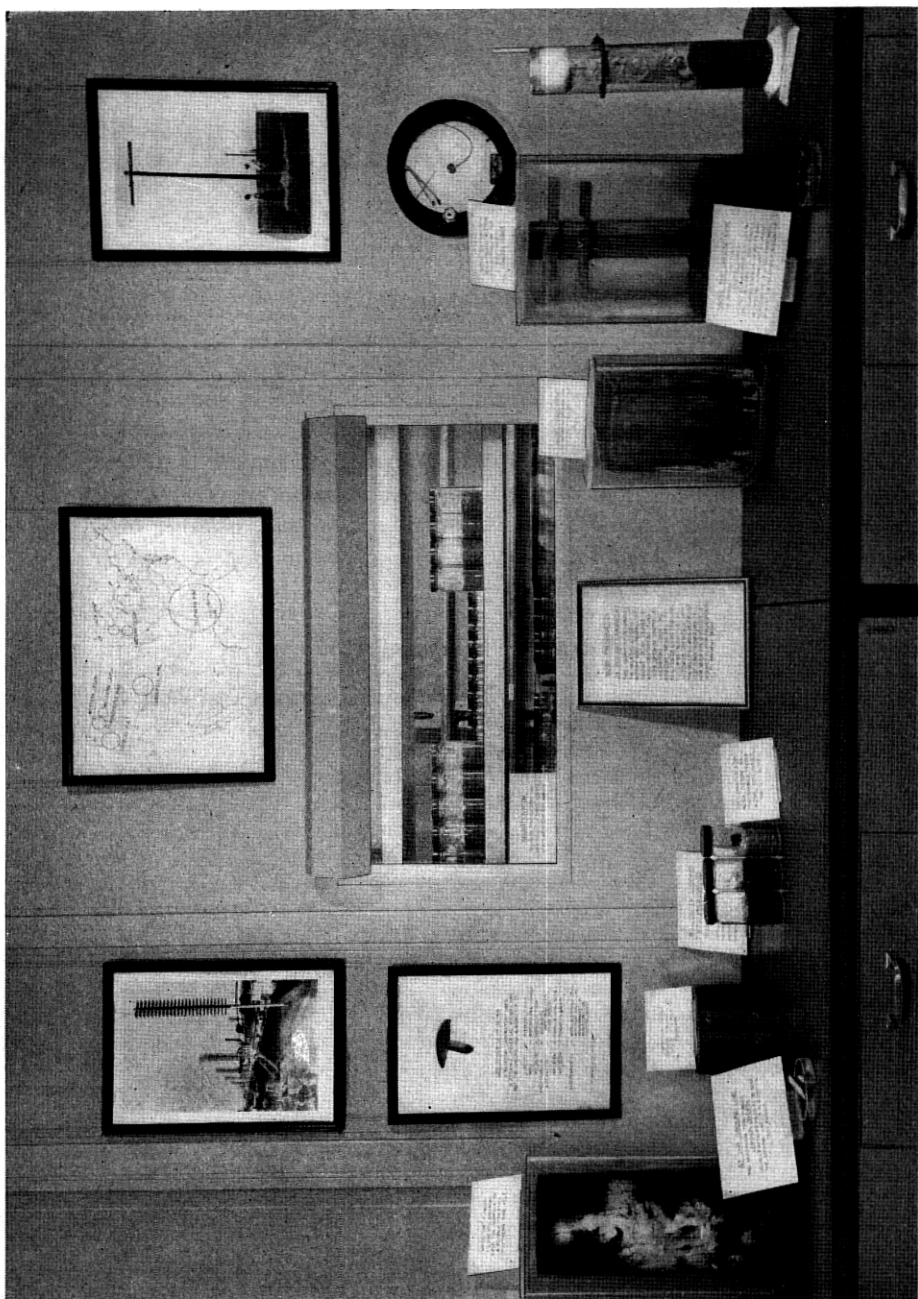
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This paper offers a review and interpretation of laboratory and field experiments aimed at determining the necessary protective threshold quantities of wood preservatives. It details the procedure followed in the soil-block tests at Bell Telephone Laboratories, Incorporated. Discussion of specific criticisms of the techniques involved and replies to these criticisms are included. The paper also presents for the first time a correlation of the results obtained from soil-block culture tests, outdoor exposure tests on stakes and on pole-diameter posts as well as pole line experience.

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EVALUATION BY TREATED $\frac{3}{4}$ -INCH SOUTHERN PINE SAPWOOD STAKES IN TEST PLOTS

Rating the Condition of the Stakes

One of the general and unavoidable difficulties in experiments involving exposure of small specimens in test plots is arriving at a measure of the inspector's judgment of the condition of the individual specimens at each inspection period. Bell Laboratories' investigators have used a system of numbers beginning with 10 as the highest, and running down in single steps to 0, to define the various gradations of destruction shown in the specimens as they pass from perfectly sound to the state of "failed" units. This system has been considered by some as slightly cumbersome; but it is a truly effective method of depreciation rating in a continuous series of inspections. In such a series any minor errors of judgment in one season can be corrected in the next. Slow depreciation can be recorded in the upper ratings until progressive destruction becomes clearly evident. Most observers of test plot experiments on treated wood specimens use a series of five numbers for five condition categories, about as follows:

10.0 Sound—no decay.

7.5 Surface soft—suspicious of decay.

5.0 Slight—positive decay.

2.5 Severe—deep decay.

0.0 Failed—almost complete loss of strength.

Some, like Rennerfelt⁸⁸ for example, use this system upside down, with 0 for no decay, and 10 for failure. The writer has proposed⁸⁵ the use of a new 5-number depreciation system, with the same definitions, based on the logarithms of the above 5 figures, and rounded off to 10, 9, 7, 4 and 0 respectively. This simplifies the Bell Telephone Laboratories' 11 division, 10-0, system while retaining the advantage of slow depreciation at first; and at the same time it avoids the sudden, and in the writer's opinion, unjustified drop from 10 to 7.5 in the arithmetic series for suspicious-of-decay specimens. In the following presentation and inter-

pretation of the behavior of some of the $\frac{3}{4}$ -inch stakes in the Gulfport test plot the recorded per cent condition of the stakes at any one inspection period has been translated into terms of the proposed 5-number log base system.

The stakes were carefully sawed and planed units, $\frac{3}{4}$ -inch square in cross-section.^{12, 69} Before treatment the stakes were selected so that they would represent the normal distribution of density in the material available. Table IX shows the analyses of the four different creosotes used to treat the stakes in respective 4- and 8-pound groups. Both empty-cell and full-cell treatments were employed. The full-cell treatments were made with toluene as a diluent in order to provide more uniform and lower controlled retentions in the treated specimens. The empty cell specimens were sorted after treatment to retain the middle group of retentions, with a view to eliminating as far as practicable some of the factors in the empty-cell treatment variation. All of the stakes were treated between March 11 and March 26, 1941, and they were all placed in the plot in the approximate period from April 8 to April 22, 1941. The distribution of retentions in the 8-pound stakes set out in the Gulfport plot are shown in Table X, along with data on average retention, standard deviation, and coefficient of variability.

TABLE IX — ANALYSES, WATER-FREE BASIS, OF FOUR CREOSOTES
USED IN TREATING $\frac{3}{4}$ -INCH SOUTHERN PINE
SAPWOOD STAKES
1941 series; Gulfport test plot

Creosote BTL No.....	5283	5286B	5286A	5285A
Specific gravity, 38/15.5°C.....	1.055	1.053	1.068	1.111
Distillation, per cent, cumulative				
To 210°C.....	2.4	4.6	5.1	0.5
210-235.....	13.2	22.4	20.3	4.2
235-270.....	38.1	49.3	41.1	18.8
270-300.....	51.1	60.5	50.0	28.7
300-315.....	58.4	65.6	53.5	33.1
315-355.....	81.1	80.7	67.3	53.2
Residue.....	18.8	18.6	32.5	46.7
Total.....	99.9	99.3	99.8	99.9
Sulph. res., gm/100 ml.....	3.4	1.6	0.7	0.7
Tar acids, gm/100 ml.....	7.8	5.7	4.0	4.0

TABLE X—DISTRIBUTION OF RETENTIONS,* LB/CU FT AT
TREATMENT, OF FOUR CREOSOTES, 8 LB EMPTY-CELL
(EC) AND FULL-CELL (FC) GROUPS

¾-inch southern pine sapwood stakes; 1941 series; Gulfport test plot.

Creosote, BTL No.	5283		5286B		5286A		5285A	
lb/cu ft	EC	FC	EC	FC	EC	FC	EC	FC
6.4	—	—	2	—	2	—	2	—
6.5	—	—	—	—	—	2	—	—
6.6	—	—	4	—	6	2	2	—
6.7	—	—	—	—	—	2	—	—
6.8	—	—	—	2	—	—	4	—
6.9	—	—	—	—	—	2	—	—
7.0	4	2	2	—	—	—	2	—
7.1	—	1	—	—	—	2	—	2
7.2	6	3	8	—	4	4	—	2
7.3	—	2	—	2	—	2	—	6
7.4	6	4	4	—	2	4	6	2
7.5	—	—	—	4	—	2	—	2
7.6	4	2	4	—	8	6	2	3
7.7	—	2	—	2	—	—	—	3
7.8	—	4	2	4	4	—	4	4
7.9	—	6	—	6	—	—	—	7
8.0	2	3	2	—	4	—	6	2
8.1	—	1	—	4	—	—	—	2
8.2	—	2	—	4	—	2	—	1
8.3	2	2	4	2	—	2	—	5
8.4	—	—	—	2	—	—	—	2
8.5	4	—	—	—	4	4	6	—
8.6	—	1	—	—	—	—	—	1
8.7	—	—	—	2	4	2	—	—
8.8	—	1	2	—	—	—	—	—
8.9	2	4	—	2	2	2	2	6
9.1	2	—	—	2	—	—	2	—
9.3	—	—	4	2	—	—	2	—
9.4	—	1	—	—	—	—	—	—
9.5	8	—	—	—	—	—	—	—
9.6	—	1	—	—	—	—	—	—
9.7	—	1	2	—	2	—	—	—
9.8	—	1	—	—	—	—	—	—
12.5	—	—	2	—	—	—	—	—

n

	40	44	42	40	42	40	40	50
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Average Retention lb/cu ft

	8.12	8.02	7.97	8.07	7.76	7.59	7.79	7.91
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Standard Deviation

	0.913	0.723	1.288	0.514	0.839	0.694	0.758	0.523
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Coefficient of Variation

	11.24	9.01	16.16	6.37	10.81	9.14	9.73	6.61
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* All retentions were calculated from weights before and after treatment. The full cell (FC) treatments were made with toluene-creosote solutions.

TABLE XI — INSPECTION RATINGS

$\frac{3}{4}$ -inch southern pine sapwood stakes in test 6 and 7 years; 1941 series; Gulfport test plot.

Creosote BTL No.	Treat- ment	n	Average retention at treatment lb/cu ft	Test period years	Number and per cent of specimens rated					Average per cent condition
					10	9	7	4	0	
5283	EC	40	8.12	6	29 72.5	8 20.0	1 2.5	— —	2 5.0	92.2
				7	17 42.5	17 42.5	2 5.0	1 2.5	3 7.5	85.6
	FC	44	8.02	6	16 36.4	22 50.0	1 2.3	3 6.8	2 4.6	85.7
				7	2 4.6	19 43.2	14 31.8	3 6.8	6 13.6	68.4
5286B	EC	42	7.97	6	25 59.5	10 23.8	4 9.5	1 2.4	2 4.8	88.6
				7	9 21.4	14 33.3	10 23.8	4 9.5	5 11.9	72.0
	FC	40	8.07	6	10 25.0	27 67.5	1 2.5	1 2.5	1 2.5	88.5
				7	1 2.5	19 47.5	18 45.0	— —	2 5.0	76.8
5286A	EC	42	7.76	6	25 59.5	15 35.7	1 2.4	1 2.4	— —	96.5
				7	8 19.0	32 76.2	— —	— —	2 4.8	87.6
	FC	40	7.59	6	10 25.0	27 67.5	1 2.5	1 2.5	1 2.5	89.0
				7	8 20.0	20 50.0	8 20.0	1 2.5	3 7.5	80.0
5285A	EC	40	7.79	6	36 90.0	4 10.0	— —	— —	— —	99.0
				7	29 72.5	9 22.5	— —	2 5.0	— —	95.0
5285A	FC	50	7.91	6	35 70.0	12 24.0	— —	2 4.0	1 2.0	93.2
				7	17 34.0	23 46.0	4 8.0	2 4.0	4 8.0	82.6

Depreciation Curves for $\frac{3}{4}$ Inch Stakes

Under the conditions at Gulfport the depreciation curves for the creosotes — particularly the low residue oils — show an increased downward pitch at the 6th to 7th year of exposure. The relative proportion of the stakes rated respectively at 10, 9, 7, 4 and 0 at the 6- and 7-year inspections, are shown by number and per cent in Table XI. The change within the one year interval is particularly striking in the 10 and 9 columns. The rating and the distribution of retentions of creosote 5283, empty-cell treatment, at 6 and 7 years, respectively, are shown in Figs.

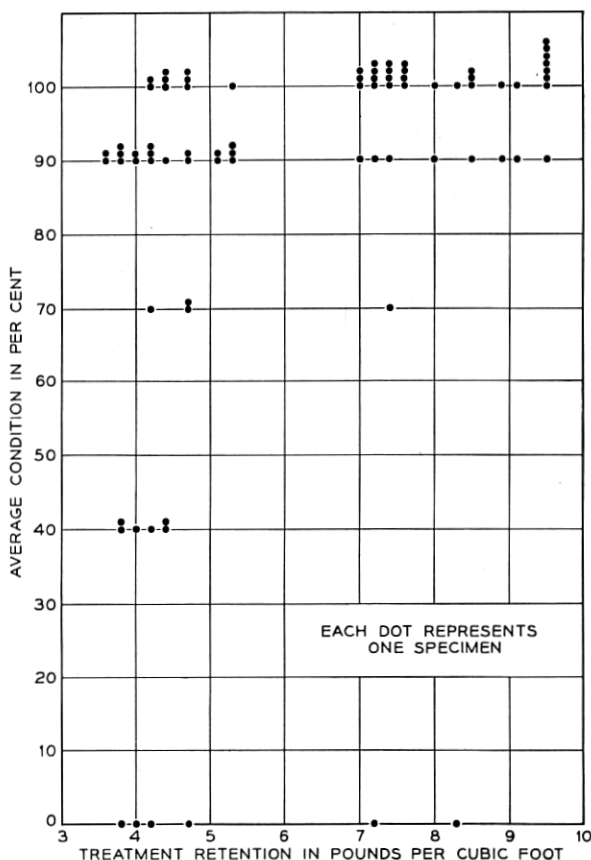


Fig. 15—Distribution of ratings in relation to retention by weight at treatment; creosote No. 5283, $\frac{3}{4}$ -inch southern pine sapwood stakes; 1941 series, empty-cell treatment; six years exposure; Gulfport test plot. See text and companion Figs. 16, 17 and 18.

creosote 5286B at 9.3, 9.7 and 12.5 pounds (see Table X) apparently have not operated to increase the average life of the group treated with this oil as much as appears to be the case in the group treated with creosote 5283. The difference in behavior at the 6-7 year interval of the stakes that were treated by a full cell process with treating solutions made by dissolving the creosote in toluene is even more marked than it is in the empty cell groups.

The average per cent condition of the stakes over the 9-year test period up to 1950 is shown in Table XII. Data are included on the number of stakes in each lot, and on the average treatment retention in

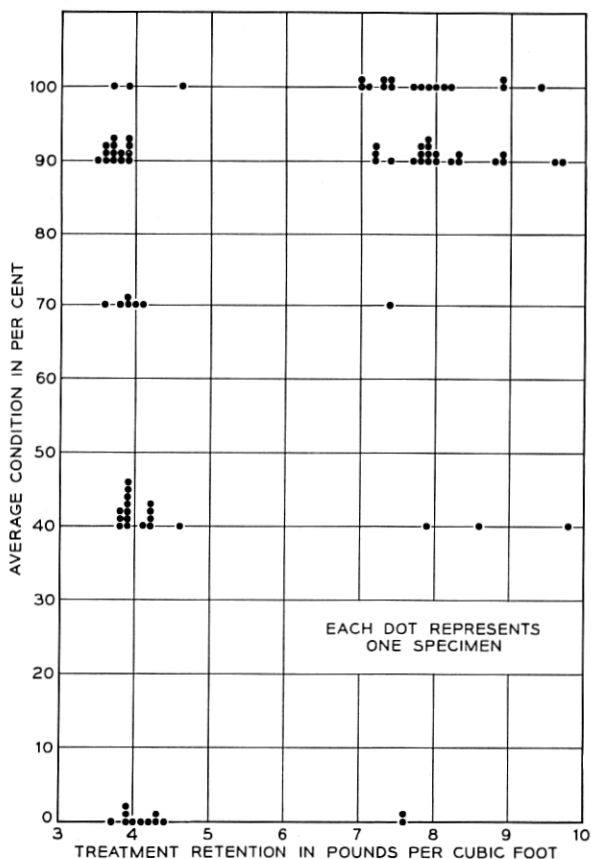


Fig. 17—Distribution of ratings in relation to retention by weight at treatment; creosote No. 5283, full-cell treatment (toluene dilution), six years exposure.

pounds per cubic foot for the respective groups. Two groups of stakes treated with greensalt K^{74, 79} at 0.57 and 1.17 pounds per cubic foot, respectively, are included in the table for comparison.

Depreciation curves for the 4- and 8-pound groups for the four creosotes are shown in Figs. 19, 20, 21 and 22. A depreciation curve for greensalt K specimens treated with 1.17 pound per cubic foot is included in Fig. 19.

Estimating Threshold Retentions and Average Life

In presenting the following discussion of a theoretical approach to the estimation of threshold retentions and average life no attempt has been

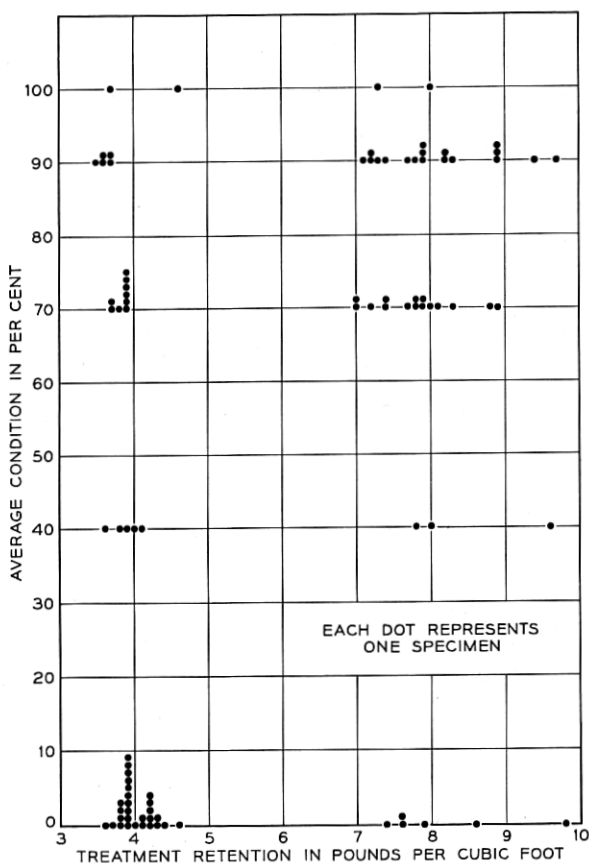


Fig. 18—Distribution of ratings in relation to retention by weight at treatment; creosote No. 5283, full-cell treatment (toluene dilution), seven years exposure.

made to separate the possible effects of the attack by different fungi or combinations of such fungi. The basic data are the figures reported by the inspectors of the stakes; and data on the actual organisms involved are very difficult — if not impossible — to obtain at the time of inspection. For the present purpose then, any differences in rate of decay by different organisms or in different parts of the test plot are all blanketed under the per cent condition averages.

TABLE XII — AVERAGE PER CENT CONDITION OF $\frac{3}{4}$ -INCH SOUTHERN PINE SAPWOOD STAKES TREATED WITH FOUR CREOSOTES, AND WITH GREENSALT K

9 years in test; 1951 series; Gulfport test plot.

Creosote BTL No.	Treatment	n	Average retention at treatment lb/cu ft	Years in test					
				1	3	6	7	8	9
				Average per cent condition					
5283	EC	40	4.38	100	97	74	50	32	13
	EC	40	8.12	100	99	92	86	74	55
	FC	49	3.93	99	86	54	30	16	8
	FC	44	8.02	99	96	86	68	54	31
5286B	EC	42	4.23	100	93	67	40	21	12
	EC	42	7.97	100	100	89	72	54	40
	FC	40	4.03	100	86	56	29	10	1
	FC	40	8.07	100	97	89	77	47	32
5286A	EC	42	3.93	100	96	76	57	42	28
	EC	42	7.76	100	100	97	88	82	72
	FC	40	4.08	100	93	84	64	44	16
	FC	40	7.59	100	96	89	80	72	53
5285A	EC	40	4.15	100	99	91	75	62	53
	EC	40	7.79	100	100	99	95	92	90
	FC	51	3.92	100	94	78	61	46	24
	FC	50	7.91	100	97	93	83	76	62
Greensalt K									
	FC	99	0.57*	100	98	84	80	72	67
	FC	100	1.17	100	99	96	88	86	79

* lb/cu ft of dry salt.

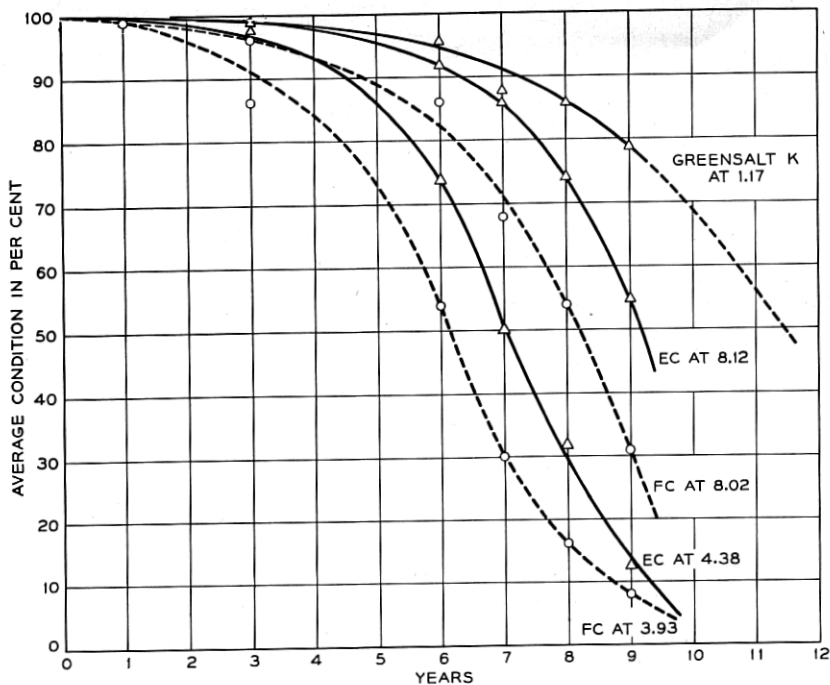


Fig. 19—Depreciation curves for $\frac{3}{4}$ -inch southern pine sapwood stakes treated with creosote, BTL No. 5283, empty-cell and full-cell (toluene dilution) processes, and with greensalt K; Gulfport test plot. See text, Tables XII-XIII, and companion Figs. 20, 21 and 22.

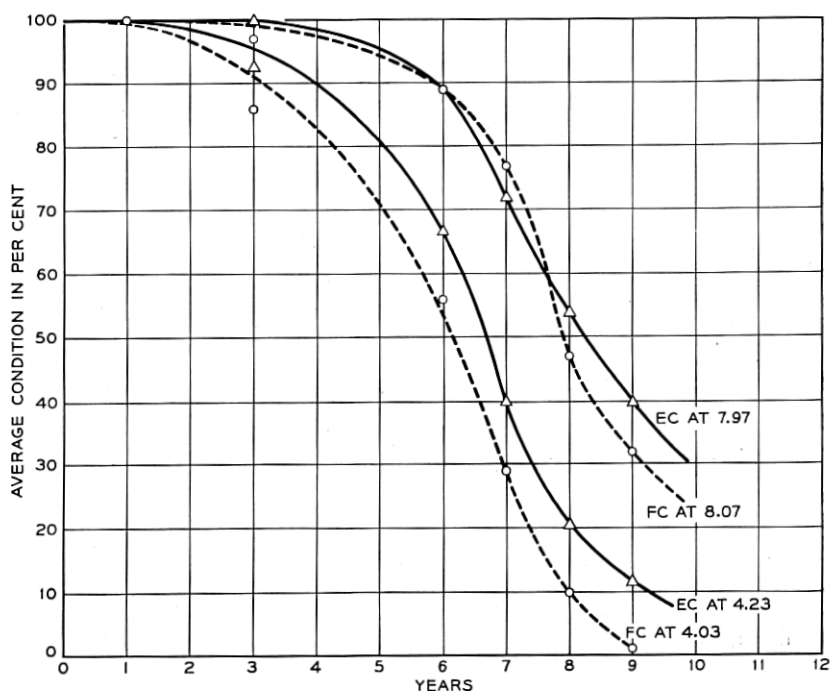


Fig. 20—Depreciation curves for $\frac{3}{4}$ -inch southern pine sapwood stakes treated with creosote, BTL No. 5286B.

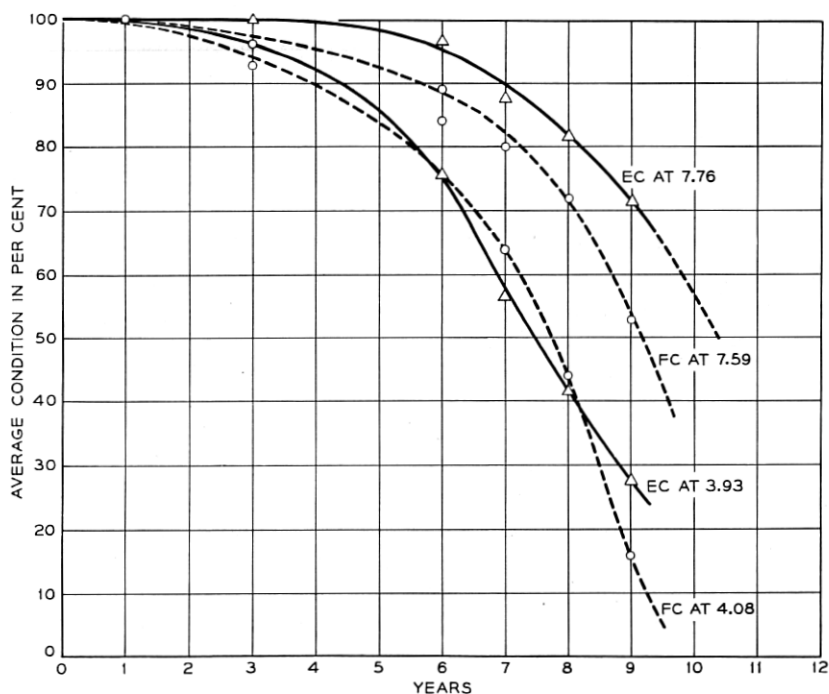


Fig. 21—Depreciation curves for $\frac{3}{4}$ -inch southern pine sapwood stakes treated with creosote, BTL 5286A.

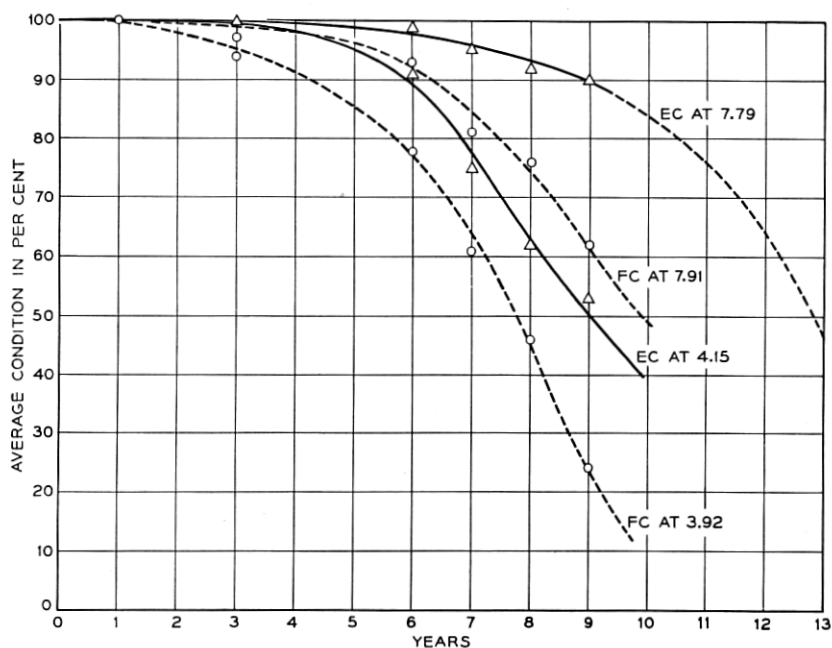


Fig. 22—Depreciation curves for $\frac{3}{4}$ -inch southern pine sapwood stakes treated with creosote, BTL-5285A.

The straight lines in Figs. 23, 24, 25 and 26 are drawn through the average points for per cent condition and retention of the respective 4- and 8-pound groups, and projected to intercept the 100 per cent condition line. The empty-cell data are represented by the solid line and the full-cell data by dashes. The threshold concentrations necessary to prevent all decay are estimated from the intersection of the gradient lines and the 100 per cent condition line. This method assumes that the relation of condition to treatment retention is linear at or near the threshold, providing the average condition points through which the lines are drawn are established by the logarithmic based rating system described. The method also resembles in a way the procedure of the Madison investigators who have used the intersection of straight lines drawn through operational losses and decay weight losses in estimating the threshold retention in creosoted blocks.³⁹ As in the case of the latter the method would probably be more precise if one had more points for average condition at average retention nearer the thresholds. At any event the system represented by Figs. 23-26 seems to be about the only one that indicates probable thresholds for these particular creosotes and these particular sets of data.

TABLE XIII — ESTIMATED THRESHOLD RETENTION AND AVERAGE LIFE

$\frac{3}{4}$ -inch southern pine sapwood stakes; (see Tables X-XII); 1941 series; Gulfport test plot.

Creosote No.	Treatment	Years in test			Average life-years*	
		3	6	7	"4 lb"	"8 lb"
		Estimated thresholds lb/cu ft*				
5283	EC	9.7	9.7	9.6	7.0	9.2
	FC	9.6	9.8	11.4	6.1	8.2
5286B	EC	8.0	9.8	11.3	6.7	8.3
	FC	9.2	9.4	10.0	6.2	7.9
5286A	EC	7.7	8.3	9.3	7.5	10.4
	FC	12.2	Indet.	11.9	7.8	9.2
5285A	EC	7.8	8.3	8.7	9.0	12.8
	FC	11.8	9.7	11.1	7.8	9.9
Greensalt K	FC (0.57)	—			10.5	
	FC (1.17)	—	1.4	2.1		11.4

* See text for additional data on method used in estimating the threshold retentions at treatment and the average life figures.

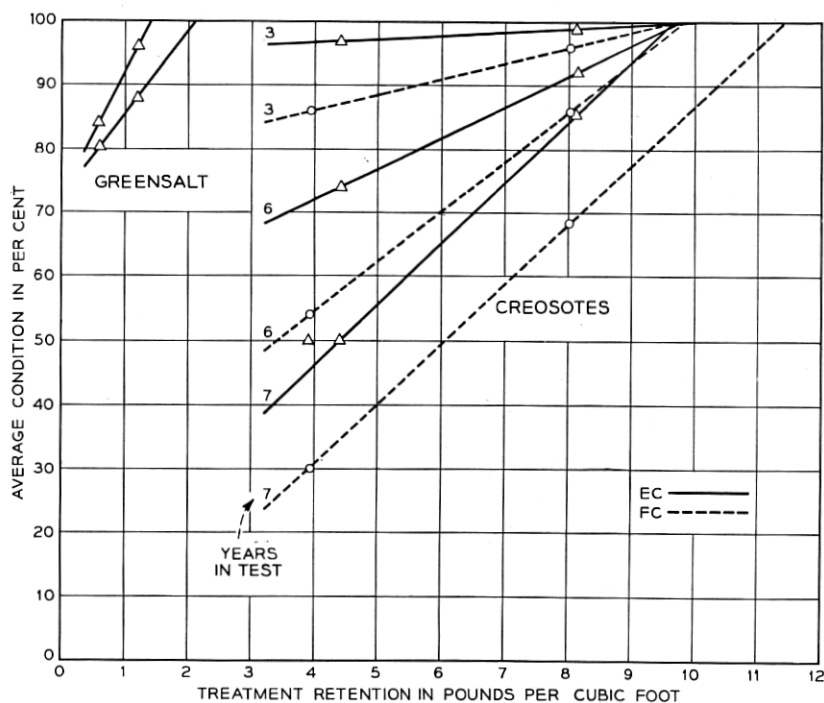


Fig. 23—Theoretical lines for estimating threshold retention for creosote No. 5283, in $\frac{3}{4}$ -inch southern pine sapwood stakes, empty-cell and full-cell (toluene dilution) treatments, and for greensalt K; Gulfport test plot. See text, Tables XI and XIII, and companion Figs. 24, 25 and 26.

The tendency for the 7-year lines to fall off to the right shows the effect of increasing decay in the 8-pound group. In the higher residue creosotes the earlier decay of the toluene dilute 8-pound treated specimens — that is, specimens that were treated below the threshold retention — tends to pull the lines so far down as to spoil their usefulness as tools for estimating thresholds. Obviously the slopes of the lines will be influenced by the depreciation rating of the 4-pound as well as the 8-pound groups. Furthermore it would appear that the utility of the specimens treated with toluene-creosote solutions for estimating thresholds does not extend much beyond the 6th year of exposure under conditions such as those prevailing at the Gulfport plot.

The estimated thresholds at the 3-, 6- and 7-year inspection periods, and the estimated average life values for the different groups are summarized in Table XIII. The average life is estimated from the intersection of the depreciation curves and the 50 per cent condition lines. There

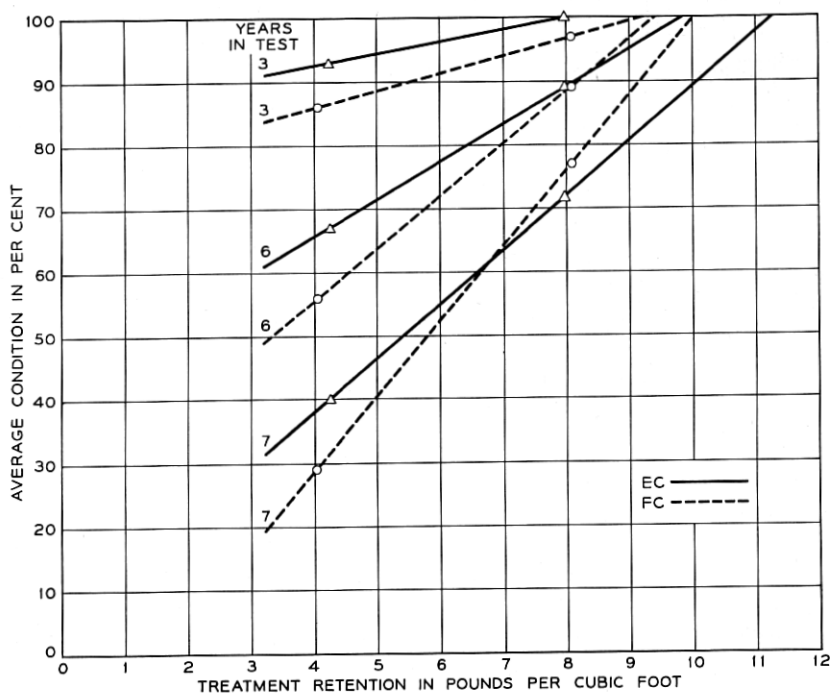


Fig. 24—Theoretical lines for estimating threshold retention for creosote No. 5286B; 3/4-inch southern pine sapwood stakes.

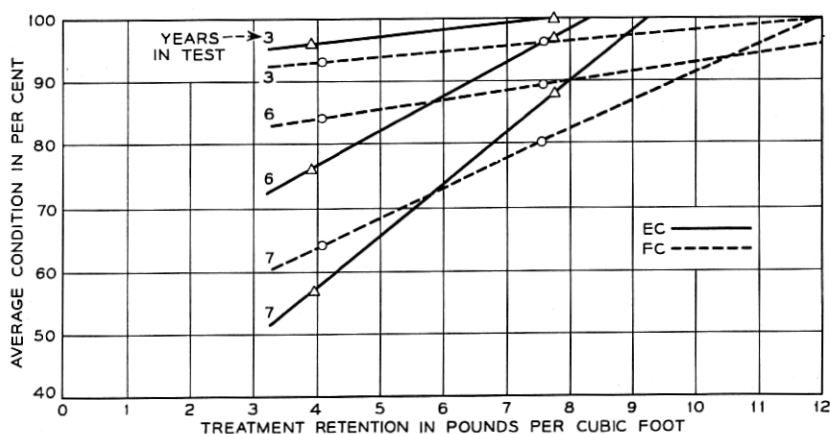


Fig. 25—Theoretical lines for estimating threshold retention for creosote No. 5286A; 3/4-inch southern pine sapwood stakes.

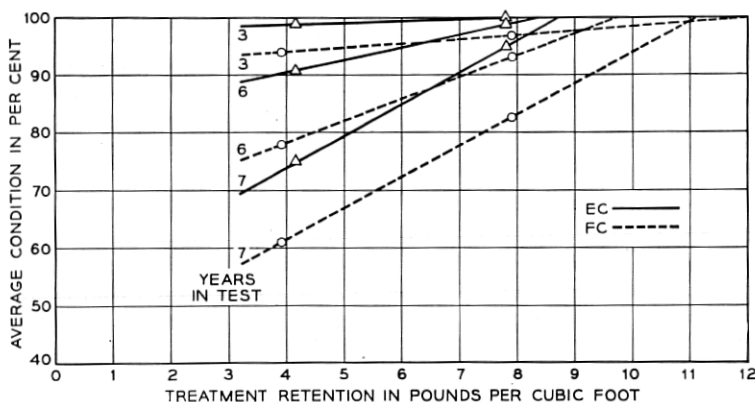


Fig. 26—Theoretical lines for estimating threshold retention for creosote No. 5285A; $\frac{3}{4}$ -inch southern pine sapwood stakes.

will inevitably be some difference of opinion as to which level to use. In the case of $\frac{3}{4}$ -inch treated stakes it is quite evident that the preservative is no longer functioning effectively if the stakes have reached a decay rating of 7 or less. In such small specimens it is questionable whether any purpose is served by allowing them to stay in the ground under the conditions at the Gulfport test plot until they practically fall over by being completely destroyed at the ground line.

Anyone who has worked with small test plot specimens will appreciate the many difficulties in the way of establishing standard procedure for determining the "failed" point or the end point of specimen life. On somewhat larger stakes Rennerfelt⁸⁸ has used a strength testing apparatus. To test the fitness of small poles in line some Associated Operating Companies have used a spring scale dynamometer which is slipped onto the base of a pike pole. In actual utility plant experience it is obvious that it is impossible to wait for the complete decay of the wood unit. Elaborate tables have been worked out as guides for pole line inspectors to let them know how far decay can go under given load conditions before a pole has to be removed from line. Generally speaking, such removal must occur at some period well in advance of the time that the specimen would have rotted clear through at the ground line. In the writer's opinion it might be preferable to estimate the average life for $\frac{3}{4}$ -inch stakes from the point where the depreciation curve passes downward through the 60 per cent condition line, leaving *all* the units in any given series in test until that time, except of course the stakes that may have actually rotted off earlier.

Study of Table XIII indicates clearly that both threshold and average

life estimates for any given set of small specimens will vary, depending on the time at which the estimates are made. Taking the 6-year inspection data as perhaps representing the best figures from which to make threshold and average life estimates of this kind, it appears that in the case of all of the four creosotes something more than 8 pounds of creosote per cubic foot was necessary to protect the $\frac{3}{4}$ -inch stake specimens against decay. There seems to be no material difference in the performance of creosotes 5283 and 5286B as far as the estimated thresholds are concerned. For both the empty-cell and full-cell treatments it appears to be somewhere in the neighborhood of 9.5 pounds or above. In the case of the higher residue oils 5286A and 5285A there appears to be a significant difference between ratings obtained from the empty-cell specimens and from the toluene dilute full-cell specimens. Estimates of average life from the 8-pound empty cell specimens appear to be significantly higher than estimates from the 8-pound full cell toluene-creosote specimens, except in the case of oil No. 5286B (Table XIII). The estimated thresholds for the full-cell toluene-creosote specimens lie within the same general magnitude as the retention at treatment thresholds found in the soil-block tests. (Cf. Tables XIII and XXXV).

How far one is justified in comparing straight 8-pound empty-cell treatments and 8-pound full-cell toluene-creosote treatments in $\frac{3}{4}$ -inch stakes is still not clear. It certainly cannot be done without taking into account the much greater variability in the retentions in empty cell stakes and the different but difficult to describe variations in distribution of the creosote from outside to inside, particularly in the case of empty cell treatments with high residue oil. Unless the empty-cell stakes are carefully selected within limited variations from the average retention, it has been found that the empty-cell coefficient of variability for retention in an 8-pound treatment, for example, may run as high as 35-40 per cent, against a coefficient of 8-10 per cent only for companion full-cell treatments with toluene-creosote solutions. The comparisons among the latter treatments appear to be more rational and fairer; and they may give a truer picture of effective threshold requirements.

The analysis of the stake test data here presented is intended merely to illustrate one set of procedures that may be used in the interpretation of small stake tests. The four sets of data are part of a much larger group of results that are being worked up for publication. Among the latter there are numerous lots indicating that truly protective thresholds of creosote for $\frac{3}{4}$ -inch stakes lie somewhere between 10 and 12 pounds per cubic foot, which is not far out of line with the estimates given above. Final analysis and publication will either confirm or modify such estimates.

EVALUATION BY TREATED POLE-DIAMETER POSTS IN TEST PLOTS

Reference was made in the introduction to the fact that Bell Telephone Laboratories' experience over the last quarter of a century in the evaluation of preservatives by the use of pole-diameter posts in the Gulfport test plot was reviewed by Lumsden⁷⁶ in April, 1952, before the American Wood-Preservers' Association. A supplementary analysis and interpretation of some of the same evidence is attempted in the following paragraphs.

Data for 185 of the posts, the time of placement and the number of years in test, and the general condition of the posts as of the 1950 inspection, are shown in Table XIV. The data for the individual posts of these

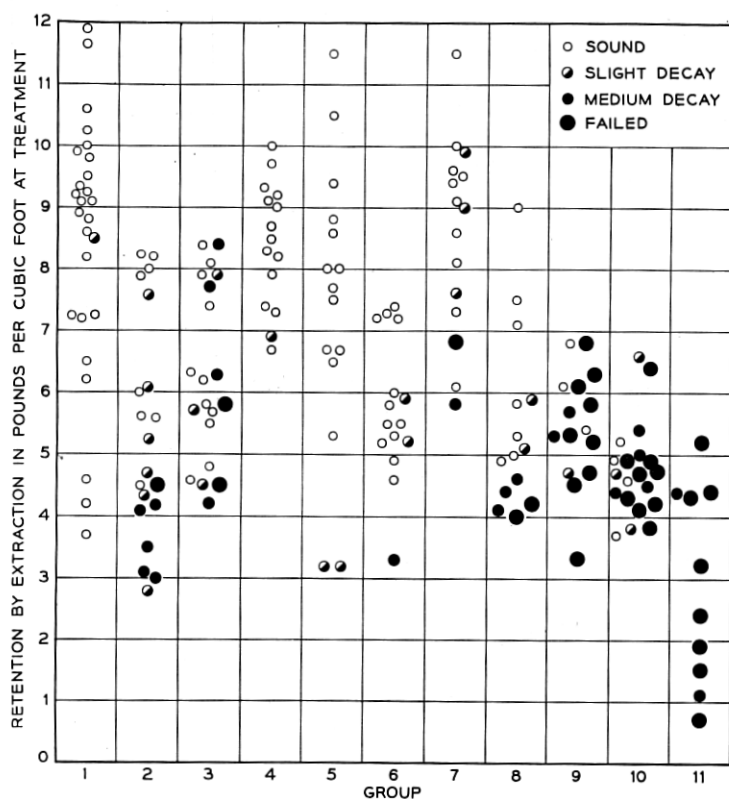


Fig. 27—The relation of creosote retention in lb/cu ft at treatment, by toluene extraction, and the rated condition of pole-diameter southern pine test posts, by post groups; 1950 inspection; Gulfport test plot. See text and Table XIV.

TABLE XIV — CREOSOTED SOUTHERN PINE TEST POSTS
Groups, years in test, and condition at 1950 inspection; Gulfport test plot.

Group*	Year placed	Years in test	n	Sound	Decaying		Failed
					Slight	Medium	
1	1931	19	26	25	1	—	—
2	1931	19	20	8	6	5	1
3	1931	19	20	11	3	4	2
4	1936	14	15	14	1	—	—
5	1935	15	15	13	2	—	—
6	1935	15	15	12	2	1	—
7	1935	15	15	10	3	1	1
8	1935	15	14	7	2	3	2
9	1935	15	15	3	1	2	9
10	1936	14	20	4	3	4	9
11	1935	15	10	—	—	2	8
Totals.....			185	107	24	22	32

* For analyses of creosotes, see Table II in Lumsden's report⁷⁶, Am. Wood Preservers' Assoc., Proc., 1952.

TABLE XV — CREOSOTED SOUTHERN PINE TEST POSTS

Relation between average treatment retentions by zones, by toluene extraction, and condition of the posts at the 1950 inspection, Gulfport test plot.

Condition	Group	n	Average retention at treatment 1b/cu ft					
			Zones, inches				Remainder of treated sapwood	Whole cross section
			Outer ¼	Next ¼	Next ½	Next 1		
Sound	5-10	64	13.1	9.9	8.5	6.4	5.2	7.4
Decaying	5	2	9.7	7.3	4.9	—	3.2	4.2
	6	3	8.8	5.5	4.8	—	4.2	4.8
	7	4	13.1	11.6	11.8	11.1	6.5	8.1
	8	5	10.9	8.7	7.4	5.0	2.9	5.8
	9	3	12.7	9.2	7.0	2.9	2.9	5.2
	10	7	10.1	5.2	3.9	3.6	3.5	4.3
	Total	24	10.9	7.7	6.5	4.3	3.9	5.4
Failed	7	1	13.7	9.2	11.7	—	4.7	6.7
	8	2	9.2	7.4	6.8	—	3.6	5.1
	9	9	13.3	10.0	6.2	3.7	3.6	5.6
	10	9	9.2	4.9	4.0	3.2	3.3	4.2
	Total	21	11.1	7.4	5.6	3.5	3.5	5.0
	Overall	109	12.3	8.9	7.5	5.5	4.6	6.5

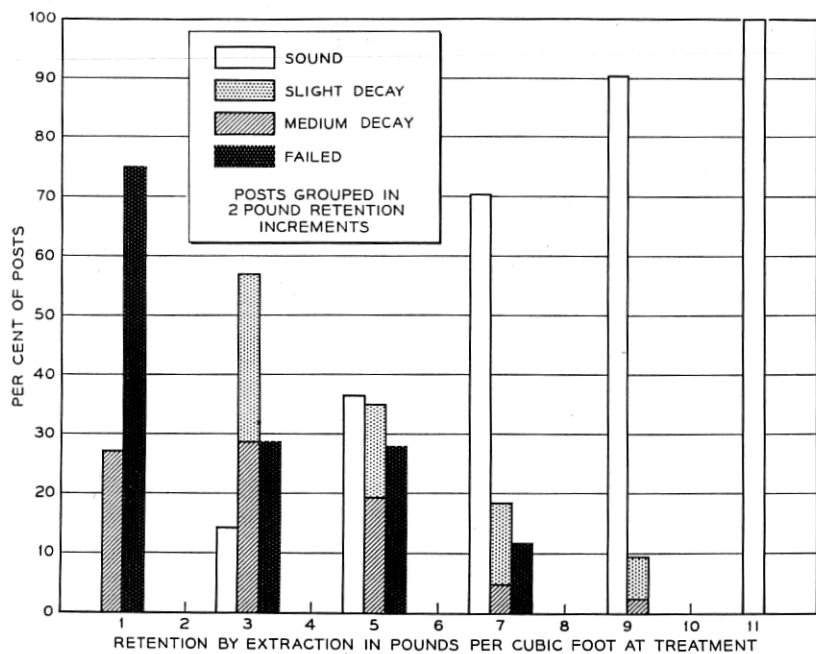


Fig. 28—The relation of creosote retention in lb/cu ft at treatment by toluene extraction, and the rated condition of pole-diameter test posts, by retention groups; 1950 inspection; Gulfport test plot. See text and Table XVI.

eleven groups are shown graphically in Fig. 27. The relation of retention by extraction at the time of treatment to the sound, decaying and failed specimens is clearly evident. All of the failures and the very great majority of the remaining decaying poles are below the 7-pound retention line. Only two cases of medium decay occur above the 7.5 pound line. It should be borne in mind particularly at this point that these retention levels represent the retentions found by extraction in the whole cross sections of the posts *as soon as practicable* after the posts were treated. The significance of these over-all retentions and of the calculated retentions in the outer one-inch layer of the posts will be discussed later. Since the data were calculated in terms of oven-dry weight and volume of the extracted wood the values may be a little high.

For Groups 5 to 10 inclusive data are available on the retention in zones, that is, in the outer quarter inch, the next quarter inch, the next half inch, the next one inch, and the remainder of the treated sapwood. These data were obtained by appropriate cutting of the samples into zones and pooling for extraction the parts that came from the same zones,

either in sectors cut from discs or in boring samples. A summary of the distribution of the retentions will be found in Table XV.

If the overall retention data for post groups 1-11 are distributed in 2-pound retention lots the evidence falls into the categories represented by Table XVI and Fig. 28. The inference is clear. One would expect a satisfactory service life if the treatment retention by extraction, based on the whole cross section, was at or above 8 pounds per cubic foot. This is perhaps over-simplification. The variations shown in Tables XIV and XV in the behavior of the posts in Groups 7 and 9, and possibly to a certain extent in Group 8, disturb what looks like reasonably straight reasoning.

In order to extend the reasoning and provide another method of interpretation, let it be assumed that the posts average 8 inches in diameter at the ground line, which is close to their actual size. Calculation of the pounds per cubic foot in the outer inch of such average posts is shown in Table XVII. The influence of posts in groups 7 and 9 on the averages is still evident. If there are differences in the efficiency of the

TABLE XVI — CREOSOTED SOUTHERN PINE TEST POSTS

Condition at 1950 inspection by retention groups; 14-19 years in test; Gulfport test plot.

Retention* lb/cu ft at treatment	n	Condition of ground section, number and per cent			
		Sound	Decaying		Failed
			Slight	Medium	
1. 10.0-11.9	10 100.0	10 100.0	0 —	0 —	0 —
2. 8.0- 9.9	42 100.0	38 90.4	3 7.2	1 2.4	0 —
3. 6.0- 7.9	44 100.0	31 70.4	6 13.6	2 4.6	5 11.4
4. 4.0- 5.9	71 100.0	26 36.6	11 15.5	14 19.7	20 28.2
5. 2.0- 3.9	14 100.0	2 14.3	4 28.5	4 28.6	4 28.6
6. 0.0- 1.9	4 100.0	0 —	0 —	1 25.0	3 75.0
Totals	185 100.0	107 57.8	24 13.0	22 11.9	32 17.3

* The distribution into retention lots was made on the basis of both weight and extraction data, depending on information applicable to the eleven groups in Table XIV.

creosotes in these groups, the differences are not at present real and tangible because they are masked by other factors, among which distribution of the preservative, and retention and penetration variables seem most important. The over-all indications are that in general one should insist on something more than 8, and probably more than 9, pounds of creosote per cubic foot, at the time of treatment, *in the outer inch* of the ground section of a southern pine pole. This, in simple terms, is in line with the conclusions reached about threshold retention requirements from the laboratory tests of creosoted $\frac{3}{4}$ -inch cubes and from test plot results on $\frac{3}{4}$ -inch stakes.

It is much harder to rate treated test posts in terms of per cent condition than it is to rate small specimens. Fig. 28 must be considered therefore as a generalization from the plot inspection data. It must be interpreted with the help of Fig. 27 and the retention by zones data in Tables XV and XVII. The reader who is at all familiar with pole line service records will recognize how much more detailed information there is for these test posts than there generally is for the ordinary pole line. Yet it is practically impossible to draw up precise, unequivocal statements about the results in certain of the post series. The conclusions must be broad.

The structure of the wood in a pole, the distribution of moisture content, the variation in density of the wood from the outside to the inside annual rings, and the distribution of creosote from the outside toward the inside all go to make up a resultant that is obviously complicated. Some of these factors have been reduced to a schematic figure (Fig. 29)

TABLE XVII — CREOSOTED SOUTHERN PINE TEST POSTS

Relation between average treatment retention in outer inch of sapwood and condition of posts at the 1950 inspection; Gulfport test plot.

Group	Average retention at treatment in outer 1 inch of sapwood					
	Sound		Decaying		Failed	
	n	lb/cu ft	n	lb/cu ft	n	lb/cu ft
5-10	64	10.14*	—	—	—	—
5	—	—	2	7.13	—	—
6	—	—	3	6.09	—	—
7	—	—	4	12.11	1	11.61
8	—	—	5	8.71	2	7.90
9	—	—	3	9.38	9	9.15
10	—	—	7	5.95	9	5.95
Totals	64	10.14	24	8.03	21	7.59

* All calculations are made on the basis of an 8-inch diameter at the point of sampling.

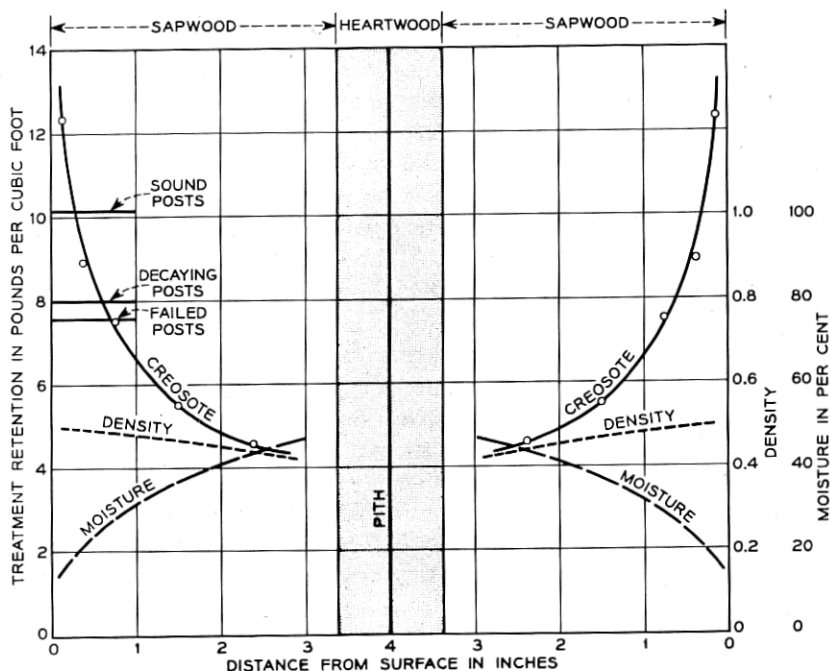


Fig. 29—Schematic diagram of a longitudinal section of a creosoted southern pine test post, showing average distribution of creosote in lb/cu ft; average density (oven-dry weight and volume as tested); average moisture content, oven-dry wood basis; and the retention levels in the outer inch of sound, decaying and failed specimens. See text.

representing a longitudinal section of an 8 inch diameter southern pine test post. The sound, decaying and failed retention levels calculated for the outer one inch (Table XVII) of such a diagrammatic post are also shown. When one considers all the variations in the wood itself and in the treatment, the site and environmental conditions where the pole is used, and the probability of the incidence of decay, the above general conclusions with respect to necessary retention seem reasonable and practicable. To refine and narrow the conclusions by repeating the post tests with the same creosotes — if one could get them — would certainly require the use of at least some posts with higher retentions and an obligatory assay of all of the treated specimens to be sure that the required retentions were actually in the wood in the right places. Apparently practical answers to questions about such required retentions can be answered very much more quickly by repeated series of soil-block tests in which many of the variables can be controlled.

EVALUATION BY POLE TEST LINES AND BY LINE EXPERIENCE; SERVICE TESTS

In 1932 a condensed report⁸⁴ of American Telephone and Telegraph Company experience with creosoted pole test lines, including brush, open-tank and pressure treatments, and covering northern (eastern) cedar, southern white cedar, chestnut and southern pine poles, ends with the following conclusions, among other generalizations:

"Because of the fact that many of the test specimens are still in service, conclusions can be reached only in the case of some of the less important problems whose solution has been sought. The possibility of extending the life of poles through preservative treatment is abundantly demonstrated, but the capabilities of the more effective processes of treatment (studies) can as yet only be estimated.

"... the indications are that in the cases not already affected, the beginning of decay attack will mainly be dependent upon changes in the quantity and the composition of the preservative retained in the individual poles."

This was only twenty years ago. Even in 1932 this service test report was more valuable as history than as a technical base for treatment specifications. The report reconfirmed the common knowledge that durable timbers like northern cedar and chestnut were better line units if properly butt-treated with creosote, and that adequate penetration and absorption of creosote were essential factors in the economy of pressure treatment of non-durable southern pine poles. However, by 1932

(a) The type of creosote used for treating the relatively large heart and small sapwood southern pine poles for the famous Washington-Norfolk and Montgomery-New Orleans lines was no longer available commercially;

(b) The type of virgin pine timber used was becoming scarcer and scarcer;

(c) The supply of commercial chestnut pole timber was just about completely exhausted;

(d) New and improved methods of butt treatment involving the use of machines for incising the ground section were being applied to northern cedar and to western cedar poles, and besides, the use of northern cedar was gradually shrinking to limited areas in telephone plant in the North-eastern and Lake States; and

(e) Because of vastly increased competition in the pole treating industry, and because the chestnut supply had failed, and because the excessive bleeding of creosote from the old style "12-pound" full-cell

southern pine poles — like those in the cited lines — made them unacceptable either as replacements for chestnut or for new construction in large sections of the northern and central telephone plant areas, and for basic reasons of economy, new specifications for creosoted southern pine poles were being issued. In preparing these specifications the Laboratories reduced the retention requirements to 8 pounds of creosote per cubic foot and provided for treatment by an empty cell process with a view to providing a clean pole, and set definite quality limits on penetration to insure an economic service life. These moves were made after careful experiment, and after analysis of 8-pound treatment results. The experience in the pole test lines had an indirect effect rather than a determining effect on the proposed new penetration requirements.

The Laboratories by 1932 had been operating the Gulfport test plot for 7 years and the Chester test plot for 3 years. Research and development programs on laboratory and test plot evaluation procedures^{123, 124, 129} were well under way. The aim of this broad program was to determine the necessary requirements for preservatives, for retention, for treatment and for penetration *before* the poles were placed in line; which is quite different from the philosophy of depending on service tests or records to reveal at some later date what was done wrong in the first place.

The reader should not get the idea that service tests can be dispensed with entirely. Material in service in the telephone plant is always under observation, casual or intensive, and the development of any obvious faults is corrected by the application of results of more and perhaps better laboratory experiments. In very many cases the faults are discovered in the laboratory or test plot before they are found in the field. However, one can never brush aside the insistent and determining effect of long and satisfactory field experience with treated wood.

For example, when the behavior of creosoted poles in line is good the service tests take on what seems to be the outstanding characteristic of their present function, namely that of a comforting confirmation of previous conclusions. The results of some of the Laboratories' analyses of the relation between penetration and decay in creosoted poles were published in July, 1936, in this Journal²⁹ and again in 1939.³ These results confirmed the previous actions involved in specifying an empty cell treatment and a retention of 8 pounds of creosote per cubic foot. The poles were clean, they were being accepted generally for line construction, and the incidence of infection and probable early failure seemed to be about in line with anticipation.

Emphasis in the last two papers cited was on penetration, which is easily determined, relatively speaking, and not on retention. The relation

of low retention to decay showed up in the Gulfport plot,⁷⁶ and it was explained by Laboratories' extraction and analysis of the creosote in the decaying or failed test stakes and test posts. One may say that such results confirmed suspicions; and they did, because creosoted poles in line — which were "related" to the decaying posts at Gulfport — were found on inspection to be behaving badly. It is believed that corrective measures in the way of supplemental ground line treatment were taken in time to give the poorly treated poles a reasonably satisfactory life.

In another striking set of circumstances, however, a number of cases of unprecedented premature failure of pine poles in line treated with a mixture of creosote and copper naphthenate petroleum revealed that the preservative solution had gone out of control and that in consequence the poles had not received the specified protective amounts of copper. Bell Telephone Laboratories' analyses of parts of the decaying poles showed that the decaying areas contained less than, and the sound areas more than, the Madison soil-block threshold of approximately 0.08 lb/cu ft of copper as metal.

These poles were immediately traced back to the supplier by their brand label. Present day pole treatment specifications in general require branding of each pole unit with symbols or code letters for the supplier's plant, the species of timber, the year of treatment, and the class and length of the pole. Each such pole becomes automatically a unit in a sort of universal service test in Bell System pole lines.

Service tests and service records are a significant part of the over-all process of evaluating wood preservatives, but insistence on service records as the most important criterion simply perpetuates the reputation of established preservatives and forever blocks or seriously impedes the development of new and promising materials. In the very nature of the case the results of service tests can be stated in the form of broad generalizations only. The truly technical approach must be made through better methods of measuring the effectiveness of preservative materials by accelerated field tests on a sufficiently large number of small stakes or by controlled laboratory experiments such as the soil-block tests.

DISCUSSION

There are a number of things in connection with the soil-block test procedure, its interpretation and the correlation of its results with the results of other evaluation methods that require further discussion. For example, questions have been raised vigorously on such matters as:

- (a) The effect of variation in growth rate and density of the wood;

- (b) The size and shape of the test block;
- (c) The use of toluene or any other diluent with creosote to get low retention;
- (d) The distribution of the preservative in the block;
- (e) The practice of heat sterilization of the creosoted blocks;
- (f) The whole philosophy of the weathering procedure; and
- (g) The methods of control assay, such as weight, creosote extraction, analysis of extracted creosote, and lime fusion chloride determinations as applied to pentachlorophenol.

None of these questions can be simply brushed aside, in spite of the fact that some of the points raised have a little of the nature of quasi-technical road blocks that may temporarily slow the approach to *any* standard laboratory test for creosote. Experimental work now under way at Madison⁴¹ and our own laboratories at Murray Hill will help answer some of the questions. The following discussion will at least explain the nature of the problems involved.

Density and Growth Rate

Data on the relation of block density and absorption at treatment have already been presented (Tables I, IV, V and VI). It is not yet evident that either density or growth rate has any material effect on the treatment retention thresholds for creosote when the tests are run on outdoor (or equally depleted) weathered blocks, and when the steps in the gradient retentions are properly spaced. When some present experiments are finished the matter may be resolved more conclusively.

Size and Shape of the Test Blocks

Some critics of the soil-block test have objected rather strenuously to the $\frac{3}{4}$ -inch cube because the two transverse faces are so close to each other. The inference is that either (a) the preservative, and in this case creosote is usually meant, is lost too rapidly through the end grain of the wood or (b) that in the evaporation process, or in the course of the weathering procedures, the preservative may be concentrated on the transverse faces. The validity of these contentions is under investigation. However, the importance of block size and shape can be greatly exaggerated.

One need not assume, in order to plan for comparative tests of oil preservatives, that a block must or can be cut so that creosote will be lost from it at exactly the same rate or manner as creosote may be lost radially from the concentric annual rings of a post or pole. Block shape

has varied almost as much as the test procedure employed. In a laboratory working plan for a study of the "Efficiency of Various Wood Preservatives," with the subtitle "The Efficiency of Various Wood Preservatives in Resisting the Attack of Fungi in Pure Cultures," signed by C. J. Humphrey, dated July 10, 1913, and approved by Howard F. Weiss, at that time director of the young U. S. Forest Products Laboratory, Humphrey proposed the use of eastern hemlock heartwood, cut into pieces measuring $1\frac{1}{4} \times 1\frac{1}{4} \times 2$ inches, which were to be treated and quartered longitudinally "to reduce the size sufficiently to allow their introduction into the (Erlenmeyer) flask." (Writer's italics.)

Untreated wood blocks were to be used as culture media, and thirty days after inoculation the test pieces were to be put into the flasks and shaken up with the inoculated blocks. The test fungus was to be *Lentinus lepideus*. The culture period was to be 9 months, with culture blocks and mycelium "kept in a condition moist and warm enough for active growth" throughout the test period. Coincidentally, Humphrey⁶¹ and his colleagues were developing the broad foundation for the Petri dish toxicity test. The proposed wood block tests never reached a really satisfactory experimental level for treated wood, although they were employed for comparative natural durability tests.⁵⁷

Breazzano^{20, 21} in the same year experimented with blocks of beech wood cut from treated ties and measuring about $9 \times 2 \times 1$ cm. As has been pointed out earlier in this paper he recommended blocks measuring $4 \times 2 \times 1$ cm as a tentative standard, and later, in 1922, blocks $4 \times 4 \times 2$ cm. were accepted as standard in Italy.²² In the case of the latter the larger faces were to be transverse faces, cut across the grain.

Howe⁵⁶ reports tests of small sticks (blocks) of southern pine, measuring $\frac{3}{8} \times \frac{3}{8} \times 6$ inches, that were treated with salt preservatives and later inserted in "8-inch sterilized test tubes containing about 10 cc of standard malt agar." He also used sets of four small sticks in Petri dishes, placing them on 10 cc of nutrient agar medium that covered the bottom of the dishes. The fungus used was called *Fomes annosus*. To supplement these tests he mixed ground-up treated wood in different concentrations with agar media, and tested his mixes against this same *Fomes annosus* and a number of other wood-destroying fungi.

Howe and Curtin³² were working on a broad plan aimed at correlating laboratory tests with test plot tests made, e.g., at Matawan, N. J., and with experience in line.

Snell¹⁰⁷ argued that one might obtain good growth by placing thin blocks of wood in tubes with agar, but that the growth of the test fungus on the agar and on the wood might be influenced by diffusion of the

preservative into the agar medium. He proposed the use of thin plaques of wood, measuring $\frac{1}{8} \times 3 \times 3$ inches. These could be soaked to any required concentration of preservative and then tested by placing them over wet filter paper in Petri dishes. The test specimen would be supported just above the wet filter paper on sterilized wood strips. Inoculation was to be by the simple process of placing a small square of agar plus the growing test fungus directly on the upper surface of the test "block." Similarity to natural conditions and the control of moisture content of the wood were considered to be decided advantages for the method.

Cislak (see discussion of Snell and Shipley¹⁰⁸) used similar plaques of wood measuring 4 x 4 inches and about $\frac{1}{28}$ -inch thick for experiments on evaporation and permanency of creosote.

Rhodes, Roche and Gillander⁹⁰ used blocks measuring $\frac{1}{2} \times \frac{1}{2} \times 3$ inches.

The European standard^{33, 45} block measures 5 x 2.5 x 1.5 cm, with the long axis in the direction of the grain.

Schulze, Theden and Starfinger, in addition to the standard block, used "half" blocks, i.e., blocks measuring 5 x 2.5 x 0.75 cm. All factors considered, they do not regard the thinner block as an advantage, and they have held to the standard size.^{54 (1)}

Lutz⁷⁷ in 1935 suggested the use of 2 x 2 x 5 cm. blocks, with the long sides dressed parallel to the fibers of the wood. He also used blocks measuring 1 x 1 x 5 cm.

Alliot² favored blocks measuring 5.0 x 1.0 x 0.5 cm. in the longitudinal, tangential and radial directions, respectively, for a French standard test.

In his recent tests^{51, 52} Harrow has used $1\frac{1}{2} \times 1\frac{5}{16} \times \frac{7}{8}$ inch blocks.

Sedziak¹⁰⁶ uses $\frac{3}{4}$ -inch cubes cut from $\frac{3}{4}$ -inch stakes *after treatment*.

The National Wood Manufacturers' Association¹⁰⁹ standard block size is 1.25 inches on the radial surface, 1.75 inches on the tangential surface and 0.25 inch thick, i.e., in the longitudinal direction of the grain.

Various size blocks have been used at Madison in soil-block tests of natural durability, but two sizes only have been employed commonly since 1944 in the above mentioned soil-block tests and agar-block tests. The soil-block is the $\frac{3}{4}$ -inch cube, generally drilled with a $\frac{1}{8}$ -inch hole in the center of a tangential face. The agar-block was cut with two broad transverse surfaces measuring $\frac{3}{4} \times 1\frac{1}{2}$ inches, and with a distance along the grain of only $\frac{3}{8}$ inch; and it was not drilled.⁴¹ The Madison agar-block is basically the same sort of a block as the one described in the previous paragraph, and it resembles the Breazzano blocks²¹ as far as maximum transverse surface exposure is concerned.

The list is incomplete, but it serves to illustrate one important point about laboratory evaluation tests, and that is the inevitable variation that creeps naturally into explorative research. In the writer's experience and opinion much of the variation in block size has been the result of a sort of forced adaptation, on the part of the investigator, to the size and shape of his laboratory glassware, coupled with certain practical problems of block procurement and manufacture. Obviously it is easy to use thin sticks in test tubes, thin sticks or plaques in Petri dishes, and relatively flat blocks in Kolle flasks. The $\frac{3}{4}$ -inch cubes, which in essence, as has been stated before, are simply sections of the $\frac{3}{4}$ -inch stake, handle easily in the soil-block cultures.

Criticism of the shape of the $\frac{3}{4}$ -inch cube did not become pointed until after the publication of the first papers^{95, 35} on the Laboratories' cooperative work with the Madison laboratory. It is argued — as mentioned before — that the evaporation of creosote from such blocks is unfairly rapid. However, the losses reported by Rhodes et al,⁸⁹ which will be discussed later, indicate that separation of the transverse faces will not prevent creosote evaporation. Assuming properly calculated gradient retentions, and the use of weathered blocks, it does not appear likely that the shape of the blocks — if kept constant within any given comparative test series — will affect the location of the treatment threshold retention.

Toluene as a Diluent for Creosote Treating Solutions

This subject is most controversial in this country. The use of acetone or chloroform has been widely accepted in Europe, but Schulze and Becker¹⁰⁴ warn that the use of any diluent may change the rate of evaporation of given creosote fractions, and may affect the rate of evaporation of whole creosote. However, there are points in the debate which can be stressed, namely:

- (a) The treatment of test blocks to low and uniform retentions without a diluent is extremely difficult, if not practically impossible;
- (b) The rate of loss of creosote by evaporation, and any change in the character of that loss that may result from toluene dilution might be evident in freshly treated blocks but not in weathered blocks; and
- (c) The volatile fractions of undiluted creosote are lost fairly rapidly from small saplings¹²⁵ and from test blocks; and it is assumed that the use of toluene does not cause loss in the fraction above 355°C.

Discussion of Item (c) will be resumed in the section on weathering. In view of the perplexing character of this toluene dilution question steps are being taken to find out what happens. In the meantime the toluene diluent which has not been found to exert any measurable toxic effect in

the soil-block tests at Madison⁴¹ must be used to secure gradient retentions down to and below the threshold level.

The Distribution of the Preservative in the Block

The opinion has been expressed by a number of investigators^{14, 22, 23, 100} that it is difficult to secure uniform distribution of the preservative in the test block, and that evaporation would cause concentration of certain preservatives on the block surface, where the toxic material might influence the behavior of the blocks in the test cultures. If such a concentration occurs the result would be to fix the threshold at lower over-all treatment retention than it would be if the preservative were not concentrated, for example, at the transverse faces. One is likely to agree that uniform distribution of some creosotes might be difficult without the use of a diluent, particularly in the low retention groups. Rhodes et al⁸⁹ were apparently satisfied that they got a fairly good distribution with a 16.7-pound retention. Experience at Madison and at Murray Hill indicates that the blocks are saturated with the toluene-creosote solution or with the toluene-penta-petroleum solution; and this confirms the ideas of Schulze, Theden and Starfinger^{54 (1)} on the way oven-dried test blocks take up the treating solutions.

Data on the distribution of residual creosote in weathered blocks is presented in Table XXX, and discussed in the section on weathering. Preliminary assay of blocks treated with penta-petroleum, (a) just after treatment, (b) after weathering and (c) after testing show that the pentachlorophenol concentration is slightly *lower* at the transverse faces than it is in the middle section of the blocks. The same seems to hold true for the copper metal in blocks treated with copper naphthenate in toluene-petroleum.

It should be pointed out here that preservatives are rather fortunately distributed in treated wood in such a way that the outer fibers or annual rings normally contain much higher concentrations of the toxic material than is found in the inner fibers. (See Tables XV and XVII and Fig. 29).

Heat Sterilization of the Treated Blocks

In pure culture test experiments some form of sterilization must be used to avoid contamination by other organisms than the test fungi. Most frequently such sterilization, for the minimum necessary time, is accomplished by flashing the treated block through a hot flame or by steaming at 100°C and atmospheric pressure. Either of these procedures

might cause a measurable loss of volatile creosote fractions from freshly treated blocks; but such losses appear to be negligible in the case of weathered blocks. At Bell Telephone Laboratories control data on these possible losses are being determined by extraction of control blocks after the sterilization phase. Such blocks are run through all the steps in the bioassay procedure up to planting in the soil-cultures. It will be recognized by anyone familiar with pressure treating methods that the 100°C temperature, usually held for 15 minutes only, represents a much gentler set of conditions than the after-treatment steaming for several hours at 240–259°F which is permitted in many specifications for creosoted poles.

The Weathering of Creosote and Creosoted Wood

Creosote is a remarkably good wood preservative, and nothing in the following paragraphs is intended to detract from its reputation in that respect. Sometimes the creosote oozes or bleeds from the surfaces of treated units such as poles and crossarms, especially on hot, sunny days; and when such bleeding occurs the treated material is unsatisfactory for use in many parts of the telephone plant. In order to prevent bleeding difficulties and the consequent unhappy employee and public relations that result, the retention requirements have been held down to the commercial standard level of 8 pounds per cubic foot for southern pine poles, and the residue above 355°C limitations on the creosote have been kept in actual practice at 25 per cent or below except when war or post-war emergencies have interfered.

The creosotes vary in the proportion of readily volatile materials they contain, and these materials are lost from creosoted wood — largely by evaporation — under many different use and exposure conditions. The general facts about such losses have been reported over and over again since the turn of the century. The significance of such losses is still not broadly understood or appreciated. Their possible bearing on the weathering procedure to be used in the soil-block tests is of fundamental importance. Paraphrasing the quotation from Schmitz⁹⁹ cited in an earlier paragraph: It is really necessary to know how much creosote to inject into wood to allow for loss by volatility and to insure a residual of the preservative, remaining in sufficient amount, to protect the wood for an economical service period. Bell Laboratories has been deeply concerned with the question whether it is practicable under commercial conditions to specify enough retention at treatment to provide the necessary protective residual and at the same time require clean, satisfactory, treated poles and crossarms for Operating Company use.

General Considerations; Creosote Fractions

Before presenting definite evidence of creosote losses this seems as good a place as any to refer briefly to investigations that have been aimed at discovering what components, volatile or relatively stable, give creosotes their properties of toxicity and permanence. Three articles by Martin,⁸⁰ Rhodes⁹⁰ and Mayfield⁸¹ are the latest American papers covering the general subject, the two former dealing with the technology of hydrocarbons and creosotes and creosote production and the latter reviewing the results of tests of whole creosotes and creosote fractions.

Teesdale's short report¹¹³ in 1911 is one of the earlier records in this country of experiments aimed at determining the loss of creosote fractions from treated wood. He used a creosote with 49 per cent distilling below 250°C and a residue above 320°C of 28 per cent, which would have been about 20 per cent at 355°C. This oil was fractionated into 5 parts, I, to 205°C; II, 205–250°C; III, 250–295°C; IV, 295–320°C; and the residue above 320°C. These five fractions and a sample of creosote with a similar distillation range were used to treat air-seasoned pieces of *Pinus taeda*, mostly sapwood, cut 2 feet long from 5 to 6-inch diameter peeled posts. The retentions were about 18 pounds for the numbered fractions, 15 pounds for the residue, and 21 pounds for the whole creosote.

The treated pieces were open-piled in the laboratory for two months, with temperatures running from 60 to 80°F. At the end of that period the per cent losses of the five fractions and the creosote were respectively 34.7, 21.3, 15.9, 6.2, 4.0 and 5.4. The results were in line with expectations. The test period was short, and there were no outdoor weathering factors. Teesdale notes that the loss of the whole creosote was about the same as the losses in the two higher fractions, III and IV; and that a proportionately composited sample of the five fractions lost at the rate of fraction III, the total at the end of the two-month period being 15.8 per cent or about *three times* as great as for the whole creosote.

Loseby and Krogh⁷³ reported in 1944 on outdoor weathering tests of creosoted wood blocks; and they compared the weight losses in the blocks with evaporation losses from open Petri dishes. The creosote used was a relatively low residue oil produced at Pretoria. The residue above 355°C was 19.95 per cent, the specific gravity at 38/20°C was 1.088, and the amount distilling to 235°C was only 4 per cent. The test blocks were planed pieces of light weight *Pinus insignis* measuring 6 x 1½ x 1½ inches. The per cent moisture at the time of treatment was 10.3 per cent.

The creosote was fractionated into four parts; Fraction I, up to 270°C; Fraction II, 270–315°C; Fraction III, 315–355°C; and, Fraction IV, the

residue above 355°C. The blocks were very heavily treated by a hot and cold soaking process with the straight creosote and each of the four fractions to the following average retentions, respectively: 52.2, 45.6, 50.3, 50.2 and 23.7 pounds per cubic foot. These high retentions place the experiments out of line with most of the others cited in this paper; but the South African tests are unique in that they supply evidence on the rate of creosote losses from such high retentions.

The relative order of losses of the materials, with the one having the highest losses first, was the same for the blocks that were hung on wire outdoors and for the open Petri dish samples, namely; Fraction I, II, whole creosote, Fraction III, and the residue. The latter actually showed no significant loss in either block or dish tests, and in the blocks there was a slight increase, possibly referable to oxidation, of a maximum of 2.2 per cent at the end of the three-year period. This gain was gone by the end of the 5½-year test. Fraction III was lost more rapidly from the blocks than from the *Petri dishes*; the reverse was markedly evident for Fractions I and II; whereas the pattern for the whole creosote was very similar after the first year. The rounded figures for losses from blocks treated with the whole creosote, at the end of 1, 2, 3 and 5½ years outdoor exposure, were 36, 42, 44 and 47 per cent; and the losses from the open dishes for the same periods were 34, 40, 43 and 48 per cent respectively. One may conclude from these tests and conditions that a loss level of about 50 per cent would have been reached in about six years in blocks treated to a reported 50-pound per cubic foot retention with an undiluted creosote.

Most of the laboratory toxicity tests on fractions have been run by the agar or the agar-block method.^{54 (1), 104, 11, 101, 47} The results obtained by Schulze and Becker¹⁰⁴ are cited by Mayfield, who includes as his Fig. 1 a copy of the summary curves prepared by the Berlin investigators. The interested reader can profitably use the rather full excerpts of tabulated results of other investigators given in Mayfield's paper as an introduction to the difficulties of testing creosotes and of interpreting the results of such tests. Some of the main controversial points are brought out by Peters, Krieg and Pflug in 1937⁸³ who challenge the results of Petri dish agar toxicity tests with results of the German agar-block tests — one of the first such broad comparisons to be made. Broekhuizen²⁵ published his findings the same year in a comprehensive paper covering agar-block tests on creosotes and creosote relatives and creosote fractions. He discusses his results with different preservatives in relation to their toxic properties, their protective and preservative qualities, and the *permanence* of such qualities, and the bearing of these qualities on practical

wood preserving procedures. His discussion of the importance of "weathering" tests and of his own results with such tests make up one of the best, if not the best consideration of this important phase of evaluation tests that had appeared up to that time.

Numerous references to weathering techniques will be found in van Groenou, Rischen and van den Berge;¹¹⁸ and van Groenou's own paper¹¹⁷ in 1940 is an excellent short review of previous work, giving his views of the pros and cons of different procedures with emphasis on the essential nature of some test to determine what changes are likely to take place in a preservative as a result of leaching or evaporation.

The following examples will be interesting as illustrations of the different techniques that have been employed in testing the toxicity, or the potential preservative value of creosotes. F. H. Rhodes and Gardner⁹¹ in 1930 determined evaporation losses of creosote fractions from thin pads of dried ground Sitka spruce pulp. The whole creosote and the fractions were introduced in an ether solution. The impregnated pads were placed on top of agar cultures in Petri dishes. The test fungus was the usual *Fomes annosus*, more recently called simply Madison 517. They state that:

"It was found that under these conditions (the Petri dish covers loosely fitting — not water sealed) the more volatile preservatives vaporized from the test specimen, so that at the end of the month only a relatively small portion of the fungicide remained in the pulp."

They were using a domestic creosote with a specific gravity of 1.065 at 38/15.5°C and a residue above 355°C of 21 per cent. They tested fractions of the dead oil from which the tar acids and tar bases were removed, and fractions of the tar bases and the tar acids themselves. They determined percentage losses by evaporation for all lots by letting the treated pulp disks remain in covered Petri dishes for one month at 25°C. All of these reported losses for the dead oil occurred in fractions boiling below 316°C, for the tar acids in fractions with the same upper limit, and for the tar bases in the fractions boiling below 308°C. The losses varied inversely as the boiling range, the greater being in the low-boiling fractions, as was to be expected. The toxic limits for *Fomes annosus* that they determined showed a gradual increase as the boiling point increased, i.e., in agreement with other workers with agar tests they found the lower boiling fractions the most toxic. Their bibliography, with one or two exceptions, covers American articles only.

Rhodes and Erickson,⁹² continuing the same general technique, but substituting mechanical pine pulp for spruce pulp, showed that much

higher quantities of respective creosote fractions were required to kill *Fomes annosus* in the pulp cultures than in Petri dish agar cultures. They concluded from their experiments that "no one compound in coal-tar creosote is primarily responsible for its preservative power. . . . The fractions from water-gas tar oil are much less effective as preservatives than are those from coal-tar creosote oil. The chlorine derivatives of phenols and creosote and of naphthalene are more toxic to fungi than are the compounds from which they are obtained."

In 1933 Flerov and Popov⁴⁸ tested fractions of two creosotes by their soil-block method and compared the results with tests of the fractions emulsified in agar, following American Petri dish procedure in general. They found (English translation by Hildegard Kipp, Forest Products Laboratory):

"On wood the toxicity of the heavy fractions is considerably higher (compared to that of the lower fractions) and the most toxic fractions are those from 315 to 375°C. . . . In tests on wood, in addition to the preservative effect, the effect of the evaporation factor, which is of great importance with oily preservatives, is determined with (more or less) accuracy."

In 1951 Finholt⁴⁶ revives the use of emulsion of the creosote fractions in the old Petri dish or flask method, like history repeating itself. The reader can be excused if he senses a degree of confusion.

Creosote Losses

Baechler's 1949 paper⁵ on the toxicity of oils before and after aging would be a fitting introduction to this section. The first task is to condense into simple statements or tables some of the available data on creosote losses from treated wood.

Curtin³² cites Bond on the latter's experimental determination in 1910-11 of creosote losses from thoroughly air-seasoned red oak and maple railroad ties with approximately the same moisture content. Bond reports that:

1. Full cell treated red oak in 200 days between November 1910, and June 1911, lost 19.0 per cent; and
2. Similar empty cell treated ties lost 52.7 per cent; and
3. Full cell treated maple ties in 105 days from March to June 1911, lost 13.4 per cent; and
4. Similar empty cell treated ties lost 23.0 per cent of the creosote absorbed at treatment.

The losses were determined by weight. He shows that the losses were

greater for the lighter treatments, and that in effect the losses were relatively greater for the empty cell than for the full cell treatments. His conclusions appear to hold good through all the subsequent cited data that admit of such comparisons.

Bateman's early, 1912, work⁶ on oils extracted from two old piles that had been in service about thirty years indicated some considerable loss — in one case more than 35 per cent in the above water section — and relatively lower losses for water line and below water line sections. This confirmed a generally accepted common opinion. Losses below water line were apparently confined to the fraction distilling below 225°C in the case of the pile which he considers to have been treated with a pure coal-tar creosote. He calls *light* oils those distilling below 205°C. With the exception of the above water section of one pile all the samples of treated wood still contained about 17 pounds per cubic foot. He states:

"The creosote in the pile which was perfectly preserved contained originally at least 40 per cent of naphthalene fractions, a large portion of which remained in the wood. The creosote in the pile which was less perfectly preserved contained little or no naphthalene."

Service records on such oils resemble records from the Washington-Norfolk line and from the specimens examined by Alleman¹ in that the data have little if any bearing — other than historical — on the creosote use problems of today.

Schmitz et al^{102, 103} report a loss of 25.9 per cent after five years service in track in red oak ties treated with a 60/40 creosote-coal tar solution, compared to a loss of 17.3 per cent after three years service. The losses, determined by extraction, varied inversely as the boiling range, as was expected. There was very little loss in the 315–355°C fraction, and no loss is indicated for the fraction boiling above 355°.

Bateman in 1922,⁷ and again in 1936 (see Discussion and ¹⁰⁸) in connection with his explanation of the relation between the loss of the creosote fraction below 270°C and a formula for estimating the permanence or preservative life of creosote in treated wood, cites the earlier work of von Schrenk, Fulks and Kammerer, and Rhodes and Hosford on creosote losses from southern pine poles in the Washington-Norfolk and Montgomery-New Orleans lines of the American Telephone and Telegraph Company. Certain of the poles in question were installed in 1897 and removed in 1906 after about nine years service in line. The creosote used to treat these poles is reported to have had a specific gravity of 1.022–1.030 at 3°C above the melting point of the oil. The residue above 315°C was about 16 per cent; and the per cent naphthalene was "not less than 40 per cent." Using the pitch residue — the per cent

boiling above 315°C — as a base for calculation, along with the change in residue determined from extracted creosote, von Schrenk, Fulks and Kammerer estimated the creosote losses, above and below ground line, for five poles from the Washington-Norfolk line, that are shown in Table XVIII. The poles were old growth longleaf pine, with a high heartwood volume. They were heavily treated — to about 16 pounds — by a full cell process. There is a wide variation in the results shown in the table

TABLE XVIII — CREOSOTE LOSSES, BASED ON RESIDUE INCREASE, FROM SOUTHERN PINE POLES

9 years exposure; Washington-Norfolk line, American Telephone and Telegraph Company—Data of von Schrenk et al.

Pole No.	Average loss, per cent	
	Top	Butt
1425	43.3	2.7
29	42.4	16.4
10749	50.9	20.8
2931	58.0	32.8
9700	70.8	60.3
Overall average	53.1	26.6

TABLE XIX — CREOSOTE LOSSES, BY EXTRACTION

Southern pine test posts, aerial sections; 1926 and 1927 series; Gulfport test plot—BTL (Waterman) data.

	Exposure period, months					
	8	17	22	31	32	46
	Average loss, per cent					
<i>1926 Series</i>						
8 lb empty cell.....	16		31		41	54
12 lb full cell.....	9		21		25	34
"Light" oil.....	17		32		41	55
"Mixed" oil.....	10		22		28	38
<i>1927 Series</i>						
"Light" oil						
8 lb empty cell.....		27		44		
12 lb full cell.....		29		36		
"Heavy" oil						
8 lb empty cell.....		16		36		
12 lb full cell.....		18		30		

TABLE XX — CREOSOTE LOSSES FROM SOUTHERN PINE POSTS

Analyses of original creosote and creosote extracted from outer 1 inch below ground line of posts in test 3, 6 and 7 years; Gulfport test plot; 1925 series; 12 lb. full-cell treatments — BTL (Waterman) data.

	Original creosote	After 3 years†	After 6 years	After 7 years
Specific gravity	1.037 (38/15.5)	1.056 (60/60)	1.057 (60/60)	1.059 (60/60)
Distillation, water free basis, per cent, cumulative				
To 210°C.....	4.1	0.5	0.3	0.1
210-235.....	32.0*	4.0	3.0	3.4
235-270.....	44.8	22.6	22.1	26.3
270-300.....	57.2	38.3	39.0	43.0
300-315.....	73.8	45.9	47.2	50.7
315-355.....	88.9*	72.2	73.9	75.8
Residue.....	10.7†	26.4	25.2	24.1
Total.....	99.7	98.6	99.1	99.9
Sulph. residue, gm/100 ml.....	3.2	4.2	3.7	2.4
Tar acids, gm/100 ml.....	8.3	3.3	2.8	6.5
Estimated losses, per cent, based on residue increase.....		59.5	57.5	55.6

* To 360°C in original oil analysis.

† Above 360°C in original oil analysis.

‡ The values for 3, 6 and 7 years are averages of data for 2, 3 and 3 posts, respectively.

TABLE XXI — CREOSOTE LOSSES FROM SOUTHERN PINE POSTS

Analyses of original creosote and of creosote extracted from outer 1 inch zone below ground line of posts in test 4, 5 and 6 years; Gulfport test plot; 1926 series; 8 lb. empty-cell treatments — BTL (Waterman) data.

	Original creosote	After 4 yrs.	After 5 yrs.	After 6 yrs.
Specific gravity.....	1.044 (38/15.5)	1.080 (60/60)	1.084 (60/60)	1.131 (60/60)
Distillation, water free basis, per cent, cumulative				
to 210°C.....	1.3	0.3	0.9	—
210-235.....	34.2	1.7	2.9	—
235-270.....	60.1	15.7	21.1	—
270-300.....	74.1	38.4	39.7	6.6
300-315.....	79.9	49.2	50.0	15.2
315-355.....	96.3*	79.7	77.6	54.7
Residue.....	3.4†	19.5	21.6	44.1
Total.....	99.7	99.4	99.2	98.8
Sulph. res., gm/100 ml.....	1.7	0.5	1.1	—
Tar acids, gm/100 ml.....	8.2	2.4	2.7	3.9
Estimated losses, per cent, based on residue increase.....		69.2	72.2	86.4

* To 360°C in original oil analysis.

† Above 360°C in original oil analysis.

for the butt sections, but the top and butt figures, respectively, seem to bear some relation to each other.

R. E. Waterman in a Bell Telephone Laboratories' memorandum dated January 23, 1928, reported losses of creosote from poles removed from the Montgomery-New Orleans line and in a memorandum dated March 7, 1931, reported creosote losses, determined by periodic extractions, from the aerial sections of southern pine posts treated in 1926 and 1927. Companion posts are among the earliest lots reported on by Lumsden.⁷⁶ Part of Waterman's data are condensed in Table XIX. His figures confirm in general the conclusions reached by Bond,³² namely, that the losses were greater for the light than for the heavy treatments, for the empty cell than for the full cell treatments, and in addition, for the lighter oil than for the heavier oil. Such conclusions are in line with what might be expected from the physical characteristics of the creosotes and general knowledge of the distribution and dispersion of the creosotes in the various treatments.

The losses shown in Table XIX are rounded figures that apply to the whole cross section of the pole-diameter posts. Of more significance are data on creosote losses from the *outer 1 inch* of the *below ground* section of companion posts in the Gulfport plot. Tables XX and XXI show distillation figures for the original creosotes and for the extracted oils, from full cell and from empty cell posts, after varying exposure periods up to about seven years. The oils were both low residue creosotes. The indicated percent losses are based on the increase in the residue above 355°C — of which more later. The losses are greater for the empty cell treatments than for the full cell treatments. The fact that so much of the loss occurred within the first four years is extremely important in evaluation philosophy.

Tables XXII and XXIII present data for whole cross sections of two posts that had begun to decay and that were removed for assay four years after installation at Gulfport. Table XXII shows the original analysis of the creosote and the average analysis of the extracted oil from the two posts. The indicated loss in the ground line decay area, figured from the residue increase, was *61.1 per cent*. Table XXIII shows the distribution of creosote at treatment by zones — from the outside toward the heartwood line — from extracted borings, and the distribution of creosote after removal from test, based on extraction of sectors cut from whole cross section disks. The indicated losses, figured from average over-all retention at treatment and after removal were 65.1 and 55.5 per cent, or an average of *60.3 per cent*. This figure can be considered to be in agreement with the 61.1 per cent figure cited above.

TABLE XXII — CREOSOTE LOSSES FROM SOUTHERN PINE POSTS

Analyses of original creosote and of creosote extracted from posts after 4 years in test; 1936 series; Gulfport test plot — BTL (Waterman) data.

	Original creosote	Average, extracted creosote*
Specific gravity	1.055 (38/15.5°C)	1.134 (60/60)
Distillation, water free, per cent, cumulative		
to 210°C.....	3.4	0.2
210-235.....	17.0	0.5
235-270.....	40.4	2.1
270-300.....	53.3	7.5
300-315.....	59.6	15.0
315-355.....	81.2	51.5
Residue.....	18.3	47.0
Total.....	99.5	98.5
Sulphonation, residue, gm/100 ml.....	4.7	3.7
Tar acids, gm/100 ml.....	7.9	12.5

* Average analyses of toluene extracted oils from disks cut adjacent to decay line; posts 273 and 280. The estimated average loss, based on residue increase, is 61.1 per cent.

TABLE XXIII — CREOSOTE LOSSES FROM SOUTHERN PINE TEST POSTS

By zones, by toluene extraction; 1936 series; Gulfport test plot — BTL (Waterman) data.

Post No.	Years in Test	Retention, by extraction lb/cu ft						Remainder of treated sapwood
		Whole cross section	Zones, inches					
			Outer ¼	Next ¼	Next ½	Next 1		
Original retentions, at treatment								
273	—	3.8*	6.9	3.5	3.8	3.9	3.0	
280	—	4.7*	11.2	4.1	3.3	1.5	3.5	
Retention after removal								
273	4	1.69†	1.51	1.20	1.34	1.76	2.50	
280	4	1.64‡§	2.78	1.32	1.90	1.63	1.10	

* Average analyses of boring samples.

† Average analyses of sectors cut from a disk taken 3 inches above maximum decay line.

‡ Average analyses of sectors cut from two disks taken 3 inches above and 3 inches below the maximum decay line. (See Table XXII for analyses of original and extracted creosote.)

§ Average loss, estimated from retentions at treatment and after removal:
For whole cross section..... 60.3 per cent

For outer 1 inch

Post 273..... 70.3 per cent
Post 280..... 64.0 per cent
Average..... 67.2 per cent

Calculated losses in the outer 1 inch of these two posts — assuming 8-inch diameter — are 64.0 and 70.3 per cent, respectively, or an average of 67.2 per cent; and this figure corresponds very closely with the four-year loss figure of 69.2 per cent calculated for the empty cell posts in Table XXI. The posts represented in Tables XXII and XXIII were obviously treated to retentions that were too low to be effective; but they illustrate what is likely to happen when too low retentions of highly volatile light creosotes are used in wood in contact with the ground.

Bateman⁷ discusses a laboratory experiment to determine creosote losses, over a 70-day period from pieces of round post sections, 5 inches in diameter and 2 feet long; and he extends his comparison to data derived from experiments conducted for the San Francisco Marine Piling Commission. He reports the following treatment data for the three creosotes involved:

1. 18 lb/cu ft of a creosote with 92 per cent distilling below 275°C;
2. 10 lb/cu ft of a creosote with 40 per cent distilling below 275°C; and
3. 27.5 lb/cu ft of a creosote with 42 per cent distilling below 275°C.

His comparative loss data are condensed in Table XXIV.

Waterman and Williams¹²⁵ report creosote losses based on periodic extractions of comparable lots of specimens from treated round southern pine *saplings* exposed in the Gulfport test plot. Their data are condensed

TABLE XXIV — CREOSOTE LOSSES

By weight, from round southern pine post sections*, and from pile sections—Madison (Bateman) data.

Exposure period, days	Group†		
	18 lb/cu ft	10 lb/cu ft	27.5 lb/cu ft
	Average loss, per cent		
10	16.0	7.0	1.3
20	22.5	9.5	—
30	28.0	12.0	3.7
40	32.0	13.5	—
50	36.0	15.5	—
60	39.0	17.0	—
70	42.0	18.5	—
90	—	—	7.3
222	—	—	16.6
475	—	—	24.5
510	—	—	25.3
785	—	—	38.2

* Five inch diameter posts, 2 feet long.

† See text for group and creosote data.

TABLE XXV — CREOSOTE LOSSES FROM ROUND SOUTHERN PINE SAPLINGS

By extraction; Gulfport test plot—BTL (Waterman and Williams) data.

	Years in test					
	1		2		3	
	n	Av. loss, per cent	n	Av. loss, per cent	n	Av. loss, per cent
Empty-cell treatments; 3.5-15.1 lb/cu ft						
Above ground line.....	14	35.5	10	55.8	3	55.9
Below ground line.....	14	40.4	10	58.8	3	61.6
Full-cell treatments; 14.0-38 lb/cu ft						
Above ground line.....	9	22.7	7	44.2	4	59.8
Below ground line.....	9	13.9	7	39.2	4	42.4

TABLE XXVI — ANALYSES OF CREOSOTES USED IN WEATHERING WHEEL AND OUTDOOR EXPOSURE TESTS

Southern pine sapwood blocks—Koppers (Rhodes et al.) data.

	Creosote I	Creosote II
Specific gravity at 38/15.5°C.....	1.064	1.081
Distillation, per cent, cumulative		
0-210°C.....	3.8	1.8
210-235.....	21.2	13.7
235-270.....	41.4	30.2
270-315.....	59.8	46.3
315-355.....	80.9	64.9
Residue above 355°C.....	18.7	34.9
Water.....	0.6	0.6
Specific gravity of fractions		
235-315°.....	1.039	1.037
315-355°.....	1.106	1.104

TABLE XXVII — CREOSOTE LOSSES FROM SOUTHERN PINE SAPWOOD BLOCKS

Ether extraction; weathering wheel tests—Koppers (Rhodes et al.) data.

Exposure period, weeks	Average loss, * per cent	
	Creosote I	Creosote II
0	0.0	0.0
1	33.7	28.4
3	50.1	40.3
5	57.2	45.3
9	64.7	52.3

* Treatment retention 16.7 lb/cu ft.

in Table XXV. The loss figures are averages for all the creosotes used. It will be noted that there is a large variation in the treatment retention groups and that the losses were more rapid and definitely higher for the lower retention empty-cell specimens. Preliminary estimates from data on creosoted $\frac{3}{4}$ -inch square stakes at Gulfport indicate losses of about the same order of magnitude, with the trend in the direction of relatively higher figures than those for the round saplings. This is in line with expectation because of the use of the toluene diluent and the practice of controlling the treatments to secure lower than threshold retentions in both full-cell and empty-cell treatments.

Creosote Losses from Treated Blocks

E. O. Rhodes and his colleagues have published two excellent papers^{50, 89} that are most significant in a discussion of creosote losses from treated wood blocks. They used two creosotes, I and II, the analyses of which are shown in Table XXVI. Southern pine sapwood blocks measuring 0.5 x 0.5 x 3.0 inches, with the long axis in the direction of the grain, were treated and exposed on a weathering wheel in the laboratory, and also out of doors. The laboratory test specimens were treated to a retention of 16.7 pounds per cubic foot by soaking the blocks, heated to 105°C, in creosote at 100°C, in a sealed container. The creosote cooled during a five-hour soaking period to 40–50°C. The authors felt that the retention of about 16.5 pounds would facilitate extraction recovery of enough oil for analysis, and that "the treated portion of a tie or pole probably contains about this amount of oil." Bell Telephone Laboratories' experience has shown (Fig. 29) that the retention in the commercially treated 8-pound post averages only about 12 pounds per cubic foot in the outer $\frac{1}{4}$ inch of wood and that the retention drops off rapidly in the wood farther beneath the surface. The Rhodes blocks, therefore, must be regarded as heavily treated.

Losses of creosote were determined by ether extraction, and the change in character of the preservative was determined by distillation of the extracted oil. Corrections were made for resin extracted with the creosote. The losses of creosote from the test blocks on the weathering wheel are shown in condensed form in Table XXVII.

The authors report creosote loss from blocks treated to a 15.0-pound retention and exposed outdoors during the winter as 44.4 per cent; and similar blocks treated to a 16.5-pound retention lost 47.1 per cent in a nine-week exposure period during the summer. The results of the experiments were taken to mean that the losses were of about the same character in the outdoor winter and summer exposure tests, and that the blocks

weathered on the wheel in the same way they did outdoors. Rhodes reaffirms his conclusions about the weathering wheel tests in 1936 in a discussion of the Snell and Shipley paper¹⁰⁸ in these words:

"Our consideration of this problem convinced us, and Snell and Shipley agree with the opinion, that natural weathering produced by heat and cold, rain and wind, involves not only evaporation but water leaching and mechanical losses of whole oil by water or by bleeding. . . . To simulate these conditions, we exposed blocks of wood treated with the test creosotes to variations in temperature, to moving water and to moving air. . . . In fact, we believe that our wood-block exposure tests include most, if not all, of the factors of natural weathering. . . ."

Now to go back a bit to Curtin's 1926³² experiments, this time referring to his own weathering tests on creosoted wood. Table XXVIII is an interpretation of the results he obtained by exposing small blocks, cut from pressure treated 2 x 4 inch southern pine sapwood stakes, to natural out-of-door weathering. His procedure was extremely severe, but he was aiming at an extreme accelerated test for permanency. After treatment his 2 x 4 inch sample pieces were held in storage under cover for 2 months; and then they were cut up so that the test blocks were about 2 x $\frac{1}{8}$ x $\frac{3}{8}$ inches. These small pieces were exposed 15 feet above the ground on wooden trays for a four-month period from September, 1926, to January, 1927, and for a ten-month period from September, 1926, to July, 1927. Losses from the 2 x 4 pieces that must have occurred during their two-month storage period would increase the loss figures shown in the table. The losses are obviously greater for the lighter treatments, and for the lower residue oil.

TABLE XXVIII — CREOSOTE LOSSES FROM SOUTHERN PINE BLOCKS*

Outdoor weathering tests—Based on data by Curtin.³²

Creosote†	Trt. No.	Average retention lb/cu ft			Average loss, per cent	
		At treatment	After 4 mos.	After 10 mos.	After 4 mos.	After 10 mos.
1	1	23.18	19.69	17.13	15.1	26.1
1	2	17.13	11.69	10.88	31.8	36.5
1	3	19.38	6.50	5.44	37.4	47.6
2	1	26.19	18.75	15.00	28.4	42.7

* See text for description of blocks

† Creosote number 1 was a domestic oil, specific gravity 1.056 and residue above 355°C of about 26 per cent; number 2 was a British oil, specific gravity, 1.068 and residue above 355°C of about 19 per cent.

In their roof exposure, outdoor weathering tests Duncan and Richards^{35, 39} have regularly found losses of creosote, by weight, in $\frac{3}{4}$ -inch blocks treated with creosotes having residues above 355°C of 20-30 per cent, to run in the neighborhood of 45-50 per cent. All treatments were made by a full-cell vacuum process with toluene creosote solutions. The over-all exposure period consisted of three stages,³⁹ a three-week conditioning period in a constant humidity and temperature room at 30 per cent relative humidity and 80°F, a sixty-day outdoor exposure on a rack on the roof, and a three-week reconditioning to approximately constant weight in the same humidity room. The losses were figured from the original conditioned weight of the blocks, the creosote retention at treatment, and the calculated amount of creosote remaining as indicated by the weight of the weathered and reconditioned blocks. A condensed and simplified summary for two creosotes and two block shapes^{39, 41} is shown in Table XXIX.

As might be expected under identical weathering conditions, the losses were higher in the case of the $\frac{3}{8} \times \frac{3}{4} \times 1\frac{1}{2}$ inch blocks — with twice as much end grain exposed as the $\frac{3}{4}$ " drilled cubes — which presumably resulted in an accelerated longitudinal evaporation. In other words, a given percentage loss of creosote is arrived at sooner in the case of the block with greater transverse surface area. The loss increase amounts to about 7 per cent at the 8 to 10-pound retention level. This loss is about twice as great as it would be if calculated on the basis of the increased

TABLE XXIX — CREOSOTE LOSSES FROM LONGLEAF
SOUTHERN PINE SAPWOOD BLOCKS

Outdoor weathering tests—Madison (Duncan and Richards) data.

Treatment retention lb/cu ft	Creosote number		
	Coop. No. 2	5340	5340
	Creosote loss, per cent, by weight		
18*	44.0†	—†	—‡
16	45.0	—	—
14	45.9	—	—
12	47.0	—	—
10	48.0	42.5	50.0
8	49.5	44.3	51.2
6	51.3	46.5	54.6
4	54.3	49.8	60.0
2	60.0	54.5	75.0

* Retention by full-cell treatment under vacuum with toluene-creosote solutions.

† $\frac{3}{4}$ -inch cubes.

‡ $\frac{3}{8} \times \frac{3}{4} \times 1\frac{1}{2}$ inches.

surface area only of the flat blocks. The losses are interpreted in general to mean that the exposure conditions were not as severe as those in the Rhodes⁸⁹ weathering wheel tests, and in line with his outdoor tests.

The results of recent determination at Bell Telephone Laboratories of creosote losses from $\frac{3}{4}$ -inch cube blocks, by toluene extraction, are shown in Table XXX. The blocks were divided into thirds before extraction (Fig. 30) and the respective parts were further divided and then pooled in the extractor.

The blocks in each group of twenty were selected to represent the whole gradient of treatments with an average treatment retention for each group of 6.05 pounds per cubic foot. The average density of the blocks, oven-dry weight and volume basis, was the same for each group, namely 0.56. The oil used was cooperative creosote No. 11, a 50/50 blend of British vertical retort tar creosote and British coke oven tar creosote,^{5, 12, 39} diluted as usual with toluene. The exposure period outdoors was sixty days at the Chester Field Station between January 4 and March 3, 1952, plus several days exposure on a bench in a steam heated laboratory both before and after the outdoor period. The average loss of 40.3 per cent is considered to be in line with losses for the same creosote in the Madison tests, considering the factor of the winter climate at Chester.

The distribution of the residual creosote in the test blocks as shown by the averages reported may be considered to be remarkably uniform. The differences in these averages are not regarded as statistically significant. Further discussion of losses from weathered blocks will be resumed in later paragraphs.

TABLE XXX — CREOSOTE LOSSES FROM LOBLOLLY-SHORTLEAF SOUTHERN PINE SAPWOOD BLOCKS, AND LB/CU FT REMAINING

By toluene extraction; outdoor winter weathering tests — BTL (Snoke and MacAllister) data.

Lot No.	n	Average retention at treatment, lb/cu ft	lb/cu ft remaining			
			Outer* third	Middle third	Outer third	Whole block
1	20	6.05	3.62	3.43	3.93	3.66
1	20	6.05	3.49	3.49	3.68	3.55

Average lb/cu ft of creosote remaining;

in outer thirds..... 3.68

in middle thirds..... 3.56

in whole blocks..... 3.61

Average loss of creosote; 2.44 lb/cu ft, or 40.3 per cent.

* $\frac{3}{4}$ -inch cubes. See cutting diagram in Fig. 30.

Creosote Losses from Impregnated Filter Paper

There have been numerous criticisms of the use of creosote loss figures obtained by evaporation from open dishes in any consideration of creosote permanence.¹⁰⁸ The use of the losses reported in this section may be criticized in a similar manner, but they represent extreme acceleration and they seem to have a bearing on the interpretation of any weathering

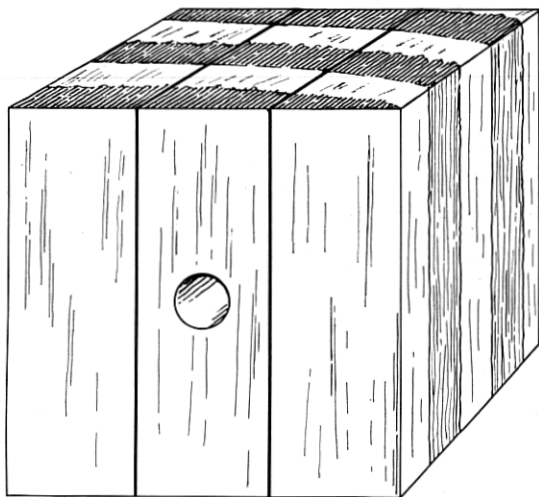


Fig. 30—Diagram of cutting plan for dividing a weathered creosoted block into three approximately equal parts for determination of residual creosote by toluene extraction.

tests in which evaporation plays the major part. Some years ago the late Heinrich T. Boving of Bell Telephone Laboratories, ran an extensive series of evaporation experiments on eight so-called Fulweiler creosotes. He used impregnated strips of filter paper, hung on quartz springs in protecting glass apparatus and exposed to a constant flow of air with no turbulence, and under constant temperature and humidity. His experiments were performed with great care. Repetitions gave excellent agreement. A condensation of his loss figures, rounded off to whole numbers, for seven- and fourteen-day exposure periods, are shown in Table XXXI. Let it be stated unequivocally at this point that it is recognized that there are important physical differences in the wood fiber combinations represented by filter paper, wood blocks, small round saplings, posts and poles; but the *quantitative* loss data seem to fall sooner or later into a similar pattern for all these test media, under the various condi-

tions, and with the assumptions made. About the difference in the *qualitative* changes that take place during creosote losses there is much, but not enough, information.

An Interpretation of Creosote Losses

Frosch,⁴⁹ in describing certain physical characteristics of the Fulweiler oils, states that they may be considered as truly viscous solutions in

TABLE XXXI — CREOSOTE LOSSES, BY WEIGHT, FROM IMPREGNATED FILTER PAPER, AND CALCULATED INCREASE IN RESIDUE BTL (Boving) data

Fulweiler creosote No.	Original residue above 335° C, per cent	Per cent loss		Calculated residue*	
		in 7 days	in 14 days	in 7 days	in 14 days
1	43.84	31	—	63.5	—
2	40.25	32	36	59.2	62.9
3	31.11	36	—	48.6	—
4	28.52	38	—	46.0	—
5	20.85	44	—	37.2	—
6	15.49	48	—	29.8	—
7	12.21	54	—	26.5	—
8	8.49	61	68	21.8	26.5

* Assuming that all loss occurs in fraction below 355°C.

TABLE XXXII — THEORETICAL CHANGES IN CREOSOTE

Loss of volatile fractions by evaporation; amount remaining of total fraction below 355°C.

1	2	Time period 1*					Time period 2				
		3	4	5	6	7	8	9	10	11	12
Fulweiler creosote No.	Assumed treatment retention lb/cu ft	Per cent loss	Residual oil lb/cu ft	Calculated per cent residue above 355°C in residual oil	More than 355°	Less than 355°	Per cent loss	Residual oil lb/cu ft	Calculated per cent residue above 355°C in residual oil	More than 355°	Less than 355°
					lb/cu ft					lb/cu ft	
2	8.00	32	5.44	59.2	3.22	2.22	36	5.12	62.9	3.22	1.90
8	8.00	61	3.12	21.8	0.68	2.44	68	2.56	26.5	0.68	1.88
2	10.00	32	6.80	59.2	4.03	2.77	36	6.40	62.9	4.03	2.37
8	10.00	61	3.90	21.8	0.85	3.05	68	3.20	26.5	0.85	2.35
2	12.00	32	8.16	59.2	4.83	3.33	36	7.68	62.9	4.83	2.85
8	12.00	61	4.68	21.8	1.02	3.66	68	3.84	26.5	1.02	2.82

* Time periods 1 and 2 represent exposures that would result in the per cent losses determined from Boving's 7 and 14 day tests, respectively. See text.

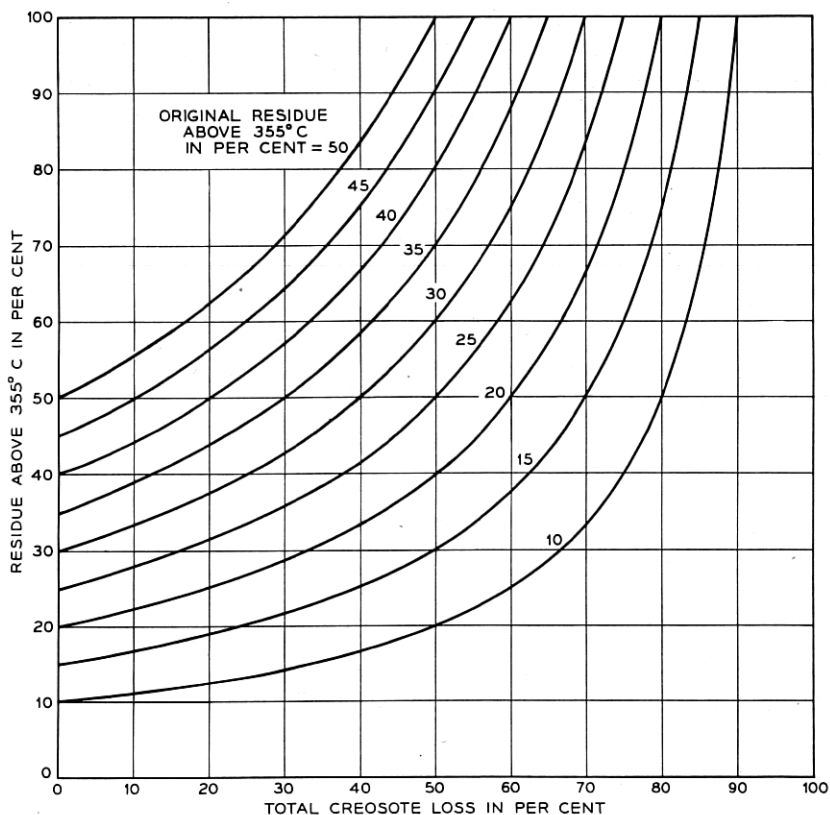


Fig. 31—Theoretical relation of the per cent increases in the residue above 355°C and the per cent losses of total creosote.

which the fraction below 355°C is the solvent and the fraction above 355°C is the solute; and that this condition does not hold for any other temperature point.

Let it be assumed that one may dodge the inferences of the results obtained by Hudson and Baechler⁵⁹ about the increase that may occur in the residue above 355°C as a result of oxidation. Here one would be in agreement with Schmitz et al.^{102 p. 270} Let it be assumed further that all loss takes place in the fraction below 355°C, and that the residue above 355°C is inert, (cf. Loseby and Krogh⁷³). The residue in the eight creosotes used by Boving would increase in their appropriate ratios to the figures shown for seven and fourteen days in Table XXXI. Losses for creosotes of different initial residues and consequent residue increases can then be represented by a family of curves for residue increase with

quantitative loss such as those shown in Fig. 31. Now let it be assumed that these hypotheses can be applied to treated wood, with full realization that the application may err in the direction of oversimplification. Bateman and Cislak accept the general principles of the 355°C division point between volatile and nonvolatile creosote constituents in debating the theoretical aspects of creosote losses.¹⁰⁸ Rhodes⁸⁹ indicates some actual loss in the fraction above 355°C in his test blocks, possibly in part the result of oil displacement in the water phase of his tests, rather than any increase that might occur as a result of oxidation.⁵⁹ For the purpose at hand in this paper it is convenient to use the relations shown in the percent loss — residue increase curves in Fig. 31.

Table XXXII represents a theoretical approach to what would happen to the gross characteristics of the oils if, in some time period X, the losses from creosoted wood treated to 8-, 10- and 12-pound retentions became

TABLE XXXIII — CREOSOTE LOSSES* FROM SOUTHERN PINE SAPWOOD BLOCKS†

Calculations of residual fractions below 355°C; weathering wheel tests.‡

1	2	3	4	5	6	7	8
Exposure period, weeks	Creosote loss, per cent	Creosote remaining		By extraction		By calculation§	
		per cent	lb/cu ft	Residue above 355°C, per cent	Residual fraction be- low 355°C lb/cu ft	Residue above 355°C, per cent	Residual fraction be- low 355°C lb/cu ft
		Creosote I					
0	0.0	100.0	16.70	19.0	13.53	19.0	13.53
1	33.7	66.3	11.07	25.2	8.28	28.7	7.89
3	50.1	49.9	8.33	34.6	5.45	38.1	5.16
5	57.2	42.8	7.15	40.2	4.28	44.4	3.97
9	64.7	35.3	5.90	48.7	3.03	53.8	2.73
12	66.5	33.5	5.59	56.3	2.44	56.7	2.42
Creosote II							
0	0.0	100.0	16.70	33.6	11.09	33.6	11.09
1	28.4	71.6	11.96	43.7	6.73	46.9	6.34
3	40.3	59.7	9.97	51.4	4.85	56.3	4.36
5	45.3	54.7	9.14	57.3	3.90	61.4	3.53
9	52.3	47.7	7.97	60.8	3.12	70.4	2.36
12¶	55.0	45.0	7.50	65.0	2.53	74.7	1.89

* Losses based on ether extraction.

† Blocks $\frac{1}{2}$ x $\frac{1}{2}$ x 3 inches.

‡ See Bibliography, References 50 and 89.

§ Assuming that all loss occurs in the fraction below 355°C.

|| Retention by soaking in undiluted creosote.

¶ The 12-week figures were calculated from extrapolations of the loss curves.

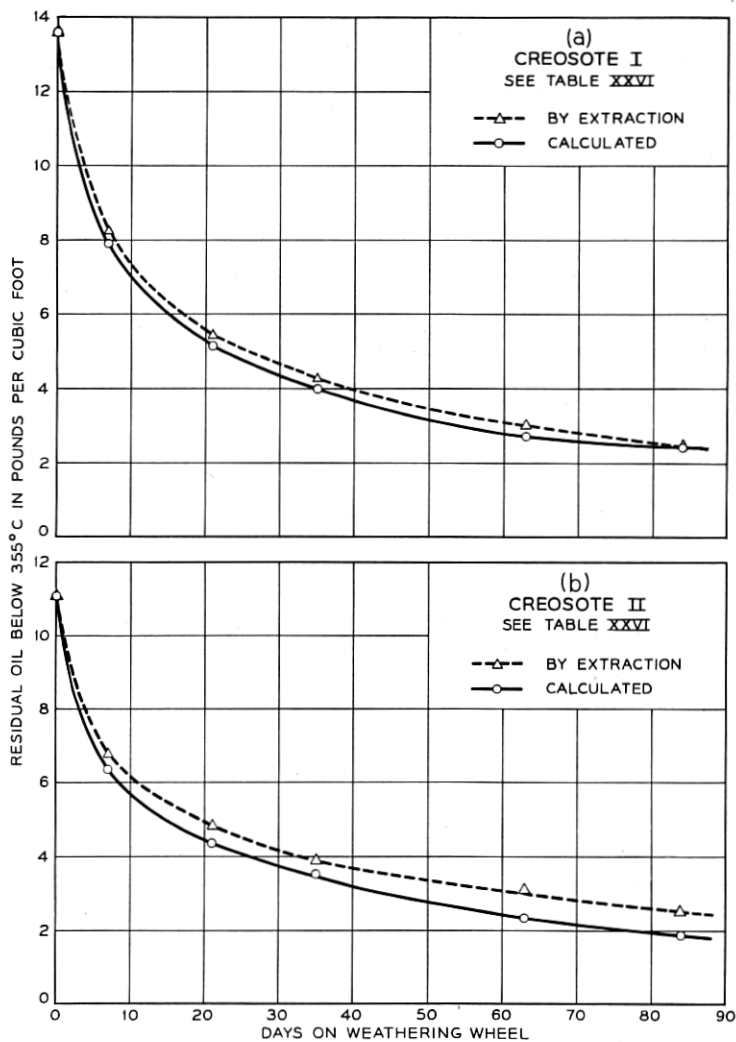


Fig. 32—Creosote losses; lb/cu ft of the fraction below 355°C remaining in the test blocks, by calculation and by ether extraction, based on weathering wheel test data. See text. (a) Creosote I, 19.0 per cent residue above 355°C. (b) Creosote II, 33.6 per cent residue above 355°C.

as great as those determined by the Boving evaporation experiments. The columns have been numbered to facilitate reference. Attention is directed to Columns 6 and 7, and to columns 11 and 12. It will be noted that at the end of Time Period 1 the calculated amounts of material boiling below 355°C in creosotes 2 and 8 are approaching the same magnitude in the respective 8-, 10- and 12-pound treatment groups; and by the end of Time Period 2 (columns 11 and 12), the calculated amounts in each respective treatment group are practically the same for these two creosotes that originally had residues above 355°C of 40.25 and 8.49 respectively. Actually, of course, the time periods would not be the same for the respective 8-, 10- and 12-pound treatments, but — on the basis of loss data already presented — would probably be relatively shorter for the lower retention group and relatively longer for the higher retention group.

Table XXXIII illustrates two methods of arriving at estimates of the per cent and amount of creosote remaining in the respective fractions above and below 355°C, by extraction data and by calculation of the

TABLE XXXIV — CALCULATED AMOUNT OF CREOSOTE FRACTION BELOW 355°C REMAINING AFTER VARIOUS EXPOSURE PERIODS UNDER WEATHERING WHEEL TEST CONDITIONS

Exposure period, weeks	Treatment retentions—lb/cu ft		
	16.7	10.0	8.0
	Residual fraction below 355°C lb/cu ft		
Creosote I			
0	13.53	8.10	6.48
1	7.89	4.96	3.46
3	5.16	3.44	<u>2.16</u>
5	3.97	<u>2.57</u>	<u>1.59</u>
9	2.73	<u>1.82</u>	.99
12	<u>2.42*</u>	1.46	.84
Creosote II			
0	11.09	6.64	6.11
1	6.34	4.03	<u>2.72</u>
3	4.36	2.91	<u>1.85</u>
5	3.53	<u>2.35</u>	1.37
9	<u>2.36</u>	<u>1.87</u>	.81
12	<u>1.89</u>	1.58	.59

* See text for significance of horizontal lines, page 485.

theoretical residue increase related to the given loss data. The loss data are those of Rhodes et al (loc. cit.) (see Table XXVII) with extrapolation to a twelve-week period; column 2. The same holds for the residue data in column 5, from which the residual pounds per cubic foot below 355°C of Column 6 were calculated, by applying the respective percent residues to the pounds per cubic foot of total creosote remaining in the blocks, column 4. The figures in column 8 were calculated in the same way by using the theoretical percent residue figures in column 7. The data in columns 7 and 8 are represented by the curves in Fig. 32, for Creosotes I and II. The curves approach each other closely enough for the purposes of the present interpretation, particularly in the case of the 19 per cent residue oil.

Now, supposing that Rhodes et al had used two lower retentions, say 10 pounds and 8 pounds in their experiments in addition to the 16.7 pounds; and assuming that their losses would be of about the same magnitudes for the 16.7 and 10-pound treatments and slightly higher for the 8-pound treatment, one would come out with theoretical calculations like those shown in Table XXXIV. The significance of the magnitudes of these amounts below 355°C will be suggested in the following paragraphs.

The Gross Characteristics of the Residual Creosotes in Soil-Block Tests of Weathered Blocks

Madison test data for average losses of creosotes by weight, from treated blocks, during the periods of weathering and reconditioning are rounded off and plotted in Fig. 33. The usual procedure was to bring the weathered blocks into the laboratory and place them under constant temperature and humidity of 80°F and 30 per cent until the blocks came to approximate weight equilibrium. Examination of the figure indicates very definitely (1) heavier losses in the lower retentions respectively for all of the creosotes, and (2) lower losses for the higher residue oils than for the lower residue oils. These conclusions follow naturally from a consideration of the distillation ranges of the oils and the treatment conditions, confirming the inferences from other loss data already discussed.

These losses of creosote from 8- to 10-pound groups of weathered treated wood blocks seem to be in the neighborhood of 40-50 per cent by weight or by extraction. The aim of the weathering techniques now being developed at Bell Telephone Laboratories is to reach some definable end point in the protective life of the preservative that will reflect the end point or failure point of test plot specimens or poles in line.

Other things being equal, one can then consider and plan for treatment retentions sufficiently high to assure a protective residual of a given preservative for a long, economical service life of the treated plant units. This service life is not necessarily an indefinitely long life, nor is it the maximum physical life that might be obtained by using relatively larger quantities of preservatives than might be consistent with cleanliness requirements. Implicit is the idea of avoiding at all times heavy maintenance and replacement costs on account of decay, but particularly in the early life of the line. To accomplish these ends it is necessary to know how much preservative to use.

The following interpretation of the results of soil-block tests is confined to weathered block experiments, for the practical reasons previously cited. The interpretation is not offered as something entirely new; but it looks like a good working hypothesis, and it seems to help in explaining what may be happening in laboratory soil-block tests of creosote.

TABLE XXXV — SUMMARY AND INTERPRETATION OF SOIL-BLOCK TESTS

Weathered, creosoted southern pine sapwood blocks; creosote losses; amounts and gross characteristics of residual oils at threshold retentions for *Lentinus lepidus*.

1	2	3	4	5	6	7	8	9	10	11
Item	Creosote No.*	Specific gravity 38/ 15.5°C	Residue above 355°C per cent	Threshold lb/cu ft	Per cent loss	Creosote loss lb/cu ft	Residual creosote lb/cu ft	Calculated residue above 355°C	Residual creosote	
									>355°C lb/cu ft	<355°C lb/cu ft
1	1	1.065	18.5	9.8	53.1	5.2	4.6	39.4	1.81	2.79
2	7	1.077	20.5	9.0	47.8	4.3	4.7	39.3	1.85	2.85
3	2	1.081	30.6	10.2	47.1	4.8	5.4	57.8	3.12	2.28
4	6	1.093	34.2	9.0	37.8	3.4	5.6	55.0	3.08	2.52
5	3	1.108	50.4	12.2	30.3	3.7	8.5	72.3	6.15	2.35
6	8	1.115	53.2	9.4	25.5	2.4	7.0	71.4	5.00	2.00
7	9a		21.2	5.7	40.4	2.3	3.4	35.6	1.21	2.19
8	9	1.001	20.0	5.8	43.1	2.5	3.3	35.1	1.16	2.14
9	10a		14.4	6.7	50.9	3.4	3.3	29.4	0.97	2.23
10	10	1.068	15.2	6.9	52.2	3.6	3.3	31.8	1.05	2.25
11	11	1.038	18.0	6.5	47.7	3.1	3.4	34.4	1.17	2.23
12	M1	1.107	41.9	8.0	33.8	2.7	5.3	63.3	3.35	1.95
13	M2	1.070	18.1	8.3	50.6	4.2	4.1	36.6	1.51	2.59
14	BTL 5340	1.088	20.9	7.5	46.6	3.5	4.0	39.1	1.57	2.43

* Creosotes 1, 2, 3, 6, 7, 8, 9, 10 and 11 are those in use in the Cooperative Creosote Tests (see Bibliography, References 12 and 39). Oils 9a and 10a are samples from the same lots as numbers 9 and 10. (See Bibliography, Reference 36.) For oils M1 and M2 see Bibliography, References 37 and 38. Creosote 5340 is shown in Table II.

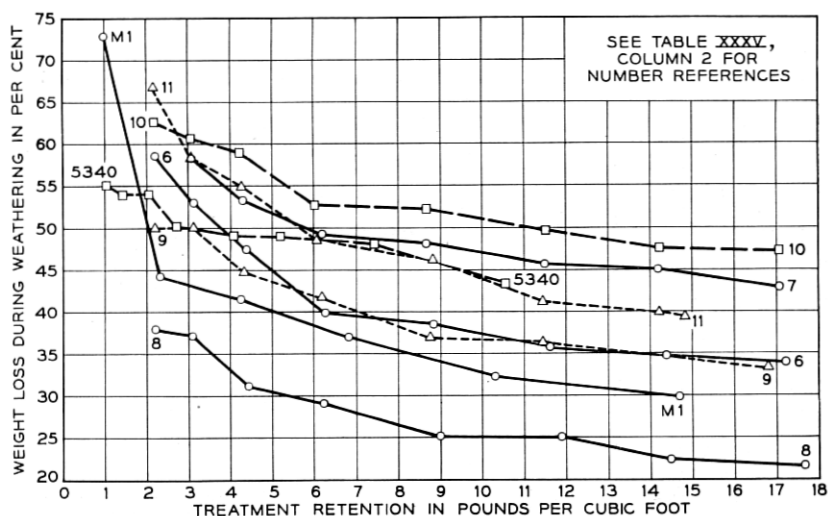


Fig. 33—Creosote losses in weathered, $\frac{3}{4}$ -inch cube, southern pine sapwood test blocks; relation of per cent loss of total creosote to original residue above 355°C and retention at treatment, lb/cu ft. All treatments were made with toluene creosote solutions. The total elapsed time from treatment to final reconditioned weight was about 105 days, including 60 days outdoor exposure on the Laboratory roof at Madison, Wis. See Table XXXV for number references.

Table XXXV is a condensed set of data on fourteen lots of weathered, creosoted $\frac{3}{4}$ -inch cube blocks. All of the tests were run at the Forest Products Laboratory at Madison, Wisconsin, in the Division of Forest Pathology, under the direct supervision of the same investigator, Dr. Catherine G. Duncan.^{36, 37, 38, 39, 41} The test fungus was *Lentinus lepideus*, Mad. 534. Ten of the tests have been run in cooperation with Bell Telephone Laboratories, and four have been run more or less concurrently with other cooperators. The technique for handling the weathered blocks has been essentially the same, and it has been rigidly controlled, except for the vagaries of the weather itself, at all essential points.

The data for the creosotes (Cols. 3 and 4), for the thresholds (Col. 5), and for the amount of residual oil in the blocks at the time they were placed in test (Col. 8), and the per cent and amount lost (Cols. 6 and 7) are all taken from the published reports or from manuscripts either ready³⁶ or in preparation for publication.⁴¹ The writer has calculated the residues in the residual oils, and the respective amounts remaining above and below 355°C (Cols. 9, 10 and 11) in pounds per cubic foot, on the assumption that all the loss occurred in the fractions boiling below 355°C . Particular attention is directed to the figures in Col. 11 — the calculated amounts remaining of the fractions boiling below 355°C . In terms of

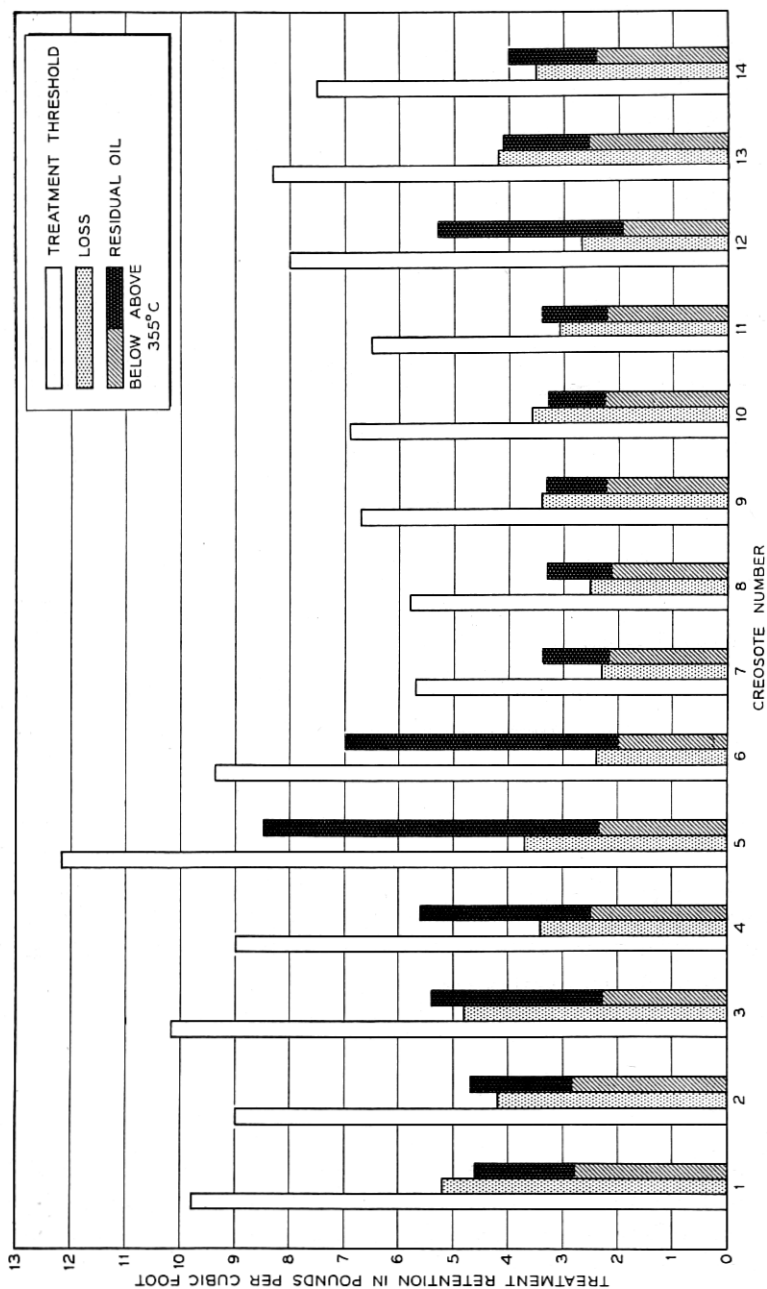


Fig. 34—Soil-block tests of fourteen creosotes against *Lentinus lepideus*; weathered southern pine sapwood blocks; comparison of treatment thresholds, losses during weathering and reconditioning, and gross characteristics of residual creosotes in terms of calculated amounts remaining of fractions above and below 355°C; based on Madison data. See Table XXXV.

pounds per cubic foot the over-all picture of the relative threshold amounts of creosote at the time of treatment, the oil lost, and the calculated proportional parts of the residual oil — after weathering — above and below 355°C, are shown graphically in Fig. 34. All of the data are in terms of pounds per cubic foot. If one bears in mind that the thresholds (Col. 5, Table XXXV) as given by the Madison investigators were located by a combination of visual observation of the blocks and extrapolation of straight lines through the weight loss data one may conclude that *in all of these tests the results were essentially the same for all the*

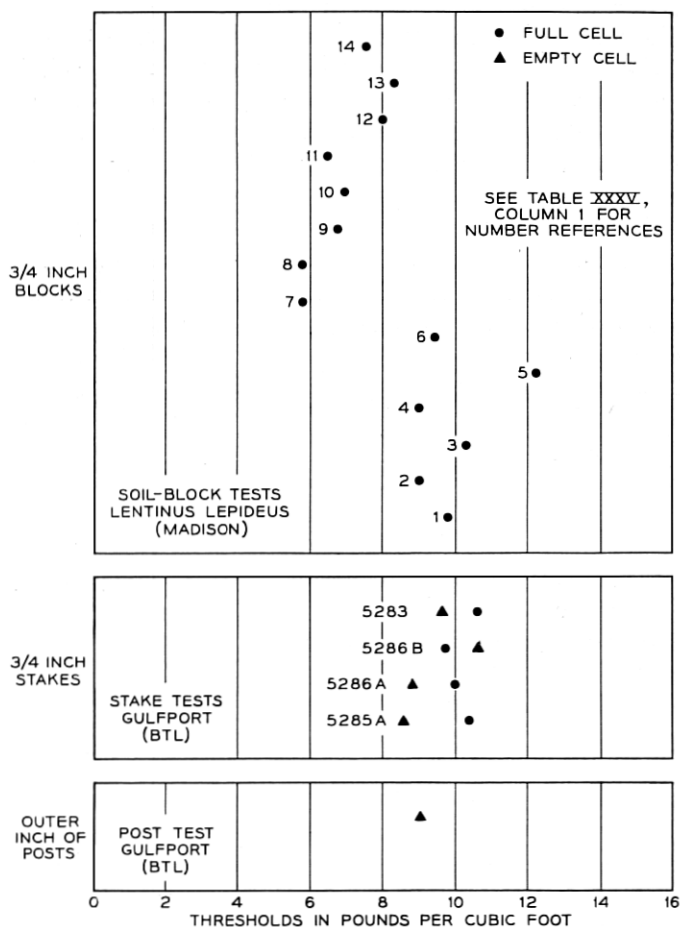


Fig. 35—Relative values of creosote thresholds by soil-block tests, $\frac{3}{4}$ -inch stake tests, and post tests.

creosotes with respect to the indicated threshold amounts distilling below 355°C. Statistically, the figures in Table XXXV Col. 11, are not significantly different. Slight changes in estimating the thresholds would conceivably bring them all to approximately the same level. In any given set of experiments the level would also vary with the duration (and types) of a different weathering cycle.

It will be recognized that no attempt has been made to separate or define the gross components of the fraction remaining below 355°C. This calls for more study, and for the development of refined methods of extraction and assay by weight and by distillation. Also, no attempt has been made to interpret the value or significance of the residue above 355°C, either because of its possible retardation of the evaporation of lower fractions, or because of some potential mechanical blocking effect. The whole interpretation is based on the simple division of the creosotes into two parts, the part distilling below 355°C and the part distilling above 355°C. Refinement will depend upon better future experimental evidence.

As an illustration of the application of the hypotheses discussed in the preceding paragraphs, one may reexamine the data from the weathering wheel experiments.⁸⁹ Rhodes, Roche and Gillander used one creosote retention only in their weathering wheel experiments, namely 16.7 pounds per cubic foot. In commenting on their work C. S. Reeve^{87 p. 78-79} noted Rhodes' emphasis on "the fact that toxicity without permanence is just as worthless as permanence without toxicity". Reeve and his colleagues carried out somewhat similar weathering experiments using slabs of wood about $\frac{1}{8}$ inch thick, exposing the treated pieces to somewhat lower temperatures than those in the Rhodes' experiments and "following a procedure with circulated air, and heat, and water". The plan of the experiments called for the conduct of "weathering cycles with reduced increments of various oils in order to get down finally to a percent of impregnation right at the end of a weathering cycle which would actually yield a rotting specimen of *Lentinus lepideus*". The work had not progressed far enough to accomplish this end, but Reeve says "The results . . . are in very close corroboration of what Mr. Rhodes has found. In other words, our loss curves with different oils running from relatively low residues to relatively high residues, have been almost parallel, I believe, with the loss curves which he has shown . . ."

Rhodes et al used essentially an agar-block method for testing their weathered blocks against *Lentinus lepideus*, Mad. 534, the same strain as that used in the Madison tests. The residual creosotes in the blocks treated with Oil I and Oil II (Table XXVI) are calculated to have been

5.90 and 7.97 pounds per cubic foot at the end of the nine-week weathering cycle (Table XXXIII); and by extraction these residuals contained 3.03 and 3.12 pounds per cubic foot of the fractions distilling below 355°C. On the basis of residue change alone these amounts are calculated at 2.73 and 2.36 pounds respectively. Rhodes states^{89 p. 76} "In no case was a weathered specimen attacked by the fungus". Paraphrasing his next sentence, this proved that both Creosote I and Creosote II at a treatment retention of 16.7 pounds per cubic foot "were affording adequate protection at the end of nine weeks, equivalent to many years of actual service". Was there any reason to expect such specimens to decay?

It is easier to attempt an answer to that question now than it was in 1934. Duncan⁴¹ shows a treatment threshold retention for "conditioned", i.e., unweathered, blocks of 1.6 pounds per cubic foot by agar-block tests, which is in close agreement with an average of 1.56 calculated from recent European tests reported by Schulze, Theden and Starfinger.⁵⁴⁽¹⁾ Of more importance for the question at hand is Duncan's weathered agar-block creosote threshold, given as 5.0 pounds per cubic foot. The loss in creosote at this 5 pound level was 57 per cent, which left 2.2 pounds per cubic foot of residual creosote in the blocks. The residue is calculated to have risen, as a result of weathering losses of the lower fractions, from an original 20.9 per cent (Table II) to 48.5 per cent; and on the basis of this figure the 2.2 pounds of residual oil consisted of 1.07 pounds per cubic foot of the fraction above 355°C and 1.13 pounds of the fraction below 355°C.

The Rhodes' nine-week weathered blocks still contained roughly two to three times this amount below 355°C. One may conclude that the nine weeks weathered blocks should not have shown decay under the culture conditions; and certainly none of the more briefly weathered blocks should have shown evidence of attack. If original retentions of 10 and 8 pounds of creosote had been used in the experiments the test fungus might have attacked nine and twelve weeks weathered 8-pound blocks treated with either Creosote I or Creosote II (Table XXXIV).

Using the results of the Madison tests shown in Table XXXV and Fig. 34 as indices of what might have happened if Rhodes, Roche and Gillander had used 16.7, 10.0 and 8.0 pounds at treatment and if — instead of employing an approximate agar-block technique — they had run their evaluation tests by the soil-block method, one would have expected decay to show up as indicated by the horizontal lines in the data columns in Table XXXIV. In other words, in the 16.7 pound treatments *Lentinus lepideus* would probably have attacked the blocks if they

had been weathered twelve weeks; and the 10.0 blocks would have been attacked at the end of five weeks weathering; while the 8-pound blocks would have been attacked after three weeks and about two weeks in the case of Creosote I and II, respectively. Under all these assumptions the treatment threshold for creosote in these block tests would probably have been set at above 8 pounds, and possibly around 9 pounds or more per cubic foot, in 1934; which would agree very well with the evidence obtained from soil-block tests, $\frac{3}{4}$ -inch stake tests, and test posts that has been presented in this paper.

Older records show very substantial quantities of the fraction below 355°C remaining in well treated wood after long service. Alleman's 1907 paper¹ is a classic. Writing of the increasing use of creosoted wood he states: "Recent reports . . . have clearly shown that, while proper treatment gives remarkably good results, much of this timber was not properly treated and has not lasted as it should". All of which in his opinion "... makes it imperative that we should know, as completely as possible, just what constitutes efficient creosote treatment. The different sorts of oils are believed to have different preservative values when injected into timber, but there is, unfortunately, a lack of uniformity in opinion".

Alleman chose, as the best method for finding some of the answers, an extraction of oils from treated timber that had given good service. As solvents he used absolute alcohol and subsequently anhydrous benzene. He fractionated the extracted creosotes with a view to determining the character of the oils, deciding to make his cuts so as to collect the distillate as follows: I to 170°C; II 170–205°C; III 205–245°C, which he regarded as the naphthalene fraction; IV 245–270°C; V 270–320°C; VI 320–420°C; and VII the residue above 420°C.

The wood from which he extracted the creosotes consisted of ties, mostly British, piles from England and the United States, paving blocks, and a section of creosoted wood duct removed in perfect condition after fourteen years service in Bell Telephone plant in Philadelphia. The English piles had been in service forty-three years, the other old samples all averaged a little over twenty years. Alleman's extractions showed — after all these years in use — that there were on the average over 9 pounds per cubic foot of oil remaining in the ties and English piles; nearly 9 pounds remaining in the conduit; and about 16 pounds remaining in the American piles and the paving blocks.

The writer has calculated the residue above 355°C in these extracted oils to have varied from about 23 to about 42 per cent; and the pounds per cubic foot of oil distilling below 355°C remaining in the treated wood

ran from a low of about 5.5 pounds to about 11.0 pounds. The wood had apparently remained sound. Alleman cites the difficulties of arriving at precise judgments but concludes "that 10 pounds of creosote per cubic foot is ample for railroad ties, and that piles require from 10 to 20 pounds" according to location.

Alleman's discussion of the relative amounts of *light* and *heavy* oil that might be desirable are not applicable to present day oils and commercial conditions. His perplexities remain — in almost identical form or in modernized version — and his extraction results are a long way from those reported by Lumsden,⁷⁶ and those cited elsewhere in this paper. Breazzano was beginning the application of biological tests in Italy²⁰ with a view to correlating chemical and fungicidal characteristics of preservatives. This type of endeavor was later to be pressed vigorously by Bateman,⁸ whose work has already been mentioned.

If the interpretation offered is supported by additional experiments already under way the Madison data in Table XXXV and Figs. 34 and 35 will be recognized as representing one of the most consistent series of laboratory tests for the evaluation of creosotes that has ever been run.

The Evaluation of Greensalt

The satisfactory performance of posts and poles treated with greensalt^{74, 79} has been reported in Lumsden's paper.⁷⁶ So far the very satisfactory results at the Gulfport test plot are accurate indices of what has been found by examination of poles in line. The only data on greensalt treated $\frac{3}{4}$ -inch stake tests reported in this paper are shown in Table XII and Figs. 19 and 23. Summaries of additional stake data are now in preparation for publication.

The indications are that the threshold for greensalt under Gulfport conditions is 1.42–2.1 pounds per cubic foot for $\frac{3}{4}$ -inch stakes. The average life estimates for the two treatment groups — 0.57 and 1.17 pounds of dry salt per cubic foot — (Table XIII) compare most favorably with the estimates for the four creosotes in the same table. The number of greensalt specimens is large enough to warrant the conclusion that the lines used for estimating the threshold in Fig. 23, in their trend to fall off to the right soon after the sixth year of exposure, indicate that particular period as a critical one for comparisons and interpretations. Commercially treated southern pine poles meeting the standard specification requirements for retention — 1 pound of dry salt per cubic foot — have about 2.0 pounds of dry salt in the outer inch. The agreement between the stake and post tests seems good.

In soil-block tests^{70, 95} *Poria incrassata*, *Poria monticola* and *Lenzites trabea* have been resistant to greensalt K, whereas *Lentinus lepideus* is very susceptible. The writer interprets the results of these three types of evaluation procedure, by soil-block, by $\frac{3}{4}$ -inch stakes, and by posts or poles to mean that:

1. The conditions for decay, as far as *Lenzites trabea* are concerned, are much more favorable in the soil-block culture bottle than they are in the above ground part of a pole under normal outdoor exposure conditions;

2. The incidence of attack or infection at the ground line by *Lenzites trabea*, *Poria incrassata* and *Poria monticola* at Gulfport is relatively rare; and that

3. The success of greensalt K in southern pine poles may be attributed in large part to the susceptibility of the ubiquitous *Lentinus lepideus* to the combination of salts in the greensalt preservative.

Incidentally one may cite the following example of confirming results in tests of another salt preservative. Harrow's⁵¹ experiments with soil-block tests resulted in locating a threshold for zinc chloride on unweathered blocks at 0.28 pounds per cubic foot (at treatment) for *Lenzites trabea*, Madison 617 and 0.53 for *Poria vaporaria*. Richards and Addoms⁹⁵ found approximately 0.25 for Madison 617, and approximately 0.50 for *Poria monticola*, Madison 698. These two *Porias* are possibly the same species. The similarity in the thresholds appears to be the definite result of following the same technique, rather than a haphazard coincidence.

The Evaluation of Pentachlorophenol

The highly toxic properties of pentachlorophenol have been established by exhaustive Petri dish agar toximetric tests.⁵³ A 5 per cent solution of penta in a light petroleum solvent is the preservative of reference in the recommended standard test for evaluating oil-soluble wood preservatives of the National Wood Manufacturers Association.¹⁰⁹ This test, as pointed out previously, is an agar-block test. Duncan reports⁴¹ that threshold determinations based on soil-block tests of a 5 per cent solution of penta in petroleum (cf. Tables V and VI) against *Lenzites trabea*, a critical fungus for this preservative, have not varied more than ± 0.2 pounds from 4.8 pounds per cubic foot in 7 series of weathered block experiments over a five-year period. Recent Bell Telephone Laboratories' soil-block tests confirm this result, with the same fungus and the same petroleum carrier.

These results are confirmed at the Laboratories' Gulfport test plot

(unpublished data). In two separate series — 1937 and 1938 — $\frac{3}{4}$ -inch stakes were treated at retentions slightly below the threshold cited by Duncan⁴¹ with 5 per cent solutions of pentachlorophenol in light petroleum (gas oil) and with two coal tar creosotes. The performance after six and seven years was approximately the same for both the penta solutions and the creosotes. However, completely favorable results on test posts have been reported for Gulfport⁷⁶ and Saucier, Miss., test plots. Early tests on 2 x 4 inch stakes are now being critically examined, and more tests are in progress.¹⁸ Penta treated posts are installed in the Saucier plot, where they are under periodic observation and comparison along with posts treated with the cooperative creosotes.¹² All of these experiments will greatly facilitate correlation of the results of different test methods.

As far as pole line tests are concerned one can only echo the report⁷⁶ that up to this time not one of the tens of thousands of poles in line that were treated with either straight penta-petroleum or with mixtures of penta-petroleum and creosote have been reported as failing because of decay.

Swedish Creosote Evaluation Tests

Rennerfelt and Starkenberg⁸⁸ report that of fourteen stakes measuring 1.5 x 1.5 x 100 cm, that were cut from the middle and inner sapwood of creosoted Scotch pine poles, none are sound after ten years in the test plot (May, 1950). The stakes were rated as 3 with slight decay, 8 with medium decay, and 3 with severe decay. Apparently these results cannot be correlated with definite treatment retentions.

On the other hand, the same authors state that stakes measuring 2 x 5 x 50 cm, treated to an average retention of 5.55 pounds per cubic foot of creosote (undiluted) have all decayed in a 4.5-year exposure period in greenhouse decay chamber tests. Additional experiments have been started, presumably with stakes at higher retentions, "in order to determine whether it is possible to correlate results from such decay chamber experiments with the results obtained in field and service tests."

In another series of experiments Rennerfelt and Starkenberg find after seven years (as of May, 1950) that creosoted stakes measuring 2 x 5 x 50 cm are showing different degrees of resistance to wood-destroying fungi in their four different test plots. The difference in behavior in different test plots — which is more or less to be expected — holds true for salt as well as creosote treatments. Creosote and Bolidens (zinc-chromium-arsenic) are the better performing preservatives, with creosote in

the lead. However, there have been a total of three failures in the creosoted stakes treated with average retentions of 3.6 (two stakes) and 5.6 (one stake) pounds of creosote. Stakes treated to an 8.6 pound retention are showing slight to medium decay, and in two plots slight decay has been found on a total of three stakes treated to a 12.1-pound retention. The stakes are all treated without the addition of any diluent, such as toluene, to the creosote. The reader, bearing in mind the differences in the site conditions, can make interesting comparisons between the small stake test results obtained in Sweden and in the Gulfport test plot.

Of further interest, however, is the fact that in the Swedish tests, round posts treated with average retentions of 5.37 and 5.80 pounds per cubic foot are all rated as sound after seven years exposure. One can assume that at treatment the outer annual rings of such posts contained 8 pounds or more of creosote and this amount has been sufficient to protect the posts in the Swedish climate. Rennerfelt has stated personally to the writer that one would have to proceed with caution in Sweden in the direction of increasing the creosote retention for poles, on account of public reaction against bleeding. His test results — the only ones of their kind available from Europe to the writer's knowledge — seem to be in line with Bell Telephone Laboratories findings. They would be more interesting if he had used soil-block tests for correlation.

Shortening the Bioassay Test

Besides speeding up the weathering period by the use of a weathering wheel, or by the method of alternating water and controlled heat cycles now being developed at Bell Telephone Laboratories, there are two other avenues of approach to shortening the bioassay test. One is the use of thin wood veneer test units in place of wood blocks, in the methods of impregnation and exposure to fungus action proposed by Breazzano^{23, 72} and Hopkins and Coldwell.⁵⁵ Breazzano claims a maximum of accuracy because of uniform distribution in thin pieces of wood, 0.6–0.7 mm, of the preservative to be tested and because the fungus attack and passage *through* the thin strip gives a quick visual indication of the necessary protective threshold. He also claims advantages for his Italian method because it is not necessary to use any culture medium at all — he exposed his wood strips over water only (cf. Waterman et al¹²⁶) — and because no tedious weighing techniques and record making are required. His arguments are intriguing, but his method seems to be quite out of question for testing toxicity-permanence relations of volatile preservatives like creosote. Evaporation losses would be very rapid, close to those

reported by Cislak in discussion following the presentation of the Snell and Shipley paper,¹⁰⁸ and would approach those obtained by Boving in his impregnated filter paper experiments (see Table XXXI). Furthermore, the results of tests such as his, which involve the principle of inoculation by placing "fungus on wood," instead of "wood on fungus" as in the soil-block test, require a lot of translation to interpret their significance in practical wood preservation. Rabanus⁸⁶ brought this matter out into the debate very clearly 20 years ago. Liese et al⁷² answer Breazzano's objections to the block test.

There have been no further reports on the procedures followed by Hopkins and Coldwell. Their methods are subject to some of the same criticisms that have been mentioned in the preceding paragraphs, particularly if one were to consider such techniques in a search for a way to speed up the culture tests. However, whether one agrees with them or not, their introduction of the idea of applying strength tests leads directly to a discussion of strength losses, as against weight losses, as criteria for establishing preservative thresholds.

Toughness or Impact Tests for Determining Preservative Effectiveness

Trendelenburg¹¹⁵ published in 1940 his scheme for testing the strength of treated blocks that had been exposed to fungus attack. He was aiming at a technique that would shorten the time period of fungus tests on wood, but he was also looking for some other criterion than weight loss as a measure of fungus attack. He used the relative impact strength values of matched sound and decaying test specimens as indices of the degree of decay. Boards were carefully quarter-sawed first into pieces of double specimen width plus saw kerf tangentially, and of double specimen length. From these blanks four test specimens were cut that measured 8.5 x 8.5 x 120.0 mm. The pairs were considered to be matched laterally and vertically since every effort was made to cut them from the same annual rings. One piece from each pair was exposed to fungus attack and the other served as a control. The fungus cultures were made in Kolle flasks on malt agar. Specimens were placed radial side down directly on the growing fungus surface. In the pendulum testing machine the impact load was always applied to the upper radial face, so that the lower more or less infected radial face represented the tension side of the specimen in the breaking test.

Trendelenburg showed that the per cent change in strength caused by the test fungus in the early stages of decay was much more pronounced than the concomitant changes in weight or density. He called

attention to the fact that the German Standard³³ for testing wood preservatives contains a stipulation that a weight loss of less than 5 per cent shall not be considered significant unless there is visual evidence of actual wood destruction by the test fungus. He presents data to show that a loss of 50 per cent of the relative original impact strength in spruce and fir occurred after about fifteen days in test, and that the weight loss for the test pieces was *about 3 per cent only*. Impact strength seems to be affected much more than the bending or compression strength. He was confident that his method would not only shorten the time of the bioassay test but also give more reliable and more significant results than those based on weight loss alone.

After Trendelenburg's death his ideas have been further tested and developed by von Pechmann and Schaile.¹¹⁹ In addition to trying out the suitability of the strength test procedure, they have explored the changes in the wood structure with the microscope, and, as decay progressed, they have determined the gross relation between weight loss and solubility in sodium hydroxide.

They present as an example comparable data for the German Standard agar-block test and a test run by Trendelenburg's method, using pine wood (presumably *Pinus sylvestris*) and the test fungus *Coniophora cerebella*, against a proprietary preservative. The absorption of the preservative was essentially the same in each test; but not enough preservative was used to permit determination of the threshold. The main results were that in the impact strength test procedure in fifty days the strength reduction was 66.4 per cent and the weight loss 12.7 per cent; whereas in the standard agar-block procedure in four months the weight loss was only 2.0 per cent. Von Pechmann and Schaile feel that it is possible to save $2\frac{1}{2}$ to 3 months time by using the strength test technique, and that with proper attention to detail the results will be more definite and just as reliable as those obtained in the longer period required for the standard agar-block tests.

The precise Trendelenburg technique has not been tried out in this country in any comparative tests on wood preservatives but toughness test data on small specimens of wood and veneer, sound, fungus stained and decayed, treated and untreated, have been accumulating at the Forest Products Laboratory at Madison, Wis. The fact that strength loss begins earlier and may increase more rapidly than weight loss or than change in specific gravity in natural infections in the heartwood was shown by the writer²⁷ and his colleagues shortly after the end of World War I. Confirming data were secured in later tests.^{28, 98} Scheffer⁹⁷ showed the same results, on a more definite basis, by growing *Polystictus versicolor*

on red gum sapwood in large test tubes and testing matched specimens for various strength properties as decay progressed.

The Trendelenburg technique has possibilities, but it will be some time before one can say whether it is practicable to take full advantage of it in developing supplemental bioassay tests. The one outstanding difficulty in the way of extensive use of strength tests on small specimens lies in the procurement, in the very variable southern pine, for example, of a requisite quantity of straight grained quarter sawed wood for the manufacture of the *matched blocks*. Small scale check tests are practicable, according to the writer's experience. The cost of personal supervision and manufacture of any large number of specimens would appear in advance to be exorbitant. Still, strength loss as a result of attack in treated wood is important, and Trendelenburg's ideas may win more proponents, if only as a supplemental procedure, after the soil-block technique has become more firmly established and appreciated.

OTHER ACCELERATED BIOASSAY TESTS

There are a number of items that must be mentioned before bringing this long paper toward its conclusion. There are, for example, other types of outdoor exposure tests on wood and of laboratory block tests than those cited. Two types only will be used as illustrations of the efforts that are being made to evaluate preservatives by other than the traditional service test, namely: Verrall's^{120, 121} *above ground* outdoor testing procedure, and the experiments of Tippo et al¹¹⁴ with large block tests devised to determine effective concentrations of preservatives for prevention or control of decay in wooden ships.

Since the soil-block test is essentially a laboratory simulation of controlled ground line conditions, there is a need for some other type of test that will approximate the above ground conditions to which treated wood may be exposed. Verrall treats pieces of dressed nominal 2 x 4 inch southern pine sapwood and exposes them to the varying wet and dry, hot and cold weather conditions at Saucier, Miss. One of two pieces has a 45° end cut. This end cut is toe-nailed to the side of the other piece, which is then nailed upright on a supporting treated or untreated rail support, with the V up. This permits maximum hazard as far as catching water is concerned. His results are furnishing valuable information about pentachlorophenol, copper naphthenate and organic mercury compounds, for example. His techniques are applicable to other preservative problems, and other investigators are using his scheme in Canada for general studies,^{105, 106} and at Ann Arbor, Mich., for testing the amount

of preservative needed in the upper, or above ground section, of thin sapwood poles.

The work of Tippo and his associates represents one phase of an extensive set of experiments in which large ($6 \times 5 \times \frac{7}{8}$ inch) and small ($3 \times 5 \times \frac{7}{8}$ inch) specimens are made up to simulate a butt-block assembly, and exposed to the attack of certain critical fungi by adding to the block assembly another inoculum block ($3 \times 5 \times \frac{7}{8}$ inch) that has been thoroughly infected. The assembled units are kept in a warm and practically saturated atmosphere until the reaction of the fungus to the different preservatives can be determined. This work is being expanded in view of the importance of minimizing decay in wooden ships.

The point to be made here is that Verrall's tests and Tippo's tests should be evaluated carefully before the service test program is broadened extensively.

OTHER OBSERVATIONS

Some of the results of Suolahti's interesting studies¹¹¹ on the influence of wood at a distance on the intensity and direction of growth of fungus filaments (mycelium) have been confirmed by preliminary experiments at Bell Laboratories. Small sterilized southern pine sapwood blocks enclosed in either test tube or Petri dish cultures exert a positive pull on the filaments that is effective over a distance of several centimeters. The growth of the mycelium is more luxuriant, and the filaments are definitely drawn in the direction of the wood. Without attempting any interpretation of the significance of this phenomenon one may be permitted to point out that such studies strongly support the very great desirability — if not the necessity — of using wood in any studies directed toward evaluation of wood preservatives.

In view of the nearly forty years of prior work both here and in Europe, in which it was definitely established that certain higher fungi were the principal causes of decay in wood, it is hard to see why Weiss¹²⁸ spent so much time and such careful work on trying to test wood preservatives by using bacteria as his bioassay agents. Following the presentation of his paper before the Society of Chemical Industries in 1911 some of Weiss's critics pointed out that his methods were unrealistic as far as oil preservatives were concerned, one of his commentators suggesting that the proper approach to the problem of preservative evaluation was *to test treated and untreated wood*, (unsterilized and sterilized) under conditions favorable for fungus growth.

Tamura in 1931¹¹² used an assembly of two pieces of treated wood

molding between which he inserted a properly sized piece of untreated wood, the whole being exposed over the surface of an agar culture of the test fungus. He did not attempt to add a block of infected wood to his setup as Trippo did; but his procedure illustrates an attempt of some twenty years ago to test the protective action of preservatives in the laboratory. His statement that sterilization might drive off a significant amount of volatile preservative from freshly treated blocks, but that the sterilizing process would probably have very little effect on the preservative residual in weathered blocks anticipated similar views expressed in the present paper.

For testing initial toxicities one can still use the Petri dish or agar flask method; but it is about as unrealistic as Weiss's procedure as far as tests of toxicity and permanence of wood preservatives are concerned. The results can be presented for their academic interest, and the investigator can keep safely aloof from the perilous practical problems of wood preservation unless he attempts to translate his data into terms of *permanence and preservative value*. Then his practical colleagues as well as his technical friends point out to him, truly with a vengeance, the error and unrealistic character of his efforts.

It may be, as Rabanus⁸⁶ has suggested, that closely similar results can be obtained by the agar and by the agar-block method in the case of certain definite toxic chemicals, particularly water soluble ones. If so, the Petri dish or agar-flask method could be used with such preservatives, and the results of the tests could be applied in practice, *after* such agreement between agar and block tests was firmly proven and established.

It is completely unrealistic to attempt to arrive at significant values for the volatile fractions of creosote by confining them within a tightly closed culture dish. If such materials are really transient their toxic function can operate only during the early life of the treated wood. In a whole creosote, for example, the relatively higher toxicity of the volatile low boiling fractions supplies an important initial power to the preservative, which power is evidently diminished as the volatiles leave the treated wood. The degree of change in toxicity, as measured by the agar method, in new and aged or weathered creosotes, as shown by the works of Snell and Shipley,¹⁰⁸ Schmitz et al,^{102, 103} Baechler⁵ and others is distinctly realistic as an index of how an oil may be altered by time and weather — at least with respect to a measure of its toxic properties. Such changes have great practical significance when minimum quantities of a preservative are employed, either for reasons of economy or in order to insure cleanliness in the treated product. The trouble is that the results of the Petri dish or agar-flask method do not indicate directly *how much*

preservative to use with a view to providing the necessary residual and effective preservative.

The agar-block tests are somewhat better in this respect. While additional proof is necessary the results of such tests may be good indices of retention requirements for certain water-borne preservatives. The comparison of agar-block and soil-block tests made by Warner and Krause¹²² is incomplete, and therefore somewhat unsatisfactory, particularly in view of the title of their article. They do not follow through, and they repeat some of the inferential objections raised by others as to the effect of soil differences and methods of interpretation. The comparison by Finholt et al^{46, 47} is also inconclusive chiefly because of the nature and design of their experiments. So also, but to a lesser degree, are the comparisons that one may draw from the first two papers on the soil-block and agar-block tests from Madison,^{95, 35} which after all, were in the nature of preliminary or reconnaissance studies, introducing the first trials of an outdoor weathering technique, and developing the necessary steps in the broader and more comprehensive plans followed later.

Duncan⁴¹ has now brought out a full scale comprehensive study of the agar-block and soil-block techniques, which, however, deals with oil type preservatives only. She shows definitely — as was indicated in the earlier Madison work — that the test fungi are more aggressive under the more natural and more realistic environment of the soil-block cultures. Definite evidence of the very important better control of moisture content in the soil-block tests is presented. The soil-block thresholds for the different preservatives are generally higher in the soil-block tests, although the *order* of effectiveness is essentially the same.

Sedziak's¹⁰⁶ recent paper comparing results of his tests on buried soil-blocks and results of tests by a soil-block technique approximate to that used at Madison and at Bell Laboratories is not convincing with respect to the implied superiority of the buried block method. His paper covers work begun after the early soil-block was started at Bell Telephone Laboratories, but before the extensive experiments at Madison were initiated. Satisfactory comparison of the work of the Madison and Ottawa laboratories is difficult because Sedziak has used a different set of test organisms, including the European *Coniophora cerebella* for example, and *Lenzites sepiaria*, which is apparently not a satisfactory discriminating organism for creosote and pentachlorophenol. He has omitted the very critical *Lentinus lepideus* that has been employed at Madison since 1944. While he interprets threshold retentions for penta and for copper naphthenate that are close to those obtained at Madison, the steps in his gradient retentions leave one wishing that the Madison and Ottawa

plans could have been more closely harmonized before the most recent Ottawa work was started, at least to the point of some tests with the same procedures and same test fungi. However, nothing in Sedziak's results negates the general conclusions reached at Madison and at Bell Laboratories about the value of the present soil-block technique for the testing of creosote and other oil type preservatives.

CONCLUSIONS

1. In the course of this paper evidence and interpretations have been presented to show that the soil-block technique incorporating a weathering procedure is a practical, rapid method of bioassay and that the results obtained from this method are in general agreement with accelerated stake and long time pole-diameter post tests on the same or similar preservatives. For example, it is shown that a creosote retention at treatment of 9-10 pounds per cubic foot is necessary to insure a satisfactory degree of preservative permanence in test blocks, in $\frac{3}{4}$ -inch stakes and in the outer 1 inch of test posts. There is no reason to believe that this minimum limit does not apply to the outer 1 inch of poles in line.

2. The good reputation of well creosoted material is reaffirmed by these findings. Moreover, they show why failures have occurred and indicate what should be done to forestall such failures.

3. Since the results of the block tests are essentially the same as the results of the much longer stake and post tests, the block test data can be used at once as a basis for the establishment of the necessary amounts of the respective preservatives distribution in the wood where they will do the most good. The possibilities of bleeding increase as the retention is increased, so the bioassay technique becomes an essential tool for closer appraisal of effective wood preserving power.

4. It is important now to recognize that the soil-block results with creosote, for example, reveal the fact that the results of the European agar-block tests are — in all cases — too low to represent indices of actual requirements in treated wood. Therefore, the results for creosote tabulated by Schulze, Theden and Starfinger^{54 (1)} have to be corrected upward by some multiplying factor; perhaps of the order of three or four, before they can be correlated with the results on blocks, stakes and posts presented in this paper. For the true scientific solution of the problems of these different techniques, perhaps an international task force may be required.

5. The interpretations presented in this paper indicate that the use of

the Laboratories' controlled weathering procedure will provide a means for determining truly effective threshold retentions for oil-type and salt-type preservatives for comparable service requirements.

6. These threshold determinations are supplying data that would have been most valuable in planning retention gradients in small stake and pole diameter post tests in test plots; and, in general, the bioassay tests explain and confirm the unequivocal results of experience.

7. Through the soil-block test a ready method is available for use in the quality control of a wood treater's current product at plants where large supplies of preservatives are received from one or more constant sources and are stored in bulk. No present method of bioassay control is sufficiently rapid to be effective or practicable on mixed samples taken at treating plants receiving preservatives at frequent intervals in small lots from varied sources.

8. The soil-block development may soon make it possible to reach approximately the ideal in which the long-time service tests of treated material in line will confirm the Laboratories' rapid test results with respect to preservative requirements. Coupled with results-type requirements (wherein the end product — not the steps of manufacture and treatment — are defined) viz., (a) retention *in the wood*, (b) penetration, and (c) cleanliness, there will then be even better assurance than at the present of the quality performance always expected under Bell System specifications.

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It is hoped that the preceding pages will be accepted by the reader for what they are — condensed results of teamwork over the years — into which have been woven some individual ideas, opinions and interpretations. The writer is responsible for the literature review and for the selection of the items discussed. In one way or another various members of the Timber Products Group of Bell Telephone Laboratories have contributed to the collection and analysis of the supporting data. Thanks are due especially for help on the soil-block section to J. Leutritz, Jr., L. R. Snoke and Ruth Ann MacAllister; on the $\frac{3}{4}$ -inch stake section to J. Leutritz, Jr.; on the text post and pole line sections to G. Q. Lumsden and A. H. Hearn; on the review and editing of the manuscript to R. J. Nossaman, F. F. Farnsworth and G. Q. Lumsden; on the zealous checking and assembly of test, tables and figures to Jean E. Perry; and on the correlation of the Madison cooperative test data to Catherine G. Duncan. Dorothy Storm's untiring efforts as a secretarial task force are deeply

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