# OIL TAR CREOSOTE

## FOR WOOD PRESERVATION

by

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#### OIL TAR CREOSOTE FOR WOOD PRESERVATION

### Introduction

Wood preservation in the Pacific Northwest timber industry occupies an increasingly important position. Within the last three 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 non-pressure tanks available for treating operations (2).\* Since the Pacific Coast has about 62 percent (32) of the Nation's sawtimber stand and about 24 percent (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 regain 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 pre-fabricated wooden structures employing creosote treatment and ring connectors have put wood structures in a position capable of competing with steel and reenforced 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.

\* Numbers in parentheses refer to literature citations.

There is at present an actual demand for creosote for wood preservation in 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 percent of the annual requirement is imported from England, Scotland, Belgium, Japan and Germany. Since the imported oil must reach the consumer in cargo shipments, over 50 percent 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

Pacific Coast	·	1936	<u>1937</u>
	distillate	5,463,400	4,726,306
	distillate	7,830,427	7,501,293
United States			
	distillate	124,456,892	82,137,128
	distillate	30,256,107	35,225,781

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 creesete 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 World War.

In searching for another supply of creosote oil, it is found that a oreosote 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 four to five million gallons of this oil tar creosote. 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 pre-

serving wood is open to much question, the action of these factors will be discussed at length later in Part II of this paper.

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 resume of the manufacturing processes of creosote and the theory of preservative action is included.

Since oil tar creosote, as manufactured by the Portland Gas and Coke Company, has never been generally placed 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. However, as will be shown later, the two creosotes are not synonomous.

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

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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.

## 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. It has been found that untreated cross-ties have a life of around five years, while preserved ties have a life of twenty-seven years (26). It has also been shown that untreated marine piling may not last a year, while creosoted piling may have an indefinite life due to its ability to resist decay and marine borers.

Treating wood serves a two-fold 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 real 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 creesote 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, Lowery, and Reuping processes are still essentially the same today as when they were first employed.

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

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. However, for all outside work, the

salt treatments cannot be recommended as being as effective as creosote because of their susceptibility to leaching with water. The railroads only use zinc chloride 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 Fine Association. The use of Pentachlorophenol in the Permatol solution has suggested the possibilities of using this chemical in oil tar creosote with the possibility of obtaining a much better product for wood preservation than is now available in coal tar creosote.

# Requirements of a Good Preservative

Toxicity. The first and most desirable requirement for a wood preservative is that it must be toxic to the organism which 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 toredo, and xylotra.

<u>Permanence</u>. 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 H O) is 184 degrees

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.

<u>Penetration.</u> In order to be effective, a preservative must have the capacity for deeply penetrating the wood. Penetration is of course commercially affected by using heat to lower the viscosity of the liquid and by pressure to force the preservative into the wood. A preservative which will not penetrate offers little advantage to the wood which may subsequently develop checks below the penetration line.

<u>Non-corresive and non-harmful to the wood</u>. Preservatives should not be corresive to metal fastenings, nor should they affect the strength of the wood. A preservative such as corresive sublimate is somewhat corresive to metal, and zinc chloride, if not properly handled in the treating process, may affect the strength qualities of the wood. (26)

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 biohlorids of mercury, lead and arsenic compounds.

<u>Readily available at low cost</u>. 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.

#### 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 partially water soluble. Gurtin (14) has shown that the action of fungi produces an acid condition equal to phy. It follows, therefore, that although a preservative may not be sufficiently toxic under neutral conditions, the slightly acid condition caused by the fungue 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 beta-naphthol. 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 .0014 percent (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 Henningson 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 leas toxic, but more soluble constituents.

Excerpts of some of the discussion regarding the conclusions of this paper are given on page 35 of this report.

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 Seidenschur, who found that the neutral cile of precepte were as equally 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 uncoubtedly true that there is a certain amount of sealing action which tends to close the pit apertures and out down the free flow of water, and also if the cell wall is filled to refusal with cil, 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 fungue 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 texic material to be present in the areas most likely to attack. It must be pointed out, however, that the character of the creceote oils change during periods of service. This change has an offect on the ability of the crocacte to act as a satisfactory preservative over a period of time. This is pointed out by Scheltz, von Schrenk, and Kammorer (h2) who conclude among other things that, "As repeatedly pointed out previously, changes in the character of the creoectes 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 creasate is low, appears to be due. In part at least, to the movement of the toxic constituents of the presence in the inner layers to that in the outer layers of wood."

<u>Permanence</u> is related to both the character of the toxic material and to the mechanical action of the preservative. As has slready 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 pre-

vent 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 pentechlorophenol is an excellent preservative. This chemical gives a loss of 0.00003 grams per square inch per hour at 50 degrees 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 cile, boiling below 205 degrees 0. will not stay in the timber, but that the heavy cils containing a high percentage of anthracene cils 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 creesets 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 lost 34.7 percent of the original amount of oil after two months, fraction II, 205 to 250 lost 21.3 percent after two months, while the original creosote oil lost only 5.4 percent. It is only logical to believe that the higher the boiling point, the less will be the volatility.

#### 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.

## 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 percent in water as the carrying medium. It follows, therefore, that the wood must be partially dry in order that the desired penetrations and absorptions can be obtained. The water may be subsequently dried out, leaving approximately one-half 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 .35 percent in agar (38). Zinc chloride has been used more extensively in the United States than in any other country. During and following the World War many users of creesote had to turn to zinc chloride because of the lack of an adequate supply of creesote from European countries. Chromated zinc chloride is said to be composed of 80 to 82 percent 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 Flouride has many of the same properties as sinc chloride. The toxic point is reported as .25 percent on agar (38). In addition to the tendency of this salt to leach is the disadvantage of having sodium flouride form an insoluble precipitate with calcium. This fact prohibits the use of sodium flouride in contact with limestone or lime water.

Mercuric chloride. Due to the high toxicity of this salt reported as .005 percent (9), only a one percent 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% to 16% years service as compared to creosote of 23 years service.

<u>Copper sulphate</u>, with a toxic point of less than .065 percent in agar (9). Copper sulphate, like mercuric chloride, is corrosive to iron and steel and therefore requires special treating apparatus. Also like sodium flouride, it will react with calcium to form precipitates.

## Proservative 011s

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.

<u>Coal tar creesete</u>, obtained mainly from the heavy oils in the distillation of coal tar have been set as the standard for the oils used in wood preservation. As will be shown later, creesete 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 which inhibit the growth of such organisms. The toxic point for coal tar ercesete has been variously reported. The main objection to the use of creesete appears to be the odor and the fact that wood treated with ercesete cannot be readily painted. Although 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.

<u>Water gas tar creesete</u> 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 point 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.

<u>Coal tar</u> 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 dilutent for creosote oil and finds considerable use in this manner. <u>Creosote coal tar solutions</u> are included in the American Wood Preservers' Association specifications (5), but the specifications do not permit the use of more than 20 percent of coal tar.

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

<u>Petroleum oils</u> as a class are non-toxic. They cannot in themselves be recommended for preservatives although the sealing action of the heavier oils may for a time inhibit the growth of fungi.

<u>Creosote petroleum mixtures</u> 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.

### Chemicals in Solution

Although organic chemicals have not been widely used 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 betanaphthol 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 six years of service, the posts treated with 13.6 lbs. of grade no. 1 creosote were all sound, whereas the posts treated with 7.1 lbs. absorption of tetrachlorophenol were 100 percent destroyed. Also, the posts treated with 30 percent B-naphthol, 10 percent

pine oil, and 67 percent petroleum oilwere 87.5 percent destroyed. This test would indicate that heither the petroleum or 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% oresylic acid and 95% petroleum as a solution to preserve railway ties has 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 sromatic hydrocarbons, would not be stable in the word.

#### PART II

# Comparison of oil tar, coal tar and water gas tar creosotes

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 creosete, as adopted by the American Wood Preservers' Association (3), The American Railway Engineering Association, and the A.S.T.M. are as follows:

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

2. It shall contain not more than 3 percent of water.

3. It shall contain not more than 0.5 percent of matter insoluble in benzol.

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

5. The distillate on a water free basis shall be within the following limits:

Up to 210 degrees C., not more than 5 percent.

Up to 235 degrees C., not more than 25 percent.

6. 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 degrees C., although creosotes containing high residues generally have higher viscocities, 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

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anthracenes 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.

<u>Water gas tar creosote.</u> 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, do 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. The specifications are as follows:

1. The oil shall be a distillate of water gas tar and shall comply with the following requirements:

2. It shall contain not more than 3 percent of water.

3. It shall contain not more than 0.5 percent insoluble in benzol.

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

5. The distillate, based on water free oil, shall be within the following limits:

Up to 210 degrees C., not more than 5 percent.

Up to 235 degrees C., not more than 25 percent.

Up to 355 degrees C., not less than 70 percent.

6. The oil shall yield not more than 2 percent of coke residue.

7. 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 degrees C. cannot exceed 30 percent which is an effort to guard against the excessive amounts of high boiling oils which Bateman (11) points out are apt to be of low toxicity.

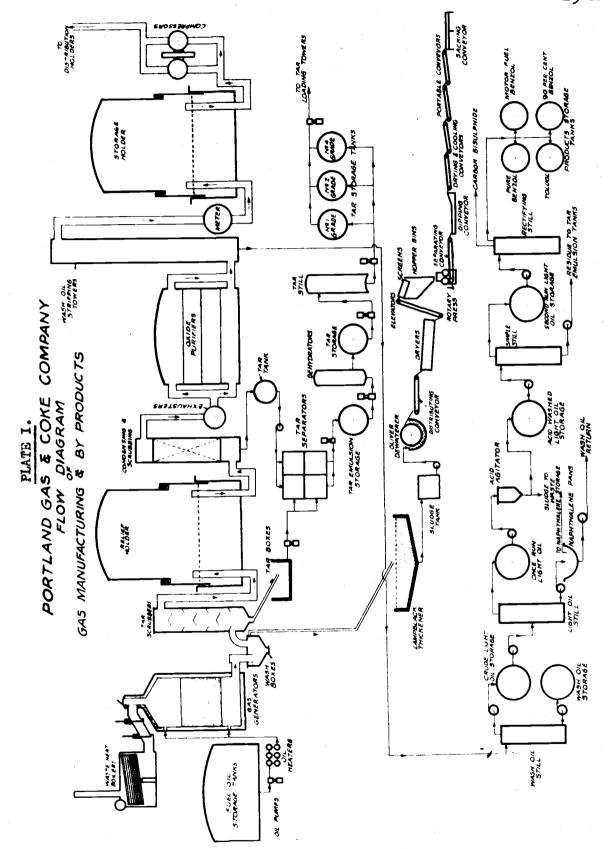
Since oil tar creosote, as such, has never been placed on the market nor has it been used as a wood preservative, no specifications have ever been prepared. It appears likely, however, that the grade 1 coal tar creosote specifications would be suitable for oil tar creosote except that the specific gravity specification could be lowered slightly without seriously affecting the quantity of the high boiling fractions.

#### 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 resume 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.

<u>Oil Tar Creosote</u> 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 the flow chart of the gas plant. (Plate I)

The gas generators are heated until the temperature reaches approximately 1800 degrees 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 stock 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 lampblack is deposited, thence passing up through a scrubbing tower having a system of baffles, over which a continuous stream of water



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is passing. The tar recovered in this process flows out into the tar separating vats, where the excess water is removed. From the separating vats, 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. However, it is only logical to believe that the creosote oils are almost as complex as the coal tar creosote, with the exception of the phenols and bases. Cil 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. Deans and Downe (15) in studies of water gas tar found that it contained thiophen, benzene, toluene, xylene, mesitylene naphthalene and anthracene. In fact, every similarity existed between the oil which they examined and coal tar creosote, except for the absence of tar acids, bases, and 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 which 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 Fourth (Untersuchungen eines Olgasteers, Munchen 1904) who found all of the constituents of coal ter, including even phenols and traces of bases, but did not find carbon diculphide 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 temperature 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. Henzene, toluene, naphthalene, phenathrene, 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 solds 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 in-

dividual 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 creesote is obtained from distilling the tars accumulating from the production of water gas. Water gas tar may be considered an oil tar, but the procedure used in producing the gas and tar is similar, 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 partially combusted and pass on through the carburetor and superheater. An additional air supply in the carburetor and superheater allow complete combustion to take place, thoroughly heating these parts which, according to Bateman (8), reach a temperature of lifel degree 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 a product of the coal reaction, but is from the cracking of the oil. Insufficient cracking, of course, will result in a lower yield of gas and a greater amount of paraffin in the tar.

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

The manufacture of coal tar creosote. Creosote, as defined by Lunge (3), .... distillates from coal tar which 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 degrees 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:

	Coel	
Gas	Tar	Coke
Light 011	Heavy 011	Pitch
	Creosote	

The tars which 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 Eateman (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 the type of producer most 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 ovens, 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 degrees F. to 2012 degrees F., the yield of aromatics increased in about the same ratio as the paraffins decreased with temperature rise. In the unwashed coal, the aromatics

yielded about 47 percent of the distillate at 932 to a maximum of 84.5 percent of the distillate at 1832 degrees F. The olefine content changed only slightly. The tar acid content decreased from 17 percent at 932 degrees F. to about 2.3 percent at 2012 degrees 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 creosetes 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.

## The Controversial Point of Tar Acids and Tar Bases

It has long been felt that the rar acids in creosote oil was 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 "Ter Acids" is applied to those constituents of ter which 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)

Compound	СНО	Boiling Point Deg. C.
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 "	8-10- 1	225
1-3-2 "	8-10- 1	212
1-3-11 "	8-10- 1	209
1-3-5 "	8-10- 1	219
ī-4-2 "	8-10- 1	209
a-naphthol	10-8-1	280
b-naphthol	10- 8- 1	286
c-naphthol	<u>14-10- 1</u>	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:

Compound	CH	I N		Boiling Point Deg. C.
Pyridene	5-	7-	1	115 (Other pyridenes
Quinoline	9-	7-	1	238 at higher B.P.)
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 makes 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 degrees C., most of the tars will be expected to yield around four or five percent 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. The tar acid content of normal commercial coal tar creosote, however, can be expected to yield around five percent 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 used for this purpose:

Larkin (29), commenting on the work of Bateman and Henningson (11):

"The toxic principles of Greosote." Referring to the work of Coisneand, taken from "Preservation of Timbers by Use of Antiseptics" (1885) by Samuel Boulton; Coisene used 5 samples as follows: 1, one with 15% tar acids; 2, another with 15% tar acids; 3, one with 8% tar acids; 4, one with 4% 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 eils....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 semi-solid constituents of the tar oils were present; in 12 of them naphthalene; and 3,

only small percentages remained of oils distilling below 450 degrees F. In a majority of cases, from 60 to 75 percent of the total bulk of the substances retained in the wood did not distill until after a temperature of 600 degrees 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. Seidenschur, Stendal, Germany in 1909:

"Anthracene oils 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. Callert, "Quantity and Character of Creosote in Well Preserved Timbers" (1):

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%, 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 crecsote. 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 degrees 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 staying qualities.

Von Schrenk, Herman: "Significance of Toxicity Determinations from a Practical Standpoint" (46):

(1) Points out that American croosote practice has been built on European practice.

(2) Quotes from Furgeson'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 tar acids, finds itself exalted to the most essential constituents. 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 creosote differing widely in their composition."

(3) High boiling constituents of creosote have comparatively high toxic values.

Lunge, George: "Coal Tar and Ammonia," 5th Ed. 1916, D. Van Nostrand & Co., (30), Seidenschur (Zangew. 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 zine 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 pehnols. 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

"• **3**8 chloride. Later on Seidenschur (Chem Zeit., 1909, No. 77) impregnated wood sleepers with progressive quantities of acid free tar oils and exposed to the wood destroying fungi, to dry rot fungus (Merulius lacrymans) and to polyporus vaporarius. The oil was emulsionated by rosin soda scap, the emulsion containing the equivalent of 6 percent 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 affects a ninefold security. From impregnated sleepers which 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 5., 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 degrees and 320 degrees 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.F.A. Proc. 1914, page 216. (17)

This report deals with specially prepared oils which were used for observation for attack of marine borers. The specimens of piling were treated with fractions of crossote by re-distilling a good grade of coal tar crossote as follows:

Fraction	I	0-205	Deg.	C.	Tar acid oils
¥1	11	205-250	tt 🐪	11	Naphthalene oils
11	III	250-295	11	11	Deed oil
<b>\$</b> \$	IV	295-320	ti -	ŧ:	Anthracene oil
<b>#</b>	٧	Above 320	教	Ħ	Regidue

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

Fraction I	Very severe attack by toredo
" II	Very severe attack by toredo
" III	Medium attack by toredo
" IV	Slight attack by toredo
* V	Practically sound
Water gas tar	Slight attack by toredo
Coal tar creosote	

Rhodes, R. H. and Gardner, F. 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. Eng. Chem. Vol. 24, page 772, July 1932. (41)

The authors, citing the diversity of opinion regarding the presence of a barren or non-taxic 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 crecosote. The summarization of their work is cited: "In coal tar crecosote, there are substances varying greatly in their toxicity to wood destroying fungi. Although there may be certain substances in coal tar crecsote 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 4 fractions, from which 8 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 degrees C. Washing this fraction alternately for 3 hour periods with 30 percent sulfuric acid and a 15 percent solution of sodium hydroxide, and removing a white crystaline material which formed on cooling did not greatly change its toxicity."

In the same paper, the authors point to the work of Charitschoff (J. Russ. Phys. Chem. Soc., ht, 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 is 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 degrees C., and the water soluble products."

Schmitz, Von Schrenk and Kammerer (12):

In their studies of the quality and toxicity of coal tar creesote oil extracted from red oak ties after long periods of service found that 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 high or higher than that of the creosote extracted from zones 2 and 3." (This amounted to from 0.5 percent to 1.3 percent) 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:

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

2. 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.

3. The removal of tar acids and bases from coal tar creosote does not appreciably affect its toxicity.

4. Creosote extractions, after long periods of service, and in spite of their tar acid content, are likely to show comparatively low toxic gualities.

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 non-toxic 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.

## Additions of Petroleum 011 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 dilutents for creosote oil have been used by railroads for treating ties and other purposes. The use of these dilutents 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 percent 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  $(h_0)$  concludes that with the samples of creosote tested, a 50-50 mixture will reduce the toxicity 1/7th and a 25-75 mixture will reduce the toxicity 1/25th, etc. In addition to the disadvantage of lower toxicities of these mixtures is also the fact that the mixtures will not penetrate the wood so readily because of the higher viscosity of the mixture.

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.

#### PART III.

## Physical Properties of Each of the Creosotes Tested

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 hydrocarbons 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 propertion 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."

<u>Color</u>. Oil tar creosote remembles water gas tar creosote, but as has already been shown, there is more liklihood of a greater percent 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, was found to be present. The color imparted to Ponderosa pine sapwood blocks was almost coal black.

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

### 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.

<u>011</u> T	ar Creosote	Coal	Tar Creosote
Temp.º C.	Specific Gravity	Temp.º C.	Specific Gravity
14.8 23.0 33.0 42.0	1.037 1.032 1.024 1.018	19.5 29.0 38.0 47.0	1.077 1.070 1.064 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 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 .01. This lower gravity may be compensated for in part by the nature of the percentage of the higher boiling fractions in the distillate.

#### 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 accumulative distillation up to 270 is approximately the same for both samples of creosote. Above 270, however, the oil tar creosote shows to be higher boiling. From 315 to 355 there is a difference of about 10% in the curves for oil tar and coal tar creosote samples examined.

This difference in the distillation range shows up in the volatilization tests, particularly in the watch glass tests, viz., the higher boiling oil shows less volatilization.

Distillation 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:

Fraction No.	Temperature Deg. C.	% by Weight over	Tar Acids	Acoumu- lative
123456	0-210 210-235 235-270 270-315 315-355 Residue above 355	6.69 6.17 26.17 23.82 23.00 14.17	0.094 0.083 0.521 0.417 0.729 0.000	6.69 12.86 39.03 62.85 85.85 100.02
			1.8hh	

Coal Tar Creosote

Fraction No.	Temperature Deg. C.	% by Weight over	Tar Acids	Accumu- lative
127456	0+210 210-235 235-270 270-315 315-355 Residue above 355	4.77 10.16 21.96 17.38 20.98 24.30	0.0 0.103 0.157 0.0 0.107 0.367	4.77 19.93 36.89 54.27 75.25 99.55

## Gasco Tar Creosote

"Since the tar acid determination upon the small fractions is 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 0.36 percent by weight of tar acids. Note that the determinations on Gasco checked the totals 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 percent to be representative since the Gasco material checked so closely."

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.

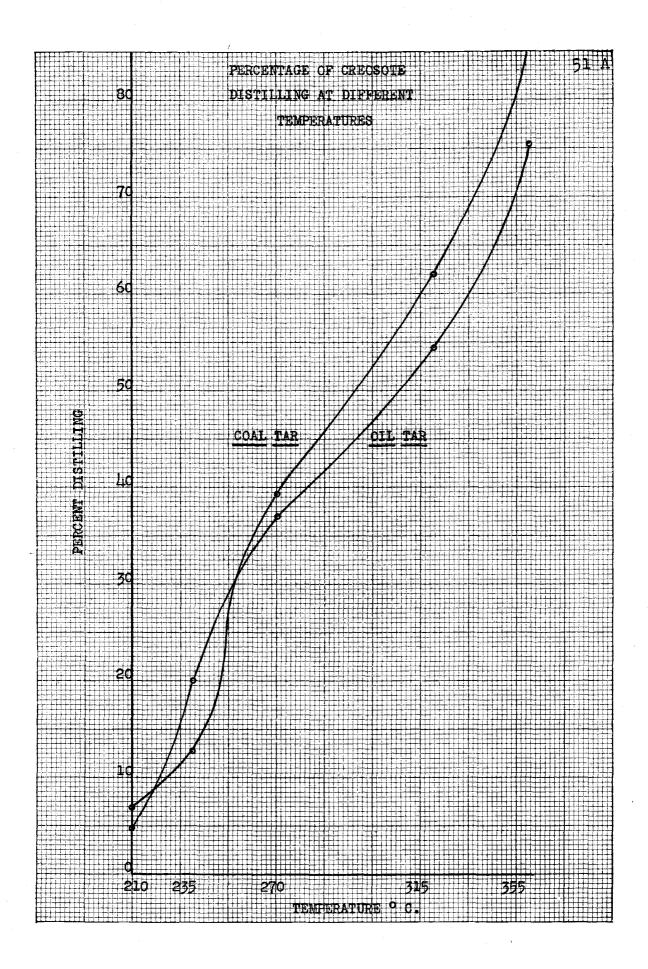
	011 Tar Creosote	Coal Ter Creosote	A.W.P.A. Specifications
Water, % by Vol.	0.1	2.6	Not over 3%
Matter insoluble in Benzol	0.01%	0.3%	Not over 0.5%
Specific Gravity, 38/15.5	1.019	1.062	Not less than 1.03
Distillation up to 210 to 235 to 270 to 315 to 355 Residue Loss	4.5% Wt. 15.1 37.3 52.4 72.7 26.2 1.1	1.7% Wt. 8.7 36.6 60.8 81.5 17.0	Not over 5% Not over 25%
Coke Residue, % of Original	0.11	1.76	Not over 2%
Tar Acids, % by Volume	None	4.7	None

## 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 fungue growth, four wood blocks, prepared as described under Pathological Study, were prepared with each of the following preservatives:

- 1. Coal tar creosote 100%
- 2. 011 tar creosote 100%
- 3. Untreated control

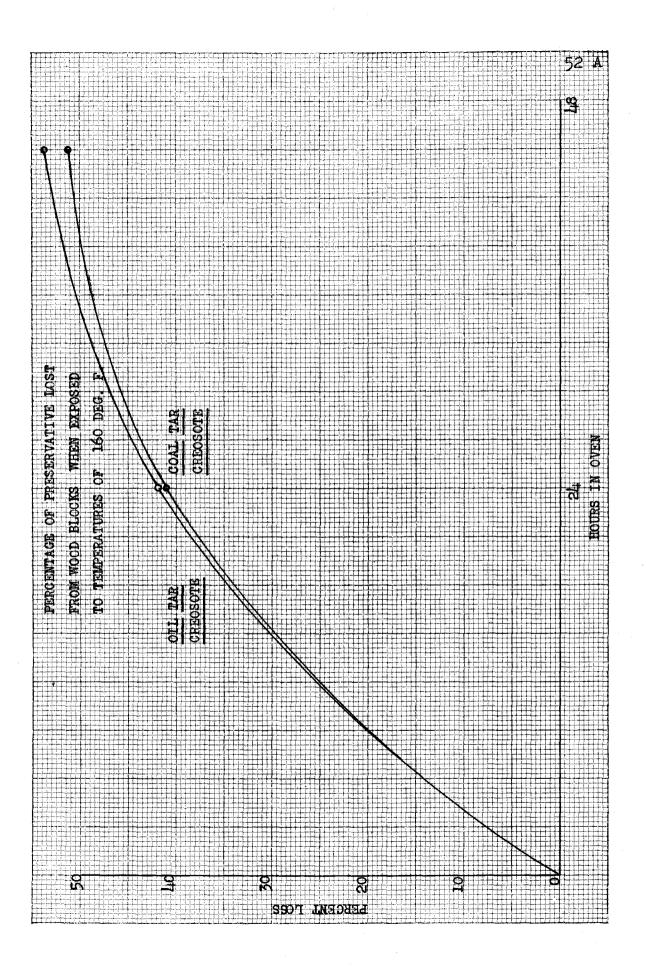
Each set of these blocks was placed in the oven and



held at a temperature of  $160^{\circ}$  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 percent 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 curves shows that the oil tar crecsote 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.

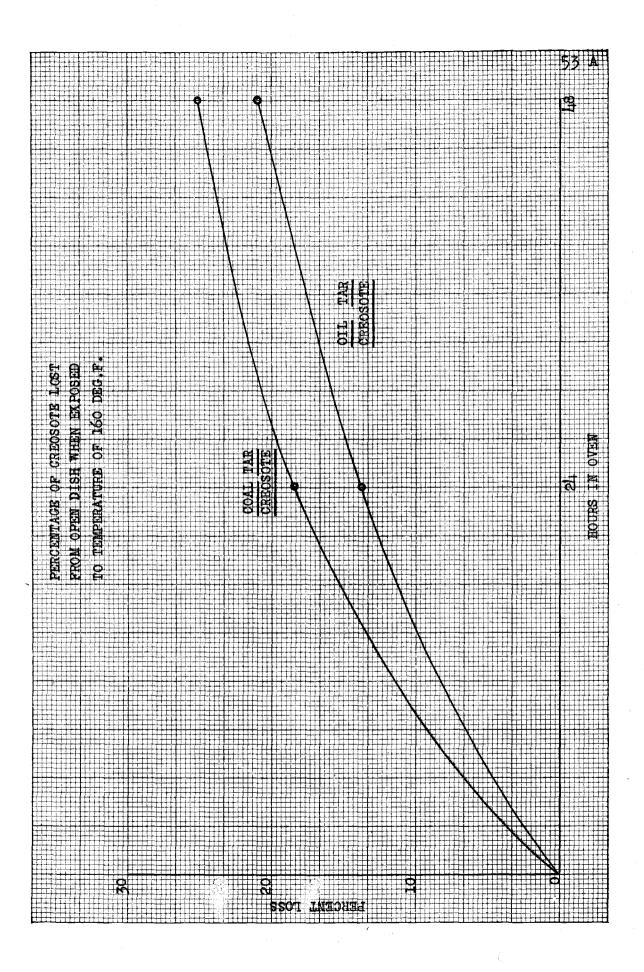


Semple	Preserva- tive taken up Wt. in Grams	Wt. of Pres. left after 24 hrs.	% Lost in 24 hrs.	Wt. of Pres. left after 24 hrs.	% Lost in 48 hrs.
Oil Tar Creosote Ave	2.90 2.337 2.680 2.977 2.727	1.642 1.444 1.526 <u>1.642</u> 1.563	42.5 38.2 41.5 44.9 41.8	1.277 1.195 1.276 <u>1.402</u> 1.287	56.0 53.4 52.2 52.9 53.6
Coal Tar Creosote Ave	2.896 3.191 3.348 2.697 3.033	1.525 1.896 2.134 1.629 1.796	47.5 40.5 36.4 39.6 41.0	1.405 1.701 1.576 <u>1.129</u> 1.452	53.0 46.5 47.0 58.0 51.1

Preceding table shows the result of volatility tests on wood blocks preserved in the usual fashion and placed in an oven at 160 degrees F. for 48 hours. Weights taken at the end of 24 and 48 hour periods. It was assumed that the moisture in the wood blocks was almost entirely evaporated after 24 hours at 160 degrees 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 cc of the preservative fluid, as nearly as could be measured with a burette, calibrated in tenths of a cc, was measured out into open glass dishes which 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 degrees F. and left for 24 hours. At the twenty-four 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 in the following table and graphs:

Wt. of fluid in grams be- fore placing in oven	Wt. of fluid in grams at end of 24 hrs.	% Loss	Wt. of fluid in grams at end of 48 hrs.	% Loss
011 tar creosote 20.869	16.97	13.88	15.37	21.03
Coal tar creosote 21.20	17.40	18.39	15.898	25.09

## Leaching Tests with Water

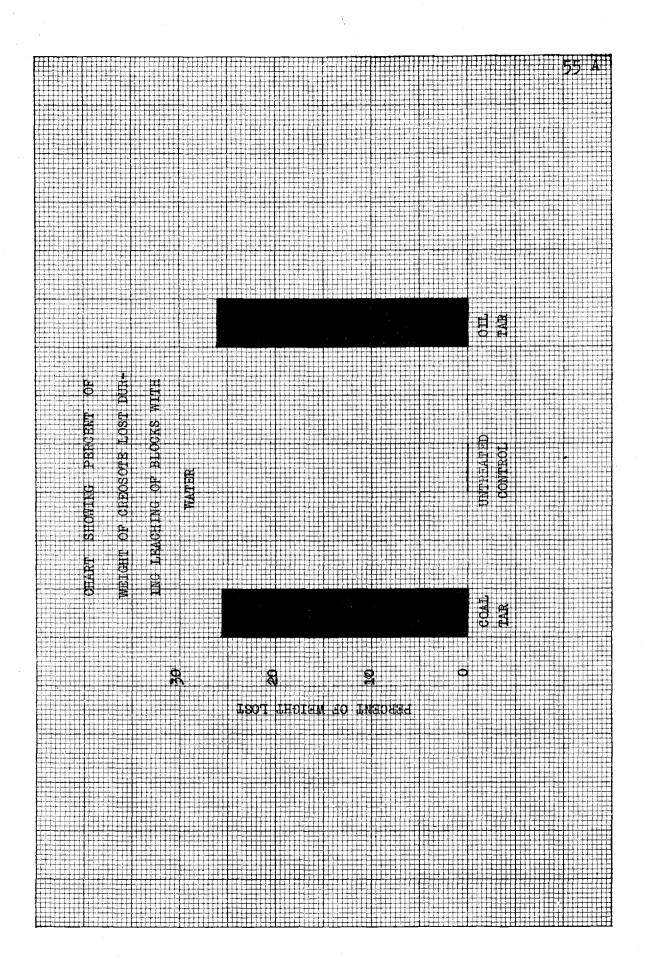
Leaching tests were made under conditions similar to those used by Hubert (24). Ponderosa pine sapwood blocks were cut to a size of  $\frac{1}{2} \times 1\frac{1}{2} \times 2$  inches so that the blocks could be subsequently used in Kolle flask tests. The blocks were conditioned in a desiccator over a saturated neutral salt solution until they had reached an equilibrium of 12.24% moisture content. Six blocks were selected,

weighed and then treated in the way as described under Pathological Study with the following:

No.	No. of Blocks Preservative		
	2	Coal tar creosote 100%	
	2	011 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 clear 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 which supplied the water. All blocks were subjected to this method of leaching for a period of 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 eight 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 amount and percent of the preservative lost during the leaching process was found as shown in the following table:

	ginal Wt.of eservative	Pinal Wt. of Preservative	% Lost	Ave. % Lost
Oil tar creosote	3.020 3.060	2.175 2.572	27.98 24.20	26.09
Coal tar crecsote	3.25 3.08	2.41 2.205	22.7 28.4	25.55
Untreated Control	0.000	0.000		

The accompanying bar chart also shows the relationship of each of the preservatives so tested to the percent of weight lost. It will be noted that there is no striking difference between any of the preservatives tested. The coal tar creosote shows a slight advantage in the matter of resistance to leaching.

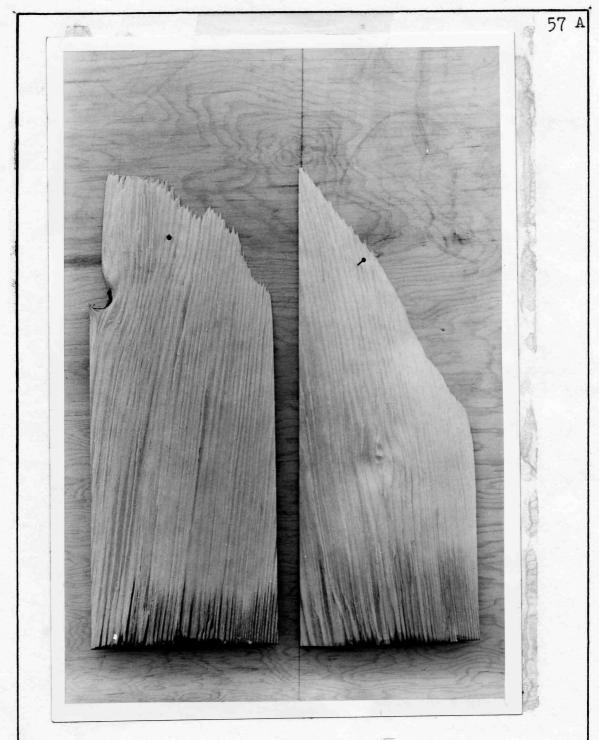
The leached blocks were then placed in Kolle flasks cultures and incubated. The results are shown later in the report.

#### 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 x 6 Fonderosa pine were sawed from the same board; one piece was set on end in } inch of coal tar creosote and the other set on end in a inch of oil tar creosote and allowed to stand for twenty minutes. After exposing to the air of the laboratory for two 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 plate 2. It was believed that this was a fair measure of the comparable penetrating ability of the two creosotes tested. However, it was deemed advisable to continue the study of penetration and absorption under commercial pressure treating conditions.

Thirty-one Douglas fir posts were selected and treated at the pilot plant of the Pope and Talbot Lumber Company creosoting plant in St. Helens, Oregon. The log of the treating operation is as follows:



## PLATE NO. 2

End penetration of coal tar creosote (left) and oil tar creosote (right) on 2 x 6 Ponderosa pine during 20 minute dipping period.

#### R. H. Rawson

#### TREATMENT REPORT

FOR Oregon State College St. Helens, Oregon at ON Test Posts 9/20 . 1939 CLIENT'S ORDER NO. PLANT ORDER NO. & OUR NO. PLANT REPORT NO. 1 RECORD OF BATH Started, Date 9/20/39 Hr. 5:30 p.m. Ended, Date Hour Hours in Bath TEMPERATURE VACUUM At Start 190 Deg. F. Min. Vacuum 24 Inches 190 " 11 Ħ Maximum Max. 21 W fi 190 At End AIR PRESSURE OR INITIAL VACUUM Started 7:30 Ended 8:30 Lbs. Ins. 50 Hours 1 OIL PRESSURE 9/21/39 Er. 8:45 Started Ended 9/21 Hr. 9:45 Hours under pressure 1 PRESSURES TEMPERATURES UNDER PRESSURE At Start 5 lbs. At Start 135 At End 130 " At End 135 Maximum 135 EXPANSION BATH Started 9/21/39 Ur. 9:45 Ended 9/21/39 Hours in Exp. Bath Hr. 11:45 TEMPERATURES During Expansion Bath 135 deg. F. 208 " " At Start Maximum At End ŧŧ. 208 耕 FINAL VACUUM Started 12 N Ended 1 p.m. Total 1 hrs. 00 min. Max. Inches Min. Inches TOTAL TREATING TIME Hrs. 19 Min. 30 MATERIAL No. Pos. Dimensions Length Lin.or Bd. Ft. Cubic Ft. 31 4 x 4 51 210' 17.5 Portland Gas & Coke Company creosote used in treatment of this material.

# 4" x 4" - 5'0" POSTS

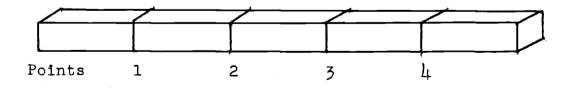
# TREATED WITH GASCO CREOSOTE

P-1 $0.627$ $23.50$ $26.125$ $36.85$ $2.625$ $4.19$ $5.10$ $9.29$ P-2 $0.589$ $21.125$ $25.625$ $32.11$ $1.500$ $2.55$ $4.31$ $6.86$ P-3 $0.627$ $21.375$ $26.000$ $33.51$ $1.625$ $2.59$ $4.15$ $7.04$ P-4 $0.608$ $18.750$ $20.875$ $35.19$ $2.125$ $3.50$ $3.67$ $7.37$ P-5 $0.608$ $20.500$ $23.125$ $32.27$ $2.625$ $4.32$ $3.59$ $6.58$ P-7 $0.608$ $21.375$ $24.000$ $31.46$ $2.125$ $3.50$ $3.63$ $7.13$ P-8 $0.608$ $25.875$ $26.375$ $40.15$ $.500$ $0.82$ $6.667$ $7.48$ P-9 $0.609$ $23.500$ $26.250$ $28.72$ $.625$ $0.94$ $3.75$ $4.16$ P-10 $0.664$ $29.625$ $30.250$ $28.72$ $1.750$ $2.975$ $4.03$ $4.04$ P-11 $0.590$ $23.750$ $25.250$ $36.22$ $1.750$ $2.91$ $8.18$ P-12 $0.590$ $23.750$ $22.000$ $34.03$ $2.375$ $4.03$ $4.04$ $8.07$ P-14 $0.590$ $22.000$ $24.375$ $32.59$ $2.375$ $4.03$ $4.04$ $8.07$ P-15 $0.622$ $25.750$ $27.900$ $36.58$ $1.250$ $1.91$ $5.55$ $7.56$ P-16 $0.590$ $23.750$ $24.875$ $37.51$ $1.125$ $1.99$ $5.65$	Piece Volu No. in cu.i			percent	t #		Correc- tion in #/CF	Net Gain in #/CF
Average 3.35 4.27 7.62	$\begin{array}{c} P-2 & 0.56 \\ P-3 & 0.66 \\ P-3 & 0.66 \\ P-4 & 0.66 \\ P-5 & 0.66 \\ P-7 & 0.66 \\ P-7 & 0.66 \\ P-10 & 0.56 \\ P-10 & 0.56 \\ P-112 & 0.56 \\ P-12 & 0.66 \\ P-12 & 0.66 \\ P-22 & $	39       24.125         24.375       24.375         24.375       24.375         28.750       24.375         28.750       24.750         29.24.750       24.750         21.570       24.750         22.4.750       24.750         24.750       24.750         21.570       21.570         22.5.3750       23.750         22.2.3.750       22.0.000         20.20.000       20.125         20.20.000       20.125         20.20.000       20.3750         22.21.1250       22.1.1250         22.21.1250       27.750         24.25.500       27.750	25.625 26.000 23.1250 26.005 26.005 26.005 26.005 26.005 26.005 26.005 26.005 26.005 225.6000 225.60000 225.60000 225.60000 225.60000 225.600000000000000000000000000000000000	32.14197165322369816669902072885589913 3352.344586553267399902072885589913 44486653326739990208207288589913	$\begin{array}{c} 1.500\\ 1.625\\ 2.125\\ 2.005\\ 2.500\\ 2.5502\\ 1.8755\\ 1.87550\\ 1.87550\\ 1.827550\\ 1.827550\\ 1.8275$	22343304023342144344451134451	44333364355345554332334852332	967776778488787799677785097794 28047083869824786679176093073945 197794
		3 (13.300			64.000	3.35	4.27	7.62

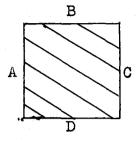
These posts, which were placed in the Oregon State College School of Forestry Test grounds, have retained an average absorption of 7.62 pounds of preservative per cubic foot as specified by the federal specification TT-W-556 and the American Rood Preservers' specification 4d or 5 b (1933.

Now that the results of absorption under the given treating conditions have been observed it becomes desirable to investigate the corresponding penetrations. Posts nos. 26 and 28 were out in even foot lengths in order to more generally observe the penetration. These points, as chown by the diagramatic out, show the following penetrations.

The following table shows the depth of genetration of posts no. 26 and no. 28. A wafer was sawn from the cross soction of each post at one feet intervals according to the following diagram:



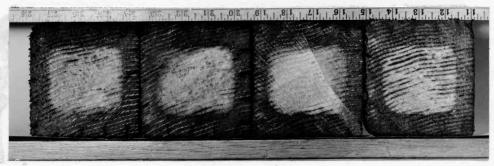
The penetration was then measured midway between the corners as follows:



The incisor marks showed a penetration of approximately four-tenths of an inch from the surface.

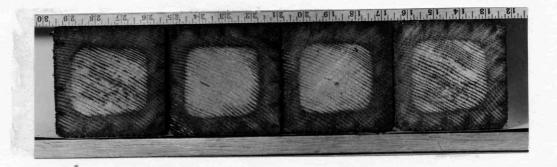
Post 28

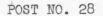
	Point 1	Point 2	Point 3	Point 4
A	.65 inches	.65 inches	.55 inches	.55 inches
В	•75 "	.85 "	.90 *	.80 "
C	.60 *	.60 "	.60 "	.70 "
D	.80 "	.80 "	.80 "	.85 "
Ave.	.70 inches	.725 inches	.712 inches	.725 inches
Post	26			
A	.60 inches	.60 inches	.65 inches	.80 inches
В	.80 "	.60 "	•75 *	.65 "
			- ( <i>J</i> )	,
C	.65 *	.80 "	.80 "	.65 "
C D	.65 <b>*</b> .80 *	.80 " .65 "		÷



POST NO. 26

Treated with oil tar creosote under commercial conditions. The section here shown corresponds to the designated points mentioned in the table. Point 1 is at left, reading 2, 3, and 4, left to right.

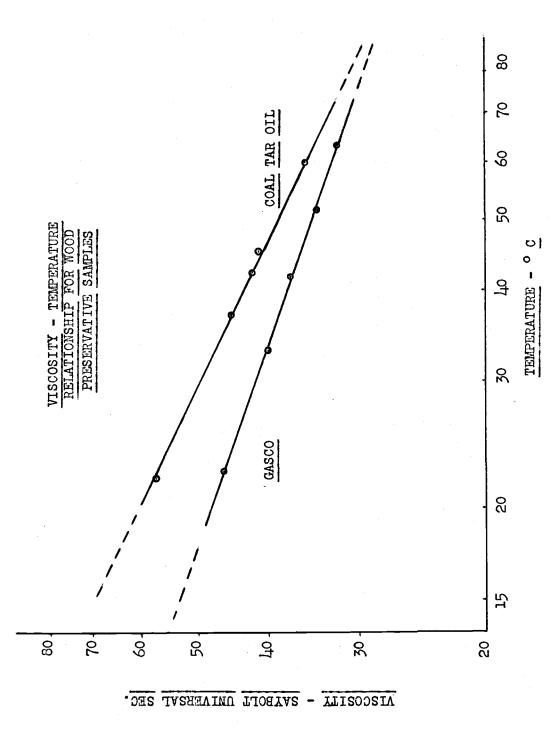




Treated with oil tar creosote under commercial conditions. The section here shown corresponds to the designated points mentioned in the table. Point 1 is at left, reading 2, 3, and 4, left to right.

61

D



61 B

An examination of the included photographs 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 in which deep penetrations have been secured.

One of the factors which bears on the penetrating ability of a liquid is the viscosity. Tests on the Saybolt Viscosity at various temperatures for oil tar creosote and for the coal tar creosote tested are shown in the following table and accompanying graph. The difference indicates a better penetrating ability of oil tar creosote.

Coal	Tar Oil	Gesc	
Temp. ° C.	Saybolt Sec.	Temp. ° C.	Saybolt Sec.
22.0	57	22.5	46
37.0	超	33.0 42.0	40 37
45.0	4 <u>1</u> 35	51.5 63.5	34 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.

### 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 relative flash points of the two creosotes under investigation. The flash point, determined by the Cleveland open cup method (18), was found to be Coal Tar Creosote cil 215° F., Oil Tar Creosote oil 175° F.

#### PART IV.

### Pathological Study

The objective of this portion of the study was to determine the relative toxicity 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 series 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. This was done and certain differences in procedure were followed in the second series than in the first. The procedure of the second series and tabulation of results therefore follow the description and tabulation of results as found in the first sories of tests.

### 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 media and inoculate with the desired fungues.

The other methods, known as the wood block method, or the European 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 Flemming (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, has become increasingly important in the United States, and has become a generally accepted procedure (33) among workers in the

United States, it was 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. It follows, therefore, 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.

### 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 .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.

### Preparation of Wood Samples

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

### 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 ununiform 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 four weeks. It was found that by this procedure a uniform moisture content of 11.97 percent was obtained. Moisture content was found by weighing every tenth block and placing in a drying oven at 104 degrees 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.

#### Selection of Fungue

After examining the work of a number of investigators who have done considerable work on toxicity of wood pre-

servatives, and upon the recommendation of Dr. 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 in 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.

#### Preparation of Flasks and Media

The malt agar media was prepared by the following formula: (U.S.D.A. Bulletin 346)

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

One hundred cc of the prepared media was poured into each Kolle flask. The flask plugged with cotton and the flasks then sterilized in an autoclave under 15 pounds gauge of 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. at which time most of the flasks had developed a mat over the entire surface of the agar media. A few flasks inoculated with Poria incrassata required slightly longer for the fungus mat to completely cover the surface of the media. All contaminations were rejected and new media prepared for the contaminated flasks. At the end of the two weeks' incubation period most of the flasks were ready to receive the preserved wood blocks.

### 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 eliminate any error. Since the weights checked almost identically, this 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 separatery funnel and hose outlet for a vacuum pump. The desired number of blocks 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 drawn for a period of thirty minutes. The preservative of the desired concentration was then let into the jar without breaking the vacuum after which the wood blocks were allowed to stand in the preservative for thirty minutes. After im-

pregnation the blocks were removed, placed on glass rods and allowed to drain and stand for 24 hours in the laboratory after which the blocks were 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 get the dilutions, Stoddard Solvent was used as the non-toxic oil. All percentages are expressed in percentages of 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.

Coal Tar Creosote

The following concentrations were used:

No.	Percent	CC Creosote	CC Solvent
1	100.	100.	0.
2	50.	50.	67.3
3	12.	11.16	110.27
4	6.	5.57	117.79
2	4.	5.93	192.5
6	1.	2.22	295.74
7	.8	1.78	272.6
8	.6	1.33	273.0
9	•5	1.68	397.91
10	.[	1.187	398.39
11	•3	.89	398.8
12	.2	.762	699.1
13	.1	.371	1.99.1.9
14	.05	.185	199.74
			TEF # T & TE .

After conditioning, weighing, impregnation, 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. The 3 millimeter glass rods which 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 which might be on the surface, neither would it kill or 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 sides of the block.

Four wood blocks were impregnated with each of the concentrations listed on the preceding page, two blocks being placed in the flask with Lenzites trabea and two blocks in flask with Poria incressata.

### Results

After incubation for a period of eight 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 - oven dry weight equals weight of preservative. Finished Preserved Weight - oven dry weight equals weight of preservative

at conclusion of tests.

Original weight of preservative - final weight of preservative x 100 equals Original weight of

preservative.

Percent 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 as found as described above was then used as the basis for computing the percent of weight lost from the wood blocks. The percentage is based 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:

Table showing loss in weight of individual sample blocks impregnated with different concentrations of Coal Tar Creosote after incubation period of 8 weeks with Lenzites trabes.

Concen- tration percent	Sam- ple	Computed Oven Dry Weight	Adjusted Oven Dry Weight	Moisture Free Wt. after in- cubation	Percent Loss in Weight	Ave. % Loss
100	A B C	4.544 4.410 4.465	6.67 6.58	6.96 6.41 4.52	attack	
50	A B C	4.877 4.772 5.123	5.58 5.49	5.85 5.36 3.70	рания в 8.2	
12	A B C	4.656 4.331	4.866 4.541	en en generen en sen die der Generen der einen einen der einen der einen der einen der einen der einen der eine	No	
6	A B C	4.914 4.342 4.518	5.02 4.45	4.93 4.30 2.75	1.7 3.3 39.1	2.5
4	A B C	4.903 4.528 4.419	4.979 4.604	3.84 4.18 2.65	22.9 9.0 40.0	15.9
1	A B C	4.812 4.673 4.383	4.83 4.692	3.31 3.39 3.23	31.2 28.0 26.3	29.6
.8	A B C	4.289 4.909 4.640	4.304 4.924	3.31 3.48 3.13	22.8 29.3 32.3	26.
.6	A B C	4 <b>.702</b> 4.968 4.556		4.31 3.51 3.04	8.5 29.3 33.2	18.9
.5	A B C	4.686 4.753 4.908		3.72 3.89 3.32	20.5 18.1 32.3	<u>19.3</u>
.4	A B C	4.562 4.565 4.899	<b>4 14 2 2 2 19 19 19 19 19 19.</b>	3.37 3.39 3.75	26.1 25.7 23.4	25.9
-3	A We B C	t 4.449 4.440 4.811	an a shara a s	4.02 3.05 3.51	9.6	31.7
.2	A B C	4.611 4.460 4.901		3.49 3.45 3.70	24.3 22.6 24.1	23.4
.1	A B C We	4.700 4.601 t 4.722		3.48 3.11 4.35	31.7 28.1 24.3 22.6 24.1 25.9 32.4 8.	29.1
.05	A B C	4.565 4.418		3.95 3.67	12.3 16.2	17.2

Desiccation

Table showing loss in weight of individual sample blocks that have been impregnated with different concentrations of Coal Tar Creosote and after an incubation period of 8 weeks with Poria incrassata.

Concen- tration percent	Sam- p <b>le</b>	Computed Oven Dry Weight	Adjusted Oven Dry Weight	Noisture Free Wt. after in- cubation	Percent Loss in Weight	Ave. % Loss
100	A B C	4.445 4.374 4.632	6.31 6.30	6.44 6.42 4.73		
50	A B C	4.418 4.463 4.959	5.23 5.31	5.39 5.46 5.19	e s c t s c t	and the second
12	A B C	4.352 4.619 5.250	4.544 4.811	4.43 4.71 5.12	2.	
6	A B C	5.124 4.427 4.304	5.239 4.542	5.32 4.53 2.34	45.6	anguleysi (14) (19) (19)
4	A B C	4.563 4.688 4.621	4.64 4.76	4.65 4.76 4.64 we		
1	A B C	4.408 4.438	4.467 4.457	an a	2.	and a state of the
.8	A B C	4.417 4.430 4.466	4.432	3.29 3.78 1.75	25.7 14.9 60.7	20.3
.6	A B C	4.947 4.423 4.690	4.509 4.435	3.94 3.05 3.00	12.6 31.2 36.0	21.9
.5	A B C	4.779 4.913	4.788 4.923			
.4	A B C	4.533 4.427 4.458		3.36 4.00 3.14	26.0 9.6 29.5	17.8
•3	A we B C	t 5.00 4.586 5.198		4.93 3.66 2.62	1.4 20.1 49.5	20.1
.2	A B C	4.779 4.515 4.498		3.53 3.27 4.55 we		26.8
	A B C	4.592 4.622 4.665		3.46 3.47 3.91	24.6 24.9 16.2	<u> 24.7</u>
.95	A we B C	t 4.530 4.811 4.616		4.54 3.42 3.25	31. 29.7	<u>31.</u>

Benzol added to solvent.

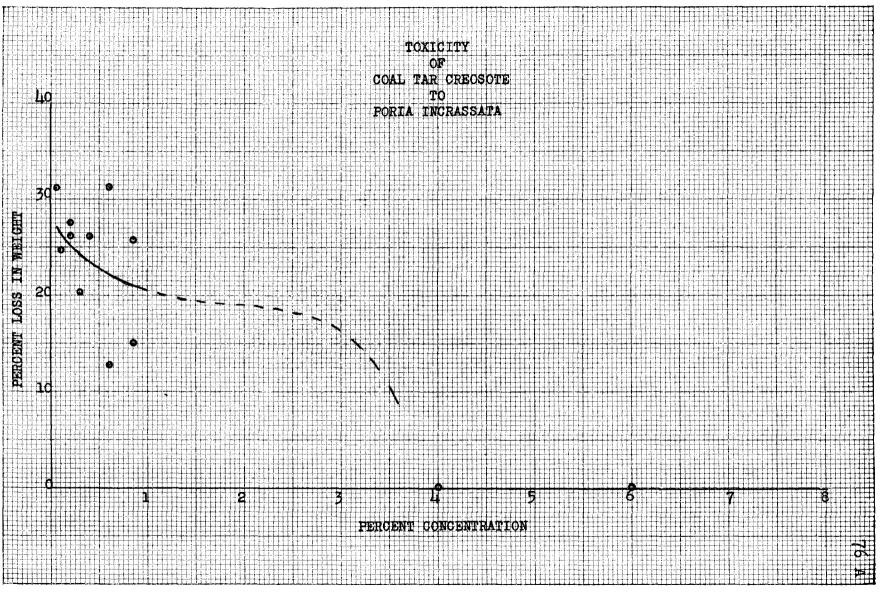


Table showing loss in weight of individual sample blocks that have been impregnated with different concentrations of 0il Tar Greosote after an incubation period of 8 weeks with Lenzites trabes. C is control.

Concen- tration percent	Sam- ple	Computed Oven Dry Weight	Adjusted Oven Dry Weight	Moisture Free Wt. after in- cubation	Percent Loss in Weight	Ave. % Loss
100	A B C	4.496 4.631 4.078	6.27 6.57 5.078	6.04 6.37 5.08		
50	A B C	4.396 4.403 4.317	5.07 5.11 4.317	5.11 5.01 4.15	attaok	
12	A B C	4.414 5.082	4.486 5.17		2	
6	A B C	4.509 4.407 5.265	4.593 4.47 5.265	4.17 4.48 3.40	9.2	<u>4.46</u> 21.84
4	A B C	4.476 4.507 4.760	4.58 4.541 4.760	4.21 3.16 3.46	8.08 30.4 37.5	
	A B C	4.438 4.435 4.658	4.451 4.548 4.658	3.165 3.16 3.67	28.9 28.9 21.2	28.9
.8	A B C	4.262 4.575 4.692	4.277 4.590 4.692	3.26 3.53 3.45	23.3 23.0 24.3	23.3
.6	A B C	4.404 4.785 4.540	4.415 4.798 4.540	3.50 4.00 3.02	25.2 16.6 33.4	<u>20.9</u>
•5	A B C	4.624 4.352 4.625	4.633 4.361 4.625	4.02 3.58 3.12	13.2 17.7 32.5	<u>15.4</u>
.4	A B C	4.765 4.410 4.376		3.74 3.40 3.15	21.4 22.9 28.0	<u>22.1</u>
•3	A B C	4.691 4.503		3.13 3.11	33.2 33.2	<u>33.2</u>
.2	A B C	4.688 4.465 4.702		3.58 3.80	23.6 14.8	<u> 19.2</u>
.1	A B C	4.314 4.330 4.907		3:12 3.40	28.1 27.4 30.7	27.7
.05	A B C	4.691 4.205 4.395	an a	3.46 3.23 3.00	26. 23.1 31.7	<u>24.5</u>

Table showing loss in weight of individual sample blocks that have been impregnated with different concentrations of Oil Tar Creosote after an incubation of 8 weeks with Poria incrassata.

Concen- tration percent	Sam- ple	Computed Oven Dry Weight	Adjusted Oven Dry Weight	Moisture Free Wt. after in- cubation	Percent Loss in Weight	Ave. % Loss
100	A B C	4.616 4.290	5.88 5.62	6.09 5.71		
50	A B C	4.503 14.595 14.906	5.10 5.15	5.10 5.20 4.99		
12	A B C	4.441 5.106 5.125	4.56 5.234	4.51 5.10	attack	
6	A B C	4.390 4.767 4.499	4.50 4.88	4.51 4.91 4.52	ŝ	
	A B C	4.419 4.701 4.454	4.496 4.77	4.51 4.83 4.44		
1	A B C	4.387 4.543 4.910	4.406 4.562	3.55 3.75 3.00	19.4 17.8 39.	18.6
.8	A B C	4.582 4.607 4.464	4.597 4.622	3.51 3.86 2.31	21.4 16.4 48.2	18.9
.6	A B C	4.702 4.454 4.632	4.713 4.465	3.33 3.66 2.84	29.4 18.0 38.9	23.7
•5	A B C	4.511 4.463 4.313	4.521 4.473	2.84 3.74 2.73	37 <b>.2</b> 16.4 36.9	21.8
•1	A B C	4.319 4.780 4.526	4.321 4.782	3.93 W	et 8.8 et 40.8	<b>41.</b>
•3	A B C	4.387 4.653 3.943		3.23 3.65 2.82	26.3 21.5 28.5	23.9
.2	A B C	4.459 4.909 4.416	wet	4.34 3.90 2.95	20.5 33.1	20.5
.1	A B C	4.557 4.532 4.146	******	3.80 3.99 3.03	16.6 11.9 32.1	<u>14.2</u>
.05		4.141 4.692 4.543		3.71 4.06 3.112	16.4 13.4 24.7	14.9

Table showing the percent of weight of untreated wood lost when exposed to 8 weeks incubation with fungus in Kolle flasks.

A. Lenzites trabea

		Sam- ple	Orig. Wt. at 12% Moisture Content	Computed oven dry wt. of wood alone	Dry Wt. at end of 8 wks.period	% Loss
Control Flask	2	A wet B	5.000	4.454	3.81 3.29	14.2 32.7
Flask	1	A B C	4.848 5.25 5.258	4.319 4.677 4.684	3.48 3.18	25.4 32.1

B. Porta incresseta

Flask	3	A B C	5.52 4.923 5.98	4.918 4.386 4.437	· · · · · · · · · · · · · · · · · · ·	· · · · · · · · · · · · · · · · · · ·
Flask	4	A B C	5.347 5.15 5.018	4.763 4.588 4.471	4.01 3.46 3.65	15.8 24.3 18.3

#### Check, or Second Series of Kolle Flask Tests

### Objective

The objective of this series of tests was 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 were 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.

### .Frocedure

Certain changes in procedure were followed in the second series of tests in order to comply with the standard procedure (33).

Since these changes seemed desirable the procedure for the second series is given in brief form:

1. Kiln dried Ponderosa pine blocks,  $1-3/8 \ge 2$  inches cross section by 1/4 inch in thickness were used.

2. Blocks conditioned for 21 days in a desiccator over a saturated solution of sodium bromide at a temperature of 23 degrees C. 3. After conditioning, the blocks were weighed to the nearest 1/100 gram.

4. 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.

5. Preparation of Preservative. Both coal tar and oil tar crecsote were diluted with benzol on the basis of weight to give the following concentrations:

1	percent	Creosote
3	<b>1</b>	
6	Ħ	*
8	11	Ħ

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

5	percent	Creosote
10	韓	

100 " Benzol was used in treating four control blocks in order to study the action of benzol.

6. Impregnation of Test Blocks. After weighing, 14 blocks were placed in a one 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 be completely covered with the solution. The blocks were weighed immediately after being removed from the treating solution.

7. 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.

8. Incubation. 10 of the 14 blocks were placed in kolle flasks on 1/8 inch glass rods. The fungus in the kolle flask had grown for a period of over two weeks and formed a mat over the surface of the media. The four remaining treated blocks were placed in kolle flasks over sterile agar. The 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 degrees C. for 64 days. Seventeen conditioned untreated blocks were also incubated with the fungus to serve as control blocks.

9. 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.

10. Computations. The computations were made in accordance with the following proportion formula:

Weight of re- Weight of re- Oven Dry Weight conditioned : conditioned :: of reference : X treated refer- treated blocks blocks ence 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 percent weight loss of the blocks due to fungus attack was then computed as follows:

Computed Oven \_ Actual Oven x 100 Dry Weight Bry Weight = % Loss in Weight Computed Oven Dry Weight

11. Results. These are shown in the tabulated form and also in graphic form by plotting percent loss in weight over concentration.

### Resistance to Volatilization

12. Twenty-sight blocks were treated with a 5% and also 28 blocks were treated with a 10% solution of creosote in Benzol.

13. 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.

14. The test blocks were exposed to a temperature of  $160^{\circ}$  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 as in item 10.

15. Results. Shown in tabular and graphic form. Resistance to Leaching by Water

16. Using the blocks subjected to the volatilization test (item 14), 14 treated blocks were placed in a one quart container, a vacuum drawn for 15 minutes and the vacuum broken with 1000 cc. of water. The water was then changed every hour for 7 hours the first two days, and every day for the remaining three 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 four days in the laboratory air, and then reconditioned for seven days as under item 2. The reference blocks were removed, oven dried, and weighed. The remainder of the blocks were incubated as in items 8 and 9.

17. Results. Shown in tabular and graphic form.

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 percent loss in weight of the blocks calculated.

The first 8 tables concern the toxicity tests made on blocks treated with 1, 3, 6 and 8 percent solutions of the creosote in benzol. The following 4 tables show the progress of the blocks treated with 5 and 10 percent solutions of creosote in benzol. The first 14 blocks in the 5 and 10 percent 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.

### 46.42 cc Benzol 3.72 cc Creosote

# Coal Tar Creosote 1% Solution in Benzol

Block No.	Original Condition- ed weight	Wt.After Treating	Wet wt. end	Computed Oven Dry Weight	Oven Dry Wt.	\$ Loss
C-1-1 C-1-2 C-1-3 C-1-4 C-1-5	4.41 4.35 4.46 4.46	10.36 10.46 10.20 10.58 10.84	10.25 8.26 4.80	4.10 4.04 4.14	2.10 1.92 <u>1.91</u> 4.18 R	50.0 50.0 60.2
Aver	4.0 <u>5</u> 4.555	10.01		Aver.	<u>4.28</u> R 4.23	

4406.4 co Benzol 3.87 co Creosote

# 011 Ter Creosote 1% Solution in Benzol

	Original Condition- ed weight	Wt.After Treating	Wet wt. end	Computed Oven Dry Weight	Oven Dry Wt.	% Loss
0-1-1- 0-1-2 0-1-3	x 4.53 4.53 <u>4.22</u>	10.12 10.54 10.07	8.23 5.32 5.58	4.18 4.18 3.89	2.71 2.90 2.64	35.1 30.62 32.1
0-1-4 0-1-5	4.60 4.58	10.28 10.36			4.27 R 4.20 R	
Aver	• 4.59			Aver.	4.235	

### 437.h cc Benzol 11.62 cc Creosote

Block No.	Origin- al con- dition- ed Wt.	Wt.After Treating	Wt.After Condition- ing	Wet Wt End		Dry Wt.	% Loss
0-3-1 0-3-2 0-3-3 0-3-4 0-3-5 0-3-6 0-3-7 0-3-8 0-3-9 0-3-10	4.23 4.47 4.25 4.275 4.215 4.32 4.175 4.175 4.15	7.65 7.49 7.68 7.60 7.60 7.42 7.42 7.26	4.28 4.49 4.275 4.30 4.265 4.36 4.25 4.20 4.20 4.18	4.31 5.22 5.75 10.00 5.21 5.02 5.39 5.10	3.89 4.08 3.98 3.98 3.98 3.98 3.98 3.98 3.98 3.9	2.62 2.66 2.52 3.54 2.52 2.52 2.52 2.15 2.15 2.50 2.41	32.6 35.9.2 91.1 315.7 25.7 25.7 25.7 25.7 25.7 25.7 25.7 2
0-3-11 0-3-12 0-3-13 0-3-14 Aver	4.16	7.74 7.47 7.00 7.54	4.45 4.18 4.06 4.26 4.238		3.72	R R R	

# 011 Tar Creosote 3% Solution in Benzol

Moisture Content = 10.7% and 11.05%

### 437.4 cc Benzol 11.16 cc Creosote

# Coal Tar Greesote 3% Solution in Benzol

Block No.	Origin- al con- dition- ed Wt.	Wt.After Treating	Wt.After Condition- ing	Wet Wt. End	Com- puted Oven Dry Wt.	Dry Wt.	loss
C-3-1 C-3-2 C-3-3 C-3-4 C-3-5 C-3-5 C-3-6 C-3-6 C-3-7 C-3-8 C-3-9 C-3-10 C-3-11 C-3-12 C-3-14 C-3-14 C-3-14 C-3-14	4.50	7.00 7.49 7.526 7.526 7.5420 7.131 7.567 7.567 7.567 7.567 7.23	4.06 4.39 4.455 4.22 4.33 4.29 4.33 4.29 4.13 4.29 4.13 4.29 4.13 4.29 4.13 4.29 4.13 4.29 4.13 4.29 4.13 4.29 4.13 4.29 4.13 5 4.15 5 4.22 4.22 4.23 4.23 5 4.22 4.23 5 4.22 4.23 5 4.22 4.23 5 5 4.22 4.23 5 5 4.22 4.23 5 5 4.22 4.23 5 5 5 4.22 4.23 5 5 4.22 4.23 5 5 5 4.22 4.23 5 5 5 4.22 4.23 5 5 4.22 4.23 5 5 4.22 4.23 5 5 4.22 4.23 5 5 4.22 4.23 5 5 4.23 5 5 4.22 4.23 5 5 4.23 5 5 4.23 5 5 4.23 5 5 4.23 5 5 4.23 5 5 4.23 5 5 4.23 5 5 4.23 5 4.23 5 5 4.23 5 4.23 5 5 4.23 5 4.23 5 4.23 5 4.23 5 4.23 5 4.23 5 4.23 5 4.23 5 4.23 5 4.23 5 4.23 5 5 4.23 5 5 4.23 5 5 4.23 5 5 4.23 5 7 4.25 5 7 4.25 5 7 5 4.25 7 5 4.25 7 5 4.25 7 5 4.25 7 5 7 5 4.25 7 5 7 4.25 7 5 7 4.10 7 5 7 5 7 5 4.10 7 5 7 5 4.10 7 5 7 5 7 5 4.10 7 5 7 5 7 5 7 5 7 5 7 5 7 5 7 5 7 5 7	4.95 8.99 5.67 8.72 12.85 5.00	3.78 4.09 4.15 3.93 4.04 3.94 4.09 4.09 4.08 5 4.08	2.31 3.37 2.59 3.32 3.15 3.37 3.37 3.37 3.60 2.57 4.03 R R 4.03 R R 4.05	

Moisture Content = 9.69% and 11.6%

### 423.9 cc Benzol 23.2 cc Creosote

# 011 Tar Creesote 6% Solution in Benzol

Block No.			Wt.After Wet Wt. Condition- End ing	Com- puted Oven Dry Wt	Oven Dry Wt.	% Loss
1234567890	4.85 4.56 4.55 4.59 4.56 4.80 4.86 5.10 4.84	7.94 7.76 7.47 7.59 7.59 7.60 7.84 8.24 7.95	$\begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	4.49 4.190 4.03 7.39 4.03 97 4.43 4.43 4.43 4.43 4.43 4.43 4.43 4.4	3.86 3.98 3.44 3.64 3.64 3.66 3.60 3.95 3.99 3.84 3.84	11.2 9.9 13.5 9.7 9.8 9.1 24.8 7.8 7.8 12.8 13.3
11 R 12 R 13 R 14 R Aver.1	4.62 4.765 4.93 4.63 R 4.736	7.63 8.00 8.01 7.82	4.70 4.84 5.00 4.72 4.82 Aver.0ven	Dry R	4.27 4.42 4.50 4.20 4.347	

R = Reference Blocks Moisture Content = 10.27 and 10.47% See also M.C. on 5% oil tar = 10.94%

### 414.8 cc Benzol 31.0 cc Creosote

# 011 Tar Creosote 8% Solution in Benzol

Block Origin- No. al con- dition- ed Wt.				Com- puted Oven Dry Wt.	Oven % Dry Wt.	Loss
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	7.53 7.00 7.35 7.10 7.48 7.29 7.39 7.35 7.28	4.72 4.185 4.495 4.545 4.545 4.51 4.51 4.54	5.88 5.37 5.17	4.22 4.72 4.06 4.98 4.88 4.88 4.06	3.86 3.53 3.92 3.89 4.16 3.68 4.00 3.74 3.97	852.1.3.30.6.2
0-8-11 4.31 0-8-12 4.175 0-8-13 4.55 0-8-14 4.305	7.22 7.36 7.42 7.35	4.44 4.315 4.67 4.425			3.95 R 3.84 4 <b>.22</b> R <u>3.95</u> R	
Aver. 4.425		4.46			3.99	
0-8-4 4.35	7.35	4.465		4.04	3.92	2.9
Moisture	Content	10.09%	and 1	1.0%		

414.8 cc Benzol 29.76 cc Creosote

Block No.	Origin- al con- dition- ed Wt.		Wt.After Condition ing		Com- puted Oven Dry Wt	Dry Wt.	Loss
C-3- 1 C-8- 2 C-8- 3 C-8- 4 C-8- 5 C-8- 5 C-8- 7 C-8- 7 C-8- 9 C-8-10	4.69 4.36 4.22 4.17 4.46 4.56 4.57 4.315 4.322	7.47 7.40 7.40 7.19 7.58 7.81 7.25 7.35	4.845 4.52 4.37 4.30 4.56 4.70 4.68 4.435 4.455	5.05 6.50 6.73 5.25	4.43 4.15 4.05 99 4.35 4.35 4.35 4.30 7 4.09	4.39 4.03 3.95 3.83 4.22 4.16 4.21 3.98	0.9840.30.40.49
C-8-11 C-8-12 C-8-13 C-8-14 Aver.	4.32 4.75 4.33 <u>4.36</u>	7.10 4.52 7.27 7.56	4.435 4.88 4.43 4.47 4.554			4.04 R 4.32 R 4.30 R 4.08 4.185	

# Coal Tar Creosote 8% Solution in Benzol

Moisture Content = 10.38%, 10.88%, 10.5% and 10.03%

# 423.9 cc. Benzol 22.3 cc Creosote

# Coal Tar Creosote 6% Solution in Benzol

Block No.	Origin- al con- dition- ed Wt.		Wt.After Condition- ing	Wet Wt End	. Com- puted Oven Dry Wt	Oven % Lry Wt.	LOSS
X-6- 1 X-6- 2 X-6- 3 X-6- 4 X-6- 5 X-6- 5 X-6- 5 X-6- 7 X-6- 8 X-6- 9 X-6-10	4-36 351 4-4-4-3 36 351 4-4-4-4 5-4-4-29	7.09 7.36 7.19 7.19 7.19 7.19 7.19 7.19 7.19 7.28 7.28 7.28 7.28 7.28	4.33 4.44 4.45 4.44 4.44 5.95 4.44 5.95 4.55 4.5	9.55 13.34 11.60 13.16 13.14 7.65 7.65 7.03	3.87 990 3.990 3.990 3.997 3.997 4.403 7.44 8.87	3.86 4.00 4.01 3.92 3.94 3.98 4.31 4.08 3.96	0.0 0.0 0.0 0.0 0.0 5.7 4.0 0.0
X-6-11 X-6-12 X-6-13 X-6-14 Aver	4.34 4.40 4.35 <u>4.25</u> . 4.37	7.20 7.20 6.50 7.30	4.42 4.47 4.42 4.32 4.407			3.96 R 3.92 R 4.00 R <u>3.88 R</u> 3.94	

# 1262.6 cc Benzol 58.1 cc Creosote

### 011 Tar Creosote 5% Solution in Benzol

Block Origin- No. al con- dition- ed Wt.	Wt.After Treating	Wt.After Condition- ing	Wet Wt End	Dry Wt	Oven Dry Wt.	% Loss
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	7.50 7.67 7.49 8.01 7.61 7.57 7.82 8.23	4.36 4.722 4.57 4.55 3.37 4.05 5.01	6.07 6.82 6.60 13.42 11.66 6.65 11.00 8.00 8.49	3.96 1.39 1.256 1.256 1.256 1.256 1.256 1.256 1.256 1.256 1.256 1.256 1.256 1.256 1.256 1.256 1.45 1.256 1.45 1.45 1.45 1.45 1.45 1.45 1.45 1.45	2.35 2.45 3.93 3.64 3.79 2.90	46.5 35.72 35.0 31.0 31.5 34.6 36.2
0-5-11 4.28 0-5-12 4.83 0-5-13 4.64 0-5-14 4.715 Aver. 4.616	7.27 7.95 7.68 7.90	4.34 4.87 4.71 4.78 4.675			3.95 4.44 4.28 4.34	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	7.88 7.680 7.680 7.680 7.680 7.680 7.680 7.685 7.685 7.885 7.685 7.685 7.685 7.48 7.48	4.7175896015511	11.78 13.84 12.92 11.48 6.13 13.50 13.10 13.75 11.50	4.25 4.12 3.4 4.93 4.19 3.8 8 8 117 4 4.07	4.00 30 59 30 30 30 30 30 30 30 30 30 30 30 44 44 30 30 30 30 30 44 44 30 30 30 30 30 44 44 30 30 30 30 30 30 30 30 30 30 30 30 30	5.88 2.2 18.7 30.7 1.0 24.6 0.7 1.7
Aver. 4.24		4.255			3.84	

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

# 1014.6 cc Benzel 96.89 cc Creosote

011	Tar	Cre	osote	10%	Solution	1n	Benzol

Block No.	Origin- al con- ditkon- ed Wt.	Wt.After Treating	Wt.After Condition- ing	Wet Wt End	. Com- puted Oven Dry Wt	Oven Dry Wt.	% Loss
0-10- 0-10- 0-10- 0-10- 0-10-	4.59 4.59 4.58 4.58 4.45 4.45 4.45 4.45 4.45 4.45	7.56 8.04 7.63 7.63 7.539 7.539 7.55 7.55 7.65 7.65 7.65 7.65 7.69 7.69 7.69 7.69 7.69 7.69 7.69 7.69	4.63 4.68 4.475 4.69 4.69 4.77 4.99 4.99	8.55 7.60 7.08 8.59 9.00 13.82 6.72 12.79	4.271125737094	4.19 4.19 4.19 5.90 5.12 4.19 5.12	1.8 1.00 2.00 2.00 2.00 2.00 2.00 2.00 2.00
Aver	. 4.69		4.89		4.40		
0-10-19 0-10-19 0-10-19 0-10-19 0-10-29 0-10-29 0-10-29 0-10-29 0-10-29	4.66 4.66 4.38 5.06 4.72 4.46 4.66	7.92 7.63 7.51 7.93 7.28 7.28 7.54 7.26 7.33 6.98	4.60	5.61 9.19 7.32 6.90 10.52 9.70 10.55	4.30 4.20 4.21 4.28 4.01 4.57 4.30 4.307 4.307 4.28 4.30 4.30 4.30 4.30 4.30 4.30 4.30 4.30	4.17220062543.665	3.0 1.9 0.6 12.0 12.5 31.7
0-10-29 0-10-29 0-10-29 0-10-28	5 4.29	7.40 7.02 7.46 7.50	4.84 4.41 4.78 4.77		4.29 3.90 4.21 4.23		
Aver	4.59		4.70		4.16		

Moisture Content = 12.7%, 10.4% and 11.02%

## 1262.6 cc Benzol 55.7 cc Creosote

Coal Tar	Creosote	5%	Solution	1n	Benzol

Block No.	Origin- al con- dition- ed Wt.	Wt.After Treating	Wt.After Condition- ing	Wet Wt End	. Com- puted Oven Dry Wt.	Oven Dry Wt.	% Loss
x-8- 1 x-8- 2 x-8- 3 x-8- 3 x-8- 4 x-8- 5 x-8- 6 x-8- 7 x-8- 6 x-8- 7 x-8- 8 x-8- 9 x-8-10 x-8-11 x-8-12 x-8-13 x-8-14	44.44.44.44.44.44.44.44.44.44.44.44.44.	7.75 7.334 7.3460 7.3460 7.32 7.388 7.540 7.540 7.72 7.540 7.72 7.72 7.72 7.72 7.72 7.72 7.72 7.7	5.00 4.66 4.41 4.285 4.335 4.355 4.35 4.35 4.35 4.35 4.35 4	11.58 9.85 12.52 5.68 12.31 5.00 5.30 11.00 7.70	4.51 4.21 3.98 3.87 4.41 3.91 3.92 3.77 4.21 4.13	2.67 2.80 3.48 2.95 3.09 2.95 3.09 2.75 2.44 4.10 4.86 3.40 3.40	36.1 33.4 128.3 23.1 23.1 27.6 9
Aver.	4.39		4.429			3.997	?
x-8-15 x-8-16 x-8-17 x-8-18 x-8-19 x-8-20 x-8-21 x-8-22 x-8-23 x-8-24 x-8-25 x-8-24 x-8-25 x-8-26 x-8-27 x-8-28	4.80 4.80 4.80 4.80 4.80 4.25 4.25 4.25 4.25 2.52 0.8 4.4 4.87 4.87 4.87 4.87 4.87 4.87 4.8	7.44 7.32 7.59 7.30 7.49 7.16 7.32 7.32 7.32 7.35	4.87 4.21 4.38 4.32 4.58 4.58 4.55 4.87 4.55 4.89 4.93 4.93 4.93	9.00 9.17 9.52 7.52 11.62 8.22 6.72 6.18	4.40 4.391 3.96 3.91 4.10 4.40 4.11	3.63 3.00 2.58 2.49 3.00 3.31 3.00 3.60 3.60 3.60 3.00 4.43 4.43 4.43 4.43	17.5 21.4 34.8 36.3 27.5 14.4 26.8 18.1 27.0
Aver.	4.726	>	4.78			4.322	25

# 1116.1 cc Benzol 102.3 cc Creosote

Block No.		Wt.After Treating	Wt.After Condition- ing	Wet Wt. End	Com- puted Oven Dry Wt.	Dry Wt.	% Loss
X-10- X-10- X-10- X-10- X-10-	234560 44.560 44.560 44.562 44.561 44.861 44.861 44.861 44.861 44.861 44.861 44.861 44.861 44.860 44.5600 44.5600 44.5600 44.5600 44.560000000000000000000000000000	7.845 7.77 8.19 8.00 8.14 7.96 8.29 7.61 8.30 7.58 7.78 7.78 7.78 7.31 8.01 7.46	4.66 4.73 5.05 4.72 4.84 4.92 4.63 5.09 4.63 5.09 4.75 4.75 4.75 4.75 4.76	5.63 7.48 8.00 5.20 5.55 10.38 13.68 12.18	4.2176777738001 4.4.52377738001 4.4.4.4.4.4.4.4.4.4.4.4.4.4.4.4.4.4.4.	3.80 3.75 3.05 3.75 3.27 5.29 4.07 4.251 3.89	9.7 12.6 33.1 11.9 18.6 20.2 2.6 7.5 9.7
Aver	. 4.55		4.76		4.30		
X-10-19 X-10-19 X-10-19 X-10-19 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29 X-10-29	4.49 4.49 4.49 4.49 4.78 4.78 4.78 4.78 4.78 4.78 4.78 4.78 4.78 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 4.65 12 12 12 12 12 12 12 12	7.43 8.09 8.08 7.59 7.57 7.61 8.23 8.17	4.64 4.85 4.66 4.65 4.65 4.69 5.81 5.89 4.71 4.90 4.71 4.90 33 4.83	7.25 7.31 6.00 8.00 11.93 7.40 10.72 7.62 7.55	4.	3.16 3.13 2.78 2.29 3.00 3.52 2.73 3.16	22.8 347.9 317.4 317.4 39.8 39.8
Aver	. 4.65		4.87		4.295		

Coal Tar Creosote 10% Solution in Benzol

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

#### CONTROLS

Key	Original Conditioned Weight	Net Wt. End	Computed Oven Dry Weight	Oven Dry Wt.	% Loss
A-1 A-2 A-3 A-4 A-5	4.33 4.505 4.69 4.56 4.68	10.76	3.904 4.06 4.38 4.12 4.22	3.07 3.22 3.52 2.90 3.00	21.4 20.7 14.9 28.36 28.9
	Moisture Conten	t • 0.T.	- 6% - 10.94	X.	
B-1 B-2 B-3 B-4 B-5 B-6	4.38 4.37 4.23 3.935 4.295 4.29	8.17 8.32 8.34 10.20	3.86 3.94 3.817 3.55 3.876 3.763	3.18 2.43 2.57 2.41 2.40 2.49	17.6* 38.3 32.16 32.1 38.0 33.85
	Moisture Conten	t · 0.7.	and C.T. at 3%	- 10.7	5
C-1 C-2 C-3 C-4 C-5 C-6	4.68 4.47 4.26 4.43 4.24 4.97	7.00 6.15 10.15 6.26	4.039 3.84 3.99 3.82 4.391	3.36 3.00 3.00 3.08 2.65 2.96	20.4 25.7 21.87 23.2 30.6 32.59
			r untr		

Moisture Content = 10.85%

# BENZOL TREATED CONTROLS

Block No.	Original Conditioned Weight	Wet Wt.	Computed Oven Dry Weight	Oven Lry Wt.	% Loss
1234	4.16	10.45	3.85	2.37	38.4
	4.90	6.45	4.54	2.85	37.2
	4.17	6.08	3.86	2.51	34.9
	4.265	9.24	3.94	2.76	30.05

Aver. 35.1

Moisture Content = Noom Dry - 8%

\* Six weeks

#### Results

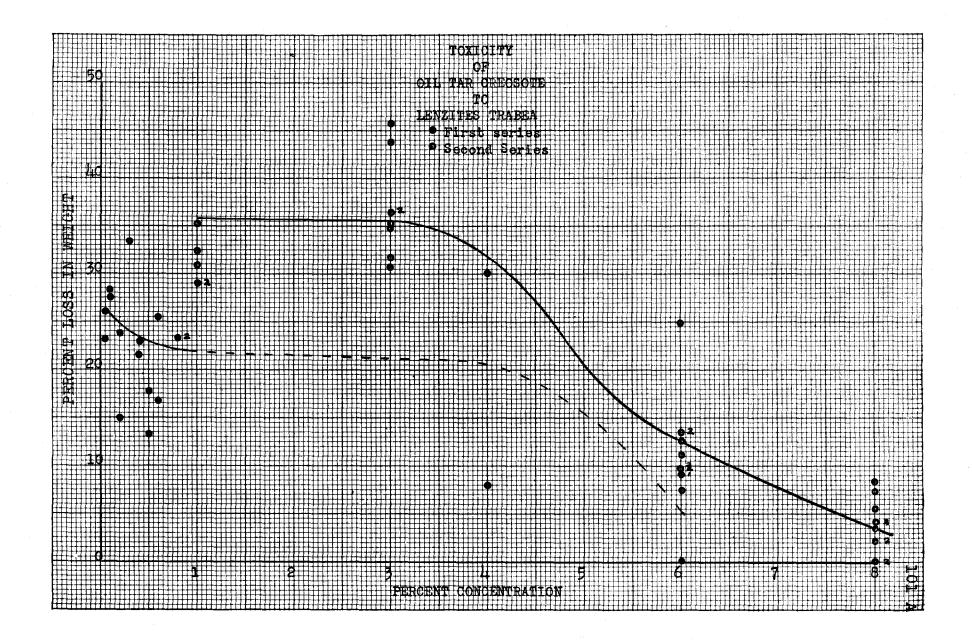
The results of the second series of toxicity tests are given in the following tables. The results of the first series are not tabulated because it was decided that the first series was inconclusive due to insufficient sampling. The percentages which are underlined have not been considered as true samples because of probable experimental error, either the samples had taken up too much moisture from the agar, were dried out during the incubation period, or had deviated too far from the mean. Summary table showing the percent loss in weight of wood blocks treated with different concentrations of creosote wnd subsequently exposed to Lenzites trabea for 64 days.

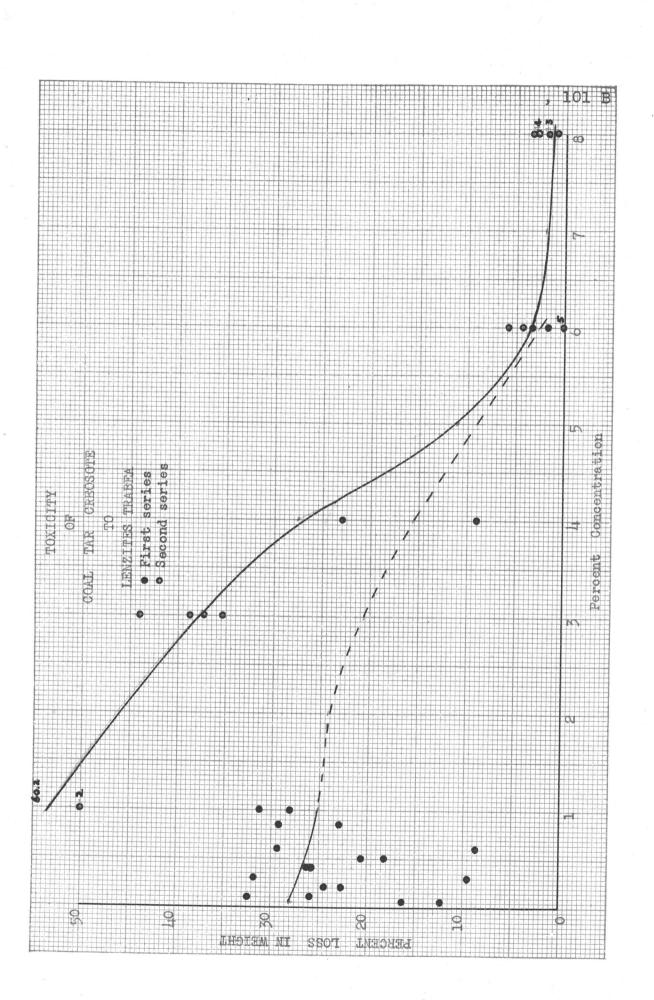
011 Tar Creosoted Samples						
Block No.	Cone	entration of T	reating Solution	on <b>s</b>		
	1%	3%	6%	8%		
1 2 3 4 5 6 7 8 9 10	35.1 30.62 32.1	32.6 34.8 35.2 9.4 11.1 31.8 45.5 43.7 36.2 36.5	11.2 9.9 13.5 9.7 9.8 9.1 24.8 7.5 12.8 13.3	8.5621		
Nean	32.61	37.03	12.16	3.78		

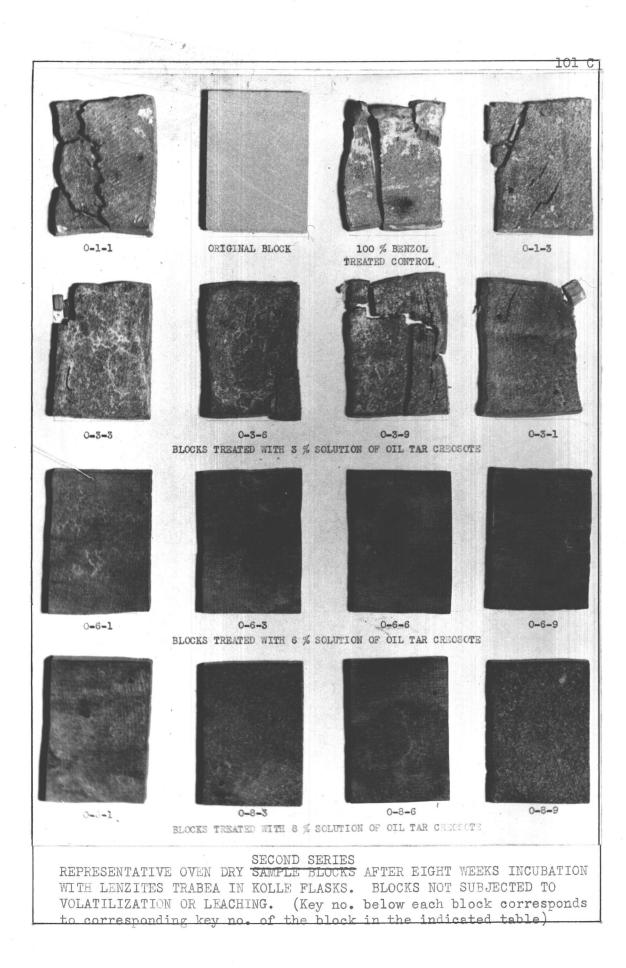
#### Coal Tar Creosoted Samples

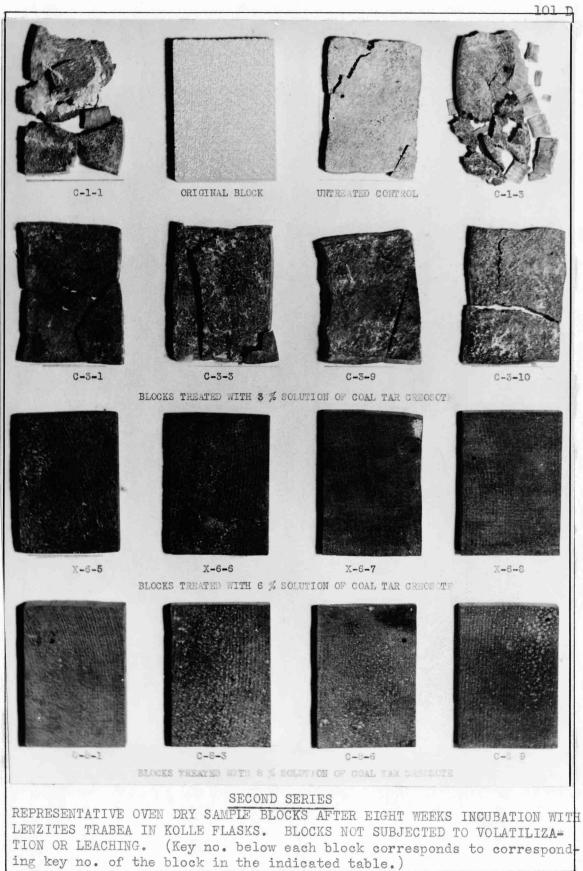
1234567	50.0 50.0 60.2	$   \begin{array}{r}     38.7 \\     \underline{17.6} \\     \overline{37.3} \\     \underline{15.6} \\     21.7 \\     8.4 \\     14.5 \\   \end{array} $	$ \begin{array}{c} 0.0 \\ 0.0 \\ 0.0 \\ 0.0 \\ 0.0 \\ 0.0 \\ 5.7 \\ \end{array} $	.9 2.8 1.4 3.0 3.3 3.0
8 9 10 Mean	53.4	$\frac{10.0}{35.3}$ 39. 37.57	4.3 0.0 <u>0.0</u> 1.43	2.0 2.4 2.7 2.61

The above data is also shown on the accompanying graphs. The results of the first or orientation series is also shown on the same graph.









The results of the second series of tests on volatilization and leaching are shown in the following table.

Table showing the percent loss in weight of treated wood blocks after volatilization at 160 degrees F. in the oven for 24 hours and subsequently incubated with Lenzites trabea for 8 weeks.

Cr	l Tar cosote lution	Coal Tar Creosote 5% Solution	Oil Tar Creosote 10% Solution	Coal Tar Creosote 10% Solution
	5.3 7.0 14.5 31.0 34.6 35.7 36.2 36.2 46.5 40st	$   \begin{array}{r}     12.5 \\     21.1 \\     23.2 \\     27.3 \\     33.1 \\     33.4 \\     34.6 \\     36.1 \\     40.9 \\     48.3 \\   \end{array} $	0.0 0.0 1.3 1.8 2.7 2.8 3.5 6.3 6.8 22.2	2.6 4.2 7.5 9.7 9.7 11.9 12.6 18.3 20.6 23.1
Mean	36.7	33.1	5.92	16.56

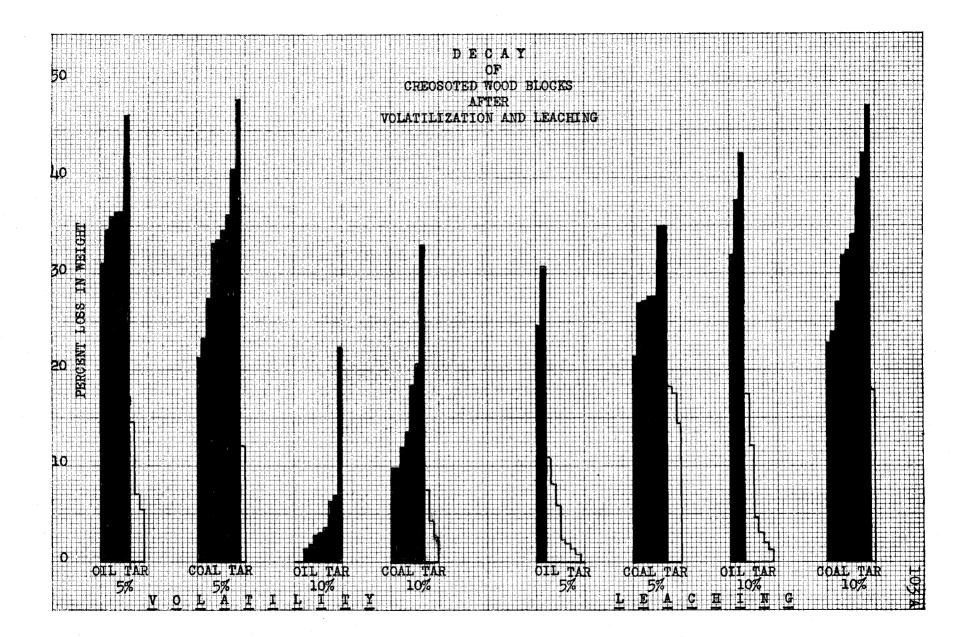
(Samples above the horizontal line rejected on the basis of experimental error, i.e., samples varied too greatly from mean due to high moisture content of sample during incubation or dessication of sample during incubation.)

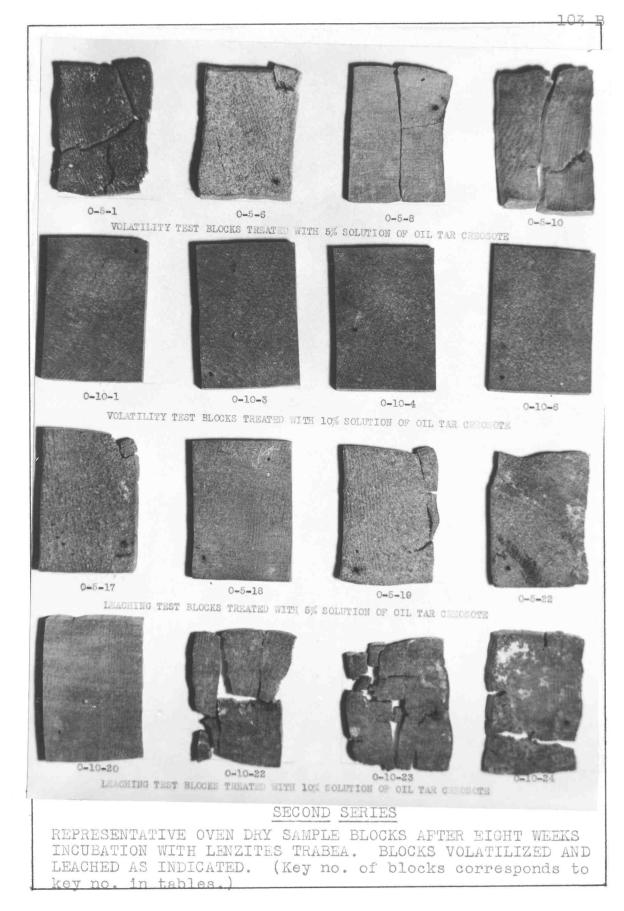
The above results are shown in the accompanying Bar Chart. Each horizontal division on the chart indicates one sample. Table showing the percent loss in weight of treated wood blocks after volatilization at 160 degrees F. for 24 hours, leaching with water for 5 days, and subsequently incubated with Lenzites trabea for a period of 8 weeks.

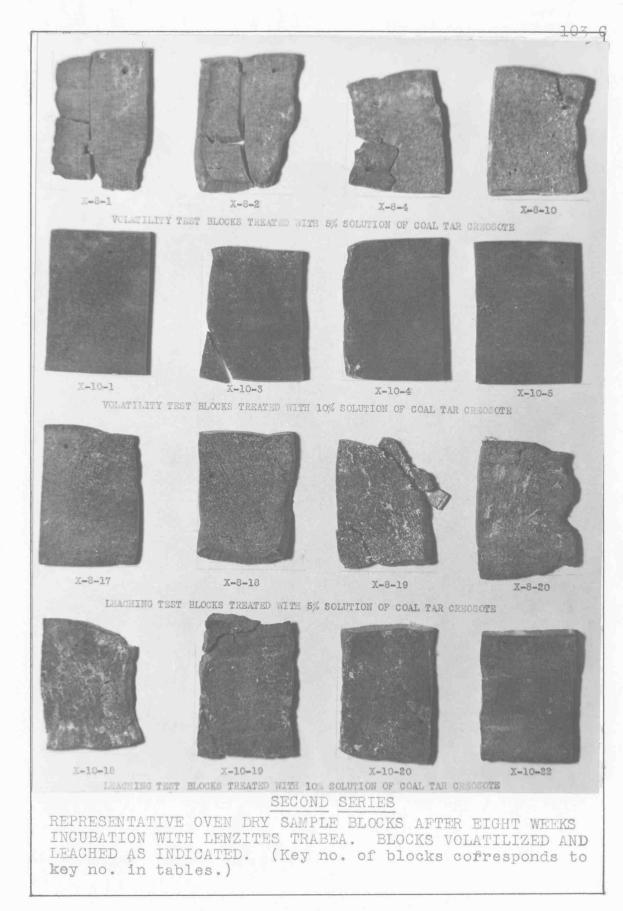
011 Tar Creosote 5% Solution	Coal Tar Creosots 5% Solution	011 Tar Creosote 10% Solution	Coal Tar Crecsote 10% Solution
0.7	14.4	0.0	$\frac{17.8}{22.7}$
1.7 1.7	$\frac{18.1}{21.4}$	1.9	23.8
2.2 5.88	26.8 27.0	4.6 12.0	31.9 32.3
$\frac{10.7}{21.5}$	2(.5 34.8	$\frac{17.4}{31.7}$	34.1 39.9
30.7	34.8 Lost	42.5	特进
Mean 27.65	28.71	37.26	33.48

(Samples above the horizontal line rejected as shown above.)

The above results are shown in the accompanying Bar Chart. Each horizontal division on the chart indicates one sample.







#### Discussion

An inspection of the data indicates 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 fact that the oil tar creosote has a higher percentage of high boiling oils than the coal tar creosote.

An examination of the data shows that there is no significant difference in the attack of the fungus when the blocks were treated with concentrations of less than 3 percent. In fact the percent of wood lost through decay in the samples so treated exceeds the loss in the untreated control blocks. At the low concentrations the greater percent loss may be attributed to the stimulating action of the dilute solutions. This stimulation has been established by other workers as a likely happening. (2h)

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

Examination of the treated blocks showed definite decay in the 6% coal ter treated blocks, While the remaining 5 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% oil ter creosoted blocks there was one sample, namely no. 7, which 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 bhought desirable to get as severe a test as possible in evaluating the two oils. This in spite of probable error.

According to the data presented here the average loss in the coal tar creosoted blocks, using an 8% solution was more than the average loss in the wood blocks treated with a 6% solution of coal tar creosote. A recheck of the computations and of the oven dry weights was made without changing

the results. However, if the standard error of the average is considered the apparent discrepancy is reduced to .55 percent 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 percent solution of coal tar creosote showed no apparent disintegration due to decay.

An examination of the blocks treated with an 8% 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 percent loss in the blocks treated with a ten percent solution of oil tar was found to be 5.92 as against an average loss of 16.56 percent in the blocks treated with a 10 percent 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 on the bar chart in the uncolored portion of the chart. There appeared to be no significant difference in the samples treated with a 5% solution of Oil tar creosote on those treated with a 5% solution of coal tar creosote.

In the leaching tests, on the basis of the accepted samples, the difference, if any, appears to be 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 percent as against a loss of 33.48 for the coal tar creosoted samples. A difference of 3.78 percent. It is believed that this difference is not significant. However, if all of the samples were to be used in computing the average, the average percent 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% solution instead of a 10% 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 which was present in sufficient quantities in the 10 percent treated blocks but not in sufficient quantity in the 5 percent treated blocks to cause active stimulation to the fungus. The maximum loss of 38 percent in the untreated control blocks as compared to the maximum loss of 47 percent in the blocks treated with the ten percent solution of coal tar creosote would lead one to believe that the latter case was 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 that the maximum losses in both the concentrations appear in the coal tar crecsoted 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 sample studied in its ability to act as a wood preservative. This is on the basis that a wood preservative must not only be toxic but have the ability to stay in place under service conditions.

#### PART V.

#### 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 one to believe that their presence may not be essential for a long lasting preservative. This opinion is substantiated by the pathological studies in Part IV 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 creosote.

B. The percent 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 percent of the weight of creosote lost from open dish volatility tests was found to be appreciably more in the case of coal tar creosote.

3. The comparable leaching tests, based on the percent 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 trabes 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 crossote tested appears to be somewhat higher than the cil tar creosote. The inhibiting point for the growth of Lenzites trabes appears to be slightly in excess of an eight percent treating solution for both oils. The inhibiting effect of coal tar creosote appears to be slightly greater than the cil tar creosote when the wood blocks were treated with both the 6 and 8 percent 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 the sample of coal tar tested. This was especially true at room temperatures. This factor is of importance when considering the dispension of the oil in the wood after the treating process.

6. The cil 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 .01.

7. The high temperatures used in the cracking of petroleum oil residium produces a tar which contains a very high percentage of aromatic hydrocarbons. As has been shown by literature citations, the aromatic hydrocarbons are more toxic than the phenols.

8. It is believed that the oil tar crossote possesses all of the requirements of a good wood preservative. It is almost as toxic as the standard grade 1 coal tar crossote tested yet 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 coest 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 which 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 which are entirely comparable to coal tar creosote.

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#### APPENDIX A

#### Statistical Summary

In order to determine the slope of the curve as well as determining the probable accuracy of the data, the curves were balanced and the standard deviation, probable error, and standard error of the average computed.

An examination of the toxicity curves for the first series of tests against both Lenzites trabea and Poria incrassata will reveal the absence of significant data above the 1% concentration point. It was decided therefore to use the data above 1% merely as control points for the samples between 0 and 1% concentration.

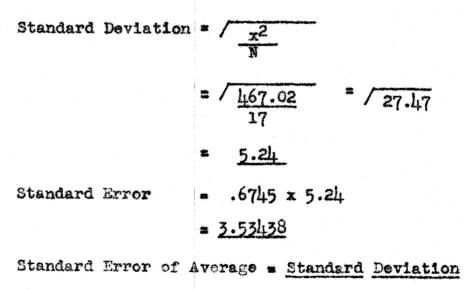
It should be explained that the <u>standard deviation</u> is a measure of the grouping of the samples and of course the smaller the deviation the greater is the probability that the data represents an accurate average.

The <u>probable error</u> as computed from the standard deviation indicates that approximately 68 times out of 100, any random sample taken under similar conditions will fall either below or above the true average, within the limits indicated by the computation.

The standard error of the average means that no matter how many samples are taken, the average of all of the samples taken will not move, either above or below the present average, more than the units indicated by the computation.

### Statistical Analysis for Oil Tar Creosote against Lenzites trabea between O gnd 1% Concentrations

Deviation	Deviation <sup>2</sup>
- 2.4	88.36 88.36
- 9.4 - 5.7	32.49
-4.9 - 2.9	24.01 8.41
- 1.5	2.28
- 0.6 0.0	.36
0.0	0.00
1.0 1.7	1.00
2.2	4.84
2.9	7.84 8.41
7.2 7.2	51.84 51.84
9.7	94.09
	467.02



 $= \frac{5.24}{17}$ = 1.27

#### Statistical Analysis for Coal Tar Creosote against Lenzites trabea

Deviation		Deviation <sup>2</sup>
+ 4.4 - 2.1 - 2.8 - 4.5		19.36 4.41 7.84 20.25
+ 5.1 + 0.0 - 5.2		26.01 0.0 1.60 27.04
- 7.7 + 3.7 - 2.6 + 3.9		59.29 13.69 6.76 15.21
72.8 76.0		7.84 <u>36.00</u> 245.30
Standard Deviat	ion = /	x <sup>2</sup>

			n
			$= \frac{21+5.30}{14}$
			= 4.19
Standard	Error		= 4.19 x .6745
			<b>a</b> 2.726
Standard	Error	of	Average = / N
			- <u>4.19</u>
			/11-
			= 1.116

Note: The samples at .05% concentration although plotted were not used in the average because the media had dried out and hence were not characteristic samples.

> One sample each at .3 and .6% concentration were not used because they had obviously picked up moisture from the agar and hence were not characteristic samples.

### Statistical Analysis for Coal Tar Creosote against Poria incrassata

Deviation	$Deviation^2$
$ \begin{array}{r} -1.6\\ -1.3\\ -3.8\\ -9.3\\ -6.2\\ \neq4.0\\ \neq1.2\\ \neq1.5\\ \neq2.7\\ \neq8.0\\ \neq4.7\\ \end{array} $	2.56 1.69 14.4 86.49 38.44 16.00 1.44 2.25 7.29 64.00
7 4 • (	256.65

Standard	Deviation	$-\frac{256.65}{11}$ = $-\frac{23.32}{11}$
		• 4.83
Standard	Error	= .6745 x 4.83
		• 3.2578
Standard	Error of	Average = $\frac{4.83}{11}$
	-	= 3.32

Note: Three samples showed only comparatively small loss in weight due to moisture conditions and hence were not used in the average.

## Statistical Analysis for Oil Tar Creosote against Poria incrassata

Deviation	Deviation <sup>2</sup>
-11.2 - 10.0 - 7.0 - 5.9 - 2.0 - 5.9 - 2.0 - 2	125.4 100.0 49.0 42.25 15.21 8.41 4.0 4.0 25 .16 310.96 256.00 72.25 18.49 1.0
	1007.38

Standard	Deviation	$= \frac{1007.38}{15} =$	67.16
Standard	Error	<ul> <li>8.19</li> <li>.6745 x 8.19</li> <li>5.524</li> </ul>	
Standard	Error of /	$1 \text{ verage} = \frac{5.54}{15}$ = 1.427	= <u>5.54</u> 3.88

Statistical analysis for the curves, the second series of tests of the toxicity of creosote against Lenzites trabea.

011 Tar (	Creosote Curve	Coal Tar Creos	ote Curve
Deviation	Deviation <sup>2</sup>	Deviation	Deviation <sup>2</sup>
	.04 $.36$ $.49$ $1.00$ $2.56$ $2.56$ $4.34$ $4.84$ $6.25$ $9.61$ $10.24$ $14.44$ $15.21$ $16.00$ $22.09$ $27.04$ $.09$ $.16$ $.25$ $.36$ $.49$ $1.21$ $1.69$ $3.24$ $12.25$ $22.09$ $62.41$ $94.09$ $158.76$ $510.10$	3 - 2.3 - 3.25 - 3.25 - 3.25 - 3.25 - 3.40 - 1.05 - 1.1 - 1.2 - 1.1 - 1.2 - 1.4 - 1.2 - 1.4 - 1.8 - 1.8 - 2.1 - 2.2 - 2.45 7 	$\frac{1.77}{23}$ = 2.63 = 1.7739
Standard	Deviation = $\frac{510}{31}$ .		$= \frac{2.63}{23}$
Standard	Error = 4.05 x	.6745 = 2.732	<b>= .</b> 546
Standard	Error of Average :	$\frac{4.05}{31}$ = .727	

#### APPENDIX B

## Cresol-Greesote and Pentachlorophenol Creosote Solutions

#### The Addition of Cresylic Acid and Its Significance

In order to meet all of the requirements of the tests for tar acid content as set up by the American Wood Preservers' Association, in their requirements for creosote oil, it has been suggested that cresol, probably better known as cresylic acid, be added to the oil tar creosote.

The addition of 5 percent by weight of cresol would still make the manufacture of the cil tar creosote possible. As has been shown, however, by Gellerman and Schmitz and Rhodes, the presence of cresylic acid may not be one of the main attributes of creosote oil.

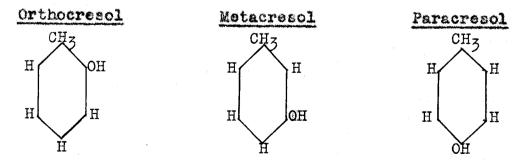
It was proposed, therefore, to make a series of comparable determinations between the straight oil tar creosotes and oil tar creosotes with the addition of 5 percent cresylic acid, the results of which are shown at the end of this report.

Past experiences with creyslic acid added to petroleum fuel cil to form the proprietary preservative known as "Cresoil" have not been too encouraging (12,48). However, it must be pointed out that in the first place, petroleum fuel cil is almost entirely of paraffin composition and in itself not toxic. Cresol, on the other hand, being an aromatic would have somewhat different characteristics which would affect its miscibility with the paraffins as compared with what could be expected with miscibility with other aromatics. "Aromatics are more miscible with aromatics than with paraffins" (18). It therefore follows that cresol in mixture with the aromatic oil tar creesote would be more likely to possess staying qualities than if mixed with petroleum fuel oil as is the case with "Cresol."

In order to more clearly show the composition of cresol, the following excerpts from Spielman (44), are given: Commercial cresol or cresylic acid contains the following isomers:

Isomer	Percent in Commercial Cresol	Boiling Point Deg. C.
Orthocresol	40	191
Paracresol	35	202
Metacresol	25	202

The structure of cresol, C7H80, is shown by the following arrangement:



The specific gravity of the cresol used for admixture with oil tar croosote for the purpose of this study was found to be 1.024 at 22 deg. C. Supply furnished by Van Waters and Rogers, Inc., industrial chemists, Seattle, Washington, and Portland, Oregon.

Dr. Hubert (24) used p-cresol in his studies on the preservation of mill work. Although 25 chemicals and 18 proprietary preservatives were tested, the list did not include either coal tar creosote or water gas tar creosote. In order to arrive at any comparable basis for these creosote oils, more work, such as this present study, was found to be needed. Hubert's findings on p-cresol are given here for the purpose of demonstrating the effectiveness of straight p-cresol as a preservative.

Toxicity in Wood (Kolle flask method):

Full treatment: Percent loss in weight - 40 Untreated: """""" 64.3 Volatility (After exposing to 160 deg. F. for 48 hours and 2 months in flask):

Full treatment:	Percent	1035	in	weight	26.8
Untreated:	¥	Ħ	群	ÿ	52.8

Leaching by water:

Full treatment	Percent	loss	in	weight	42.2
Untreated:	17	11	Ħ	ñ	47.5

On this basis, it would seem that the leaching effect of water on p-cresol was much more effective in reducing the toxicity than the high temperature.

# The Additions of Pentachlorophenol and Its Significance

The work of Dr. Hubert (24) and others have demonstrated the effectiveness of organic chemicals in mineral solvents. If it is true that organic substances in mixture (as shown by Zehl, 41) have a toxicity equal to the summation of each component, then pentachlorophenol added to a creosote should be equal to the summation of the toxicity of both the creosote and the pentachlorophenol. The work of Bateman does not wholly agree with that of Zehl, but he concludes that the toxicity is greater than when each is used alone. Also when one organic and one inorganic or a mixture of two inorganic substances are used, Zehl found that the effect of the toxicity was greater than the summation of the components of the mixture. It follows, therefore, that pentachlorophenol (C60150H) added to oil tar creosote would be very likely to have a toxicity greater than when each was used separately.

It has been shown by Bateman and Baechler (9) the killing point of pentachlorophenol is .002 against Fomes annosus. This is an especially low concentration when considering the solubility of this chemical. The solubility is reported (31) to be 0.0014 percent at 20 deg. C. The solubility factor plus the low volatility, reported as not measurable at 25 deg. C. (32), plus the high toxicity lends significance to the use of pentachlorophenol as a wood preservative under any and all conditions. It has also been found that penta-

chlorophenol is readily soluble in oil tar creosote. Schwarz (43) reports a maximum solubility of 40 percent and it has been found in the laboratory that  $2\frac{1}{2}$  to 5 percent solutions are not difficult to obtain at room temperatures.

Hubert (24) has probably done more recent investigative work on the use of chemicals in solution than any other worker. Of the large number of preservatives used in his investigations, pentachlorophenol was rated as the best preservative on the basis of toxicity (agar and wood) volatility, leaching and cost. Other preservatives in order of rating were: 2, tetrachlorophenol; 3, p-tert - Butylphenol; and 4, phenyl mercury cleate.

Using the results of Hubert's investigations it was concluded that pentachlorophenol would be the best of several possible chemicals to use in mixture with oil tar creosote for producing a preservative of high toxicity, and permanence at a reasonable cost.

#### Volatility Tests on Wood Blocks

Tests, identical with those described previously under wood block volatility tests, were made using the following combination of preservatives:

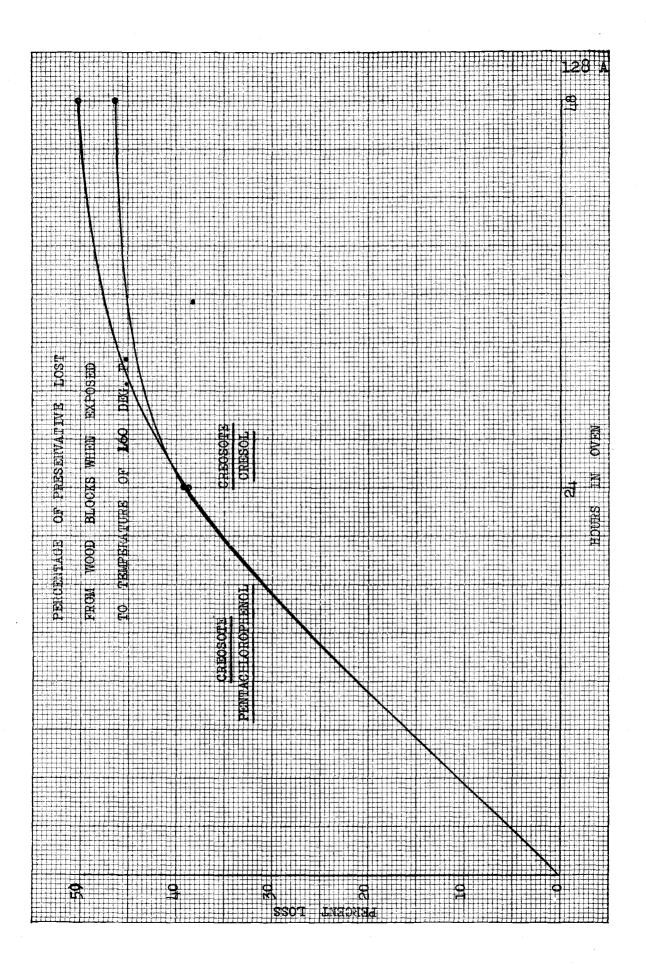
1. 011 tar creosote - 95%; Creso1 - 5%

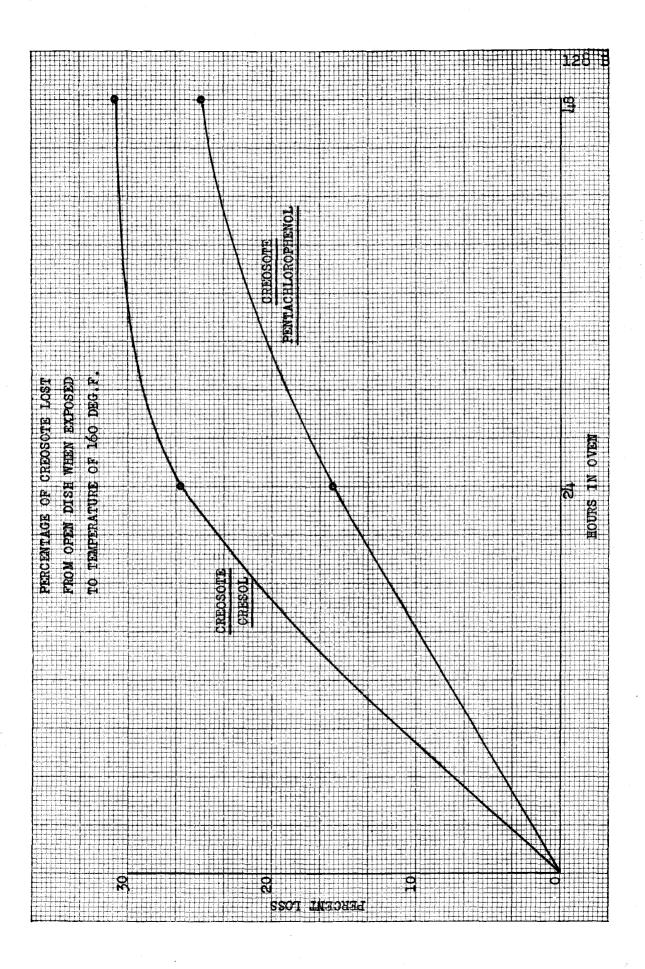
2. 011 tar creosote - 971%; pentachlorophenol - 21%

The following table shows the results of volatility tests on wood blocks preserved in the usual fashion and placed in an oven at 160 deg. F. for 48 hours. Weights were taken at the end of the forty-eight hour period. It was assumed that the moisture in the wood blocks was entirely evaporated after 24 hours at 160 deg. F. The calculated oven dry weights were therefore used as a basis for computing the percentage of weight lost.

Sample	Preser- vative taken up Wt. in Grams	Wt.of Pre- servative left after 24 hrs.	% Lost in 24 hrs.	Wt.of Pre- servative left after 48 hrs.	% Lost
0il Tar Creosote / 2.5% Penta- chloro- phenol	2.333 2.483 2.501 2.365	1.346 1.575 1.539 1.339	42.2 36.2 38.4 39.0	1.096 1.191 1.185 1.320	53.1 52.0 52.4 44.1
011 Tar Creosote / 5% Cresol	2.013 2.090 1.982 2.051	1.126 1.306 1.270 1.225	44.06 37.4 35.9 35.4	.906 1.232 1.185 1.034	54.5 40.8 40.0 50.1

The following graph shows the average percentage of weight lost during the volatilization period as computed from the table above.





Watch Glass Volatility Tests were made according to the procedure previously outlined. The results are shown in the following tables:

in Grans be-	of Fluid n Grams at d of 24 hrs	% Loss	Wt.of Fluid in grams at End of 48 hr	% Loss
0il Tar Greesote / 2.5% Penta- chlorophenol				
20.608	16.868	15.71	14.593	24.33
0il Tar Creoscte / 5% Cresc1				
20.39	15.05	26.14	13.935	30.72

The accompanying curves show quite readily the comparable volatilities of these two mixtures.

Leaching tests were made according to the procedure described in the main report to ascertain the comparative resistance to leaching of the two solutions: ereosote plus crosol and creosote plus pentachlorophenol. The results of these tests are as follows:

Preservative	Original Wt. of Preservative	Final Wt.of Preservative	% Lost	Aver. % Lost
011 Tar Crocsots <u>7 5% Cresol</u>	2.87 3.078	2.13 2.173	25.75 29.07	دبا. 27
011 Tar Creosote / 2.5% Penta- chlorophenol	3.091 3.094	2.155 2.417	30.2 21.81	26.5

The following bar chart shows the comparison of the percentage of weight lost due to leaching by water.

Table showing the loss in weight of wood blocks when treated with varying amounts of Pentachlorophenol in solution with Stoddard Solvent and exposed to incubation for 8 weeks with fungus.

## Table A. Lenzites trabea

Preservative Pentachloro- phenol	Sam- ple	Orig.Wt.of 12% Block	Wt.after Treating	Computed Oven Dry Weight	Wt.at* end of 8 weeks
5 %	A B	5.012 4.956	5.017 4.96	4.488 4.436	4.52 4.41
2.5%	A B	5.061 5.015	5.082 5.028	4.530 4.488	4.76
L %	A B	4.942 4.97	4.890 4.91	4.423	4.42
.2%	A B	5.300 4.973	5.253 4.92	4.744 4.451	
•5%	A B	5.18 5.08	5.16	4.636	4.63 4.48
.025%	A B	5.303 5.174	5.270 5.130	4.747 4.631	3.15 ** 2.66 ***

## Table B. Poria incressete

5 %	A B	5.011 5.323	4.487 4.766	4.52 *
2.5%	A B	5.239 4.785	4.682 4.285	4
L \$	A B	5.351 4.842	4.791 4.335	4.78
.2%	A B	4.972 5.301	4.454 4.747	4.42
•5%	A B C	5.111 5.202 5.079	4.575 4.658 4.547	4.56 4.60 4.37

\* Dried in oven \*\* 33.6% loss \*\*\* 42.5% loss Table showing the percent of weight of wood lost when wood was treated with 1% oil tar creosote plus 1% Pentachlorophenol and exposed to Poria incrassata for 8 weeks in Kolle flask. C is untreated control.

Sample	Oven Dry Wt. (Computed)	Adjusted Oven Dry Weight	Wt. at end of Test	% Loss
A	4.842	4.851	4.55	6.6
B C	5.30 4.139	5.319	4.47 2.51	15.9 39.3

Table showing the percent of weight of wood lost when wood was treated with 6% oil tar creosote plus 1/10 of 1% Pentachlorophenol and exposed to Lenzites trabea for 8 weeks in Kolle flasks.

Sample	Oven Dry Wt. (Computed)	Adjusted Oven Dry Weight	Wt. at end of Test	% Loss
A	4.351	4.460	4.400*	-
B	5.119	5.228	5.20 *	

\* Dried in oven

Table showing the effect of an incubation period of 8 weeks on the retention of preservative in the wood sample blocks after the preserved blocks were exposed to leaching by water. No attack on preserved blocks. C is untreated control.

Preser- vative	Sam- ple	Orig.Wt. at 12% Moisture Content		Leaching	Computed Oven Dry Wt.of wood alone	Dry Wt. at end of 8 wks. Period
100% 611 Tar Creo- sote L. trabea	A B C	5.425 5.25 4.783	8.445 8.31	7.60 7.822	4.869 4.686 4.261	
011 Tar Creosote 5% Cresol L. trabea		5.49 5.02 5.065	8.36 8.098	7.62 7.193	4.901 4.481 4.512	6.42 6.08 3.56
011 Tar Creosote 2.5% Pen- tachloro- phenol L. trabea		5.44 5.431 5.327	8.531 8.435	7.595 7.758	4.856 4.768 4.746	6.55 6.64
Coal Tar Creosote 100% L. trabea	A B C	5.22 4.73 5.751	8.47 7.81	7.63 6.935	4.659 4.222 4.901	6.56 5.76 5.01
Untreated	A B C	5.01	annan - an an Arly (1997) "Alatan (1997) "An an Arly (1997)" (1997)	5.01	4.472	3.65
<b>ala an in 1977 with the state of state</b>	U.	4.952	-		4.412	2.97

Table showing the percent of weight of untreated wood lost when exposed to 8 weeks incubation with fungus in Kolle flasks.

## A. Lenzites trabea

Preser- vative	Sample	Orig. Wt. at 12% Moisture Content	Computed Oven Dry Wt. of Wood Alone	Dry Wt. at End of 8 Wks. Period	% Loss
Control Flask 2	A wet B	5.000 5.42	4.454 4.829	3.81 3.29	14.2 32.7
Flask 1	A B C	4.848 5.25 5.258	4.319 4.677 4.684	3.48 3.18	25.4 32.1

### B. Poria incressata

Flask	3	A B C	5.52 4.923 5.98	4.918 4.386 4.437		
Flask	4	A B C	5.347 5.15 5.018	4.763 4.588 4.471	4.01 3.46 3.65	15.8 24.3 18.3

Table showing effect of incubation period of 8 weeks on the retention of preservative in the wood sample blocks. After the blocks were exposed to 160 deg. F. for 48 hours. No attack on treated blocks.

Preser- vative and Fungus	Sam- ple	Orig.Wt. at 12% m.c.	Wt.after Treating	160° for 48 hrs.	Computed Oven Dry Wt. on Wood Alone	Dry Wt. at end of incubation
011 Tar Crecsote 100% L.trabea	A B C	5.20 5.088 5.39	8.10 8.065	5.91 5.935	4.633 4.533 4.802	5.85 5.89 3.05
Oil Tar Creosote 5%Cresol L.trabea	A B C	5.169 4.887 5.34	7.220 6.900	5.639 5.26	4.605 4.354 4.757	5.76 5.36 3.03
011 Tar Crecsote 2.5% Pen- tachloro- phenol L.trabea	A B C	5.337 5.367 5.317	7.82 7.868	5.946 5.966	4.755 4.781 4.737	6.16 6.19 2.85
Coal Tar Creosote 100% L.trabea	A B C	5.282 5.793 4.805	8.63 8.49	6.282 6.29	4.706 5.161 4.281	6.26 6.32 2.66
011 Tar Creosote 100% P.in- crassata	A B C	5.013 5.370 5.703	7.350 8.050	5.661 6.06	4.466 4.784 5.081	5.64 6.04 3.46
Oil Tar Creo. 5% Cresol P. incrassata	A B C	4.923 5.295 5.505	6.905 7.385	5.49 5.949	4.305 4.717 4.904	5.56 6.023 4.76
Oil Tar Creo.2.5% Pentachlor ophenol P. Incrassata		5.00 5.012 5.406	8.191 7.998	6.155 5.87	4.454 4.465 4.816	5.99 5.72
Coal Tar Creo.100% P. incras- sata	A B C	5.00 5.333 5.627	7.333 7.708	5.55 6.071	4.454 4.751 5.013	5.56 6.05 3.23

Table showing effect of incubation period on the retention of preservative in the wood sample blocks. Blocks treated with oil tar creesote and varying amounts of Gresol and incubated with Lenzites trabes for 8 weeks. No attack on treated blocks.

Preserva- tive Cresol %	Sam- ple	Orig.Wt. at 12% Moisture Content	Wt. after impregna- tion and desiccator	Computed Oven Dry Wt.of Wood Alone	Actual Wt. at end of Incubation Period
5	A B	5.249 5.218	7.263 7.245	4.700	6.60
2.5	A B C	5.336 5.37 4.946	8.560 8.20	4.769 4.804 4.429	7.13 6.73
1	A B	4.834	7.308 8.093	4.296 4.849	<u>4.46 *</u> 5.84 7.34
•5	A B	5:171	8.000 7.745	4.572	6.33 7.00
.1	A B	5.21 4.798	8.18 7.395	4.665	6.63 6.35
.025	A B	4.841 6.022	7.422 9.038	4.334 5.391	6.21 7.85

Table same as above except that sample blocks were exposed to Poria incrassata. No attack on treated blocks.

5	A B	4.941	7.185	4.424	5.72
2.5	A B	4.97	7.625	4.615	6.29
1	A B	5.43	8.327 7.853	4.117 4.862 4.552	<u>6.86</u> 6.91
•5	A B	5.099 4.97	8.020 7.868	4.566	<u> </u>
.1	A B C	5.101 5.46 5.50	7.985 8.478	4.567 4.889 4.925	6.81 6.86
.025	A B	5.148 4.828	8.462 7.55	4.878	<u>     4.09    </u> 5.98

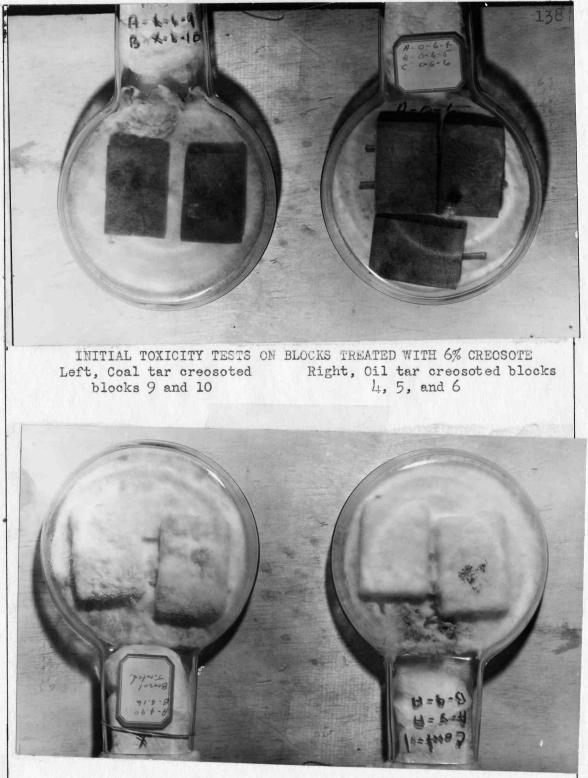
\* Untreated

Table showing effect of incubation on the retention of preservative in the wood sample blocks. Blocks treated with oil tar creosote and varying amounts of Pentachlorophenol. No attack on treated blocks when incubated 8 weeks with Lenzites trabea.

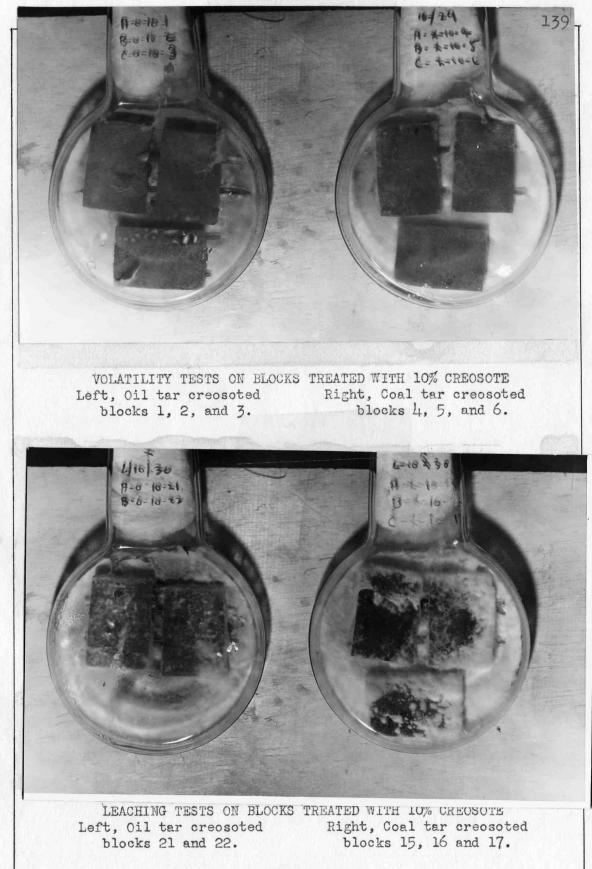
Preserva- tive % Per tachloro- phencl in <u>mixture</u>	n-ple	Orig.Wt.st 12% Mois- ture Content		Computed Oven Dry Wt.of Wood Alone	Actual Wt. at End of Incubation Period
5	A B	4.984 5.169	7.360 7.541	4.440 4.605	6.14 6.32
2.5	A B	4.920 7.000	4.995	4.383 4.450	6.07
1.0	A B	4.900 7.188	4.869 7.185	4.364 4.338	6.00 5.95
•5	A B	5.584 7.645	5.531 7.910	4.975 4.992	6.49 6.87
•1	A B	4.79 7.773	5.027 5.899	4.267 4.478	and and a state of the
.025	A B C	5.00 7.112	5.597 7.691	4.454 4.986 4.214	5.89 6.68 4.30

Table same as above except that sample blocks were exposed to Poria incrassata. No attack in treated blocks.

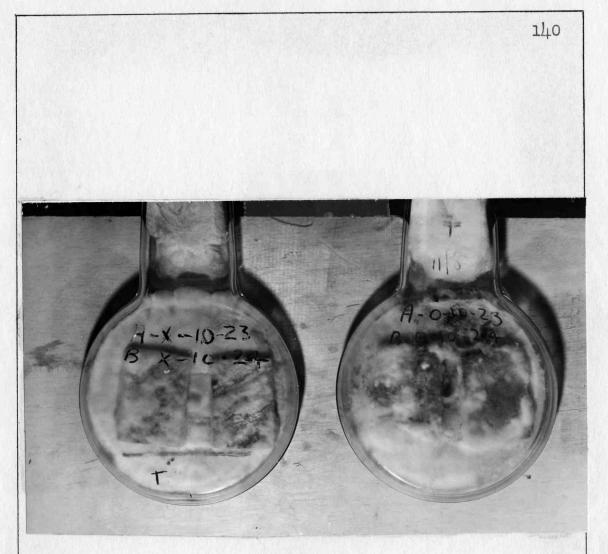
5	A B	5.140 4.827	7.841 7.235	4.602 4.332	6.47 6.01
2.5	A B	5.096 5.669	7.51 8.005	4.563 5.067	<b>498 9 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 </b>
1	A B	5.411 5.34	7.71 7.79	4.845 4.781	6.65
-5	A B	5.289 5.134	8.00	4.736	6.43 6.21
•1	A B	4.600 5.155	7.168 8.04	4.119 4.616	5.90
.025	A B	5.239	7.360 7.132	4.691 4.521	6.20 6.06



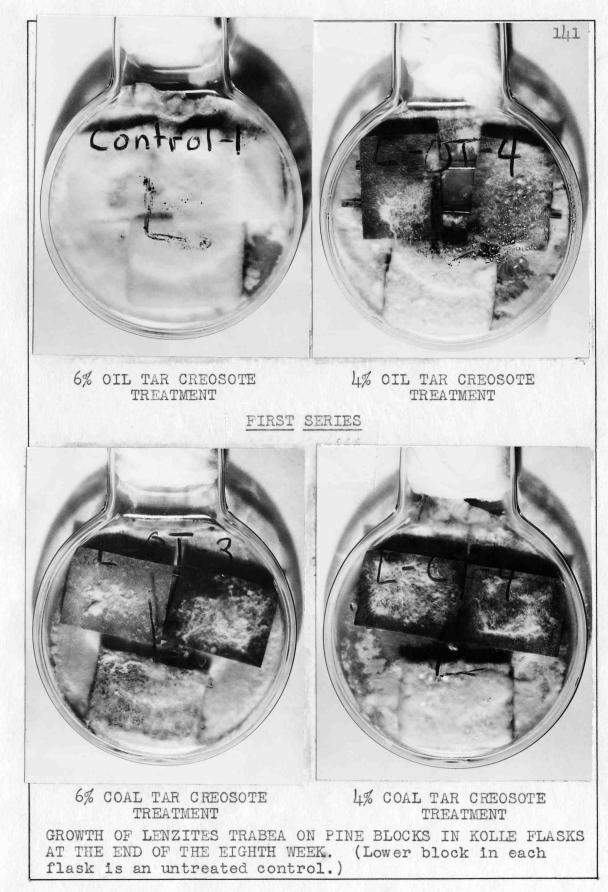
Untreated Control Blocks Benzol Treated Control Blocks

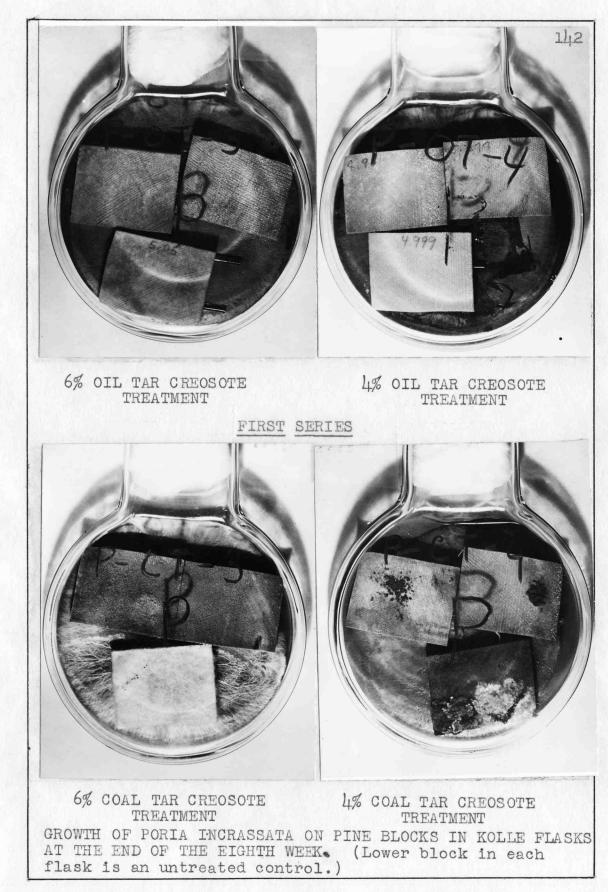


### PHOTOGRAPHIC EXHIBIT OF RESULTS



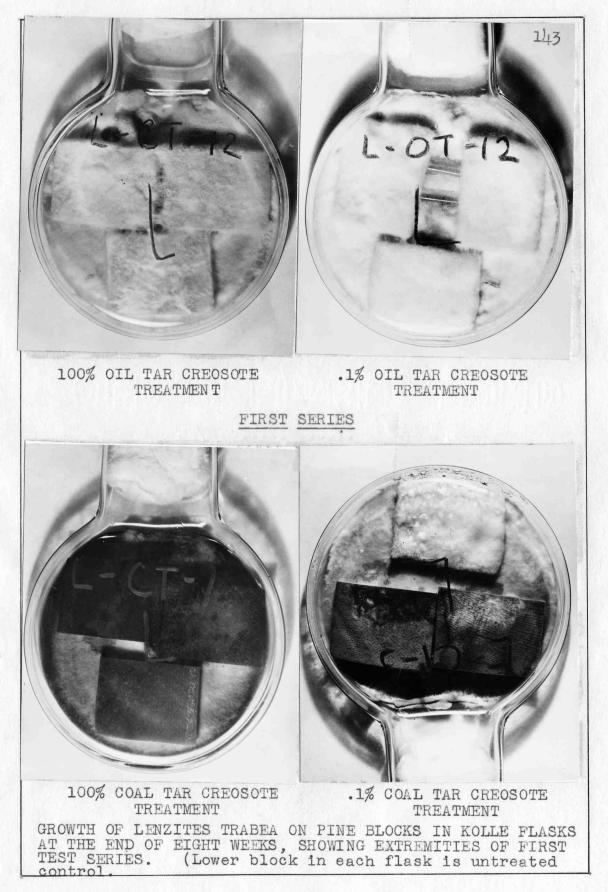
LEACHING TESTS ON BLOCKS TREATED WITH 10% CREOSOTE Left, Coal tar creosoted Right, Oil tar creosoted blocks 23 and 24. blocks 23 and 24.





### PHOTOGRAPHIC EXHIBITS OF RESULTS

FIRST SERIES



UNTREATED CONTROL BLOCKS ON CULTURES OF LENZITES TRABEA FOR A PERIOD OF 8 WEEKS.



UNTREATED CONTROL BLOCKS ON CULTURES OF PORIA INCRASSATA FOR A PERIOD OF 8 WEEKS.

