EXPERIMENTS ON TOXICITY, LEACHING, AND FIRE-RETARDING EFFECTIVENESS OF WOLMAN SALTS

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EXPERIMENTS ON TOXICITY, LEACHING, AND FIRE-RETARDING EFFECTIVENESS OF WOLMAN SALTS

By

R. H. BAECHLER, Chemist[1]

Forest Products Laboratory,[2] Forest Service
U. S. Department of Agriculture

The purpose of the experiments[3] described in this report was to study properties of the Wolman salts, Triolith and Tanalith, with regard to: (1) toxicity to the wood-destroying fungi Coniophora cerebella, Lentinus lepideus, Lenzites trabea, Madison No. 517, Polyporus vaporarius, Poria incrassata; (2) resistance to leaching from small blocks of impregnated wood repeatedly submerged in water; (3) resistance of the treated, leached blocks to attack by the organisms listed above; and (4) fire resistance of treated wood.

Tanalith (Tanalith-U) normally contains sodium fluoride, dinitrophenol, sodium chromate, and sodium arsenate. Triolith (Triolith-U) normally contains sodium fluoride, dinitrophenol, and potassium bichromate.[4] The composition of these two preservatives is covered by U. S. Patent No. 1,957,873, to K. H. Wolman, May 8, 1934. A supply of these preservatives was provided for the experiments by the American Lumber and Treating Company. Analyses made by the Forest Products Laboratory showed that the composition of the preservatives tested was substantially as represented.

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[1]Acknowledgement is made to E. Bateman, C. A. Harrison, and Geo. M. Hunt of the Forest Products Laboratory for assistance in the work here described and for aid in the preparation of this report, and to C. A. Richards of the Bureau of Plant Industry for furnishing the fungus samples.

[2]Maintained at Madison, Wis., in cooperation with the University of Wisconsin.

[3]These experiments were made by the Forest Products Laboratory at the request of the American Lumber and Treating Company, Chicago, Ill., and at their expense.

[4]The term Tanalith throughout this report refers to the new formula often called Tanalith-U and Triolith similarly refers to Triolith-U.

RESULTS OF EXPERIMENTS

Toxicity

The toxicity was determined by the American method, which is described in Industrial and Engineering Chemistry, Analytical Edition, Volume 2, page 361, October 1930. The method consists in placing fungus transplants on malt-agar culture media, in which different concentrations of the chemical under test have been incorporated, and maintaining them under conditions favorable to fungus growth for 28 days. When the concentration is found that is just sufficient to prevent the growth of the fungus transplant, a further test is made to determine whether the fungus has been killed or is merely prevented from growing. The percentage of chemical that is just sufficient to kill the fungus is called the killing concentration. The results obtained with the two preservatives and the six fungi used are given in table 1.

Table 1.--Killing concentrations of Tanalith and Triolith measured against six fungi

Fungus	Killing concentrations[1]	
	Tanalith	Triolith
Lenzites trabea	0.30	0.12
Madison No. 517	.20	.16
Lentinus lepideus	.12	.15
Coniophora cerebella	.04	.015
Poria incrassata	.015	.05
Polyporus vaporarius	.01	.015

[1] Expressed as a percentage of the weight of the culture medium.

Leaching Tests

The leaching tests were made on small blocks of southern yellow pine sapwood 1.5 by 2.5 by 5 cm (approximately 0.6 by 1 by 2 inches) in size. They were first conditioned to a moisture content of about 14 to 15 percent by allowing them to come to constant weight in a room kept at 80° F. and 75 percent relative humidity, then treated to approximate refusal with a 2 percent solution of Tanalith or Triolith. The treatment was made by placing the blocks in a container in a small treating cylinder, drawing a vacuum of about 28 inches for 1 hour, then allowing preservative solution to be drawn into the container while the vacuum was maintained, and finally applying air pressure of 60 to 70 pounds per square inch for an hour. The preservative solutions were at room temperature. The individual blocks were weighed before and after treatment in order to determine the amount of preservative absorbed. After treatment they were dried again to a moisture content of 14 to 15 percent by allowing them to come to constant weight in the 75 percent humidity room. The absorptions obtained are shown in table 2.

Table 2.--Absorptions of Tanalith and Triolith in small blocks to be used for leaching tests.

Preservative	Amounts of dry salt absorbed		
	Maximum	Minimum	Average of 20 blocks
	Pounds per cubic foot	Pounds per cubic foot	Pounds per cubic foot
Tanalith................	0.926	0.877	0.904
Triolith................	.893	.815	.850

After drying under these conditions for 53 days, the blocks were leached by the following method: Ten blocks of Tanalith treated wood were placed in each of two vacuum bottles and ten blocks of Triolith treated wood in each of two other bottles -- a total of 20 blocks for each preservative. A vacuum of about 28 inches was then drawn and, with the vacuum pump still running, 500 cc of distilled water was drawn into each bottle. The vacuum was then released, causing a rapid absorption of water by the blocks so that most of them sank while the rest floated very low in the water. The flasks were then shaken occasionally and at the end of an hour the leaching water was poured off and saved for analysis. Another 500 cc portion of distilled water was then poured over the blocks in each flask (omitting the vacuum) and, with occasional shaking, left for 2 hours, after which it was poured off and saved for analysis. In a similar manner the blocks were leached for a period of 4 hours and then for 31 periods of 8 hours each. Only one 8-hour leaching was made in any 24-hour period and between leachings the blocks were left in the stoppered bottles, without water.

The water from each leaching was made up to exactly 500 cc by adding distilled water and kept separately. Analyses for the principal ingredients in each preservative were made of the leaching water from each of the first four leachings. After that, leachings were combined as shown below, by taking 100 cc from each of the leachings included in the respective groups and analyzing the composite samples:

5th to 6th period, inclusive (2 leachings)
7th to 10th period, inclusive (4 leachings)
11th to 18th period, inclusive (8 leachings)
19th to 34th period, inclusive (16 leachings)

The chromate in the leaching water was determined by the well-known method of titrating the iodine liberated from potassium iodide in acid solution. Because of the small amount of chromate present, N/100 sodium thiosulphate was used. The method was checked against small amounts of chromate and was found to be satisfactory. The method used for the determination of arsenate was checked likewise. It consisted of reducing the arsenate to arsenite with potassium iodide, boiling off the iodine and titrating the arsenite with N/50 iodine in the presence of an excess of sodium bicarbonate.

The method used for the determination of fluoride was to precipitate it as calcium fluoride as described in Scott's "Standard Methods of Chemical Analysis" and elsewhere. Chromate and arsenate were first removed by precipitation of their silver salts, the excess silver in the filtrate being removed by the addition of sodium chloride and filtration.

The leaching of the dinitrophenol was not studied since no simple accurate method was known for the determination of small amounts of that chemical in the presence of wood extracts. The color of dinitrophenol persisted in all leachings, however, indicating that an appreciable quantity remained in the wood after the final leaching.

Table 3 gives the weight of sodium fluoride and potassium dichromate leached from the Triolith treated blocks and similar data for sodium fluoride, sodium chromate, and sodium arsenate from the Tanalith treated blocks. Figures 1 and 2 show the same data calculated on a percentage basis. It is evident from the figures that both the chromates and dichromates were almost nonleachable and that the arsenate was much less leachable than the sodium fluoride, or at least its rate was much slower. In making the calculations for figures 1 and 2 it has been assumed that the difference in weight before and after treatment, multiplied by the concentration of the treating solutions, gave the amount of salts retained by the wood. This amount then becomes the basis for the percentage calculation and is 100 percent.

In order to obtain information as to whether or not the amount or the rate of leaching of the sodium fluoride was affected in any way by the presence of either the chromate or the arsenate, or both, a third series of blocks matching those used for Triolith and Tanalith was treated with sodium fluoride alone. The procedure of treating, drying, and leaching of these blocks was identical with the procedure used for the Wolman salts,

Table 3.--Amounts in grams of Tanalith and Triolith leached from treated blocks

Leaching cycle	Tanalith						Triolith			
	Arsenate[1]		Chromate[2]		Fluoride[3]		Chromate[4]		Fluoride[3]	
	Flask 3	Flask 4	Flask 3	Flask 4	Flask 3	Flask 4	Flask 1	Flask 2	Flask 1	Flask 2
1	0.0033	0.0025	0.00080	0.00080	0.0259	0.0167	0.00085	0.00085	0.0904	0.0770
2	.0017	.0017	.00173	.00173	.0129	.0194	.00193	.00193	.0210	.0188
3	.0067	.0067	.00173	.00146	.0285	.0280	.00181	.00181	.0156	.0404
4	.0013	.0014	.00186	.00186	.0818	.0764	.00145	.00181	.0700	.0775
5 to 6, inclusive	.0225	.0225	.00146	.00146	.0740	.0915	.00120	.00121	.1320	.1240
7 to 10, inclusive	.0209	.0217	.00053	.00027	.0786	.0860	.00048	.00024	.1334	.1495
11 to 18, inclusive	.0250	.0267	.00053	.00053	.1080	.1118	.00048	.00048	.2904	.2290
19 to 34, inclusive	.0334	.0350	.00266	.00160	.0646	.0880	.00210	.00120	.1420	.1240
Total..............	.1148	.1182	.01130	.00971	.4743	.5178	.01030	.00953	.8948	.8402
Initial amount	.627	.631	1.023	1.030	.629	.633	.919	.908	1.264	1.250
Percentage leached	18.3	18.7	1.1	0.9	75.4	81.8	1.1	1.0	70.8	67.2

[1]Calculated as sodium arsenate.

[2]Calculated as sodium chromate.

[3]Calculated as sodium fluoride.

[4]Calculated as potassium dichromate.

but the work was done at a different time. The initial amount injected, as caclucated from the treating records and the amounts in grams leached out at the various periods, are given in table 4. The same data calculated to a percentage basis are given in figure 3. The leaching curve for sodium fluoride from Triolith has been plotted for comparison. The curve for the leaching of sodium fluoride from Triolith treated wood seemingly crosses the curve for the straight sodium fluoride treatment. This is believed to be due to inaccuracies inherent in the experimental methods employed.

Table 4.--Amounts in grams of sodium fluoride leached from treated blocks

Leaching cycle	Sodium fluoride leached	
	Flask 5	Flask 6
1	0.4690	0.4890
2	.3270	.3600
3	.2590	.2270
4	.2770	.2840
5 to 6, inclusive	.3110	.2630
7 to 10, inclusive	.1010	.0940
11 to 18, inclusive	.0110	.0110
19 to 34, inclusive	.0240	.0280
Total	1.7790	1.7560
Initial amount	2.553	2.552
Percentage leached	69.7	68.8

The amount of sodium fluoride leached in 32 periods was practically the same for both cases, being about 69 percent. At the beginning, the sodium fluoride leached much faster when present alone than when the chromate was used in combination in the treatment. As the leaching progressed, the total amount leached approached the same value. The last three-fourths of the leaching curve for the sodium fluoride alone shows that little or no more could be obtained even in a very long time.

Other investigators have reported somewhat higher amounts of sodium flouride leached from blocks treated with straight sodium fluoride and with mixtures corresponding to Wolman salts. It was at first believed that the rather low results obtained in these tests might be due to the fact that the leachings were stored in glass containers for some time before the analyses were begun, thus permitting some reaction between the fluoride and the glass. An opportunity to check this supposition presented itself when the leaching tests were repeated to provide additional blocks for tests on the resistance to fungus attack to be described later. The leachings from two of the flasks containing Triolith treated blocks and two containing Tanalith treated blocks were made alkaline at once and then analyzed for fluoride. The results were only slightly higher than those obtained in the first test, showing that the low values cannot be explained by a reaction between the fluoride and the glass. Some of the leached Triolith treated blocks were analyzed for sodium fluoride according to an unpublished method developed at the Forest Products Laboratories of Canada. The amount thus found, when added to the total amount found in the leachings, gave a total recovery of 87.6 percent of the amount originally present in the treated blocks as calculated from the treating records.

The method used for determining sodium fluoride in the leachings, according to Scott,[2] gives results which are slightly low, owing to the solubility of calcium fluoride. This error increases, on the percentage basis, as the amount of fluoride in the sample decreases. It is believed, therefore, that the leaching curves for sodium fluoride are somewhat low in all cases. The fluoride determinations from the first series of leachings are given here in preference to those obtained in the second series since the former are more comparable with those obtained with straight sodium fluoride-treated blocks.

Resistance of Leached Blocks to Fungus Attack

After the leaching was completed, the leached blocks were returned to a 75 percent humidity room for drying. After drying for 114 days, ten Tanalith blocks and ten Triolith blocks were brought to a moisture content of about 50 percent by putting them in a vacuum flask with a measured amount of water, drawing a vacuum of about 28 inches for about a minute and then releasing the vacuum. They were then sterilized for one-half hour in saturated steam at atmospheric pressure. Untreated blocks of the same kind were similarly moistened and sterilized in a separate container. The blocks were then exposed to vigorously growing pure cultures of *Coniophora cerebella* and *Lenzites trabea* in Kolle flasks, one treated and one untreated block to a flask, and five flasks for each fungus and each preservative. The blocks were laid on glass rods in the flasks to prevent their becoming too saturated with moisture from the malt-agar culture medium upon which the fungi were growing. The flasks were kept at room temperature for four months, after which the blocks were removed, inspected for decay, dried at 105°, and weighed to determine their loss in weight.

The results are given in table 5, series A. In all flasks the untreated control was covered with mycelium and in practically all cases it was

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[2] Scott's "Standard Methods of Chemical Analysis," Fourth Ed., Vol. 1, p. 217.

Table 5.--Condition of leached blocks after four months' exposure to fungi in Kolle flasks

Fungus used	Preservative	Loss in weight: Maximum	Loss in weight: Minimum	Loss in weight: Average	Condition of blocks
		Percent	Percent	Percent	
SERIES A					
Coniophora cerebella	Triolith	3.9	1.3	2.2	5 blocks no apparent attack, 1 block had light mycelial growth on one side.
Do	None	39.0	27.0	34.7	5 blocks covered with mycelium, 4 discolored and softened.
Do	Tanalith	2.5	1.4	1.9	Light growth over 3 blocks, none over 2.
Do	None	26.5	17.2	21.2	All blocks covered with mycelium, discolored, softened.
Lenzites trabea	Triolith	13.8	3.9	9.2	Slight attack on all 5 blocks.
Do	None	42.3	17.8	27.3	All blocks covered with mycelium, discolored, and softened in springwood.
Do	Tanalith	47.8	17.9	38.9	All blocks severely attacked.
Do	None	45.4	29.9	37.2	do
SERIES B (RETEST)					
Coniophora cerebella	Triolith	11.3	3.0	6.5	All blocks covered with mycelium, 3 show slight softening.
Do	None	62.0	38.2	54.3	All blocks severely attacked and greatly reduced in size.
Do	Tanalith	3.8	3.2	3.6	All blocks covered lightly with mycelium, very little softening.
Do	None	50.0	42.1	45.5	All blocks severely attacked and greatly reduced in size.
Lenzites trabea	Triolith	13.4	5.5	9.7	Covered with mycelium, 3 considerably softened, 2 slightly softened.
Do	None	63.0	24.3	42.5	3 blocks severely attacked and reduced in size, 2 soft but not reduced.
Do	Tanalith	41.5	27.3	34.2	All blocks severely attacked and reduced in size.
Do	None	36.2	32.4	34.4	do
Madison No. 517	Triolith	4.5	4.0	4.2	Light growth on 4 blocks, no softening.
Do	None	21.8	4.0	12.9	All more or less covered with mycelium, some softening in all blocks.
Do	Tanalith	3.0	2.5	2.8	Unattacked.
Do	None	13.8	6.7	10.6	Almost covered with light mycelium, very little softening.
Polyporus vaporarius	Triolith	2.9	1.8	2.4	4 show dark spots, no softening, 1 shows a few soft spots.
Do	None	27.2	20.7	24.3	All blocks covered with mycelium and softened on all sides.
Do	Tanalith	9.5	3.1	6.7	Light mycelial growth on all blocks, very little softening.
Do	None	32.0	26.5	28.9	All blocks covered with mycelium and softened on all sides.
Poria incrassata	Triolith	5.9	3.3	4.7	All blocks covered with mycelium, very little softening.
Do	None	43.6	27.6	34.5	All blocks severely attacked and reduced in size.
Do	Tanalith	5.9	4.0	5.0	All blocks covered with light mycelium, very little softening.
Do	None	50.2	40.9	47.3	All blocks severely attacked and reduced in size.
Lentinus lepideus	Triolith	5.3	4.0	4.7	All blocks covered with mycelium, very little softening.
Do	None	44.6	35.5	42.1	All blocks severely attacked and reduced in size.
Do	Tanalith	2.7	2.1	2.5	All blocks covered with light mycelium, a few dark spots, practically no softening.
Do	None	40.8	27.7	34.4	All blocks severely attacked and reduced in size.

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severely attacked by the organism, being discolored and softened. Coniophora cerebella was able to establish itself only to a slight extent on the treated and leached blocks although it spread over part of the surface of several blocks. In these particular blocks the toxic material which remained did not kill the organism in contact with the wood although it protected the wood from invasion during the period of the tests. Lenzites trabea attacked the leached Triolith blocks slightly, whereas the leached Tanalith blocks were about as severely attacked by Lenzites trabea as were the controls.

At the request of the cooperator, these tests were repeated with the same organisms and with four additional organisms namely, Madison 517, Lentinus lepideus, Polyporus vaporarius, and Poria incrassata. The results of this second series of tests are given in table 5, series B. In this series Coniophora cerebella again attacked the treated and leached blocks only to a very slight extent. Lenzites trabea seemed to attack the leached Triolith treated blocks more severely than it had in the first series, whereas the Tanalith treated blocks were again about as severely attacked as were the controls. Madison 517, Polyporus vaporarius, Poria incrassata, and Lentinus lepideus showed little or no attack on the leached treated blocks. Madison 517 attacked the controls to only a moderate extent; in all other cases the controls were badly decayed.

Fire Resistance of Treated Wood

In order to obtain an indication of the extent to which the fire resistance of wood may be influenced by treatment with Tanalith and Triolith, sapwood boards of southern yellow pine were treated to approximate refusal with 2 percent solutions of these salts, dried, and tested in the Forest Products Laboratory fire tube. The treatments were made by the full-cell process, under a pressure of about 150 pounds per square inch for 4 hours at solution temperatures of about 170° to 180° F. The boards were 7/8 by 7-1/2 inches by 5 feet 3 inches in size. They were dried to about 12 or 13 percent moisture content before treatment and again after treatment, by leaving them for several weeks in a room maintained at 65 percent relative humidity at a temperature of 80° F.

After reaching approximate equilibrium in the 65 percent humidity room, the boards were moved into a room maintained at 30 percent relative humidity and 80° F. and left until they had reached equilibrium (at about 6 to 7 percent moisture content). Two of the boards treated with each preservative were then surfaced (on both sides) to a net thickness of 3/4 inch and cut into fire-tube test specimens as indicated in figure 4, after which the specimens were returned to the 30 percent humidity room and kept there until tested in the fire tube.

Table 6 shows the absorptions obtained with each preservative in the six boards of its charge and indicates the boards selected for test. It may be noted that the absorption of dry salt in pounds per cubic foot is two to three times the minimum absorption of these salts commonly recommended in the commercial treatment of lumber with these preservatives (for dry situations a minimum of 0.25 pound per cubic foot and for general outdoor use a minimum of 0.3 pound per cubic foot). The absorptions in the boards tested were considerably higher than the amount of salt that might be found in the outer inch of a timber of tie size or larger with an absorption of 0.3 pound per cubic foot and much too high to be representative of ordinary 1 and 2 inch lumber commercially treated.

Table 6.--Absorption in boards treated for fire-tube tests

Preservative	Board No.	Absorption		Remarks
		Solution	Dry salt	
		Pounds per cubic foot	Pounds per cubic foot	
Tanalith..	(785-3	42.9	0.86	
	(795-3	42.9	.86	
	(781-3	43.0	.86	Tested in fire tube
	(798-2	40.3	.81	........do...............
	(793-1	41.0	.82	
	(786-3	40.8	.82	
Triolith..	(785-2	43.9	.88	........do...............
	(791-1	43.6	.87	
	(798-1	41.1	.82	
	(842-3	39.5	.79	........do...............
	(840-1	40.3	.81	
	(788-3	39.6	.79	

The fire resistance of the treated wood was tested in the fire tube by the standard method in use with this apparatus at the Forest Products Laboratory.[6] The test consists in hanging the test specimen (3/8 by 3/4 by 40 inches in size) in a ventilated tube on a balance arm and exposing the lower 10 inches of the specimen to a gas flame for 4 minutes. The gas flame is then removed and the specimen allowed to burn at will. A pointer attached to the balance arm indicates the percentage loss in weight as the wood is consumed during the test and a thermocouple at the top of the tube shows the temperatures attained at that point.

[6] T. R. Truax and C. A. Harrison, Amer. Soc. Testing Materials, Proc. 29 (Pt. 2), 973-989 (1929); Geo. M. Hunt, T. R. Truax, and C. A. Harrison, Amer. Wood Pres. Assoc. Proc., 1930, p. 130-159.

Table 7 shows the results obtained in the tests on the boards treated with Triolith and Tanalith. For comparative purposes previously published data[7] are also given for untreated wood and for wood treated with diammonium phosphate. The Tanalith and Triolith treated specimens showed somewhat lower weight loss than the untreated specimens but no reduction in maximum temperature. This is in accord with the general experience that absorptions of less than 1 pound of salt per cubic foot do not produce an important reduction in the loss in weight during test. The data given for diammonium phosphate, one of the most effective fireproofing chemicals, illustrate the point. The Tanalith and Triolith treated specimens showed some tendency to continue glowing after flaming ceased and this may be related to the high temperatures shown. Normal absorptions of Tanalith and Triolith would have shown still lower effectiveness.

Table 7.--Results of fire-tube tests on Tanalith and Triolith treated boards of southern yellow pine compared with untreated boards and boards treated with diammonium phosphate

Preservative	Board No.	Average absorption	Test results: Loss in weight when flaming ceased	Test results: Maximum temperature	Test results: Tendency to glow	Test results: Number test specimens averaged
		Pounds per cubic foot	Percent	°C.		
Tanalith..	(798-2	0.81	66	860	Glowed	5
	(781-3	.86	69	880	...do...	5
Triolith..	(842-3	.79	70	875	...do...	5
	(785-2	.88	73	875	...do...	5
Untreated	....	....	84	782	...do...	16
Diammonium phosphate	....	.90	69	717	None	16
Do......	....	3.23	22	191	...do...	16
Do......	....	5.15	18	168	...do...	16

[7] Geo. M. Hunt, T. R. Truax, and C. A. Harrison, Amer. Wood Pres. Assoc. Proc., 1931, p. 117.

To summarize, it may be said that the absorptions of Tanalith and Triolith ordinarily used in commercial treatment have no marked effect on the fire resistance of the treated wood, as measured by the fire-tube test, but the small effect noted is in the direction of increasing fire resistance.

Summary

The toxicity determinations on Triolith and Tanalith showed that both are very toxic to six typical wood destroying fungi and have adequate toxicity for wood preserving purposes.

In the severe leaching tests, the chromium salts proved highly resistant to leaching, the total amount leached throughout the test being only about 1 percent. About one-fifth of the arsenate leached from the Tanalith treated blocks, which is much higher than the proportion of chromium salts leached but still indicates considerable resistance. The sodium fluoride leached out more rapidly and completely than the other salts, the average amount leached falling between 70 and 80 percent of the amount originally injected. It was found that the leaching of sodium fluoride from Triolith treated blocks started at a slower rate than from similar blocks treated with straight sodium fluoride, although the total amount leached at the end of the test was approximately the same in both cases.

When the leached blocks were exposed to fungus attack in Kolle flasks, it was found that the leaching had been carried so far that they were not immune to attack but, against all but one fungus, they showed much greater resistance than the untreated control blocks. The fungus *Lenzites trabea*, which is known to be arsenic tolerant, attacked the leached Tanalith treated blocks about as severely as it did the untreated blocks.

Lumber treated with 0.79 to 0.88 pound of Wolman salts per cubic foot of wood showed no significant resistance to fire in the Forest Products Laboratory fire-tube test.

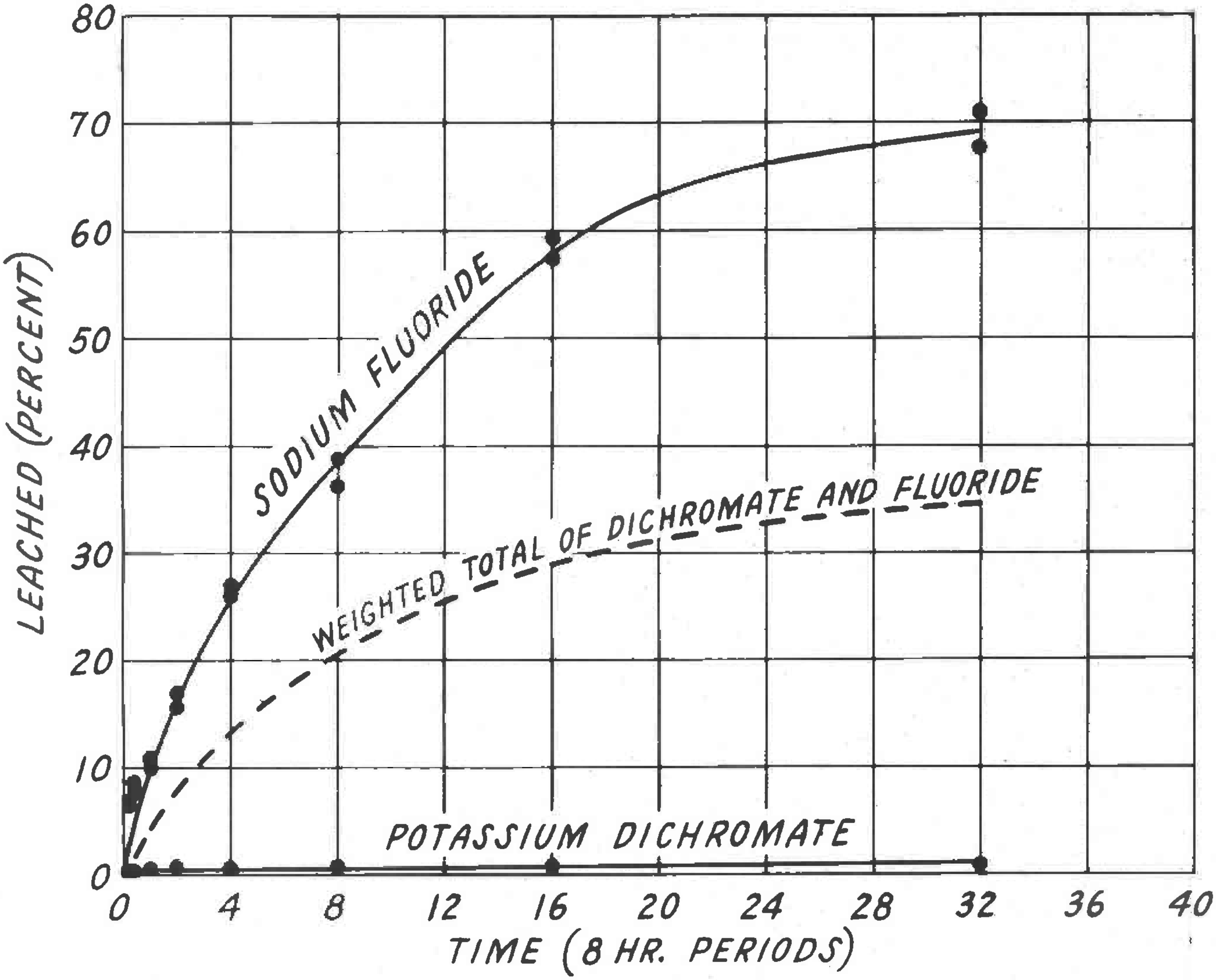

FIG. 1
LEACHING OF TRIOLITH INGREDIENTS

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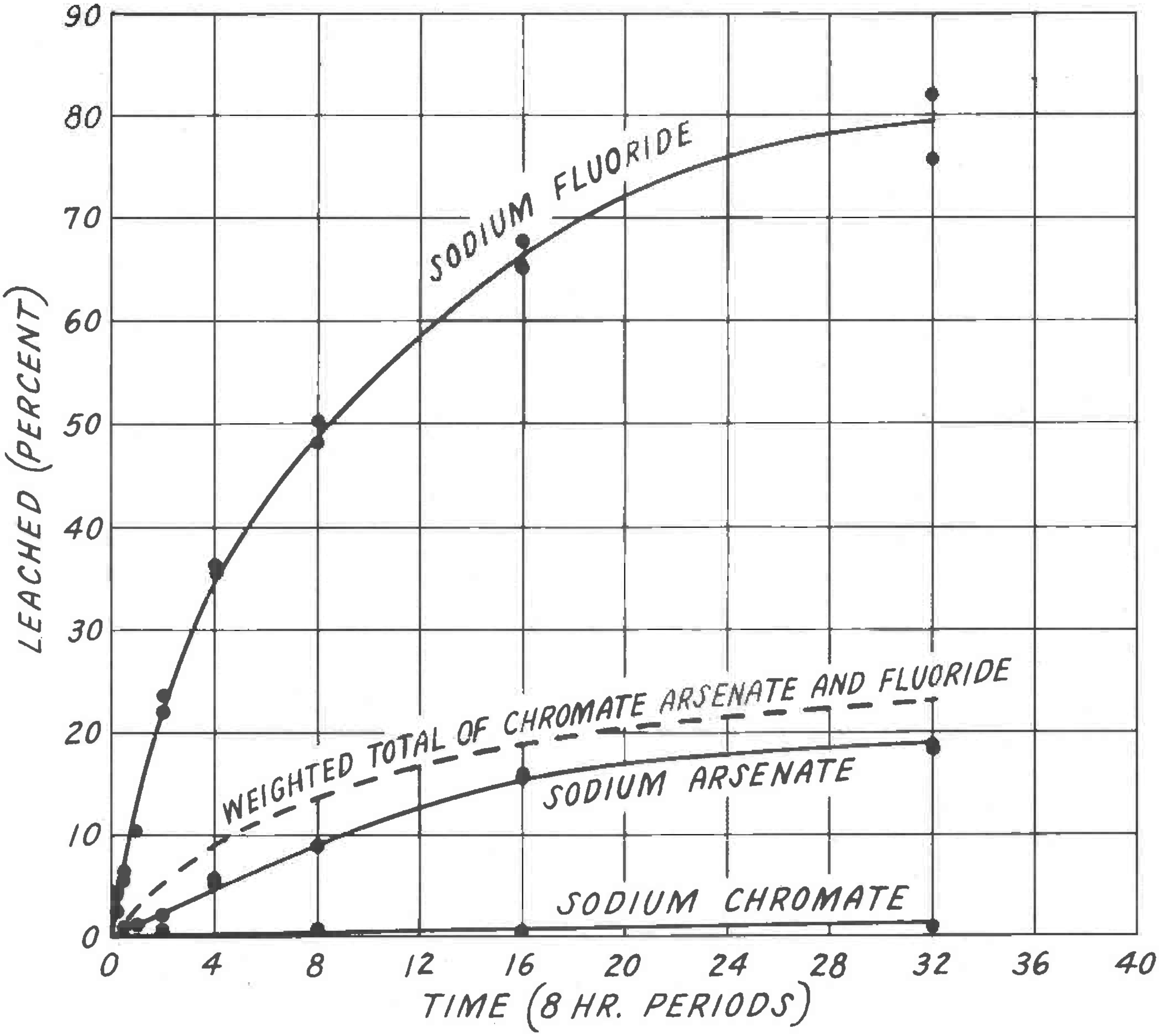

FIG. 2
LEACHING OF TANALITH INGREDIENTS

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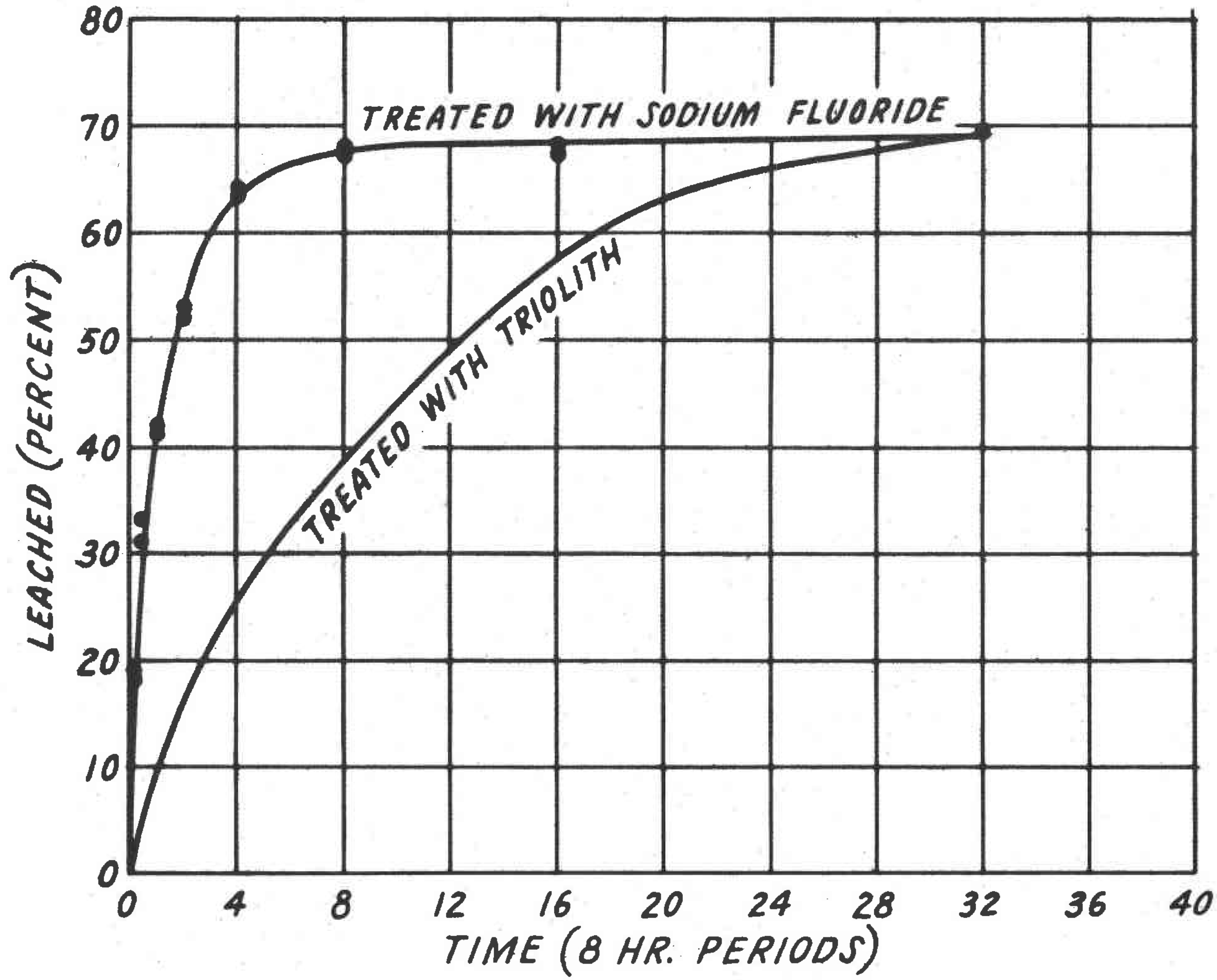

FIG. 3
LEACHING OF SODIUM FLUORIDE

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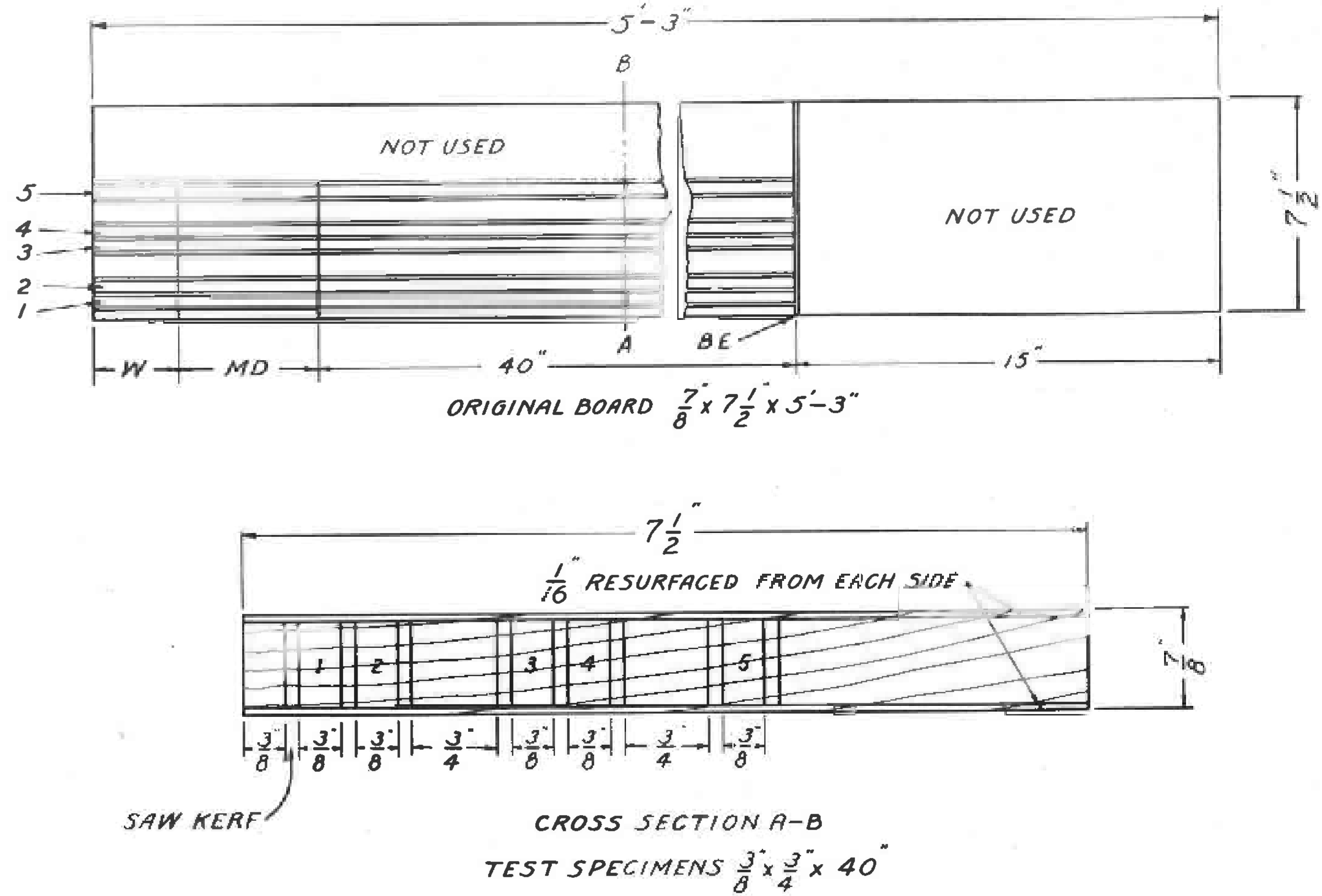

SPECIMENS 1, 2, 3, 4 & 5 INCLUDED IN TEST
W-ABOUT 3" CUT OFF OF ENDS OF SPECIMENS AND WASTED
MD-ABOUT 5" USED AS MOISTURE DISCS
BE-BOTTOM END OF SPECIMENS IN TEST

FIG. 4-METHOD OF CUTTING SPECIMENS FOR FIRE TUBE TESTS

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