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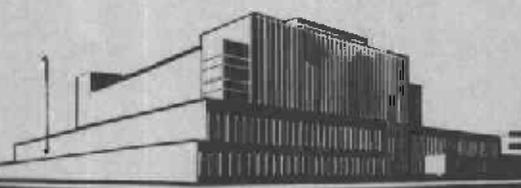
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A SURVEY OF THE PROPERTIES OF COMMERCIAL WATER REPELLENTS AND RELATED PRODUCTS

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FOREST PRODUCTS LABORATORY
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UNITED STATES DEPARTMENT OF AGRICULTURE
FOREST SERVICE

In Cooperation with the University of Wisconsin

A SURVEY OF THE PROPERTIES OF COMMERCIAL
WATER REPELLENTS AND RELATED PRODUCTS¹

By

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In cooperation with the Office of Production
Research and Development, of the War Production Board

A survey was made by the Forest Products Laboratory of the properties of 55 commercial water repellents, water repellent preservatives, wood sealers, and preservative wood sealers to determine the character and range of effectiveness of the products now on the market as a guide for the Laboratory in further studies of water-repellent preservatives. Although the primary interest was in water repellents, sealers were included for two reasons; first, terminology in the field is still confused so that sealers may be obtained when water repellents are requested and vice versa; and second, the inclusion of some sealers was desirable to bring out the contrasts between sealers and water repellents. Definitions of water repellents, sealers, and related products are listed in Appendix I of this report.

Each proprietary product tested was submitted by one of 19 manufacturers or dealers. Most of the products undoubtedly were of separate manufacture but a few cases of duplication of a product from the manufacturer and from a dealer under another trade name may have occurred. Five products were used at two dilutions each because the manufacturer's directions proposed more than one dilution for use. In this report the commercial products are identified only by code numbers.

In addition to the commercial products, eight familiar materials of known composition were included in the study to furnish background for appraising the water repellency of the commercial products. The known materials were:

¹Paints for the paint holdout and paintability tests were designed and made by Don F. Laughnan, Technologist, and the lacquer by A. C. Schwebs, Chemist. The original date of issue of this report was October 1945.

²Maintained at Madison, Wis., in cooperation with the University of Wisconsin.
Report No. 1495

(1) phenolic-resin spar varnish complying with Army-Navy Aeronautical Specification AN-TT-V-118 (since replaced by Specification AN-V-26) applied in three full coats to form a film of substantial thickness over all surfaces of the test specimens; (2) raw linseed oil, undiluted, applied by a single dip, (3) coal-tar creosote, (4) raw tung oil, (5 to 8 inclusive) four formulas for diluted mixtures of linseed oil made in accordance with Army, Corps of Engineers, Tentative Specification T-2288.

Details of the test methods followed are given in Appendix II to this report. Results of the tests are reported in table 1. In column 1 of the table the reference materials of known composition are identified by the numbers XI to X8, inclusive. The commercial products are numbered 1 to 60 followed by a letter designating the manufacturer or dealer so that several from a single source can be recognized without revealing the source. The products are arranged in the table in decreasing order of water repellency for test specimens dipped once for 10 seconds (column 5, subcolumn headed "1/10").

Comparison of Commercial Products with Materials of Known Composition

83% in table 1.
The most effective product tested was the coating of phenolic-resin varnish, which rated (93) percent in effectiveness. Still higher results are obtainable with thicker coatings of varnish or with coatings of many kinds of paints or enamels (see Forest Products Laboratory Rept. No. 1396, table 1, in which the moisture-excluding effectiveness of 3 coats of a similar varnish was 69 percent when tested by the more severe technique used for studying coatings). The best commercial water repellent in table 1 rated 83 percent but it had poor paintability. The best commercial water repellent with acceptable paintability (except with lacquer) rated 77 percent.

Raw linseed oil, undiluted, proved 50 percent in water repellency, raw tung oil 40 percent and coal-tar creosote 42 percent. Since the drying oils antedate the commercial products by many years in use as water repellents for wood, the undiluted drying oils may well be taken as a yardstick for evaluating commercial products. There are 31 commercial products listed above raw tung oil and 29 above raw linseed oil in table 1. Below raw tung oil there are 29 products, which may be considered poor water repellents though some of them may be useful for other purposes.

Diluted linseed oil, containing 20 to 25 percent linseed oil dissolved in volatile solvent, proved to be very low in water repellency. Nevertheless such solutions of linseed oil have sometimes been proposed as water repellents.

Character of Product (Table 1, Column 2)

Lack of established terminology has led to the use of the terms "sealer" and "water repellent" more or less synonymously. This Laboratory believes that

clear distinction should be drawn between them. In Appendix I to this report are given some definitions now under consideration by the Preservative Standards Advisory Committee of the National Door Manufacturers' Association. The commercial products tested ranged from those that were good water repellents under the proposed definitions to those that were good wood sealers; in between were products of intermediate character that were neither good water repellents nor good sealers. Among the 31 commercial products listed above raw tung oil in table 1, 28 must be classified as water repellents on the basis of their relatively deep penetration into end-grainsapwood, feeble tendency to "hold out" subsequent coatings, little or no drying on tinplate, and relatively low viscosity, yet 8 of them have the syllable "seal" or the word "sealer" in their trade names. The 3 products among the 31 superior to raw tung oil that had the properties of sealers were called sealers by their manufacturers. Among the 29 commercial products listed below raw tung oil 19 may be classified as water repellents, although 9 contained "seal" or "sealer" in the trade name. Eight had the properties of sealers; six of the eight were so designated by the manufacturer and two had only noncommittal catalog numbers. Of the 2 remaining among the last 29, one was called a sealer but must be considered a poor one because of its lack of ability to "hold out" subsequent coatings and the other (38D) was a water repellent designed for textiles and not classifiable among either sealers or water repellents for wood.

Preservative Content (Table 1, Column 3)

The manufacturer's statement of content of preservatives was accepted without confirmation by laboratory test. Among 12 commercial sealers 7 were preservative sealers in 4 of which the preservative was chlorinated phenol, in 2 phenylmercury oleate, but for 1 the nature of the preservative was not disclosed. Among 47 commercial water repellents 14 contained no preservative and 33 were water-repellent preservatives in which chlorinated phenol was used in 24, copper naphthenate in 1, phenylmercury oleate in 5, and undisclosed preservative in 3.

Water Repellency (Table 1, Column 5)

All products except the varnish, XI, were tested for water repellency when applied by dipping the test specimens once for 10 seconds (subcolumn headed "1/10"), which is the usual testing procedure for water repellents when using the swellograph method. A selected group of 18 products, chosen to include 9 sealers and 9 water repellents of a wide range in effectiveness, were later retested when applied by dipping once for 20 seconds (subcolumn "1/20") and again when applied by dipping twice for 10 seconds each time, with 24 hours between for drying (subcolumn "2/10"). These extra tests were made to compare sealers and water repellents when applied by the method commonly used when sealers are chosen primarily for their moisture-retarding effect.

Some government specifications for water repellents set 45 percent as the minimum acceptable value. The results indicate that this is a reasonable

minimum for specification purposes. When application was by one dip for 10 seconds there were 28 water repellents or water-repellent preservatives with water repellency greater than 45 percent. Only three products having the limited penetration of sealers had more than 45 percent water repellency when applied by dipping once. Of the three, two were very viscous products that tended to leave an appreciable coating over the wood (unless the excess was wiped off) and the third (No. 14Q) gained a much higher rating than Nos. 39Q and 40Q, which were said to be the same sealer except for addition of preservatives. No. 14Q had been tested previously at several times with results more nearly comparable with those of Nos. 39Q and 40Q. Among the commercial products below 45 percent in water repellency there were 9 sealers and 19 products classed as water repellents.

Change in the time of dipping from 10 seconds to 20 seconds did not change the water repellency proportionately. For the nine water repellents tested both ways the average for 10 seconds of dipping was 54 percent, and for 20 seconds was 55 percent. For the nine sealers tested both ways the average for 10 seconds was 29 percent, and for 20 seconds was 30 percent. The individual cases in which there was a significant difference are subject to the following possible explanations: Number 14Q, as already pointed out, seems to have given abnormally high results in these tests, 26P and 28P were products of such high consistency that variability of single applications was to be expected, 31R was found to diminish steadily in water repellency as time passed (still later results were as low as 28 percent), and 52R and 58M were in the region of very low water repellency where the test method is known to give widely varying results.

When application was made by dipping twice, allowing time for drying between dips, an important difference was found between good water repellents and good sealers. For the nine water repellents tested by the three methods of application the average water repellency was 55 percent for one dip for 20 seconds and 64 percent for two dips for 10 seconds each, an increase of only 9 percent. If the averages are taken for the six highest of these nine repellents the comparison is between 73 percent for single dipping and 79 percent for double dipping, an increase of only 6 percent. For the nine sealers tested by the three methods of application, however, the averages were 30 percent for single dipping and 61 percent for double dipping, an increase of 31 percent. When application is by two dips with time for drying between, sealers apparently are capable of producing as much water repellency as can be obtained with water repellents.

The water repellency measured in these experiments relates to the period soon after treatment of end-grain specimens of sapwood of ponderosa pine. There are few systematic data on the possible changes in water repellency as the treated wood ages but practical observations suggest that the water repellency does not change rapidly during aging indoors and that it persists for at least a few months even on exposure to the weather without further protection by coatings. Numerically the data on water repellency would undoubtedly vary on test specimens of other sizes, shapes, grain direction, species, and even on heartwood instead of sapwood of ponderosa pine. Relatively, however, it

is presumed that those products that perform well in the present test will likewise perform well on other kinds of wood. Further inquiry into the validity of the present assumptions belongs in future studies of the practical serviceableness of water repellents and sealers rather than in this survey of commercial products.

Absorption of Treatment by Swellograph Specimens (Table 1, Column 6)

The amount of water repellent or sealer absorbed per specimen was determined by weighing the test specimens for the swellograph measurements immediately before and after dipping. Considering the averages for the nine water repellents and the nine sealers that were tested by all three methods of application, it appears that in any one method of application sealers are taken up in somewhat larger quantity by weight than are water repellents. Thus for one dip for 10 seconds the average absorption of the nine water repellents was 2.9 grams and of the nine sealers 3.6 grams per specimen. Since sealers are slightly higher in specific gravity than are water repellents the difference by volume was not quite so great. Presumably, the higher viscosity of the sealers, averaging more than 800 millipoises for the nine sealers in question compared with 20 millipoises for the nine repellents, caused the specimens to hold a thicker film of liquid as they were withdrawn from the dip tank. For brief dipping the thickness of this liquid film seems to be more important in determining absorption than is the depth of penetration into the wood finally attained. Further evidence to that effect is the fact that the absorption was no greater, in fact slightly less, in the 20-second dip than in the 10-second dip, both for water repellents and for sealers (for nine repellents 2.9 grams per specimen in 10 seconds and 2.7 grams in 20 seconds; for nine sealers 3.6 grams in 10 seconds and 3.2 grams in 20 seconds).

When application was in two dips with time for drying between, the absorption both of water repellents and of sealers was materially increased. For the nine repellents the absorption for one 20-second dip was 2.7 grams per specimen and for two 10-second dips it was 4.0 grams per specimen. For the nine sealers the corresponding data were 3.2 and 4.1 grams per specimen. The two-dip method resulted in nearly equal total absorption for both repellents and sealers, namely, 4.0 and 4.1 grams per specimen, respectively. The second dip contributed less to the total absorption than the first one did; for water repellents the absorption during the second dip was about half that during the first and for sealers the absorption during the second dip was less than a third of that during the first.

Penetration Test (Table 1, Column 7)

The test for penetration into the end-grain of pine sapwood, which is discussed in Appendix II, is the principal basis for differentiating between water repellents and sealers. Water repellents exhibit penetration of type C, that is, relatively deep penetration in both springwood and summerwood. For the

28 repellents rating 45 percent or more in water repellency the penetration ranged between $7/8$ and $2-5/8$ inches, and averaged 1.78 inch. For the 19 repellents below 45 percent in water repellency the penetration ranged between 1 and 2 inches, and averaged 1.60 inch. The better water repellents, on the average, may therefore have slightly better penetrating properties but the difference is small.

Raw linseed oil, No. X2, penetrated $2-5/8$ inches. Linseed oil diluted with mineral spirits, Numbers X5 to X8, penetrated $1-1/4$ to $1-3/4$ inches.

Wood sealers usually exhibit type-A penetration, that is, $1/6$ to $1/8$ inch into end-grain springwood and summerwood. When diluted with solvent to a viscosity below 500 millipoises they may, and at viscosity below 100 millipoises usually do, exhibit type-B penetration, that is, one-eighth to one-fourth inch in end-grain springwood but as much as an inch in end-grain summerwood.

Paint Holdout (Table 1, Column 8)

In the paint holdout test, high numbers indicate that application of the water repellent or sealer to wood, later followed by coating with a certain semi-gloss enamel, produced a coating of medium gloss which in turn means that the repellent or sealer prevented the absorption of much oil from the enamel. On bare wood the enamel dried with gloss varying from 1.72 to less than 0.92 unit on different specimens. The holdout test is described in detail in Appendix II.

For the water repellents that rated 45 percent or higher in water repellency, the average gloss in the holdout test was 1.83 units, the maximum was 3.64, and the minimum was 0.97 unit. The one repellent giving the maximum of 3.64 was a concentrate intended to be diluted with 1.5 times its volume of solvent before use, in which case the gloss was 2.42 units. For the water repellents that rated lower than 45 percent in water repellency, the average gloss in the holdout test was 2.33 units, the maximum was 4.81, and the minimum was 1.12 units. For the products classified as sealers the average gloss in the holdout test was 6.31 units, the maximum was 10.16, and the minimum was 1.89 units.

In general, good water repellents that penetrate wood relatively deeply have poor holdout properties. A wood sealer can hardly be considered satisfactory unless it has good holdout properties but if it does it cannot be expected to exhibit the depth of penetration obtainable with water repellents.

Paintability (Table 1, Columns 9 and 10)

Details of the test used for paintability, that is, the effect of treatment of wood with water repellent or sealer on the drying and adhesion of coatings applied subsequently, are given in Appendix II. In table 1, columns 9 and 10, the symbol 0 means that treatment of the wood before coating failed to alter drying or adhesion observably, -1 means that the drying or adhesion was slight-

but not seriously impaired, -2 that it was seriously impaired but not to the point of making painting impracticable, and -3 that drying was prevented for at least 24 hours or that adhesion was so poor that the coating came off of its own accord.

Among the 60 commercial products tested, only 16 were recorded as entirely free from deleterious effect on drying or adhesion of any coating tried. Among the 16 were 10 water repellents and 6 sealers. There were another 16 products, 4 sealers and 12 repellents, with -1 ratings for one or more coatings but no -2 or -3 ratings. Rating of -3 for at least one coating was recorded for seven products, all of them water repellents of 45 percent or greater water repellency. Of the seven, only one is said by the manufacturer to be unsuitable for painting.

There was much more interference with drying and adhesion of coatings by the products that showed 45 percent or greater water repellency than by those of lower water repellency. Among the repellents and sealers in the more effective group all but one sealer and one repellent gave precipitates in the acetone precipitation test (column 13 of table 1). Of the 28 repellents and 3 sealers in the more effective group, 2 repellents exhibited no interference with any coating, 2 sealers and 6 repellents were rated -1 for at least one coating, 1 sealer and 13 repellents were rated -2 for one or more coatings, and 7 repellents were rated -3 for one or more coatings. Of the 28 repellents and sealers of less than 45 percent water repellency, only 8 of which formed a precipitate in the acetone precipitation test, none was rated -3 for any coating, 4 were rated -2 for at least one coating, 10 were -1 for at least one coating, and 14 were rated free from interference with any coating. It is noteworthy that 6 out of a total of 12 sealers were found to impair drying or adhesion of at least one coating, two of the sealers receiving -2 ratings for at least one coating.

Of 22 products that failed to form a precipitate in the acetone precipitation test, 11 exhibited no interference with any coating, 9 were rated -1 for at least one coating, and 2 were rated -2 for at least one coating. It appears, therefore, that there may be interference with painting by sealers or water repellents that contain no wax. The worst interference, however, was found among the 37 repellents and sealers that, from the precipitates formed in the acetone precipitation test, may be presumed to contain substantial amounts of wax. Nevertheless there were 8 repellents and 1 sealer, with high water repellency and substantial precipitates in the acetone test, that showed no -2 or -3 ratings for paintability, and 6 that showed no more than one -2 rating for paintability.

The six coatings used in the paintability tests differed greatly in sensitivity to treatment of the wood with repellents or sealers. Coating A was a white, linseed-oil house paint, coating B was a medium green, alkyd-resin trim paint, D was a white enamel containing no lead or zinc pigments, E was an interior varnish, F was an exterior varnish, and coating G was a white lacquer. No repellent or sealer interfered with the adhesion of coatings A, E, or F and their drying was retarded by eight, seven, and nine products, respectively. The retardation rated as much as -2 only for one product for coating A and 2 products for coating F. Coating B proved very sensitive to retardation in drying; its drying was

slowed slightly by 5 products, markedly by 18 products, and was prevented by 3 products. The adhesion of coating B was impaired slightly by 3 products and markedly by 1 product. Coating D was of intermediate sensitivity; it was retarded in drying slightly by 13 products, markedly by 7 products, and its adhesion was impaired slightly by 5 products, markedly by 1 product. Coating G proved to be very sensitive with respect to adhesion though not to retarded drying; one repellent prevented its drying entirely but no other product seemed to affect its drying, whereas the adhesion was slightly impaired by 15 products, markedly by 6 products, and was prevented almost entirely by 3 products.

Drying on Tinplate (Table 1, Column 11)

The behavior of a thin film of water repellent or sealer spread on a nonabsorptive surface such as tinplate reveals important characteristics of the product. All products classed as sealers formed varnish-like films within 24 hours, although one of them showed persistent after-tackiness, as though the drier usually present in varnish had been omitted, and two formed soft films easily gouged with a finger nail. The two sealers forming soft films also contained much material precipitated by acetone (column 13, table 1).

Among the water repellents of 45 percent or greater water repellency 17 exhibited little or no drying properties and left wet films even after 72 hours on tinplate. Another seven formed dry films of a definitely wax-like nature, not at all like varnish, one formed a grease-like film, two formed tacky films, and one a hard film. Among the water repellents of less than 45 percent water repellency, 1 left a wet film, 1 a greasy film, 6 tacky films, 1 a soft but varnish-like film, and 10 left hard films like varnishes.

Sludging Test (Table 1, Column 12)

For the sludging test 40 milliliters of the water repellent or sealer was chilled to 32° to 35° F., centrifuged, and the volume of precipitate formed, if any, was measured. It is considered significant of the stability of the solution during shipment or storage in cold weather and also of the presence of much wax in the product. Where a very small amount of precipitate is recorded it might have been pentachlorophenol.

Among the products with 45 percent or greater water repellency only two, one a water repellent and one a sealer, failed to form a precipitate in the sludging test. With 5 of the remaining repellents the precipitate amounted to a trace only, 2 repellents were recorded as "slight" in precipitation, 1 formed a cloudiness that did not settle out, two formed only 0.2 or 0.5 milliliter of precipitate, and 19 formed 1 to 40 milliliters of precipitate. The three that formed 40 milliliters of precipitate congealed to jellies in the refrigerator. In all cases the precipitate redissolved on returning to 70° F., although it was often necessary to stir the solution to regain uniformity of composition.

Among the sealers and repellents of less than 45 percent water repellency 20 formed no precipitate, 3 formed only a trace, 2 formed 0.5 milliliter, 1 formed 1.5 milliliter, and 1 formed 5 milliliters of precipitate. Those that formed 0.5 milliliter or more of precipitate were in the group above 20 percent water repellency and only one product of the group with 20 to 45 percent water repellency failed to form a measurable precipitate.

Acetone Precipitation Test (Table 1, Column 13)

The acetone precipitation test is considered a test for content of wax although it is not certain that wax is the only possible ingredient that can be precipitated by acetone at 32° to 35° F. or that the volume of precipitate is quantitatively proportional to the content of the wax. The results of the acetone precipitation test were nearly, but not exactly, parallel to the results of the sludging test.

Among the products of 45 percent or greater water repellency only two gave no precipitate with acetone. Two products gave less than 1 milliliter of precipitate, 15 gave 1 to 5 milliliters, 6 gave 6 to 10 milliliters, and 5 gave 10 to 24 milliliters. Among the products of 20 to 45 percent water repellency one gave no precipitate with acetone, one gave 0.75 milliliter, two gave 1.5 milliliters, and one gave 11.0 milliliters. Among products of less than 20 percent water repellency 23 gave no precipitate with acetone, 2 gave 0.25 milliliter, and 1 gave 0.5 milliliter.

Viscosity (Table 1, Column 14)

The viscosity of water repellents of 45 percent or greater water repellency ranged between 9.6 and 49.0 millipoises (No. 4K with 73.1 millipoises was a concentrate to be diluted before use). The three sealers in that range of water repellency had viscosities of 361.8 millipoises or more. For the repellents of less than 45 percent water repellency the viscosity lay between 8.6 and 165.1 millipoises and for the sealers in that range of water repellency the viscosity varied from 31.2 to 2,310 millipoises.

Flash Point (Table 1, Column 15)

To avoid undue hazard of fire it is desirable that products used in open tanks in woodworking plants have as high a flash point as is consistent with reasonable speed of evaporation after wood has been treated. A minimum flash point of 100° F. for water repellents seems to be generally recognized as desirable. Nevertheless, 17 water repellents were found to have less than 100° F. flash point by the Tagliabue closed-cup method. Only seven of them were among the repellents of 45 percent or greater water repellency but one of the seven flashed at 63°, one at 79°, two at 86°, one at 89°, one at 92°, and one at 99° F. There were 20 water repellents of high water repellency with flash points between 100° and 116° F.

Low flash points were even more prevalent among the water repellents of less than 45 percent water repellency. Only nine in that group had flash points of 100° F. or more, one flashed at 39°, one at 40°, one at 42°, one at 59°, one at 62°, one at 74°, two at 96°, and two at 99° F. Only one sealer flashed above 100° F., two had flash points below 30°, one at 35°, one at 43°, one at 71°, two at 75°, and one at 94° F.

Nonvolatile Content (Table 1, Column 16)

Nonvolatile content was determined by the method of drying a small sample at 105° C. (221° F.) for 3 hours. The method is subject to small errors for all oxidizing materials that change in weight when exposed to warm air and for gelatinous materials that tend to retain solvent. It is subject to large errors for materials that contain substances such as chlorinated phenols, which volatilize appreciably but not completely at 105° C. (221° F.). For 28 commercial products the manufacturer's statement of content of nonvolatile was available or could be calculated from information disclosed about the composition. In many products there was satisfactory agreement between the nonvolatile content reported by the manufacturer and that found on testing but there were also a number of serious discrepancies. For products for which the manufacturer stated a minimum content of nonvolatile the amount found was sometimes greater by as much as 6.6 percent. Number 13C, for example, reported 15 percent minimum, but was found to contain 21.6 percent. For products containing chlorinated phenols the nonvolatile content found was usually less, often as much as 4 percent less than that reported.

For water repellents having 45 percent or greater water repellency the content of nonvolatile by weight as determined by analysis varied from as little as 6.2 percent to 32.4 percent (excluding a concentrate to be diluted for use). The average was 16.5 percent. Among these the water repellents without preservatives varied from 6.2 to 32.4, and averaged 15.7 percent. The water-repellent preservatives containing at least 5 percent chlorinated phenols varied from 11.3 to 25.0, and averaged 16.9 percent. Apparently water repellency as high as 77 percent can be obtained with the concentration of water-repellent ingredients being no greater than 10 percent by weight. For water repellents rating below 45 percent in water repellency the nonvolatile content varied from 8.9 to 26.3 percent, and averaged 16.3 percent. For wood sealers, on the other hand, the nonvolatile content ranged from 18.9 to 44.7, and averaged 28.7 percent by weight.

APPENDIX I -- Definitions

The following definitions of water repellent, water-repellent preservative, wood sealer, and preservative wood sealer were prepared by a subcommittee of the Preservative Standards Advisory Committee, National Door Manufacturers' Assn. to be submitted to the Advisory Comm. for action at its next meeting:

"Water Repellent for Wood

"An N.S.P. water repellent for wood (a short way of saying nonswelling, paintable water repellent for wood) is a liquid or a solution that penetrates and continues for some time to spread into wood even when applied by nonpressure methods; that does not swell wood during application; and, after drying, leaves the wood essentially unaltered in odor, in smoothness of surface, in dimensions and shape, does not adversely affect the color of wood, leaves the surface of the wood free from objectionable contamination with oily, gummy, or powdery ingredients of the water repellent, or a coating of appreciable thickness, leaves the wood amenable to the application of the paints, enamels, varnishes, or other wood finishes commonly used on the wooden products for which the water repellent is offered, and imparts to the wood the water repellency consistent with the other requirements.

"Wood Sealer

"A wood sealer is a kind of varnish or lacquer that, when applied by any of the methods customary in finishing wood, penetrates into the wood just enough to leave no coating of appreciable thickness on the surface and, on drying, leaves the lumens of the wood cells and pores nearest the surface largely occupied by nonvolatile ingredients of the sealer in such manner that one or more of the following purposes is accomplished: (a) finishing materials applied subsequently remain almost entirely on the surface without loss from penetration into lumens of the wood, (b) the surface is rendered relatively nonabsorptive of liquids and easy to clean when soiled, (c) a decorative finish of a character not attainable with surface coatings is achieved, (d) the property of moisture exclusion is provided to the degree consistent with the other requirements.

"Preservative Wood Sealer

"A preservative wood sealer is a wood sealer containing fungicide, or fungicide and insecticide.

"Water-repellent Preservative for Wood

"An N.S.P. water-repellent preservative for wood (a short way of saying non-swelling, paintable water-repellent preservative for wood) is a wood preservative that has in addition, the properties of a water repellent for wood."

APPENDIX II - Methods of Testing

The following methods of testing were used in obtaining the data reported in table 1:

Water Repellency (Table 1, Column 5)

Test specimens were cut from a selected stock of 2- by 10-inch flat-grain planks of sapwood of ponderosa pine. The specimens were in the form of thin wafers one-fourth inch in the longitudinal direction, 1-1/2 inches in the radial direction, and 8 inches in the tangential direction. A pair of adjacent specimens from each of 5 different planks, 10 specimens in all, constituted a set for one water-repellency test. All specimens were stored in 65 percent relative humidity at 80° F. until needed. One specimen of each pair in a set was then treated with the water repellent to be tested. Usually by dipping for 10 seconds but in other cases by one of the methods described in footnotes 4 and 13 of table 1, after which all specimens were allowed to dry in 65 percent relative humidity at 80° F. for 7 days.

The rates of swelling of the treated and the untreated specimens when submerged in water at room temperature were recorded in a swellograph. Figure 1 shows three complete swellographs. The test specimen is mounted in a standard which has a sliding bar moved by the swelling of the sample. A differential pulley transmits the motion, magnified 10 times, to a floating pen that scribes the motion on coordinate paper. The paper is mounted on a drum of 24-inch circumference which is rotated by a synchronous clock motor at the rate of one revolution in 12 hours. A cylinder containing water is used to immerse the specimen. The curve drawn on the coordinate paper by the pen records the rate at which the specimen swells, a gentle slope indicating slow swelling and a steep slope rapid swelling.

Swelling of each test specimen was recorded for at least 30 minutes of immersion in water. The last specimens in the swellographs at the end of each working day were usually allowed to remain in place until the next morning by which time swelling equilibrium was reached even for the most effective water repellents tested. This was done to detect products having antishrink properties, that is, the ability to reduce the amount of swelling when equilibrium is reached. No products with antishrink properties were observed among those listed in table 1. Figure 2 shows typical curves recorded by the swellograph. The charts removed from the swellograph drum are turned upside down to view the curves in the conventional orientation.

Water repellency \underline{W} was calculated as follows: if \underline{S}_{ul} is the swelling after 30 minutes in water of the untreated specimen from plank 1 and \underline{S}_{t1} the swelling after 30 minutes in water of the treated specimen from plank 1, then the water repellency in percent found on plank 1 is

$$\underline{W}_1 = \frac{\underline{S}_{ul} - \underline{S}_{t1}}{\underline{S}_{ul}} \times 100$$

In similar fashion the water repellency (\underline{W}_2 to \underline{W}_5 inclusive), on each of the other planks, was calculated. The arithmetic mean of the five results was taken as the average water repellency of the product, \underline{W}_{av} . The variation among the values \underline{W}_1 to \underline{W}_5 inclusive, which was often considerable, may be of significance; hence the standard deviation of the mean was calculated from the customary formula,

$$\sqrt{\frac{\sum \underline{d}^2}{n(n-1)}}$$

where $\sum \underline{d}^2$ is the sum of the squares of the differences $\underline{W}_{av} - \underline{W}_1$, $\underline{W}_{av} - \underline{W}_2$, ...etc. and n is the number of observations.

Treating Solution Absorbed (Table 1, Column 6)

The weight of water-repellent solution absorbed per specimen is not usually determined in routine swellograph tests but the information was considered desirable for this report. Each test specimen was weighed to the nearest 0.1 gram immediately before and after dipping in the treating solution. Where the specimen was dipped twice the weight recorded in column 6 is the sum of the weights absorbed in the two dips. The amount taken up in the first dip was always considerably greater than that in the second. The determinations were subject to appreciable error because draining after dipping was not always complete when the second weighing was made and some loss of volatile took place during draining and weighing.

Penetration Test (Table 1, Column 7)

Test specimens were 1/2 inch by 1/2 inch by 5 inches (longitudinal dimension) of sapwood of ponderosa pine at the moisture content attained after storage in the laboratory. Conditioning of the specimens to a precise moisture content is unnecessary for the penetration test as long as they are kept in an air-dry condition. Three specimens were used for each test of a product. A sample of the product to be tested was placed in a 100 milliliter beaker in sufficient amount to form a layer one-half inch deep. Dry Sudan-red dye was dissolved in the product in the amount necessary to produce a blood-red color. By means of a suitable jig the three test specimens were inserted vertically into the beaker

until their bottom 1/2- by 1/2-inch ends (of end-grain wood) were one-fourth inch beneath the surface of the solution. After 3 minutes the specimens were removed from the solution, allowed to stand in the laboratory for 1 week, then sawed into halves 1/4 by 1/2 by 6 inches, and the freshly sawed surfaces planed with the strokes of the plane always from the undipped to the dipped end of the specimen. The planed surfaces were then examined for type and depth of penetration.

Figure 3 illustrates the three types of penetration that are sometimes observed with water repellents or sealers: Type A, penetration from end-grain surface, usually about 3/16 inch as in A(1), and deeper penetration at times in the summerwood only as in A(2); Type B, two definite zones of penetration, one of which is about 3/16 inch from the end-grain surface but the other, of lighter color, may extend an inch or more in both springwood and summerwood; Type C, penetration in one clearly defined zone only, extending more than three-fourths inch in both springwood and summerwood.

Paint Holdout (Table 1, Column 8)

Test specimens for the paint-holdout test were pieces of Eastern white pine 1/4 inch by 2-5/8 inches by 12 inches in size, planed and sandpapered on the face to be painted. Preliminary experiments indicated that it was not necessary to differentiate between heartwood and sapwood. One test specimen was used for each product tested and 4 specimens were taken for untreated controls. Application of water repellent or sealer was by dipping for 3 minutes followed by 24 hours for drying after which a coat of enamel was applied.

A semigloss enamel was designed to reveal loss of oil to the wood by decrease in the degree of gloss of the coating when dry. The enamel was made as follows:

Titanium dioxide (Titanox AA-10)	1.55 pounds
Basic sulfate white lead	1.04 "
Zinc oxide (Azo 32-33)	1.83 "
Magnesium silicate (Asbestine X)	2.10 "
Bodied linseed oil (viscosity 23)	2.35 "
Mineral spirits	1.81 "
Dipentene	1.46 "
Pine oil	0.15 "
Lead-manganese naphthenate drier	0.07 "

12.36 pounds, makes 1 gallon

Calculated by the method described in U.S.D.A. Technical Bulletin 804, the opaque pigment was 0.259 gallon, the total pigment was 0.194 gallon, the nonvolatile was 0.486 gallon per gallon and the ratio of pigment to nonvolatile was 0.40. The enamel was tinted medium blue-gray with a small amount of lampblack in oil to facilitate observation of differences in gloss because the lower the gloss the lighter the color of the dry coating.

The enamel was applied in one full coat by brushing and was allowed to dry at least 24 hours before observing the degree of gloss. Gloss was then recorded numerically with a Hunter multipurpose reflectometer using the attachment for 75° angle of incidence. For setting the reflectometer, a National Bureau of Standards depolished glass plaque with a gloss of 9.3 at 60° angle of incidence was placed on the aperture, the gloss scale set at 5.0, and the instrument adjusted to bring the galvanometer to zero. The resulting scale of values was an arbitrary one but it made it possible to read the gloss of all of the specimens, which ranged from 0.92 to 10.16, with the 75° attachment. Each specimen was observed for gloss at 3 different portions of the surface and the mean of the 3 readings was reported. At high levels of gloss the 3 readings agreed closely but at low levels of gloss they often varied considerably as would be expected in view of the variability of wood surfaces. For the 4 untreated control specimens the average gloss readings were respectively 1.72, 1.50, 1.39, and less than 0.92.

Paintability (Table 1, Columns 9 and 10)

Test specimens for the paintability tests were of Eastern white pine 1/4 by 2-5/8 by 12 inches in size, planed and sandpapered on the face to be painted, each specimen having at least some heartwood in the face to be painted. It is believed that treated heartwood is more likely to exhibit impaired paintability than is treated sapwood. Seven specimens were prepared for each product to be tested. Sealers and water repellents were applied by dipping the specimens one-half their length (that is, 6 inches) for 3 minutes and letting them stand in the laboratory for 24 hours before applying the first coat of paint, varnish, or lacquer over all of one face. One specimen was prepared for each of the possible combinations of 57 water repellents or sealers with 7 paint systems, making a total of 399 specimens. Lacquer was applied by spraying; all other coatings were applied by brushing. The first coat was allowed to dry for 24 hours after which it was observed for its condition of dryness over the treated and untreated halves of the test specimen and its so-called adhesion on the two halves was tested by scratching with the edge of a small coin. A second coat of paint, varnish, or lacquer was then applied (except on specimens on which the first coat failed to dry), the specimens were allowed to stand 24 hours, and the observations of dryness and adhesion were repeated. The observations reported in columns 9 and 10 of table 1 were those after applying the second coat except where the first coat failed to dry or to adhere, when the paintability was recorded as -3.

The seven paints, varnishes, and lacquers used were as follows:

A. A white, exterior house paint classified according to USDA Technical Bulletin 804 as group TLZ, type 3B, grade 1 made as follows:

Titanium dioxide (Titanox A)	0.906 pounds
Basic carbonate white lead	4.068 "
Zinc oxide (XX50 and XX503)	3.434 "
Magnesium silicate (Asbestine X)	2.054 "

Linseed oil	5.200 pounds
Mineral spirits	.320 "
Lead-manganese naphthenate drier	.167 "
	<u>16.149</u> pounds, makes 1 gallon

The equivalent opaque pigment was 0.263 gallon, the total pigment was 0.260 gallon, the nonvolatile was 0.928 gallon per gallon, and the ratio of pigment to nonvolatile was 0.280.

B. A trim paint of medium chrome green color made with a long-oil alkyd-resin vehicle, classified according to U.S.D.A. Technical Bulletin 804 as group CL (e), type 1A, grade 2 made as follows:

C. P. chrome green, medium	4.985 pounds
Alkyd resin (Duraplex D-63) solution	4.985 "
Aliphatic mineral spirits	1.017 "
Aromatic mineral spirits	.677 "
	<u>11.664</u> pounds, makes 1 gallon

Naphthenate driers were added to the extent of 0.4 percent lead, 0.04 percent cobalt, and 0.0175 percent manganese based on the resin content. (The Alkyd resin solution contained about 63 percent nonvolatile by volume.) The equivalent opaque pigment was 0.340 gallon, total pigment was 0.136 gallon, nonvolatile was 0.522 gallon per gallon, and the ratio of pigment to nonvolatile was 0.26. Like most commercial trim paints, this one was slow in drying and had a strong tendency to after-tackiness.

C. An interior enamel undercoater made with an ester gum and linseed oil varnish of 15-gallon length in oil (to be used under enamel D) made as follows:

Titanium dioxide (Titanox AA-LO)	0.968 pounds
Titanium-calcium pigment	3.876 "
Magnesium silicate (Asbestine X)	2.262 "
Ester gum varnish	2.946 "
Mineral spirits	2.212 "
	<u>12.264</u> pounds, makes 1 gallon

The varnish contained 51.5 percent nonvolatile by volume. The undercoater contained 0.275 gallon of equivalent opaque pigment, 0.275 gallon of total pigment, and 0.473 gallon of nonvolatile per gallon, and the ratio of pigment to nonvolatile was 0.572. Enamel undercoater C was used as a first coat only, after which a finishing coat of enamel D was applied. Results with the system of undercoater and enamel are omitted in table 1 but are recorded in the original records of the work.

D. A white, interior enamel made with an ester gum and linseed oil varnish of 20-gallon length in oil, U.S.D.A. classification group T(e), Type 5, grade 1 made as follows:

Titanium dioxide (Titanox AA-10)	2.552 pounds
Magnesium silicate (Asbestine X)	3.358 "
Ester gum varnish	4.547 "
Mineral spirits	.757 "
Dipentene	.410 "
	<u>11.624</u> pounds, makes 1 gallon

The varnish contained 68.2 percent nonvolatile by volume. The enamel contained 0.330 gallon of equivalent opaque pigment, 0.220 gallon of total pigment, 0.633 gallon of nonvolatile per gallon, and the ratio of pigment to nonvolatile was 0.348.

E. An interior varnish made from ester gum and linseed oil at 20-gallon length in oil:

Ester gum, acid number 10 to 16	100 pounds
Bodied linseed oil, viscosity Q	20 gallons
Mineral spirits	32.7 "
Lead naphthenate solution	5.33 pounds
Cobalt naphthenate solution	2.14 "

Cooking log: Three-fifths of the ester gum and all the linseed oil was heated to 305° C. (581° F.) in 50 minutes. It was held until a 12-inch string came off the paddle (about 2 hours) and then was checked with the rest of the ester gum. It was removed from the fire and let cool for 20 minutes. The mineral spirits and the driers were added. The nonvolatile content was 54.5 percent by weight and the viscosity was D (Gardner-Holdt scale).

F. An exterior varnish made with modified phenolic resin, linseed oil, and tung oil at 25-gallon length in oil:

Modified phenolic resin (Bakelite BR-2963)	100 pounds
Raw tung oil	8-1/3 gallons
Bodied linseed oil, viscosity Q	16-2/3 "
Mineral spirits	45 "
Lead linoleate solution	5 pounds
Cobalt naphthenate solution	1 pound

Cooking log: All the resin, tung oil, and one-third of the linseed oil was heated to 293° C. (559° F.) in 60 minutes. The rest of the linseed oil was added and 271° C. (520° F.) was regained in 70 minutes. The lead linoleate was added and stirred thoroughly. After removal from the fire, when the temperature dropped to 213° C. (415° F.) (about 80 minutes) the mineral spirits were added. The cobalt naphthenate was added when the mixture was cool. The nonvolatile content was 56.8 percent by weight and the viscosity was B (Gardner-Holdt scale).

G. A white lacquer enamel for interior use containing 45 percent by weight of nitrocellulose (1/2-second viscosity), 35 percent alkyd resin, and 20 percent

plasticizers (tricresyl phosphate and blown castor oil) in the nonvolatile vehicle and pigmented with titanium dioxide:

Titanium dioxide	2.355	pounds
Nitrocellulose, 1/2-second viscosity	1.750	"
Alkyd resin (Rezyl 12)	1.360	"
Tricresyl phosphate	.285	"
Blown castor oil (Baker's No. 15)	.493	"
Nitrocellulose solvents	2.625	"
Toluol	1.360	"
Xylol	.207	"
	<u>10.435</u>	pounds, makes 1 gallon

The nitrocellulose was purchased in solution in the nitrocellulose solvents, the nature of which was not revealed. The lacquer enamel contained 59.2 percent total solids by weight, 42.6 percent by volume. The ratio of pigment to nonvolatile by volume was 0.167. For application by spraying, the lacquer enamel was thinned with an equal volume of reducer consisting of 60 percent toluol, 30 percent butyl acetate, and 10 percent butyl alcohol by weight.

Drying on Tinplate (Table 1, Column 11)

The water repellent or sealer was flowed on a piece of tinplate and allowed to stand in the laboratory. The condition of the film was observed after 24 hours and after 72 hours.

Sludging Test (Table 1, Column 12)

Forty milliliters of the water repellent or sealer was placed in a 50-milliliter centrifuge tube with conical bottom and a graduated scale, chilled to 32° to 35° F. by placing in a refrigerator for at least an hour, and then quickly placed in a centrifuge and rotated for 3 minutes at 2,000 revolutions per minute with the meniscus about 7 centimeters from the axis of rotation. The centrifugal force exerted was calculated to range from more than 300 times gravity at the meniscus to 800 times gravity at the tip of the whirling tube. After centrifuging, the volume of precipitate was read by means of the graduated scale. When the precipitate was recorded as 40 milliliters the entire solution congealed to a jelly.

Acetone Precipitation Test (Table 1, Column 13)

Ten milliliters of sample were placed in a centrifuge tube similar to that used for the sludging test, 30 milliliters of cold acetone was added, the mixture stirred and the tube placed in the refrigerator for at least 1 hour. The tube was then whirled in the centrifuge and the volume of precipitate observed. Paraffin wax or other waxes are precipitated by cold acetone. No varnishes or

resin solutions so far tested were precipitated but it is not certain that there are no possible ingredients other than wax that are precipitated.

Viscosity (Table 1, Column 14)

Viscosity was determined at 80° F. by means of Ostwald viscometers calibrated with viscosity standards certified by the National Bureau of Standards.

Flash Point (Table 1, Column 15)

Flash point was determined by the Tagliabue closed-cup method following the procedure described in American Society for Testing Materials, Standards 1944, Part II, page 984, method D56-36 and in Federal Specification TT-P-141a, method 429.1.

Nonvolatile Content (Table 1, Column 16)

Nonvolatile content was determined by the procedure of Federal Specification TT-P-141a, method 404.1, namely, by drying a weighed sample of approximately 1.5 grams at 105° C. (221° F.) for 3 hours.

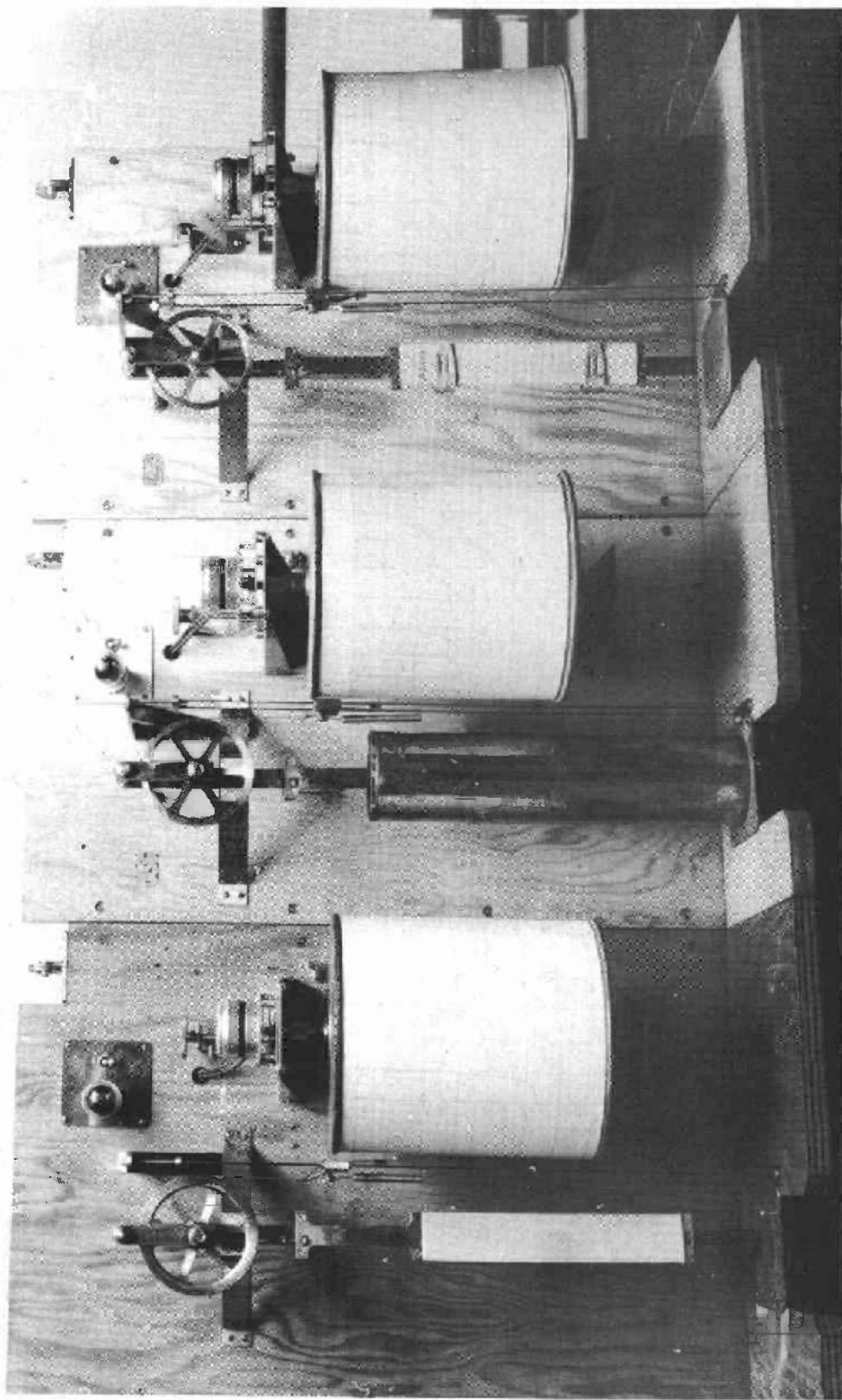


Figure 1.--Three swellographs. The one in the center shows the cylinder removed in place that submerges the specimen; the ones at left and right with the cylinder removed show specimens in place in the standard with the sliding bar resting on top of the specimens. Before immersing in water the specimens are clipped, as shown in the machine at the right, to prevent bowing while swelling. Upward movement of the sliding bar is transmitted through the differential pulley at a 1-to-10 ratio to the floating pen that scribes on the coordinate paper mounted on the drum which in turn is rotated once in 12 hours by a synchronous clock motor. A light mounted in the plate containing the toggle switch above the motor indicates when the motor is operating.

Z M 66060 F

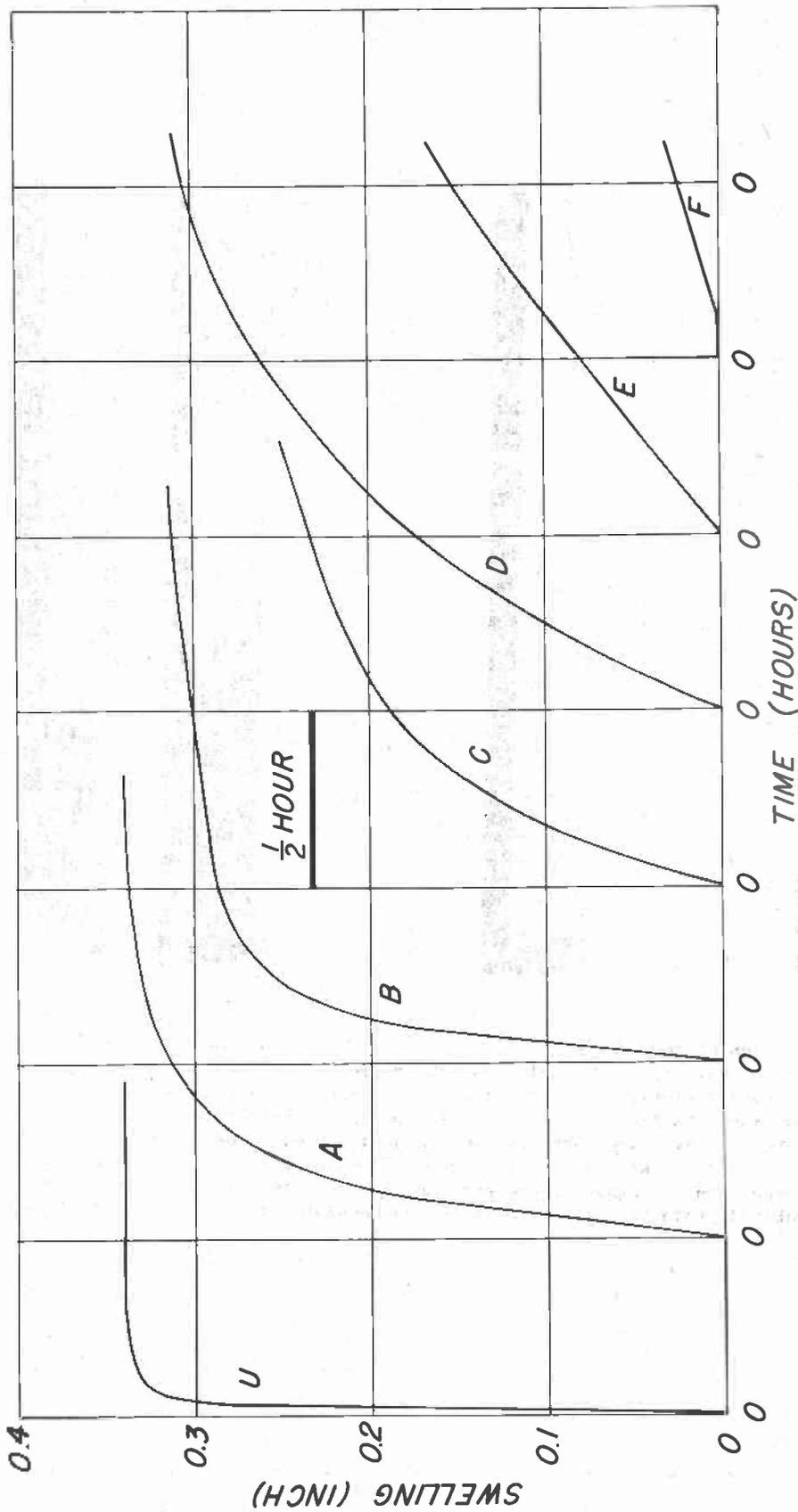


Figure 2.--Typical charts scribed by swellograph. Curve U is for untreated wood. Curves A to E inclusive are for wood treated respectively with: A, a water repellent of 5 percent effectiveness; B, a sealer of 16 percent effectiveness; C, a water repellent of 45 percent effectiveness; D, undiluted linseed oil of 50 percent effectiveness; E, a water repellent of 77 percent effectiveness. Curve F is for wood covered with three coats of phenolic-resin varnish of 93 percent effectiveness. Note that curve F runs along the horizontal axis for 7-1/2 minutes before water penetrates the coating and begins to swell the wood.

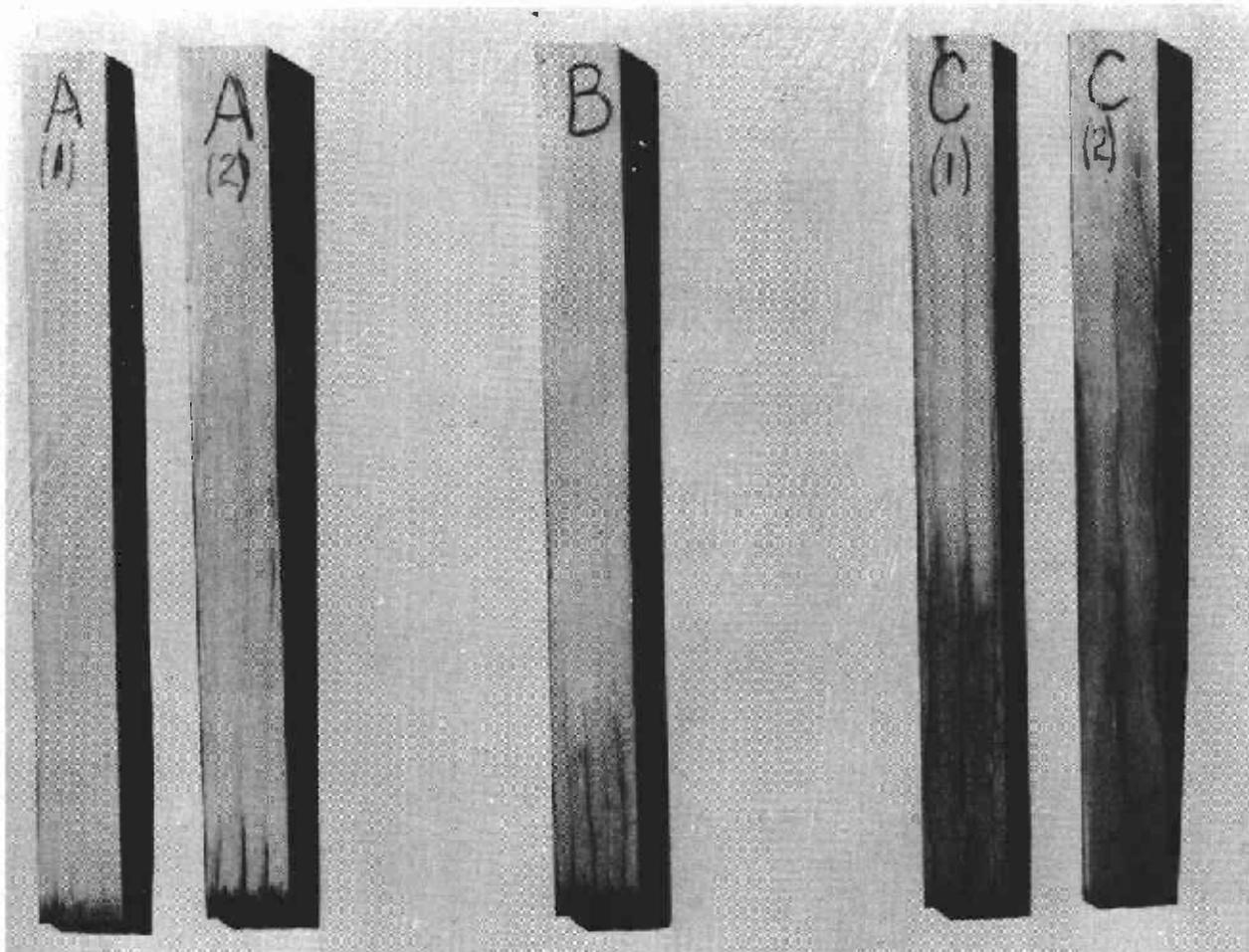


Figure 3.--Three types of penetration obtained in the penetration test for water repellents and sealers. Specimens A(1) and A(2) represent the shallow penetration typical of good wood sealers, sometimes with deeper penetration in summerwood than in springwood as shown in A(2). Specimen C(1) represents typical penetration and specimen C(2) exceptionally deep penetration found with good water repellents. Specimen B, in which there are two zones of penetration of which the shallower one is darker in color than the other, represents an intermediate condition often found in poorly formulated products that are neither good sealers nor good water repellents.

APPENDIX III -- Composition of Water Repellents

The commercial water repellents and water-repellent preservatives tested by the Forest Products Laboratory were of undisclosed composition except for the nature and amount of the preservative ingredient, if any, such as chlorinated phenols, copper naphthenate, or phenylmercury oleate. It is not the custom of the industry to report the ingredients that impart the property of water repellency without undue interference with subsequent painting of wood that has been treated with the product.

Further studies were made by the Forest Products Laboratory to discover preparations of known composition that possess properties similar to those of the commercial water repellents as shown in table 1, particularly the properties of wood water repellency, good penetration, and satisfactory paintability. A number of formulations were found that, when tested by the methods used for the survey, matched the properties of the best commercial products closely but none of the Laboratory formulations was superior to the better commercial products.

Although many natural and synthetic resins, varnishes, and miscellaneous ingredients were tested, it was not found possible to achieve the desired degree of water repellency in a single application without incorporating a moderate proportion, usually from 2 to 3 percent, of a hydrocarbon wax such as paraffin wax or ceresin. Vegetable waxes and other true waxes proved much less effective and resins or varnishes of all kinds still less so. Hydrocarbon wax, then, was the essential ingredient for water repellency in all of the satisfactory laboratory formulas. Wax alone, however, was harmful to subsequent painting. A sufficient addition of drying oil or resin was found necessary to keep the wax from interfering too much with painting. Many drying oils and resins were found to serve the purpose. Some of the less expensive resins were just as satisfactory as the more costly ones. Resins and drying oils were dissolved in the solvent without much heating because highly viscous or polymerized ingredients tend to impair penetration into the wood.

Six formulas that were considered satisfactory in all of the tests are listed in this appendix. They are selected as illustrative of suitable compositions, not because any one of them is considered outstandingly meritorious. Many other formulations could be added. None of them has been prepared on a scale larger than laboratory batches. Further study on a larger scale should be made before using any of them for commercial production. Moreover, production for use should be closely supervised by technical men well versed in the art of making paint and varnish products. The solvents and some of the ingredients are highly inflammable and can be seriously harmful to workmen unless handled with suitable precautions. It is strongly recommended that users of water repellents and related products purchase them from competent manufacturers and do not attempt to make them for themselves.

<u>Formula WRP-1</u>	<u>Percent by weight</u>
Raw linseed oil.....	17.0
Paraffin wax.....	3.0
Pentachlorophenol.....	5.0
Aliphatic mineral spirits.....	37.5
Aromatic mineral spirits.....	<u>37.5</u>
	100.0

<u>Formula WRP-2</u>	
Ester gum.....	6.5
Raw linseed oil.....	6.5
Paraffin wax.....	2.0
Pentachlorophenol.....	5.0
Aliphatic mineral spirits.....	40.0
Aromatic mineral spirits.....	<u>40.0</u>
	100.0

<u>Formula WRP-4</u>	
Long-oil alkyd resin.....	13.0
Paraffin wax.....	2.0
Pentachlorophenol.....	5.0
Aliphatic mineral spirits.....	40.0
Aromatic mineral spirits.....	<u>40.0</u>
	100.0

Formula WRP-5
 Coumarone-indene resin in place of the long-oil alkyd resin in WRP-4

<u>Formula WRP-6</u>	
Ester gum.....	7.0
Paraffin wax.....	3.0
Pentachlorophenol.....	5.0
Aliphatic mineral spirits.....	42.5
Aromatic mineral spirits.....	<u>42.5</u>
	100.0

<u>Formula WRP-7</u>	
Methyl ester of hydrogenated rosin.....	8.0
Paraffin wax.....	2.0
Pentachlorophenol.....	5.0
Aliphatic mineral spirits.....	42.5
Aromatic mineral spirits.....	<u>42.5</u>
	100.0