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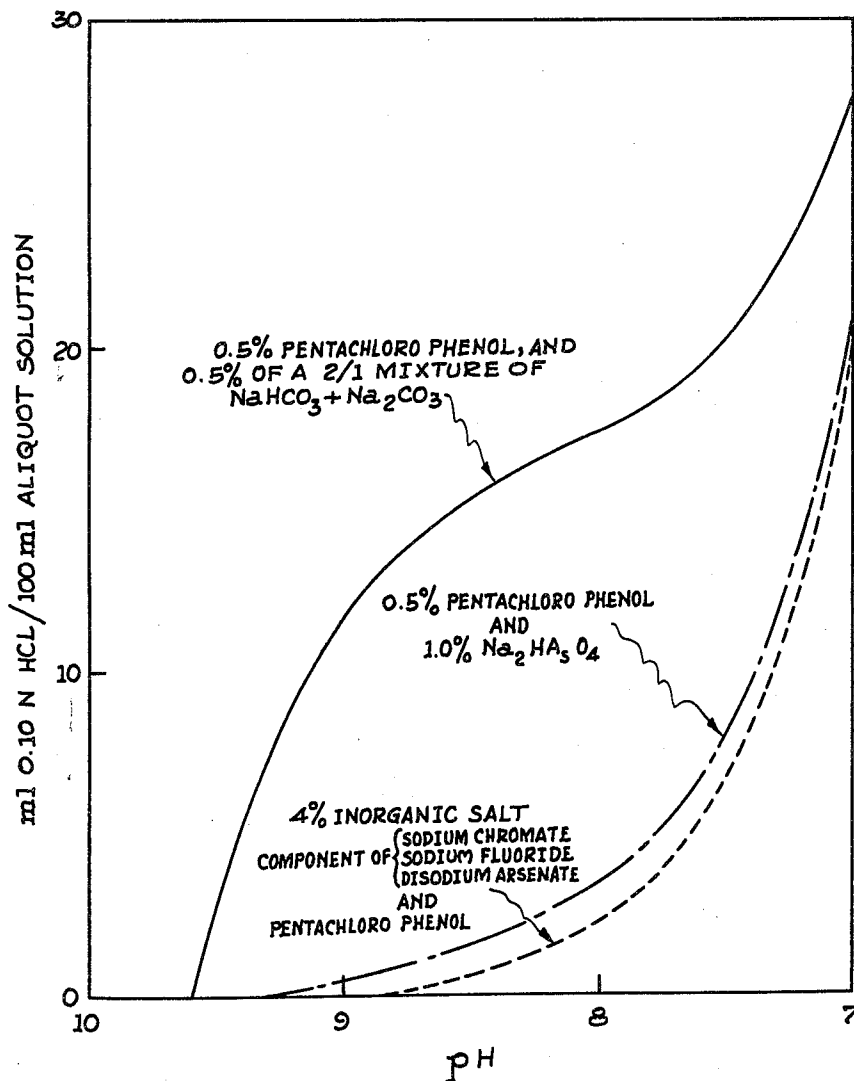
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WOOD PRESERVATIVE

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COMPARATIVE BUFFERING CAPACITY
OF VARIOUS CHEMICALS ON SOLUTIONS
CONTAINING SODIUM PENTACHLOROPHENATE



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WOOD PRESERVATIVE

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This invention relates in general to compositions and processes adapted for use in preservation of wood, as from fungi and termites, and more particularly to a composition for, process of, and product for, the combined preservation of wood with a non-staining, non-sludging composition.

Wood treated with a typical preservative composition composed of inorganic components typified by sodium fluoride, sodium chromate or potassium dichromate, sodium arsenate, and an organic component 2,4-dinitrophenol, possesses high resistance to attack by fungi and termites and is clean, odorless, and paintable. However, the dinitrophenol component is leached from the wood by rain or condensed moisture. In areas, such as Florida, where slab construction is common, and such preserved wood is suitable, this leaching causes the staining of the slab and is objectionable. When the rain falls on the wood, the coloring leached from the wood penetrates other objects, such as porous terrazzo floors, etc., and the resulting stain cannot be removed, even by grinding. Other porous materials, such as plaster, are often stained as well.

To overcome this staining and bleeding problem, it has been proposed to substitute chlorinated and nitrated aryl arsonic acids for the dinitrophenol, but this proposal has not been entirely satisfactory because the substituted aryl arsonic acids are more expensive than dinitrophenol, and contrary to the proposals, the aryl arsonic acids are less toxic to wood-destroying fungi than is dinitrophenol.

We have found unexpectedly that sodium pentachlorophenate can be used with the foregoing salts, such as alkali metal fluoride, chromates, and arsenates, to provide an excellent non-staining, non-sludging preservative for wood. Although sodium pentachlorophenate is a colorless, water-soluble salt with high toxicity to fungi, it had not been heretofore used for the impregnation of wood because the acidic nature of the wood causes precipitation of pentachlorophenol when the pH of the sodium pentachlorophenate solution falls slightly below 7 for technical grade material. After it is introduced into the wood, the pentachlorophenate is changed back to pentachlorophenol by the acidity of the wood and possibly through reaction with carbon dioxide. Because of the precipitation due to the acidity of the wood, the pentachlorophenate generally does not penetrate the wood deeply. While the material does afford some protection from decay, the protection is less satisfactory than that protection which can be obtained with oil solutions of chlorinated phenol. The use of aqueous solutions of sodium pentachlorophenate therefore has been heretofore limited to the dipping of green lumber in a solution of the pentachlorophenate to prevent blue staining during seasoning.

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To prevent this precipitation, it has been proposed to incorporate a mixture of sodium carbonate and sodium bicarbonate in the composition to act as a buffer. Such formulation forms a solution that has an initial pH of about 9.6 and has a buffer capacity that is high between this value and a pH of about 8.5. When the buffer solution is applied to wood, the acidic nature, about pH 3–4 of the wood, lowers the pH of this solution sufficiently to precipitate pentachlorophenol in the solution and on the surface of the wood; so these solutions do not penetrate the wood sufficiently for commercial purposes unless the original pH of the solution is very high, almost 10. To use such a high pH formulation to prevent this sludging action, is not feasible, since such high alkalinity has a tendency to damage the wood. Wood is readily attacked by alkalis; the hemicellulose is extracted, resins are saponified, and the lignin becomes soluble. Today's practice to prevent delignification calls for operation at a pH below 8.5 to avoid attack of the wood by alkali. The approximate value for precipitation of free pentachlorophenol is pH 7, which is below the 7.2 to 7.8 American Wood Preserving Association (AWPA) range for salts of the aforesaid typical composition. Hence, it has been thought not feasible to use the sodium pentachlorophenate for preservation of wood with alkali as a buffer, to prevent the sludging that otherwise would take place as a result of the acid in the wood, because the amount of alkali for buffering to avoid sludging and permit deep penetration, would result in too great a destructive attack on wood.

In accordance with the present invention, we have found that formulations can be successively attained for avoiding staining of wood or sludging of this component while retaining the advantages as to economy, ready availability of materials, easy solubility and toxicity of formulations employing dinitrophenol and still be within the 7.2 to 7.8 pH range required to prevent precipitating pentachlorophenol in the wood as aforesaid, by employing an organic component typified by the salt sodium pentachlorophenate, instead of the organic component dinitrophenol in the aforesaid typified composition, and in the same ratio as customarily employed with the other inorganic components aforesaid, that is, in the ratio of sodium pentachlorophenate 1:disodium arsenate 2:sodium chromate 3:sodium fluoride 2. Solutions of from 1% to 10% of the salts are possible and totally effective for the purpose.

Unexpectedly, we have now found that the presence of the inorganic components of the typified composition, prevents the precipitation of the organic component typified by sodium pentachlorophenate during the pressure-impregnation treatment of the wood. The buffering effect of said inorganic components on the acid from the wood during the pressure treatment of the wood takes place well above the critical 7.0 pH for precipitation of pentachlorophenol. When wood is treated with a salt solution of the typified composition containing sodium pentachlorophenate in place of the customary dinitrophenol, the pH changes from 9.4 to 7.4 with no sludge formation and complete penetration. This solution, when used

again to treat fresh wood, showed a pH change only from 7.4 to 7.2. Again, the solution was still clear, and complete penetration of the wood by pentachlorophenol had taken place. From this, it is apparent that the mixture of the inorganic components sodium chromate, disodium hydrogen arsenate, and sodium fluoride, buffers the solution against acid groups in the wood and allows penetration of the wood with sodium pentachlorophenate without precipitation of pentachlorophenol outside the wood.

According to the invention therefore there is provided a composition for use in the preservation of timber, with a non-staining and non-sludging action, comprising components by weight typified by the following: 12.5% sodium pentachlorophenate, 25% disodium arsenate, 37.5% sodium chromate, and 25% sodium fluoride.

In pursuance of the invention, it was further found that the relatively low solubility of sodium fluoride is a limiting factor which prevents preparation of solutions of high concentration while maintaining the formulated gram ratio of salts 1:2:3:2. It was also found that the presence of NaF and Na_2CrO_4 in the mixture had little effect on the buffering capacity.

Accordingly, the invention further comprehends a formulation of the component typified by sodium pentachlorophenate with the component typified by Na_2HAsO_4 , disodium arsenate, in the gram ratios 1:2 with a maximum salt concentration of 30%, and for a 1:2:3 ratio of the sodium pentachlorophenate component and the disodium arsenate and the sodium chromate components, a maximum concentration of 20%.

According to a still further feature of the invention, there is provided a composition as aforesaid comprising components by gram weight typified by the following: 10 g. sodium pentachlorophenate and 20 g. disodium arsenate without either or both the sodium chromate and sodium fluoride.

In further pursuance of the invention, it was found that since technical pentachlorophenol also contains less highly chlorinated phenols, such as trichlorophenols, than other alkali-metal salts of a polychlorophenol would be equally useful for most aspects of the composition. It was found that 2,4,6-trichlorophenol; 4,6-dichlororesorcinol; and 2,4,6-trichlororesorcinol, were highly effective and highly toxic to wood-destroying fungi, non-staining and compatible with the inorganic components disodium arsenate, sodium chromate and sodium fluoride, without precipitating free polychlorophenol outside the wood over the desired pH range. The buffering action of the component inorganic salts was even more marked with the 2,4,6-trichlorophenol, since it required 27 ml. of 0.1 N HCl to reduce the pH from 9.8 to 6.5 and cause precipitation of the trichlorophenol in the presence of the component inorganic salts, compared to 3.5 ml. required to reduce the pH from 10.9 to 6.8 and cause precipitation in the absence of the component inorganic salts. The additional hydroxyl group in the resorcinol increases the water solubility of the compounds 4,6-dichlororesorcinol and 2,4,6-trichlororesorcinol sufficiently that no precipitation problems exist at the concentrations of interest in this novel application to wood for preserving the same.

According to a further feature of the invention, the component typified by sodium pentachlorophenate in the aforesaid typified composition therefore may be, and is, an alkali-metal salt of polychlorophenol, and more particularly, at least one of the group consisting of pentachlorophenol; 2,4,6-trichlorophenol; 4,6-dichlororesorcinol; and 2,4,6-trichlororesorcinol. The alkali-metal for all components may be sodium or potassium. Ammonia alkalinity seems to be weak.

The maximum salt concentration for the four component mixtures while maintaining the formulated ratio, is about 10%. For a mixture of only the two components,

sodium pentachlorophenate and disodium arsenate in a ratio of 1:2, the maximum salt concentration is 30%, and for a 1:2:3 ratio of sodium pentachlorophenate, disodium arsenate and sodium chromate, the maximum concentration is 20%.

When employing a solution containing 10% (10 g.) of sodium pentachlorophenate and 20% (20 g.) of disodium arsenate for treating wood to a retention of 30 lbs. per cu. ft., the retention of free penta, if complete conversion from the sodium salt to the free phenol took place, would be 2.8 lbs. per cu. ft., or about ten times the retention of penta usually aimed at with oil solvents. Even a solution of 1.25% (10 g.) sodium pentachlorophenate and 2.5% (20 g.) Na_2HAsO_4 , 3.75% (30 g.) Na_2CrO_4 , and 2.5% (20 g.) NaF, which has the proper ratios of all materials, gives a free pentachlorophenol retention of 0.35 lb. per cu. ft., which is slightly higher than the 0.3 lb. per cu. ft. considered necessary with treatments using oil type solvent.

The sodium pentachlorophenate is water-soluble and compatible with the three inorganic components at pH values above 7. The resorcinol salts are water-soluble and compatible over the entire pH range, but are more expensive than the first mentioned salt. Because of the adequate reserve buffer capacity of these component salt solutions between pH 8.5 and 7, it is desirable to avoid treatment with solutions of pH greater than 8.5. This will prevent damage to the wood by more alkaline solutions and ensure the precipitation of insoluble pentachlorophenol on prolonged standing. The use of a mixture of sodium dichromate and sodium chromate in preparing the dry mix, provides the desired pH when the dry salts are dissolved in water. The safe lower pH limit for these solutions to prevent precipitation is probably 7.2. If the pH is lowered, due to the use of the same solution in successive treatments of wood, and the addition of make-up salts does not raise it sufficiently, adjustment of pH may be made by the addition of soda ash. If the pH is too high, i.e., above pH 8.5, neutralization with acid is recommended to avoid alkaline damage of the wood.

The new compositions may be used in the preservation of timber, sawn wood, and other wood products by the conventional empty and full cell methods of impregnation. For example, the wood is placed in a standard treating cylinder and a vacuum pulled until a manometer shows at least 22", for about one-half hour. This is then followed by submerging the wood with the solution of a mixture of the components aforesaid under pressure at up to 200 p.s.i.g. at up to 200° F. to penetrate the wood with the solution for several hours until the wood becomes impregnated by a solution of the components alkali-metal salt of a polychlorophenol and one of the group consisting of mono-, di- and tri-alkali-metal arsenates, with or without, either or both, one of the group consisting of alkali-metal chromate and alkali-metal dichromate, and an alkali-metal fluoride. At the conclusion of the process, the cylinder and wood may be subjected to a vacuum to remove excess impregnant material from the wood.

The invention also comprises the aforesaid process, and the product of the process, that is, a wood product, in the form of timber or sawn wood, having an impregnant therein consisting of the hereinafter claimed components.

The following data may be illustrative in understanding and ascertaining the invention, percentages being by weight, unless otherwise specified.

A desirable non-staining, non-sludging salt formulation for wood preservation consists of the inorganic salts 37.5% sodium chromate, 25% disodium arsenate, 25% sodium fluoride, and the organic component 12.5% sodium pentachlorophenate.

Table I shows the results of tests on the effectiveness of sodium pentachlorophenate 4,6-dichlororesorcinol and 2,4,6-trichlororesorcinol in inhibiting the growth of six species of wood-destroying fungi at a concentration of 0.01% in agar.

TABLE I
Toxicity Against Wood-Destroying Fungi on Agar Plates

Compound	Concentration, percent	Four Weeks' Growth 1 of—					
		<i>Poria monticola</i> Madison 698	<i>Polyporus versicolor</i> Madison 697	<i>Lenzites trabea</i> Madison 617	<i>Poria incrassata</i> Madison 563	<i>Lentinus lepideus</i> Madison 534	<i>Coniophora puteana</i> Madison 515
Sodium Pentachlorophenate.	0.01	0	0	0	0	0	0
	0.001	0	9	0	0	0	0
	0.005	0	9	0	0	0	0
4,6-Dichlororesorcinol-----	0.1	0	0	0	0	0	0
	0.01	0	9	0	0	0	9
	0.001	9	9	9	9	9	0
2,4,6-Trichlororesorcinol----	0.1	0	0	0	0	0	0
	0.01	0	9	0	0	0	0
	0.001	9	9	9	9	9	9

1 Numbers indicate diameter of growth in centimeters.

In the treatment of wood with sodium pentachlorophenate alone, a 0.1% sodium pentachlorophenate solution which possessed an initial pH of 9.8, was used to treat by the full cell process, end coated southern yellow pine board 1½ x 4 x 12 inches. After treatment, the pH of the solution was 7.4 and it was still clear. Penetration of pentachlorophenol to the center of the board was shown by the color test of Sakornbut and Morrill (Anal. Chem. 27, 1259-61 (1935)). However, when the pH of the sodium pentachlorophenate solution was adjusted to 7.9 before treatment, a sludge formed in the solution during treatment, and free pentachlorophenol precipitated on the surface of the wood. The post treatment pH of the solution was 6.1. A second treatment caused the solution pH to fall to 5.2. Precipitation of technical pentachlorophenol takes place at pH 6.7-6.9. Unless the original pH of the solution is very high (almost 10) the acidic nature of the wood lowers the pH of a sodium pentachlorophenate solution sufficiently to precipitate pentachlorophenol in the solution and on the surface of the wood.

We have now found that when sodium pentachlorophenate in the presence of the inorganic salt components sodium chromate, disodium hydrogen arsenate and sodium fluoride is used to treat wood, the precipitation that has been experienced heretofore, does not occur. When wood, for example, is treated with a 1% salt solution containing 0.1% sodium pentachlorophenate in place of the customary 0.125% dinitrophenol, the pH change is from 9.4 to 7.4 with no sludge formation and with complete penetration of the wood. We have also found that when the solution is used again to treat fresh wood, the pH changes only from 7.4 to 7.2. Again, the solution is still clear and complete penetration of the wood by the sodium pentachlorophenate has taken place. From this, it is apparent that the inorganic components sodium chromate, disodium hydrogen arsenate and sodium fluoride buffer the solution against the acid groups in the wood and allow penetration of sodium pentachlorophenate without precipitation.

Confirmatory experiments also illustrate this buffering action. A 2% salt solution of said typified composition containing sodium pentachlorophenate had an initial pH of 8.5 when technical grade chemicals and tap water were used. Six charges of semi-green southern pine posts were treated using a full cell process following presteaming. After the completion of each charge the pH of the solution remaining was measured. It was then made up to original volume and the pH measured. The results were as follows:

Charge Number	pH Before Treatment	pH After Treatment
1-----	8.5	8.1
2-----	8.3	7.8
3-----	7.95	7.7
4-----	7.7	7.7
5-----	7.7	7.6
6-----	7.7	7.6

This buffering effect takes place well above the critical pH for precipitation of pentachlorophenol.

The FIGURE of the drawing shows the greater improvement in buffering capacity of the inorganic components (1) Na₂HAsO₄, NaF, and Na₂CrO₄, and (2) Na₂HAsO₄, for sodium pentachlorophenate in comparison to the buffering capacity of a mixture of sodium bicarbonate (NaHCO₃) and sodium carbonate (Na₂CO₃).

From this figure of the drawing, it will be apparent that the presence of the NaF and Na₂CrO₄ in the organic salts, has little effect on the buffering capacity.

Further examination of the figure shows that sodium carbonate, sodium bicarbonate formulation has an initial pH of 9.6 and that the buffer capacity is high between this value and a pH value of about 8.5. The sodium arsenate formulations, in contrast, have their greatest buffer capacity between pH 8 and pH 7. The approximate value for precipitation of free pentachlorophenol is pH 7.

The use of sodium arsenate provides the high buffer capacity needed to prevent precipitation of free pentachlorophenol, and a toxicity itself, that adds to the toxicity of the sodium pentachlorophenate. The formulations containing sodium fluoride and sodium chromate, in addition to the sodium arsenate for conjoint use with sodium pentachlorophenate, are non-staining.

A quantitative measurement of the buffering action was made by titrating 100 ml. of 2% inorganic salt solution of sodium chromate, disodium hydrogen arsenate, and sodium fluoride containing as organic component 0.25% pure sodium pentachlorophenate and 100 ml. of aqueous solution containing only 0.25% pure sodium pentachlorophenate with 0.1 N HCl and 0.1 N acetic acid. This measurement shows that in the absence of the inorganic salt components, only 2.6 ml. of 0.1 N HCl were required to reduce the pH from 11.3 to 7.1 where precipitation of pure pentachlorophenol took place. In the presence of the inorganic salt components, 12.0 ml. of 0.1 N HCl were required to reduce the pH from 10.3 to 7.0 and cause precipitation. Only 4.0 ml. of this acid was needed to reduce the pH to the normal inorganic salt pH value of 7.8. The buffering action is most marked in the normal pH range for inorganic salt solutions. Results with acetic acid were the same.

Titration carried out on solutions containing 2,4,6-trichlorophenol show similar results. The buffering action of the inorganic salts was even more marked. It required 27 ml. of 0.1 N HCl to reduce the pH from 9.8 to 6.5 and cause precipitation in the presence of inorganic salts compared to 3.5 ml. required to reduce the pH from 10.9 to 6.8 and cause precipitation in their absence.

4,6-dichlororesorcinol and 2,4,6-trichlororesorcinol are highly toxic to wood-destroying fungi, non-staining, non-sludging and compatible with the inorganic salt components without precipitating over the entire pH range. The additional hydroxyl group in the resorcinol increases the water solubility of these compounds sufficiently that no precipitation problem exists at the concentrations of interest in this application.

The following information indicates the solubility limits of the claimed non-staining mixture of the inorganic salts and its components. Results show that the maximum salt concentration for these salts while maintaining the formulated ratio of components is about 10%. For a mixture of sodium pentachlorophenate and disodium arsenate in a 1:2 ratio, the maximum salt concentration is 30%, and for a 1:2:3 ratio of sodium pentachlorophenate, disodium arsenate and sodium chromate, the maximum salt concentration is 20%.

Sodium pentachlorophenate may be purchased or may be readily prepared as follows: To 472.28 g. of distilled H₂O containing 7.51 g. of reagent grade NaOH, was added 50.00 g. of pure pentachlorophenol. After mixing for one hour at room temperature, some undissolved pentachlorophenol remained. Since these two compounds were reacted in molar ratios, complete solution should have resulted. To this solution 10% NaOH was added until all of the penta had dissolved.

The high pH of this solution (12.3) indicated that there was excess NaOH in the solution. An excess of pentachlorophenol was added to the solution until no more dissolved, and the unreacted penta was removed by filtration. The pH of this solution was 8.73. A 10.00 g. aliquot was placed in a beaker and evaporated in an 80° C. forced air oven over the weekend, and then dried for an additional two hours at 100° C. From the ten grams of solution 1.77 g. of solid remained for a solution strength of 17.7%. All of the following solutions were prepared from this sodium pentachlorophenate solution.

Solubility limits in water: The solutions as prepared contained all or part of the components of the non-staining, non-sludging mixture of salts, as shown in Table II.

EXAMPLES

A 1.2% inorganic salt solution was prepared by mixing the following materials:

	G.
5 NaF -----	1.50
Na ₂ HAsO ₄ +494 g. H ₂ O-----	1.50
Na ₂ CrO ₄ -----	2.25
Sodium pentachlorophenate-----	0.75
	6.00

The pH of this solution after mixing was 9.60. When HCl was slowly added to an aliquot of this solution, turbidity developed at pH of 6.7, and pentachlorophenol precipitated at a pH of 6.6. From this test, it appeared that sodium pentachlorophenate was compatible with inorganic salts in the desired pH range.

The method used to detect qualitatively pentachlorophenol in wood was adapted from a paper by S. S. Sakornbut and H. L. Morrill of the Monsanto Chemical Company. This paper entitled "Detection of Pentachlorophenol in Treated Wood" was published in "Analytical Chemistry," 27, 1259-61 (1955). In this determination, pentachlorophenol is oxidized to chloranil by chlorine dioxide. When wood containing chloranil is sprayed with the leuco base of crystal violet (reduced form), the leuco base is oxidized to crystal violet, and a purple color develops. The leuco base is a 1% solution in xylene and Skellysolve E (petroleum ether).

Two southern yellow pine samples with dimensions of 1½ x 4 x 12 inches were end coated with two applications of resorcinol formaldehyde resin. After drying, they were treated full cell with either a 1% inorganic salt solution containing 0.1% sodium pentachlorophen-

TABLE II
Data for Water Solutions Containing All or Part of the
Components of Inorganic Salts

Soln. No.	Sodium Pentachlorophenate	Na ₂ HAsO ₄	Na ₂ CrO ₄	NaF	Total Solids	H ₂ O	Total	pH	Remarks
1-----	10.0 g., 10.0%.	20.0 g., 20.0%.	30.0 g., 30.0%.	none-----	60 g., 60%.	40 g., 40%.	100 g., 100%.	-----	Large amts. of insoluble salts.
2-----	10.0 g., 5.0%.	20.0 g., 10.0%.	30.0 g., 15.0%.	none-----	60 g., 30%.	140 g., 70%.	200 g., 100%.	-----	Considerable amt. of insoluble salts.
3-----	10.0 g., 3.0%.	20.0 g., 6.0%.	30.0 g., 9.0%.	3.0 g., 0.9%.	63 g., 19%.	270 g., 81%.	333 g., 100%.	8.5	Solution only slightly turbid.
4-----	10.0 g., 2.0%.	20.0 g., 4.0%.	30.0 g., 6.0%.	6.8 g., 1.36%.	67 g., 13.4%.	433 g., 87%.	500 g., 100%.	8.5	Do.
5-----	10.0 g., 1.25%.	20.0 g., 2.5%.	30.0 g., 3.75%.	20.0 g., 2.5%.	80 g., 10%.	720 g., 90%.	800 g., 100%.	8.5	Do.
6-----	10.0 g., 10.0%.	20.0 g., 20%.	none-----	none-----	30 g., 30%.	70 g., 70%.	100 g., 100%.	8.6	Completely soluble.
7-----	10.0 g., 3.33%.	20.0 g., 6.67%.	30.0 g., 10.0%.	none-----	60 g., 20%.	240 g., 80%.	300 g., 100%.	8.6	Do.

Results of this series of tests show that the relatively low solubility of sodium fluoride is the limiting factor which prevents preparation of solutions of high concentration while maintaining the formulated ratio of salts. This ratio is:

Sodium pentachlorophenate-----	1
Disodium arsenate-----	2
Sodium chromate-----	3
Sodium fluoride-----	2

Another important feature appears from the results listed in the table. If solution number 6 were used to treat wood to a retention of 30 lbs./cu. ft., the retention of free penta, if complete conversion from the sodium salt to the free phenol took place, would be 2.8 lbs./cu. ft., or about 10 times the retention of penta usually aimed at with oil solvents. Even solution 5, which has the proper ratio of all materials, would give a free penta retention of 0.35 lb./cu. ft. which is slightly higher than 0.3 lb./cu. ft. considered necessary with treatments using oil type solvents.

ate aforesaid (pH 9.45), or 0.1% sodium pentachlorophenate solution (pH 9.85). The post treatment pH of these solutions was 7.35. After drying these flat grained samples were split in half longitudinally parallel to the radial surface. Then one-half section from each sample was similarly split into four sections so that four sections with dimension of 1½ x ½ x 12 inches resulted. By this method of cutting, penetration of pentachlorophenol could be measured in increments from the outer radial surface to the center of the sample. From each of these strips (1½ x ½ x 12 inches) were cut sections equidistant from each end three inches long, so the final dimensions of the samples to be tested were 1½ x ½ x 3 inches. Sectioning samples in this manner removed areas with end grain penetration, and gave a good indication of the tangential and radial penetration of the penta in inorganic salts.

The above samples and untreated pine controls were oxidized for 30 minutes in chlorine dioxide generated by mixing glacial acetic acid with sodium chloride. After oxidation, the samples were aired for 30 minutes

to remove ClO_2 , then sprayed with the 1% leuco base solution. The samples were immediately placed in a nitrogen atmosphere to prevent air oxidation which would cause a color to develop. A purple color developed on the original outer surfaces of the treated samples in one to two minutes, and the inner surfaces formed the purple color within 10 minutes. A slight color appeared on the control after about 20 minutes, probably because of oxidation by the entrapped air.

Another series of tests was made on additional end coated samples. One sample was treated with the aforesaid inorganic modified salts containing sodium pentachlorophenate with an initial pH of 7.35, and the other with inorganic salts without the penta or dinitrophenol component. The post treatment pH of the modified solution was 7.18. This test was made to determine if some other component of inorganic salts would oxidize the leuco base to its colored form, and if the normal pH of the solution would still allow penetration. After treating and testing similar to the first series of tests, the purple color developed on the samples treated with the inorganic salt solution containing sodium chlorophenate, but no color formed on the other sample or the controls.

Results of this test indicate that complete penetration of sodium pentachlorophenate into pine is readily accomplished when incorporated into inorganic salt solutions at allowable pH values.

What is claimed is:

1. A method of treating wood with a non-staining, non-sludging wood preserving composition, comprising

impregnating wood with 1-10% aqueous solution of a mixture consisting essentially of about 1 part sodium pentachlorophenate, about 2 parts of an alkali metal arsenate, about 3 parts of a water-soluble chromium salt, and about 2 parts of an alkali metal fluoride, said solution having a pH of 7.2-8.5.

2. A method of treating wood with a non-staining, non-sludging wood preserving composition, comprising impregnating wood with a 1-20% aqueous solution of a mixture consisting essentially of about 1 part sodium pentachlorophenate, about 2 parts of an alkali metal arsenate, and about 3 parts of a water-soluble chromium salt, said solution having a pH of 7.2-8.5.

3. A method of treating wood with a non-staining, non-sludging wood preserving composition, comprising impregnating wood with a 1-30% aqueous solution of a mixture consisting essentially of about 1 part sodium pentachlorophenate and about 2 parts of an alkali metal arsenate, said solution having a pH of 7.2-8.5.

References Cited in the file of this patent

UNITED STATES PATENTS

1,465,603	Gohmert	Aug. 21, 1923
1,544,013	Kitchin	June 30, 1925
1,957,873	Wolman	May 8, 1934
2,012,976	Schmittutz	Sept. 3, 1935
2,322,633	Hitchens	June 22, 1943
2,344,019	Bostrum	Mar. 14, 1944
2,784,139	Cutler	Mar. 5, 1957
2,895,848	Baker	July 21, 1959