A COMPARISON OF WATER REPELLENCY TEST METHODS AS USED FOR WOOD

and

COMPARISON OF THREE COMMERCIAL OIL-SOLUBLE WOOD PRESERVING SOLUTIONS

May 13, 1948

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Ann Arbor, Michigan
May 13, 1948

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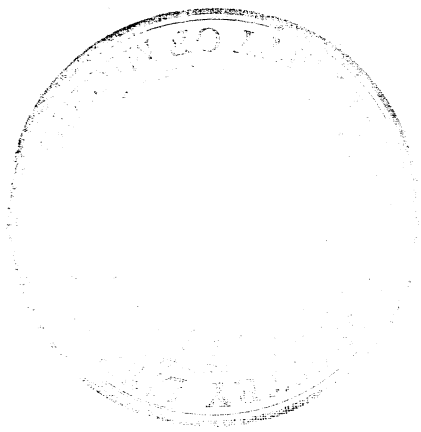
Dear Professor Patronsky:

These reports on two topics are respectively submitted in compliance with requirements for the degree of Master of Wood Technology.

Sincerely,

Duane L. Konaga

Duane L. Konaga



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Duane L. Kenaga

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SUMMARY

The Wood Technology Laboratory of the University of Michigan carried out a series of tests on five water repellent solutions by three different methods in cooperation with a program sponsored by the National Door Manufacturers Association in an attempt to determine a standard method of evaluating water repellent solutions. This report is made only on the work done here.

Method 1, the Protection Products 7-day Method, was determined to be probably the best method, but also would not be entirely satisfactory due to variability of results between different boards.

Method 2, comparison with boiled linseed oil as a standard, was determined to give results too low for a standard.

Method 3, comparison with a 0.5% by weight wax-mineral spirits solution was considered to be usable but not as reliable as Method 1.

The study also showed that there is a large variability of effectiveness of a solution on different boards. Repeated tests with a solution on the same board showed little variation. On this basis it was recommended that a study be made of a method by which wood material would first be selected in an attempt to reduce variability of results.

INTRODUCTION

Because of the inherent nature of wood, methods for evaluating water repellents solutions have been difficult to establish.(1) In an attempt to agree on a standard test the National Door Manufacturers Association, Inc., Chicago, Illinois, set up a program to determine which of three methods would be the most acceptable.

As stated in Appendix A, five water repellent solutions were to be supplied by the N.D.M.A. Tests were to be made with these solutions by three specified methods, and results forwarded to them. This report is made on the phase of the work carried out in the Wood Technology Laboratory of the Forestry School, University of Michigan, and represents only a portion of the total N.D.M.A. program.

PROCEDURE

The procedure used for conducting the series of tests is detailed in Appendixes A and B. Each test was carried out as follows:

1. Ten different clear, kiln dried, straight grain ponderosa pine sapwood boards were selected. Selection of sapwood was based on the benzidene-sodium nitrite test for differentiation of heart from sap in pine wood. See Appendix C.

2. Test blocks, or wafers, were 1/4" slices taken from the boards previously machined to 1-3/8" x 1-5/8", the 1/4" dimension being in the direction of the grain, and the growth rings being almost straight and parallel to the 1-5/8" dimension.

3. Specific gravity, rings per inch, regularity of growth, and grain deviation from the horizontal were determined for each test board. These physical characteristics are presented in Table 8, columns 2, 3, 4, and 5, page 15.

4. Four wafers were cut and numbered in rotation from each board. Wafers Nos. 1 and 2 were used for Method 1, wafers No. 3 for Method 2, and wafers No. 4 for Method 3.

5. The wafers were conditioned for at least one week in a relative humidity room at 80°F and 80% relative humidity, which is equivalent to an equilibrium moisture content of 15%. Since the standard test requires a moisture content of 12%, the blocks were left in the laboratory for 16 hours. Previous experiment had shown that the blocks fell to a 12% m.c. in this period under the prevailing conditions.

6. Five test solutions were furnished by the N.D.M.A., one solution to be used for each test in Method 1. Method 2 was set up to utilize raw linseed oil. However, the work reported on by this paper was done with boiled linseed oil as the standard solution. In Method 3 the standard solution consisted of 0.5% paraffin wax, melting point 128°F., in mineral spirits. The standard solutions of Method 2 and 3 were used as a minimum of water repellency against which the test solutions of Method 1 were compared.

7. At approximately the same time and in the same manner, the ten No. 1 wafers were dipped for 30 seconds in the test solution; the ten No. 3 wafers were dipped for 10 seconds in the boiled linseed oil; and the ten No. 4 wafers were dipped for 10 seconds in the wax-mineral spirits solution. Wafers No. 2 remained untreated to serve as controls. They were at all times kept under the same moisture conditions.

8. The treated wafers and controls were reconditioned for 1 week as given in step 5.

9. Each block was measured to the nearest thousandth of an inch in the 1-5/8" direction. This was done by means of a dial gage graduated in thousandths of an inch mounted on a horizontal metal plate. A rigid metal bar mounted on the plate is placed at such a distance from the spindle of the gage that it will accommodate the wafer and a second removable metal bar. The wafer is placed between the two metal bars which are machined

to touch the 1/4" x 1-3/8" surfaces only in center areas of 1/8" x 1-3/8" cross section. By this method irregularities in the surface of the wood block are evened out.

10. After the wafers were measured, they were immersed in a constant temperature water bath held at 80°F. Constant immersion was made possible by means of suitably built wire racks. Handling one wafer per 1/2 minute, the measurements and immersions were continued for 30 minutes, at which time they were removed in the same order of rotation at 1/2-minute intervals and again measured. By this method, measurements on each block were taken immediately before and after a 30-minute immersion period.

11. The percent effectiveness of the test solution or standard solution was calculated by dividing the difference between the amount of swelling of the treated and untreated wafer by the amount of swelling of the untreated wafer.

RESULTS

The results of the five tests which are summarized in Table 1, page 5, are depicted in Figure 1, page 6. Tables 2 through 6, pages 7 to 12 are the results of the five tests.

The figures given in Table 1 also include the standard deviation calculated by the accepted formula, $\sigma = \pm\sqrt{\frac{\Sigma(x)^2}{n}}$

DISCUSSION OF RESULTS

Evaluation of Test Methods

The summary in Table 1, page 5, shows the effectiveness of the test solutions by Method 1 and the effectiveness of the standard solutions of Methods 2 and 3 to which the test solutions can be compared. By Method 2, the effectiveness of boiled linseed oil is about 20 % to 25 % effective with a

Table 1. SUMMARY OF RESULTS OF TESTS.

(See Tables 2 to 6.)

Test No.	Average Percent Effectiveness*		
	Method 1. Test Solutions.	Method 2. Linseed Oil. BOILED	Method 3. Wax-Mineral Spirits Solution.
1.	66.2 ±3.0	21.5 ±10.1	52.7 ±9.3
2.	8.8 ±7.3	20.5 ±10.2	51.5 ±8.1
3.	14.9 ±7.7	26.0 ±10.8	55.8 ±5.0
4.	12.8 ±8.8	24.7 ±12.8	48.4 ±8.9
5.	66.5 ±3.7	25.3 ± 9.6	53.1 ±6.3

*Standard deviation included.

SUMMARY OF RESULTS OF TESTS

CROSS HATCHING INDICATES STANDARD DEVIATION - PLUS AND MINUS

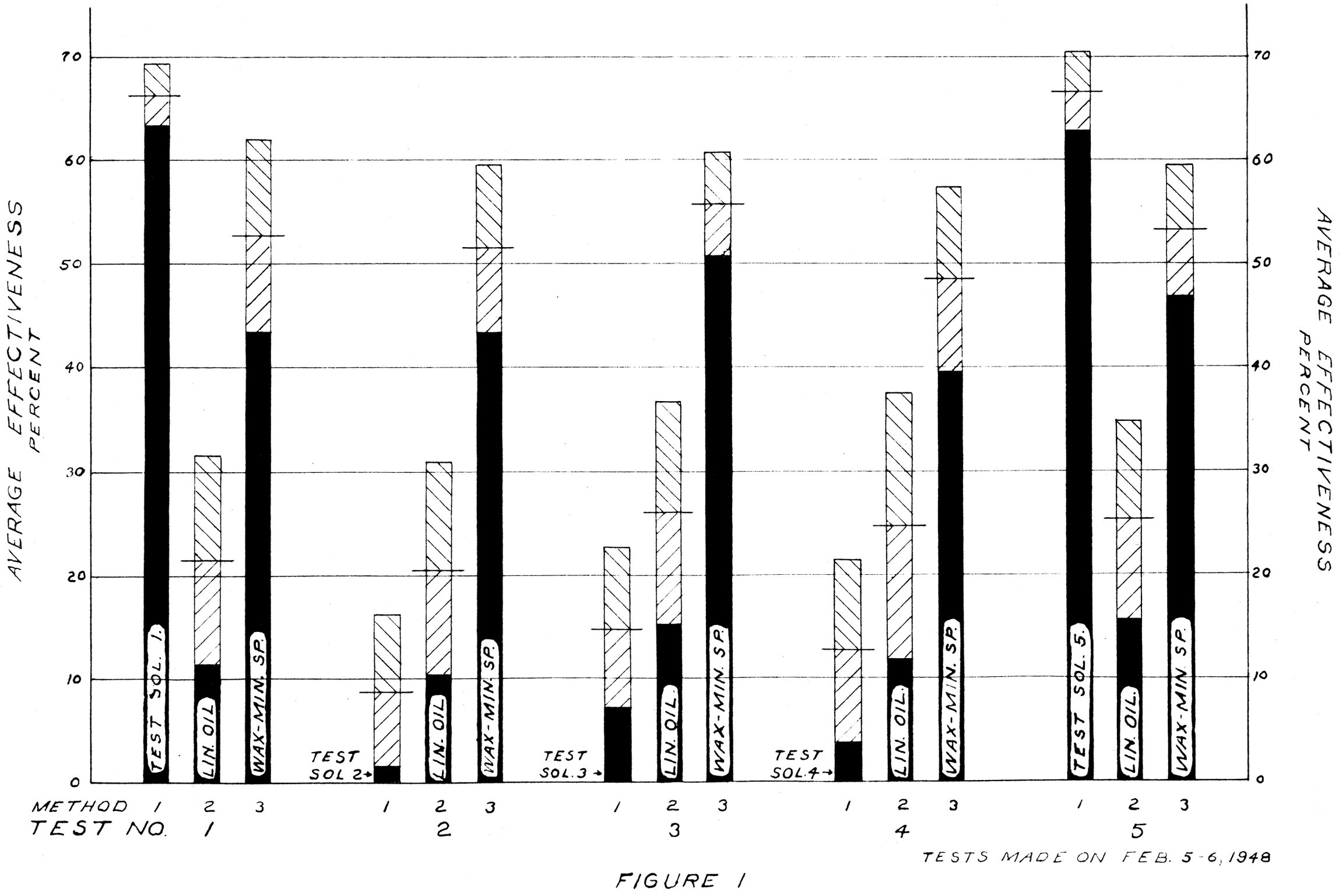

FIGURE 1

Table 2. RESULTS OF TEST 1.

Board No.	Swelling of untreated wafers.	Method 1			Method 2			Method 3		
		Swelling of wafers treated with Solution 1.	Difference in swelling.	Effectiveness of Solution 1.	Swelling of wafers treated with Linseed Oil.	Difference in swelling.	Effectiveness of Linseed Oil.	Swelling of wafers treated with Wax-Mineral Spirits Solution.	Difference in swelling.	Effectiveness of Wax-Mineral Spirits Solution.
1	2	3	4	5	6	7	8	9	10	11
	A	B	A - B	$\frac{A-B}{A} \times 100$	C	A-C	$\frac{A-C}{A} \times 100$	D	A-D	$\frac{A-D}{A} \times 100$
	inches	inches	inches	percent	inches	inches	percent	inches	inches	percent
6	0.067	0.024	0.043	64.17	0.056	0.011	16.42	0.039	0.026	38.80
8	0.067	0.021	0.046	68.65	0.041	0.026	38.80	0.028	0.039	58.21
9	0.066	0.026	0.040	60.61	0.056	0.010	15.15	0.029	0.037	56.06
10	0.086	0.026	0.060	69.77	0.069	0.015	17.44	0.033	0.053	61.63
11	0.075	0.024	0.051	68.00	0.057	0.018	24.00	0.028	0.047	62.66
12	0.076	0.028	0.048	63.15	0.069	0.007	9.21	0.049	0.027	35.53
13	0.079	0.026	0.053	67.08	0.073	0.006	7.59	0.045	0.034	43.04
14	0.053	0.018	0.035	63.04	0.037	0.016	30.19	0.023	0.030	56.60
15	0.061	0.020	0.041	67.21	0.039	0.022	36.06	0.027	0.034	55.74
16	0.090	0.027	0.063	70.00	0.072	0.018	20.00	0.037	0.053	58.89
Ave.	0.072	0.024	0.048	66.17	0.057	0.015	21.49	0.034	0.038	52.72

Table 3. RESULTS OF TEST 2.

Board No.	Swelling of untreated wafers.	Method 1			Method 2			Method 3		
		Swelling of wafers treated with Solution 2.	Difference in swelling.	Effectiveness of Solution 2	Swelling of wafers treated with Linseed Oil.	Difference in swelling.	Effectiveness of Linseed Oil.	Swelling of wafers treated with Wax-Mineral Spirit Solution.	Difference in swelling.	Effectiveness of Wax-Mineral Spirit Solution.
1	2	3	4	5	6	7	8	9	10	11
	A	B	A-B	$\frac{A-B}{A} \times 100$	C	A-C	$\frac{A-C}{A} \times 100$	D	A-D	$\frac{A-D}{A} \times 100$
	inches	inches	inches	percent	inches	inches	percent	inches	inches	percent
6	0.064	0.063	0.001	1.56	0.055	0.009	14.06	0.040	0.024	37.50
8	0.067	0.052	0.015	22.38	0.043	0.024	35.82	0.029	0.036	53.73
9	0.065	0.054	0.011	16.92	0.057	0.008	12.31	0.031	0.034	52.31
10	0.086	0.074	0.012	13.95	0.065	0.021	24.42	0.038	0.048	55.81
11	0.073	0.063	0.010	13.70	0.055	0.018	24.66	0.027	0.046	63.01
12	0.076	0.076	0.000	0.00	0.071	0.005	6.58	0.048	0.028	36.84
13	0.081	0.078	0.003	3.70	0.072	0.009	11.11	0.044	0.037	45.68
14	0.051	0.052	0.000	0.00	0.036	0.015	29.41	0.023	0.028	54.90
15	0.063	0.058	0.005	7.94	0.041	0.022	34.92	0.027	0.036	57.14
16	0.089	0.082	0.007	7.87	0.079	0.010	11.23	0.037	0.052	58.43
Ave.	0.071	0.065	0.006	8.81	0.060	0.014	20.45	0.034	0.037	51.53

Table 4. RESULTS OF TEST 3.

Board No.	Swelling of untreated wafers.	Method 1			Method 2			Method 3		
		Swelling of wafers treated with Solution 3.	Difference in swelling.	Effectiveness of Solution 3.	Swelling of wafers treated with Linseed Oil.	Difference in swelling.	Effectiveness of Linseed Oil.	Swelling of wafers treated with Wax-Mineral Spirit Solution.	Difference in swelling.	Effectiveness of Wax-Mineral Spirit Solution.
1	2	3	4	5	6	7	8	9	10	11
	A	B	A-B	$\frac{A-B}{A} \times 100$	C	A-C	$\frac{A-C}{A} \times 100$	D	A-D	$\frac{A-D}{A} \times 100$
	inches	inches	inches	percent	inches	inches	percent	inches	inches	percent
6	0.070	0.066	0.004	5.71	0.051	0.019	27.14	0.036	0.034	48.57
8	0.068	0.054	0.014	20.59	0.038	0.030	44.12	0.029	0.039	57.35
9	0.067	0.059	0.008	19.40	0.055	0.012	17.91	0.029	0.038	56.72
10	0.089	0.076	0.013	14.61	0.063	0.026	29.21	0.037	0.052	58.43
11	0.075	0.067	0.008	10.67	0.051	0.024	32.00	0.030	0.045	60.00
12	0.078	0.073	0.005	6.41	0.071	0.007	8.97	0.045	0.033	42.31
13	0.083	0.067	0.016	19.28	0.074	0.009	8.43	0.036	0.047	56.63
14	0.052	0.048	0.004	7.69	0.039	0.013	25.00	0.024	0.028	53.85
15	0.065	0.057	0.008	12.31	0.042	0.023	35.38	0.029	0.036	55.38
16	0.092	0.062	0.030	32.61	0.063	0.029	31.52	0.038	0.054	58.69
Ave.	0.074	0.063	0.011	14.92	0.055	0.019	25.97	0.033	0.041	54.79

Table 5. RESULTS OF TEST 4.

Board No.	Swelling of untreated wafers.	Method 1			Method 2			Method 3		
		Swelling of wafers treated with Solution 4.	Difference in swelling.	Effectiveness of Solution 4.	Swelling of wafers treated with Linseed Oil.	Difference in swelling.	Effectiveness of Linseed Oil.	Swelling of wafers treated with Wax-Mineral Spirit Solution.	Difference in swelling.	Effectiveness of Wax-Mineral Spirit Solution.
1	2	3	4	5	6	7	8	9	10	11
	A	B	A-B	$\frac{A-B}{A} \times 100$	C	A-C	$\frac{A-C}{A} \times 100$	D	A-D	$\frac{A-D}{A} \times 100$
	inches	inches	inches	percent	inches	inches	percent	inches	inches	percent
1	0.063	0.049	0.014	22.22	0.049	0.014	22.22	0.035	0.028	44.44
6	0.069	0.066	0.003	4.35	0.056	0.013	18.84	0.042	0.027	39.13
8	0.069	0.057	0.012	17.39	0.043	0.026	37.68	0.033	0.026	37.68
9	0.069	0.061	0.008	11.59	0.063	0.006	8.69	0.033	0.026	37.68
10	0.086	0.080	0.006	6.98	0.059	0.027	31.39	0.037	0.049	56.97
11	0.070	0.054	0.016	22.86	0.046	0.024	34.28	0.028	0.042	60.00
12	0.077	0.071	0.006	7.79	0.074	0.003	3.89	0.047	0.030	38.96
14	0.056	0.052	0.004	7.14	0.034	0.022	39.28	0.027	0.029	51.78
15	0.063	0.055	0.008	12.70	0.042	0.025	39.68	0.028	0.035	55.55
16	0.088	0.075	0.013	14.77	0.078	0.010	11.36	0.034	0.054	61.36
Ave.	0.071	0.062	0.009	12.78	0.054	0.017	24.73	0.034	0.035	48.36

Table 6. RESULTS OF TEST 5.

Board No.	Swelling of untreated wafers.	Method 1			Method 2			Method 3		
		Swelling of wafers treated with Solution 5.	Difference in swelling.	Effectiveness of Solution 5.	Swelling of wafers treated with Linseed Oil.	Difference in swelling.	Effectiveness of Linseed Oil.	Swelling of wafers treated with Wax-Mineral Spirit Solution.	Difference in swelling.	Effectiveness of Wax-Mineral Spirit Solution.
1	2	3	4	5	6	7	8	9	10	11
	A	B	A-B	$\frac{A-B}{A} \times 100$	C	A-C	$\frac{A-C}{A} \times 100$	D	A-D	$\frac{A-D}{A} \times 100$
	inches	inches	inches	percent	inches	inches	percent	inches	inches	percent
1	0.062	0.024	0.038	61.29	0.048	0.014	22.58	0.033	0.029	46.77
6	0.068	0.027	0.041	60.29	0.056	0.012	17.64	0.038	0.030	44.12
8	0.068	0.021	0.047	69.11	0.042	0.026	38.23	0.029	0.039	57.35
9	0.068	0.024	0.044	64.70	0.057	0.011	16.17	0.034	0.034	50.00
10	0.086	0.025	0.062	72.09	0.059	0.027	31.39	0.035	0.051	59.30
11	0.067	0.023	0.044	65.67	0.047	0.020	29.85	0.026	0.041	61.19
12	0.077	0.024	0.053	68.83	0.072	0.005	6.49	0.044	0.033	42.86
14	0.055	0.019	0.036	65.45	0.034	0.021	38.18	0.024	0.031	56.36
15	0.062	0.021	0.041	66.12	0.044	0.018	29.03	0.027	0.035	56.45
16	0.086	0.025	0.061	70.93	0.066	0.020	23.25	0.037	0.049	56.97
Ave.	0.070	0.023	0.047	66.45	0.053	0.017	25.28	0.033	0.037	53.14

deviation of approximately ±10%. The low effectiveness would preclude this method from any consideration as a standard solution.

The wax-mineral spirits solution has an effectiveness of about 50%, with a deviation of ±8%. This variability is quite large but the method could be used as a standard against which to compare water repellent solutions if enough specimens were used. It would indicate that a solution is higher or lower than 50% effective.

There is, however, a further drawback to the use of a wax-mineral spirits solution as a standard. Patronsky has shown that an extremely small variation in wax concentration results in a large change of effectiveness (2).

Since only one variable solution, the test solution, is used in Method 1 instead of also introducing a second variable standard solution, Method 1 is apparently the best method of the three for evaluating the repellent qualities of water repellent solutions. However, Patronsky found that replicate tests with any given solution on a large number of boards by this method gives a solution effectiveness variation of approximately 10%.(2)

Variability of Test Material.

As has been pointed out there is a great variation in results between boards. A close analysis of the Tables 2 through 6, pages 7 to 12, reveals that some test boards were consistently effective or ineffective. An attempt was made

to classify these boards according to their effectiveness and to correlate them with their various physical characteristics. The correlation was made as follows:

1. For each test 10 boards were used. They were ranked from 1 to 10 according to their effectiveness for each method in all tests. The board with the lowest effectiveness graded 1, the highest 10, and the ones in between were graded according to their relative positions. These ranks ~~were~~ ARE given in Table 7, page 14, columns 2 through 16.

2. The ranks of each board averaged for each method of all tests are found in Table 7, columns 17, 18 and 19. Column 17 is the average rank of all boards treated with boiled linseed oil, and is also the average of columns 3, 6, 9, 12, and 15. Column 18 is the average rank of all boards treated with the wax-mineral spirits solution and is likewise the average of columns 4, 7, 10, 13, and 16. Column 19, the average rank of each board in all solutions, is the average of all columns 2 through 16.

3. The classification of each board is then obtained by rerating the averages of columns 17, 18 and 19 of Table 7 from 1 to 11 based on lowest to highest effectiveness. This classification is found in Table 8, page 15, columns 6, 7, and 8, where the effectiveness of each board is compared to the physical characteristics.

Table 7. CLASSIFICATION OF BOARDS IN ORDER OF PERCENT EFFECTIVENESS

(Based on a lowest effectiveness of 1, and a highest effectiveness of 10.)

Board No.	Rank															Average Rank		
	Test 1			Test 2			Test 3			Test 4			Test 5					
	Solution 1.	Linseed Oil.	Wax-Mineral Spirits.	Solution 2.	Linseed Oil.	Wax-Mineral Spirits.	Solution 3.	Linseed Oil.	Wax-Mineral Spirits.	Solution 4.	Linseed Oil.	Wax-Mineral Spirits.	Solution 5.	Linseed Oil.	Wax-Mineral Spirits.	Linseed Oil.	Wax-Mineral Spirits.	All Solutions.
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
1										9	5	5	2	4	3	4.5	4.0	4.7
6	4	4	2	3	5	2	1	5	2	1	4	4	1	3	2	4.2	2.4	2.9
8	8	10	7	10	10	5	9	10	7	8	8	1	8	10	8	9.6	5.6	7.9
9	1	3	5	9	4	4	8	3	5	5	2	2	3	2	4	2.8	5.0	4.0
10	9	5	9	8	6	7	6	6	8	2	6	8	10	8	9	6.2	8.2	7.1
11	7	7	10	7	7	10	4	8	10	10	7	9	5	7	10	7.2	9.8	7.9
12	3	2	1	2	1	1	2	2	1	4	1	3	7	1	1	1.4	1.4	2.1
13	5	1	3	4	2	3	7	1	6							1.3	4.0	3.6
14	2	8	6	1	8	6	3	4	3	3	9	6	4	9	5	7.6	5.2	5.1
15	6	9	4	6	9	8	5	9	4	6	10	7	6	6	6	8.6	5.8	6.7
16	10	6	8	5	3	9	10	7	9	7	3	10	9	5	7	4.8	8.6	7.2

Table 8. COMPARISON BETWEEN THE PHYSICAL CHARACTERISTICS OF THE PONDEROSA PINE SAPWOOD BOARDS USED FOR THE WATER REPELLENT TESTS AND THEIR CLASSIFICATION BASED ON PERCENT EFFECTIVENESS.

Classification is determined by averaging the ranks established for each board in its relation to all the boards in ability to repel water for each treatment and rerating these averages from 1 to 11 -- lowest to highest. (See Table 7.)

Board No.	Physical Characteristics				Classification		
	Specific Gravity. wgr. + vol. o.d.	Rings per inch.	Width of rings.	Deviation from flat grain in degrees.	Linseed Oil.	Wax-Mineral Spirits Solution	All Solutions.
1	2	3	4	5	6	7	8
1	-	14	Uniform	0	5	4	5
6	0.39	20	Irregular	5	4	2	2
8	0.36	14	Very irreg.	4	11	7	10
9	0.38	24	Irregular	0	3	5	4
10	0.36	18	Irregular	7	7	9	8
11	0.38	18	Uniform	8	8	11	11
12	0.35	28	Uniform	8	2	1	1
13	0.38	46	Uniform	12	1	3	3
14	0.42	21	Irregular	0	9	6	6
15	0.37	18	Very irreg.	0	10	8	7
16	0.40	26	Uniform	0	6	10	9

4. To facilitate comparison, graphs are presented in Figures 2, 3, and 4, pages 17, 18, and 19, in which specific gravity and number of growth rings per inch have been plotted against effectiveness.

As can be discerned from Figures 2, 3, and 4, there is no correlation between specific gravity and effectiveness. There is also little correlation between number of growth rings per inch and effectiveness. However, Figure 4 does indicate the tendency that fast-growing material has a higher effectiveness. There is not enough evidence at hand to indicate more than this possible relation. In fact, other investigators (1, 2) have been unable to correlate any physical property with effectiveness.

As previously mentioned the variation in results is largely between boards. Table 9, page 20, shows the effectiveness of the wax-mineral spirits solution for replicate tests on different boards. The standard deviation based on five tests on nine different boards was approximately $\pm 2\%$, exceeding $\pm 4\%$ in only one board. Similarly, Patronsky found that results are reproducible on the same wood material within an approximate 5% range.(2)

CONCLUSION

The work reported by this paper shows that there is an extreme variability in the effectiveness of a water repellent solution between different boards. However, on the same

COMPARISON BETWEEN SPECIFIC GRAVITY AND NUMBER OF GROWTH RINGS PER INCH, AND THE CLASSIFICATION OF EFFECTIVENESS FOR ALL REPELLENT SOLUTIONS OF THE TREATED PONDEROSA PINE SAPWOOD BOARDS.

CLASSIFICATION OF EFFECTIVENESS IS BASED ON AVERAGING THE RANKS ESTABLISHED FOR EACH BOARD IN ITS RELATION TO ALL THE BOARDS IN ABILITY TO REPEL WATER FOR EACH TREATMENT AND RERATING THESE AVERAGES FROM 1 TO 11. (SEE TABLES 7 AND 8.)

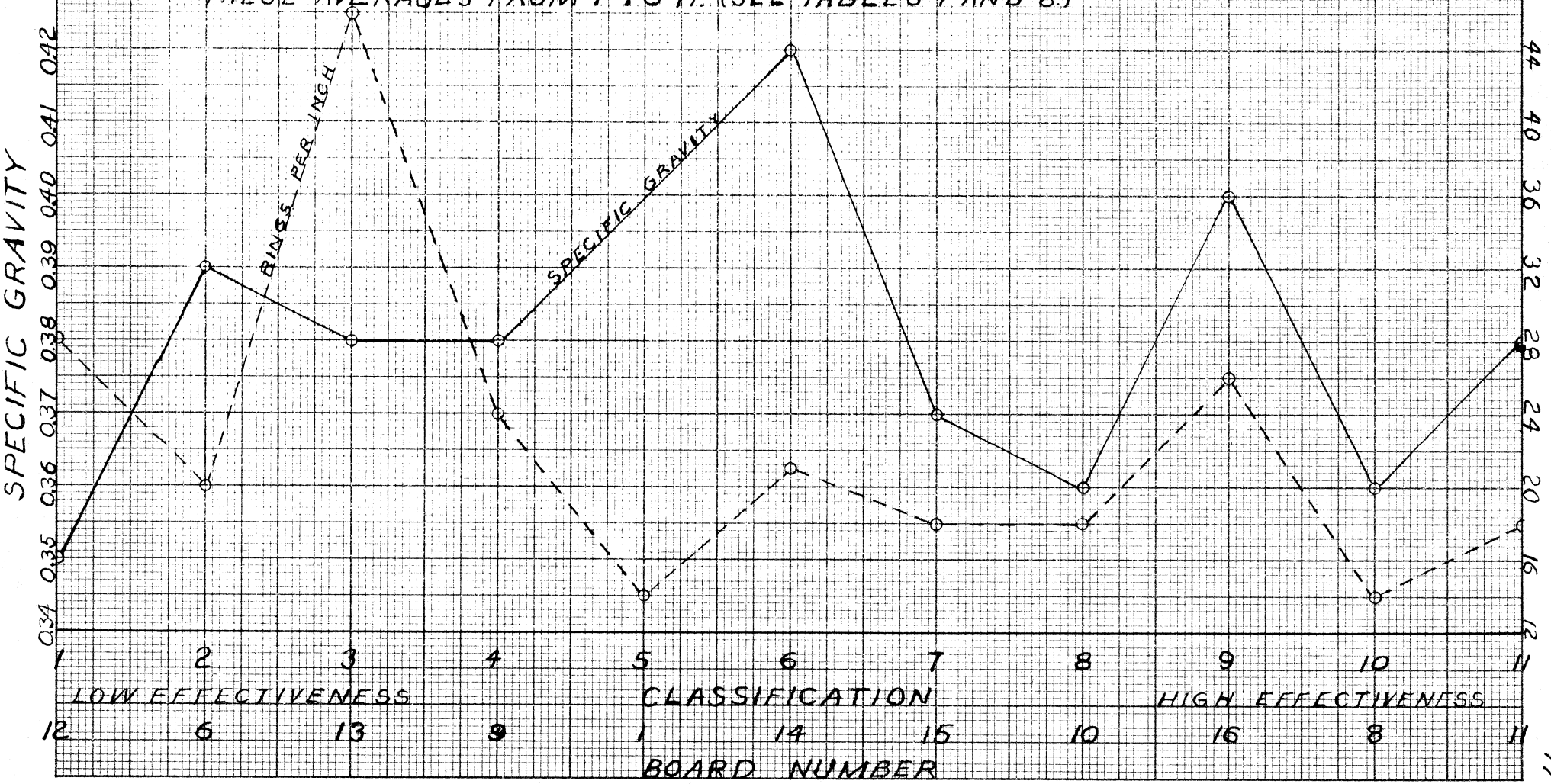

FIGURE 2

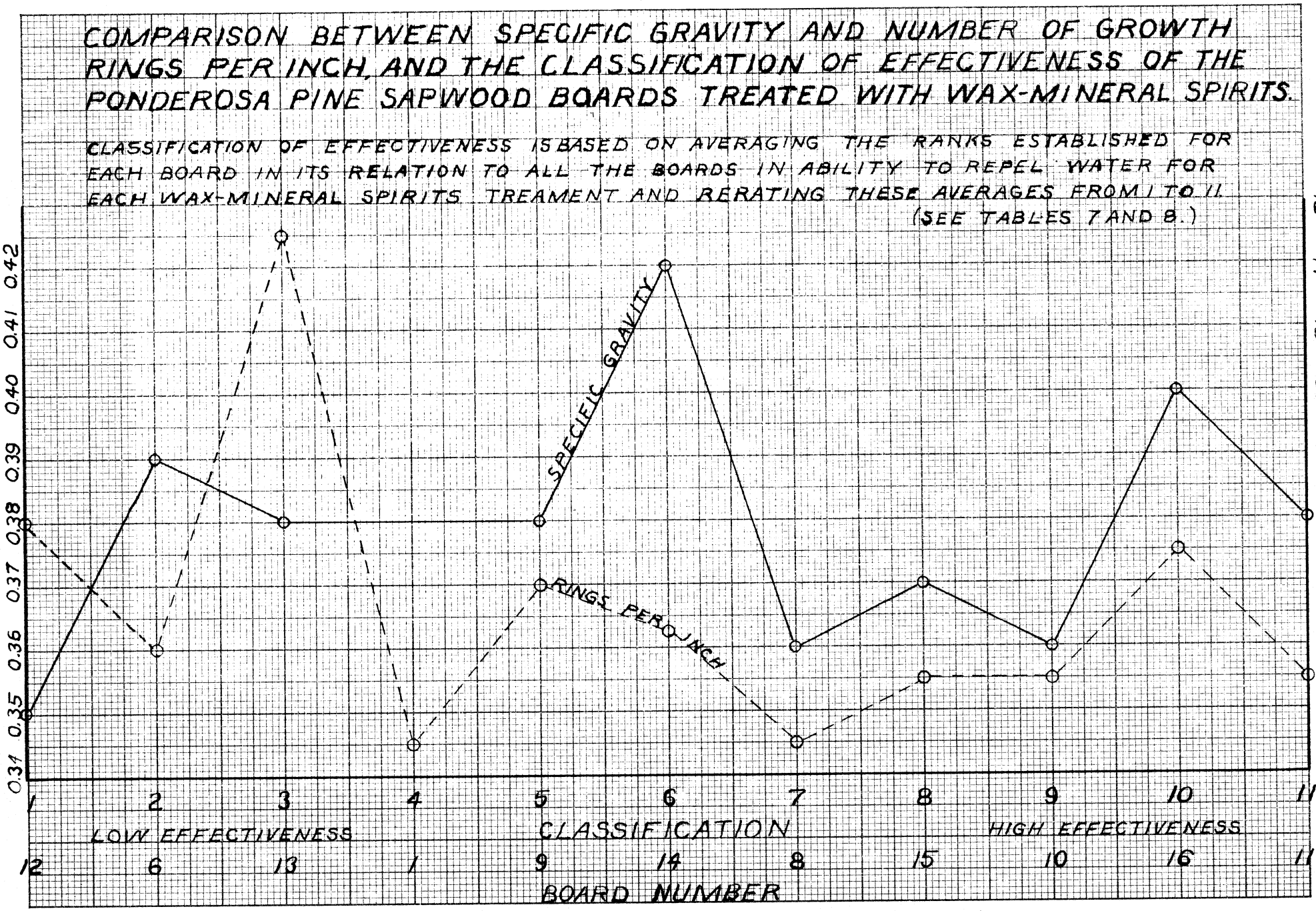

FIGURE 3

COMPARISON BETWEEN SPECIFIC GRAVITY AND NUMBER OF GROWTH RINGS PER INCH, AND THE CLASSIFICATION OF EFFECTIVENESS OF THE PONDEROSA PINE SAPWOOD BOARDS TREATED WITH LINSEED OIL.

CLASSIFICATION IS BASED ON AVERAGING THE RANKS ESTABLISHED FOR EACH BOARD IN ITS RELATION TO ALL THE BOARDS IN ABILITY TO REPEL WATER FOR EACH LINSEED OIL TREATMENT AND RERATING THE AVERAGES FROM 1 TO N. (SEE TABLES 7 AND 8.)

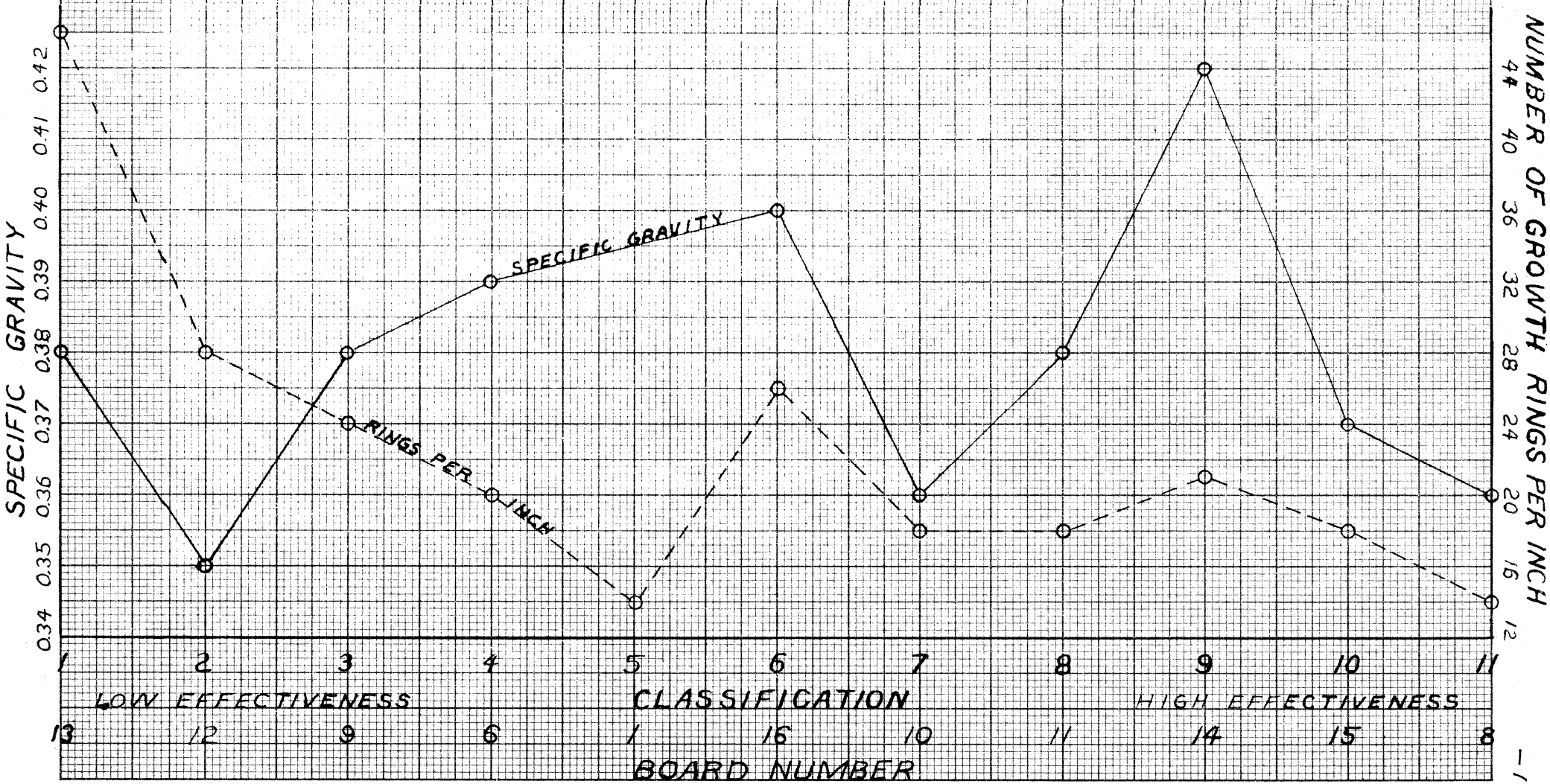

FIGURE 4

Table 9. VARIABILITY OF REPLICATE TESTS OF WATER REPELLENCY ON THE SAME WOOD MATERIAL WITH A 0.5 PERCENT WAX-MINERAL SPIRITS SOLUTION.

Board No.	Percent effectiveness of wax-mineral spirits solution.					
	Test 1	Test 2	Test 3	Test 4	Test 5	Average*
6	38.80	37.50	48.57	39.13	44.12	41.62 ±4.1
8	58.21	53.73	57.35	52.17	57.35	55.76 ±2.3
9	56.06	52.31	56.72	52.17	50.00	53.45 ±2.5
10	61.63	55.81	58.43	56.97	59.30	58.43 ±2.0
11	62.66	63.01	60.00	60.00	61.19	61.37 ±1.6
12	35.53	36.84	42.31	38.96	42.86	39.30 ±2.9
14	56.60	54.90	53.85	51.78	56.36	54.70 ±1.8
15	55.74	57.14	55.38	55.55	56.45	56.05 ±0.6
16	58.89	58.43	58.69	61.36	56.97	58.86 ±1.4

*Includes standard deviation.

board, the effectiveness of a given water repellent does not vary greatly.

The variability between boards is not easily correlated with any readily recognizable physical characteristic such as specific gravity, ring count, or regularity of growth.

Method 2, in which boiled linseed oil is used as a standard for comparison, is unsatisfactory due to its low effectiveness.

Method 3, which utilized a 0.5% by weight wax-mineral spirits solution as a standard, is not very satisfactory on two counts: (1) results among different boards are quite variable, and (2) the effectiveness of the solution is extremely sensitive to a slight change in concentration.

Method 1, the Protection Products 7-day Method, which compares the swelling of an untreated specimen to a treated specimen from the same board, is probably the best method of the three. However, the extreme variability inherent between different boards indicates that the method will not be entirely satisfactory.

RECOMMENDATIONS

It is recommended that a further method of evaluating the effectiveness of water repellent solution be investigated. This method would entail selection of ponderosa pine sapwood material that ranged in effectiveness between 45% and 55% (or other suitable range to be also determined)

when treated with a carefully prepared 0.5% by weight wax-mineral spirits solution. This selected material would then be used for testing water repellent solutions by either Method 1 or 3. By this careful selection of testing material, it is hoped that extreme variability between boards can be eliminated.

REFERENCES

1. Browne, F. L., and A. C. Schwebs. 1944. A study of methods of measuring the water repellency of water repellents and water-repellent preservatives for wood. Forest Products Laboratory Mimeograph No. R1453. 37 pp.

2. Patronsky, L. A. 1945. Study of a method for the determination of effectiveness of water repellent solutions and the development of a paraffin wax-mineral spirit control solution. (Unpublished report for the National Door Manufacturers Association, Inc.) 35 pp.

APPENDIX A

EXCERPT FROM THE MINUTES OF THE MEETING OF THE PRESERVATIVE STANDARDS ADVISORY COMMITTEE OF THE NATIONAL DOOR MANUFACTURERS ASSOCIATION

held at

The Forest Products Laboratory, Madison, Wis.
Sept. 11-12, 1947

13. The chairman appointed a committee consisting of Dr. Browne, Dr. Stout, Mr. Garlick and Mr. Lance to prepare a recommendation for conducting laboratory tests of water-repellent solutions as recommended at the meeting of April 4-5, 1946, and report as soon as possible. A recess was taken to enable the committee to prepare its report.

Following the recess, Secretary Lance read the report of the above committee as follows:

"The committee appointed at this meeting to recommend laboratory test methods for water-repellency, hereby recommends for the series of tests by the cooperating laboratories as previously authorized, the three following methods, among which the final decision is to be made.

"Method No. 1 is the present tentative method involving pairs of specimens from ten (10) different boards and measurements by either the swellograph or the Protection Products 7-day method." (Details of these methods will be furnished by NDMA.)

"Methods 2 and 3 - In Method 2 the standard solution consisting of undiluted raw linseed oil and in Method 3 a standard solution consisting of .5% (one-half per cent) by weight of paraffin wax, melting point 128°F, in mineral spirits, is used as the minimum of water-repellency against which a water repellent is compared. From each of the ten (10) boards prescribed for Method 1, four adjacent test specimens shall be taken, of which the first two shall be used for Method 1, the third for Method 2, and the fourth for Method 3. The third and fourth specimens shall be dipped in their standard solutions for ten (10) seconds in the same manner and at approximately the same time as the first specimen is treated with the water-repellent.

"In reporting results by Methods 2 and 3, it should be reported merely whether the average swelling of the specimens treated with the water-repellent is or is not less than that of the specimens treated with the standard solutions."

APPENDIX B

WATER REPELLENT TESTING METHOD
Developed by
Protection Products Manufacturing Company

Material

Use clear, kiln dried, straight grained ponderosa pine sapwood. The test blocks are to be 1/4" slices taken from a piece 1-5/8" x 1-3/8" in cross section, with the annual rings running practically straight and parallel to the 1-5/8" dimension, and the longitudinal grain parallel to the length of the piece.

Use ten blocks for each treatment to be tested, and ten for untreated controls. The blocks from different boards for the different groups must be selected in rotation as they are cut from the stock so that each group will be comparable with the other groups.

Conditioning

Condition the blocks at 80°F; 60% R.H. for one week before dipping.

Preparation

Dip the blocks for thirty seconds in the solution to be tested. Drain, dry, and condition the blocks for a week by keeping them in an atmosphere held at 60% relative humidity and 80° F. The untreated blocks must be kept under the same moisture conditions as the treated ones at all times.

Test Method

Measure each block to the nearest thousandth of an inch in the 1-5/8" direction. This can best be done by means of a dial gauge on a suitable mounting of glass or metal. Handling the blocks in a definite order, immerse each in water held at 80° F. immediately after taking its measurement. When a thirty minute interval has elapsed, go through the set of blocks in the same order, removing each from the water, measuring it and replacing it.

Results

Average the readings in each ten block group and determine the swelling of the wood by subtracting the initial reading from the final reading. The efficiency of the treatment is found by dividing the difference between swelling of the treated and untreated blocks by the swelling of the untreated blocks in the same time interval.

Equipment

The equipment necessary for the test is:

(1) A conditioning chamber maintained at 60% relative humidity. This may be a room, the atmosphere of which is regulated by a humidifier controlled by a humidistat, or it may be a small case containing a saturated solution of sodium bromide.

(2) A measuring instrument for the blocks. This should be a dial gauge graduated in thousandths of an inch, mounted on a horizontal plate of glass or metal, using an accurately machined metal bar at least 2" long at the bottom and at the edge of the test piece.

(3) A constant temperature water bath and a frame for holding the blocks under water. The design of the frame is not important but a simple holder can be made of a small floating board studded on the under side with finishing nails 3/8" apart to act as separators for the blocks which float under the board.

APPENDIX C

NEW METHOD FOR THE DIFFERENTIATION OF HEART AND SAP IN PINE WOOD

by

J. E. Koch and W. Krieg

(Chemeker Zeitung 62 (15): 140-141, Feb. 19, 1938)

1. Prepare a solution of 5 grams benzidine in 25 grams of HCl (about 25%) and 970 grams water.

2. Prepare a 10% solution of sodium nitrite.

3. Keep the two solutions separate.

4. Immediately before testing pour equal amounts of the solutions together. Either dip the wood in the solution or paint it on the surface. After treating the wood it is advisable to wash it with water or blot up the excess solution because the colors then hold better.

5. This treatment colors the heartwood dark red-brown, the sapwood yellow, after a time the sapwood takes on a dark yellow or brown color. Nevertheless, the difference between the heartwood and sapwood color remains distinct.

From translation made by
Dr. Elois Gerry.
Forest Products Lab.

6/7/39 NDMA

COMPARISON OF THREE COMMERCIAL OIL-SOLUBLE WOOD PRESERVING SOLUTIONS

May 13, 1948

Duane L. Kenaga

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Appendix

A. N.D.M.A. Method of Testing Oil-Soluble Wood Preservatives for Toxic Properties Using Wood Blocks Uniformly Impregnated i

B. Differentiation of Heart and Sap in Pine Wood..... iv

SUMMARY

The toxic properties of three oil-soluble commercial wood preservative solutions, containing respectively 5% pentachlorophenol, 20% copper naphthenate (2% copper metal), and 20% zinc naphthenate (2% zinc metal), were compared. The testing was conducted with the N.D.M.A. Method of Testing Oil-Soluble Wood Preservatives for Toxic Properties Using Wood Blocks Uniformly Impregnated. The test was conducted on two different boards of ponderosa pine sapwood material with pentachlorophenol and copper naphthenate, and on three other boards with zinc naphthenate, copper naphthenate, and pentachlorophenol.

Results tended to show that both the pentachlorophenol and copper naphthenate solutions were satisfactory, with no discernable difference between them. The zinc naphthenate solution was not considered satisfactory. Both of the naphthenate solutions supported the growth of contaminating molds.

Literature cited herein notes that copper naphthenate, although not satisfactory for millwork because of its characteristic green color, is being used in creosote-petroleum oil mixtures. Zinc naphthenate, although possessing comparatively low fungicidal properties, has certain other properties which might eventually insure its use as a preservative material.

The N.D.M.A. method was examined in the light of three criticisms advanced to condemn the test. Suggestions have

been made for further investigations which will aid in correcting the conditions creating the aforementioned criticisms.

INTRODUCTION

Wood preservatives of the oil-soluble type are becoming increasingly important in the wood preservative industry. Creosote has been and will continue to be the major wood preservative (13). However, the inherent nature of creosote makes it undesirable to use with such items as window sash, doors, and other millwork. On the other hand, most oil-soluble preservatives have properties that make them suitable for millwork utilization (2, 13).

The oil-soluble preservatives which have appeared on the market in recent years under various trade names contain many different toxic compounds (13). Pentachlorophenol has gained a reputation as one of the better compounds available (7, 8, 10, 11, 12). Metal naphthenates such as copper and zinc have by comparison only recently appeared on the market as preservatives, and little can be found in the preservative literature concerning their ability to prevent decay.

This paper is a report on the comparison of the toxic properties of three commercial preservatives of the oil-soluble type containing respectively pentachlorophenol, copper naphthenate, and zinc naphthenate as evaluated by a laboratory test.

TEST MATERIALS

The National Door Manufacturers' Association's Method of Testing Oil-Soluble Wood Preservatives for Toxic Properties Using Wood Blocks Uniformly Impregnated was used to

compare the toxic qualities of each of the three commercial preservatives. See Appendix A. Materials as specified by this method were used.

Wood Material.

Straight grain ponderosa pine sapwood, kiln dried, of average density, and free from defects, was selected. Table 1, page 5, gives the characteristics of the five boards chosen for this investigation. Selection of sapwood was based on the benzidene-sodium nitrite test for differentiation of heart from sap in pine wood. See Appendix B.

Test blocks were cut from surfaced boards 1 3/8" by 1 5/8" in cross section, the test blocks or wafers being cut 1/4" thick in such a manner that the longitudinal grain was parallel to the 1/4" dimension and the growth rings parallel to the 1 5/8" dimension.

Preservative Solutions.

A well-known commercial preservative solution containing 5% pentachlorophenol was used. Hereafter in this report the solution will be referred to as the pentachlorophenol solution.

The copper and zinc naphthenates were supplied as concentrates containing 80% by weight of metal naphthenate or 8% by weight of metal. For millwork, the company supplying the concentrates market solutions which contain 20% by weight of copper or zinc naphthenate. Therefore, solutions containing 20% by weight of copper and zinc naphthenate respectively (2% metal) were made by diluting the concentrate with half mineral spirits and half Stoddard's solvent. In this

Table 1. PHYSICAL CHARACTERISTICS OF PONDEROSA PINE SAPWOOD TEST BOARDS.

Test no.	Board no.	Rings per inch.	Specific gravity. Vol. & wgt. O.D.	Width of rings	Deviation from flat grain in degrees.
1	9	24	0.37	Irregular	0
2	13	46	0.38	Uniform	10
3	14	21	0.42	Irregular	0
4	11	18	0.38	Uniform	8
5	20	18	0.41	Irregular	5

report the solutions will be referred to as copper and zinc naphthenate solutions.

Wood-Rotter

The test fungus specified for inoculating the specimens was Lenzites trabea. The strain used in this investigation was secured from the Protection Products Manufacturing Company, Kalamazoo, Michigan. Originally the fungus had been obtained from the U. S. Forest Products Laboratory, Madison, Wisconsin.

TEST METHOD

Previous experience at the Wood Technology Laboratory of the University of Michigan indicated that results obtained by the N.D.M.A. method were inclined to vary considerably. It was therefore decided to repeat the tests on several different pieces of wood material. Tests 1 and 2, made on boards 9 and 13, compare the pentachlorophenol and copper naphthenate solutions; Tests 3, 4, and 5, made on boards 14, 11, and 20 respectively, compare the pentachlorophenol, copper naphthenate, and zinc naphthenate solutions.

The procedure for each test was as follows:

1. Fifty-six wafers, numbered in rotation as cut from each board, were used as shown in Table 2, page 8. For each test solution a set of fourteen blocks was required. Another set of fourteen blocks was needed for an untreated control. In each set, ten blocks were utilized as test specimens and four were used as reference specimens.

2. All blocks were conditioned in a relative humitity room at 15% E.M.C. for at least three weeks. This is the only major deviation made from the method as given in Appendix B. The saturated solution of sodium bromide did not work successfully, so it became necessary to use a relative humidity room being held at 15% E.M.C.

3. The test blocks were weighed to an accuracy of two decimal places.

4. The volume of the blocks was determined by measuring the length and width of every fifth wafer with an accurate scale and the thickness with a micrometer.

5. The conditioned blocks were treated with test solutions diluted 50% with Stoddards solvent. The impregnations were made by placing the fourteen specimens in a 2-liter, heavy-walled, filter flask, separating and weighting the wafers with glass marbles. A vacuum of 125 mm. of mercury was drawn on the flask for 30 minutes.

After the vacuum pump was shut off, enough solution to cover the blocks was introduced by means of a separatory funnel. The treatment was carried out at approximately 70° F.

After 15 minutes the vacuum was broken and blocks removed. The excess liquid was then blotted off and the blocks weighed immediately.

6. The untreated control specimens were individually wrapped in small squares of paper and steam sterilized for 10 minutes at 100° C.

7. The treated and control blocks were reconditioned in the relative humidity room for twenty days. Previous to starting the incubation period the treated blocks were reweighed.

8. Twenty-one days before commencing the incubation period, Lenzites trabea cultures were prepared in 100 by 20 mm. petri dishes. The nutrient agar for growth was made as follows:

Difco bacto agar	25 grams
Diastastic malt extract	25 grams
Distilled water	1000 grams

After the agar was dissolved, a suitable amount was poured in each dish. The dishes were then sterilized for 20 minutes at 15 p.s.i. Inoculations were made with fungus that was approximately fourteen days old.

Table 2. METHOD BY WHICH BLOCKS WERE SELECTED IN ROTATING ORDER FROM EACH BOARD FOR CONTROL OR TREATED SPECIMENS.

Numbers 1 to 56 represent block numbers as cut in succession from the same board.

Use of block.	Treatment			
	Pentachlorophenol	Copper Naphthenate	Untreated control	Zinc Naphthenate
Test	1	2	3	4
Test	5	6	7	8
Reference	9	10	11	12
Test	13	14	15	16
Test	17	18	19	20
Reference	21	22	23	24
Test	25	26	27	28
Test	29	30	31	32
Reference	33	34	35	36
Test	37	38	39	40
Test	41	42	43	44
Reference	45	46	47	48
Test	49	50	51	52
Test	53	54	55	56

9. After the prescribed times mentioned previously, ten blocks from each set of fourteen, which includes ten untreated control test specimens and ten treated test specimens from each solution, were placed on 1/8" diameter "V"-shaped glass rods over the prepared fungus mats. The remaining sixteen reference blocks were placed over uninoculated agar.

10. The specimens were incubated for sixty-three days at 80° F.

11. After the incubation period the blocks were removed from the fungus mats, scraped clean of mycelium oven-dried, and weighed.

12. The following calculations were made:

a. Absorption in terms of pounds of toxic compound per cubic foot of wood material.

b. Percent loss in weight due to decay determined as follows:

where,

W = weight of reconditioned treated test blocks, or of conditioned untreated control blocks.

G = weight of reconditioned treated reference blocks, or of conditioned untreated control reference blocks.

R = oven-dry weight of treated reference blocks at end of test, or of untreated control reference blocks at end of test.

F = oven-dry weight of treated test blocks or untreated control blocks after incubation.

then,

$WR/G = Z$, computed oven-dry weight of test blocks before incubation.

and,

$100(Z - F)/Z = E$, per cent loss in weight due to decay.

RESULTS

The results of Tests 1 through 5 conducted with boards 9, 13, 14, 11, and 20 respectively, are presented in Tables 4 through 8, pages 12 to 16. Table 3, page 11, summarizes the results of each of the tests and indicates the absorption of active toxicant in each treatment.

Loss in weight caused by decay of the untreated wafers varied about 15% from 31.95% in Test 1 to 46.22% in Test 4. The standard deviation of percent loss in weight calculated by the standard formula, $\sigma \sqrt{\frac{\Sigma x^2}{M}}$, varied from ±5.75% in Test 5 to ±8.78% in Test 2.

Attention is called to Figures 1 through 5, pages 17 to 19, which are photographs of typical fungus growths as found at the completion of the incubation period of each test. Figures 6 through 10, pages 20 to 22, are photographs of the blocks at the completion of the test.

The absorption of pentachlorophenol varied from 0.232 lbs. per cu. ft. to 0.30 lbs. per cu. ft. The loss of weight due to decay in all tests was under 1-1/2% except for Test 3 which was 2.86%.

The absorption of copper naphthenate expressed as pounds of copper metal per cubic foot of wood varied in five tests from 0.912 to 1.160. The loss of weight caused by decay was under 1-1/4% in each test except for Test 3 where the loss amounted to 2.24%.

Table 3. PERFORMANCE OF THREE OIL-SOLUBLE PRESERVATIVES IN REPEATED TESTS AS EVALUATED BY THE N.D.M.A. METHOD.

(Summary of results in Tables 4, 5, 6, 7, and 8.)

Test no.	Board no.	Treatment	Absorption of active in lbs. per cu. ft.*	Percent weight loss due to decay.**
1	9	Untreated		31.95 ± 7.63
		Pentachlorophenol	0.248	1.03
		Copper naphthenate	0.938	+0.09***
2	13	Untreated		36.15 ± 8.78
		Pentachlorophenol	0.289	+0.64***
		Copper naphthenate	1.160	0.17
3	14	Untreated		39.54 ± 7.81
		Pentachlorophenol	0.232	2.86
		Copper naphthenate	0.912	2.24
		Zinc naphthenate	0.894	+0.03***
4	11	Untreated		46.22 ± 6.61
		Pentachlorophenol	0.301	1.44
		Copper naphthenate	0.968	1.26
		Zinc naphthenate	0.948	3.00
5	20	Untreated		40.55 ± 5.75
		Pentachlorophenol	0.296	+0.28***
		Copper naphthenate	0.968	0.15
		Zinc naphthenate	0.930	1.28

* Active absorption given in lbs./cu.ft. of copper or zinc metal.

** Includes standard deviation of the untreated specimens.

*** + indicates gain in weight.

Table 4. RESULTS OF TEST 1 MADE ON BOARD 9.

	Block no.	Wgt. of test blocks before incubation	Wgt. of reference blocks before incubation	Wgt. of reference blocks after incubation (oven dried)	Correction factor for loss other than decay	Calculated O. D. wgt. of test blocks before incubation	O. D. wgt. of test blocks after incubation	Loss in wgt. of test blocks due to decay	Percent loss in wgt. of test blocks due to decay
		grams W	grams G	grams R	R/G	grams $Z=\frac{WR}{G}$	grams F	grams Z - F	percent $E=\frac{Z-F}{Z} \times 100$
	1	2	3	4	5	6	7	8	9
Untreated control Reference	11		3.76	3.32	0.883				
	23		3.88	3.44	0.880				
	35		3.86	3.40	0.880				
	47		3.70	3.28	0.886				
	Ave.				0.882				
Test	3	3.71				3.27	1.76	1.51	46.17
	7	3.81				3.36	2.55	0.81	24.10
	15	3.91				3.45	2.22	1.23	35.65
	19	4.03				3.55	2666	0.89	25.07
	27	3.86				3.40	2.24	1.16	34.11
	31	3.84				3.39	2.68	0.71	20.94
	39	3.79				3.34	1.93	1.41	42.21
	43	3.79				3.34	2.44	0.90	26.94
	51	3.84				3.39	2.32	1.07	31.56
	55	3.95				3.48	2.34	1.14	32.75
	Ave.								31.95 ±7.6**
Pentachlorophenol Reference	9		4.18	3.60	0.861				
	21		4.29	3.69	0.860				
	33		4.15	3.57	0.860				
	45		4.09	3.51	0.858				
	Ave.				0.860				
Test	1	4.21				3.62	3.59	0.03	00.83
	5	4.04				3.48	3.43	0.05	01.44
	13	4.24				3.65	3.61	0.04	01.09
	17	4.22				3.63	3.60	0.03	0.82
	25	4.14				3.56	3.51	0.05	1.40
	29	4.16				3.58	3.54	0.04	1.11
	37	4.15				3.57	3.55	0.02	0.56
	41	4.20				3.61	3.58	0.03	0.83
	49	4.20				3.61	3.56	0.05	1.38
	53	4.09				3.52	3.49	0.03	0.85
	Ave.								1.03
Copper Naphthenate Reference	10		3.92	3.39	0.864				
	22		4.07	3.50	0.859				
	34		4.04	3.48	0.861				
	46		4.03	3.46	0.858				
	Ave.				0.860				
Test	2	3.94				3.39	3.39	0.00	0.00
	6	3.97				3.41	3.42	+0.01*	+ 0.29*
	14	4.16				3.58	3.58	0.00	0.00
	18	4.04				3.48	3.49	+0.01*	+ 0.28*
	26	3.97				3.41	3.41	0.00	0.00
	30	4.02				3.46	3.46	0.00	0.00
	38	4.02				3.46	3.45	0.01	0.29
	42	4.04				3.48	3.48	0.00	0.00
	50	4.05				3.48	3.48	0.00	0.00
	54	4.05				3.48	3.48	0.00	0.00
	Ave.								+ 0.05*

* + indicates gain in weight.
** Standard deviation included.

Table 5. RESULTS OF TEST 2 MADE ON BOARD 13.

	Block no.	Wgt. of test blocks before incubation	Wgt. of reference blocks before incubation	Wgt. of reference blocks after incubation (oven dried)	Correction factor for loss other than decay	Calculated O. D. wgt. of test blocks before incubation	O. D. wgt. of test blocks after incubation	Loss in wgt. of test blocks due to decay	Percent loss in wgt. of test blocks due to decay
		grams W	grams G	grams R	R/G	grams $Z = \frac{WR}{G}$	grams F	grams Z - F	percent $E = \frac{Z-F}{Z} \times 100$
	1	2	3	4	5	6	7	8	9
Untreated control Reference	11		3.8[illegible]	3.44	0.888				
	23		3.82	3.38	0.884				
	35		3.83	3.39	0.885				
	47		3.88	3.41	0.878				
	Ave.				0.884				
Test	3	3.83				3.39	1.97	1.42	41.88
	7	3.86				3.41	2.13	1.28	37.53
	15	3.85				3.40	2.40	1.00	29.41
	19	3.85				3.40	1.90	1.50	44.11
	27	3.77				3.33	2.11	1.22	36.63
	31	3.80				3.36	1.96	1.40	41.66
	39	3.89				3.44	2.26	1.18	34.30
	43	3.94				3.48	2.63	0.85	24.42
	51	4.06	3.59			3.59	1.99	1.60	44.56
	55	3.77				3.33	2.43	0.90	27.02
	Ave.								36.15 ±8.87**
Pentachlorophenol Reference	9		4.25	3.65	0.859				
	21		4.09	3.50	0.855				
	33		4.13	3.53	0.854				
	45		4.33	3.70	0.854				
	Ave.				0.855				
Test	1	4.22				3.61	3.62	+0.01*	+00.27*
	5	4.20				3.59	3.61	+0.02*	+00.55*
	13	4.22				3.61	3.64	+0.03*	+00.83*
	17	4.13				3.53	3.52	0.01	00.29
	25	4.13				3.53	3.55	+0.02*	+00.56*
	29	4.17				3.57	3.59	+0.02*	+00.56*
	37	4.18				3.57	3.63	+0.06*	+01.68*
	41	4.21				3.60	3.64	+0.04*	+01.11*
	49	4.21				3.60	3.63	+0.03*	+00.83*
	53	4.19				3.58	3.59	+0.01*	+00.27*
	Ave.								+00.64*
Copper Naphthenate Reference	10		4.12	3.54	0.859				
	22		4.02	3.45	0.860				
	34		3.95	3.38	0.855				
	46		4.06	3.48	0.857				
	Ave.				0.858				
Test	2	4.01				3.44	3.45	+0.01*	+00.29*
	6	4.09				3.51	3.50	0.01	00.28
	14	4.08				3.50	3.51	+0.01*	+00.28*
	18	4.07				3.49	3.49	0.00	00.00
	26	3.97				3.41	3.42	+0.01*	+00.29
	30	3.93				3.37	3.36	0.01	00.30
	38	4.04				3.47	3.48	+0.01*	+00.28*
	42	4.11				3.53	3.50	0.03	00.85
	50	4.11				3.53	3.50	0.03	00.85
	54	4.13				3.55	3.53	0.02	00.56
	Ave.								00.17

* + indicates gain in weight.
** Standard deviation included.

Table 6. RESULTS OF TEST 3 MADE ON BOARD 14.

	Block no.	Wgt. of test blocks before incubation	Wgt. of reference blocks before incubation	Wgt. of reference blocks after incubation (oven dried)	Correction factor for loss other than decay	Calculated O. D. wgt. of test blocks before incubation	O. D. wgt. of test blocks after incubation	Loss in wgt. of test blocks due to decay	Percent loss in wgt. of test blocks due to decay
		grams W	grams G	grams R	R/G	grams $Z = \frac{WR}{G}$	grams F	grams Z - F	percent $E = \frac{Z-F}{Z} \times 100$
		1	2	3	4	5	6	7	8	9
Untreated control Reference	11		4.31	3.79	0.879					
	23		4.47	3.96	0.885					
	35		4.58	4.05	0.884					
	47		4.50	3.98	0.884					
	Ave.				0.8855					
Test	3	4.23				3.75	1.88	1.87	49.87	
	7	4.30				3.81	1.90	1.91	50.13	
	15	4.43				3.92	2.60	1.26	32.14	
	19	4.44				3.93	2.52	1.41	35.87	
	27	4.42				3.91	2.93	0.98	25.06	
	31	4.62				4.09	2.28	1.81	44.25	
	39	4.55				4.03	2.67	1.36	33.74	
	43	4.55				4.03	2.36	1.67	41.43	
	51	4.44				3.93	2.40	1.53	39.93	
	55	4.46				3.95	2.25	1.70	43.03	
	Ave.								39.54	±7.81**
Pentachlorophenol Reference	9		4.60	4.09	0.889					
	21		4.69	4.16	0.886					
	33		4.86	4.31	0.886					
	45		4.76	4.24	0.890					
	Ave.				0.888					
Test	1	4.24				3.77	3.63	0.14	3.71	
	5	4.39				3.90	3.78	0.12	3.17	
	13	4.62				4.10	3.97	0.13	3.17	
	17	4.62				4.10	3.99	0.11	2.68	
	25	4.66				4.14	4.07	0.07	1.69	
	29	4.80				4.26	4.12	0.14	3.28	
	37	4.83				4.29	4.16	0.13	3.03	
	41	4.77				4.24	4.08	0.16	3.69	
	49	4.73				4.20	4.13	0.07	1.59	
	53	4.68				4.16	4.05	0.11	2.64	
	Ave.								2.86	
Copper Naphthenate Reference	10		4.49	3.97	0.884					
	22		4.55	4.02	0.883					
	34		4.78	4.23	0.884					
	46		4.62	4.10	0.887					
	Ave.				0.8845					
Test	2	4.23				3.74	3.58"	----	----	
	6	4.48				3.96	3.91	0.05	1.26	
	14	4.51				3.99	3.75"	----	-----	
	18	4.51				3.99	3.89	0.10	2.51	
	26	4.61				4.08	3.97	0.11	2.69	
	30	4.66				4.12	4.00	0.12	2.91	
	38	4.69				4.15	3.87"	----	----	
	42	4.69				4.15	4.07	0.07	1.68	
	50	4.58				4.05	3.95	0.10	2.46	
	54	4.55				4.02	3.93	0.09	2.23	
	Ave.								2.24	
Zinc Naphthenate Reference	12		4.43	3.88	0.869					
	24		4.37	3.82	0.874					
Test	36		4.64	4.06	0,875					
	48		4.55	3.99	0.869					
	Ave.				0.872					
Test	4	4.28				3.73	3.70	0.03	0.80	
	8	4.40				3.84	3.89	+0.05*	+1.30*	
	16	4.45				3.88	3.96	+0.08*	+2.06*	
	20	4.51				3.39	3.93	0.00	0.00	
	28	4.56				3.98	3.98	0.00	0.00	
	32	4.68				4.08	4.01	0.07	1.17	
	40	4.60				4.01	4.05	+0.04*	+0.99*	
	44	4.59				4.00	3.98	0.02	0.50	
	52	4.55				3.97	3.91	0.06	1.51	
	56	4.52				3.94	3.96	+0.02*	+0.51*	
									+0.03*	

" Scorched in oven-drying.
* + indicates gain in weight.
** Standard deviation included.

Table 7. RESULTS OF TEST 4 MADE ON BOARD 11.

	Block no.	Wgt. of test blocks before incubation	Wgt. of reference blocks before incubation	O. D. wgt. of reference blocks after incubation	Correction factor for loss other than decay	Calculated O. D. wgt. of test blocks before incubation	O. D. wgt. of test blocks after incubation	Loss in wgt. of test blocks due to decay	Percent loss in wgt. of test blocks due to decay
		grams	grams	grams		grams	grams	grams	percent
		W	G	R	R/G	$Z=\frac{WR}{G}$	F	Z - F	$E=\frac{Z-F}{Z}$ x 100
		1	2	3	4	5	6	7	8	9
Untreated control Reference	11		3.73	3.29	0.882				
	23		3.99	3.50	0.877				
	35		3.68	3.25	0.883				
	47		3.78	3.33	0.881				
	Ave.				0.881				
Test	3	3.70				3.26	1.89	1.37	42.03
	7	3.61				3.18	2.07	1.11	34.91
	15	3.76				3.31	1.79	1.52	45.92
	19	3.76				3.31	1.49	1.82	54.98
	27	3.69				3.25	1.85	1.40	43.08
	31	3.74				3.29	1.77	1.52	46.20
	39	3.88				3.42	1.94	1.48	43.27
	43	3.75				3.30	1.76	1.54	46.67
	51	3.68				3.24	1.29	1.95	60.19
	55	3.63				3.20	1.76	1.44	45.00
	Ave.								46.22 ±6.62**
Pentachlorophenol Reference	9		4.04	3.53	0.874				
	21		4.20	3.66	0.870				
	33		4.08	3.56	0.873				
	45		4.06	3.56	0.877				
	Ave.				0.8735				
Test	1	4.09				3.57	3.52	0.05	1.40
	5	4.03				3.52	3.48	0.04	1.14
	13	4.06				3.55	3.51	0.04	1.13
	17	3.99				3.49	3.43	0.06	1.72
	25	4.06				3.55	3.49	0.06	1.69
	29	4.01				3.50	3.44	0.06	1.71
	37	4.06				3.55	3.49	0.06	1.69
	41	4.20				3.67	3.63	0.04	1.09
	49	4.08				3.56	3.51	0.05	1.40
	53	4.01				3.50	3.45	0.05	1.43
	Ave.								1.44
Copper Naphthenate Reference	10		3.86	3.39	0.878				
	22		4.07	3.58	0.880				
	34		3.90	3.42	0.877				
	46		3.86	3.41	0.883				
	Ave.				0.8795				
Test	2	3.83				3.37	3.33	0.04	1.19
	6	3.81				3.35	3.31	0.04	1.19
	14	3.86				3.39	3.35	0.04	1.18
	18	3.91				3.44	3.40	0.04	1.16
	26	3.80				3.34	3.31	0.03	0.90
	30	3.91				3.44	3.39	0.05	1.45
	38	3.98				3.50	3.44	0.06	1.71
	42	3.92				3.45	3.40	0.05	1.45
	50	3.89				3.42	3.37	0.05	1.46
	54	3.82				3.36	3.33	0.03	0.89
	Ave.								1.26
Zinc Naphthenate Reference	12		3.83	3.36	0.877				
	24		3.87	3.40	0.879				
	36		3.91	3.43	0.877				
	48		3.83	3.36	0.877				
	Ave.				0.8775				
Test	4	3.80				3.33	3.29	0.04	1.20
	8	3.77				3.31	3.24	0.07	2.12
	16	3.90				3.42	3.39	0.03	0.88
	20	3.93				3.45	3.35	0.10*	----
	28	3.83				3.36	3.34	0.02*	----
	32	3.78				3.32	3.30	0.02	0.60
	40	3.96				3.47	3.14	0.33	9.51
	44	3.87				3.40	3.13	0.27	7.94
	52	3.83				3.36	3.33	0.03	0.89
	56	3.83				3.36	3.33	0.03	0.89
	Ave.								3.00

* Contaminated by mold.
** Standard deviation included.

Table 8. RESULTS OF TEST 5 MADE ON BOARD 20.

	Block no.	Wgt. of test blocks before incubation	Wgt. of reference blocks before incubation	O. D. wgt. of reference blocks after incubation	Correction factor for loss other than decay	Calculated O. D. wgt. of test blocks before incubation	O. D. wgt. of test blocks after incubation	Loss in wgt. of test blocks due to decay	Percent loss in wgt. of test blocks due to decay
		grams W	grams G	grams R	R/G	grams $Z = \frac{WR}{G}$	grams F	grams Z - F	percent $E = \frac{Z-F}{Z} \times 100$
		1	2	3	4	5	6	7	8	9
Untreated control Reference	11			4.09	3.62	0.885				
	23			4.07	3.57	0.877				
	35			4.17	3.65	0.875				
	47			4.14	3.19"	---				
	Ave.					0.879				
Test	3	4.19					3.68	1.98	1.70	46.20
	7	3.98					3.50	2.17	1.33	38.00
	15	4.01					3.52	2.18	1.34	38.07
	19	4.13					3.63	2.65	0.98	27.00
	27	4.14					3.64	2.10	1.54	42.31
	31	4.14					3.64	2.13	1.51	41.48
	39	4.13					3.63	1.86	1.77	48.76
	43	4.07					3.58	1.96	1.62	45.25
	51	4.07					3.58	2.11	1.47	41.06
	55	4.20					3.69	2.31	1.38	37.39
	Ave.									40.55 ±5.75**
Pentachlorlphenol Reference	9			4.31	3.72	0.863				
	21			4.33	3.76	0.868				
	33			4.45	3.87	0.870				
	45			4.45	3.87	0.870				
	Ave.					0.868				
Test	1	4.23					3.67	3.67	0.00	0.00
	5	4.36					3.78	3.81	+0.03*	+0.79*
	13	4.38					3.80	3.81	+0.01*	+0.26*
	17	4.38					3.80	3.80	0.00	0.00
	25	4.40					3.82	3.85	+0.03*	+0.78
	29	4.47					3.88	3.89	+0.01*	+0.25
	37	4.39					3.81	3.79	0.02	0.52
	41	4.38					3.80	3.80	0.00	0.00
	49	4.45					3.86	3.88	+0.02*	+0.51*
	53	4.46					3.87	3.90	+0.03	+0.77*
	Ave.									+0.28*
Copper Naphthenate Reference	10			4.14	3.62	0.874				
	22			4.18	3.65	0.873				
	34			4.26	3.72	0.873				
	46			4.27	3.73	0.873				
	Ave.					0.873				
Test	2	4.07					3.55	3.58	+0.03*	+0.84*
	6	4.18					3.65	3.58	0.07	1.91
	14	4.23					3.69	3.69	0.00	0.00
	18	4.19					3.66	3.64	0.02	0.54
	26	4.23					3.69	3.69	0.00	0.00
	30	4.30					3.75	3.75	0.00	0.00
	38	4.19					3.66	3.66	0.00	0.00
	42	4.25					3.71	3.70"	----	----
	50	4.20					3.67	3.69	+0.02*	+0.54*
	54	4.28					3.74	3.73	0.01	0.26
	Ave.									0.15
Zinc Naphthenate Reference	12			4.18	3.66	0.875				
	24			4.17	3.67	0.880				
	36			4.26	3.74	0.877				
	48			4.27	3.75	0.878				
	Ave.					0.878				
Test	4	4.14					3.63	3.60	0.03	0.82
	8	4.12					3.62	3.58	0.04	1.10
	16	4.15					3.64	3.61	0.03	0.82
	20	4.18					3.67	3.62	0.05	1.36
	28	4.20					3.69	3.63	0.06	1.62
	32	4.23					3.71	3.69"	----	----
	40	4.19					3.68	3.62"	----	----
	44	4.25					3.73	3.67	0.06	1.60
	52	4.19					3.68	3.62	0.06	1.63
	56	4.32					3.79	3.75"	-----	----
	Ave.									1.28

" Contaminated by mold.
* + indicates gain in weight.
** Standard deviation included.

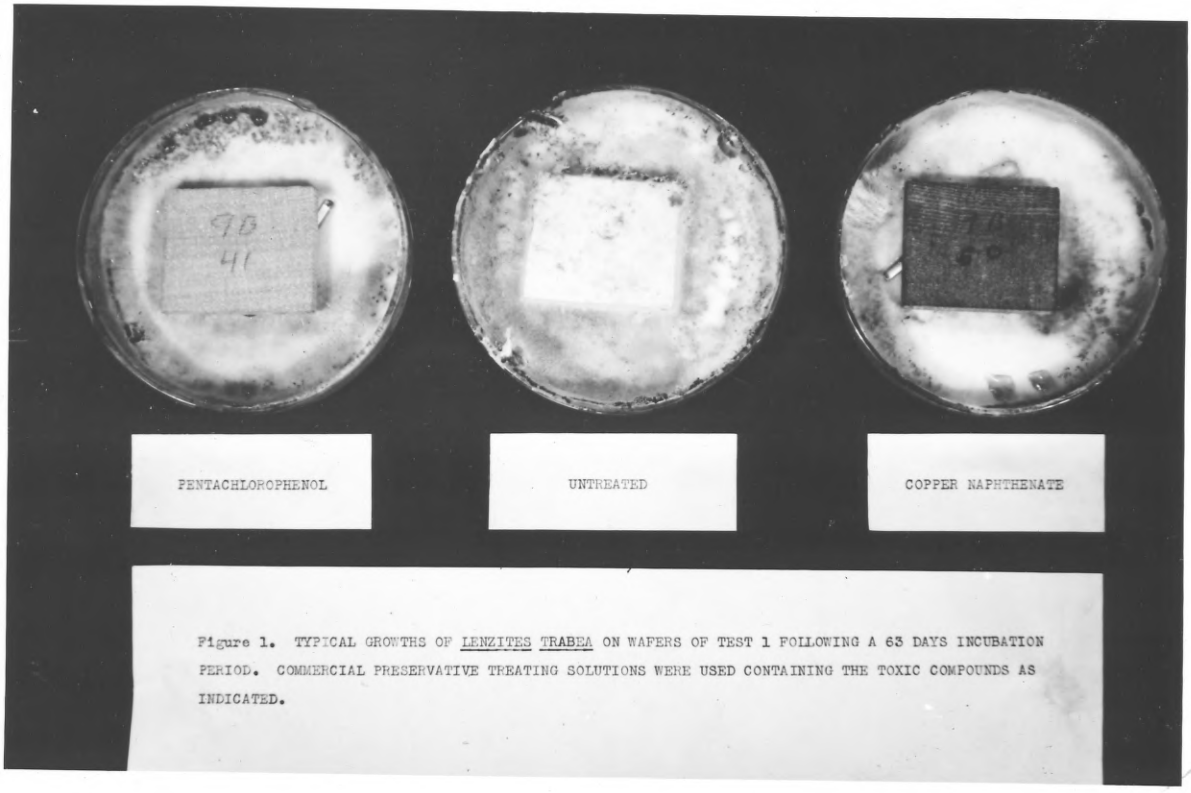


Figure 1. TYPICAL GROWTHS OF LENZITES TRABEA ON WAFERS OF TEST 1 FOLLOWING A 63 DAYS INCUBATION PERIOD. COMMERCIAL PRESERVATIVE TREATING SOLUTIONS WERE USED CONTAINING THE TOXIC COMPOUNDS AS INDICATED.

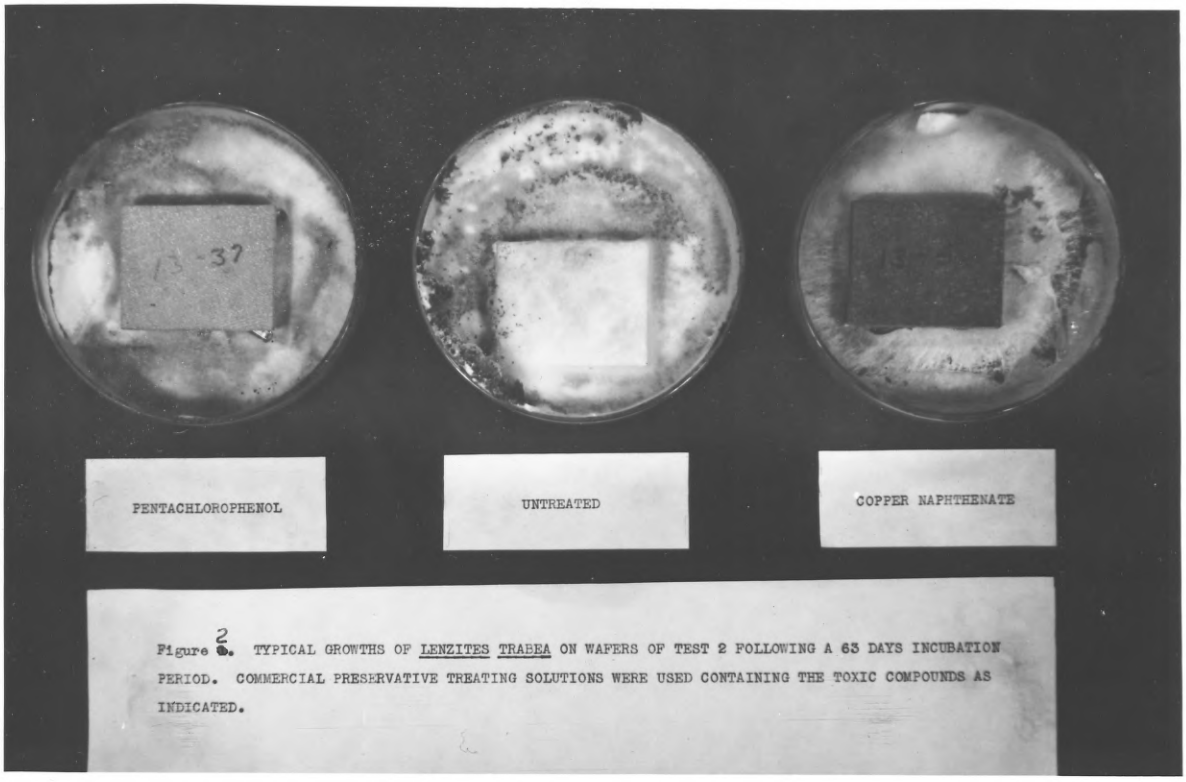


Figure 2. TYPICAL GROWTHS OF LENZITES TRABEA ON WAFERS OF TEST 2 FOLLOWING A 63 DAYS INCUBATION PERIOD. COMMERCIAL PRESERVATIVE TREATING SOLUTIONS WERE USED CONTAINING THE TOXIC COMPOUNDS AS INDICATED.

FIG. 2.

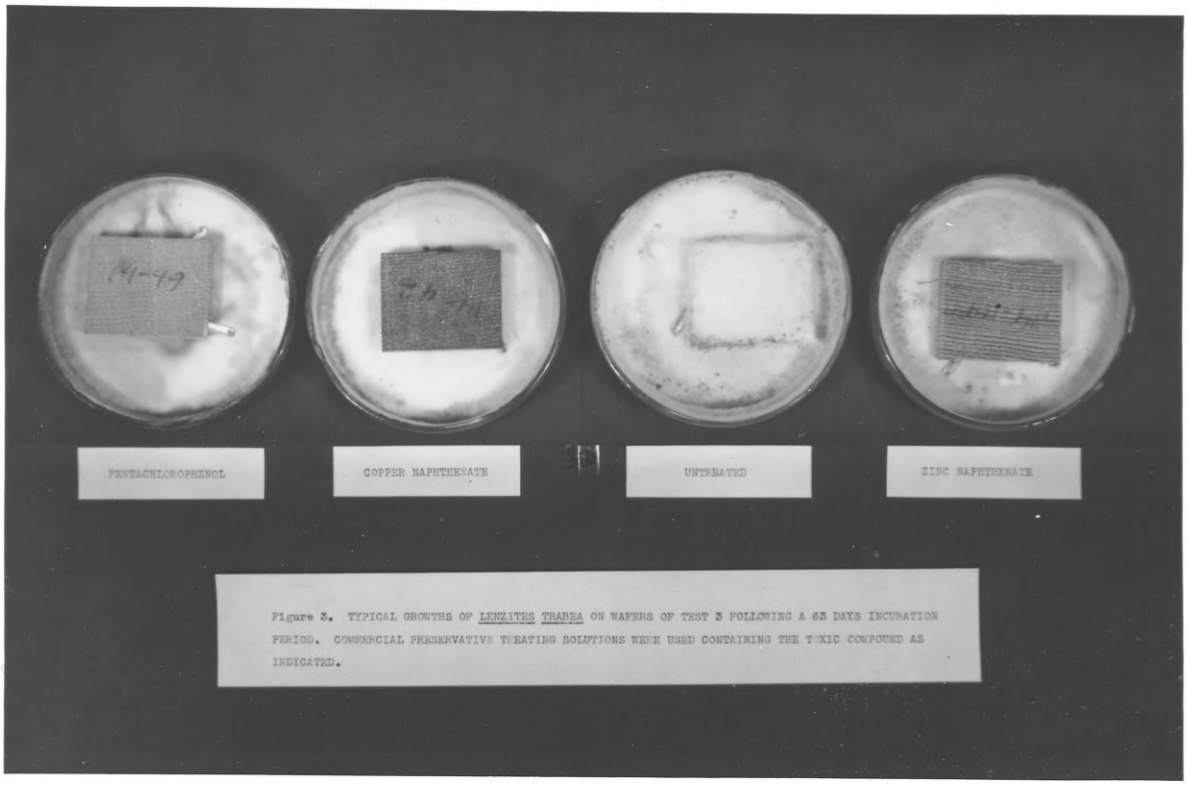
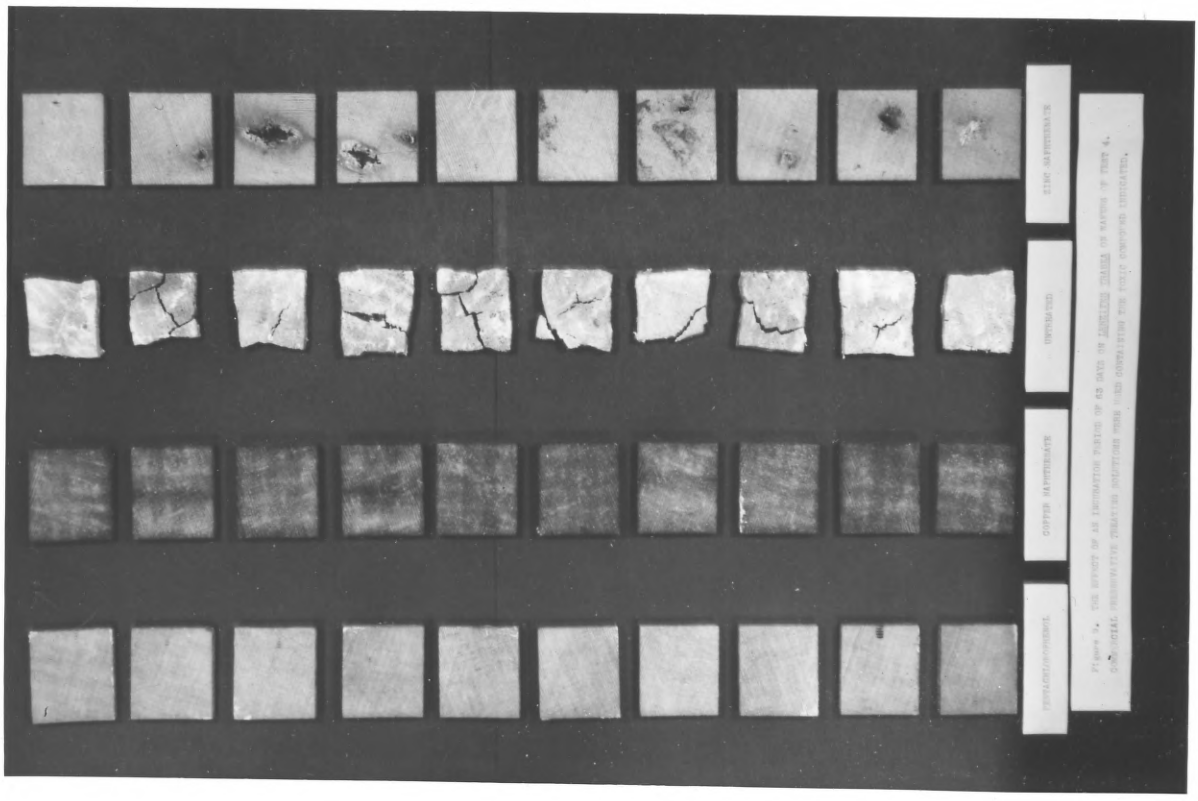


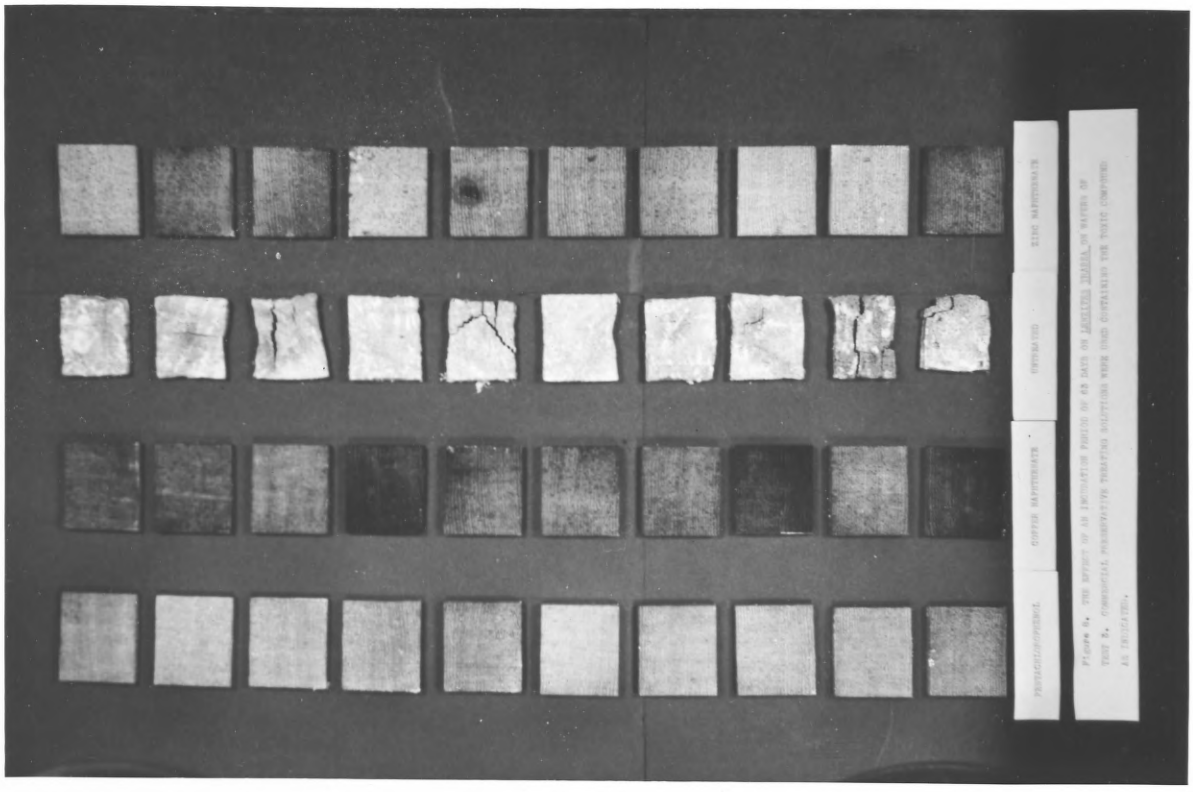
Figure 3. TYPICAL GROWTHS OF LENZITES TRABEA ON WAFERS OF TEST 3 FOLLOWING A 63 DAYS INCUBATION PERIOD. COMMERCIAL PRESERVATIVE TREATING SOLUTIONS WERE USED CONTAINING THE TOXIC COMPOUND AS INDICATED.

Figure 4. TYPICAL GROWTHS OF LENZITES TRABEA ON WAFERS OF TEST 5 FOLLOWING A 63 DAYS INCUBATION PERIOD. COMMERCIAL PRESERVATIVE TREATING SOLUTIONS WERE USED CONTAINING THE TOXIC COMPOUND AS INDICATED.

FIG. 4. TEST 5

Figure 5. TYPICAL GROWTHS OF LENZITES TRABEA ON WAFERS OF TEST 4 FOLLOWING A 63 DAYS INCUBATION PERIOD. COMMERCIAL PRESERVATIVE TREATING SOLUTIONS WERE USED CONTAINING THE TOXIC COMPOUND AS INDICATED.


ZINC HAYDENHAITE
COPPER HAYDENHAITE
UNTREATED
PENTACHLOROPHENOL


ZINC HAYDENHAITE
COPPER HAYDENHAITE
UNTREATED
PENTACHLOROPHENOL

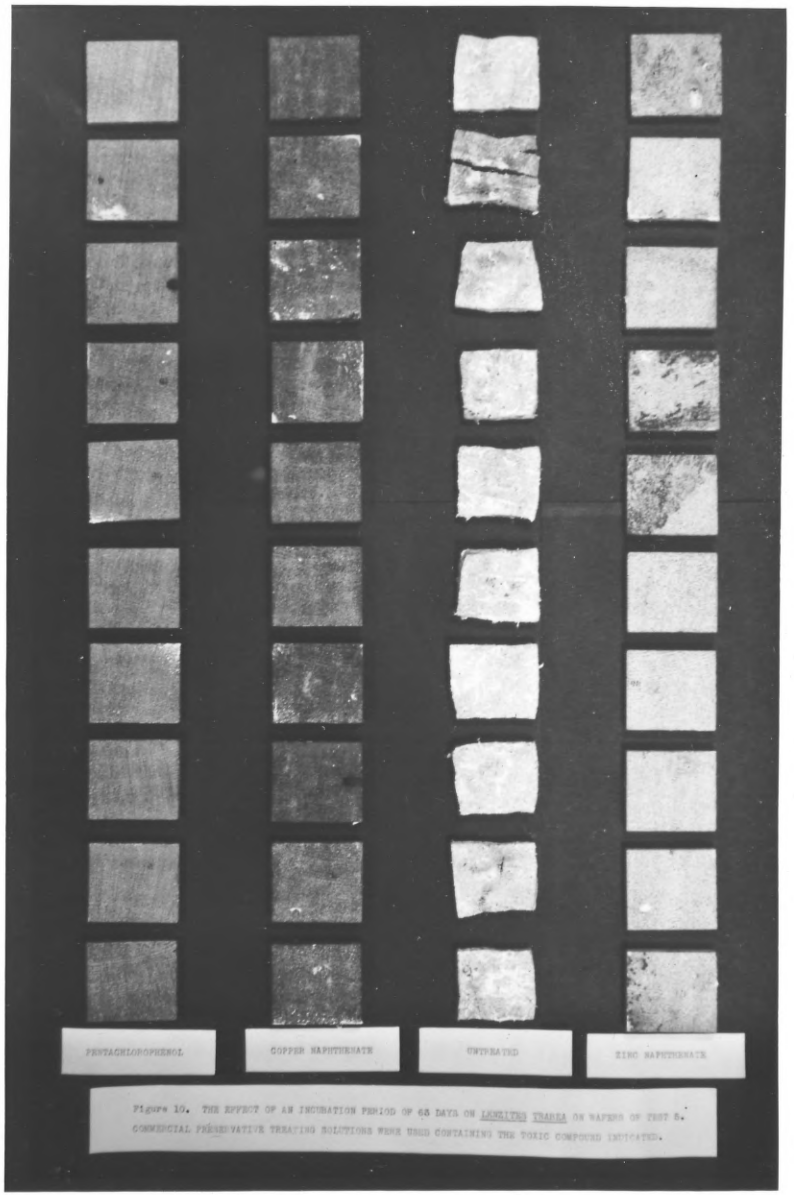


Figure 10. THE EFFECT OF AN INCUBATION PERIOD OF 65 DAYS ON LEACHING TRAYS ON WAFERS OF TEST 8. COMMERCIAL PRESERVATIVE TREATING SOLUTIONS WERE USED CONTAINING THE TOXIC COMPOUND INDICATED.

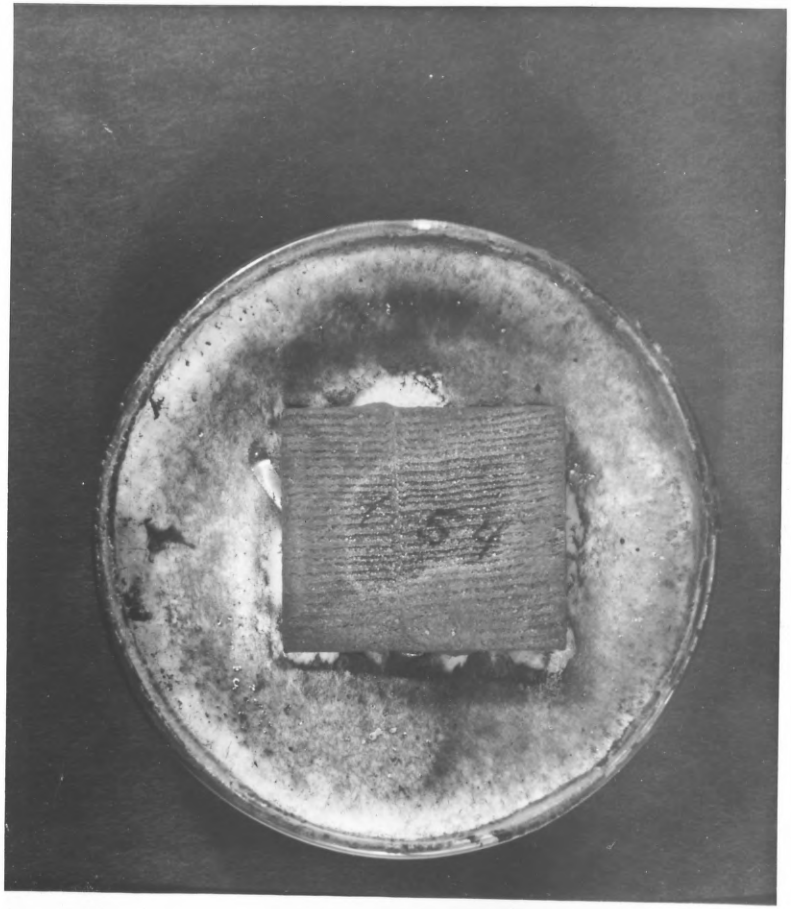


Figure 11. MOLD CONTAMINATION ON WAFER TREATED WITH COPPER NAPHTHENATE PRESERVATIVE SOLUTION. WAFER IS FROM AMONG THOSE OF TEST 3.

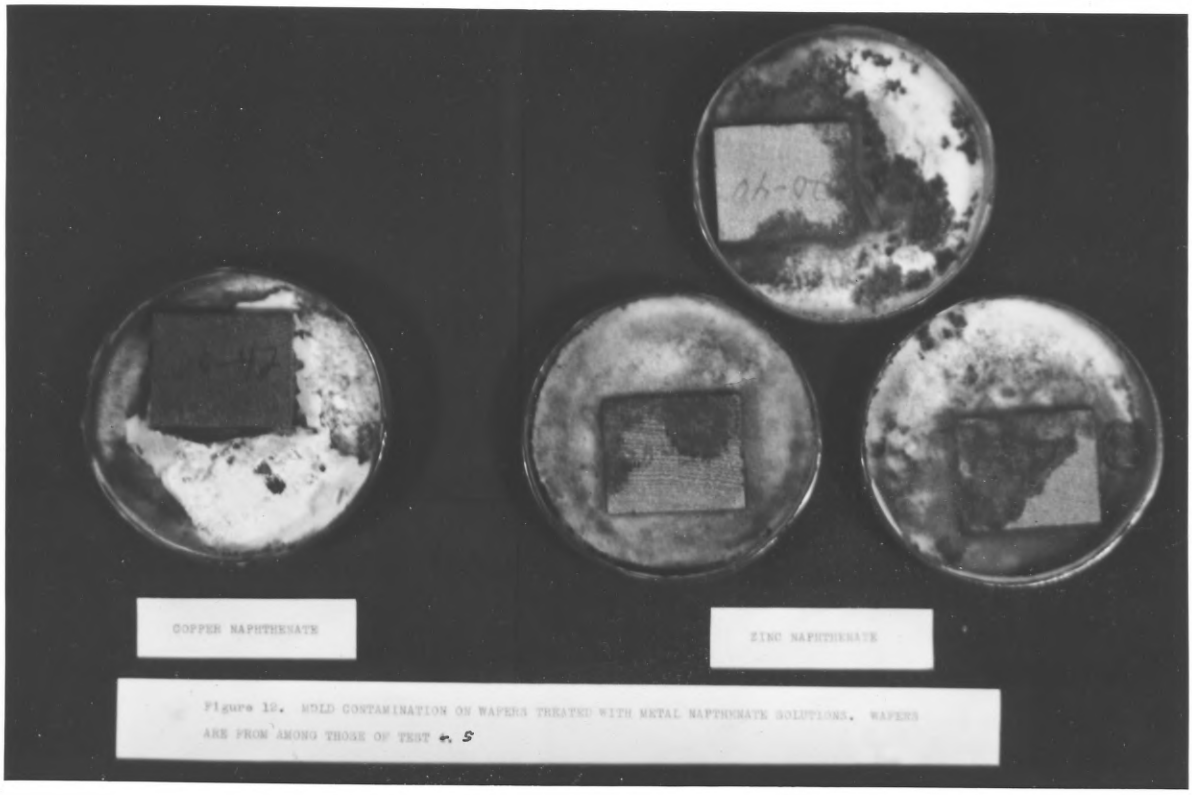


Figure 10. MOLD CONTAMINATION ON WAFERS TREATED WITH METAL NAPTHENATE SOLUTIONS. WAFERS ARE FROM AMONG THOSE OF TEST # 5

TEST 5.

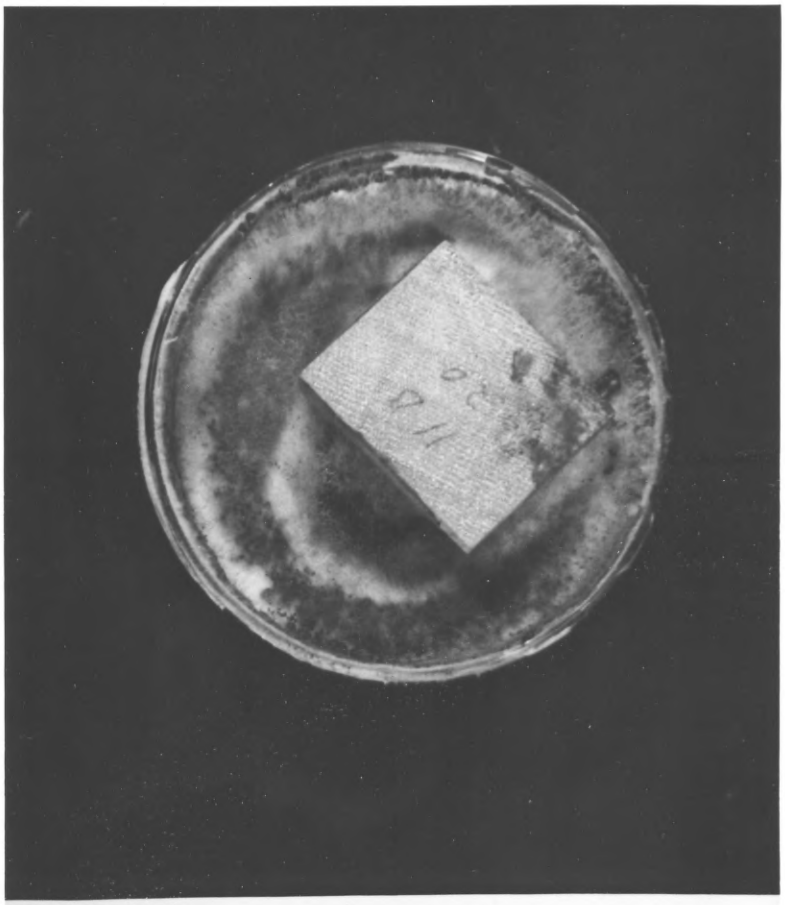


Figure 13. MOLD CONTAMINATION ON WAFER TREATED WITH ZINC NAPHTHENATE PRESERVATIVE SOLUTION. WAFER IS FROM AMONG THOSE OF TEST 4.

The absorption of zinc naphthenate in Test 3, 4, and 5 varied from 0.894 lbs. of zinc metal per cu. ft. of wood to 0.848 lbs. of zinc metal per cu. ft. of wood. The loss of weight varied from 0% in Test 3 to 3.00% in Test 4.

In Test 4 decay holes were rotted through the center of six of the blocks treated with zinc naphthenate. This is shown in the photograph of Figure 9, page 21.

Mold contamination was encountered on a few naphthenate treated blocks of Tests 3, 4, and 5. Figure 11, page 22, is a photograph of mold growing on one copper naphthenate treated block of Test 3. Figure 12, page 23, is a photograph of mold growing on one copper naphthenate treated block and three zinc naphthenate treated blocks of Test 5. Figure 13, page 24, is another photograph of mold growth on a zinc naphthenate treated block from Test 4.

DISCUSSION OF RESULTS

Toxic Qualities of Preservative Solutions.

Pentachlorophenol. As was mentioned in the introduction of this report, pentachlorophenol is one of the better oil-soluble preservatives. The literature concerning the utilization of this compound as a preservative is large and voluminous. Certain references have already been noted and little need be added, except to point out that its reputation is well-sustained by tests conducted here. The average maximum weight loss that occurred in Test 3 of 2.86% cannot be considered excessive. This amounts to a loss in weight of 0.12 grams in a wafer originally

weighing 4.12 grams oven-dried. Furthermore, in five tests there were no visual signs of decay on any of the pentachlorophenol treated blocks.

Copper Naphthenate. Copper naphthenate is a relatively new preservative. Commercial quantities were used for the first time in 1946 (5, 20). It is green in color, soluble in light oils, inexpensive, and resistant to leaching (5). At high concentrations it is quite viscous.

The results of the tests indicate that the 20% concentration (2% copper metal) is as good a wood preservative as a 5% pentachlorophenol solution, since there are no visable signs of decay on any blocks treated with either solution. However, Kirkpatrick states that laboratory tests indicate a relatively poor rating for copper naphthenate, although he does not specify the type of laboratory tests conducted or concentrations used (14).

Kirkpatrick also states that the metallic naphthenates seem to have an "affinity" for the cellulosic fibers (14). This "affinity" theory accounts for the discrepancies between laboratory and field tests conducted in the South which have shown copper naphthenate to have as great or greater efficiency than pentachlorophenol. As to the chemical combination that copper makes with the cellulosic fibers, Kirkpatrick is unable to state. However, he is apparently certain that the constitution will ultimately be recognized.

The Forest Products Laboratory has investigated copper naphthenate by field tests conducted at a test plot in

Mississippi. Stakes nominally 2" by 4" by 18" were treated with various concentrations of copper naphthenate by several methods (1). The average life of ten stakes brush-treated with a 2% copper metal solution was 3.7 years. When given a 3-minute dip in the same solution, five stakes were completely destroyed and five partially destroyed in 4-1/2 years. Only one of ten stakes pressure treated with a 0.11% copper metal solution showed signs of decay in 4-1/2 years. There was no decay or termite attack on stakes pressure treated with more potent toxic solutions of copper naphthenate over the same period.

Reports originating from Trinidad by Berry and Cater on the effect of copper naphthenate as a wood preservative were somewhat confused when it was discovered that the gas-oil carrier was also exhibiting preservative qualities (3, 4). Ten white pine specimens treated with a solution of 2-1/2% copper naphthenate in gas-oil by a hot-and-cold open tank process were perfectly sound at the completion of six years in a "graveyard". However, eight of ten stakes treated in the same manner with only the gas-oil carrier were unattacked in the same period, thus creating confusion as to the exact effectiveness of copper naphthenate.

Verrall found that over a 3-1/2 - 4-year exposure period, test units of southern yellow pine soaked for 30 minutes in a 4.5% copper naphthenate solution were slightly better preserved than test units similarly treated with a 5% pentachlorophenol solution (21). The test units were specifically designed to allow rain seepage at joints in

outdoor, above ground exposure. A two coat brush treatment with an 18% copper naphthenate solution also proved effective over a 3-1/2 - 4-year period.

Work reported by Harkom and Sedziak of progress made on laboratory and service tests of pentachlorophenol and copper naphthenate noted that under the conditions and solvents used, copper naphthenate was more resistant to removal in accelerated weather tests of heating and leaching cycles; and that after such cycles, blocks treated with copper naphthenate were more resistant to decay (9). These conclusions are based on extensive work which is still continuing. They used pressure treatments and two different solvents for each of two concentrations of each preservative. In the laboratory blocks were tested for decay resistance with three fungi by means of a burial test in soil.

Even though copper naphthenate must be considered as an effective preservative, its use in millwork is limited because of the inherent green color that it imparts to the wood. A formula utilizing a cobalt drier has been found successful to prevent bleeding, thus making it possible to paint wood treated with copper naphthenate (5). However, this is only a remedy applicable in certain situations, and does not eliminate the undesirable color characteristic. Other uses for copper naphthenate include fence posts and stained shingles (6, 22). Also pressure treatments of ties, piling, poles, and construction timber have been undertaken with solutions of creosote and petroleum containing copper naphthenate (1, 20).

Another disadvantage of copper naphthenate must also be recognized--its ineffectiveness in controlling certain molds. Contaminations of a few of the test specimens in this work indicate that some molds are not inhibited by copper naphthenate treated wood. See Figures 11 and 12, pages 22 and 23. Richards also noted this fact when the black mold, Aspergillus niger, was encountered making a heavy attack on blocks treated with copper naphthenate while conducting certain tests (18). This tolerance that is shown towards some molds might become a limiting influence on the use of copper naphthenate as a wood preservative.

Zinc Naphthenate. There seems to be little known about zinc naphthenate as a wood preservative. There is little literature available mentioning the possibilities of its use.

Morriss states that zinc naphthenate has the advantage of less odor, no irritation, and a quicker drying reaction than the general chlorinated phenol compounds (17). The fact that it is also colorless, insoluble in water, and soluble in oil, immediately suggests its use for millwork preservation. By the application of zinc naphthenate as a fungicide for fabrics, it has been demonstrated that this compound is only about one-half as potent as copper naphthenate (5).

The work done here stresses the fact that as a wood preservative zinc naphthenate is not as potent as copper naphthenate. In Test 4, holes developed in the mid-section of six blocks. See Figure 9, page 21. The fact that this occurred in only one of three tests indicates probable

border-line toxicity. With one wood material zinc naphthenate will be satisfactory at this concentration but will fail with another wood material.

At the time the Forest Products Laboratory conducted the investigation in the South with copper naphthenate, they also tested zinc naphthenate (1). Ten stakes dipped for 3 minutes in a 17% solution (2% zinc metal) had an average life of 2.2 years. Five of ten stakes pressure treated with a 1% solution (0.12% zinc metal) were untouched by decay or termites after 4-1/2 years. When pressure treated with a 7.5% solution (0.88% zinc metal) all stakes were sound after 4-1/2 years. The indication in this test was that a copper naphthenate solution containing 0.29% copper metal is equal in toxicity to a zinc naphthenate solution containing 0.88% zinc metal.

As was true with copper naphthenate, zinc naphthenate is also tolerant to certain molds. Figures 12 and 13, pages 23 and 24, are photographs of mold contaminations found existing on zinc naphthenate treated blocks. Unpublished work conducted in the Wood Utilization Laboratory of the University of Michigan also noted the same fact in a similar test with a lower concentration of zinc naphthenate.

The future of zinc naphthenate as a preservative must to a large extent depend upon its ability to compete with pentachlorophenol. If the amount of zinc naphthenate required for a concentration high enough to meet the toxic standards of pentachlorophenol can be purchased at a comparable price, it could conceivably become widely used for

dip treatments because of its quick-drying, non-irritative qualities.

There is also the possibility that zinc naphthenate can be combined with some of the chlorinated phenol compounds to reduce irritationmand improve permanency . Krause found that a solution containing 0.1% phenyl mercury oleate and 1% zinc naphthenate was very promising for control of both decay and blue stain when tested by the N.D.M.A. method (15).

Criticisms of the N.D.M.A. Test Method.

The N.D.M.A. test method for evaluating the toxic properties of oil-soluble preservatives has been criticized for various reasons which may or may not be justified. For the purpose of understanding the weaknesses inherent in the method, it becomes worthwhile to note these criticisms.

Moisture Content Improper. An improper moisture content has been advanced as a reason for lack of optimum decay conditions in the N.D.M.A. test method (15). If this is so, it could be expected that quite variable results will be obtained when conducting tests with this method. An examination of the results tabulated in Table 3, page 11, does show considerable variability among the untreated specimens. The loss in weight among five different boards when subjected to identical conditions varied in weight loss due to decay by approximately 15%. The tests conducted on one of five boards resulted in a maximum standard deviation among ten test blocks of ± 8.78%. The minimum standard deviation for the same number of test specimens from another board was ± 5.75%. This large variation in the amount

of decay between boards can probably be ascribed to two reasons: (1) the inherent differences in wood material of the same species, and (2) the differences in moisture content established in each test specimen after the test has gotten underway. This second reason accounts in even a larger measure for the variation between test blocks from the same board. It is, of course, generally impossible to control the nature of the wood material, but the moisture content is capable of control.

According to Snell, the optimum moisture content for decay in woods of low specific gravity is close to 150% (19). Since the N.D.M.A. test depends upon the fungus to derive the necessary moisture from the agar, it therefore stands to reason that decay will not proceed uniformly between blocks over a given period. The particular block on which the fungus obtains the first start will transport more moisture to the block, and decay will proceed at a more rapid pace; the metabolic water produced will in turn favor the decay process, thus resulting in the lack of uniformity of decay between test blocks.

The N.D.M.A. test recognizes the moisture problem. The test specifies that the block be supported above the agar on glass rods, preventing direct moisture transfer between the agar and the block. Krause points out that the method works fairly well for untreated blocks (15). However, he contends that it is another matter to expect a fungus to attack and supply the necessary moisture to a block heavily loaded with a preservative. Tests conducted by him with

the N.D.M.A. method showed that at the completion of the test the moisture content of the untreated blocks averaged 250%, whereas the treated blocks averaged 38 to 40% moisture. It should also be pointed out that since little decay took place, there was little metabolic water built up in the treated blocks. But the point he wishes to make is that if the treated blocks had initially ~~shown~~ the optimum moisture content, conditions would more nearly approach normal usage and the fungus would be aided, giving a better and more reliable test.

In an attempt to prove this idea, Krause conducted exploratory tests with blocks 1" thick by 2" by 2-1/2" on the tangential surface. After dip-treating for 3 minutes at a depth of 4", the blocks were placed ir jars on a bed of glass cloth. Sterile water was added to provide a high moisture content, and after a certain period, an inoculum block was placed on top of the test block. Unfortunately, at the time he presented his paper at the meeting of the Forest Products Research Society, the results of these tests were inconclusive.

Richards and Addoms have also noted the effect of a suitable moisture content for favorable decay conditions (18). While evaluating the soil-culture technique or Leutritz's method of evaluating preservatives (16), they found that soil containing 31% moisture caused more decay than soil containing 25% moisture, which in turn caused more decay than soil containing 18-1/2% moisture.

In the light of the work mentioned (15, 18), it would seem that an investigation should be conducted with blocks conditioned at higher calculated moisture contents. For instance, after conditioning the blocks for a given period at 12% E.M.C., the blocks could be weighed, then placed individually in containers in such a manner that they would come to a desired calculated moisture content. Then the blocks could be placed on the fungus mats as specified in the N.D.M.A. method.

Impregnation too Heavy. Another criticism of the N.D.M.A. test method slightly mentioned heretofore, is that the test specimens are too heavily loaded with the preservative. As a result of heavy loading, there is sufficient toxic vapors given off to accumulate and inhibit if not kill the test fungus.(15). In ordinary usage, the amount of toxic vapor given off by any of the preservatives mentioned in this report is insignificantly small. Furthermore, it is seldom that treated wood would ever be used where vapors accumulate. However, in closed flasks or petri dishes, heavily impregnated blocks will cause this unnatural and undesirable result.

The N.D.M.A. test method specifies impregnating by pressure to reduce variability of absorption encountered in dip treatments. Since there are so many other variables that are uncontrollable or not as easily controlled, it is undoubtedly best to obtain as uniform impregnation between

blocks as possible. The test specifies diluting the preservative solution 50% with an equal volume of Stoddards solvent. The objection to overloading could be met by diluting the preservative solution to 33-1/3% or 25%, continuing pressure impregnation to obtain uniform penetration.

One Test Fungus Inadequate. Perhaps the most justifiable criticism leveled at the N.D.M.A. method is that it requires use of only one test fungus. Certain fungi are more tolerant of particular preservative compounds than are other fungi. That the naphthenates are tolerant to some molds has already been demonstrated in this report. It is therefore evident that several test organisms should be selected on the basis of the products to be protected (18) and utilized in the test method.

CONCLUSIONS

The work presented by this report tends to show that a 20% solution of copper naphthenate (2% copper metal) can be depended upon to give as reliable protection as given by a 5% solution of pentachlorophenol. Under the conditions established in this test, the copper naphthenate solution was as satisfactory as the commercial pentachlorophenol solution. No visible decay had taken place on the test blocks of either preservative in five tests. However, the wafers treated with the 20% copper naphthenate solution supported contaminating mold growths.

The 20% solution of zinc naphthenate (2% zinc metal) tested under the same conditions could not be classed as

satisfactory. Quite visible evidence of decay was noted on several test blocks in one of three tests. Literature cited notes that the fungicidal qualities of zinc naphthenate are only about one-half as potent as are those of copper naphthenate. Blocks treated with the zinc naphthenate solution also supported the growth of mold contaminations.

Literature cited herein has shown that the use of copper naphthenate, although limited in its use for millwork because of its characteristic green color, promises to be valuable for use in creosote mixtures and other uses where color is of little importance.

The possibilities of utilizing zinc naphthenate as a wood preservative have been little explored. However, this compound does not have certain obnoxious properties possessed by the chlorinated phenols which would indicate a possible market.

Three criticisms have been directed against the N.D.M.A. test method as used here for evaluating oil-soluble preservatives. They are: (1) moisture content too low for optimum decay conditions, (2) test blocks too heavily loaded with preservative for a satisfactory test, and (3) one test fungus insufficient to evaluate the effectiveness of a preservative. It was pointed out that further investigations should be made: (1) to determine if a higher moisture content would be more desirable, and to determine the best method of obtaining this moisture content, (2) to determine if the test solutions should be diluted more than the test now specifies, and (3) to consider and select other test fungi most suitable to use with this test method.

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APPENDIX A

N.D.M.A. Method of Testing Oil-Soluble Wood Preservatives for Toxic Properties Using Wood Blocks Uniformly Impregnated

Taken from "Toximetric Method for Oil-Soluble Wood Preservatives", Ernest E. Hubert. Ind. and Eng. Chem. Anal. Edition 12, 138-140, 1940.

Testing Materials

1. Selection of Material and Test Blocks. The wood used for the preparation of test blocks shall be average density kiln-dried Ponderosa pine sapwood. It shall be straight-grained and free of blemishes and defects. Blocks 0.25 inch thick (longitudinal direction) shall be cut from surfaced pieces of wood measuring 1.375 inches on the radial surface and 2 inches on the tangential surface. The surfaces of the blocks shall be smooth, parallel, and free of slivers and dust and each test piece shall be numbered.

For each test, ten blocks shall be treated and exposed to each fungus used, and four similarly treated reference blocks shall be placed over uninoculated agar. To serve as controls, ten untreated sterilized blocks shall be exposed to each fungus used and four untreated blocks (reference blocks for the controls) shall be placed over uninoculated agar.

2. Conditioning of Test Blocks. Condition all blocks on glass rods to a moisture content of 12.4 $\pm$ 0.5% by exposure for 20 days to a saturated solution of sodium bromide ($NaBr.2H_2O$) at 26° to 28° C., or condition in an accurately controlled humidity room at 60% R. H. and 80° F.

3. Weighing. Weigh the test blocks accurately to two decimal places in the humidity room or from a desiccator containing a saturated sodium bromide solution to maintain the moisture content desired when handling the blocks for weighing. Record original weight as O.

4. Volume of test blocks. Measure the first and sixth test block in each set of 10 with an accurate millimeter scale recording length and width to the second decimal place. Measure the block at the middle of each of the four sides with a micrometer to determine the thickness. Compute volume and record as V.

5. Culture Material. Kolle flasks or other suitable glass containers shall be used. Make up the nutrient agar for the growth of the test fungi as follows:

Difco bacto agar	25 grams
Trommer's plain diastasic malt extract	25 grams
Distilled water	1000 grams

After dissolving, pour into each container sufficient agar to support vigorous growth for the duration of the test, stopper, and sterilize, autoclaving for 20 minutes at 15 pounds pressure. Inoculate each flask by placing at the center of the medium surface an inoculum block about 1 cm. square with the mycelium side upward. The pure culture of the test fungus shall not be older than 14 days and shall be grown at 26-28 degrees C.

6. Test Fungi. Test fungi of approved strains obtained from the Forest Products Laboratory shall be used. The test fungus for decay shall be F.P.L. No. 617, Lenzites trabea.

The inoculated containers shall be incubated at 26° to 28° C. for a period of 21 days, after which the test blocks shall be placed in them.

Testing Methods

7. Preparation of Preservative. Preservative shall be tested in dilution and type of solvent recommended by the manufacturers in half-strength solution obtained by diluting with an equal volume of Stoddard solvent naphtha.

8. Impregnation of Test Blocks. Place all 14 blocks to be treated in a 2-liter, heavy-walled filter flask and weight them down with glass marbles. Fit a 1-liter separatory funnel in the rubber stopper, connect the flask with a manometer and the vacuum line, and exhaust the flask to a pressure of 125 ± 2 mm. of mercury, for a period of 30 minutes. At a temperature of about 25° C. place enough treating solution in the separatory funnel to cover the blocks later.

Cut off the vacuum and introduce the treating solution without admitting air. Shake the flask to remove air bubbles from the blocks. Allow the solution to stand for 15 minutes, then break the vacuum.

After 5 minutes remove treated blocks, blot off excess liquid and keep the blocks until weighed in a desiccator over the treating solution used. Weigh the blocks soon after treating and record weight as T.

9. Reconditioning. Place treated blocks on glass rods, dry them 13 days in the laboratory air, and then recondition them for 7 days under conditions specified in Item 2. Reweigh the blocks after the 20-day period and record weight of the test and reference blocks as W and G respectively.

10. Incubation. Place 10 similarly treated test blocks in the containers on two glass rods of 0.125 inch (0.32 cm.) in

diameter, resting on the surface of the fungus mat. Similarly place ten steam-sterilized (10 minutes at 100°) untreated test blocks after conditioning as indicated in Item 2, in other containers. Place 4 treated reference blocks on rods over uninoculated agar. Incubate at 26° to 28° for 63 days.

11. End of Test. At the end of the 63-day incubation period remove the treated and untreated test blocks, scrape clean of mycelium, oven-dry, weigh, and record weight as F. Oven-dry the reference blocks and record as R. Controls should show at least 25% loss in weight due to decay.

12. Computations.

Volume of test blocks = V

T - O = A, grams of treating solution in test block.

$\frac{A}{V}$ = B, absorption in grams per cc.

B x 62.44 = C, absorption in pounds per cu. ft.

$\frac{C \times \%\text{ of toxicant}}{100}$ = D, absorption of toxicant in pounds per cu. ft.

W = weight of reconditioned treated blocks and conditioned blocks in case of controls (Item 9).

R = oven-dry weight of treated reference blocks at end of test, and of untreated reference blocks at end of test in case of controls.

G = weight of reconditioned treated reference blocks, and of conditioned untreated reference blocks in case of controls.

$\frac{WR}{G}$ = Z, computed oven-dry weight of test blocks before incubation.

F = oven-dry weight of treated and control blocks after incubation.

$\frac{Z - F}{Z} \times 100$ = E, % of loss in weight due to decay.

APPENDIX B

NEW METHOD FOR THE DIFFERENTIATION OF HEART AND SAP IN PINE WOOD

by

J. E. Koch and W. Krieg

(Chemeker Zeitung 62(15): 140-141, Feb. 19, 1938)

1. Prepare a solution of 5 grams benzidine in 25 grams of HCl (about 25%) and 970 grams water.

2. Prepare a 10% solution of sodium nitrite.

3. Keep the two solutions separate.

4. Immediately before testing pour equal amounts of the solutions together. Either dip the wood in the solution or paint it on the surface. After treating the wood it is advisable to wash it with water or blot up the excess solution because the colors then hold better.

5. This treatment colors the heartwood dark red-brown, the sapwood yellow, after a time the sapwood takes on a dark yellow or brown color. Nevertheless, the difference between the heartwood and sapwood color remains distinct.

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6/7/39 NDMA