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WOOD PRESERVATIVE GREASE COMPOSITION

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ABSTRACT OF THE DISCLOSURE

A wood preservative composition thickened with a combination of micro-crystalline wax, water-soluble non-ionic surfactant and water (a gel-promoter) and combined with a mixture of an organic wood preservative liquid, and an alkaline inorganic alkali metal salt. This wood preservative composition is combinable with various dispersing agents, bentonite compounds and inert inorganic fillers. The individual components of the wood preservative composition are intermixed and heated, cooled and then milled to obtain a grease-like material that is applied to wood at areas of probable deterioration to preserve the wood throughout various climate conditions.

This invention relates to a method and composition of preservation of wood, particle board, hard board, and related structural fibrous materials, which method and composition are designed to increase the performance in wood preservation, simplify the application to the wood, and improve the overall results thus obtained.

There are many methods of applying preservatives to wood prior to the time the wood is put to use. The composition of the instant invention may have utility in this respect, although it is intended primarily for the use on wood already in place, such as poles, posts, sills, toe plates, stair risers, etc.; and the instant invention will be described primarily in connection with this use.

One of the chief uses for the present invention is in extending the usable life of poles already in use. Some of these poles may have been in place many years and the application of the instant composition thereto will add substantial years of use to the pole. These poles which were poorly pretreated, or pretreated with inferior preservatives may be again treated, while in place, with the instant composition along the deteriorating areas of the poles. Poles which are removed from one location to be used in another, usually at a different depth, can be advantageously treated with the composition of the instant invention.

The instant composition is a grease or a grease-like material, which may be applied by hand spreading, by a mechanical grease gun, or on a paper or plastic film or bandage-like wrapping. On poles it is applied at the ground line area, where rot is most likely to occur (however, it may be applied to the whole post or any area thereof). There are at present several wood preservative greases known and in use. Each of these greases, however, leaves much to be desired. The composition of the instant invention overcomes almost all of these difficulties or objections encountered in the use of the prior art compositions. For example, in British Patent No. 699,207

(published November 4, 1953) reference is made to a grease-like substance which can be applied to wooden objects along the ground line. The greases described by the British patent are oil base materials thickened with soaps such as aluminum or calcium stearate. Such greases are difficult to manufacture, the costs are inordinately high, and they have the disadvantage that the number of oils that can be used as the solvent for the active fungicide is limited. Also, such greases have a marked tendency to bleed at room temperature and even more predominating bleeding is frequently encountered in storage and in the field during summer. In addition, the preservative portions of such greases sometimes does not penetrate the wood well enough, and quite often certain desirable basic inorganic salts are incompatible with such greases.

Other preservative gels or greases now on the market have various disadvantages which have been overcome by the product of the instant invention. These disadvantages include poor resistance to water, inadequate penetration of wood, difficulty of application, high costs, doubtful preservative value, bleeding of the fluid, tendency to change in consistency, and incompatibility with certain inorganic salts.

In the instant invention, an organic wood preservative liquid is thickened with a micro-crystalline wax, a water-soluble non-ionic surfactant, and a gel promoter, sufficient to impart a grease-like consistency to the composition, and this grease-like composition is applied to wood. It has been found that the thickening compound consisting of a micro-crystalline wax and a water-soluble non-ionic surfactant and a gel promoter has unique properties in that it is capable of imparting the desired consistency to the grease-like composition, while permitting penetration of the wood preservative to a sufficient extent into the wood to which the grease is applied. This thickening compound produces a distinctly superior grease in that bleeding is substantially eliminated, a further wax coating is imparted to the post and it is compatible with the desired basic inorganic salts. Also the material is easily compounded and applied.

It is therefore an important object of the present invention to provide an improved composition and method for wood preservation.

It is a further object of the instant invention to provide an improved grease-like wood-preserving composition that does not tend to bleed.

It is another object of the instant invention to provide an improved grease-like composition that is easily applied to wood for the preservation thereof, comprising an organic wood preservative liquid and a thickening compound composed of a micro-crystalline wax, a water-soluble non-ionic surfactant and a gel promoter.

Yet another object of the instant invention is to provide an improved method of preserving wood that comprises milling or subjecting to shear a composition comprising an organic wood preservative liquid and a thickening compound composed of a micro-crystalline wax, a water-soluble non-ionic surfactant and a gel promoter (with one of many standard colloid mills or homogenizers) and applying the composition to wood.

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A further object of the instant invention is to provide an improved grease-like composition which is compatible with basic salts containing water of hydration (water of crystallization).

Other and further objects, features and advantages of the present invention will become apparent to those skilled in the art from the following detailed disclosure thereof.

The invention consists of a wood preservative composition comprising, in its more specific aspects, an organic wood preservative liquid, an alkaline inorganic alkali metal salt and a micro-crystalline wax, a water-soluble non-ionic surfactant and a gel promoter in an amount sufficient to impart a grease-like consistency to said composition.

The organic wood preservative liquid used in the practice of the instant invention acts as a diluent for the composition and additionally functions as a spreading agent for the final composition. This liquid may be a solution of wood preservative in an organic liquid carrier (such as a solution of pentachlorophenol in an aromatic petroleum oil) or it may be a liquid preservative (oil) per se as in the case of creosote. Whenever such liquid is used, however, it preferably contains an appreciable amount of pentachlorophenol. By "creosote" is meant materials so designated by chemists which are, in fact, organic mixtures derived from coal tar or wood tar and prepared commercially as wood preserving liquids, examples of which include coal tar creosote, coke oven tar creosote, water gas tar creosote, petroleum oil creosote, wood creoste, and mixtures thereof. Each such creosote may be used in the practice of the instant invention as a functional oil per se, and/or it may be used with or without additional solvents or carriers, such as petroleum oil; or they may be used with additional preservatives such as copper naphthenate or pentachlorophenol (or other poly tri to penta halo phenol compounds).

The amount of organic wood preservative liquid in the final composition of the invention depends to a substantial extent upon the viscosity of this liquid and also on the nature of the molecules comprising the liquid; and it may range from about 60% for thin liquids to as much as about 90–94% for thick liquids. (As used herein, the terms "percent," "parts," and "weight fraction" mean percent, parts, or weight fraction by weight, unless otherwise designated.)

As mentioned, the organic wood preservative liquid used in the practice of the invention may be a liquid preservative per se such as creosote. Also, it may be a preservative such as a polyvalent metal naphthenate such as zinc or copper naphthenate dissolved in a suitable (preferably non-polar) solvent, such as mineral oil. The preferred organic wood preservative liquid of this type is a solution of copper naphthenate in a mineral oil; and the resulting grease-like composition preferably contains at least 2% of the metal (Cu) and preferably 10% to 30% of the copper naphthenate is used.

The organic fungicide found to be most desirable for the use in the practice of the instant invention is pentachlorophenol. The amount of pentachlorophenol in the final grease-like composition should be at an approximate minimum of 2% and is preferably within the range of 5% to 30% of the weight of the composition. Thus, pentachlorophenol is utilized in the instant grease composition in amounts ranging from 2% to 50% (and up to 30%). The pentachlorophenol is incorporated in a wood preservative liquid first and a thickening agent is added subsequently. The solvent used for pentachlorophenol in the liquid is preferably a mineral oil; and the best results are obtained using an aromatic petroleum oil or sometimes called an aromatic gas oil or a heavy or light recycle oil. A preferred oil is a heavy recycled oil having a boiling point of approximately 600–700° F. Other oils which may also be used include cracking stock, slurry oils, heavy aromatic gas oils and medium aromatic gas

oils, examples being Amoco heavy thermal side cut No. 105, Amoco No. 102E oil, Tidewater No. 200 diesel oil. Tidewater CGB oil, Denver No. 3 oil, Magnolia lube oil extract No. 465E, aromatic HB oil, heavy aromatic naphtha, Shell medium aromatic oil, and Deep Rock heavy cycle oil. Other solvents for the organic fungicide here mentioned include naphthenic oils, petroleum tar gas oil, linseed oil, xylene, mineral spirits, and butyl Cellosolve. The last mentioned solvent will dissolve up to 60% pentachlorophenol and can be used to make a grease containing 50% pentachlorophenol for special purposes. An aromatic petroleum oil such as an aromatic HB oil is preferred for the use in the practice of the instant invention with pentachlorophenol, because of proper viscosity and good solvent power for pentachlorophenol.

The thickening agent employed in the practice of the instant invention for obtaining a grease-like consistency in the final composition is a compound of a water-soluble non-ionic surfactant, a micro-crystalline wax, and a gel promoter. The water-soluble non-ionic surfactant has the general formula:

$$X—Y—(RO—_n—H$$

wherein X is selected from a group consisting of C_6–C_{20} alkyl, and C_6–C_{12} alkyl benzene radical; Y is sulfur, or preferably oxygen; each R is a C_2–C_4 alkylene radical, n is an integer from 1–20. Such water-soluble non-ionic surfactant may be further described as being a polyalkylene glycol ether with a molecular weight being substantially in the range of 90 to 12,500. The water-soluble non-ionic surfactants, as defined hereinbefore, as used in amounts in the range of 2% to 7% of the weight of the composition. Such compounds are well known in the commercial world by various trade names such as Tergitol-XD (polyalkylene glycol ether), Tergitol NP–12 (nonylphenyl polyethylene glycol ether), Tergitol-NXP (nonylphenyl polyethylene glycol ether), Tergitol 12–P–15 (dodecylphenyl polyethylene glycol ether), Lissopal-NX (nonylphenyl ethylene oxide condensate), Sterox-NK (alkylphenol ethyl oxide adduct) Sterox-NL (alkylphenol ethyl oxide adduct), Sterox-SK (polyoxyethylene thioether), Triton X–45 (iso-octyl phenyl polyethoxyethanol OPE–5), Triton X–100 (iso-octyl phenyl polyethoxyethanol OPE 9–10), Triton N–101 (nonylphenyl polyethoxyethanol NPE 9–10), Triton N–128 (nonylphenyl polyethoxyethanol NPE 12–13), or similar compounds.

The micro-crystalline waxes used are obtainable through the dewaxing process of lubricating-oil fractions of petroleum from the residual lubricating fractions of the crude oil thereof. Micro-crystalline wax, Amorphous wax, Petrolatum wax and Petroleum Ceresine are the various names given to this series of hydrocarbon waxes of high melting point (140° to 200° F.) which are isolatable from certain types of crude petroleum oils. They possess the important characteristics of being an amorphous, or minute crystalline structure with excellent adhesive properties and are quite flexible in thin polished films over a wide range of temperatures, also they possess good sealing strength. Micro-crystalline waxes may be defined as having a melting point range from preferably about 170° F. upwards, being relatively hard and containing very little oil. Typical commercial examples of micro-crystalline waxes are: Petrolite C–1035, Monton Type 434, Petrolite C–200–7, Monton Type 431, Petrolite C–500, Recrystallized Micro-814, Monton Type 16, Besquare 190B, Besquare 190–A, Besquare 180–B and Besquare 180–A. Preferably, amounts in the range of 6% to 12%, by weight of the composition, of microcrystalline wax are utilized.

It will be understood that the exemplary microcrystalline waxes and water-soluble non-ionic surfactants are merely examples and that the invention is not limited to the use of such materials but that other like materials having the desired properties might be used.

Although the combination of a polyalkene glycol ether,

a micro-crystalline wax, and water (generally, amounts of water in the range of 0.5% to 1.75% of the weight of the composition are used) is particularly useful in the practice of the instant invention as a thickener for the final grease composition, it will be noted that some of the above described thickener combinations may be replaced in part by an inert inorganic filler such as finely divided silica and diatomaceous earth, or finely divided calcium silicate. In general the total amount of the said thickener combination and inorganic filler (if any is used) should be such as to effect the desirable thickening of the grease composition and this amount may range from about 5% to 40% of the final grease composition. The weight ratio of the composition of a polyalkene glycol ether, a micro-crystalline wax, and water to the inorganic filler may range from 1:10 to 10:1.

In addition, another ingredient which has been found to be of particular importance in improving the penetration and fungicide protection, particularly in the case of wet or moist wood, is an alkaline inorganic alkali metal salt. Such salts exclude substantially neutral salts such as sodium chloride, but include the alkaline salts of the alkali metals (i.e. sodium and potassium) such as sodium fluoride, sodium carbonate, borax (i.e., anhydrous to $Na_2B_4O_7$·$10H_2O$), sodium bicarbonate, potassium carbonate, and potassium fluoride. The group of compounds found to be most useful for this purpose are sodium and potassium fluorides, carbonates, bicarbonates and tetraborates. Sodium tetraborate is preferred. Such salts are preferably added with the inorganic filler (if any is used), after the addition of the other ingredients. Such salts may be employed in amounts ranging from 0.5% to 20% of the final grease-like composition, and preferably in amounts ranging from 1 to 20% thereof.

It has been found that there is a unique synergistic effect resulting from the combination of such inorganic salts, in combinaiton with a non-ionic water-soluble polyalkene glycol ether, a micro-crystalline wax, and water, and the organic wood preservative liquid, containing pentachlorophenol. This combination of ingredients achieves distinctly superior stability as well as penetration of the pentachlorophenol into the wood, particularly when the wood is moist, for example, having a moisture content of 10% to 50%. For example, it has been found that the penetration of the pentachlorophenol at an appreciable distance from the outer of the wood may be as much as 6% to 7% greater if the alkaline inorganic salt is incorporated in the grease-like composition.

In addition, a dispersing agent may be employed (to achieve still better stability and/or a penetrating effect) with the thickener combination of a polyalkene glycol ether, a micro-crystalline wax, and water. The amount of dispersing agent employed is 0.5% to 5% of the final grease composition, or 5% to 20% of the amount of the above described thickener combination used. The dispersing agent is an organic polar solvent such as methanol, acetone or the like (preferably a low molecular weight, alcohol, ketone, or ester containing not more than about 6 carbon atoms such as methanol, ethanol, isopropanol, propanol, butanol, acetone, diethyl ketone, methyl acetone, ethyl acetone, propylene carbonate, ethylene carbonate, etc.). In the practice of the instant invention the dispersing agent (if any is used) is added to the grease composition after the thickener combination, but before any inert filler or alkaline inorganic salts are added.

Further, although the combination of a polyalkene glycol ether, a micro-crystalline wax, and water is particularly useful in the practice of the instant invention as in formulating a thickener for the final grease composition, it will be noted that some of the thickener combination, above described, may be also replaced by certain of the bentonite compounds. Such bentonite compounds are composed of montmorillonite mineral in which at least part of the cation content of the mineral has been replaced by an organic nitrogen base. Clays that swell at least to some extent on being contacted with water and contain as a primary constituent a mineral of the group known as montmorillonites are generally referred to as bentonite. Such clays, which contain exchangeable alkali metal atoms either naturally or after treatment, constitute the raw minerals employed in making the bentonite-organic compounds which may be used in the practice of the instant invention. The bentonite-organic base compounds are preferably prepared as described in U.S. Patent No. 2,033,856 (issued Mar. 10, 1936 and incorporated herein by reference), by bringing together the bentonite and the organic-nitrogen base in the presence of aqueous mineral acid to effect base exchange; the bentonite-organic base compounds are so formulated as to be of sufficient alkalinity as to be titratable with mineral acids.

The organic-nitrogen bases include cyclic, aliphatic and heterocyclic amines, such as decyl amine, dodecyl amine, tetradecyl amine, hexadecyl amine, octadecyl amine, hexadecyl ammonium acetate, octadecyl ammonium acetate, dimethyl dioctyl ammonium acetate, dimethyl didodecyl ammonium acetate, dimethyl dodecyl hexadecyl ammonium acetate, dimethyl dioctyl ammonium acetate, dimethyl hexadecyl octadecyl ammonium acetate, dimethyl dioctadecyl ammonium acetate, and the corresponding chlorides and quaternary ammonium chlorides. The preferred organic-nitrogen bases from the quaternary ammonium group are those in which the N substituents are aliphatic groups containing at least one alkyl group with a total with at least ten to tewelve carbon atoms. When aliphatic amines are used they preferably contain at least ten to twelve carbon atoms.

In general, the total amount of bentonite compound (if any is used) and the combination of a polyalkene glycol ether, a micro-crystalline wax and water, should be such as to effect the desired thickening of the final grease composition and this amount may range from about 3.5% to about 5.5%. The weight ratio of the bentonite compound to the polyalkene glycol ether being substantially equal. Typical amine treated bentonites which may be used in the practice of the instant invention are: Baragel-24, Bentone-30, Baragel-27, Baragel, Bentone-34 and Bentone-70-C.

In the practice of the instant invention, the organic wood preserving liquid is first provided (in the form of creosote) or prepared by dissolving the organic fungicide in a suitable solvent therefor, such as mineral oil, such solution is slowly heated to 195°–210° F. Then a micro-crystalline wax is blended into the solution at the previously mentioned temperature, until a uniform solution is obtained. It is then cooled rapidly with continuous stirring until a temperature of 75°–85° F. is reached. (Such cooling may be effected by placing a water jacket on the heating kettle or any other arrangement whereby substantial controlled cooling of the solution may be achieved.) Addition of the polyalkene glycol ether (and bentonite, if desired) is then made with continuous stirring. The temperature of the micro-crystalline wax-organic wood preservative blend must be above the pour point of the polyalkene glycol ether for best results. Thereafter water is added immediately and stirring is continued for approximately five minutes. Then, any suitable inorganic filler material to be employed is added, also at this point, if desired, the relatively small quantity of methanol, acetone or other organic polar solvent used as a dispersing agent is added. The resulting material is a liquid slurry, which is then passed through a homogenizer or colloid mill wherein it is subjected to 1000 to 8000 pounds per square inch of shear, preferably at 2500 pounds per square inch of shear, and the slurry is thereby converted to a grease-like gel. Inorganic filler, is used, plus the alkaline inorganic alkali metal salt is preferably added next.

The foregoing procedure is carried out using the following formulations (which are prepared initially without in-

organic salt) and the results indicated in connection with each of these examples are the results obtained:

and for the purposes of the invention "greases" have approximately 400 or less units penetration.

TABLE I

Ingredients [1]	Examples 1	2	3	4	5	6	7	8	9
Micro-Crystalline Wax (Petrolite C1035(190-A)	0.120	0.120	0.090	0.120	0.100	0.110	0.110	0.0325	0.0250
Polyalkene Glycol Ether (Tergitol-XD)	0.070	0.040	0.050	0.030	0.030	0.030	0.020	0.050	0.045
Water	0.0175	0.0100	0.0125	0.0075	0.0075	0.0075	0.0050	0.0125	0.0075
Bentonite Compound [2]								0.040	0.0450
Preservative Solution [3]	(A) 0.7925	(B) 0.8300	5C) 0.8475	(D) 0.8425	(E) & (F) 0.8625	(G) 0.8625	(H) 0.8650	(I) 0.8650	(J) .8675
Methanol									0.010
Percent Oil Release	40	45	48.6	50	52.6	53.3	57.5	55.4	44-48
ASTM Penetration Unworked	256	255	304	260	305	277	293	276	310
ASTM Penetration Worked	318	318	344	324	356	343	354	335	320

[1] Ingredients are given in weight fractions of total composition.
[2] Bentonite compound used was Dicetyldimethyl ammonium bentonite but any like material may be used.
[3] Preservative Solutions consist of the organic-wood preservative liquid (such as creosol, or pentachlorophenol dissolved in a suitable mineral oil); such preservative solution may be any of the following:

		Wt. Fractions
Solution A:	Shell Medium Aromatic Oil	.8465
	Pentachlorophenol	.1535
Solution B:	Aromatic HB Oil	.843
	Pentachlorophenol	.157
Solution C:	Aromatic HB Oil	.859
	Pentachlorophenol	.141
Solution D:	Heavy Aromatic Naphtha	.8615
	Pentachlorophenol	.1385
Solution E:	Aromatic HB Oil	.750
	Pentachlorophenol	.115
	Silica [Hi-Sil 233]	.135
Solution F:	Coal Tar Creosote	.481
	Heavy Aromatic Naphtha	.438
	Pentachlorophenol	.081
Solution G:	Tidewater Diesel Oil	.908
	Pentachlorophenol	.092
Solution H:	Aromatic HB Oil	.7893
	Pentachlorophenol	.0984
	Calcium Silicate (Microcel-E)	.1123
Solution I:	Aromatic HB Oil	.8295
	Pentachlorophenol	.1705
Solution J:	Deep Rock Heavy Cycle Oil	.8695
	Pentachlorophenol	.1305

It may be seen, as shown in Table I above, that decreasing any component of the gellation system (micro-crystalline wax, polyalkene glycol ether, or water) increases the oil release and tendency toward bleeding. It was also found that increasing the polyalkene glycol ether (Tergitol-XD) content suppressed bleeding more than did corresponding increases of the micro-crystalline wax or of the water content.

The National Lubricating Grease Institute developed and adopted a consistency classification in 1941. This classification, which is based on penetration obtained by ASTM Method D-217, is shown in Table 21.1. When the Federal Government put into effect a tax on lubricating oils (see Regulation 44, Section 314.40, Use of Terms) the above method of test was recognized, as can be seen from the following quotation:

"The term 'lubricating oils' does not include products of the type commonly known as grease. Oleaginous substances which are classed as grease and which contain oil are not subject to the tax when of a worked consistency of less than 390 penetration units, or an unworked consistency of less than 360 penetration units, by the method of test of the American Society for Testing Materials D-217-33-%."

TABLE 21.1.—NLGI LUBRICATING GREASE CONSISTENCY CLASSIFICATION

Consistency Number:	ASTM worked penetration at 77° F.
0	355 to 385
1	310 to 340
2	265 to 295
3	220 to 250
4	175 to 205
5	130 to 160
6	85 to 115

ASTM Method D-217-52T does not provide for tests of lubricating greases softer than those having a penetration of about 400 units, but some of the greases of the invention have a penetration of 400 units or slightly over,

Limited data on other micro-crystalline waxes were obtained as follows:

TABLE II

Ingredients	Tests 1	2	3	4	5
Petrolite C-1035 [1]:					
190-W	0.100				
190-B		0.100			
190-A			0.100		
180-A				0.100	
180-B					0.100
Tergitol-XD	0.0650	0.0650	0.0650	0.0650	0.0650
Preservative Solution (B)[2]	0.8265	0.8265	0.8265	0.8265	0.8265
Water	0.0085	0.0085	0.0085	0.0085	0.0085
ASTM Penetration Unworked	260	290	270	335	329
ASTM Penetration Worked	323	344	354	7,400	7,400

[1] Ingredients are given as weight fractions of total compositions.
[2] Refer to Table I for composition of Solution B.

As a specified example, using the previously quoted formulation of Example I, pentachlorophenol (10 parts) is dissolved in Shell Medium Aromatic Oil (79.3 parts). Then, the above solution is heated to 195°-210° F. and the micro-crystalline wax [Petrolite C-1035 (190-A)] is blended into the said solution at this temperature. When the solution of pentachlorophenol in Shell Medium Aromatic Oil with Petrolite C-1035 (190-A) has attained uniformity it is cooled rapidly to 75°-95° F. (using a water jacket on the kettle, or any other such suitable device whereby the slurry is cooled to the required temperature). Next, the polyalkene glycol ether (Tergitol-XD) is stirred into the slurry of pentachlorophenol in Shell Medium Aromatic Oil with Petrolite C-1035 (190-A). The temperature of this "wax-oil" blend must be above the pour point of the Tergitol-XD for the best results. Water is then added immediately to the slurry of pentachlorophenol in Shell Medium Aromatic Oil with Petrolite C-1035 (190-A) and Tergitol-XD and stirred thoroughly for approximately five minutes, at which stage this material is a relatively free flowing slurry. The material is then passed through a homogenizer (wherein

it is subject to about 2500 pounds per square inch of shear) and the resulting material obtained from the homogenizer is a grease-like gel having the properties described in connection with Example 1. The same procedure is employed in preparing each of the other formulations set forth herein, to obtain the results specified.

The grease of Example 1 is applied to poles in use (along the ground line) and found to effectively preserve the wood. In areas of rainfall of about 15 inches or more per year, this grease-like material is applied to an area on the pole extending from about 2 inches above the ground line to about 14 to 20 inches below the ground line. In arid regions, the grease-like material is applied from approximately the ground line to as much as 3 or more feet below the ground line. The instant grease-like material is also applied to the exposed top of pole and found to be an effective preservative in this respect also. Each of the aforementioned examples of grease-like material are used in substantially the manner just described to obtain effective wood preservation and prolonging the life of poles in use.

Particularly in the case of moist wood having a moisture content of 10% to 50%, it has been found that the penetration of the compositions of Example 1 through 9, as seen in Table I, is greatly improved by the addition thereto of an alkaline inorganic alkali metal salt in relatively small amounts. In each case, the inorganic salt is added, by thoroughly mixing, after the other ingredients have been processed through the homogenizer to obtain a grease-like gel. A typical formulation for this purpose is set forth in the following example:

Example 10

Preservation solution C (refer to Table–I)	0.7925
Micro-crystalline wax [Petrolite C–1035 (190–A)]	0.100
Polyalkene glycol ether (Tergitol–XD)	0.060
Water	0.0075
Borax (anhydrous to $Na_2B_4O_7.10H_2O$)	0.050

ASTM penetration (worked)—400+; all other properties good.

Pole stubs of southern yellow pine placed in the soil are found to have an equilibrium moisture content at the ground line (under test conditions) of 35% to 45% by weight of the dry wood; it is found that these pole stubs are effectively preserved by the application of the grease-like material in Example 10. In such cases where the moisture content is high, a preferred formulation is that of Example 10 which specifies the use of a basic, water-soluble, preservative salt such as Borax (anhydrous to $Na_2B_4O_7.10H_2O$) in the grease-like composition. Distinctly superior penetration of the wood is obtained, as evidenced by a uniform higher concentration of pentachlorophenol within the body of the wood, if an alkaline inorganic alkali metal salt such as sodium tetraborate (anhydrous to $Na_2B_4O_7.10H_2O$), is used.

Corresponding superior results in penetration of moist wood are obtained using in the procedure of Example 10 any of the following compositions:

Composition A.—Sodium fluoride in an amount equal to 5% of the weight of the grease-like material of Example 1 is admixed therewith and found to impart superior wood penetration thereto.

Composition B.—To the grease-like composition of Example 2, there is added 5% by weight of sodium carbonate, and such addition is found to distinctly enhance wood penetration in moist wood.

Composition C.—To the grease-like composition of Example 3, there is added 5% by weight of borax and it is found that superior wood penetration is achieved thereby.

Composition D.—Sodium tetraborate (anhydrous to $Na_2B_4O_7.10H_2O$) in amounts equivalent to 0.5%, 1%, 2%, 4%, 5% and 10% by weight is added in separate portions to the grease-like compositions of Examples 4 and 5 and each resulting composition exhibits superior wood penetration with test pieces of southern yellow pine having a moisture content of 35% to 40%. The superior penetration is more noticeable in the case of the compositions containing 5% and 10% sodium tetraborate.

Composition E.—Sodium bicarbonate in amounts equal to 5% by weight of the grease-like composition of Examples 6 and 7 is added thereto, and the moist wood penetration of such compositions is noticeably improved.

Composition F.—Potassium fluoride is added in amounts equal to 5%, by weight, to the grease-like composition of Examples 8 and 9 and it is found that the addition of potassium fluoride improves the moist wood penetration of the compositions.

Example 11

As previously mentioned, the superior pentachlorophenol wood penetration obtained by the use of the alkaline inorganic metal salt in a grease-like composition amounts to an improvement of as much as 600% to 700%. This may be demonstrated numerically. For example, pieces of untreated southern yellow pine posts were set in the ground in the manner in which telephone poles would be placed. After several weeks during which rainfall was heavy and the wood picked up substantial amounts of moisture, these pieces were removed and treated by the application of compositions X and Y (below). The posts were then replaced in the ground. Two months after the application of the preservative grease, the posts were again removed, and boring taken and analysis made of the pounds of pentachlorophenol per cubic foot of wood, both at the "outside" which is the outer ½ inch thickness of the posts and the "inside" which is the region 1 to 2 inches from the outside surface of the posts. The results are shown in Table III below:

Composition X: Weight Fraction

Preservative Solution C (refer to Table–I)	0.8475
Micro-Crystalline Wax [Petrolite C–1035 (190–A)]	0.0900
Polyalkene glycol ether (Tergitol–XD)	0.0500
Water	0.0125

Composition Y:

Sodium Tetraborate (anhydrous to $Na_2B_4O_7.10H_2O$) in an amount equivalent to 5% of composition X is added thereto.

TABLE III

Post No.	Moisture Content, percent	Composition	Penetration Pentachlorophenol Content	
			Outside	Inside
1	28.5	X	0.755	0.060
2	31.5	X	0.684	0.080
3	36.0	X	0.785	0.113
4	31.5	X	0.852	0.176
5	31.0	X	0.585	0.060
6	45.0	Y	0.713	0.234
7	42.0	Y	0.792	0.343
8	41.0	Y	0.819	0.393
9	41.0	Y	0.852	0.343
10	42.0	Y	0.819	0.306

Comparable results are obtained using, in place of the sodium tetraborate in Composition Y, sodium fluoride, sodium carbonate, sodium bicarbonate, potassium carbonate, and/or potassium fluoride. Comparable results are also obtained using the following composition.

Composition Z: Weight Fractions

Coal Tar Creosote	0.167
Sodium Tetraborate (anhydrous to $Na_2B_4O_7.10H_2O$)	0.060
Aromatic HB Oil	0.529
Pentachlorophenol	0.087
Micro-Crystalline Wax [Petrolite C–1035 (190–A)]	0.087
Polyalkene glycol ether (Tergitol–XD)	0.057
Water	0.013

As indicated herein, the mineral oil solvent content of the composition is preferably relatively substantial, i.e., about 50% to 95%; although it is apparent that when a liquid preservative such as creosote is employed, a substantial portion of the typical aromatic petroleum oil solvent may be replaced by the "liquid" creosote, e.g., in Solution F hereinbefore described, wherein the oil content comprises 40% heavy aromatic naphtha and 44.3% coal tar creosote.

It will be understood that modifications and variations may be effected without departing from the spirit and scope of the novel concepts of the present invention.

I claim as my invention:

1. A wood preservative compound comprising at least 2% to 20% of pentachlorophenol, 50% to 95% of a mineral oil solvent for the pentachlorophenol, a combination of (a) an inert organic filler, (b) 0.5% to 20% of an alkaline inorganic alkali metal salt selected from the class consisting of borates, bicarbonates, carbonates and fluorides and (c) a composition of 6% to 12% of a microcrystalline wax, 2% to 7% of a non-ionic water-soluble polyalkene glycol ether and 0.5% to 1.75% of water, the weight ratio of (a) plus (b): (c) ranging from 1:10 to 10:1.

2. A wood preservative composition having the following formulation:

	Weight Fraction
Coal Tar Creosote	0.167
Sodium Tetraborate	0.060
Aromatic HB Oil	0.529
Pentachlorophenol	0.087
Micro-Crystalline Wax	0.087
Polyalkene glycol ether	0.057
Water	0.013

References Cited

UNITED STATES PATENTS

2,662,056	12/1953	McCarthy	252—49.5
2,775,561	12/1956	Frohmader	106—271
3,049,471	8/1962	Behr	106—15
3,063,852	11/1962	Davdin et al.	106—15
3,080,330	3/1963	Rudel et al.	260—28.5

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