TESTING WOOD PRESERVATIVES 1137



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For many of the products in industrial use, there are laboratory tests by which the utility of the material can be determined quickly and by which accurate comparisons can be made between competitive products in a short time. With such materials, it is possible to obtain an immediate and dependable evaluation of a new product that is offered in competition with older materials. This fact naturally leads to the common belief that the effectiveness of wood preservatives can also be determined quickly by simple laboratory tests. Unfortunately, this is not correct for there are no short-time laboratory tests by which the relative effectiveness of different wood preservatives can be compared with assurance that similar results will be obtained from treated wood in actual service. Laboratory tests can be made to give very useful information and frequently will show that the new material offered as a preservative is not worth testing further. When thorough laboratory tests give favorable results, a preservative can be considered promising, but it is not safe to conclude that equally favorable results will be obtained in service. It is necessary to observe the performance of a preservative over a period of years in actual use before it can be reliably evaluated or compared with other preservatives. Even then, the results may be misleading unless a considerable volume of timber under different use conditions is considered.

Observations must be made on many different installations under various conditions of service because the service life secured by the use of any preservative or process will vary as these factors vary. A few early failures of treated wood in service do not necessarily mean that the preservative or process is ineffective. The preservative may have been applied improperly or in inadequate quantity, or the conditions of service may have been abnormally severe. On the other hand, successful performance for a few years, or in a few cases, is not adequate evidence of high effectiveness. The results from a large number of installations must be considered together before the true picture is obtained.

The great disadvantage of waiting for the results of service tests is the very long time usually required for their completion. It is especially discouraging to the promoters of new preservatives, for a long period of pioneering effort is necessary and, with patented materials, the major portion of the life of the patent will have passed before the preservative is conclusively evaluated. Nevertheless, there is as yet no short cut by which the necessity for service records can be avoided.

Requirements of a Good Preservative

In order to be generally useful, a wood preservative must (a) be toxic to the organisms that injure or destroy wood, (b) have suitable permanence, (c) have satisfactory penetrating properties, (d) be safe to handle and use, (e) be harmless to wood and metal, and (f) be inexpensive and plentiful. For the treatment of certain special products, such as millwork or manufactured articles, it may also be required that the preservative be paintable, odorless, colorless, fire resistant, moisture repelling, or nonswelling, or that it have combinations of several of these or other properties. The relative importance of the different requirements depends upon the character of the product treated, the purpose for which it is to be used, and the conditions of service.

Toxicity is required in all wood preservatives, in order to make the wood poisonous to the various organisms that damage it. It seems possible for preservatives to be repellent toward some insects without being toxic, but for general effectiveness, and especially for protection against fungi and marine borers, toxicity is necessary. It is not infrequently claimed that decay prevention is accomplished by "waterproofing" the wood, but this is incorrect for no "waterproofing" material or treatment yet discovered, that is practical for common use, does more than retard the rate of water absorption. Under continued exposure to dampness, the wood can usually absorb enough water in a few weeks or months, even through the best of "waterproofing" treatment, to permit its decay. This is one of the reasons why paint does not prevent decay.

Since treated wood must last many years in order to give satisfactory service, it is apparent that a preservative must have a considerable degree of permanence. No preservative is acceptable that evaporates readily or that otherwise disappears in a short time from the wood or changes chemically into compounds that are ineffective. Benzel and turpentine are examples of chemicals that are toxic but that evaporate from the wood too quickly to give the desired protection. Complete resistance to evaporation, leaching, or chemical change is not required, but the rate at which the preservative becomes ineffective must be so slow that adequate protection is afforded over a sufficient period of time.

A preservative should not be dangerous to handle nor make the treated wood hazardous to the health of the men who must prepare it or build with it. It must not endanger persons or animals who may later

A possible exception is the impregnation of wood with synthetic resinforming materials which, as yet, is not cheap enough for common use. (See reference (4) at end of paper.)

come into contact with the treated wood or occupy structures containing it. Any preservative, if taken internally in sufficient quantity would probably cause death, but that is true of most of the chemicals in daily domestic or industrial use and is not a valid objection. Any special hazard, however, such as explosiveness, high fire danger, extreme poisoning hazard, or tendency to produce occupational disease, may prevent the commercial use of an otherwise suitable material.

Preservatives must penetrate wood satisfactorily in order to be acceptable. It is not reasonable to expect deep penetration by mere brushing, or brief immersion, although that would be highly desirable, if obtainable. Good penetration by such methods is not infrequently claimed for new preservatives, but, so far as is known, it has not been successfully attained. New preservatives, however, should penetrate as satisfactorily as those in common use, when applied by pressure or by other suitable methods. The protective zone must be deep enough so that it is not easily broken through in handling or by the ordinary wear and season checking to which wood is normally exposed in service.

Freservatives that attack wood and seriously reduce its strength are obviously unsuitable for prolonging its life. Corrosiveness to metal is also undesirable for corrosive preservatives damage the treating apparatus as well as the bolts, nails, and other metal with which the treated wood must be in contact when in use.

Low cost and a plentiful supply are important requirments in a new preservative unless it is so outstanding in other respects that its cost becomes of secondary importance. The value of the product being treated, the service life desired, and competition with other materials largely determine the permissible cost. The commonly used preservatives are low in price, when purchased in large quantities, and are not likely to be supplanted by new materials that markedly increase the cost of treatment. The wholesale prices of coal-tar creosote, for example, are commonly in the range of 12ϕ to 16ϕ per gallon; zinc chloride sells for about $3-1/2\phi$ to 5ϕ per pound in large lots, and even some of the special preservatives used for the treatment of window sash can be obtained for 25ϕ per gallon or less. Retail prices are necessarily much higher because of the greater cost of packaging, selling, and distributing.

Color, odor, and painting properties are usually not important considerations in the treatment of railway ties, piling, fence posts, and many other forms of timber that are to be used out of doors or in contact with the ground. In highway guard posts, however, and sometimes in fences around houses and estates, or in telephone or power line poles, cleanness and paintability are important items. In residences and other buildings, it is usually desirable that the preservative leave the wood just as free as possible from color and odor and as suitable as untreated wood for all types of stains, paints, varnishes, and other finishes.

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It is seldom demanded that a preservative make the wood highly resistant to fire, although this would often be of great advantage. It is a definite disadvantage, however, if the preservative increases the flammability of wood used in buildings or other structures where fire hazard is of importance.

A preservative that would prevent moisture changes in wood in service, and thus prevent shrinking and swelling, would be advantageous in many uses of wood. Since it is a requirement that cannot be met by the preservatives now in use, it is not demanded in new preservatives for general use. If such a preservative ever is developed it will undoubtedly find fields of usefulness not now met by any preservative.

A special requirement in the treatment of manufactured articles, such as furniture, millwork, store and office fixtures, and similar products, is that the preservative, when injected, shall not swell the wood, for swelling causes grain raising, distortion of plane surfaces, and severe stresses in the joints. The shrinkage that takes place during the seasoning of wood swelled in treatment leaves open joints and warped, roughened surfaces. For these reasons, water-borne preservatives are seldom used in the treatment of furniture and millwork and toxicants carried in organic, nonswelling solvents are preferred.

Laboratory Tests

Much useful information can be obtained through laboratory testing methods on toxicity, penetrating properties, corrosiveness to wood and metal, painting properties, color, odor, fire resistance, fire and explosion hazard, water repellence, and swelling. Laboratory tests also give information on various factors that affect permanence, such as volatility, leachability, and chemical stability, but not enough to obviate the necessity of service tests on materials of promising character. Health hazards to workmen handling the preservative and the treated material, and to others who may subsequently have contact with the treated wood or be influenced by it are difficult to determine accurately.

Toxicity. -- The malt-agar culture method and the wood-block method are the two general methods of making tests for the toxicity of chemicals to fungi. Both give useful, comparative information, but both are purely empirical. The results by either method are influenced very greatly by the details of manipulation as well as by the species of fungus used as the test organism. Results obtained by the two methods are not directly comparable, and often different workers using the same method differ enough in their technic to cause considerable variations in results.

The malt-agar culture method, which is the toxicity method most commonly used in the United States for wood preservatives, consists in growing the test fungus on malt-agar culture media containing different

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concentrations of the preservative under test (3). With nonvolatile materials, petri dishes may be used as containers, but, when it is desired to reduce moisture losses during the test, or to prevent evaporation and loss of volatile materials, closed Erlenmeyer flasks may be employed. After the culture media containing the different concentrations of preservative have been prepared and poured into the petri dishes or flasks, a small square of healthy mycelium cut from a specially grown culture is planted in each container. At sufficiently low concentrations, the fungus will grow and spread out over the culture medium while at higher concentrations the fungus transplant may be killed by the preservative. An intermediate concentration may be found at which the fungus will not grow, but at which it is not killed. This is called the total inhibition point. The lowest concentration at which the fungus is killed is called the killing point. These two values are expressed as percentages of the concentration of the preservative in the culture medium. In working with a new preservative, it is often desirable to use rather wide variations in concentration in a preliminary test to locate the killing point approximately, and then to determine it more accurately by a retest, using smaller variations in concentration in the vicinity of the approximate killing point. Since each set of determinations requires a month or more, a complete series of tests may have to extend over 3 to 6 months, or even longer.

The killing point values obtained vary considerably with different species of fungi (2). For a preliminary survey, it is often sufficient to use one fungus, but in any comprehensive study, several species should be employed. When using only one fungus in the agarplate toxicity test, the Forest Products Laboratory usually employs a special strain of a fungus known as Madison No. 517, which grows well in the laboratory and has been found very suitable for the purpose.

In Europe, investigators prefer to make toxicity tests by treating wood blocks with different concentrations or absorptions of the preservative under test and then placing them in flasks on mats of vigorously growing fungus (1). Untreated wood is placed in each flask with the treated wood, for comparative purposes. As in the agar-plate method, it is desirable to use several species of fungi. The exposure is usually continued for 3 or 4 months, after which the blocks are removed, examined for softness, then dried and weighed. The amount of deterioration of the wood, as indicated by loss in weight and strength, and concentration of preservative required to prevent the growth of the fungus, afford bases for comparing toxicities.

The European method gives excellent results and is widely used. Other wood block methods are also used to some extent which follow the same general principles as the European method, but vary in details $(\underline{6})$.

Numbers in parentheses refer to literature citations at the end of this discussion.

The fact that the test is made in wood, the material the preservative is intended to protect, is an advantage. A disadvantage is that the block test requires 2 or 3 times as long as that using the malt-agar culture medium.

Wood preservatives are sometimes tested for toxicity against bacteria. The results obtained are practically valueless as an indication of wood preserving effectiveness because the test organisms are not wood destroyers and their behavior is not indicative of the behavior of wood destroyers.

The results of toxicity tests must be used with caution. Differences in toxicity are too frequently assumed to be a direct measure of the relative effectiveness of preservatives, or to indicate the quantity required per cubic foot of wood to afford protection, neither of which is correct. Other properties, especially permanence, are of equal or greater importance. The most toxic preservative is valueless without sufficient permanence and comparatively low toxicity may be acceptable if permanence, cost, and other properties are favorable. Very high toxicity, when other properties are also favorable, indicates the possibility that low concentrations of the toxicant in the preservative solution, or low net absorptions of the preserving compound, may be sufficient. It is not safe to risk the protection of important structures or products upon this assumption, however, until its correctness has been demonstrated by field experiments and service records.

Penetration.—Accurate comparisons of the penetrating properties of different preservatives are difficult to make because of the great variability of wood in resistance to penetration. Even in different parts of one face of a piece of wood, the penetration frequently varies by several hundred percent. There are also wide differences in penetration in different directions of the grain. End or longitudinal penetration, for example, usually averages more than 5 times the side penetration and sometimes is as much as 50 times as great. End penetrations of 15 to 20 times the side penetration are common. There is also a difference between side penetration across the annual rings (radial penetration) and side penetration parallel to the annual rings (tangential penetration), the tangential penetration being greater, in most species. Penetration usually is very much greater in sapwood than in heartwood although there are exceptions to the rule.

For the foregoing reasons, specimens selected for comparative penetration tests should be either all sapwood or all heartwood, long enough to avoid complete penetration from the ends, and of large enough cross section to allow separate measurement of radial and tangential penetration. They should also be closely matched so as to be as nearly alike as possible in density, rate of growth, direction of grain, and other characteristics that may influence penetration. When practicable,

it is desirable to make the specimens square in cross section, with two sides showing edge grain and two sides showing flat grain. Many experimenters and promoters of new preservatives have deceived themselves and others by failing to observe the above precautions. Salesmen also, by selection of easily penetrable wood and dependence upon end penetration alone, sometimes induce prospective customers to believe that their product has remarkable penetrating properties.

While accurate quantitative comparisons of penetrating properties are not practicable, it is possible, with materials that are colored or can be made to give color reactions, to make rough comparisons and to learn whether the new material is significantly different in this respect from those in common use. The depth of penetration of creosote and other colored oils is easily distinguishable upon cutting into or splitting open the treated wood. Copper sulphate and certain other salts also have sufficient color to be seen. Zinc chloride and sodium fluoride are examples of preservatives that are colorless, but whose presence can be made visible by spraying the treated surfaces with chemicals that produce colored compounds when they react with the preservative, or that are colored themselves, but are changed in color by reaction with the preservative in the treated parts of the wood. With colorless preservatives for which no color tests have been devised, it is difficult and often practically impossible to obtain even a good approximation of the depth to which they have penetrated. The addition of dyes to colorless solutions does not give accurate indications of penetration because the dyes frequently filter out or fail to penetrate as far as the preservative. A rough indication of the probability of good penetration can be obtained, for materials that do not react with the wood, by a consideration of their viscosities for, other things being equal, lower viscosities favor deeper penetration.

When testing the penetrating properties of a proprietary preservative, consideration should be given to the claims made by its promoters and the tests made accordingly. For example, preservatives recommended for application by brush or other superficial method should be applied in that way in the test, whereas preservatives recommended only for pressure treatment should be applied by pressure.

Volatility. -- The volatility or loss of preservative by evaporation from the wood in service is governed largely by the vapor pressure of the preservative at the temperatures to which wood in service is exposed. Data on the vapor pressures of many chemicals are not so complete as may be desired, but the boiling point of most chemicals is known and constitutes a fairly good index of their volatility at ordinary atmospheric temperatures. The volatility of oils and mixtures of oils can be studied by making fractional distillation tests by the standard distillation procedure in use for coal-tar creosote, and comparing the results with those obtained from creosote. With organic solids, determinations of melting point and boiling point are useful, as well as behavior on continued heating at different temperatures. Inorganic solids seldom are volatile enough to give concern.

Leachability .-- When treated wood is in contact with water, the preservative may be gradually dissolved out of the wood by the water. No chemicals are absolutely insoluble in water, but some have such low solubility that they leach very slowly. The toxic constituents of coal-tar creosote, for example, have such low solubility in water and leach so very slowly that the creosote gives protection to structural timbers for periods as long as 50 years or more, if present in substantial quantity to begin with. Although creosoted wood usually does not last that long, its ultimate destruction is ordinarily caused by factors other than leaching. Even preservatives that are readily soluble in water, if originally injected in suitable quantities, require some years to leach to the point where they no longer afford protection. When preservatives are sufficiently soluble to make water solutions of satisfactory strength for treatment, differences in their solubility are not a measure or resistance to leaching. For example, zinc chloride is soluble in water in practically all proportions while the solubility of sodium fluoride is only about 4 percent at normal atmospheric temperatures, yet the rate at which they leach from wood is very similar.

There is no standard method of making leaching tests, but a common method is to impregnate blocks of wood, submerge them in water for definite and repeated periods of time and make analyses of the water to determine the amount of leached chemical it contains. Analyses may also be made of the wood after leaching. With some chemicals, such analyses can be made rather simply and accurately. Other chemicals require much more inconvenience, time, and expense; for some, satisfactory methods of analyses may not even be available. On the whole, comprehensive and reliable leaching tests are both expensive and time consuming.

The chemical stability of a preservative in wood is one of the most difficult properties to measure in the laboratory. It is sometimes possible to classify a material as unpromising because of its known tendency or the tendency of chemically related materials to decompose spontaneously or to react with water, oxygen, or carbon dioxide. The fact that a material reacts vigorously with some constituent of wood at a high temperature leads one to suspect that a slow reaction may take place at ordinary temperatures, even though such reaction is not at once apparent. There are no standard tests for chemical stability and when a special study of this property is required the methods of investigating it must be selected according to the nature of the chemical under consideration.

Accelerated field tests can be made by treating stakes or saplings of small cross section and placing them in the ground in a soil and climate that favor rapid deterioration. The rate of deterioration is noted by pulling the stakes and inspecting them once or twice a year. Untreated stakes should always be included for comparison. Although the test specimens are exposed to natural conditions, their small cross section and their large ratio of surface area to unit volume favor more rapid loss of preservative by leaching, volatility, and some types of chemical change than takes place in timbers of structural size. The balance between the

factors that govern the loss of preservative is not the same in small specimens as in larger pieces. Specimens 1/2 to 3/4 of an inch square and 1-1/2 feet long may be used to advantage. Common 2 by 4-inch lumber is also used occasionally. The specimens should preferably be all sapwood and complete penetration of preservative should be obtained so that no failure will result from incomplete penetration. A range of absorptions should be used with each preservative tested, including some that are above and some below the absorptions recommended for ordinary connercial use.

The conditions to which the field test specimens are exposed are more representative of severe service conditions than any laboratory test. The irregular periods of wetting, drying, freezing, thawing, and exposure to sunlight occurring in actual service, as well as the variety of chemicals, fungi, and insects present in the soil, create such complicated and varying conditions of exposure that it is not practical to simulate them in laboratory tests. Since these conditions are also constantly varying in a field test and are different in different localities, direct comparisons between preservatives can be made only when their respective test specimens are exposed at the same time and place.

While a field test of this character may properly be considered an accelerated test, it cannot be completed quickly for good preservatives may protect even these shall specimens for 10 years or more. It is also impossible to predict with accuracy from an accelerated field test how long treated structural timbers will last in service. The field test, however, next to service tests of full-sized timbers of various classes, affords the best method of comparing different preservatives and, if it is conducted carefully and on a sufficiently comprehensive scale, the results may be given considerable weight in considering comparative effectiveness.

Corrosiveness to Wood and Metal.—Wood is resistant to attack by most chemicals and is seldem damaged by preservatives. Strong acids and alkalis destroy it in time and certain other chemicals, in concentrated solution attack it. The probability of attack by a new preservative can usually be determined from its chemical composition. When tests are necessary, they may be very time consuming and expensive and may have to include comparative strength tests on treated and untreated specimens. No standard method of testing this property has been devised.

Comparative corresiveness to metal can be measured by hanging strips of metal in containers of the respective preservative solutions maintained at constant temperature, and noting their loss in weight from time to time. Another method is to drive nails or screws, made of the respective metals, into treated wood, expose the wood to different moisture and temperature conditions, and then measure the amount of deterioration of the metal by noting the loss in weight, the depth of corrosion, or the loss in strength. While there is no standard test for the purpose, it is usually possible to obtain sufficient information by the above methods to determine whether the preservative is noticeably harmful to wood or metal.

Painting properties, or the effect of a preservative on paint films applied over it, may be apparent in at least three different ways, namely, bleeding of preservative through the film, staining of the film by dissolving some of the preservative, and reduction of the normal life of the film. Coal-tar creosotes and similar oils are not satisfactorily paintable because of bleeding and staining, and they probably also reduce the serviceable life of the paint. Zinc chloride and other water-borne, inorganic preservatives, as a rule do not stain or bleed through paint, but some of them may decrease its life. Any preservative that discolors wood will, of course, reduce its suitability for transparent finishes.

Tests on the staining of paint can be made by applying paint to treated wood, in sawed and round forms, and observing the extent to which discoloration occurs. The same specimens can then be placed outdoors where they will be freely exposed to sunshine. Observations for bleeding and staining should be continued through at least 1 year. By continuing the exposure over 4 or 5 years, information can also be obtained on the life of the paint coating. For a conclusive test on the effect of the preservative on paint durability, however, it is necessary to include both treated and untreated specimens of otherwise similar character and in sufficient numbers to give averages that are reliable and are not too greatly influenced by the behavior of single specimens. Several types of paint should be used and the test should be continued long enough to require one or more repaintings. Observations made within a few months after painting are not conclusive on the question of durability, and may be very misleading. The technic of testing raint durability is not simple and a careful study of paint testing methods is desirable before important tests on the paintability of preservatives are started.

Fire Retardance.—The extent to which a preservative changes the resistance of wood to the spread of fire can be measured by the "fire-tube" test (5). This test indicates any marked change from the performance of normal untreated wood. However, it does not indicate directly the ease of ignition of the treated wood or its resistance to charring through or "penetration," upon continued exposure to flame or high temperatures. No satisfactory test is known for ease of ignition. Resistance to fire penetration can be compared by placing test specimens over a standard flame or over the opening of a furnace maintained at a definite temperature or temperature range, and observing the time required for the temperature of the outer face to rise a given number of degrees above its original temperature, or for flame to show through.

These small scale tests give very useful indications as to how the treated wood compares with the untreated and they usually are sufficient. When the necessary apparatus is available, the tests need not be very expensive unless conducted on an elaborate scale. If more conclusive evidence is required, it may be necessary to build complete structural units of treated and untreated wood and expose them to large scale, standard fire tests, which are very expensive.

Water Repellence. -- This property is confined to materials of oily, waxy, or resinous nature for inorganic salts are not water repelling. The capacity of a preservative to reduce the rate or amount of water absorption in wood can be studied by exposing treated and untreated specimens to high humidity or to water for different lengths of time and noting their respective changes in weight and dimensions. The amount of change in the treated pieces during a given period of time, compared with that in the untreated pieces, affords a measure of the effectiveness of treatment. The length of the exposure period, however, influences the results enormously. In very short exposure periods, the untreated wood may absorb considerable water while the treated specimens absorb practically none. Over a period of two or three weeks, however, it may be found that the treated wood has absorbed about as much water as the untreated. Most, if not all, treatments merely retard the rate at which water is absorbed, but do not prevent it. Untreated wood, when submerged in water, absorbs water quickly at first and then more slowly until it reaches the saturation point, after which no more water can be absorbed. The treated wood absorbs water more slowly from the beginning but, if allowed to remain in the test long enough, it ultimately absorbs just about as much as the untreated, if allowance is made for the space taken up by the preservative. The behavior is very similar when the specimens are exposed to air at high humidity, instead of to water, the principal difference being that the maximum amount that can be absorbed by the specimens is the amount required to bring the moisture content of the wood up to the equilibrium point corresponding to the air humidity. Comparative values of waterrepelling effectiveness, therefore, are practically meaningless unless the time of exposure is stated.

The fire and explosion hazard of the preservative during its preparation and storage, and during the treatment and the subsequent handling of the freshly treated wood can usually be determined sufficiently from the composition of the preservative. Water solutions of inorganic salts, of course, usually present no fire or explosion hazards. Oils and the common organic solvents have fire and explosion hazards more or less in proportion to their volatility. There are some solvents, of course, particularly those containing a high proportion of chlorine, that are volatile, but do not burn readily, if at all.

The health hazard of a new material cannot be determined quickly or easily. Usually the character of the material will give some information as to the probable health hazards connected with its preparation, handling, and use, for long experience in the industrial use of chemicals of various kinds has furnished much information of this character. When experimental work is required on a new material to learn to what extent it is dangerous to health, the experiments should be made by specialists in this field.

Other Properties. -- Some properties, such as color and odor, usually do not require formal tests for they can be readily observed. The tendency of the preservative solution to swell wood can usually be deduced from the composition of the solution, but can easily be tested by noting whether swelling takes place when wood is treated. Tests for other

special properties, if required, must be devised according to the specific requirements to be met and the general character of the material under test.

Precautions should be taken in all tests conducted, to see that the test specimens and their method of preparation are such that they give representative results. Abnormal absorptions, for example, should be avoided unless the character of the test makes them necessary. Preservatives promoted for superficial application only should not, ordinarily, be applied to the test specimens by more thorough methods. The size and shape of the test specimens should receive careful consideration so that excessive end penetration does not lead to false conclusions. Absorptions used in durability and permanence tests should be chosen with consideration for the absorptions used or recommended for commercial practice. The number of test specimens should be sufficient to give dependable averages and, in all other ways, every care should be taken to avoid reliance upon nonrepresentative material or inadequate tests.

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