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Oil-tar Creosote For Wood Preservation

By
GLENN VOORHIES

Bulletin Series, No. 13

June 1940

A Research Project of the
School of Forestry

with the Cooperation of the
Portland Gas and Coke Company

Engineering Experiment Station
Oregon State System of Higher Education
Oregon State College

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Assistant Professor of Wood Products

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Oil-tar Creosote For Wood Preservation

By

GLENN VOORHIES,
Assistant Professor of Wood Products

I. ACKNOWLEDGMENTS AND SUMMARY

1. **Acknowledgments.** The author of this paper is indebted to E. L. Hall, Vice President and Chief Engineer of the Portland Gas and Coke Company, for suggesting the problem, for supplying the material, and for constructive criticism of the working plan; to Geo. W. Gleeson, head of the Department of Chemical Engineering, for data on the physical properties of the creosote oils; to Dr. C. E. Owens, Dr. D. F. Rogers, and Dr. S. M. Zeller, of the Department of Botany, for suggestions and use of laboratory facilities; to R. H. Rawson, consulting timber engineer, for data on the actual treatment of specimen posts; to C. W. Osborne for cooperation in treating the posts in the experimental retort of the Pope and Talbot Lumber Company; to E. G. Mason, assistant dean of the School of Forestry, for helpful suggestions and allowance for time involved in the study; and to S. H. Graf, director of engineering research, for counsel and editorial assistance in preparation of this bulletin. All photographs were made by R. M. Evenden and charts were redrawn for publication by W. H. Paul.

2. **Summary.** The objective of this study was to determine the suitability of oil-tar creosote for a wood preservative.

Since oil-tar creosote, a by-product of the Portland Gas and Coke Company, has never been manufactured for or sold previously as a wood preservative, it was necessary to make both general and specific comparisons of the oil-tar creosote to other commercially accepted preservatives such as coal-tar creosote, water-gas-tar creosote, and zinc chloride. The study was pointed to those characteristics that are considered desirable qualities in a wood preservative, namely: (a) toxicity, (b) permanence (volatility and leaching), (c) penetration, (d) availability and cost, and (e) safety in handling and use.

Grade I coal-tar creosote is considered by the American Wood Preservers Association as the high standard of preservatives. With this as a major premise many comparisons have been made of coal-tar and oil-tar creosote. It has been shown that the only differences between the A.W.P.A. specifications for Grade I coal-tar creosote and oil-tar creosote are: (a) the guaranty of coal-tar origin, and (b) the fact that the specific gravity of the oil-tar creosote is 0.01 below the written specifications. It has been further shown by a study of the literature that the presence of the tar acids is apparently not necessary for a long-lasting preservative. Further, the aromatic hydrocarbons as found in the oil-tar creosote have a greater toxicity than the phenols (tar acids) in the corresponding boiling range. The conclusions of several well-known authorities have been cited in support of the belief that the tar acids are not the essential toxic material in coal-tar creosote.

The manufacturing processes for the various creosotes have been explained. It has been shown that the temperature employed in the carbonization of coal has a corresponding effect on the characteristics of the tars. It is also shown that the high-temperature cracking of California petroleum-oil residuum almost

completely changes the aliphatic hydrocarbons to aromatic hydrocarbons, which are the desirable hydrocarbons in creosote oil. Comparable distillations have been made and the findings show that the oil-tar creosote has a greater percentage of high-boiling oils (above 355 C) than the coal-tar creosote tested; this in spite of the slightly lower specific gravity. The viscosities of the two oils under test were compared and it was found that the viscosity of the oil-tar creosote was less than that of the coal-tar creosote, especially at room temperatures. This indicated an oil that would have good penetrating and diffusion properties.

In order to check the resistance to volatilization, comparable tests were made by determining the percentage of creosote lost from open dishes when exposed to temperatures approximating service conditions. No significant difference was noted in this study. A further similar check was made by studying the percentages of creosote lost from impregnated wood blocks. No significant differences were found in the latter study. Studies were also made on the resistance of impregnated wood blocks to leaching by water. No significant differences were noted.

In order to determine the action of the oil-tar creosote under actual commercial treating operations, thirty-one 4 in. x 4 in. x 5 ft Douglas-fir posts were given a standard empty-cell treatment. It was found that satisfactory penetrations and absorptions could be obtained in the usual treating procedure.

The pathological study was designed to study not only the toxicity of the creosote oil immediately after impregnation into the wood but also to study the ability of the oils to maintain their toxicity over a period of time when put into service conditions.

Two series of tests employing two representative fungi and involving the study of some 400 sample blocks were made. The first series of tests was designed as an orientation series and involved the use of several different concentrations of creosote from 0.05% to 100%. The second series of tests was designed to act as a check series and to study the toxic properties at the marginal treating solution. Certain improved procedures and solvents were used in the second series in order to comply fully with standard procedure and to eliminate some of the variable factors that were apparent in the first series of tests.

The Kolle flask method was used. This is an adaptation of the European block method and is essentially as follows: The malt-extract medium is prepared and poured into Kolle flasks, the medium then inoculated with the desired test fungus, and a mat of mycelium grown over the surface of the agar. The carefully weighed and treated blocks are then placed on glass rods on the mycelium in the flask and allowed to incubate for 63 days. At the end of the 63-day period the blocks are removed, oven dried, weighed, and the per cent loss of weight of the treated blocks taken as a measure of the ability of the preservative to inhibit or kill the growth of the fungus within the wood.

Using this procedure and on the basis of the described sampling, there is no significant difference in the ability of the two creosote oils to act as preservatives against common wood-rotting fungi. This conclusion is based on the analysis of the data from the various tests of the toxicity of the creosoted wood blocks immediately after treating, together with the analysis of the data obtained from the tests of the creosoted wood blocks after subjecting the test blocks to volatilization and leaching. Even though the initial toxicity of the coal-tar creosote was slightly greater than that of the oil-tar creosote, the coal-tar oil did not prove as satisfactory as the oil-tar creosote after the blocks were exposed to the extremes of service conditions.

Photographs, tables, graphs, and charts have been employed as aids in showing the results.

II. INTRODUCTION

1. **Present status of industry.** Wood preservation in the Pacific Northwest timber industry occupies an increasingly important position. Within the past 3 years, two salt-treating retorts and one creosote retort have been added to the plant facilities in the Northwest. With the addition of these retorts there are now 28 retorts for creosote, 5 retorts for salt treatment, and 51 nonpressure tanks available for treating operations (2).* Since the Pacific Coast has about 62 per cent (32) of the nation's sawtimber stand and about 24 per cent (37) of the forest land area of the United States, wood preservation will continue to be an important phase of the timber industry in Oregon, Washington, California, and Idaho. Even after the large stands of mature timber have been harvested there will always remain the smaller pole and sawtimber products that are admirably suited for railway ties, poles, and other types of treated construction materials. Recent developments in the field of prefabricated wooden structures employing creosote treatment and ring connectors have put wood structures in a position capable of competing with steel and re-enforced concrete construction. This is an especially important development for the timber and preservation interests because it opens the field for wider use of the product.

There is at present an actual demand for creosote for wood preservation on the Pacific Coast to the amount of about 12,000,000 gallons annually, with a potential demand of even greater amounts. The United States as a whole has used up to 150,000,000 gallons of creosote annually for wood preservation (22). Depending on the demand, 30 to 50 per cent of the annual requirement is imported from England, Scotland, Belgium, Japan, and Germany. Since the imported oil must reach the consumer in cargo shipments, more than 50 per cent of the oil that is used in the Pacific Northwest is imported.

The following table shows the origin of the creosote oil that is used on the Pacific Coast and in the entire United States:

Coal-Tar Creosote Used in Wood Preservation,
Based on Report of R. K. Helphenstine,
American Wood Preservers Association
Proceedings, 1938

| <i>Pacific Coast</i> | 1936 | 1937 |
|--------------------------|-------------|------------|
| Origin: | | |
| Domestic distillate..... | 5,463,400 | 4,726,306 |
| Imported distillate..... | 7,830,427 | 7,501,293 |
| <i>United States</i> | | |
| Origin: | | |
| Domestic distillate..... | 124,456,892 | 82,137,128 |
| Imported distillate..... | 30,256,107 | 35,225,781 |

* Numbers in parentheses refer to literature citations.

The U. S. Department of Commerce reported 58,189,527 gallons of creosote imported in 1937. It is assumed in the above tabulation that the difference in the two totals is accounted for by the fact that some of the imported creosote was resold as domestic creosote.

A local supply of creosote would have many advantages over an imported supply or even over a supply from the eastern United States. There would be no particular need for large inventories of oil at the treating plants, no need for anticipating needs over a long period of time, and no disruptions of the operations should the imported supply be suddenly shut off, as was the case during the First World War.

In searching for another supply of creosote oil, it is found that a creosote may be produced by distilling the tar residue resulting from the cracking of asphaltic-base petroleum oils in which artificial fuel gas is the main product. The plant of the Portland Gas and Coke Company has a potential annual producing capacity of some 4 to 5 million gallons of this oil-tar creosote. It appears that the oil-tar creosote produced at this plant can be made to meet all of the specifications of the Grade I creosote as written by the American Wood Preservers Association (3), with the exception of the clause demanding that the creosote be of coal-tar origin.

Since there are only very small amounts of tar acids and bases present in oil-tar creosote and since the presence of these constituents, as beneficial ingredients in preserving wood, is open to much question, the action of these factors will be discussed at length later in Chapter III of this bulletin.

Likewise, in order to show the relationship of oil-tar creosote to other wood preservatives and in order that the reader may gain a rational viewpoint of wood preservatives in general, the historical developments as well as a resumé of the manufacturing processes of creosote and the theory of preservative action are included.

Since oil-tar creosote, as manufactured by the Portland Gas and Coke Company, has never been placed generally on the market, many comparisons will be made to show its relative value as a wood preservative. The closest approach to a product similar to oil-tar creosote is found in water-gas-tar creosote. As will be shown later, however, the two creosotes are not synonymous.

Considerable effort was made to determine the possible value of oil-tar creosote. In answer to correspondence, Hunt (25) of the Forest Products Laboratory at Madison, Wisconsin, included the following remarks: "We have made no recent studies on tars or creosotes obtained from petroleum used in the production of fuel gas and are not prepared to say to what extent the product you have in mind differs from the water gas tars or creosotes used in tests started years ago. The difference between such oils from different gas plants is probably sufficient, however, to make it unsafe to generalize very much about them. If the oil about which you inquire is available in sufficient volume and uniformity to be of local importance it should be worth while to determine its toxicity and its other properties that bear on its ability to preserve wood satisfactorily. We have for a long time felt that a survey of the local tars and creosotes and their suitability for home treatment would be useful and would enable us to be more specific in our recommendations to the residents of any region, but we have not been able to undertake such a study."

The preliminary studies seemed to justify a more complete and extensive study of the toxicity and related properties of the oil-tar creosote. The methods and results of this portion of the study are contained in this paper under the heading of pathological study. It is the purpose of the pathological study to show the comparative toxicity of coal-tar creosote and oil-tar creosote.

2. **History of wood preservation.** Wood is one of the cheapest and most readily available of all building materials. Under certain conditions of use and climatic conditions it is subject to decay by fungus organisms. It is also subject to attack by certain insects and wood borers. It is true that some woods, such as Western Red Cedar, Redwood, and Black Locust, will naturally resist attack of these wood-destroying organisms because of the toxic-infiltrated materials within the wood cells. Most species, however, do not have these natural toxicants present and to render the wood durable it must be artificially preserved in order to make the use of the nondurable species economically feasible. Based on the service records of the C.B.&Q. Railroad, it has been found that untreated cross-ties have a life of 5½ years, while preserved ties have a life of 27 years (26). It has been shown also that untreated marine piling may not last a year, while creosoted piling may have an average life of 20 to 40 years or more in marine-borer-infested waters.

Treating wood serves a twofold purpose: first, it prolongs the life of the structure which makes the use of wood economically possible, and second, preservation prevents waste and therefore undue drain on the natural forest resources. When wood is used under conditions that favor fungus, insect, and borer attack, preservation is the answer to consumer satisfaction.

With the advance of science it was only natural that the building trades turn to science in an effort to find the answer to wood preservation. As early as 1705 (26) mercuric chloride was used as a wood preserver. Since that time many different types of treatments and many different materials, including organic and inorganic, have been tried. No really satisfactory substitute has been found for creosote oil for any and all types of work.

In the United States, the first treating plant was erected in 1865, employing the Bethel process. In 1902 Reuping obtained a patent for the so-called empty-cell process for treating wood with creosote oil. This method is still widely used. In 1906, Lowry obtained a patent for another method of empty-cell treatment. Although many refinements in the mechanics and techniques have since been developed, the use of the Boulton, Bethel, Lowry, and Reuping processes are still essentially the same today as when they were first employed.

The use of petroleum oil as a wood preservative has been suggested from time to time, but it has been found that petroleum oil in itself is nontoxic. In order to treat many items, such as cross-ties and piling, more economically, petroleum oil is now widely used as a diluent for creosote.

Water-soluble salts of a toxic nature are widely used as wood preservers. Among the best known are zinc chloride, sodium fluoride, and copper sulphate. The salt treatment has some advantages over creosote in that it is possible to paint over the salt-treated wood, and that the weight of the salt in the dried wood is not such an expensive item in transportation costs. For all outside work, however, the salt treatments cannot be recommended as being as effective as creosote because of their susceptibility to leaching with water. The railroads now use zinc chloride only in extremely dry climates such as New Mexico and Arizona.

Toxic chemicals in organic solvents are a comparatively new development in the wood-preservation field. Perhaps the best known of this type of preservative is the Permatol, developed by Dr. Hubert of the Western Pine Association.

3. Requirements of a good preservative.

(a) *Toxicity.* The first and most desirable requirement for a wood preservative is that it must be toxic to the organism that feeds on the wood, such as the common fungi, or to the organisms that may use the wood for a shelter, such as the marine borers, the teredo, and bankia.

(b) *Permanence.* In addition to the preservative being toxic, it must be permanent; that is, not subject to leaching by water, or subject to volatilization. A substance like carbolic acid may be extremely toxic, but since the boiling point of carbolic acid (phenol C_6H_5OH) is $184^\circ C$ (30), it lacks stability as compared to the other fractions of creosote. Mercuric chloride is known to be extremely toxic, but is readily soluble in water so that it cannot be considered as a stable wood preservative.

(c) *Penetration.* In order to be effective, a preservative must have the capacity for deeply penetrating the wood. Penetration is of course commercially effected by using heat to lower the viscosity of the liquid and by pressure to force the preservative into the wood. A preservative that will not penetrate offers little advantage to the wood that may subsequently develop seasoning checks below the penetration line.

(d) *Noncorrosive and nonharmful to the wood.* Preservatives should not be corrosive to metal fastenings, nor should they affect the strength of the wood. A preservative such as corrosive sublimate is somewhat corrosive to metal, and zinc chloride, if not properly handled in the treating process, may affect the strength qualities of the wood (26).

(e) *Safety in handling and use* is a factor in the selection of a preservative because it is desirable to prevent undue loss of the wood and treating equipment from fire as well as to prevent the toxic effects of certain chemicals on the human system. Highly inflammable materials must therefore be avoided and precautions taken in the handling and use of such toxic materials as bichloride of mercury, and lead and arsenic compounds.

(f) *Readily available at low cost.* In order that wood may be treated economically, the preservative must not only be inexpensive but also readily available in sufficient quantity to justify its use.

4. Theory of preservative action. Two factors making creosote a desirable preservative are, first, the toxic or killing action of the creosote, and second, the mechanical action of the oil tending to exclude moisture and oxygen.

As has already been mentioned, the first quality of a good preservative is that it must be toxic to the organism which it is intended to kill. The toxic material must be sufficiently soluble in the body fluids of the attacking organism to kill. The body fluids of these organisms are water soluble, therefore the preservative must be at least partly water soluble. Curtin (14) has shown that the action of fungi produces an acid condition equal to pH_4 . It follows, therefore, that although a preservative may not be sufficiently toxic under neutral conditions, the slightly acid condition caused by the fungus secretions may render the preservative soluble enough to be effective. Bateman (7) concludes in a summary of his work on experiments with the toxicity of hydrocarbons: "It seems likely from the data presented that the hydrocarbons are at least four times as toxic, molecule for molecule, as the corresponding phenols. That is, benzene is more toxic than carbolic acid, naphthalene is more toxic than betanaphthol. Hydrocarbons which of themselves are not toxic enough to inhibit fungus growth may aid to a very considerable extent when combined with other hydrocarbons."

Likewise, a compound which in itself may be only very slightly soluble, such as pentachlorophenol, with a solubility in water of 0.0014 per cent (31, 13) may be so highly toxic that there is sufficient chemical present in solution to produce a lethal dose.

The conclusions reached by Bateman and Benningson on the toxic principles of creosote (11) contain the following remarks:

(1) "The essential toxic material of coal tar creosote may be divided into two groups, viz., the hydrocarbon oils boiling below 270 degrees C, and the tar acids and bases boiling above 270 degrees C.

(2) "The hydrocarbon oils distilling below 270 degrees C are much more toxic than any other class of material in coal tar creosote, and they may be considered the essential toxic material of creosote oil.

(3) "The high boiling tar acids and tar bases may be considered the essential toxic material for high boiling distillates such as carbolineums. In this case, the hydrocarbons, although potentially very toxic, are rendered ineffective by their low solubility, leaving the work to the less toxic, but more soluble constituents."

Excerpts of some of the discussion regarding the conclusions of this paper are given in Section 4 of Chapter III.

In addition to the toxic action of preservatives, there may also be the mechanical action in the preservative, particularly in the oils. Lunge (30) points out that there are appreciable effects from the mechanical point of view, citing the work of Seidenschneur, who found that the neutral oils of creosote were equally as effective against certain organisms as when the tar acids and bases were left in. In other words, the presence of the oil in the wood and around the fibrils in the cell wall prevents the entrance of water, which is of course necessary for fungus growth. It is undoubtedly true that there is a certain amount of sealing action that tends to close the pit apertures and cut down the free flow of water, and also if the cell wall is filled to refusal with oil, there is less likelihood of appreciable amounts of water being present. These facts, coupled with a sufficiently toxic material in the oil, will prohibit the fungus growth. In addition, the flow of the reserve material from the inside of the treated timber to the outside allows a sufficient concentration of the toxic material to be present in the areas most liable to attack. It must be pointed out, however, that the character of the creosote oils changes during periods of service. This change has an effect on the ability of the creosote to act as a satisfactory preservative over a period of time. This is pointed out by Schmitz, von Schrenk, and Kammerer (42) who conclude among other things that, "As repeatedly pointed out previously, changes in the character of the creosotes in treated wood occur during the period of service. These changes are more pronounced in the outer layers of the treated wood and are less extensive in the deeper layers of the treated wood." Also, "The continued protection of the outer layers of the wood, where the toxicity of the creosote is low, appears to be due in part at least, to the movement of the toxic constituents of the creosote in the inner layers to that in the outer layers of wood."

Permanence is related to both the character of the toxic material and to the mechanical action of the preservative. As has already been shown, permanence of the preservative in the wood is a function of its solubility and its volatility. In order to be effective over a long period of time, the preservative must be soluble enough to inhibit or kill the growth of fungi, and yet insoluble enough to prevent excessive leaching. It also logically follows that the preservative should not be volatile under service conditions. From a volatility standpoint, an organic substance such as pentachlorophenol is an excellent preservative. This chemical gives a loss of 0.00003 gram per square inch per hour at 50 C (31). In the case of creosote oil, the boiling point of the various substances has a direct bearing on the volatility and hence the permanence of the

oil. Allerman (1) points out that: "It appears therefore that light oils, boiling below 205 degrees C will not stay in the timber, but that the heavy oils containing a high percentage of anthracene oils will remain almost indefinitely and protect the wood from decay and boring animals." Von Schrenk (46) also points out that the high boiling constituents of creosote have high toxic values. Teesdale (45) studied the volatilization of the different fractions of creosote, and found that after treating loblolly pine sapwood that the fraction up to 205 C lost 34.7 per cent of the original amount of oil after 2 months, fraction II, 205 C to 250 C, lost 21.3 per cent after 2 months, while the original creosote oil lost only 5.4 per cent. It is only logical to believe that the higher the boiling point, the less will be the volatility.

5. Common wood preservatives. A few of the more common wood preservatives, including salts, oils, and chemicals, will be discussed in order to show the general characteristics of each and the limitations of each type in their ability to act as a wood preservative.

(a) *Water-soluble salts.* A number of water-soluble toxic salts are now generally used as wood preservatives. All of the preservatives of this type depend upon a solution of from 4 to 10 per cent in water as the carrying medium. It follows, therefore, that the wood must be partly dry in order that the desired penetrations and absorptions can be obtained. The water may be subsequently dried out, leaving approximately $\frac{1}{2}$ pound of dry salt per cubic foot absorption. A few of the more common salts of this type are as follows:

Zinc chloride. This salt possesses most of the qualifications of a good preservative except that it lacks permanence due to its susceptibility to leaching by water. The toxic or killing point is reported as being 0.35 per cent in agar (38). Zinc chloride has been used more extensively in the United States than in any other country. During and following the First World War many users of creosote had to turn to zinc chloride because of the lack of an adequate supply of creosote from European countries. Chromated zinc chloride is said to be composed of 80 to 82 per cent zinc chloride while the remainder is sodium dichromate (26). It is claimed that the addition of the chromates makes the preservative more resistant to leaching.

Sodium fluoride has many of the same properties as zinc chloride. The toxic point is reported as 0.25 per cent in agar (38). In addition to the tendency of this salt to leach is the disadvantage of having sodium fluoride form an insoluble precipitate with calcium. This fact limits the effectiveness of sodium fluoride in contact with limestone and lime water.

Mercuric chloride. Due to the high toxicity of this salt, reported as 0.005 per cent (9), only a 1 per cent solution is generally used in the treating fluid. This salt has been widely used in Germany. Poles so treated have been reported (26) to give from 14 $\frac{1}{2}$ to 16 $\frac{1}{2}$ years service as compared to 23 years service for poles treated with creosote.

Copper sulphate, with a toxic point of less than 0.065 per cent in agar (9). Copper sulphate, like mercuric chloride, is corrosive to iron and steel and therefore requires special treating apparatus. Also like sodium fluoride, it will react with calcium to form precipitates.

(b) *Preservative oils.* The preservative oils are probably the best and most widely used of all wood preservatives. The better oils fulfill all of the requirements of a good preservative and unlike the soluble salts, the oils are more resistant to leaching. The better-known oils are briefly discussed in the following paragraphs.

Coal-tar creosote, obtained mainly from the heavy oils in the distillation of coal tar, has been set as the standard for the oils used in wood preservation. As will be shown later, creosote oil is a solution containing many individual hydrocarbons, all of which seem to be more or less toxic to wood-destroying organisms, or at least produce conditions that inhibit the growth of such organisms. The toxic point of coal-tar creosote has been variously reported. The main objection to the use of creosote appears to be the odor and the fact that wood treated with creosote cannot be readily painted, though after the wood has been exposed to the air or water for a short time the odor is materially lessened.

Anthracene oils or carbolineums are coal-tar distillates of higher gravity and higher boiling points than ordinary creosote and because of this fact are less toxic than straight creosote. These oils find their greatest use in the open-tank treatments where loss through volatility of ordinary creosote is a factor.

Water-gas-tar creosote is produced by the distillation of the tars formed in the manufacture of water gas. Water-gas-tar creosotes cannot be distinguished with certainty from coal-tar creosotes by any known chemical or physical tests (26). Water-gas-tar creosotes do not contain the tar acids or bases normally found in coal-tar creosotes. Unfortunately, no great amount of work has been done on the constituents of water-gas-tar creosote, and the toxic points of those oils studied have shown a greater percentage of concentration than the ordinary run of creosotes. The temperature at which these tars are formed no doubt has some bearing on their toxic properties.

Coal tar is not widely used as such for a wood preservative. Its toxicity is somewhat lower than coal-tar creosote, and uniform penetrations are hard to obtain. It is used, however, as a diluent for creosote oil and finds considerable use in this manner. Creosote coal-tar solutions are included in the American Wood Preservers Association Specifications (3), but the specifications do not permit the use of more than 20 per cent of coal tar.

Water-gas tar, or solutions are not included in the specifications of the A.W.P.A. except for use on paving blocks.

Petroleum oils as a class are nontoxic. They cannot in themselves be recommended as preservatives although the sealing action of the heavier oils may for a time inhibit the growth of fungi.

Creosote-petroleum mixtures are employed for the treating of ties and other timbers. Bateman (6) has shown that the toxicity of the mixtures is reduced more than in direct proportion to the amount of petroleum added. The main reason for such dilution is largely a matter of cost, although some beneficial results may result because of the lessened tendency of ties to check when the petroleum is added.

(c) *Chemicals in solution.* Although organic chemicals have not been used widely as wood preservatives, considerable work has recently been done on their use for this purpose. Hubert (24) has shown that excellent results may be expected by the use of pentachlorophenol, tetrachlorophenol, and 2-chlororthophenol in oil solutions. Wood (42) has suggested the use of beta-naphthol in petroleum oils. Hunt (27) and Snyder treated posts with dinitrochlorobenzene, dinitronaphthalene, tetrachlorophenol, with petroleum, and placed them in service in the Canal Zone to measure their respective resistance to termite and fungus attack. After 6 years of service, the posts treated with 13.6 lb of grade no. 1 creosote were all sound, whereas the posts treated with 7.1 lb absorption of tetrachlorophenol were 100 per cent destroyed. Also, the posts treated with 30 per cent B-naphthol, 10 per cent pine oil, and 87 per cent petroleum oil were 87.5 per cent destroyed. This test would indicate that neither

the petroleum nor the phenolic compounds were very successful in preventing termite attack. Reports of cresylic acid (14) being added to petroleum oils in the ratio of 5 per cent cresylic acid and 95 per cent petroleum as a solution to preserve railway ties have been made. This preservative, known as Cresoil, has failed to give adequate protection, indicating that the tar-acid content of creosote may not be the protective medium, or if so, the chemical nature of the mixture is not stable. This does not in itself mean that the cresylic acid in another medium closer to its molecular structure, such as the aromatic hydrocarbons, would not be stable in the wood.

III. COMPARISON OF OIL-TAR, COAL-TAR, AND WATER-GAS-TAR CREOSOTES

1. **Coal-tar creosote.** The creosotes obtained from the distillation of tars of different origin have many properties and constituents in common. There is, in fact, no known means of distinguishing, with certainty, whether a creosote may be of coal-tar or of water-gas-tar origin (26). It is also true that oil-tar creosote cannot be distinguished from the other creosotes. Oil-tar creosote can be made to meet all of the requirements of the specifications for coal-tar creosote.

Since the source of the distillate is petroleum, the tar-acid content is extremely low and the specific gravity may fall below the specifications as now written for coal-tar creosote. With the exception that the creosote be of coal-tar origin, the oil-tar creosote can be made to meet all of the specifications for coal-tar creosote.

The specifications for Grade I coal-tar creosote, as adopted by the American Wood Preservers Association (3), The American Railway Engineering Association, and the A.S.T.M., are as follows:

(a) The creosote shall be a distillate of coal tar or coke oven tar. It shall comply with the following requirements:

(b) It shall contain not more than 3 per cent of water.

(c) It shall contain not more than 0.5 per cent of matter insoluble in benzol.

(d) The specific gravity of the creosote at 38 C, as compared with water at 15.5 C, shall be not less than 1.03.

(e) The distillate on a water-free basis shall be within the following limits:

Up to 210 C, not more than 5 per cent.

Up to 235 C, not more than 25 per cent.

(f) The creosote shall be made in accordance with the standard methods of the American Wood Preservers Association.

Relative to the detection of the presence of tar acids, Hunt (26) points out: "This specification does not limit the amount of residue above 355 C, although creosotes containing high residues generally have higher viscosities, lower toxicities, and greater tendency to bleed from the wood than those having moderate or low residues."

Other grades of creosote oil are recognized by the American Wood Preservers Association. Among these are the specifications for creosote for brush or spray treatments, which specify higher gravity and higher boiling points for

the different fractions. This oil, when used in the open-tank treatment, will not volatilize so readily and hence there is not so great a loss as with ordinary creosote. The anthracene oils or carbolineums also have approved specifications. These oils have some of the solids (anthracene and phenanthrene) removed so that they are liquid at ordinary temperatures. These oils are quite generally used for open-tank treatments because the high-boiling, high-gravity oils, as stated above, give low volatility.

2. Water-gas-tar creosote. The American Wood Preservers Association and the American Railway Engineering Association do not recognize any standard specifications for the treatment of ties and structural timbers with water-gas-tar creosote. The American Wood Preservers Association, however, does have a specification for water-gas-tar creosote when mixed with zinc chloride for use in the Card process. This specification is included in this paper because it may justly be argued that oil-tar creosote and water-gas-tar creosote have many properties in common. These specifications are as follows:

(a) The oil shall be a distillate of water-gas tar and shall comply with the following requirements:

(b) It shall contain not more than 3 per cent of water.

(c) It shall contain not more than 0.5 per cent insoluble in benzol.

(d) The specific gravity of the oil at 38 C compared with water at 15.5 C shall not be less than 1.02.

(e) The distillate, based on water-free oil, shall be within the following limits:

Up to 210 C, not more than 5 per cent.

Up to 235 C, not more than 25 per cent.

Up to 355 C, not less than 70 per cent.

(f) The oil shall yield not more than 2 per cent of coke residue.

(g) The foregoing tests shall be made in accordance with the standard methods of the American Wood Preservers Association.

It will be noted that the residue above 355 C cannot exceed 30 per cent, which is an effort to guard against the excessive amounts of high-boiling oils that Bateman (11) points out are likely to be of low toxicity.

Since oil-tar creosote, as such, has never been placed on the market nor has it been used as a wood preservative, no specifications have ever been prepared. It appears likely, however, that the Grade I coal-tar creosote specifications would be suitable for oil-tar creosote except that the specific-gravity specification could be lowered slightly without seriously affecting the quantity of the high-boiling fractions.

3. Comparison of manufacturing processes. In order to point out more clearly the character of the tars resulting from the different manufacturing methods under consideration, a brief resumé of the main manufacturing processes will be included. This inclusion is also considered necessary to point out the difference between the distillates from oil tar and from water-gas tar.

Oil-tar creosote as manufactured by the Portland Gas and Coke Company is a distillate of the oil tars formed during the process of making gas from California fuel oils. The process can perhaps best be followed by referring to Figure 1, which depicts the flow chart of the entire gas plant.

The gas generators are heated until the temperature reaches approximately 1800 F. The heat that passes out of the generator in the exhaust gases is used

in the waste heat boilers. As the generators reach maximum temperature, the air blast is discontinued, the valve leading to the waste heat boilers is closed, and the stack valve is closed. The fuel oil is then sprayed into the generator as the gas "make" starts. The gas "make" proceeds for approximately 18 minutes, when the generator is again heated and the process continued. The gas, upon escaping from the generator, passes through a wash box where lamp-black is deposited, thence passing up through a scrubbing tower having a system of baffles, over which a continuous stream of water is passing. The tar recovered in this process flows out into the tar-separating tanks, where the excess water is removed. From the separating tanks, the tar is pumped into the steam dehydrators where practically all of the water is removed. The tar is then ready for the distillation process, which is carried on in the conventional manner.

The tars obtained in this process, by virtue of the high cracking temperature, are practically all of the aromatic series. No complete analysis of the different individual oils existing in either the creosotes or tars of this origin could be found. It is only logical, however, to believe that the creosote oils are almost as complex as the coal-tar creosote, with the exception of the phenols and bases. Oil-tar creosote can be expected to approach more closely the water-gas-tar creosotes in general composition except that by virtue of the higher cracking temperatures more aromatics and higher-boiling oils will result. Deane and Downs (15) in studies of water-gas tar found that it contained thiophene, benzene, toluene, xylene, mesitylene, naphthalene, and anthracene. In fact, every similarity existed between the oil that they examined and coal-tar creosote, except for the absence of tar acids, bases, and usually lesser amounts of free carbon. Lunge (30) cites a number of references on work that has been done with water-gas tar and some work that has been done on straight oil tars. According to Lunge, the "gas oil" was cracked by passing the vapors through red-hot tubes or retorts, during which process the vapors are decomposed into more volatile bodies. A portion of the oil escapes decomposition and can be trapped in a receiver while another portion is converted into aromatic hydrocarbons. Lunge also reports the studies of Wurth (*Untersuchungen eines Oelgasteers*, Munchen 1904) who found all of the constituents of coal tar, including even phenols and traces of bases, but did not find carbon disulphide or acridine.

Bateman (8) in his discussion of oil tars says: "Like coal tars, these fluids are exceedingly complex mixtures. The character of the hydrocarbons depends greatly upon the temperatures at which the tars were formed. Like the high temperature coal tars, the high temperature oil tars are very complex mixtures of compounds. The hydrocarbons are chiefly of the aromatic series. Benzene, toluene, naphthalene, phenanthrene, and methyl anthracene have been found in them; but so far as is known, no true anthracene has been identified in the American oil tars. They are further characterized by the almost entire absence of tar acids and tar bases, and this seems to constitute the chief difference between this type of tars and high temperature coal tar."

Further work is needed to establish definitely the individual components of California petroleum tars. It is sufficient to say, however, that the oil tars formed during the high cracking temperatures, such as are used at the plant of the Portland Gas and Coke Company, are chiefly aromatic and that the creosote obtained therefrom is sufficiently high boiling to escape any more than average loss through volatilization.

Water-gas-tar creosote is obtained from distilling the tars accumulating from the production of water gas. Water-gas tar may be considered an oil tar;

while the procedure used in producing the gas and tar is somewhat similar, it is yet quite different from the procedure used by the Portland Gas and Coke Company.

The typical gas-producing unit consists of three parts: the generator, the carburetor, and the superheater. The generator, is loaded with coke or coal and the latter heated to incandescence by means of an air blast. The gases generated during this heating of the coal are of course partly burned and pass on through the carburetor and superheater. An additional air supply in the carburetor and superheater allows complete combustion to take place, thoroughly heating these parts, which according to Bateman (8), reach a temperature of 1461 F at the base of the superheater. As soon as the correct temperature has been reached, the escape valve is closed, the gas holder valve is opened. Steam is blown through the coke bed, which results in the formation of carbon monoxide and hydrogen. As the resultant gas enters the carburetor, gas oil is sprayed on the hot brick and "cracked." The cracking continues in the superheater, yielding the gas and suspended tar. The tar is of course removed in the scrubbers and from this tar the distillation proceeds. The tar formed in this process is not chiefly a product of the coal reaction, but from the cracking of the oil. Insufficient cracking, of course, will result in a lower yield of gas and a greater amount of paraffin or asphalt in the tar, according to the source of the gas oil.

In comparing the oil tar of Portland Gas and Coke Company manufacture and standard water-gas tar, it must be pointed out that the former tars are formed at higher temperatures and are not subjected to quite as great a temperature drop during the "gas make."

Coal-tar creosote, as defined by Lunge (3), "is composed of distillates from coal tar that are midway between carbolic oil and anthracene oil. These are, in the first place, the fraction of coal tar distilling directly between 240 and 270 C and besides, from either side, the residue from the manufacture of carbolic acid, naphthalene, and anthracene."

The tar, of course, is obtained from the destructive distillation of coal in the following general manner:

| | | |
|-----------|------------------|-------|
| | <i>Coal</i> | |
| | : | |
| Gas | <i>Tar</i> | Coke |
| | : | |
| Light Oil | <i>Heavy Oil</i> | Pitch |
| | : | |
| | : | |
| | <i>Creosote</i> | |

The tars that are formed during the carbonization of the coal vary over quite wide limits in their properties, depending on the type of retort, temperature applied, and the original coal. For this reason, it may be well to briefly consider the main types of ovens and retorts used together with the temperature applied and resultant properties of the tars.

There are three general methods employed in the production of coal gas, tar, and coke. These are described in detail by Lunge (30) and more briefly by Bateman (8). The horizontal retort has the coal chamber lying in a horizontal plane. The coal chamber is usually about 18 inches wide by 15 inches high by 6 to 18 feet long. The retorts may be heated by direct heat or by gas to the required temperature until complete carbonization has taken place. The

vertical retort employs a vertical coal chamber so that the coal may be fed and the coke removed by gravity. The small retort is then heated by adjacent vertical flues and continued at the desired temperature until carbonization is complete. The inclined retort is an adaptation of this same method. The coke oven, of which there are several types, is widely employed in the United States. The ovens are somewhat long, and rectangular in shape, being from 3 to 9 feet high by 17 to 19 inches wide and up to 35 feet long. The coal is heated by means of flues adjacent to the oven while the gas passes upward into the gas main.

Fisher (16) shows, by means of a table prepared by H. M. Spiers, not only the properties of various tars when produced in different ovens and retorts, but also the properties of low-temperature tars. In general, this table shows that the specific gravity of the tar is the highest in the horizontal retorts, closely followed by the coke ovens; considerably lower in the vertical retorts; and the lowest gravity was found in the low-temperature tars. This same general order prevailed in the relationship of the percentages of the higher-boiling fractions of the distillates; that is, the horizontal retorts gave the greater percentage of higher-boiling distillate, etc., in the order named above. The reverse order held for the percentage of crude tar acids. Fisher also shows by graphic representation the effect of carbonizing temperature of Pratt coal on the yield of the various hydrocarbons. In the range of 932 F to 2012 F, the yield of aromatics increased in about the same ratio as the paraffin decreased with temperature rise. In the unwashed coal, the aromatics yielded about 47 per cent of the distillate at 932 F to a maximum of 84.5 per cent of the distillate at 1832 F. The olefin content changed only slightly. The tar-acid content decreased from 17 per cent at 932 F to about 2.3 per cent at 2012 F. This shows quite clearly the effect of temperature on the composition of the tars, which also holds true, in general, for the effect of temperature on the constituents of oil tars. It also shows quite clearly that the creosotes produced from the various types of tars are very likely to possess quite varying characteristics and quite different compounds. A part of this variation is of course overcome by means of the A.W.P.A. specifications.

4. The controversial point of tar acids and tar bases. It has long been felt that the tar acids in creosote oil were not the essential toxic material. Authorities have disagreed upon this subject, but as will be seen from an examination of the following extractions from the literature, tar acids are not, in the main, thought to be responsible for the toxic action of creosote oils.

The term "tar acids" is applied to those constituents of tar that are oxygenized. These oxygen-containing compounds may further be divided into the acidic compounds and the neutral compounds. Fisher lists 23 separate acidic compounds and 12 neutral compounds. It is the oxygen-containing compounds in the acidic group that are frequently held to be the more effective toxic element in coal-tar creosote. Chief of these compounds are as follows (30):

| <i>Compound</i> | <i>CHO</i> | <i>Boiling point Deg C</i> |
|---------------------|------------|--------------------------------|
| Phenol | 6- 6-1 | 181 |
| o-Cresol | 7- 8-1 | 191 |
| m-Cresol | 7- 8-1 | 202.8 |
| p-Cresol | 7- 8-1 | 201.8 |
| 1-2-3-Xylenol | 8-10-1 | 214 |
| 1-2-4-Xylenol | 8-10-1 | 225 |

| <i>Compound</i> | <i>CHO</i> | <i>Boiling point Deg C</i> |
|---------------------|------------|--------------------------------|
| 1-3-2-Xylenol | 8-10-1 | 212 |
| 1-3-4-Xylenol | 8-10-1 | 209 |
| 1-3-5 Xylenol | 8-10-1 | 219 |
| 1-4-2-Xylenol | 8-10-1 | 209 |
| a-Naphthol | 10- 8-1 | 280 |
| b-Naphthol | 10- 8-1 | 286 |
| c-Naphthol | 14-10-1 | 395-396 |

The term "tar bases" is applied to those constituents of tar that are nitrogenized. These nitrogen compounds, like the tar acids, may be further divided into the true bases or ammonia derivatives and the neutral nitrogen compounds. The percentage of tar bases in crude tar is relatively low (30), and for that reason, not so much toxic benefit is ascribed to the tar bases as to the tar acids. Fisher (16) lists 48 separate ammonia derivatives and 4 neutral compounds found in coal tar. The following is a list of the more generally accepted compounds found in creosote:

| <i>Compound</i> | <i>CHN</i> | <i>Boiling point Deg C</i> |
|-----------------|------------|--------------------------------|
| Pyridene | 5-7-1 | 115 (Other pyridenes at |
| Quinoline | 9-7-1 | 238 higher boiling points) |
| Acridine | 13-9-1 | 346 |

It is known that some of these bases are toxic to bacteria and fungi, but the specifications for coal-tar creosote make no reference as to the necessity of tar bases being present in order to qualify for any grade of creosote. In this paper, therefore, no further discussion of tar bases is necessary.

It has already been shown that the tar-acid content of coal tar is largely dependent on the carbonization temperature and to a lesser degree on the type of oven and the coal used. Since the normal coke oven operates at about 1000 C, most of the tars will be expected to yield around 4 or 5 per cent of tar acids. Unless the tar acids are extracted from the tar to be used for by-products (which is a usual procedure), the tar-acid content of the creosote might be expected to yield considerable quantities of tar acids. Commercial coal-tar creosote, however, can be expected to yield around 5 per cent tar acids.

The tar acids in creosote have been the subject of many investigations and discussions, particularly concerning their value as toxic agents and their permanence in the wood. The early workers ascribed the value of tar acids to their ability to coagulate albumen and hence prevent decay. This, of course, was exploded after the era of scientific investigations by such men as Lister. Since about 1885 suspicion has been directed toward the beneficial effects of tar acids in creosote to be used in wood preservation. It is the object of this portion of the paper to point out some of the opinions and findings of those who have directed their energy toward finding the truth concerning this matter. The following extractions are therefore cited for this purpose:

Larkin (29), comments on the work of Bateman and Henningson (11), "The toxic principles of Creosote," by referring to the work of Coisene, taken from "Preservation of Timbers by Use of Antiseptics" (1885) by Samuel Boulton. Coisene used 5 samples as follows: (1) one with 15 per cent tar acids; (2) another with 15 per cent tar acids; (3) one with 8 per cent tar

acids; (4) one with 4 per cent tar acids; and (5) a specially prepared oil with no tar acids. The last sample of oil produced better results than any of the other oils. He also concludes that tar acids are volatile and very soluble in water. Also, Boulton's experiments on pieces of ties in service from 16 to 32 years, when analyzed, showed (1) no tar acids detected by ordinary means; (2) in 14 of 17 samples, the semisolid constituents of the tar oils were present, in 12 of them naphthalene; and (3) only small percentages remained of oils distilling below 450 F. In a majority of cases, from 60 to 75 per cent of the total bulk of the substances retained in the wood did not distill until after a temperature of 600 F had been reached. "It is clear, therefore, that those solid timbers had been preserved by the action of the heaviest and most solid portion of the tar oils, and that the other constituents had disappeared." (Hartman, E. F., A.W.P.A. Proc. 1923, page 100, also refers to this same work of M. Coisene.)

Hartman, E. F., (19) referring to the work of F. Seidenschnur, Stendal, Germany in 1909: "Anthracene oil consisting almost entirely of neutral and high boiling oils has a greater antiseptic strength than creosote oil containing considerable quantities of tar acids." Also referring to extract from Federal Specifications Board: "Yet the highest boiling fractions which are the least poisonous stand up best in actual service because they resist evaporation and leaching."

Allerman, Dr. Gallert, "Quantity and Character of Creosote in Well Preserved Timbers" (1) says "It is worth noting that these long lived American piles contained more anthracene oils than naphthalene. Perhaps the most striking thing is the disappearance of the tar acids. It is certainly conservative to place the original tar acids at 5 per cent, yet the extracted oils show but a tenth of this amount. It is possible that these compounds on account of their hydroxyl groups have been exposed to varying amounts of water and air, to the reactive lignin portion of the wood and to the numerous compounds present in creosote. On the other hand, these phenol bodies may have been volatilized or been washed from the timbers.

"It appears, therefore, that light oils, boiling below 205 C will not remain in the timber, but the heavy oils, containing a high percentage of anthracene oil will remain almost indefinitely and protect the wood from decay and boring animals. . . . The value of tar acids has apparently been overestimated by many persons, for although it has not been proved they are valueless, they have been shown to possess poor staving qualities."

Von Schrenk, Herman: "Significance of Toxicity Determinations from a Practical Standpoint" (46): (1) Points out that American creosote practice has been built on European practice. (2) Quotes from Purgeson's paper in Boulton's, "A Century of Wood Preservation." "Still expressing the viewpoint of most of us, Mr. Boulton calls attention to the numerous specifications for creosote differing widely, each from the other. Today, thanks to the work of individuals and societies, these are rendered to a few in number. The fads and theories of earlier years have also given place to sound common sense views. Today, neither naphthalene, nor the tar acid group, finds itself exalted to the most essential constituent. The heavier oils are preferred, but it is understood that to obtain at a reasonable price a commercial article which is the by-product of another industry, considerable latitude in constituents must be allowed. Good results have been obtained with creosotes differing widely in their composition." (3) States that high-boiling constituents of creosote have comparatively high toxic values.

Lunge, George, in "Coal Tar and Ammonia" (30) states that Seidenschneur (Z. Angew. Chem., 1901, p. 437) made numerous bacteriological investigations upon the efficiency of coal-tar oil from which the acid constituents (the phenols) had been removed, for preserving wood, comparing its action with that of zinc chloride. His experiments, carried out with the employment of *Penicillium glaucum* and *Mucor mucedo*, showed that the destroying action of tar oils on these organisms has no connection whatsoever with their content of phenols. It is indifferent whether the oils contain much or little or nothing at all of these "tar acids." The action of coal tar, deprived of the acids, on those fungi was three times as strong as that of zinc chloride. Later on Seidenschneur (Chem. Zeit., 1909, No. 77) impregnated wood sleepers with progressive quantities of acid-free tar oils and exposed them to the wood-destroying fungi, to dry-rot fungus (*Merulius lacrymans*), and to *Polyporus vaporarius*. The oil was emulsified by rosin soda soap, the emulsion containing the equivalent of 6 per cent tar oil. The experiments showed that the application of 0.8 kg of tar oil per sleeper sufficed for protecting the sleeper, so that the impregnation of the sleepers on the large scale, when they take up 7 kg of oil effects a ninefold security. From impregnated sleepers that had been in the railway track for 16 years and showed a trace of deterioration by rot, an oil was extracted which consisted almost entirely of high-boiling hydrocarbons, containing neither phenols, nor bases, nor low-boiling hydrocarbons.

Reeves, Charles S., A.W.P.A. Proc. 1928, pages 42-50 (35), "The Determination of the Toxicity of Wood Preservatives." This work was conducted on wood flour and gives the toxic points of various preservatives, and graphs of growth over fractions of different oils. In discussing the results, Reeves says: "The most toxic material seems to be concentrated in the oils (coal tar distillates) boiling between 280 and 320 C. It is of particular interest to note the relative increase in toxicity of oil No. 1 after extraction of acids and bases (known as oil No. 2) which is a clear demonstration of the high preservative value of neutral hydrocarbons."

Forest Products Laboratory Report, A.W.P.A. Proc. 1914, page 216 (17), deals with specially prepared oils that were used for observation on attack of marine borers. The specimens of piling were treated with fractions of creosote by re-distilling a good grade of coal-tar creosote as follows:

| | | |
|--------------------|-------------|------------------|
| Fraction I | 0-205 C | Tar-acid oils |
| Fraction II | 205-250 C | Naphthalene oils |
| Fraction III | 250-295 C | Dead oil |
| Fraction IV | 295-320 C | Anthracene oil |
| Fraction V | Above 320 C | Residue |

In addition, a coal-tar creosote and a water-gas-tar creosote were used. Piles were placed in service March 1914, and examined January 1916, with the following results:

| | |
|-------------------------|------------------------------|
| Fraction I | Very severe attack by teredo |
| Fraction II | Very severe attack by teredo |
| Fraction III | Medium attack by teredo |
| Fraction IV | Slight attack by teredo |
| Fraction V | Practically sound |
| Water-gas tar | Slight attack by teredo |
| Coal-tar creosote | Practically sound |

Rhodes, R. N., and Gardner, P. T.: "Removal of Tar Acids and Bases and Toxicity," Ind. and Eng. Chem. 22: 167, 1930 (39). "The neutral hydrocarbons were found to be fully as effective as the phenolic compounds of the same distillation range, while the tar bases were found to be only comparatively slightly toxic. The authors believe that the desirable effects of the presence of tar acids in creosote oil are not due to the high fungicidal power of the tar acids, themselves."

Schmitz, Henry, and Buckman, Stanley: "Toxic Action of Coal Tar Creosote with Special Reference to the Existence of a Barren Non-toxic Oil." Ind. and Eng. Chem., Vol. 24, page 772, July 1932 (41). The authors, citing the diversity of opinion regarding the presence of a barren or nontoxic oil, notably the opinions of Bateman, Nowotny, Moll, and Dehnst, directed the study toward the establishment of the presence of a nontoxic oil in coal-tar creosote. The summarization of their work is cited: "In coal tar creosote, there are substances varying greatly in their toxicity to wood-destroying fungi. Although there may be certain substances in coal tar creosote which are essentially non-toxic, the presence of large amounts of non-toxic substances has yet to be demonstrated."

"So-called barren oil cannot properly be considered as non-toxic to wood destroying fungi. Although high concentrations of barren oil do not completely inhibit their growth, even relatively small amounts exert marked toxic effects."

The method in this study was to separate the original oil into four fractions, from which eight other preparations were made. Only one of these will be discussed here in order to show the general trend.

"Attention has already been called to the high toxicity of the fraction distilling below 285 C. Washing this fraction alternately for 3 hour periods with 30 per cent sulfuric acid and a 15 per cent solution of sodium hydroxide, and removing a white crystalline material which formed on cooling, did not greatly change its toxicity."

In the same paper, the authors point to the work of Charitschhoff (J. Russ. Phys. Chem. Soc., 44: 345-8, 1912) who "showed that although the phenol and nitrogenous compounds occurring in coal tar creosote by themselves are quite toxic, their presence in creosote only slightly increases the antiseptic powers of the latter."

Also in the same paper is reference to the work of Dehnst, Z. Angew., Chem. 41, 355-8, 1928: "Suffice it to say that Dehnst concludes the toxicity of coal-tar creosote towards *Coniophora cerebella* is not greatly changed by the removal of the tar acids, tar bases, naphthalene, raw anthracene, the oils boiling below 285 C, and the water soluble products."

Schmitz, Von Schrenk, and Kammerer (42): In their studies of the quality and toxicity of coal-tar-creosote oil extracted from red-oak ties after long periods of service, found that in the creosote extracted from the various zones of the ties examined: "No consistent differences were apparent in the tar acid content of the extracted creosotes. In many cases, the tar acid content of the creosotes extracted from the outer one-half inch of the tie was as high or higher than that of the creosote extracted from zones 2 and 3." (This amounted to from 0.5 per cent to 1.3 per cent.) Yet the authors point out that, "One of the most interesting facts brought out by the study of the toxicity of the extracted creosotes is the comparatively low toxicity of the creosotes extracted from the outer one-half inch of both ties." *In this case there seems to be little correlation between tar acids and toxicity.*

Pooler, F. S., and Howell, Dr. A. M., and Hunt, G. M.: Discussion from floor A.W.P.A. Proc. 1925, p. 108 (34): Regarding ties treated with an oil

of petroleum-gas-house origin, indicating a long life from 1911 to 1925—no ties removed from the track.

The above references of the work of some of the better-known workers in the field would seem to justify the following conclusions:

(a) The presence of tar acids in creosote oils may not necessarily be an index of the toxicity.

(b) The phenols found in coal-tar creosote, although initially toxic, do not tend to stay in place after the wood is put into service over long periods of time. No doubt this is because their solubility in water is generally high and the boiling point somewhat low.

(c) The removal of tar acids and bases from coal-tar creosote does not appreciably affect its toxicity.

(d) Creosote extractions, after long periods of service, and in spite of their tar-acid content, are likely to show comparatively low toxic qualities.

Previous discussion has shown that the characteristics of tars are dependent on several factors. It has been shown that the aliphatic or saturated hydrocarbons found in petroleum oils can be changed to the aromatic or unsaturated hydrocarbons by high-temperature cracking. In other words, the nontoxic hydrocarbons can be changed to toxic hydrocarbons by subjection to high temperatures. In the case of coal-tar creosote, the yield of tar acids decreases with the yield of aromatic hydrocarbons. Thus it is shown that the presence of small amounts of tar acids may indicate larger amounts of the more toxic aromatic hydrocarbons.

5. Additions of petroleum oil and mineral solvents. Petroleum oil and many of its derivatives are not toxic to fungi and wood borers when used alone. For many years (26) petroleum diluents for creosote oil have been used by railroads for treating ties and other purposes. The use of these diluents has been mainly for the purpose of reducing the cost of the treating fluid. The use of a 50-50 mixture is quite a common solution for treating ties and of course when 50 per cent fuel oil is added, the cost is reduced materially.

The additions of petroleum oils will also reduce the toxicity because they themselves are not toxic. Schmitz (40) concludes that with the samples of creosote tested, a 50-50 mixture will reduce the toxicity by one-seventh and a 25-75 mixture will reduce the toxicity by one twenty-fifth, etc. Due to the higher viscosity of the petroleum-creosote mixtures, somewhat higher temperatures are necessary to obtain desirable penetrations. It has been found, however, that the mixture has a tendency to reduce checking of the treated material because the surface of the wood remains in an oily condition and prevents rapid changes in the surface moisture content.

IV. PHYSICAL PROPERTIES OF EACH OF THE CREOSOTES TESTED

1. Properties to be compared. In order that a common ground might be had for evaluating the ability of oil-tar creosote to act as a wood preservative, some of the physical properties of this oil will be compared to a standard Grade I coal-tar creosote. These properties include the specific gravity, distillation, viscosity, penetration, absorption, volatilization by heat, leaching by water, and the flash point.

As Bateman (8) points out, "The similarity of water gas tar creosote and coal tar creosote makes it seem very probable that in general the hydro-

carbons found in the highly aromatic water gas tars are the same as those found in coal tars. Benzol, toluol, xylol, naphthalene, phenanthrene, and methyl anthracene have been identified. The most notable difference between coal tar creosotes and water gas tar creosotes is the almost complete absence of tar acids and tar bases in the latter and their presence in considerable amounts in the former. Because of the lack of these materials, the odor of water gas tar creosotes is more oily than the odor of coal tar creosotes.

"The chemical properties of water gas tar creosotes are in general the same as those of coal tar creosotes from which the tar acids and tar bases have been removed. Only a very small proportion is reacted upon by caustic soda or dilute mineral acids. Concentrated sulphuric acid forms many sulphonic acids which are identical with the sulphonic acids produced from coal tar creosotes.

"Because of the great similarity between water gas tar creosotes and coal tar creosotes, the physical properties of one material would in general be the same as those of the other. The same solvents can be used for both."

2. **Color.** Oil-tar creosote resembles water-gas-tar creosote, but as has already been shown, there is more likelihood of a greater percentage of aromatic hydrocarbons in the oil-tar creosote because of the higher cracking temperature. The oil-tar creosote was found to be a greenish-brown color, remarkably free of any sludge or precipitates. The color imparted to ponderosa pine sapwood blocks was decidedly an oily green.

The coal-tar sample used was found to be a very blackish brown. Considerable amounts of suspended material, which in the solution gave the appearance of free carbon, were found to be present. The color imparted to ponderosa pine sapwood blocks was almost coal black.

3. **Odor.** Both creosotes carried the familiar tar odor. No great difference in the character of the odor could be determined. If anything, the oil-tar creosote had a more penetrating aromatic odor than the coal-tar creosote.

4. **Specific gravity.** Specific-gravity determinations were made (18) of the two creosotes at different temperatures. Results are shown in the following table and accompanying graph.

| <i>Oil-tar creosote</i> | | <i>Coal-tar creosote</i> | |
|-------------------------|-------------------------|--------------------------|-------------------------|
| <i>Temp. C</i> | <i>Specific gravity</i> | <i>Temp. C</i> | <i>Specific gravity</i> |
| 14.8 | 1.037 | 19.5 | 1.077 |
| 23.0 | 1.032 | 29.0 | 1.070 |
| 33.0 | 1.024 | 38.0 | 1.064 |
| 42.0 | 1.018 | 47.0 | 1.057 |

Temperature-specific gravity relationships are plotted on the following graph and are essentially straight lines over the ordinary range of temperatures. It will be noted that the specific gravity of the oil-tar creosote at 38 C is approximately 1.02, slightly lower than the A.W.P.A. specifications, which call for a specific gravity of not less than 1.03, or a difference of 0.01. This lower gravity may be compensated for in part by the nature of the percentage of the higher-boiling fractions in the distillate.

5. **Distillations.** The following distillations made by two different laboratories according to standard procedure agree fairly closely in the results. As will be noted upon examination of the graph, the cumulative distillation up to 270 C is approximately the same for both samples of creosote. In the upper ranges of distillation the oil-tar creosote contains a greater amount of material of high molecular weight.

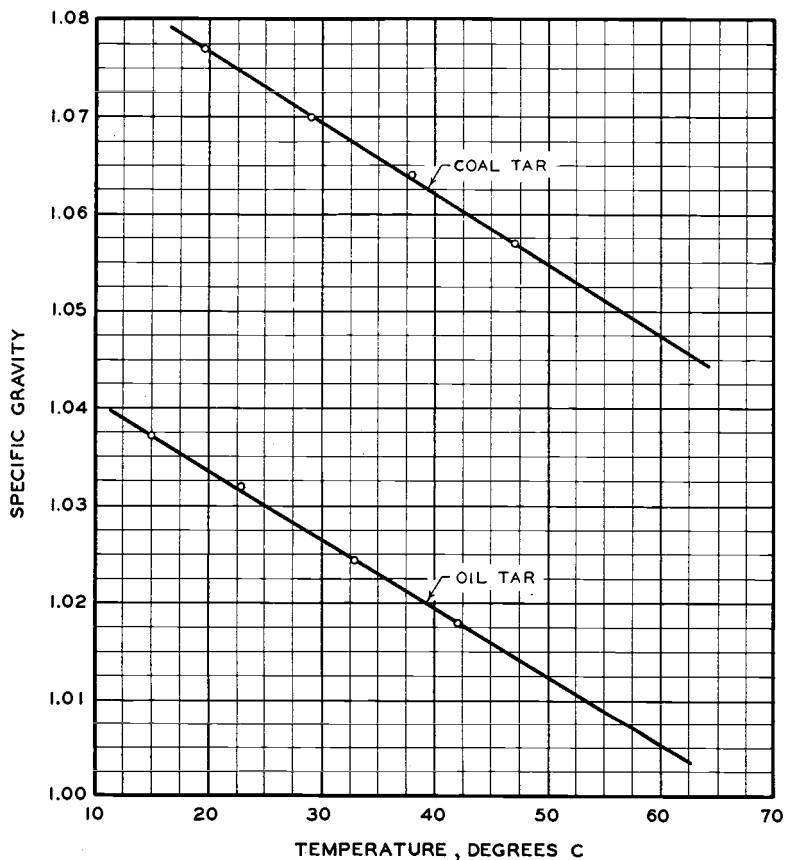


Figure 2. CHANGE IN SPECIFIC GRAVITY WITH TEMPERATURE OF CREOSOTE.

Distillations of the sample of coal-tar creosote supplied by the Pope and Talbot Lumber Company and the oil-tar creosote supplied by the Portland Gas and Coke Company were made by the Department of Chemical Engineering of the Oregon State College (18). The results follow:

Coal-Tar Creosote

| Fraction No. | Temperature C | Per cent by weight over | Tar acids per cent | Cumulative |
|--------------|-------------------|-------------------------|--------------------|------------|
| 1 | 0-210 | 6.69 | 0.094 | 6.69 |
| 2 | 210-235 | 6.17 | 0.083 | 12.86 |
| 3 | 235-270 | 26.17 | 0.521 | 39.03 |
| 4 | 270-315 | 23.82 | 0.417 | 62.85 |
| 5 | 315-355 | 23.00 | 0.729 | 85.85 |
| 6 | Residue above 355 | 14.17 | 0.000 | 100.02 |
| | | | 1.844 | |

Gasco-Tar Creosote

| Fraction No. | Temperature C | Per cent by weight over | Tar acids per cent | Cumulative |
|--------------|-------------------|-------------------------|--------------------|------------|
| 1 | 0-210 | 4.77 | 0.0 | 4.77 |
| 2 | 210-235 | 10.16 | 0.0 | 19.93 |
| 3 | 235-270 | 21.96 | 0.103 | 36.89 |
| 4 | 270-315 | 17.38 | 0.157 | 54.27 |
| 5 | 315-355 | 20.98 | 0.0 | 75.25 |
| 6 | Residue above 355 | 24.30 | 0.107 | 99.55 |
| | | | | 0.367 |

"Since the tar acid determinations upon the small fractions are not too accurate, it was decided to make determinations on the samples themselves without distillation. The coal tar creosote gave 3.62 and the Gasco tar creosote

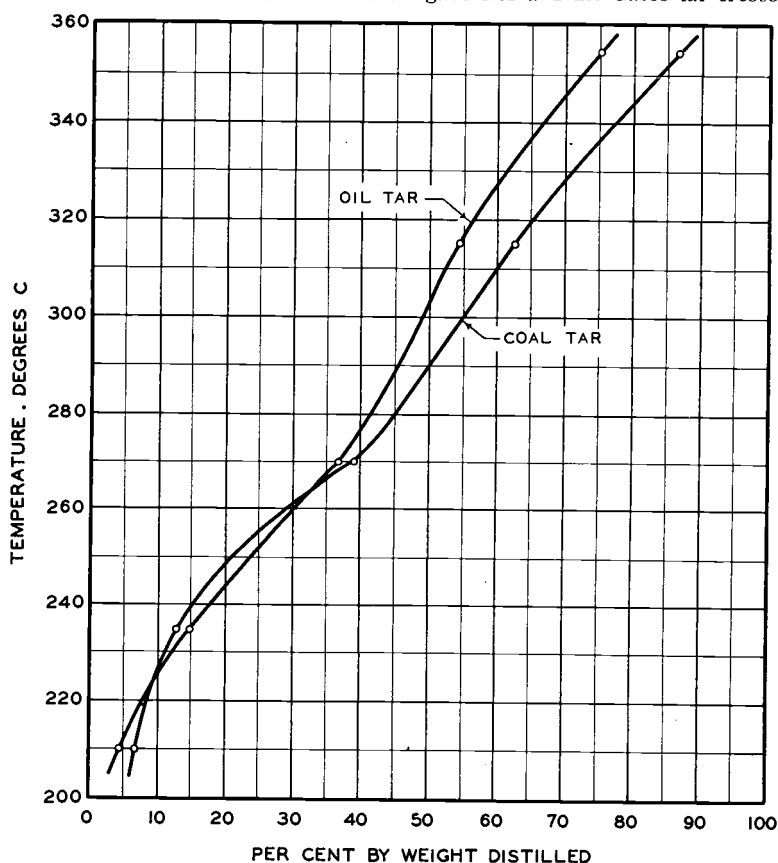


Figure 3. DISTILLATION CURVES FOR COAL-TAR AND OIL-TAR CREOSOTES.

DETERMINATIONS ARE MADE ON WEIGHT BASIS; DIFFERENCE IN CURVES ON VOLUME BASIS WOULD BE VERY SLIGHT.

0.36 per cent by weight of tar acids. Note that the determinations on Gasco checked the total of the fractions while the determination on the coal tar creosote was appreciably higher. This is due to the fact that the coal tar material tends to form an emulsion when treated without distillation and the residual tar oils which do not satisfactorily separate build up volume which appears as tar acid. I believe the value of 1.84 per cent to be representative since the Gasco material checked so closely" (18).

The following distillations were made in the laboratory of the Portland Gas and Coke Company (43). In general they check rather closely with those made at the State College.

| | <i>Oil-tar creosote</i> | <i>Coal-tar creosote</i> | <i>A.W.P.A. specifications</i> |
|----------------------------------|-----------------------------|------------------------------|------------------------------------|
| Water, per cent by volume | 0.1% | 2.6% | Not over 3% |
| Matter insoluble in benzol | 0.01% | 0.3% | Not over 0.5% |
| Specific gravity, 38/15.5 | 1.019 | 1.062 | Not less than 1.3 |

Distillation:

| | <i>Percentages by weight</i> | | |
|--|------------------------------|------|--------------|
| up to 210 C | 4.5 | 1.7 | Not over 5% |
| to 235 C | 15.1 | 8.7 | Not over 25% |
| to 270 C | 37.3 | 36.6 | |
| to 315 C | 52.4 | 60.8 | |
| to 355 C | 72.7 | 81.5 | |
| Residue | 26.2 | 17.0 | |
| Loss | 1.1 | | |
| Coke residue, per cent of original | 0.11 | 1.76 | Not over 2% |
| Tar acids, per cent by volume | None | 4.7 | None |

6. **Volatility—wood-block tests.** In order to determine the relative resistance of the preservative to volatilization after being injected into wood, and to determine the effect of this volatilization on fungus growth, four wood blocks, prepared as described in Chapter V, were prepared with each of the following preservatives:

1. Coal-tar creosote—100%
2. Oil-tar creosote— 100%
3. Untreated control

Each set of blocks was placed in the oven and held at a temperature of 160 F for 24 hours, at which time they were removed and weighed. They were again placed in the oven and left for another 24 hours at the same temperature, removed, and weighed. The per cent loss in weight was then computed on the basis of the original weight in order to show the comparative volatility of the preservatives under examination. The temperature of 160 F was used for this test on the basis described by Hubert (24) for the maximum temperature likely to be found as a direct result of exposure to the sun's rays.

Examination of the following table and the curves in Figure 4 shows that the oil-tar creosote as used in these tests is of about the same volatility as the sample of coal-tar creosote tested.

The blocks thus treated were later used in Kolle flask tests to determine the effect of volatility of toxicity. No attack by the fungus was noted.

| Sample | Preservative taken up, grams | Wt of Pres lost after 24 hr | Per cent lost in 24 hr | Wt of Pres lost after 48 hr | Per cent lost in 48 hr |
|-------------------------|------------------------------------|-----------------------------------|---------------------------|-----------------------------------|------------------------------|
| Oil-tar creosote | 2.900 | 1.243 | 42.9 | 1.625 | 56.0 |
| | 2.337 | 0.893 | 38.2 | 1.142 | 48.9 |
| | 2.680 | 1.104 | 41.2 | 1.404 | 52.4 |
| | 2.977 | 1.355 | 45.5 | 1.575 | 52.9 |
| Avg | 2.723 | 1.149 | 42.2 | 1.438 | 52.8 |
| Coal-tar creosote | 2.986 | 1.361 | 45.5 | 1.581 | 53.0 |
| | 3.191 | 1.295 | 40.6 | 1.490 | 46.6 |
| | 3.348 | 1.214 | 36.3 | 1.772 | 52.9 |
| | 2.697 | 1.068 | 39.6 | 1.568 | 58.2 |
| Avg | 3.073 | 1.234 | 40.7 | 1.603 | 52.2 |

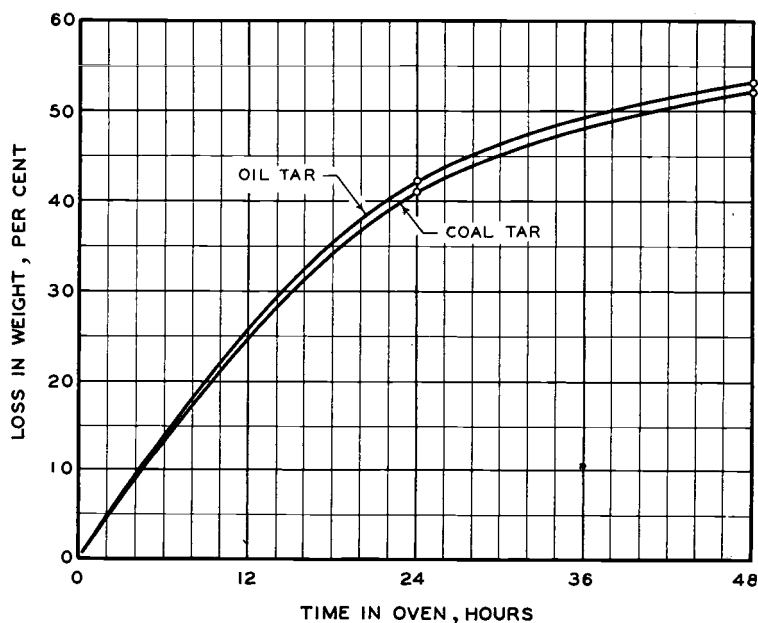


Figure 4. PRESERVATIVE LOST FROM WOOD BLOCKS WHEN EXPOSED TO TEMPERATURE OF 160 F.

Preceding table shows the result of volatility tests on wood blocks preserved in the usual fashion and placed in an oven at 160 F for 48 hours. Weights were taken at the end of the 24- and 48-hour periods. It was assumed that the moisture in the wood blocks would be almost entirely evaporated after 24 hours at 160 F. The calculated oven-dry weights were therefore used as the basis for computing the percentages expressed.

In order to further evaluate the two samples in respect to their resistance to volatilization and to check the results of the wood-block tests of volatility, watch-glass tests were made according to the following procedure:

Twenty cubic centimeters of the preservative fluid, as nearly as could be measured with a burette, calibrated in tenths of a cubic centimeter, were measured out into open glass dishes that had previously been carefully weighed. The weight of the fluid and container was then taken and recorded. The four dishes were then placed in a drying oven at 160 F and left for 24 hours. At the end of the 24-hour period the dishes were weighed and replaced in the oven. At the end of another 24 hours the weights were again taken on each of the four samples and the percentages computed. The results are shown in the following table and in Figure 5:

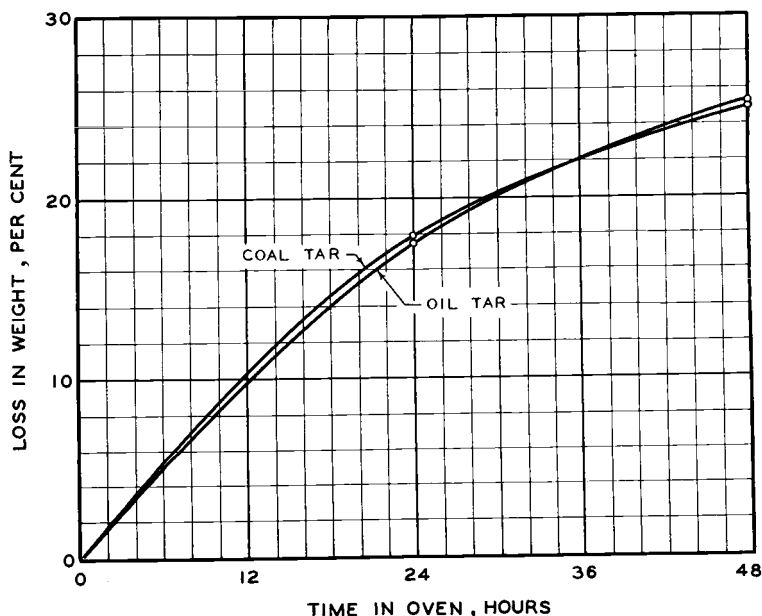


Figure 5. CREOSOTE LOST FROM OPEN DISH WHEN EXPOSED TO TEMPERATURE OF 160 F.

| <i>Wt of fluid in grams before placing in oven</i> | <i>Wt of fluid in grams at end of 24 hr</i> | <i>Per cent loss</i> | <i>Wt of fluid in grams at end of 48 hr</i> | <i>Per cent loss</i> |
|--|---|----------------------|---|----------------------|
| Oil-tar creosote 20.569 | 16.97 | 17.50 | 15.37 | 25.3 |
| Coal-tar creosote 21.20 | 17.40 | 17.92 | 15.90 | 25.0 |

7. Leaching tests with water. Leaching tests were made under conditions similar to those used by Hubert (24). Ponderosa pine sapwood blocks were cut to a size of $\frac{1}{4} \times 1\frac{1}{2} \times 2$ inches so that the blocks could be subsequently used in Kolle flask tests. The blocks were conditioned in a desiccator over a saturated neutral salt solution until they had reached an equilibrium of 12.24 per cent moisture content. Six blocks were selected, weighed, and then treated in the way described under Pathological Study in Chapter V with the following:

| <i>Number of blocks</i> | <i>Preservative</i> |
|-------------------------|-------------------------|
| 2 | Coal-tar creosote, 100% |
| 2 | Oil-tar creosote, 100% |
| 2 | Untreated controls |

As soon as the excess preservative had been removed by placing the treated blocks on clean blotting paper, the weight of each block was again taken and recorded. The blocks treated with the same preservative were then placed together in quart jars, and clean tap water was run into the jars at the rate of 20 changes per hour. The blocks remained submerged in the water near the top of the jar and were held in that position by the hose that supplied the water. All blocks were subjected to this method of leaching for a period of

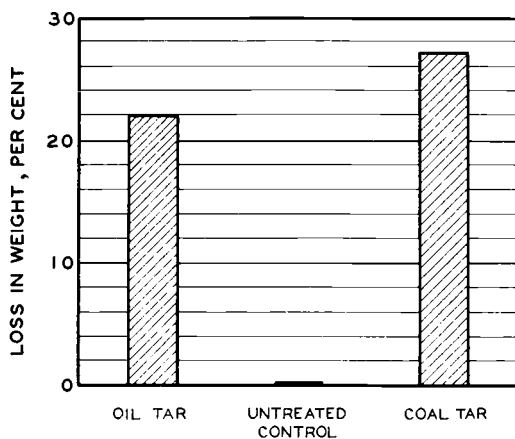


Figure 6. CREOSOTE LOST DURING LEACHING OF BLOCKS WITH WATER.

14 days except that on the fourth, sixth, eighth, eleventh, and thirteenth day the blocks were removed and placed in the atmosphere of the laboratory and left to stand for a period of 8 hours, after which they were returned to the water-leaching process. It was thought that in this way the alternate wetting and drying would more nearly approximate service conditions.

At the end of the leaching process the blocks were dried at room temperature for 48 hours and replaced in the desiccator until they had reached an equilibrium moisture content. This was verified by the weight of the untreated control blocks. After reaching equilibrium the blocks were again weighed and the amounts and percentages of the preservative lost during the leaching process were found to be as shown in the following table.

| <i>Preservative</i> | <i>Original Wt of preservative</i> | <i>Wt of preservative lost</i> | <i>Per cent lost</i> | <i>Avg per cent lost</i> |
|-------------------------|------------------------------------|--------------------------------|----------------------|--------------------------|
| Oil-tar creosote | 3.020 | 0.845 | 27.98 | |
| | 3.060 | 0.488 | 15.94 | 21.96 |
| Coal-tar creosote | 3.250 | 0.840 | 25.8 | |
| | 3.080 | 0.875 | 28.4 | 27.10 |
| Untreated control | 0.000 | 0.000 | | |

The bar chart of Figure 6 also shows the relationship of each of the preservatives so tested to the per cent of weight lost. It will be noted that although there is no striking difference between the preservatives tested, the oil-tar creosote shows a slight advantage in the matter of resistance to leaching.

The leached blocks were then placed in Kolle flask cultures and incubated. The results are shown later in the bulletin.

8. Penetration and absorption. Penetration and absorption are affected by several factors, such as temperature of the liquid, viscosity of the liquid, pressure used in the application, and the time under treatment. In the initial investigations of oil-tar creosote it was found that the oil flowed freely into the wood. As an initial study of the penetration, two short pieces of 2 in. \times 6 in. ponderosa pine were sawed from the same board; one piece was set on end in $\frac{1}{2}$ inch of coal-tar creosote and the other set on end in $\frac{1}{2}$ inch of oil-tar

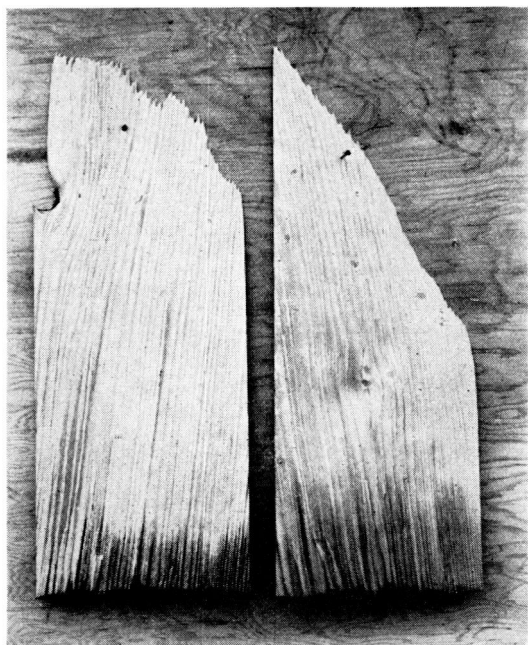


Figure 7. END PENETRATION OF COAL-TAR CREOSOTE (LEFT) AND OIL-TAR CREOSOTE (RIGHT) ON 2 \times 6 PONDEROSA PINE DURING 20-MINUTE DIPPING PERIOD.

creosote and allowed to stand for 20 minutes. After exposing to the air of the laboratory for 2 weeks, the pieces were split longitudinally and the end penetration noted. The average depth of penetration parallel with the grain was 1.25 inches for the coal-tar creosote and 2.5 inches for the oil-tar creosote. These penetrations are shown in Figure 7. It was believed that this was a fair measure of the comparable penetrating ability of the two creosotes tested. It was deemed advisable, however, to continue the study of penetration and absorption under semicommercial pressure-treating conditions.

TREATMENT REPORT

For Oregon State College at St. Helens, Oregon

On Test Posts

9/20, 1939

Client's Order No.....

Plant Order No.....

Our No.

Plant Report No. 1.....

RECORD OF BATHStarted, Date 9/20/39
HourHour 5:30 p.m.
Hours in Bath

Ended, Date.....

*Temperature**Vacuum*

At Start190 F

Min Vacuum24 inches

Maximum190 F

Max Vacuum24 inches

At End190 F

AIR PRESSURE OR INITIAL VACUUM

Started 7:30

Ended 8:30

Lb/Sq In., 50

Hours 1

OIL PRESSURE

Started 9/21/39

Hour 8:45

Ended 9/21/39

Hr 9:45

Hours under pressure, 1

*Pressures**Temperatures under pressure*

At Start5 Lb/Sq In.

At Start135 Lb/Sq In.

At End130 Lb/Sq In.

At End135 Lb/Sq In.

Maximum135 Lb/Sq In.

EXPANSION BATH

Started 9/21/39

Hour 9:45

Ended 9/21/39

Hr 11:45

Hours in expansion bath, 2

Temperatures

During Expansion Bath

At Start135 F

Maximum208 F

At End208 F

FINAL VACUUM

Started 12 N

Ended 1 pm

Total 1 hr, 60 min

Max, Inches

Min, Inches

TOTAL TREATING TIME**HR 19****Min 30****MATERIAL**

No. Pcs

Dimensions

Length

Lin or Bd Ft

Cubic Ft

31

4 X 4

5 ft

210 Bd Ft

17.5

Portland Gas and Coke Company creosote used in treatment of this material.

Inasmuch as a number of posts were to be treated and used for field tests in the School of Forestry test grounds (Post Farm), it is believed desirable to include the log of the treating process together with individual post data. These data are of value in a study of absorption and penetration and may be used for future reference in connection with the field tests as well.

Thirty-one Douglas-fir posts were selected and treated at the pilot plant of the Pope and Talbot creosoting plant at St. Helens, Oregon. This work was done under the direction of Mr. R. H. Rawson. A direct comparison

Table Showing Data on the Treatment of Douglas-Fir Posts
with Oil-Tar Creosote.

4 in. X 4 in. — 5 ft posts

| <i>Piece No.</i> | <i>Volume Cu Ft</i> | <i>Green weight Lb</i> | <i>Treated weight Lb</i> | <i>Moisture per cent before</i> | <i>Gain Lb</i> | <i>Gain Lb/Cu Ft</i> | <i>Correc- tion Lb/Cu Ft</i> | <i>Net gain Lb/Cu Ft</i> |
|--|-------------------------|--------------------------------|----------------------------------|---|--------------------|------------------------------|--|--------------------------------------|
| P- 1 | 0.627 | 23.500 | 26.125 | 36.85 | 2.625 | 4.19 | 5.10 | 9.29 |
| P- 2 | 0.589 | 24.125 | 25.625 | 32.14 | 1.500 | 2.55 | 4.31 | 6.86 |
| P- 3 | 0.627 | 24.375 | 26.000 | 33.51 | 1.625 | 2.59 | 4.45 | 7.04 |
| P- 4 | 0.608 | 18.750 | 20.875 | 35.19 | 2.125 | 3.50 | 3.87 | 7.37 |
| P- 5 | 0.608 | 20.500 | 23.125 | 32.27 | 2.625 | 4.32 | 3.58 | 7.90 |
| P- 6 | 0.627 | 24.750 | 26.750 | 29.31 | 2.000 | 3.19 | 3.39 | 6.58 |
| P- 7 | 0.608 | 21.875 | 24.000 | 31.46 | 2.125 | 3.50 | 3.63 | 7.13 |
| P- 8 | 0.608 | 25.875 | 26.375 | 40.15 | 0.500 | 0.82 | 6.66 | 7.48 |
| P- 9 | 0.609 | 23.500 | 26.000 | 34.03 | 2.500 | 4.11 | 4.55 | 8.66 |
| P-10 | 0.664 | 29.625 | 30.250 | 28.72 | 0.625 | 0.94 | 3.75 | 4.69 |
| P-11 | 0.590 | 23.500 | 25.250 | 36.02 | 1.750 | 2.97 | 5.21 | 8.18 |
| P-12 | 0.590 | 23.750 | 25.625 | 35.13 | 1.875 | 3.18 | 5.04 | 8.22 |
| P-13 | 0.608 | 20.625 | 23.000 | 33.26 | 2.375 | 3.91 | 3.83 | 7.74 |
| P-14 | 0.590 | 22.000 | 24.375 | 32.59 | 2.375 | 4.03 | 4.04 | 8.07 |
| P-15 | 0.622 | 25.750 | 27.000 | 36.58 | 1.250 | 2.01 | 5.57 | 7.58 |
| P-16 | 0.590 | 23.750 | 24.875 | 37.51 | 1.125 | 1.91 | 5.65 | 7.56 |
| P-17 | 0.599 | 20.125 | 22.750 | 33.96 | 2.625 | 4.38 | 5.29 | 9.67 |
| P-18 | 0.617 | 20.625 | 23.375 | 49.36 | 2.750 | 4.46 | 4.73 | 9.19 |
| P-19 | 0.570 | 22.000 | 24.000 | 29.29 | 2.000 | 3.51 | 3.30 | 6.81 |
| P-20 | 0.627 | 20.000 | 23.000 | 30.90 | 3.000 | 4.78 | 3.09 | 7.87 |
| P-21 | 0.627 | 20.875 | 23.500 | 29.82 | 2.625 | 4.19 | 2.97 | 7.16 |
| P-22 | 0.609 | 22.500 | 25.000 | 29.00 | 2.500 | 4.11 | 3.09 | 7.20 |
| P-23 | 0.625 | 22.875 | 26.500 | 28.77 | 3.625 | 5.80 | 3.00 | 8.80 |
| P-24 | 0.590 | 24.250 | 25.000 | 32.82 | 0.750 | 1.27 | 4.52 | 5.79 |
| P-25 | 0.627 | 23.500 | 24.750 | 39.68 | 1.250 | 1.99 | 8.04 | 10.03 |
| P-26 | 0.655 | 22.875 | 25.375 | 37.05 | 2.500 | 3.82 | 5.88 | 9.70 |
| P-27 | 0.674 | 22.125 | 25.250 | 28.85 | 3.125 | 4.64 | 2.71 | 7.35 |
| P-28 | 0.646 | 21.125 | 24.000 | 32.38 | 2.875 | 4.45 | 3.50 | 7.95 |
| P-29 | 0.636 | 22.500 | 26.000 | 32.99 | 3.500 | 5.50 | 3.93 | 9.43 |
| P-30 | 0.599 | 26.125 | 27.250 | 28.21 | 1.125 | 1.88 | 2.65 | 4.53 |
| P-31 | 0.627 | 27.750 | 28.500 | 32.63 | 0.750 | 1.20 | 4.81 | 6.01 |
| Total | 19.093 | 715.500 | 779.500 | | 64.000 | | | |
| Average | | | | | | 3.35 | 4.27 | 7.62 |
| Average moisture after treatment | | | | 18.22 | | | | |

of the penetrations and absorptions of the oil-tar creosoted posts treated in the pilot plant and other preservatives applied in commercial retorts cannot be made due to the differences in the treating plants. These posts, which were placed in the Post Farm, have retained an average absorption of 7.62 pounds

of preservative per cubic foot as specified by Federal Specifications TT-W-556 and the American Wood Preservers Specifications 4d and 5b (1933).

Now that the results of absorption under the given treating conditions have been observed it becomes desirable to investigate the corresponding penetrations. Posts 26 and 28 were cut in even foot lengths in order to more generally observe the penetration. The following table shows the depths of penetration for posts 26 and 28. Wafers were sawed from the cross-section of each post at one-foot intervals as indicated in Figure 8. Penetrations were measured midway between corners at lettered points. The incisor marks showed a penetration of approximately four-tenths of an inch from the surface.

Measured Penetrations

Post 28

| Point | Section 1 | | Section 2 | | Section 3 | | Section 4 | |
|-----------|-----------|------|-----------|------|-----------|------|-----------|------|
| A | 0.65 | inch | 0.65 | inch | 0.55 | inch | 0.55 | inch |
| B | 0.75 | inch | 0.85 | inch | 0.90 | inch | 0.80 | inch |
| C | 0.60 | inch | 0.60 | inch | 0.60 | inch | 0.70 | inch |
| D | 0.80 | inch | 0.80 | inch | 0.80 | inch | 0.85 | inch |
| Avg | 0.700 | inch | 0.725 | inch | 0.712 | inch | 0.725 | inch |

Post 26

| | | | | | | | | |
|-----------|-------|------|-------|------|-------|------|-------|------|
| A | 0.60 | inch | 0.60 | inch | 0.65 | inch | 0.80 | inch |
| B | 0.80 | inch | 0.80 | inch | 0.75 | inch | 0.65 | inch |
| C | 0.65 | inch | 0.80 | inch | 0.80 | inch | 0.65 | inch |
| D | 0.80 | inch | 0.65 | inch | 0.75 | inch | 0.70 | inch |
| Avg | 0.712 | inch | 0.712 | inch | 0.737 | inch | 0.700 | inch |

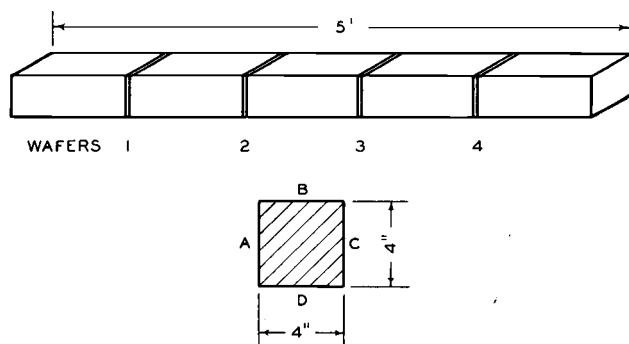


Figure 8. SKETCH INDICATING SAMPLING POINTS FOR PENETRATION MEASUREMENTS.

An examination of the photographs (Figures 9 and 10) will convey a general impression of the depth of penetration secured. It is thus shown that the oil-tar creosote, in spite of the higher than normal percentage of high-boiling oils, still maintains a very fluid state, through which deep penetrations have been obtained.

9. **Viscosity.** One of the factors that bears on the penetrating ability of a liquid is the viscosity. Tests on the Saybolt viscosities at various temperatures for oil-tar creosote and for the coal-tar creosote tested are shown in the following table and in Figure 11. The difference indicates a better penetrating ability for oil-tar creosote.

| <i>Coal-Tar Oil</i> | | <i>Gasco</i> | |
|---------------------|--------------------|---------------|--------------------|
| <i>Temp C</i> | <i>Saybolt Sec</i> | <i>Temp C</i> | <i>Saybolt Sec</i> |
| 22.0 | 57 | 22.50 | 46 |
| 37.0 | 45 | 33.0 | 40 |
| 42.5 | 42 | 42.0 | 37 |
| 45.0 | 41 | 51.5 | 34 |
| 60.0 | 35 | 63.5 | 32 |

It was not considered advisable to carry the determinations to a higher temperature because of the approach to the limit for the Saybolt instrument. In order to show further comparison with other creosote oils the viscosity data from J. D. MacLean's U. S. Dept. of Agric. Miscellaneous Publication No. 224 were used. Since his data did not contain information on specific gravity, a unit specific gravity was assumed and absolute viscosity was changed to Saybolt in order to conform to the data shown on the graph of Figure 11.

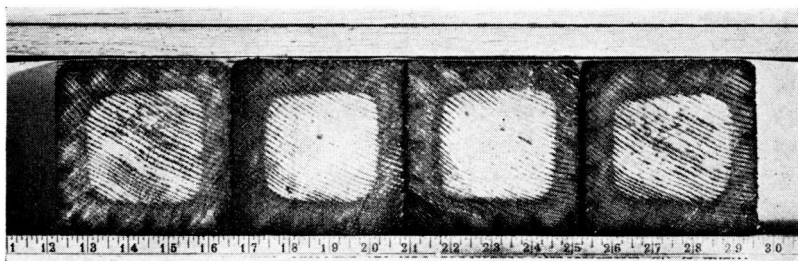


Figure 9. POST No. 26 TREATED WITH OIL-TAR CREOSOTE.

THE SECTIONS HERE SHOWN CORRESPOND TO THE SAMPLE POINTS AS DESIGNATED IN THE TEXT. SECTIONS TAKEN THREE WEEKS AFTER TREATING. POST 28 SHOWED VERY SIMILAR RESULTS.

10. **Flash point.** Since safety in handling and use of a preservative is one of the requirements, and since fires are a hazard about the creosoting operations, it was thought advisable to investigate the flash points of the two creosotes under investigation. The flash points, determined by the Cleveland open-cup method (18), were found to be 215 F for coal-tar creosote and 175 F for oil-tar creosote. Inasmuch as the lower-boiling fractions have been found to be of low, transient value in serving as a wood preservative, the commercial production of oil-tar creosote will doubtless eliminate a larger portion of the lower-boiling oils. This, in turn, should materially raise the flash point of the commercial creosote.

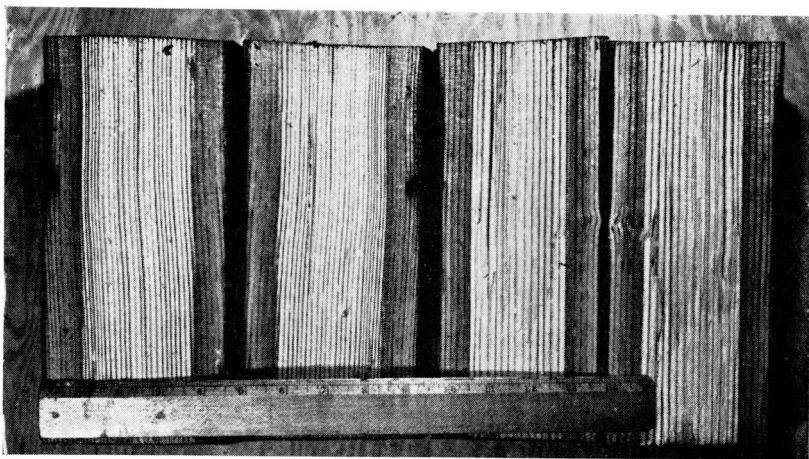


Figure 10. POSTS 26 AND 28 SPLIT 3 MONTHS AFTER TREATING TO SHOW PENETRATION OF CREOSOTE. NOTE DEPTH OF PENETRATION BELOW INCISOR MARKS.

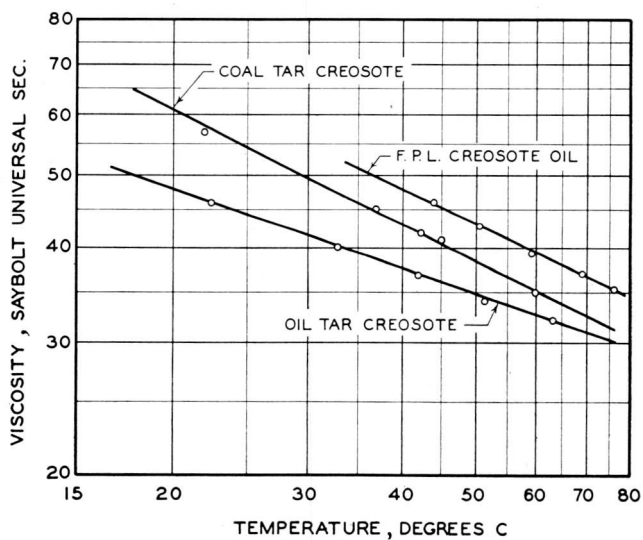


Figure 11. VISCOSITY-TEMPERATURE RELATIONSHIPS FOR WOOD-PRESERVATIVE SAMPLES.

V. PATHOLOGICAL STUDY

1. **Test program.** The objective of this portion of the study was to determine the relative toxicities of coal-tar creosote and oil-tar creosote. The procedure and results will be found in the following discussion.

The study here is divided into two parts; the first or orientation series was conducted in order to determine in a general way the relative toxicities. After completing the first series of tests it was decided that in order to make the study comprehensive and thorough a check series would be highly desirable. Such a series was run with certain differences in procedure followed in the second series. The procedure used in the second series and tabulation of results for the same follow the description and tabulation of results as for the first series of tests.

2. **Selection of method.** In searching for a satisfactory method of determining the toxicity of a preservative as well as the ability of the preservative to resist leaching and volatilization from the wood, two general methods were studied.

One of these methods, the petri-dish method, has been widely used for the determination of the toxic point of preservatives. This method, known also as the American method, has been the basis for most of the toximetric determinations made by American investigators. Humphrey and Fleming (28) describe this method quite fully. Some variations as to the type and preparation of media are used by different investigators, but all mix the preservative with the medium and inoculate with the desired fungus.

The other method, known as the wood-block method, is quite generally used by the European investigators. It is claimed, as Hunt (26) points out, that the wood-block method is more desirable since it more nearly measures the conditions under which wood will decay in service. Rabanus, Adolph, (35) concludes that the wood-block method is the better of the two. Waterman and associates (47) of the Bell Telephone Laboratories have used a special adaptation of the wood-block method, believing it superior to the agar or petri-dish method. Hatfield, Shumard, and Flenning (21) describe the general method of procedure in using the wood-block method. Hubert (24) has also used the wood-block method with excellent results.

Since the wood-block method in Kolle flasks seems to be held in high favor by European investigators, it has become increasingly important in the United States, and has become a generally accepted procedure (33) among workers in the United States. It was therefore concluded that this method would be most logical and most productive for present and future comparisons of data. It has been estimated by some workers that the procedure followed in this study may give results comparable to 25 years of service. Consequently, it is believed that in lieu of service tests this procedure is the best one available for obtaining not only the toxicity but also the action of a preservative over a period of time.

3. **Selection of wood for the host.** In order to comply with standard procedure, ponderosa pine sapwood was selected as the wood to be impregnated with the preservatives and subsequently subjected to the action of fungus. The pine sapwood was obtained from Shevlin-Hixon Company, Bend, Oregon. All of the samples used in the first study were from the same board, the wood of which showed a specific gravity of 0.506 and 29 rings per inch. Since ponderosa pine is generally considered nondurable, especially the sapwood, it was

believed that this material would represent about the worst condition in respect to the effect of the inherent durability of the wood to show in the results. Also it was believed that by selecting all of the material from one 12-foot board, all samples would have equal properties, thereby eliminating several variable factors.

4. Preparation of wood samples. The wood was allowed to season for 1 year in a heated room. The moisture content reached after this seasoning was approximately 10 per cent. The $2 \times 6 \times 12$ was then ripped and planed to $1\frac{1}{2} \times 2$ ", from which $\frac{1}{4}$ -inch sections were cut by means of a band saw.

5. Conditioning of samples. In order that all of the sample blocks be of the same moisture content, both for determining the moisture content on the oven-dry basis as well as for eliminating the factor of nonuniform moisture content at the time of impregnation, 350 sample blocks were placed in a desiccator over a saturated solution of sodium chloride and left to stand for 4 weeks. It was found that by this procedure a uniform moisture content of 11.97 per cent was obtained. Moisture content was found by weighing every tenth block and placing in a drying oven at 104 C until no further loss in weight of the sample could be detected. The percentage was then computed on the oven-dry basis, and this percentage was used for computing the oven-dry weight of the impregnated samples.

6. Selection of fungus. After examining the work of a number of investigators who have done considerable work on toxicity of wood preservatives, and upon the recommendation of Dr. E. E. Hubert, *Lenzites trabea* and *Poria incrassata* were selected as the two organisms with which to work. The basis for this selection was the fact that both of these fungi are quite generally found under actual service conditions, both react well under laboratory conditions, and both have been previously used in experimental work. In spite of the fact that the strain of *Fomes annosus*, known as Madison 517, has been more widely used in experimental work than the two selected, it is seldom found under general service conditions.

Cultures of both fungi were obtained from the Forest Products Laboratory, Madison, Wisconsin.

7. Preparation of flasks and media. The malt-agar medium was prepared by the following formula: (U.S. Dept. of Agric. Bulletin 346)

1000 cc distilled water
25 grams Difco malt extract
15 grams agar

One hundred cc of the prepared medium were poured into each Kolle flask. The flask was plugged with cotton and the flasks then sterilized in an autoclave under 15 pounds gage pressure steam for a period of 20 minutes. The flasks were then cooled and inoculated with the desired fungus. The fungus was allowed to grow for 14 days under incubation at 26 C, by which time most of the flasks had developed a mat over the entire surface of the agar medium. A few flasks inoculated with *Poria incrassata* required slightly longer for the fungus mat to completely cover the surface of the medium. All contaminations were rejected and new media prepared for the contaminated flasks. At the end of the 2 weeks' incubation period most of the flasks were ready to receive the preserved wood blocks.

8. **Treatment of wood blocks.** The wood blocks were taken from the desiccator and weighed on a delicate triple-beam balance. A number of weights were checked on the analytical balance in order to guard against any error. Since the weights checked almost identically, the former method of weighing was used. After the weights were taken and recorded, the same wood blocks were immediately placed in a jar fitted with a separatory funnel and hose outlet for a vacuum pump. Blocks to the desired number were placed in the jar, covered with glass beads, and the top of the jar sealed. The vacuum pump was then started and a vacuum of 2.2 inches of mercury absolute pressure drawn for a period of 30 minutes. The preservative of the desired concentration was then introduced without breaking the vacuum, after which the wood blocks were allowed to stand in the preservative for 30 minutes. After impregnation the blocks were removed, placed on glass rods, and allowed to drain and stand for 24 hours in the laboratory. The blocks were then replaced in the desiccator for 72 hours before weighing the second time. The second weighing was, of course, for the purpose of determining the amount of preservative taken up in each case.

In order to make the dilutions, Stoddard solvent was used as the nontoxic oil. All percentages are by weight. The specific gravity of each material used was determined and from these specific gravities the number of ml required of each substance was computed.

The following concentrations were used:

Coal-Tar Creosote

| No. | Per cent | CC Creosote | CC Solvent |
|----------|----------|-------------|------------|
| 1 | 100.0 | 100.00 | 0.00 |
| 2 | 50.0 | 50.00 | 67.30 |
| 3 | 12.0 | 11.16 | 110.27 |
| 4 | 6.0 | 5.57 | 117.79 |
| 5 | 4.0 | 5.93 | 192.50 |
| 6 | 1.0 | 2.22 | 295.74 |
| 7 | 0.8 | 1.78 | 272.60 |
| 8 | 0.6 | 1.33 | 273.00 |
| 9 | 0.5 | 1.48 | 397.91 |
| 10 | 0.4 | 1.187 | 398.39 |
| 11 | 0.3 | 0.89 | 398.80 |
| 12 | 0.2 | 0.742 | 499.10 |
| 13 | 0.1 | 0.371 | 499.49 |
| 14 | 0.05 | 0.185 | 499.74 |

After conditioning, weighing, impregnating, and again conditioning and weighing the samples were placed in the Kolle flasks. Two impregnated samples were placed in each flask, and in most cases a third block of untreated wood was placed in each flask to act as a control. Transfer of wood blocks to the flasks was made in a sterile transfer room in order to avoid contaminations. Three-millimeter glass rods were placed on top of the fungus mat to receive the wood blocks. The untreated blocks were sterilized in boiling water before placing in the flask. The impregnated blocks were not sterilized because it was thought that if the concentration of preservative was not sufficiently strong to kill any fungus spores that might be on the surface, neither would it kill nor inhibit the fungus to which it was exposed. The glass rods were used in order that the wood blocks would not pick up an excessive amount of moisture from the agar, which would inhibit the growth of the fungus. Also,

the surface of the wood being free from the agar would better accommodate the growth of the fungus on all six faces of the block.

Four wood blocks were impregnated with each of the concentrations listed, two blocks being placed in the flask with *Lenzites trabea* and two blocks in the flask of *Poria incrassata*. One untreated control block was placed in each flask along with the two treated blocks. The control block shows in the tabulations as "C".

9. Results from first series. After incubation for a period of 8 weeks at 26 C, the blocks that were exposed to *Lenzites trabea* were removed from the flask. The blocks exposed to *Poria incrassata* were removed from the flask at the end of the tenth week. The surface mycelium, if any, was brushed off and the blocks placed in a desiccator over sulphuric acid and allowed to stand until no further loss in weight could be detected. In order to allow for volatility during the incubation period the following procedure was used: 20 unattacked blocks were used for the basis:

Original preserved weight minus oven-dry weight equals weight of preservative.

Finished preserved weight minus oven-dry weight equals weight of preservative at conclusion of tests.

100 (*Original weight of preservative minus final weight of preservative*) divided by original weight of preservative equals *per cent of preservative lost during time in Kolle flask.*

This percentage of loss was then applied to each treated block to make the necessary correction for volatility.

The moisture-free weight of the wood blocks found as described above was then used as the basis for computing the per cent of weight lost on the original weight of the block before it was placed in the Kolle flask.

The results are given in the following tables and accompanying graphs:

TOXICITY TESTS, FIRST SERIES

Table Showing Loss in Weight of Individual Sample Blocks Impregnated with Different Concentrations of Coal-Tar Creosote after an Incubation Period of 8 Weeks with *Lenzites trabea*.

| Concentration, per cent | Sample (C is control) | Computed oven-dry weight | Adjusted oven-dry weight | Moisture-free Wt after incubation | Per cent loss in weight | Avg per cent loss |
|----------------------------|-----------------------------|--------------------------------|--------------------------------|---|-------------------------------|-------------------------|
| 100 | A | 4.544 | 6.670 | 6.96 | | |
| | B | 4.410 | 6.580 | 6.41 | No | |
| | C | 4.465 | | 4.52 | attack | — |
| 50 | A | 4.877 | 5.580 | 5.85 | No | |
| | B | 4.772 | 5.490 | 5.36 | attack | |
| | C | 5.123 | | 3.70 | 8.2 | — |
| 12 | A | 4.656 | 4.866 | | | |
| | B | 4.331 | 4.541 | | No | |
| | C | | | | attack | — |
| 6 | A | 4.914 | 5.020 | 4.93 | 1.7 | |
| | B | 4.342 | 4.450 | 4.30 | 3.3 | <u>2.5</u> |
| | C | 4.518 | | 2.75 | 39.1 | |
| 4 | A | 4.903 | 4.979 | 3.84 | 22.9 | |
| | B | 4.528 | 4.604 | 4.18 | 9.0 | <u>15.9</u> |
| | C | 4.419 | | 2.65 | 40.0 | |
| 1 | A | 4.812 | 4.830 | 3.31 | 31.2 | |
| | B | 4.673 | 4.692 | 3.39 | 28.0 | <u>29.6</u> |
| | C | 4.383 | | 3.23 | 26.3 | |
| 0.8 | A | 4.289 | 4.304 | 3.31 | 22.8 | |
| | B | 4.909 | 4.924 | 3.48 | 29.3 | <u>26.0</u> |
| | C | 4.640 | | 3.13 | 32.3 | |
| 0.6 | A | 4.702 | | 4.31 | 8.5 | |
| | B | 4.968 | | 3.51 | 29.3 | <u>18.9</u> |
| | C | 4.556 | | 3.04 | 33.2 | |
| 0.5 | A | 4.686 | | 3.72 | 20.5 | |
| | B | 4.753 | | 3.89 | 18.1 | <u>19.3</u> |
| | C | 4.908 | | 3.32 | 32.5 | |
| 0.4 | A | 4.562 | | 3.37 | 26.1 | |
| | B | 4.565 | | 3.39 | 25.7 | <u>25.9</u> |
| | C | 4.899 | | 3.75 | 23.4 | |
| 0.3 | A | 4.449 | | 4.02 | 9.6 | |
| | B | 4.440 | | 3.05 | 31.7 | <u>31.7</u> |
| | C | 4.811 | | 3.51 | 28.1 | |
| 0.2 | A | 4.611 | | 3.49 | 24.3 | |
| | B | 4.460 | | 3.45 | 22.6 | <u>23.4</u> |
| | C | 4.901 | | 3.70 | 24.1 | |
| 0.1 | A | 4.700 | | 3.48 | 25.9 | |
| | B | 4.601 | | 3.11 | 32.4 | <u>29.1</u> |
| | C Wet | 4.722 | | 4.35 | 8.0 | |
| 0.05* | A | 4.565 | | 3.95 | 12.3 | |
| | B | 4.418 | | 3.67 | 16.2 | <u>14.2</u> |
| | C | | | | | |

* Desiccation due to air leakage.

TOXICITY TEST, FIRST SERIES

Table Showing Loss in Weight of Individual Sample Blocks that Have Been Impregnated with Different Concentrations of Coal-Tar Creosote after an Incubation Period of 8 Weeks with *Poria incrassata*.

| Concentration, per cent | Sample (C is control) | Computed oven-dry weight | Adjusted oven-dry weight | Moisture-free Wt after incubation | Per cent loss in weight | Avg per cent loss |
|----------------------------|-----------------------------|--------------------------------|--------------------------------|---|-------------------------------|-------------------------|
| 100 | A | 4.445 | 6.310 | 6.44 | | |
| | B | 4.374 | 6.300 | 6.42 | No | |
| | C | 4.632 | | 4.73 | attack | |
| 50 | A | 4.418 | 5.230 | 5.39 | | |
| | B | 4.463 | 5.310 | 5.46 | No | |
| | C | 4.959 | | 5.19 | attack | |
| 12 | A | 4.352 | 4.544 | 4.43 | No | |
| | B | 4.619 | 4.811 | 4.71 | attack | |
| | C | 5.250 | | 5.12 | 2.0 | |
| 6 | A | 5.124 | 5.239 | 5.32 | No | |
| | B | 4.427 | 4.542 | 4.53 | attack | |
| | C | 4.304 | | 2.34 | 45.6 | |
| 4 | A | 4.563 | 4.640 | 4.65 | | |
| | B | 4.688 | 4.760 | 4.76 | No | |
| | C | 4.621 | | 4.64 (wet) | attack | |
| 1 | A | 4.408 | 4.467 | | 2.0 | |
| | B | 4.438 | 4.457 | | | |
| | C | | | | | |
| 0.8 | A | 4.417 | 4.432 | 3.29 | 25.7 | |
| | B | 4.430 | 4.445 | 3.78 | 14.9 | <u>20.3</u> |
| | C | 4.466 | | 1.75 | 60.7 | |
| 0.6* | A | 4.947 | 4.509 | 3.94 | 12.6 | |
| | B | 4.423 | 4.435 | 3.05 | 31.2 | <u>21.9</u> |
| | C | 4.690 | | 3.00 | 36.0 | |
| 0.5 | A | 4.779 | 4.788 | | | |
| | B | 4.913 | 4.923 | | | |
| | C | | | | | |
| 0.4* | A | 4.533 | | 3.36 | 26.0 | |
| | B | 4.427 | | 4.00 | 9.6 | <u>17.8</u> |
| | C | 4.458 | | 3.14 | 29.5 | |
| 0.3 | A wet | 5.000 | | 4.93 | 1.4 | |
| | B | 4.586 | | 3.66 | 20.1 | <u>20.1</u> |
| | C | 5.198 | | 2.62 | 49.5 | |
| 0.2 | A | 4.779 | | 3.53 | 26.1 | |
| | B | 4.515 | | 3.27 | 27.5 | <u>26.8</u> |
| | C | 4.498 | | 4.55 (wet) | | |
| 0.1 | A | 4.592 | | 3.46 | 24.6 | |
| | B | 4.622 | | 3.47 | 24.9 | <u>24.7</u> |
| | C | 4.665 | | 3.91 | 16.2 | |
| 0.05 | A wet | 4.530 | | 4.54 | | |
| | B | 4.811 | | 3.42 | 31.0 | <u>31.0</u> |
| | C | 4.616 | | 3.25 | 29.7 | |

* Benzol added to solvent in these samples.

Table Showing Loss in Weight of Individual Sample Blocks that Have Been Impregnated with Different Concentrations of Oil-Tar Creosote after an Incubation Period of 8 Weeks with *Lenzites trabea*.

| Concentration, per cent | Sample (C is control) | Computed oven-dry weight | Adjusted oven-dry weight | Moisture-free Wt after incubation | Per cent loss in weight | Avg per cent loss |
|----------------------------|-----------------------------|--------------------------------|--------------------------------|---|-------------------------------|-------------------------|
| 100 | A | 4.496 | 6.270 | 6.04 | | |
| | B | 4.631 | 6.570 | 6.37 | No | |
| | C | 4.078 | 5.078 | 5.08 | attack | — |
| 50 | A | 4.396 | 5.070 | 5.11 | | |
| | B | 4.403 | 5.110 | 5.01 | No | |
| | C | 4.317 | 4.317 | 4.15 | attack | — |
| 12 | A | 4.414 | 4.486 | | | |
| | B | 5.082 | 5.170 | | No | |
| | C | | | | attack | — |
| 6 | A | 4.509 | 4.593 | 4.17 | 9.2 | <u>4.46</u> |
| | B | 4.407 | 4.470 | 4.48 | | |
| | C | 5.265 | 5.265 | 3.40 | 35.6 | 21.84 |
| 4 | A | 4.476 | 4.580 | 4.21 | 8.08 | |
| | B | 4.507 | 4.541 | 3.16 | 30.4 | |
| | C | 4.760 | 4.760 | 3.46 | 37.5 | — |
| 1 | A | 4.438 | 4.451 | 3.165 | 28.9 | |
| | B | 4.435 | 4.448 | 3.16 | 28.9 | <u>28.9</u> |
| | C | 4.658 | 4.658 | 3.67 | 21.2 | |
| 0.8 | A | 4.262 | 4.277 | 3.26 | 23.3 | |
| | B | 4.575 | 4.590 | 3.53 | 23.0 | <u>23.3</u> |
| | C | 4.692 | 4.692 | 3.45 | 24.3 | |
| 0.6 | A | 4.404 | 4.415 | 3.50 | 25.2 | |
| | B | 4.785 | 4.798 | 4.00 | 16.6 | <u>20.9</u> |
| | C | 4.540 | 4.540 | 3.02 | 33.4 | |
| 0.5 | A | 4.624 | 4.633 | 4.02 | 13.2 | |
| | B | 4.352 | 4.361 | 3.58 | 17.7 | <u>15.4</u> |
| | C | 4.625 | 4.625 | 3.12 | 32.5 | |
| 0.4 | A | 4.765 | | 3.74 | 21.4 | |
| | B | 4.410 | | 3.40 | 22.9 | <u>22.1</u> |
| | C | 4.376 | | 3.15 | 28.0 | |
| 0.3 | A | | | | 33.2 | |
| | B | 4.691 | | 3.13 | | <u>33.2</u> |
| | C | 4.503 | | 3.11 | 33.2 | |
| 0.2 | A | 4.688 | | 3.58 | 23.6 | |
| | B | 4.465 | | 3.80 | 14.8 | <u>19.2</u> |
| | C | 4.702 | | | | |
| 0.1 | A | 4.314 | | 3.10 | 28.1 | |
| | B | 4.330 | | 3.14 | 27.4 | <u>27.7</u> |
| | C | 4.907 | | 3.40 | 30.7 | |
| 0.05 | A | 4.691 | | 3.46 | 26.0 | |
| | B | 4.205 | | 3.23 | 23.1 | <u>24.5</u> |
| | C | 4.395 | | 3.00 | 31.7 | |

TOXICITY TEST, FIRST SERIES

Table Showing Loss in Weight of Individual Sample Blocks That Have Been Impregnated with Different Concentrations of Oil-Tar Creosote after an Incubation Period of 8 Weeks with *Poria incrassata*.

| Concentration, per cent | Sample (C is control) | Computed oven-dry weight | Adjusted oven-dry weight | Moisture-free Wt after incubation | Per cent loss in weight | Avg per cent loss |
|----------------------------|-----------------------------|--------------------------------|--------------------------------|---|-------------------------------|-------------------------|
| 100 | A | 4.616 | 5.880 | 6.09 | | |
| | B | 4.290 | 5.620 | 5.71 | | |
| | C | | | | No attack | — |
| 50 | A | 4.503 | 5.100 | 5.10 | | |
| | B | 4.595 | 5.150 | 5.20 | | |
| | C | 4.906 | | 4.99 | No attack | — |
| 12 | A | 4.441 | 4.560 | 4.51 | | |
| | B | 5.106 | 5.234 | | No attack | — |
| | C | 5.125 | | 5.10 | | |
| 6 | A | 4.390 | 4.500 | 4.51 | | |
| | B | 4.767 | 4.880 | 4.91 | | |
| | C | 4.499 | | 4.52 | No attack | — |
| 4 | A | 4.419 | 4.496 | 4.51 | | |
| | B | 4.701 | 4.770 | 4.83 | | |
| | C | 4.454 | | 4.44 | No attack | — |
| 1 | A | 4.387 | 4.406 | 3.55 | 19.4 | |
| | B | 4.543 | 4.562 | 3.75 | 17.8 | 18.6 |
| | C | 4.910 | | 3.00 | 39.0 | |
| 0.8 | A | 4.582 | 4.597 | 3.51 | 21.4 | |
| | B | 4.607 | 4.622 | 3.86 | 16.4 | 18.9 |
| | C | 4.464 | | 2.31 | 48.2 | |
| 0.6 | A | 4.702 | 4.713 | 3.33 | 29.4 | |
| | B | 4.454 | 4.465 | 3.66 | 18.0 | 23.7 |
| | C | 4.632 | | 2.84 | 38.9 | |
| 0.5 | A | 4.511 | 4.521 | 2.84 | 37.2 | |
| | B | 4.463 | 4.473 | 3.74 | 16.4 | 21.8 |
| | C | 4.313 | | 2.73 | 36.9 | |
| 0.4 | A | 4.319 | 4.321 | 3.93 (wet) | 8.8 | |
| | B | 4.780 | 4.782 | 4.90 (wet) | | |
| | C | 4.526 | | 2.68 | 40.8 | 41.0 |
| 0.3 | A | 4.387 | | 3.23 | 26.3 | |
| | B | 4.653 | | 3.65 | 21.5 | 23.9 |
| | C | 3.943 | | 2.82 | 28.5 | |
| 0.2 | A | 4.459 (wet) | | 4.34 | | |
| | B | 4.909 | | 3.90 | 20.5 | 20.5 |
| | C | 4.416 | | 2.95 | 33.1 | |
| 0.1 | A | 4.557 | | 3.80 | 16.6 | |
| | B | 4.532 | | 3.99 | 11.9 | 14.2 |
| | C | 4.464 | | 3.03 | 32.1 | |
| 0.05 | A | 4.441 | | 3.71 | 16.4 | |
| | B | 4.692 | | 4.06 | 13.4 | 14.9 |
| | C | 4.543 | | 3.42 | 24.7 | |

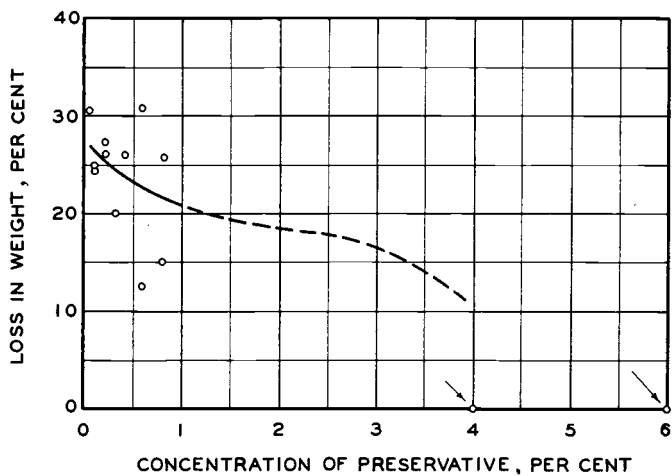


Figure 12. TOXICITY OF COAL-TAR CREOSOTE TO *Poria Incrassata*.

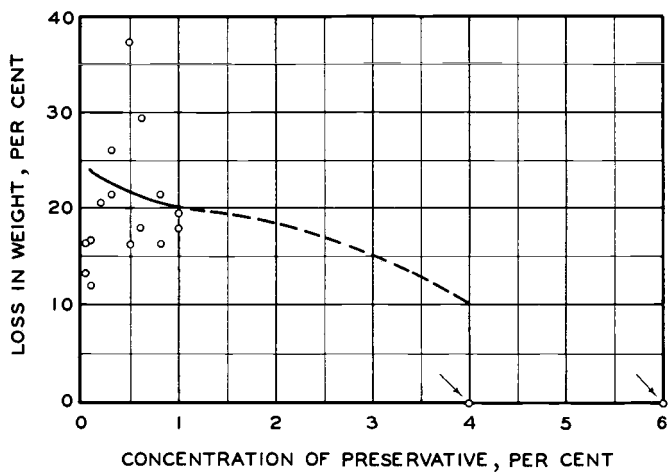


Figure 13. TOXICITY OF OIL-TAR CREOSOTE TO *Poria Incrassata*.

Table Showing the Per Cent of Weight of Untreated Wood Lost when Exposed to 8 Weeks Incubation with Fungus in Kolle Flasks.

| Sample | Orig Wt at 12 per cent moisture content | Computed oven-dry Wt of wood alone | Dry Wt at end of 8-week period | Per cent loss |
|-----------------------------------|--|---|---|------------------|
| A. <i>Lenzites trabea</i> | | | | |
| ControlA (wet) | 5.000 | 4.454 | 3.81 | 14.2 |
| Flask 2B | 5.420 | 4.829 | 3.29 | 32.7 |
| Flask 1A | 4.848 | 4.319 | | |
| B | 5.250 | 4.677 | 3.48 | 25.4 |
| C | 5.258 | 4.684 | 3.18 | 32.1 |
| B. <i>Poria incrassata</i> | | | | |
| Flask 3A | 5.520 | 4.918 | | |
| B | 4.923 | 4.386 | | |
| C | 5.980 | 4.437 | | |
| Flask 4A | 5.347 | 4.763 | 4.01 | 15.8 |
| B | 5.150 | 4.588 | 3.46 | 24.3 |
| C | 5.018 | 4.471 | 3.65 | 18.3 |

VI. SECOND SERIES OF KOLLE FLASK TESTS

1. **Objective.** The objective of this series of tests was to check and to determine more closely the comparative toxicity of the two creosotes at the inhibiting point.

These tests were made because it was found that the number of samples near the inhibiting point was not sufficient for determining accurate results. It was also necessary to check the action of the solvent in the first series of tests because it was found that the mixture of petroleum solvent and aromatic creosote formed a precipitate almost immediately in the case of the coal-tar creosote and within a few days with the oil-tar creosote.

2. **Procedure.** Certain changes in technique were followed in the second series of tests in order to comply with the standard procedure (35). Since these changes seemed desirable the procedure for the second series is given in brief form:

(a) Kiln-dried ponderosa pine blocks, $1\frac{1}{2} \times 2$ inches cross-section by $\frac{1}{4}$ inch in thickness were used.

(b) Blocks conditioned for 21 days in a desiccator over a saturated solution of sodium bromide at a temperature of 23 C.

(c) After conditioning, the blocks were weighed to the nearest 1/100 gram.

(d) Culture media and test fungus prepared as in the first series. Only one fungus, *Lenzites trabea*, was used since it apparently was more resistant to the creosote than *Poria incrassata*. This also allowed a greater number of samples to be taken.

(e) Preparation of preservative. Both coal-tar and oil-tar creosote were diluted with benzol on the basis of weight to give the following concentrations:

- 1 per cent creosote
- 3 per cent creosote
- 6 per cent creosote
- 8 per cent creosote

These concentrations were used for straight toxicity tests. For volatility and leaching tests the following concentrations were used:

5 per cent creosote
10 per cent creosote
100 per cent benzol was used in treating four control blocks in order to study the action of benzol.

(f) Impregnation of test blocks. After weighing, 14 blocks were placed in a 1-pint flask to which a separatory funnel and vacuum line were attached. The flask was exhausted for a period of 30 minutes at a pressure of 4 inches of mercury. After exhausting, the treating solution was admitted to the flask through the separatory funnel without breaking the vacuum and the solution allowed to stand for 20 minutes. The flask was filled to capacity in order that all blocks might be completely covered with the solution. The blocks were weighed immediately after being removed from the treating solution.

(g) Reconditioning. The treated blocks were placed on glass rods and dried 14 days in the laboratory air, after which they were replaced in the desiccator over the sodium bromide solution and allowed to stand for 7 days. The blocks were then removed from the desiccator and weighed.

(h) Incubation. Ten of the 14 blocks were placed in Kolle flasks on $\frac{1}{8}$ -inch glass rods. The fungus in the Kolle flask had grown for a period of more than 2 weeks and formed a mat over the surface of the media. The four remaining treated blocks were placed in Kolle flasks over sterile agar. These blocks will hereafter be referred to as reference blocks, and in the tabulations, will be marked R. The flasks were then placed in an incubator at 26 C for 64 days. Seventeen conditioned untreated blocks were also incubated with the fungus to serve as control blocks.

(i) End of test. At the end of the incubation period the blocks were removed, the surface brushed free of mycelium, and oven dried. The oven-dry weight was recorded.

(j) Computations. The computations were made in accordance with the following proportion formula:

$$\frac{\text{Weight of reconditioned treated : reference blocks}}{\text{Weight of reconditioned treated : blocks}} :: \frac{\text{Oven-dry weight of reference : X blocks}}{\text{Oven-dry weight of reference : blocks}}$$

This computation gave the computed oven-dry weight of the treated blocks as they were before submission to the test fungus.

Knowing the computed oven-dry weight of the treated blocks, the per cent weight loss of the blocks due to fungus attack was then computed as follows:

$$\frac{(\text{Computed oven-dry weight} - \text{Actual oven-dry weight}) \times 100}{\text{Computed oven-dry weight}} = \begin{matrix} \% \text{ loss} \\ \text{in weight} \end{matrix}$$

(k) Results. These are shown in the tabulated form and also in graphic form by plotting per cent loss in weight over concentration. See Figures 14 and 15.

3. Resistance to volatilization. Twenty-eight blocks were treated with a 5 per cent and also 28 blocks with a 10 per cent solution of creosote in benzol. The treated blocks were dried for 14 days in the laboratory air on glass

rods, replaced in the desiccator, and conditioned as in item 2 for 7 days. The blocks were then weighed.

The test blocks were exposed to a temperature of 160 F for 24 hours. The blocks were again reconditioned for 3 days as under item 2. Four reference blocks were oven dried and weighed. Ten of the blocks were incubated with *Lenzites trabeas* as in item 8. Computations were made as in item 10. Results are shown in tabular and graphic form. See Figure 16.

4. Resistance to leaching by water. Using the blocks subjected to the volatilization test, 14 treated blocks were placed in a 1-quart container, a vacuum drawn for 15 minutes, and the vacuum broken with 1,000 ml of water. The water was then changed every hour for 7 hours the first 2 days, and every day for the remaining 3 days. The water was drained off at the end of each day, leaving the blocks in the closed flask for the 16-hour period. At the end of the fifth day the blocks were dried for a period of 4 days in the laboratory air, and then reconditioned for 7 days as under item 2-b. The reference blocks were removed, oven-dried, and weighed. The remaining blocks were incubated as in items 2-h and 2-i. Results are shown in tabular and graphic form. See Figure 16.

The following tabulations show the progress of the individual sample blocks from the time they were first weighed from the desiccator after being conditioned, to the time the blocks were removed from the oven and the losses in weight calculated.

The first eight tables concern the toxicity tests made on blocks treated with 1, 3, 6, and 8 per cent solutions of the creosote in benzol. The following four tables show the progress of the blocks treated with 5 and 10 per cent solutions of creosote in benzol. The first 14 blocks in the 5 and 10 per cent tables were used for volatility tests and those numbered from 15 to 28 inclusive were subjected to the leaching tests as well as volatility. The last table shows the progress of the untreated control blocks and the benzol-treated control blocks in the same manner.

TOXICITY TESTS, SECOND SERIES

Coal-Tar Creosote 1% Solution in Benzol

| Block No. | 446.42 ml benzol, | | 3.72 ml creosote | | | |
|-------------|-----------------------------|-----------------------|-------------------|--------------------------|-----------------|---------------|
| | Original conditioned weight | Weight after treating | Wet weight at end | Computed oven-dry weight | Oven-dry weight | Per cent loss |
| C-1-1 | 4.41 | 10.36 | 10.25 | 4.10 | 2.10 | 50.0 |
| C-1-2 | 4.35 | 10.46 | 8.26 | 4.04 | 1.92 | 50.0 |
| C-1-3 | 4.46 | 10.20 | 4.80 | 4.14 | 1.91 | 60.2 |
| C-1-4 | 4.46 | 10.58 | | | 4.18 R | |
| C-1-5 | 4.65 | 10.84 | | | 4.28 R | |
| Avg | 4.555 | | | | Avg 4.23 | |

Oil-Tar Creosote 1% Solution in Benzol

446.4 ml benzol, 3.87 ml creosote

| | | | | | | |
|---------------|------|-------|-------|-------|-----------|-------|
| O-1-1-x | 4.53 | 10.12 | 8.23 | 4.18 | 2.71 | 35.1 |
| O-1-2 | 4.53 | 10.54 | 5.32 | 4.18 | 2.90 | 30.62 |
| O-1-3 | 4.22 | 10.07 | 5.58 | 3.89 | 2.64 | 32.1 |
| <hr/> | | | | | | |
| O-1-4 | 4.60 | 10.28 | | | 4.27 R | |
| O-1-5 | 4.58 | 10.36 | | | 4.20 R | |
| <hr/> | | | | | | |
| Avg | 4.59 | | | | Avg 4.235 | |

TOXICITY TESTS, SECOND SERIES

Coal-Tar Creosote, 3% Solution in Benzol

437.4 ml benzol, 11.16 ml creosote

| Block No. | Original conditioned weight | Weight after treating | Weight after conditioning | Wet weight at end | Computed oven-dry weight | Oven-dry weight | Per cent loss |
|--------------|-----------------------------|-----------------------|---------------------------|-------------------|--------------------------|-----------------|---------------|
| C-3-1 | 4.05 | 7.00 | 4.06 | 4.95 | 3.78 | 2.31 | 38.8 |
| C-3-2 | 4.38 | 7.49 | 4.39 | 8.99 | 4.09 | 5.37 | 17.6 |
| C-3-3 | 4.44 | 7.51 | 4.455 | 5.67 | 4.15 | 2.59 | 37.3 |
| C-3-4 | 4.21 | 7.26 | 4.22 | | 3.93 | 3.32 | 15.6 |
| C-3-5 | 4.30 | 7.58 | 4.33 | | 4.03 | 3.15 | 21.7 |
| C-3-6 | 4.36 | 7.45 | 4.34 | | 4.04 | 3.70 | 8.4 |
| C-3-7 | 4.225 | 7.28 | 4.235 | 8.72 | 3.94 | 3.37 | 14.5 |
| C-3-8 | 4.30 | 7.40 | 4.29 | 12.85 | 4.00 | 3.60 | 10.0 |
| C-3-9 | 4.14 | 7.13 | 4.13 | 5.00 | 3.85 | 2.48 | 35.3 |
| C-3-10 | 4.38 | 7.61 | 4.38 | | 4.08 | 2.57 | 37.0 |
| <hr/> | | | | | | | |
| C-3-11 | 4.50 | 7.70 | 4.47 | | | 4.07 R | |
| C-3-12 | 4.35 | 7.54 | 4.35 | | | 4.03 R | |
| C-3-13 | 4.46 | 7.67 | 4.47 | | | 4.07 R | |
| C-3-14 | 4.11 | 7.23 | 4.10 | | | 4.03 R | |
| <hr/> | | | | | | | |
| Avg | 4.355 | | 4.375 | | | 4.05 | |

Moisture content = 9.69% and 11.6%

Oil-Tar Creosote 3% Solution in Benzol

437.4 ml benzol, 11.62 ml creosote

| | | | | | | | |
|--------------|--------|------|-------|-------|--------|-------|-------|
| O-3-1 | 4.23 | 7.65 | 4.28 | 4.31 | 3.89 | 2.62 | 32.6 |
| O-3-2 | 4.47 | 7.89 | 4.49 | 5.22 | 4.08 | 2.66 | 34.8 |
| O-3-3 | 4.25 | 7.47 | 4.275 | 5.75 | 3.89 | 2.52 | 35.2 |
| O-3-4 | 4.275 | 7.68 | 4.30 | 10.00 | 3.91 | 3.54 | 9.4 |
| O-3-5 | 4.215 | 7.67 | 4.265 | | 3.88 | 3.45 | 11.1 |
| O-3-6 | 4.32 | 7.60 | 4.36 | | 3.96 | 2.70 | 31.8 |
| O-3-7 | 4.21 | 7.29 | 4.23 | 5.21 | 3.85 | 2.12 | 45.5 |
| O-3-8 | 4.175 | 7.46 | 4.20 | 5.02 | 3.82 | 2.15 | 43.7 |
| O-3-9 | 4.27 | 7.42 | 4.31 | 5.39 | 3.92 | 2.50 | 36.2 |
| O-3-10 | 4.15 | 7.23 | 4.18 | 5.10 | 3.80 | 2.41 | 36.5 |
| <hr/> | | | | | | | |
| O-3-11 | 4.41 | 7.74 | 4.45 | | 4.00 R | | |
| O-3-12 | 4.16 | 7.47 | 4.18 | | 3.78 R | | |
| O-3-13 | 4.03 | 7.00 | 4.06 | | 3.72 R | | |
| O-3-14 | 4.23 | 7.54 | 4.26 | | 3.91 R | | |
| <hr/> | | | | | | | |
| Avg | 4.2075 | | 4.238 | | 3.8525 | | |

Moisture content = 10.7% and 11.05%

TOXICITY TESTS, SECOND SERIES

Coal-Tar Creosote 6% Solution in Benzol

423.9 ml benzol, 22.3 ml creosote

| Block No. | Original conditioned weight | Weight after treating | Weight after conditioning | Wet weight at end | Computed oven-dry weight | Oven-dry weight | Per cent loss |
|--------------|-----------------------------|-----------------------|---------------------------|-------------------|--------------------------|-----------------|---------------|
| X-6-1 | 4.36 | 7.09 | 4.33 | 9.55 | 3.87 | 3.86 | 0.0 |
| X-6-2 | 4.35 | 7.36 | 4.43 | 13.34 | 3.99 | 4.00 | 0.0 |
| X-6-3 | 4.41 | 7.44 | 4.46 | 11.60 | 4.00 | 4.01 | 0.0 |
| X-6-4 | 4.31 | 7.19 | 4.39 | 13.16 | 3.93 | 3.92 | 0.0 |
| X-6-5 | 4.36 | 7.17 | 4.435 | 5.47 | 3.97 | 3.94 | 0.0 |
| X-6-6 | 4.37 | 6.76 | 4.44 | 7.85 | 3.97 | 3.98 | 0.0 |
| X-6-7 | 5.05 | 8.28 | 5.11 | 6.65 | 4.57 | 4.31 | 5.7 |
| X-6-8 | 4.84 | 7.78 | 4.92 | 7.03 | 4.40 | | 4.3 |
| X-6-9 | 4.44 | 7.28 | 4.51 | | 4.03 | 4.08 | 0.0 |
| X-6-10 | 4.29 | 7.34 | 4.32 | | 3.87 | 3.96 | 0.0 |
| <hr/> | | | | | | | |
| X-6-11 | 4.34 | 7.20 | 4.42 | | | 3.96 R | |
| X-6-12 | 4.40 | 7.20 | 4.47 | | | 3.92 R | |
| X-6-13 | 4.35 | 6.50 | 4.42 | | | 4.00 R | |
| X-6-14 | 4.25 | 7.30 | 4.32 | | | 3.88 R | |
| <hr/> | | | | | | | |
| Avg | 4.37 | | 4.407 | | | 3.94 | |

Oil-Tar Creosote 6% Solution in Benzol

423.9 ml benzol, 23.2 ml creosote

| | | | | | | | |
|------------|-------|-------|-------|----------------|-------|-------|-------|
| 1 | 4.85 | 7.94 | 4.94 | 5.84 | 4.46 | 3.86 | 11.2 |
| 2 | 4.56 | 7.76 | 4.635 | | 4.19 | 3.98 | 9.9 |
| 3 | 4.35 | 7.47 | 4.43 | | 4.00 | 3.46 | 13.5 |
| 4 | 4.39 | 7.35 | 4.46 | 5.00 | 4.03 | 3.64 | 9.7 |
| 5 | 4.56 | 7.59 | 4.62 | 5.30 | 4.17 | 3.76 | 9.8 |
| 6 | 4.41 | 7.60 | 4.46 | 6.60 | 4.03 | 3.66 | 9.1 |
| 7 | 4.80 | 7.74 | 4.86 | 6.37 | 4.39 | 3.30 | 24.8 |
| 8 | 4.66 | 7.84 | 4.725 | 9.20 | 4.27 | 3.95 | 7.5 |
| 9 | 5.10 | 8.24 | 5.19 | 6.22 | 4.69 | 4.09 | 12.8 |
| 10 | 4.84 | 7.95 | 4.91 | 6.00 | 4.43 | 3.84 | 13.3 |
| <hr/> | | | | | | | |
| 11 R | 4.62 | 7.63 | 4.70 | | | 4.27 | |
| 12 R | 4.765 | 8.00 | 4.84 | | | 4.42 | |
| 13 R | 4.93 | 8.01 | 5.00 | | | 4.50 | |
| 14 R | 4.63 | 7.82 | 4.72 | | | 4.20 | |
| <hr/> | | | | | | | |
| Avg | 4.736 | | 4.82 | Avg Oven Dry R | | 4.347 | |

R = Reference blocks

Moisture content = 10.27 and 10.47%

See also M.C. on 5% oil tar = 10.94%

TOXICITY TESTS, SECOND SERIES
Coal-Tar Creosote 8% Solution in Benzol

414.8 ml benzol, 29.76 ml creosote

| <i>Block No.</i> | <i>Original conditioned weight</i> | <i>Weight after treating</i> | <i>Weight after conditioning</i> | <i>Wet weight at end</i> | <i>Computed oven-dry weight</i> | <i>Oven-dry weight</i> | <i>Per cent loss</i> |
|------------------|------------------------------------|------------------------------|----------------------------------|--------------------------|---------------------------------|------------------------|----------------------|
| C-8-1 | 4.69 | 7.78 | 4.845 | | 4.43 | 4.39 | 0.9 |
| C-8-2 | 4.36 | 7.47 | 4.52 | | 4.15 | 4.03 | 2.8 |
| C-8-3 | 4.22 | 7.40 | 4.37 | | 4.01 | 3.95 | 1.4 |
| C-8-4 | 4.17 | 7.19 | 4.30 | 5.05 | 3.95 | 3.83 | 3.0 |
| C-8-5 | 4.46 | 7.53 | 4.56 | | 4.19 | 4.05 | 3.3 |
| C-8-6 | 4.67 | 7.08 | 4.74 | 6.50 | 4.35 | 4.22 | 3.0 |
| C-8-7 | 4.56 | 7.81 | 4.70 | | 4.31 | 4.16 | 3.4 |
| C-8-8 | 4.57 | 7.24 | 4.68 | 6.73 | 4.30 | 4.21 | 2.0 |
| C-8-9 | 4.315 | 7.15 | 4.435 | 5.25 | 4.07 | 3.97 | 2.4 |
| C-8-10 | 4.322 | 7.35 | 4.455 | | 4.09 | 3.98 | 3.9 |
| <hr/> | | | | | | | |
| C-8-11 | 4.32 | 7.10 | 4.435 | | | 4.04 R | |
| C-8-12 | 4.75 | 4.52 | 4.88 | | | 4.32 R | |
| C-8-13 | 4.33 | 7.27 | 4.43 | | | 4.30 R | |
| C-8-14 | 4.36 | 7.56 | 4.47 | | | 4.08 | |
| <hr/> | | | | | | | |
| Avg | 4.44 | | 4.554 | | | 4.185 | |

Moisture content = 10.38%, 10.88%, 10.5%, and 10.03%.

Oil-Tar Creosote 8% Solution in Benzol

414.8 ml benzol, 31.0 ml creosote

| | | | | | | | |
|--------------|-------|-------|-------|-------|-------|--------|-------|
| O-8-1 | 4.58 | 7.53 | 4.72 | | 4.22 | 3.86 | 8.5 |
| O-8-2 | 4.18 | 7.00 | 4.185 | | 4.74 | 3.53 | 5.6 |
| O-8-3 | 4.412 | 7.35 | 4.495 | | 4.02 | 5.92 | 2.2 |
| O-8-4 | 4.35 | 7.35 | 4.465 | | 4.04 | 3.92 | 2.9 |
| O-8-5 | 4.42 | 7.10 | 4.545 | | 4.06 | 3.89 | 4.1 |
| O-8-6 | 4.76 | 7.48 | 4.87 | 5.88 | 4.35 | 4.16 | 4.3 |
| O-8-7 | 4.35 | 7.21 | 4.445 | | 3.98 | 3.68 | 7.3 |
| O-8-8 | 4.417 | 7.39 | 4.51 | 5.37 | 4.03 | 4.00 | 0.0 |
| O-8-9 | 4.21 | 7.35 | 4.335 | 5.17 | 3.88 | 3.74 | 3.6 |
| O-8-10 | 4.40 | 7.28 | 4.54 | | 4.06 | 3.97 | 2.2 |
| <hr/> | | | | | | | |
| O-8-11 | 4.31 | 7.22 | 4.44 | | | 3.95 R | |
| O-8-12 | 4.175 | 7.36 | 4.315 | | | 3.84 | |
| O-8-13 | 4.55 | 7.42 | 4.67 | | | 4.22 R | |
| O-8-14 | 4.305 | 7.35 | 4.425 | | | 3.95 R | |
| <hr/> | | | | | | | |
| Avg | 4.425 | | 4.46 | | | 3.99 | |

Moisture content = 10.09% and 11.0%.

TOXICITY AND VOLATILIZATION TESTS, SECOND SERIES

Coal-Tar Creosote 5% Solution in Benzol

| Block No. | 1,262.6 ml benzol | | | | 55.7 ml creosote | | |
|--------------|-----------------------------------|-----------------------------|---------------------------------|-------------------------|--------------------------------|--------------------|---------------------|
| | Original conditioned weight | Weight after treating | Weight after conditioning | Wet weight at end | Computed oven-dry weight | Oven-dry weight | Per cent loss |
| X-8-1 | 4.95 | 7.75 | 5.00 | 11.58 | 4.51 | 2.87 | 36.1 |
| X-8-2 | 4.63 | 7.33 | 4.66 | 9.85 | 4.21 | 2.80 | 33.4 |
| X-8-3 | 4.35 | 7.34 | 4.41 | 12.52 | 3.98 | 3.48 | 12.5 |
| X-8-4 | 4.24 | 7.46 | 4.285 | 5.68 | 3.87 | 2.00 | 48.3 |
| X-8-5 | 4.85 | 7.90 | 4.88 | 12.31 | 4.41 | 2.95 | 33.1 |
| X-8-6 | 4.31 | 7.38 | 4.335 | 5.00 | 3.91 | 3.00 | 23.2 |
| X-8-7 | 4.32 | 7.21 | 4.35 | 5.30 | 3.92 | 3.09 | 21.1 |
| X-8-8 | 4.15 | 7.37 | 4.18 | | 3.77 | 2.74 | 27.3 |
| X-8-9 | 4.62 | 7.83 | 4.66 | 11.00 | 4.21 | 2.75 | 34.6 |
| X-8-10 | 4.56 | 7.68 | 4.58 | 7.70 | 4.13 | 2.44 | 40.9 |
| <hr/> | | | | | | | |
| X-8-11 | 4.49 | 7.77 | 4.535 | | | 4.10 | |
| X-8-12 | 4.35 | 7.54 | 4.40 | | | 4.00 | |
| X-8-13 | 4.21 | 7.40 | 4.29 | | | 3.86 | |
| X-8-14 | 4.42 | 7.72 | 4.48 | | | 4.03 | |
| <hr/> | | | | | | | |
| Avg | 4.39 | | 4.429 | | | 3.997 | |
| <hr/> | | | | | | | |
| X-8-15 | 4.82 | 7.88 | 4.87 | 9.00 | 4.40 | 3.63 | 17.5 |
| X-8-16 | 4.80 | 7.92 | | | 4.391 | | |
| X-8-17 | 4.205 | 7.47 | 4.21 | 9.17 | 3.81 | 3.00 | 21.4 |
| X-8-18 | 4.35 | 7.44 | 4.38 | 9.52 | 3.96 | 2.58 | 34.8 |
| X-8-19 | 4.28 | 7.32 | 4.32 | 7.52 | 3.91 | 2.49 | 36.3 |
| X-8-20 | 4.55 | 7.59 | 4.58 | 11.62 | 4.14 | 3.00 | 27.5 |
| X-8-21 | 4.22 | 7.30 | 4.28 | 8.22 | 3.87 | 3.31 | 14.4 |
| X-8-22 | 4.50 | 7.49 | 4.54 | 6.72 | 4.10 | 3.00 | 26.8 |
| X-8-23 | 4.78 | 7.16 | 4.87 | 6.12 | 4.40 | 3.60 | 18.1 |
| X-8-24 | 4.47 | 7.32 | 4.55 | 6.18 | 4.11 | 3.00 | 27.0 |
| <hr/> | | | | | | | |
| X-8-25 | 4.833 | 7.71 | 4.89 | | | 4.43 | |
| X-8-26 | 4.87 | 7.82 | 4.93 | | | 4.43 | |
| X-8-27 | 4.41 | 7.05 | 4.47 | | | 4.01 | |
| X-8-28 | 4.79 | 7.35 | 4.83 | | | 4.42 | |
| <hr/> | | | | | | | |
| Avg | 4.726 | | 4.78 | | | 4.3225 | |

TOXICITY AND VOLATILIZATION TESTS, SECOND SERIES

Oil-Tar Creosote 5% Solution in Benzol

1,262.6 ml benzol, 58.1 ml creosote

| <i>Block No.</i> | <i>Original conditioned weight</i> | <i>Weight after treating</i> | <i>Weight after conditioning</i> | <i>Wet weight at end</i> | <i>Computed oven-dry weight</i> | <i>Oven-dry weight</i> | <i>Per cent loss</i> |
|------------------|------------------------------------|------------------------------|----------------------------------|--------------------------|---------------------------------|------------------------|----------------------|
| O-5-1 | 4.32 | 7.50 | 4.36 | 6.07 | 3.96 | 2.35 | 46.5 |
| O-5-2 | 4.69 | 7.80 | 4.74 | 6.82 | 4.31 | 2.77 | 35.7 |
| O-5-3 | 4.18 | 7.67 | 4.22 | 6.60 | 3.84 | 2.45 | 36.2 |
| O-5-4 | 4.52 | 7.49 | 4.57 | 15.42 | 4.15 | 3.93 | 5.3 |
| O-5-5 | 4.64 | 8.01 | 4.67 | 11.66 | 4.25 | 3.95 | 7.0 |
| O-5-6 | 4.30 | 7.61 | 4.35 | 6.65 | 3.96 | 2.64 | 31.0 |
| O-5-7 | 4.28 | 7.70 | 4.33 | 11.00 | 3.94 | 3.37 | 14.5 |
| O-5-8 | 4.63 | 7.57 | 4.70 | | 4.27 | 2.79 | 34.6 |
| O-5-9 | 4.66 | 7.82 | 4.75 | 8.00 | 4.32 | | |
| O-5-10 | 5.00 | 8.23 | 5.01 | 8.49 | 4.35 | 2.90 | 36.2 |
| O-5-11 | 4.28 | 7.27 | 4.34 | | | 3.95 | |
| O-5-12 | 4.83 | 7.95 | 4.87 | | | 4.44 | |
| O-5-13 | 4.64 | 7.68 | 4.71 | | | 4.28 | |
| O-5-14 | 4.715 | 7.90 | 4.78 | | | 4.34 | |
| Avg | 4.616 | | 4.675 | | | 4.25 | |
| O-5-15 | 4.65 | 7.88 | 4.71 | 11.78 | 4.25 | 4.00 | 5.88 |
| O-5-16 | 4.57 | 7.71 | 4.57 | 13.84 | 4.12 | 4.03 | 2.2 |
| O-5-17 | 4.33 | 7.68 | 4.35 | 12.92 | 3.92 | 3.50 | 18.7 |
| O-5-18 | 4.75 | 7.80 | 4.78 | 11.48 | 4.30 | 3.95 | 8.1 |
| O-5-19 | 4.56 | 7.66 | 4.59 | 6.13 | 4.14 | 2.87 | 30.7 |
| O-5-20 | 4.33 | 7.23 | 4.36 | 13.50 | 3.93 | 3.95 | 1.2 |
| O-5-21 | 4.30 | 7.49 | 4.30 | | 3.88 | 3.93 | 0.0 |
| O-5-22 | 4.40 | 7.87 | 4.41 | | 3.98 | 3.00 | 24.6 |
| O-5-23 | 4.51 | 7.80 | 4.55 | 13.10 | 4.11 | 4.08 | 0.7 |
| O-5-24 | 4.45 | 7.85 | 4.515 | 13.75 | 4.07 | 4.00 | 1.7 |
| O-5-25 | 4.50 | 7.85 | 4.51 | 11.50 | 4.07 | 4.00 | 1.7 |
| O-5-26 | 4.28 | 7.63 | 4.31 | | | 3.84 | |
| O-5-27 | 4.315 | 7.79 | 4.33 | | | 3.86 | |
| O-5-28 | 4.37 | 7.47 | 4.23 | | | 3.94 | |
| O-5-29 | 4.12 | 7.48 | 4.15 | | | 3.73 | |
| Avg | 4.24 | | 4.255 | | | 3.84 | |

Moisture content = 10.81%, 10.99%, and 12.16% = 10.94%

TOXICITY AND VOLATILIZATION TESTS, SECOND SERIES

Coal-Tar Creosote 10% Solution in Benzol

1116.1 ml benzol, 102.3 ml creosote

| <i>Block No.</i> | <i>Original conditioned weight</i> | <i>Weight after treating</i> | <i>Weight after conditioning</i> | <i>Wet weight at end</i> | <i>Computed oven-dry weight</i> | <i>Oven-dry weight</i> | <i>Per cent loss</i> |
|------------------|------------------------------------|------------------------------|----------------------------------|--------------------------|---------------------------------|------------------------|----------------------|
| X-10-1 | 4.46 | 7.845 | 4.66 | 5.63 | 4.21 | 3.80 | 9.7 |
| X-10-2 | 4.52 | 7.77 | 4.73 | 7.48 | 4.27 | 3.73 | 12.6 |
| X-10-3 | 4.87 | 8.19 | 5.05 | 6.48 | 4.56 | 3.05 | 33.1 |
| X-10-4 | 4.56 | 8.00 | 4.73 | 8.00 | 4.27 | 3.76 | 11.9 |
| X-10-5 | 4.605 | 8.14 | 4.84 | 5.20 | 4.37 | 3.57 | 18.3 |
| X-10-6 | 4.52 | 7.96 | 4.72 | 5.54 | 4.27 | 5.39 | 20.6 |
| X-10-7 | 4.67 | 8.29 | 4.905 | 8.65 | 4.43 | 4.24 | 4.2 |
| X-10-8 | 4.45 | 7.61 | 4.63 | 10.38 | 4.18 | 4.07 | 2.6 |
| X-10-9 | 4.86 | 8.30 | 5.09 | 13.68 | 4.60 | 4.251 | 7.5 |
| X-10-10 | 4.61 | 7.58 | 4.775 | 12.18 | 4.31 | 3.89 | 9.7 |
| <hr/> | | | | | | | |
| X-10-11 | 4.53 | 7.78 | 4.73 | | 4.25 | | |
| X-10-12 | 4.36 | 7.31 | 4.55 | | 4.12 | | |
| X-10-13 | 4.80 | 8.01 | 5.00 | | 4.51 | | |
| X-10-14 | 4.62 | 7.46 | 4.76 | | 4.33 | | |
| <hr/> | | | | | | | |
| Avg | 4.55 | | 4.76 | | 4.30 | | |
| <hr/> | | | | | | | |
| X-10-15 | 4.45 | 7.77 | 4.64 | 7.25 | 4.09 | 3.16 | 22.7 |
| X-10-16 | 4.65 | 7.83 | 4.85 | 7.31 | 4.18 | 3.13 | 26.8 |
| X-10-17 | 4.475 | 7.69 | 4.66 | 6.00 | 4.11 | 2.78 | 32.3 |
| X-10-18 | 4.49 | 7.43 | 4.65 | 8.00 | 4.10 | 2.36 | 42.4 |
| X-10-19 | 4.74 | 8.09 | 4.94 | 11.93 | 4.36 | 2.29 | 47.4 |
| X-10-20 | 4.78 | 8.08 | 5.00 | 7.40 | 4.41 | 3.00 | 31.9 |
| X-10-21 | 4.70 | 7.59 | 4.81 | 10.72 | 4.24 | 3.52 | 17.8 |
| X-10-22 | 4.48 | 7.57 | 4.69 | 7.62 | 4.13 | 2.72 | 34.1 |
| X-10-23 | 4.60 | 7.77 | 4.77 | 7.55 | 4.21 | 2.53 | 39.9 |
| X-10-24 | 4.55 | 7.61 | 4.71 | | 4.15 | 3.16 | 23.8 |
| <hr/> | | | | | | | |
| X-10-25 | 4.71 | 8.23 | 4.91 | | 4.34 | | |
| X-10-26 | 4.69 | 8.17 | 4.90 | | 4.33 | | |
| X-10-27 | 4.635 | 7.56 | 4.83 | | 4.24 | | |
| X-10-28 | 4.58 | 8.07 | 4.83 | | 4.27 | | |
| <hr/> | | | | | | | |
| Avg | 4.65 | | 4.87 | | 4.295 | | |

Moisture content = 10.24%, 11.79%, and 10.65%

TOXICITY AND VOLATILIZATION TESTS, SECOND SERIES

Oil-Tar Creosote 10% Solution in Benzol

1014.6 ml benzol, 96.89 ml creosote

| <i>Block No.</i> | <i>Original conditioned weight</i> | <i>Weight after treating</i> | <i>Weight after conditioning</i> | <i>Wet weight at end</i> | <i>Computed oven-dry weight</i> | <i>Oven-dry weight</i> | <i>Per cent loss</i> |
|------------------|------------------------------------|------------------------------|----------------------------------|--------------------------|---------------------------------|------------------------|----------------------|
| O-10-1 | 4.58 | 7.56 | 4.75 | 8.55 | 4.27 | 4.19 | 1.8 |
| O-10-2 | 4.59 | 8.04 | 4.79 | 10.55 | 4.31 | 4.25 | 1.39 |
| O-10-3 | 4.72 | 7.79 | 4.90 | 7.60 | 4.41 | 4.11 | 6.8 |
| O-10-4 | 4.55 | 7.63 | 4.72 | 7.08 | 4.25 | 3.98 | 6.3 |
| O-10-5 | 4.48 | 7.52 | 4.63 | 8.59 | 4.17 | 4.05 | 2.8 |
| O-10-6 | 4.29 | 7.39 | 4.48 | 9.00 | 4.03 | 3.92 | 2.7 |
| O-10-7 | 4.57 | 7.50 | 4.75 | 13.82 | 4.27 | 4.12 | 3.5 |
| O-10-8 | 4.803 | 8.05 | 5.00 | 6.72 | 4.50 | 3.50 | 22.2 |
| O-10-9 | 4.37 | 7.46 | 4.55 | 12.79 | 4.09 | 4.12 | 0.0 |
| O-10-10 | 4.49 | 7.65 | 4.68 | 10.70 | 4.21 | | 0.0 |
| <hr/> | | | | | | | |
| O-10-11 | 4.59 | 7.92 | 4.77 | | 4.30 | | |
| O-10-12 | 4.70 | 7.60 | 4.87 | | 4.40 | | |
| O-10-13 | 4.70 | 7.93 | 4.93 | | 4.41 | | |
| O-10-14 | 4.78 | 8.22 | 4.99 | | 4.48 | | |
| <hr/> | | | | | | | |
| Avg | 4.69 | | 4.89 | | 4.40 | | |
| <hr/> | | | | | | | |
| O-10-15 | 4.66 | 7.92 | 4.86 | 5.61 | 4.30 | 4.17 | 3.0 |
| O-10-16 | 4.60 | 7.63 | 4.745 | 6.05 | 4.20 | 4.12 | 1.9 |
| O-10-17 | 4.665 | 7.51 | 4.76 | 9.19 | 4.21 | 4.22 | 0.0 |
| O-10-18 | 4.66 | 7.93 | 4.835 | 7.32 | 4.28 | 4.00 | 4.6 |
| O-10-19 | 4.38 | 7.22 | 4.535 | 5.24 | 4.01 | 3.96 | 1.2 |
| O-10-20 | 5.06 | 8.08 | 5.16 | 6.90 | 4.57 | 4.02 | 12.0 |
| O-10-21 | 4.72 | 7.54 | 4.86 | 10.52 | 4.30 | 3.55 | 17.4 |
| O-10-22 | 4.46 | 7.26 | 4.60 | 9.70 | 4.07 | 2.34 | 42.5 |
| O-10-23 | 4.66 | 7.33 | 4.77 | 10.10 | 4.22 | 2.63 | 37.6 |
| O-10-24 | 4.26 | 6.98 | 4.385 | 7.55 | 3.88 | 2.65 | 31.7 |
| <hr/> | | | | | | | |
| O-10-25 | 4.75 | 7.40 | 4.84 | | 4.29 | | |
| O-10-26 | 4.29 | 7.02 | 4.41 | | 3.90 | | |
| O-10-27 | 4.64 | 7.46 | 4.78 | | 4.21 | | |
| O-10-28 | 4.66 | 7.50 | 4.77 | | 4.23 | | |
| <hr/> | | | | | | | |
| Avg | 4.59 | | 4.70 | | 4.16 | | |

Moisture content = 12.7%, 10.4%, and 11.02%

Table Showing Action of *Lenzites trabea* on Benzol-Treated and Untreated Controls.

CONTROLS

| Key | Original conditioned weight | Wet weight at end | Computed oven-dry weight | Oven-dry weight | Per cent loss |
|-----------|-----------------------------|-------------------|--------------------------|-----------------|---------------|
| A-1 | 4.33 | | 3.904 | 3.07 | 21.4 |
| A-2 | 4.505 | | 4.06 | 3.22 | 20.7 |
| A-3 | 4.69 | | 4.38 | 3.52 | 14.9 |
| A-4 | 4.56 | 10.76 | 4.12 | 2.90 | 28.36 |
| A-5 | 4.68 | | 4.22 | 3.00 | 28.9 |

Moisture content = O. T. — 6% = 10.94%

| | | | | | |
|-----------|-------|-------|-------|------|-------|
| B-1 | 4.38 | | 3.86 | 3.18 | 17.6* |
| B-2 | 4.37 | | 3.94 | 2.43 | 38.3 |
| B-3 | 4.23 | 8.17 | 3.817 | 2.57 | 32.16 |
| B-4 | 3.935 | 8.32 | 3.55 | 2.41 | 32.1 |
| B-5 | 4.295 | 8.34 | 3.876 | 2.40 | 38.0 |
| B-6 | 4.29 | 10.20 | 3.763 | 2.49 | 33.85 |

Moisture content = O. T. and C. T. at 3% = 10.76%

| | | | | | |
|-----------|------|-------|-------|------|-------|
| C-1 | 4.68 | 7.00 | 4.22 | 3.36 | 20.4 |
| C-2 | 4.47 | 6.15 | 4.039 | 3.00 | 25.7 |
| C-3 | 4.26 | 10.15 | 3.84 | 3.00 | 21.87 |
| C-4 | 4.43 | | 3.99 | 3.08 | 23.2 |
| C-5 | 4.24 | | 3.82 | 2.65 | 30.6 |
| C-6 | 4.97 | 6.26 | 4.391 | 2.96 | 32.59 |

Moisture content = 10.85%

BENZOL-TREATED CONTROLS

| Block No. | Original conditioned weight | Wet weight at end | Computed oven-dry weight | Oven-dry weight | Per cent loss |
|-----------|-----------------------------|-------------------|--------------------------|-----------------|---------------|
| 1 | 4.16 | 10.45 | 3.85 | 2.37 | 38.4 |
| 2 | 4.90 | 6.45 | 4.54 | 2.85 | 37.2 |
| 3 | 4.17 | 6.08 | 3.86 | 2.51 | 34.9 |
| 4 | 4.265 | 9.24 | 3.94 | 2.76 | 30.0 |

Avg 35.1

Moisture content = Room Dry — 8%

* Six weeks.

5. **Toxicity test results (second series).** The results of the second series of toxicity tests are given in the following condensed tables. The percentages that are underlined have not been considered as true samples because of probable experimental error; the samples had taken up excessive moisture from the agar, were dried out during the incubation period, or had deviated too far from the mean.

The data on which the first series curves are based will be found in the tabulations in Section 9, Chapter V. Although the number of samples near the inhibiting point is not adequate for statistical analysis, the trend of the curve is corroborated by the results of the second series of tests.

Summary Table Showing the Per Cent Loss in Weight of Wood Blocks
Treated with Different Concentrations of Creosote and Sub-
sequently Exposed to *Lenzites trabea* for 64 Days.

OIL-TAR CREOSOTED SAMPLES

| Block No. | Concentration of treating solutions | | | |
|------------|-------------------------------------|-------------|-------|------|
| | 1% | 3% | 6% | 8% |
| 1 | 35.1 | 32.6 | 11.2 | 8.5 |
| 2 | 30.62 | 34.8 | 9.9 | 5.6 |
| 3 | 32.1 | 35.2 | 13.5 | 2.2 |
| 4 | | 9.4 | 9.7 | 4.1 |
| 5 | | <u>11.1</u> | 9.8 | 4.3 |
| 6 | | 31.8 | 9.1 | 7.3 |
| 7 | | 45.5 | 24.8 | 0.0 |
| 8 | | 43.7 | 7.5 | 3.6 |
| 9 | | 36.2 | 12.8 | 2.2 |
| 10 | | 36.5 | 13.3 | 0.0 |
| Mean | 32.61 | 37.03 | 12.16 | 3.78 |

COAL-TAR CREOSOTED SAMPLES

| | | | | |
|------------|------|-------------|------------|------------|
| 1 | 50.0 | 38.7 | 0.0 | 0.9 |
| 2 | 50.0 | <u>17.6</u> | <u>0.0</u> | 2.8 |
| 3 | 60.2 | 37.3 | <u>0.0</u> | <u>1.4</u> |
| 4 | | 15.6 | <u>0.0</u> | 3.0 |
| 5 | | <u>21.7</u> | 0.0 | 3.3 |
| 6 | | 8.4 | 0.0 | 3.0 |
| 7 | | 14.5 | 5.7 | 3.4 |
| 8 | | <u>10.0</u> | 4.3 | 2.0 |
| 9 | | 35.3 | 0.0 | 2.4 |
| 10 | | 39.0 | 0.0 | 2.7 |
| Mean | 53.4 | 37.57 | 1.43 | 2.61 |

The above data are also shown on the accompanying graphs. The results of the first or orientation series are also shown on the same graphs (Figures 14 and 15).

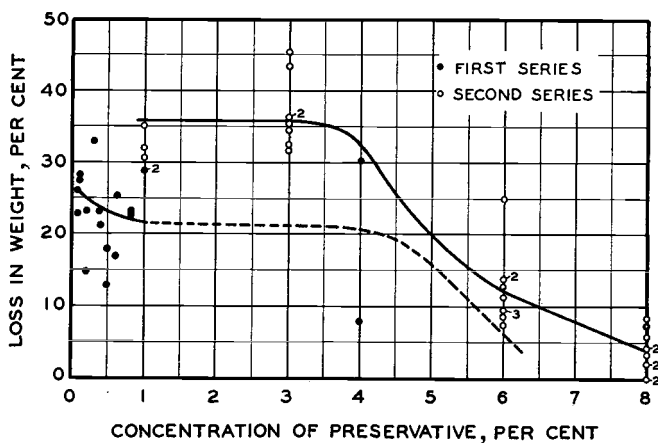


Figure 14. TOXICITY OF OIL-TAR CREOSOTE TO *Lenzites trabea*.

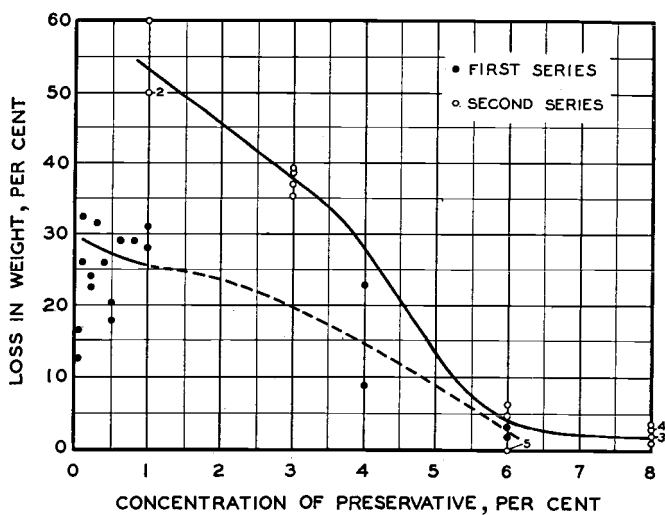
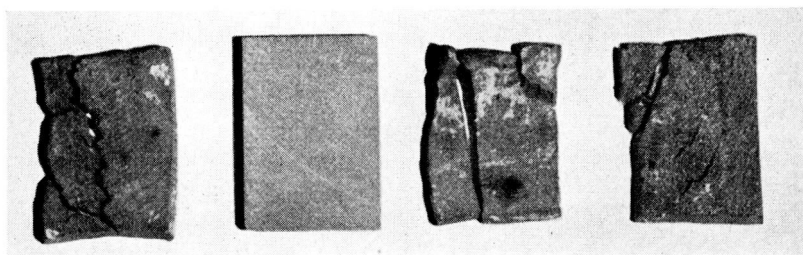


Figure 15. TOXICITY OF COAL-TAR CREOSOTE TO *Lenzites trabea*.

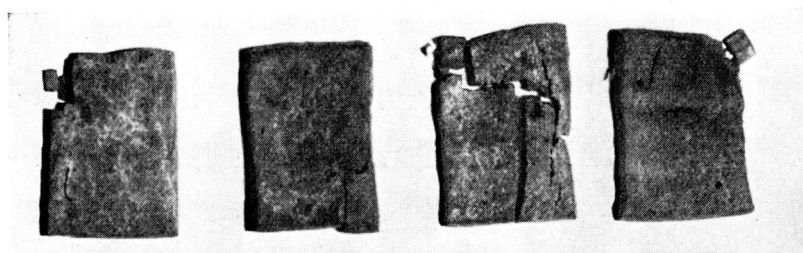


O-1-1

Original block

100% benzol
treated control

O-1-3



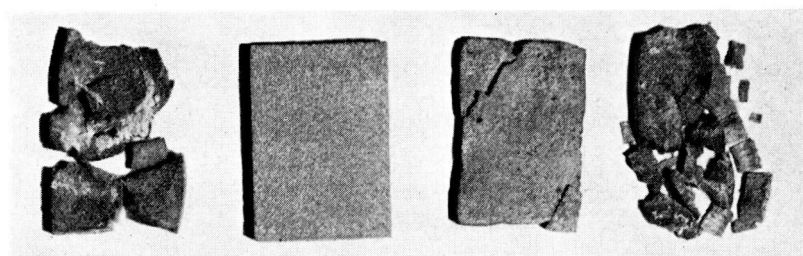
O-3-3

O-3-6

O-3-9

O-3-1

BLOCKS TREATED WITH 3% SOLUTION OF OIL-TAR CREOSOTE

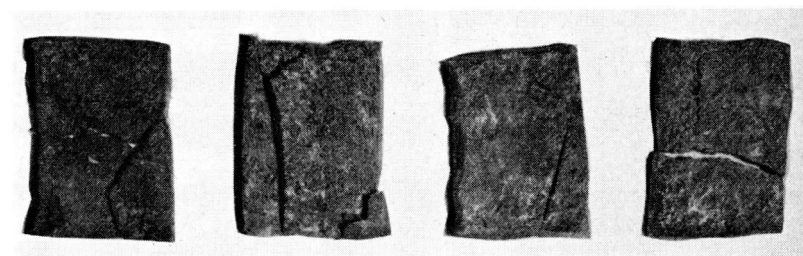


C-1-1

Original block

Untreated control

C-1-3



C-3-1

C-3-3

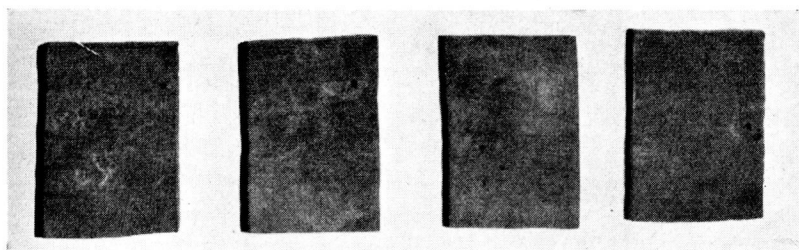
C-3-9

C-3-10

BLOCKS TREATED WITH 3% SOLUTION OF COAL-TAR CREOSOTE

Figure 16. REPRESENTATIVE OVEN-DRY SAMPLE BLOCKS FROM SECOND SERIES AFTER EIGHT WEEKS INCUBATION WITH *Lenzites trabea*.

(KEY NUMBERS OF BLOCKS CORRESPOND TO KEY NUMBERS IN TABLES.) BLOCKS NOT VOLATILIZED OR LEACHED. (TREATED WITH 1% AND 3% SOLUTIONS.)



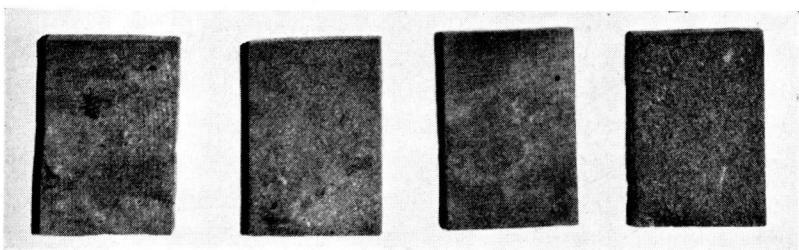
O-6-1

O-6-3

O-6-6

O-6-9

BLOCKS TREATED WITH 6% SOLUTION OF OIL-TAR CREOSOTE



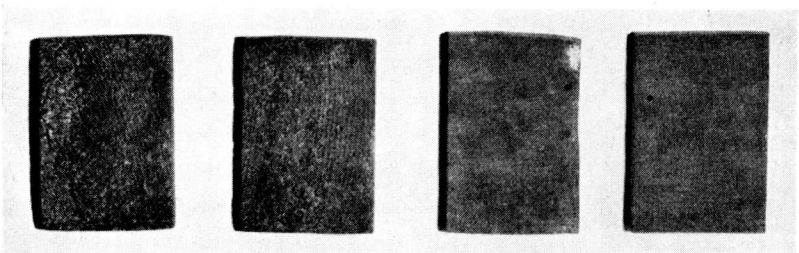
O-8-1

O-8-3

O-8-6

O-8-9

BLOCKS TREATED WITH 8% SOLUTION OF OIL-TAR CREOSOTE



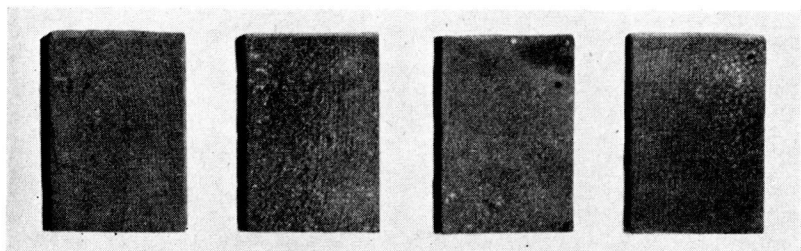
X-6-5

X-6-6

X-6-7

X-6-8

BLOCKS TREATED WITH 6% SOLUTION OF COAL-TAR CREOSOTE



C-8-1

C-8-3

C-8-6

C-8-9

BLOCKS TREATED WITH 8% SOLUTION OF COAL-TAR CREOSOTE

Figure 17. REPRESENTATIVE OVEN-DRY SAMPLE BLOCKS FROM SECOND SERIES AFTER EIGHT WEEKS INCUBATION WITH *Lenzites trabea*.
(KEY NUMBERS OF BLOCKS CORRESPOND TO KEY NUMBERS IN TABLES.) BLOCKS NOT VOLATILIZED OR LEACHED. (TREATED WITH 6% AND 8% SOLUTIONS.)

6. **Volatility and leaching test results (second series).** The results of the second series of tests on volatilization and leaching are shown in the following table.

Table Showing the Per Cent Loss in Weight of Treated Wood Blocks after Volatilization at 160 F in the Oven for 24 Hours and Subsequently Incubated with *Lenzites trabea* for 8 Weeks.

| | <i>Oil-tar creosote 5% solution</i> | <i>Coal-tar creosote 5% solution</i> | <i>Oil-tar creosote 10% solution</i> | <i>Coal-tar creosote 10% solution</i> |
|------------|---|--|--|---|
| | 5.3 | <u>12.5</u> | 0.0 | 2.6 |
| | 7.0 | 21.1 | 0.0 | 4.2 |
| | <u>14.5</u> | 23.2 | 1.3 | <u>7.5</u> |
| | 31.0 | 27.3 | 1.8 | 9.7 |
| | 34.6 | 33.1 | 2.7 | 9.7 |
| | 35.7 | 33.4 | 2.8 | 11.9 |
| | 36.2 | 34.6 | 3.5 | 12.6 |
| | 36.2 | 36.1 | 6.3 | 18.3 |
| | 46.5 | 40.9 | 6.8 | 20.6 |
| | Lost | 48.3 | 22.2 | 23.1 |
| Mean | 36.7 | 33.1 | 5.92 | 16.56 |

(Samples above the underscore lines rejected on the basis of experimental error; i.e., samples varied too greatly from mean due to high moisture content of sample during incubation or desiccation of sample during incubation.)

The above results are shown in the accompanying bar chart (Figure 18). Each horizontal division on the chart indicates one sample.

Table Showing the Per Cent Loss in Weight of Treated Wood Blocks after Volatilization at 160 F for 24 Hours, Leaching with Water for 5 Days, and Subsequently Incubated with *Lenzites trabea* for a Period of 8 Weeks.

| | <i>Oil-tar creosote 5% solution</i> | <i>Coal-tar creosote 5% solution</i> | <i>Oil-tar creosote 10% solution</i> | <i>Coal-tar creosote 10% solution</i> |
|------------|---|--|--|---|
| | 0.7 | 14.4 | 0.0 | <u>17.8</u> |
| | 1.2 | 17.5 | 1.2 | <u>22.7</u> |
| | 1.7 | <u>18.1</u> | 1.9 | 23.8 |
| | 1.7 | 21.4 | 3.0 | 26.8 |
| | 2.2 | 26.8 | 4.6 | 31.9 |
| | 5.88 | 27.0 | 12.0 | 32.3 |
| | 8.1 | 27.5 | <u>17.4</u> | 34.1 |
| | <u>10.7</u> | 34.8 | 31.7 | 39.9 |
| | 24.6 | 34.8 | 37.6 | 42.4 |
| | 30.7 | Lost | 42.5 | 47.4 |
| Mean | 27.65 | 28.71 | 37.26 | 33.48 |

(Samples above underscore lines rejected as shown above.)

The above results are shown in the accompanying bar chart (Figure 18). Each horizontal division on the chart indicates one sample.

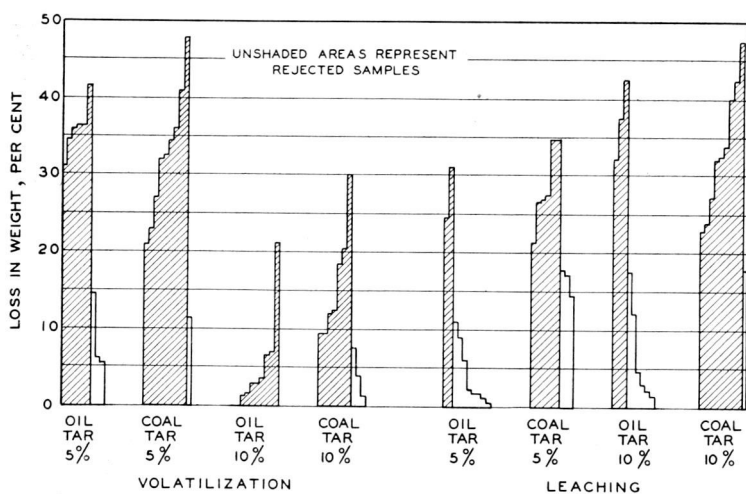
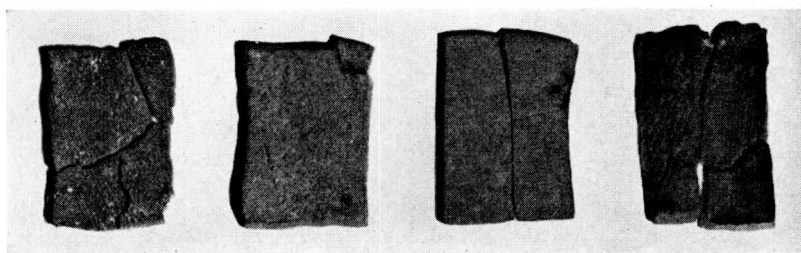
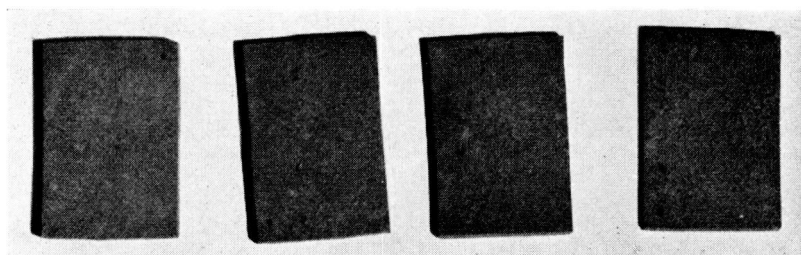


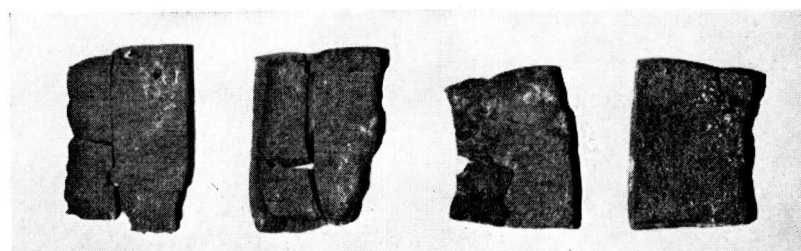
Figure 18. DECAY OF CREOSOTED WOOD BLOCKS AFTER VOLATILIZATION AND LEACHING. (UNSHADED AREAS REPRESENT REJECTED SAMPLES.)



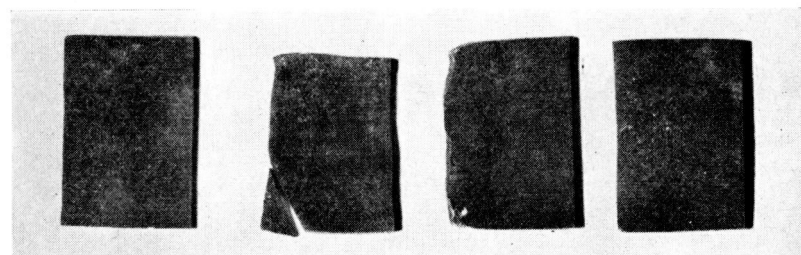
O-5-1 O-5-6 O-5-8 O-5-10
VOLATILITY TEST BLOCKS TREATED WITH 5% SOLUTION OF OIL-TAR CREOSOTE



O-10-1 O-10-3 O-10-4 O-10-6
VOLATILITY TEST BLOCKS TREATED WITH 10% SOLUTION OF OIL-TAR CREOSOTE



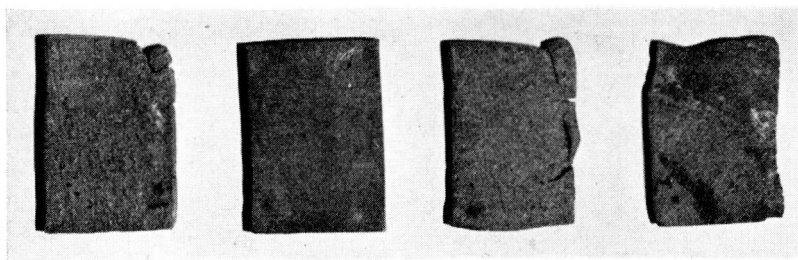
X-8-1 X-8-2 X-8-4 X-8-10
VOLATILITY TEST BLOCKS TREATED WITH 5% SOLUTION OF COAL-TAR CREOSOTE



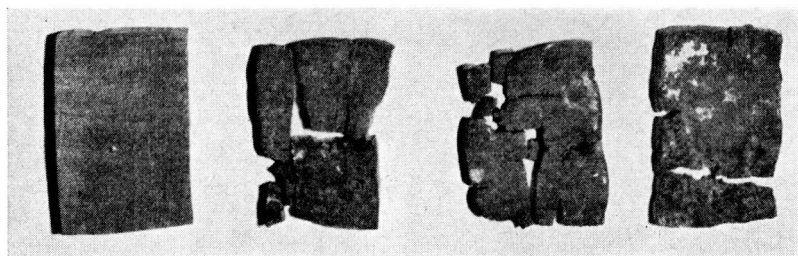
X-10-1 X-10-3 X-10-4 X-10-5
VOLATILITY TEST BLOCKS TREATED WITH 10% SOLUTION OF COAL-TAR CREOSOTE

Figure 19. REPRESENTATIVE OVEN-DRY SAMPLE BLOCKS FROM SECOND SERIES AFTER EIGHT WEEKS INCUBATION WITH *Lenzites trabea*.

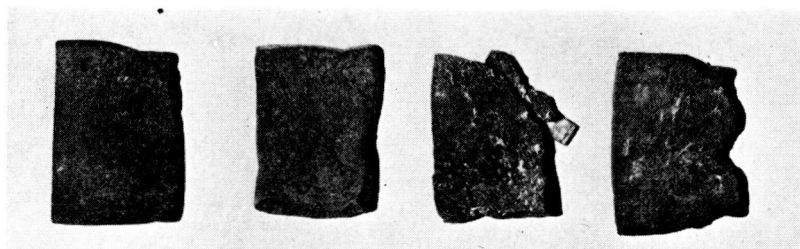
BLOCKS SUBJECTED TO THE STANDARD VOLATILIZATION TEST. (KEY NUMBERS OF BLOCKS CORRESPOND TO KEY NUMBERS IN TABLES.)



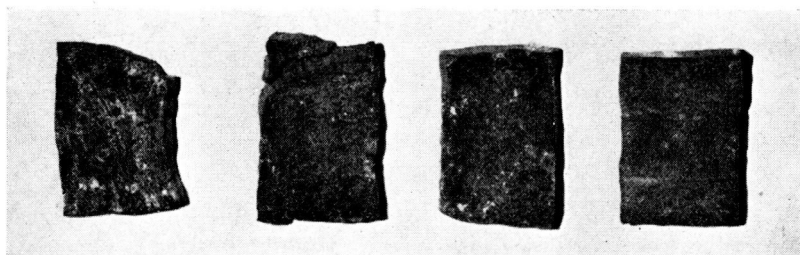
O-5-17 O-5-18 O-5-19 O-5-22
LEACHING TEST BLOCKS TREATED WITH 10% SOLUTION OF OIL-TAR CREOSOTE



O-10-20 O-10-22 O-10-23 O-10-24
LEACHING TEST BLOCKS TREATED WITH 5% SOLUTION OF COAL-TAR CREOSOTE



X-8-17 X-8-18 X-8-19 X-8-20
LEACHING TEST BLOCKS TREATED WITH 5% SOLUTION OF OIL-TAR CREOSOTE



X-10-18 X-10-19 X-10-20 X-10-22
LEACHING TEST BLOCKS TREATED WITH 10% SOLUTION OF OIL-TAR CREOSOTE

Figure 20. REPRESENTATIVE OVEN-DRY SAMPLE BLOCKS FROM SECOND SERIES AFTER EIGHT WEEKS INCUBATION WITH *Lenzites trabea*.

BLOCKS SUBJECTED TO THE STANDARD LEACHING TEST. (KEY NUMBERS OF BLOCKS CORRESPOND TO KEY NUMBERS IN TABLES.)

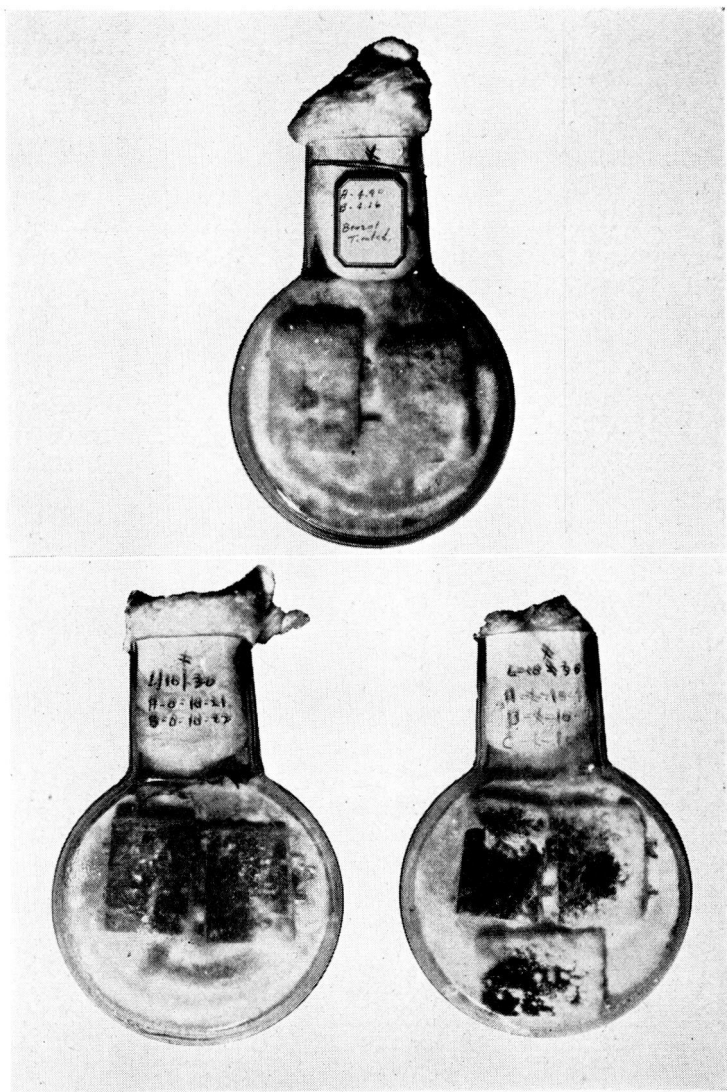


Figure 21. TEST BLOCKS TREATED WITH 10% CREOSOTE SOLUTIONS, SUBJECTED TO THE STANDARD VOLATILIZATION AND LEACHING TESTS, AND SUBSEQUENTLY INCUBATED FOR EIGHT WEEKS WITH *Lenzites trabea*.
 (Left, oil-tar-creosoted blocks 21 and 22; right, coal-tar-creosoted blocks 15, 16, and 17.) (Top: CONTROL BLOCKS TREATED WITH 100% BENZOL AND SUBSEQUENTLY INCUBATED FOR 8 WEEKS WITH *Lenzites trabea*.)

7. **Toxicity of the different fractions of oil-tar creosote.** The effectiveness of the various fractions of creosote oil to act as wood preservatives has been the subject of considerable discussion. In an attempt to answer this question in regard to oil-tar creosote as well as to learn more about the toxic properties in relation to the manufacturing procedure, an extensive study of the initial toxicity and toxicity after volatilization of the six fractions of the creosote was made.

The procedure consisted of making a standard distillation of a quantity of the whole oil and then by using the standard N.D.M.A. testing procedure (33), both the initial toxicity and toxicity after volatilization were investigated. A slight variation in the N.D.M.A. procedure was followed; namely, the substitution of benzol for the petroleum solvent recommended and the addition of

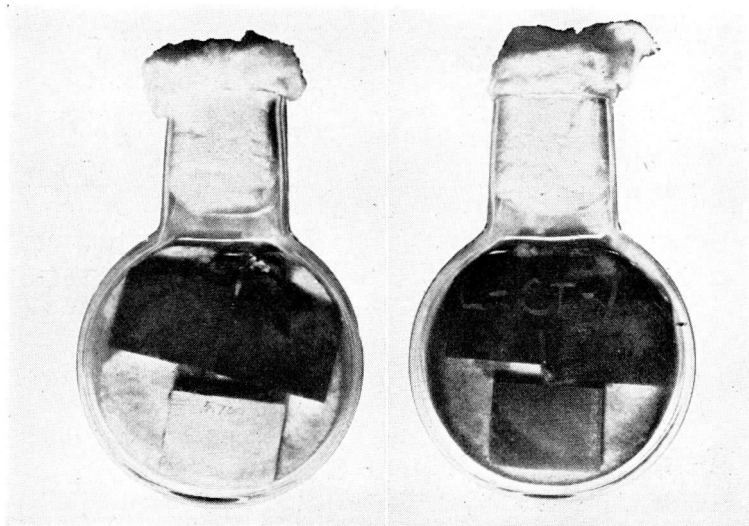


Figure 22. TYPICAL RESULTS OF THE EXPOSURE OF TREATED BLOCKS FROM THE FIRST SERIES TO LEACHING BY WATER AND SUBSEQUENT INCUBATION ON CULTURES OF *Lenzites trabea* FOR A PERIOD OF 8 WEEKS.

(Left, 100% OIL-TAR-CREOSOTE TREATMENT. Right, 100% COAL-TAR-CREOSOTE TREATMENT.)

a heavy neutral oil to prevent blooming of the crystalline materials such as naphthalene. As previously determined, benzol proved to be a desirable solvent, which, after sufficient conditioning, had no retarding effect on the growth of the test fungus, *Lenzites trabea*.

A total of more than 300 test and reference blocks were used in making the study. All of the fractions were studied for both toxicity and resistance to volatilization near the inhibiting point for the growth of the test fungus.

The general conclusion reached in this study is that the fractions that distill above 270 C are more effective, both in initial toxicity and toxicity after volatilization, in controlling the fungus *Lenzites trabea* than the fractions that

distill below 270 C. As found in this study, the fractions are arranged in the order of their effectiveness in the following order.

1. Oils distilling between 315 and 355 C.
2. Oils distilling between 270 and 315 C.
3. Oils distilling above 355 C.
4. Oils distilling between 210 and 235 C.
5. Oils distilling between 235 and 270 C.
6. Oils distilling between 0 and 210 C.

It is believed that the wood-block method used in this investigation is a much better guide to the actual effectiveness of creosote oils than tests that are made by the Petri-dish method. Thus it is shown that such compounds as the naphthalenes, although potentially toxic, are rendered ineffective by rapid volatilization, while the higher-boiling oils are both toxic and permanent.

Although not investigated in this study, it is very probable that many of the coal-tar creosotes would react similarly in the same testing procedure because of the similarity of their chemical constituents.

VII. DISCUSSION OF RESULTS

Inspection of the data discloses two significant facts: first, that the initial toxicity of the coal-tar creosote is slightly greater than the oil-tar creosote, and second, that although the coal-tar creosote may be somewhat more toxic it is less likely to stay in place under service conditions. It must be remembered that these studies are made with dilutions of the creosote oil beyond any practical commercial application. Under commercial treating conditions it is doubtful if these differences would become significant. The fact that the oil-tar creosote is more resistant to volatilization is definitely a point in favor of the oil-tar creosote. This resistance to volatilization can be correlated with the circumstance that the oil-tar creosote has a higher percentage of high-boiling oils than does the coal-tar creosote.

The data further show that there was no significant difference in the attack of the fungus when the blocks were treated with concentrations of less than 3 per cent. In fact the per cent of wood lost through decay in the samples so treated exceeds the loss in the untreated control blocks. At the low concentrations the greater per cent loss may be attributed to the stimulating action of the dilute solutions. This stimulation has been established by other workers as a likely happening (24).

As shown by the data the inhibiting action starts somewhere between the 3- and 6-per-cent concentrations. In the absence of complete data it was assumed that an increase in concentration between 3- and 6-per-cent solutions would have a proportionate effect on the per cent loss in weight of wood due to fungus attack. At the 6-per-cent concentration the oil-tar creosote lost, on the average, 12.16 per cent as against 1.43 per cent in the case of the coal-tar-treated samples.

Examination of the treated blocks showed definite decay in the 6 per cent coal-tar-creosote treated blocks, while the remaining five showed no loss. Three of the samples at this concentration were eliminated because of excessive moisture content at the end of the test. In the 6 per cent oil-tar-creosoted blocks one sample, No. 7, showed an excessive loss. Although this block was out of line with the remainder of the blocks at this concentration, it was

included in the average because it was thought desirable to get as severe a test as possible in evaluating the two oils. This is in spite of probable error.

According to the tabular data presented the average loss in the coal-tar-creosoted blocks, using an 8-per-cent solution, was more than the average loss in the wood blocks treated with a 6-per-cent solution of coal-tar creosote. A recheck of the computations and of the oven-dry weights was made without changing the results. If the standard error of the average is considered, the apparent discrepancy is reduced to 0.55 per cent, which is an error that could be attributed to variations in moisture content due to insufficient time in the desiccator after treating; however, the standard procedure was followed throughout. The blocks treated with the 8-per-cent solution of coal-tar creosote showed no apparent disintegration due to decay. An examination of the blocks treated with an 8-per-cent solution of oil-tar creosote showed blocks No. 1, 2, and 6 to be definitely attacked by the fungus. None of the other blocks gave any surface indication of decay.

In the blocks subjected to the volatilization tests, there appears to be a significant difference in their resistance to decay. The average per cent loss in the blocks treated with a 10-per-cent solution of oil-tar creosote was found to be 5.92 as against an average loss of 16.56 per cent in the blocks treated with a 10-per-cent solution of coal-tar creosote. In analyzing these data some of the samples were rejected on the same basis as mentioned previously; namely, moisture conditions or probable error. The rejected samples are shown along with the accepted samples in Figure 18 by the unshaded portions of the chart. There appeared to be no significant difference in the samples treated with a 5-per-cent solution of oil-tar creosote from those treated with a 5-per-cent solution of coal-tar creosote.

In the leaching tests, on the basis of the accepted samples, the difference, if any, appears to be slightly in favor of the coal-tar creosote. The basis of rejecting these samples was somewhat empirical but if it is assumed that the maximum losses (or the severe conditions) should govern, the basis for the rejection is sound. It was found, on this basis, that the oil-tar-creosoted samples lost 37.26 per cent as against a loss of 33.48 per cent for the coal-tar-creosoted samples: difference of 3.78 per cent. It is believed that this difference is not significant. If all of the samples were to be used in computing the average, however, the average per cent loss in the oil tar would be 15.19 and in the coal tar 32.37.

Insofar as leaching is concerned one might reason, on the basis of these results, that it would be better to treat with a 5-per-cent solution instead of a 10-per-cent solution. One of three possibilities might account for this apparent discrepancy: 1. Insufficient sampling. 2. Improper interpretation of the results. 3. The stimulating effect of some part of the creosote oil that was present in sufficient quantity in the 10-per-cent-treated blocks but not in sufficient quantity in the 5-per-cent-treated blocks to cause active stimulation to the fungus. The maximum loss of 38 per cent in the untreated control blocks as compared to the maximum loss of 47 per cent in the blocks treated with the 10-per-cent solution of coal-tar creosote would lead one to believe that the last might be the most probable answer. Further study would be desirable in order to clarify this point.

It is very interesting to note that in all cases in the volatilization and leaching tests the maximum losses in both the concentrations appear in the coal-tar-creosoted blocks.

On the basis of these results it appears that the oil-tar creosote studied is equal, if not superior, to the coal-tar creosote studied in its ability to act as

a wood preservative. This statement rests on the basis that a wood preservative must not only be toxic but have the ability to remain in place under service conditions.

VIII. CONCLUSIONS

1. An examination of the literature regarding the beneficial action of tar acids and tar bases in creosote oil to be used as a wood preservative leads to the belief that their presence may not be essential for a long-lasting preservative. This opinion is substantiated by the pathological studies in Part V of this paper.

2. The comparable volatility tests show the following significant points:

- (a) On the basis of the Kolle flask tests against the test fungus *Lenzites trabea*, the oil-tar-creosoted blocks proved to be superior to the coal-tar creosoted blocks.
- (b) The per cent of the weight of creosote lost from wood blocks treated with the whole creosote oil was found to be approximately the same in both creosotes tested.
- (c) The per cent of the weight of creosote lost from open-dish volatility tests was found to be approximately the same in both the creosotes tested.

3. The comparable leaching tests, based on the per cent of weight of creosote oil lost from impregnated wood blocks, showed no significant difference in the two creosote oils. On the basis of Kolle flask tests against the test fungus *Lenzites trabea* no significant differences were determined. The action of leaching on the toxic qualities is not entirely clear, but it is believed that the conclusions, based on the test samples, are conservative. Further tests would be desirable to clarify this point.

4. The initial toxicity of the coal-tar creosote tested appears to be somewhat higher than the oil-tar creosote. The inhibiting point for the growth of *Lenzites trabea* appears to be slightly in excess of an 8-per-cent treating solution for both oils. The inhibiting effect of coal-tar creosote appears to be slightly greater than the oil-tar creosote when the wood blocks were treated with both the 6- and 8-per-cent concentrations. The dilutions were made entirely for the purpose of studying the relative toxicities. It is believed that the higher concentrations used in commercial treatments would give adequate protection for many years.

5. From a study of the samples of wood used in the penetration tests it is shown that the oil-tar creosote possesses good penetrating qualities. Good penetration was secured when Douglas-fir posts were treated with the oil-tar creosote under commercial conditions. The viscosity of oil-tar creosote was found to be lower than that of the sample of coal-tar creosote tested. This was especially true at room temperatures. This factor is of importance when considering the dispersion of the oil in the wood after the treating process.

6. The oil-tar creosote possesses all of the requirements of the American Wood Preservers Association specifications for Grade 1 creosote with the exception of the guaranty of coal-tar origin, and a specific gravity difference of 0.01.

7. The high temperatures used in the cracking of petroleum oil residuum produce a tar that contains a very high percentage of aromatic hydrocarbons.

As shown by literature citations, the aromatic hydrocarbons are more toxic than the phenols.

8. It is believed that the oil-tar creosote possesses all of the requirements of a good wood preservative. It is almost as toxic as the standard Grade 1 coal-tar creosote tested and it is likely to stay in place better because of greater resistance to volatilization. It possesses good penetrating qualities and a low viscosity. It can be made in commercial quantities and is readily available for Pacific Coast consumption.

9. It is suggested that further study of the leaching characteristics would be desirable in order to clarify the results of this study. A study of the literature did not reveal any comparable tests by the Kolle flask method.

Also, it is suggested that studies that would show the resistance of oil-tar-creosoted wood to attack by marine borers and termites would be desirable. These studies would be in the nature of substantiating evidence, because as has been shown, the oil-tar creosote possesses characteristics that are entirely comparable to coal-tar creosote.

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