The Evaluation of Wood Preservatives Part I

Interpretation and Correlation of the Results of Laboratory Soil-Block Tests and Outdoor Test Plot Experience, with Special Reference to Oil-Type Materials

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This paper offers a review and interpretation of laboratory and field experiments aimed at determining the necessary protective threshold quantities of wood preservatives. It details the procedure followed in the soil-block tests at the Bell Telephone Laboratories, Incorporated. Discussion of specific criticisms of the techniques involved and replies to these criticisms are included. The paper also presents for the first time a correlation of the results obtained from soil-block culture tests, outdoor exposure tests on stakes and on pole-diameter posts as well as pole line experience. It demonstrates that the same levels for toxicity-permanence requirements (thresholds) are obtained from the three different types of accelerated experimental evaluations. There is every reason to believe that the same limits apply for the outer inch of sapwood in pine poles in line.

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INTRODUCTION

In discussing the problems involved in the evaluation of wood preservatives over the years, it has generally been found necessary to orient the audience — in this case the readers of this JOURNAL — in the field of biology, and particularly in the field of biological tests involving wood-

destroying fungi. It is impractical to expect from such tests the degree of accuracy in results that one would look for as a matter of course in certain types of well conducted physical or chemical experiments. One can, however, look for high reliability in the biological sense. In the half science, half art of wood preservation there is as yet no generally acceptable laboratory technique for measuring the preservative value of a given material. Although much development work has been done, both here and abroad, in an effort to promote standard laboratory procedures, their proponents have had very little success in bringing into line the techniques used in the various areas. The interest of the Bell System in establishing a standard bioassay test will become convincingly evident as this story unfolds.

When the first American telephone lines were built there was an adequate supply of naturally durable pole timber in northern cedar and chestnut forests. The chestnut trees have been killed by a fungus disease, the chestnut blight; and the chestnut supply failed completely about twenty years ago. Northern cedar trees are not straight enough nor large enough nor plentiful enough to meet the demands of the power and communication utilities, but they are still used to some extent in the Lake States area. Usually they are incised at the ground line by toothed machines; and they are then given a preservative treatment with creosote or with pentachlorophenol in petroleum to prolong the life of the butt and ground line section.

In the northern and western states the increasing demands for poles 35 feet and longer brought in western red cedar, a straight and nearly perfectly shaped pole tree. The present Bell System use of the species is relatively small, about 4 per cent of the total annual production. Butt treatment of western red as well as northern cedar began in earnest about thirty years ago. This procedure protects the ground section. Many western cedars are now full length treated because, although the species is durable, the tops and sapwood layers are subject to infection and decay, sometimes after a relatively short service life.

In the South and Southeast the great favorite is naturally the southern pine pole, full length pressure-treated with creosote. Such poles made their way in the Bell System as far north as Memphis and Washington by the turn of the century. Their use increased rapidly after World War I, and they moved into virtually all parts of the country. They now make up about 73 per cent of the telephone pole plant. New treatment procedures for southern pine employing pentachlorophenol in petroleum applied by pressure processes are now under way at a number of plants.

Pressure-treated Douglas fir and butt-treated western red cedar dom-

inate other species on the West Coast and in the Pacific Northwest, while pressure and non-pressure treated lodgepole pine poles are favored in the Mountain States area. Pressure-treated jack pine and ponderosa pine move into telephone plant in small quantities in the Lake States and in the California areas, respectively.

To render telephone service the Bell System has some 20,000,000 wood poles carrying its wires and cables. Many of these poles are used jointly with the power companies. Since poles of the joint use sizes are not available in sufficient quantities in the southern pine forest to meet all the demands of the utilities all of the time it is inevitable that western cedar, Douglas fir, lodgepole pine, red pine and western larch should move into various parts of the System, either for the direct and sole use of the Operating Companies or for joint use.

The pole plant is continually changing. Pole species from the Northwest vary greatly in their treatability and they are generally harder to treat than southern pine. It is not possible to use traditional creosote pressure treatments for some of these species without running the risk of objectionable exudation, or bleeding, of the creosote.

The development of practical specifications for the application of new preservatives such as pentachlorophenol and greensalt, as well as the various types of creosote, to all of the pole species now used in Bell System plant calls for setting as exactly as possible necessary protective quantities of the various preservative materials. This is particularly true in view of the fact that for normal telephone use as well as for joint use it is absolutely essential to deliver to the Operating Companies poles that are clean and satisfactory for use in all types of telephone lines, without compromising on the question of adequate physical life for the treated units. This purpose is back of the Laboratories' efforts to develop bioassay tests that come as close as practicable to measuring the necessary protective amount of any given preservative, and to predicting its relative permanence in poles and crossarms in plant.

It has been pointed out in earlier papers[30, 31, 76] that Bell Laboratories' concept of preservative evaluation involves (a) laboratory evaluation tests, (b) test plot experiments with small stakes, (c) similar tests of pole size specimens, and (d) test lines selected for long time observation. The latter are chosen with the cooperation of the Operating Companies. Lumsden[76] has recently presented a summary of a quarter century of experience with pole-diameter posts in one test plot located at Gulfport, Mississippi. The principal aims of the present paper are to interpret the results of various laboratory methods of preservative evaluation, and to

indicate how these results may possibly be correlated with test plot and field experience.

A SHORT HISTORY OF THE DEVELOPMENT OF LABORATORY EVALUATION PROCEDURES

The practice of laboratory evaluation of wood preservatives developed along different lines in Europe and in the United States. Here the Petri dish method was the early favorite.[61, 100] The basic scheme of this test is to use agar culture media containing gradient concentrations of the preservative material to be tested, and to employ various easily grown test fungi as indicators of inhibiting or lethal doses. The same scheme is employed with stoppered Erlenmeyer flasks. The fungus now known as Madison 517, formerly referred to as *Fomes annosus*, has been used most frequently as the standard test organism although other fungi were also used.[101]

In the culture phase of the European standard agar-block method[33] the test fungi are grown in Kolle flasks on a malt agar medium. The impregnated wood test blocks are supported on glass "benches" just above the surface of the agar and the growing test fungus. Wood pulp or paper boards saturated with malt extract are used by some investigators[9, 54 (1)] in place of the agar medium alone. Generally an untreated block and a treated block are placed together in the same flask.

The concept of a test for wood preservatives that motivated the proponents of the German agar-block method was broad enough to include selection of the test blocks and test fungi, treatment and handling procedures except weathering tests, culture technique, determination of the protection boundary, and directions for reporting the results. Differences in the behavior of water solutions of single chemical compounds such as sodium fluoride, and of volatile oily preservatives such as the creosotes were recognized; and provision was made for dealing with both types of materials.

The formalizing of both the Petri dish agar method in the United States and of the agar-block method in Europe developed as a result of conferences called by Dr. Hermann von Schrenk, the first in St. Louis in 1929, and the second in Berlin in 1930. The Laboratories' representatives at the St. Louis conference were the writer and R. E. Waterman. The action taken at St. Louis was published by Schmitz in 1930.[100]

In a previous paper[99] about a year earlier, Schmitz had discussed various laboratory test procedures, and had offered an "improvement" in the Petri dish technique based on the idea of preventing evaporation of volatile materials. Some of his statements at that time now seem by

hindsight to have something of the character of a judgment before trial; but their bearing on the questions under discussion and their possible effect in retarding the development of more realistic methods appear to be important enough to warrant quoting at this time. For example, with special reference to Petri dish agar tests he says:

"The determination of the toxicity of relatively volatile substances, such as coal tar creosote, is particularly difficult, owing to the control of the loss of preservative during the sterilization process. In order to prevent this loss, it is proposed to place the preservative in small sealed glass ampules, which are later broken to liberate the preservative to form preservative-agar mixtures of any desired concentration."

He considers laboratory tests of toxicity of preservatives to have little or no application in commercial practice, and his opinions are definitely stated as follows:

"Toxicity studies deal only with the poisonous properties of a wood preservative, and therefore they do not give a complete picture of the value of any particular substance as a wood preservative. . . .

"*For commercial work, however, it is of interest to know the amount of material that must be initially injected into the wood to maintain the desired amount of preservative for a definite period of time.* (Author's italics). Laboratory studies of the toxicity of wood preservatives do not give this information. Attempts to calculate the amount of material which must be injected into the wood from laboratory studies of toxicity are, therefore, based upon an erroneous conception of the value of such studies."

Writing about the laboratory use of impregnated blocks of wood in testing wood preservatives, which was already well under way in Europe, he says that by using wood one may obtain conditions more or less closely resembling but not identical with conditions in actual service; but one would not only have to use a solvent in treating to low retentions, but there would be difficulties in obtaining an even distribution of the preservative in the wood. Furthermore:

"Getting rid of the solvent would require considerable time, during which a considerable loss of creosote would occur. . . .

"The composition of the creosote in the impregnated wood after the solvent has evaporated may be quite different from that of the original sample. More important still, the movement of the solvent in the wood during drying would cause an uneven distribution of the creosote."

The reader will bear in mind that these opinions were expressed in advance of the St. Louis and Berlin conferences on laboratory evaluation methods. Schmitz repeated them essentially in his 1930 paper, saying, for instance:

"Toximetric values are not in themselves an index of the wood preserv-

ing value of the substance tested. Other factors, such as leaching, volatility, chemical stability, penetrability, cost, cleanliness, etc., must all be considered in the final evaluation of a wood preservative."

With respect to the European wood block test, he felt that

" . . . until more confidence can be placed in the even distribution of the preservative in the test block(s) their use will be greatly limited."

He has maintained his arguments with a high degree of consistency in later papers, and they have unquestionably influenced American thought on laboratory procedures and their practical application.

The Petri dish method adopted as a possible American standard procedure at the 1929 St. Louis meeting followed closely the techniques that had been developed and published by Humphrey et al.,[61] Batemen[8] and Richards.[94] Bell Telephone Laboratories made an intensive study of the Petri dish method during this period. The data obtained were never organized for publication since it was felt that the required evaluation of toxicity and permanence of toxicity of preservatives could not be obtained by the Petri dish test.

European workers would accept neither the Petri dish test method nor Madison 517 as the test fungus. In 1931, about a year after the Berlin conference, and four years before Liese et al[71] reported on the task force development of the agar-block method, A. Rabanus of the I. G. Farbenindustrie Aktiengesellschaft, Germany, published his "Die toximetrische Prüfung von Holzkonservierungsmitteln" (Toximetric testing of wood preservatives).[86] A somewhat expurgated and amended translation of this paper was presented to the American Wood-Preservers' Association in 1933. In the writer's opinion much of the force of the Rabanus argument was lost in the translation. The emphasis on the relative merits of the agar toximetric test and of the agar-block test was considerably diluted; and the cautiously guarded but nonetheless positive philosophy on the possibilities of using the results of agar-block tests in actual wood preserving practice was made water thin.

Apparently there was an understanding that subsequent to the 1930 conference in Berlin[71] tests by the agar-block method would be run in the United States. For this project samples of creosotes as well as Scotch pine wood blocks were sent to a number of workers; but to the writer's knowledge no treated blocks were ever tested, or if they were no results were ever published. At Bell Telephone Laboratories some of the untreated Scotch pine blocks were put through preliminary trials with the Kolle flask technique,[125] and also a considerable number of plate and flask agar toxicity tests were run with the two sample creosotes. The inconsistency of the results — as far as translation to practical wood

preservation was concerned — was a strong stimulant toward the Laboratories' development of a block test, referred to later. It was more or less general information at the time that agar toximetric tests with native and European strains of test fungi were being run in other laboratories in this country; but again — as far as the writer knows — the results were not published.

In the meantime, and, as in the case of the Rabanus article cited above, before the publication of the Liese[71] report, Flerov and Popov[48] published in 1933 in German the basic general principles of a soil-block test. The significance of the article by these two Russian investigators was apparently completely lost on American workers until the publication in England in 1946 of Cartwright and Findlay's "Decay of Timber and its Prevention."[26] Findlay had been a member of the Berlin conference. Flerov and Popov were familiar with the discussions and result of the conference, and decided in favor of the soil base for their cultures after a critical review of the various methods then in use. Their proposals to all intents and purposes were unknown here.

Van den Berge's comprehensive thesis[116] on "Testing the Suitability of Fungicides for Wood Preservation" appeared in Dutch in 1934. A mimeographed English translation was made available soon after for limited distribution. European workers were about ready to confine the use of the agar toximetric test to determining relative toxicities only of various preservatives *in an agar medium*. Liese and his colleagues[71] summarized the arguments and experiments on the agar-block method in 1935, and launched it into a status of general acceptance in Europe and Great Britain. The British[24, 45] and German[33] editions of the standard were issued in 1939. The Rumanian version[110] — closely following the German — came out in 1950. Jacquiot,[64] Lutz[77] and Alliot[2] worked out proposals for standard procedures that would be more comprehensive and in their opinion better applicable to wood preservation research in France.

The Petri dish — and later the stoppered Erlenmeyer flask — agar methods continued to be used by many American investigators for testing wood preservatives, and there is no denying a certain utility in these methods for developing information about fungus poisons. The persistency of the agar techniques can be traced through publications by Richards,[94] Schmitz,[99, 101] Snell and Shipley,[108] Schmitz, Buckman and von Schrenk,[102] Schmitz, von Schrenk and Kammerer,[103] Bland[14] and Hatfield.[53] Baechler still uses the closed flask-agar method and the fungus called Madison 517 for determining basic toximetric values;[4, 5] and Finholt[46] has recently been bold enough to state that "Fungitoxic materials

can be evaluated as wood preservatives by mixing the toxic substances with a malt extract agar solution and then testing the mix against standard fungi."

Flerov and Popov had used sand in their preliminary experiments, but they were by no means the first to do so (see Falck[44]). Rabanus[86] had reported his experiments with sand-block cultures two years earlier. He placed a pair of wood blocks — one treated and one untreated — on glass rods on wet sand in Erlenmeyer flasks; and after sterilization he inoculated the blocks directly with his test fungi. He points out that in this procedure the conditions were less favorable for the fungi than when the treated wood is placed above or on a vigorously growing culture, as in the agar-block test.

Since the papers by Rabanus and by Flerov and Popov appeared in the same journal, one can assume that the latter knew of Rabanus' work. How much any of them knew of still earlier work by Breazzano is uncertain. His work in Italy,[20, 21, 22, 23] begun in the first decade of the century, is evidence of the intense interest of the management of the Italian railroads in some practical laboratory means for testing wood preservatives that would provide results sooner and with more definiteness than the traditional service tests. Parts of Breazzano's report of Oct. 9, 1913 are worth quoting in full from the English translation as historical background information. He reviews the situation as he sees it, and says:

"New systems and various substances for injection into wood are constantly being put on the market by industrial concerns, so that the Railway Administration finds itself confronted by an ever increasing number of processes to be examined and tested for efficiency."

By 1910 the Railway Experimental Institute

" . . . is well on the way toward testing the efficacy of a system of wood preservation by a method which gives dependable results even after a few months of observation.

" . . . after making use also of the advice on the subject received directly from Prof. Tubeuf and from Netzsch's laboratory . . . positive results were obtained with the following technique:

"On the bottom of an Erlenmeyer flask of 200 ml capacity was placed a thin layer of sand." After sterilization in dry heat at 180°C "sterilized water was poured on the sand to moisten it well. Then there was placed on the sand the sample of wood, of dimensions about 9 x 2 x 1 cm., with one end resting on the damp sand and the other on the inside wall of the flask."

The whole setup was sterilized in an autoclave at 120°C for about 20

minutes. The wood was inoculated by placing a piece of a culture of *Coniophora cerebella*, grown on agar medium, directly on the wood. Breazzano states that the wood was kept moist enough because of the water in the sand, that the fungus grew luxuriantly, and that "the development of the fungus was evidently at the expense of the wood, since no other nutritive substance was at its disposal."

He used blocks cut from treated beech ties. The fungus grew readily and he concludes that the treatment was not effective. He ends this early report with the statement:

" . . . If the experiment is carried on under carefully defined conditions the various methods proposed for immunizing woods can be judged all by the same standard."

Breazzano presented his method at Pisa in 1919, and in 1922[21] the principles of the sand-block culture were proposed as standard procedure (for Italy) for evaluating wood preservatives. Precise directions were given for the whole test technique, with important modification of the cultures, as indicated in the steps outlined below:

1. Sterilize by dry heat, at 180°C, "soyka" boxes 8 cm in diameter and 4 cm in height "in which is first placed a layer of sand 1 cm deep".
2. Prepare blocks of wood — treated and untreated — 4 x 4 x 2 cm, cutting them so that the broader faces will be transverse sections; and place these test blocks broad face down on the sand.
3. Sterilize at 100°C for one hour.
4. After sterilizing and cooling add sterile water in an amount that will be slightly in excess of what the sand can absorb.
5. After the wood blocks become moist plant *Coniophora cerebella* — without carrying over any agar medium with the transplant.
6. Incubate the "soyka" box cultures in a covered crystallizing dish in a dark place for one month at 20–25°C; and "Take care that in this time the water which the sand absorbs does not evaporate completely, and add sterile water when necessary."

At the end of the test the wood blocks were to be examined for decay; and if there was any doubt the wood was to be sectioned and examined microscopically for the presence of wood-destroying fungus hyphae (threads).

In retrospect the subsequent changes involving the use of soil instead of sand, and in the testing of blocks specially treated for the experiment, seem like refinements of Breazzano's methods. He later shifted to the use of very thin pieces of treated wood for his test specimens,[22] [23] severely criticizing the agar-block method that grew out of the Berlin conference as time consuming and inaccurate (loc. cit.).[72]

In Bell Telephone Laboratories, R. E. Waterman and his colleagues started work on a wood-over-water block method for testing wood preservatives soon after the St. Louis conference, and they published their early results in 1937 and 1938.[67, 126, 127] Their block was a ¾-inch cube with a hole drilled through it in the approximate center of a transverse face. The ¾-inch cubes simply represented sections of the ¾-inch square stakes that had been substituted for round saplings[69] in the small specimen test plot experiments. The hole served a double purpose — it facilitated handling the blocks during drying and sorting operations[126] and it served as a point of entrance of moisture, which was purposely provided for the block by means of a wood wick.

Leutritz[70] formalized a soil-block test completely independently of Flerov and Popov, and published his method in this JOURNAL (Vol. 25) in 1946, following an earlier short article in 1939[68] suggesting soil as a culture medium.

Beginning in the summer of 1944 and continuing until June 30, 1951, Bell Telephone Laboratories subsidized in part a series of studies by the Madison Branch of the Division of Forest Pathology, of the United States Department of Agriculture, Bureau of Plant Industry, in cooperation with the Forest Products Laboratory at Madison, Wisconsin. The results of these studies and of parallel investigations have appeared in eight papers[16, 35, 36, 37, 38, 39, 40, 95] from 1947 to date. The differences between the agar-block and the soil-block techniques, and the results obtained in comparable test series by the two methods are of fundamental importance. They are presented and discussed at length in a paper by Duncan.[41] Already some 40,000 blocks have been tested by the soil-block method at Madison, with 75 oil-type preservatives. Both at Madison and at Bell Telephone Laboratories, Murray Hill, additional work aimed at further refining of the soil-block technique is under way.

Subsequent to discussions of the new soil-block techniques between representatives of Bell Telephone Laboratories and of the Forest Products Laboratories of Canada, Sedziak[106] has developed a soil-block test involving burying the block in the soil all but one corner; and instead of placing it on a fungus culture growing on feeder blocks, he inoculates a corner of the test block directly.

For a general review of laboratory and test plot methods for evaluating wood preservatives the interested reader should have available, in addition to Cartwright and Findlay's book,[26] at least two more recent books, namely "Wood Preservation During the Last 50 Years" by van Groenou, Rischen and van den Berge,[118] and the third edition of Holzkonservierung by Mahlke-Troschel-Liese.[78] Hunt and Garratt[62] survey wood preservation

with particular reference to the American scene. The works of Boyce,[19] Baxter[10] and Hubert[58] should be consulted for general information on wood-destroying fungi and the pathology of timber products. Kaufert[65] prepared a concise bibliography of pertinent articles in 1949. For a fuller coverage the book by van Groenou, Rischen and van den Berge will be found most stimulating.

Much of the European work on the testing and application of wood preservatives has been summarized in challenging form by the investigators at the Berlin-Dahlem testing station.[54] In this memorial volume, the first paper, by Schulze, Theden and Starfinger, is a compilation of the results of comparative laboratory tests of wood preservatives by the agar-block method. So much work has been done that the ingenious graphical summary table is about 12 feet long; and even then the authors have omitted many results because the conditions of the standard test[33] were not observed. Becker[54(2)] brings up to date the results of testing insecticides in the second article; Becker,[54(3)] in the next paper, summarizes tests for termite control; and Becker and Schulze[54(4)] in the fourth article cover laboratory tests of preservative materials for the control of marine borers. Six additional articles on subjects directly related to wood preservation complete an excellent supplement to the Mahlke-Troschel-Liese book already cited. The emphasis is, somewhat naturally, centered on the work of the Berlin station.

Rennerfelt and his colleagues[42, 43, 88] are conducting a series of laboratory, decay chamber and test plot experiments in Sweden, aimed at evaluating wood preservatives for use in that country, and at possible correlation of experimental results with actual experience.

Bienfait and Hof[13] are working in Holland on what appear to be the broadest test post experiments in Europe at the present time, under both land and water exposure conditions. Their tests of 10 preservatives and some 3350 posts of Douglas fir, Scotch pine, European larch, Sitka spruce, poplar and willow rival Bell Telephone Laboratories' installations in four test plots at Gulfport, Miss., Orange Park, Fla., Chester, N. J., and Limon, Colo.[69, 75, 76] and the Forest Products Laboratory installations in Mississippi.[17] Bienfait and Hof, like Rennerfelt, have been using the standard European agar-block test in their plan for correlation of laboratory and field results. No report on the Holland tests has appeared since 1948.

Narayanamurti and his associates[82] in their first interim report on laboratory and field tests of creosotes of Indian origin present the results of some fifteen years work at the Forest Research Institute at Dehra Dun, indicating from still another quarter the compelling force that is

leading to the development of preservative evaluation methods to supplement or partly displace long and uncertain service tests. The authors present a mass of information on six different creosotes, on four creosote fractions, and on mixtures of the creosote with fuel oil of Persian origin. Sal (*Shorea robusta*) railway ties were used for the field trials. Many of the data are condensed into graphs that are small and difficult to read. The findings in general are favorable to the creosote-petroleum blends. The writer, on the basis of personal experience, is dubious about either the theoretical or practical significance, in experiments of the type reported, of the values given for standard deviations and standard errors. The scope of the work entitles it to more complete review than is practicable at this particular time and place.

Bell Telephone Laboratories are represented in a group carrying on comprehensive cooperative investigations of pedigreed creosotes on which four papers have already been published.[5, 12, 39, 93]

The results of outdoor tests of small stakes and fence posts are issued periodically by the Forest Products Laboratory at Madison, Wis.[15, 17, 18, 63] In this connection, the Proceedings of the American Wood-Preservers' Association are in the class of required reading. Additional references will be cited at appropriate points in the succeeding paragraphs.

Data will first be presented on some of the experience of Bell Telephone Laboratories and others with laboratory soil-block tests, with outdoor tests of small stakes, of pole-diameter posts, and with pole test lines in evaluating wood preservatives. Through analysis and discussion an attempt will be made to interpret the significance of the results obtained by the various evaluation procedures and to correlate the evidence. Emphasis will be placed naturally on creosote and pentachlorophenol because of their great importance to the Bell System pole plant. The writer intends to support his interpretations with experimental data wherever possible, reserving the privilege in some cases to make suggestions as to possible significance, even though complete technical proof may be lacking at present.

EVALUATION BY SOIL-BLOCK TESTS

General Procedures

Soil-block cultures have been described in a number of papers[95, 96, 52, 38, 39] since Leutritz presented his method in this JOURNAL in 1946.[70] Some of the following statements, therefore, will be repetition; but the intent is to outline the technique employed at Bell Telephone Laboratories as a base for later discussion.

The culture jars are wide mouth cylindrical 8-ounce bottles, provided with screw caps. The moisture content of the soil is predetermined on a representative sample, and enough distilled water is placed in the bottle so that when the soil is added its moisture content will be somewhat above 40 per cent by weight. The bottles are filled approximately half-full of screened field top soil — which means about 140 grams of an oven-dried sandy loam. The soil handles better if it is reasonably dry so that it can be poured through a suitable funnel; and putting the water in bottles before one puts in the soil results in a practically clean glass surface on the inside of the bottle above the soil level.

Two southern pine sapwood feeder blocks, measuring $1\frac{3}{8}$ inches in the direction of the grain by $\frac{3}{4}$ inch by approximately $\frac{5}{32}$ inch (35 x 20 x 4.0–4.5 mm) are placed carefully on the flattened soil surface, as shown in Fig. 1(a). The soil and feeder block setups are then sterilized for one-half hour at a pressure of 15 pounds per square inch, after which they are allowed to cool in the autoclave.

Inoculation is accomplished by carefully placing a piece of inoculum, cut from a fresh Petri dish culture of what may be called a standard test organism, at or near the middle of the feeder block surfaces. Under

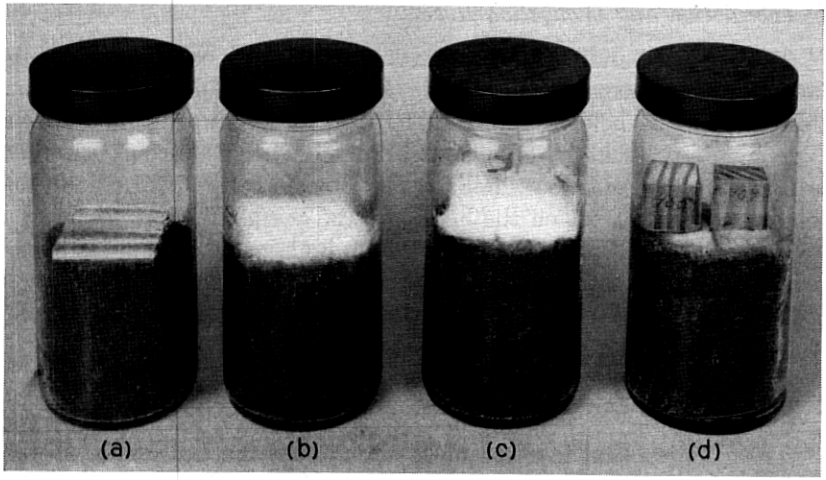

Fig 1—Four eight-ounce cylindrical bottles illustrating the soil-block cultures: (a) Bottle half-full of top soil, containing 40 per cent moisture on an oven-dry soil basis, with two flat feeder blocks of southern pine sapwood on top. (b) A sixteen-day old culture ready to receive the impregnated southern pine sapwood test blocks. (c) Two laboratory weathered test blocks from the same series treated to a below threshold concentration of pentachlorophenol 0.051 lb dry penta/cu ft, attacked by the test fungus, *Lenzites trabea*. (d) Two test blocks, laboratory weathered, treated to a retention of 0.194 lb dry penta/cu ft, near the threshold retention, showing resistance to fungus attack.

the temperature and humidity conditions of the incubation room, the growth of the fungus mycelium covers the feeder blocks in about two weeks and the fungus threads are then well started downward into the soil.

Treated test blocks are weathered and then conditioned under controlled temperature and humidity to approximate constant weight. They are then sterilized, along with untreated control blocks, in an autoclave for 15 minutes at 100°C, atmospheric pressure.

As a rule two treated blocks having approximately the same retention of preservative are placed together in a single test bottle. The incubation period is three months, in an incubation room held at a temperature of 80 ± 2°F and at a relative humidity of 70 ± 2 per cent. At the end of this period the cultures are taken down. This means that the blocks are removed from the bottles, brushed free of fungus mycelium, and weighed immediately. They are given a preliminary examination for decay evidence, and then reconditioned, under the same temperature and humidity conditions as before sterilization, to approximate constant weight. Fungus attack is determined by observation and by weight losses. The general setup of the cultures is illustrated in Fig. 1 (a–d).

Inoculation and Incubation Rooms

To facilitate handling the soil-block cultures, an inoculation room and an incubation room have been built (Fig. 2) at the Murray Hill Laboratories. Both are held at approximately the same temperature and relative humidity, that is, 80°F and 70 per cent. The inoculation room serves as a lock chamber, and passage from it to the incubation room has a negligible effect on the humidity and temperature of the incubation room. The latter is provided with an illuminated double plate glass window (Fig. 3), so that the interior can be exhibited without the necessity of entering the room. This window is fitted with a heavy roller shade, and the room ordinarily is kept dark.

Soil Characteristics and Moisture Content

The question that is asked most often about the cultures is whether a standard soil is used. European and American criticism has been definitely directed[78, 122] at the fact that the use of different soils might have so much effect upon the growth and the reaction of the test fungi in the cultures that quite different results would be obtained by investigators in different laboratories. This possibility is recognized; but the evidence to date seems to point to the general conclusion that perhaps the prin-

Fig. 2—Soil-block cultures on the shelves in the incubation room. The unpainted wood shelves come to equilibrium with the temperature and relative humidity and thus are a factor in keeping the conditions stable. The back edges of the shelves are set away from the wall to provide spaces for air circulation. The front edges of the shelves are provided with metal labeling strips.

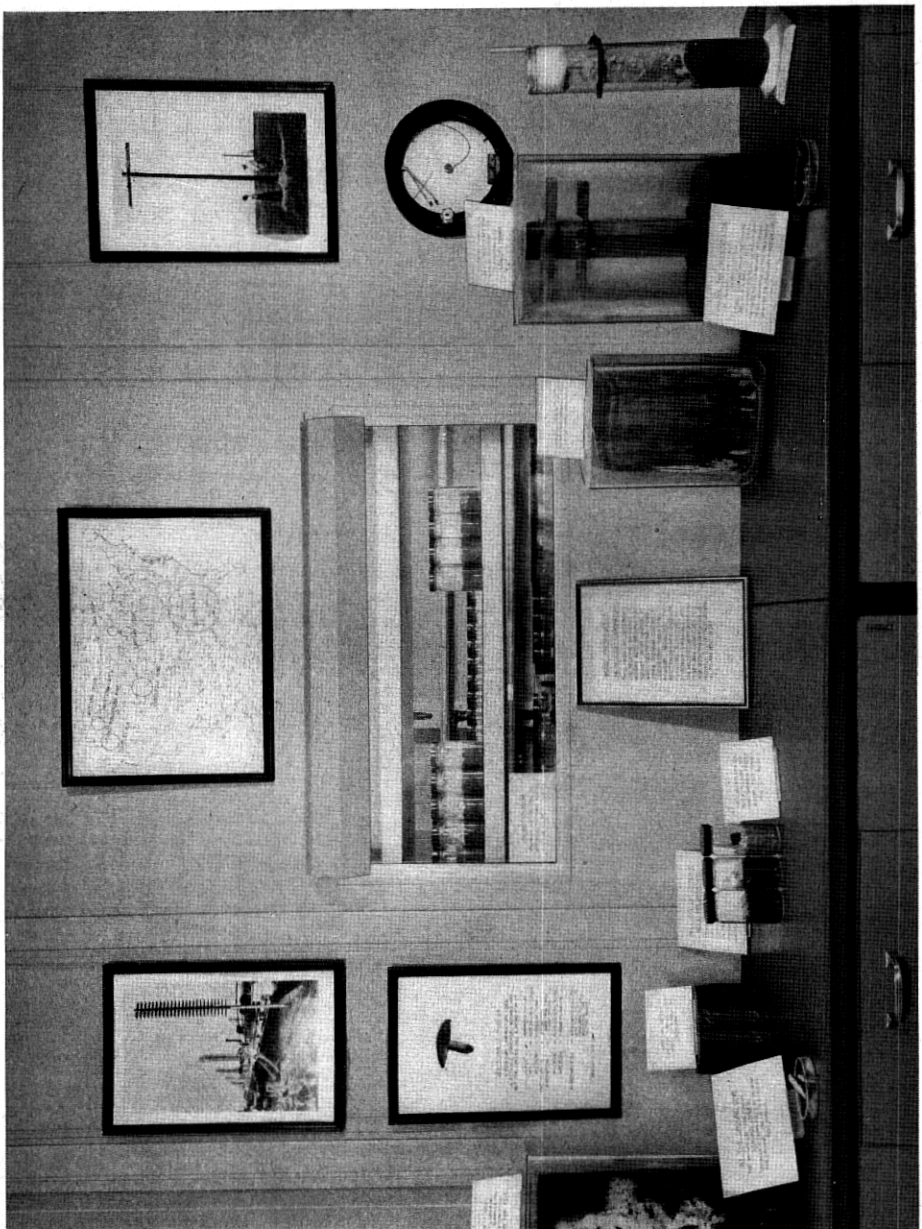

Fig. 3—The "Biossay Corner" at Bell Telephone Laboratories, Murray Hill. Centered in the illustration of the display

cipal and most important factor in the soil-block culture is the moisture holding capacity and content of the soil, rather than its nutrient function. If continued experimentation supports this conclusion it would not be necessary to limit the type of soils used except within rather broad limits. It also appears that the size and thickness of the feeder block now employed introduces enough wood into the culture bottle to mask any minor variations in the soil itself. The all important thing is to have enough water in the soil *throughout the test period* to keep the air above the soil essentially at 100 per cent humidity and the blocks at about fiber saturation — say about 27 per cent, oven-dry weight basis.

The soil in use at Bell Laboratories at present is obtained from a plot that has been set aside at the Chester (N. J.) Test Station. This plot has been fallow for twenty-five years. It supports a general grassy flora. The soil is a sandy loam with the following general description:

pH	4.9–5.0
Available magnesium	37.5 lb/acre
Available phosphorus	4.5 lb/acre
Available potassium	70.0 lb/acre
Organic matter	3.0 per cent

The cultures at the Forest Products Laboratory[41] have been made with a silt loam having a pH between 5.5 and 6.0. Bell Telephone Laboratories' tests have indicated the desirability of avoiding soils of either very sandy or very heavy clay types. The soil from the Chester Test Station described above is being used in all cultures, and there is a sufficient layer of top soil on the reserved plot to make parallel cultures for a good many years. Until such time as more definite and positive information on the effect of minor variations in the soil type are determined it is generally agreed that all of the comparative tests in any given series at least should be run on the same soil. Experimental work is now under way to determine the possible advantage of the addition of Krilium* to the soil in the culture bottles to maintain porosity and an even, high moisture holding capacity.

After the test blocks are placed in the culture bottles and during the course of the ninety-day incubation period the screw caps are left *loose*. The general technique followed in making up the soil cultures, as far as moisture is concerned, parallels that used at Madison. The moisture content is close to that recommended by Flerov and Popov,[48] namely 40–50 per cent of the weight of the soil plus the feeder block, with distilled water added during the test period, if necessary to maintain good

* An acrylonitrile product of the Monsanto Chemical Company.

growth of the test fungus. Breazzano[21] thoroughly saturated the sand base in his test cultures. Leutritz[70] and Harrow[51, 52] working with tightly closed culture jars found a 25 per cent level in the soil to be satisfactory.

Flerov and Popov state after special control tests "that replacement of (the) sand by soil had no effect on the results of the tests and only shortened their duration." Duncan[41] has found from her tests that variations in moisture content and soil type affect the degree of fungus attack only and that they do not change the determination of the treatment threshold concentration in any given set of test blocks.

Even-Aged Cultures

The thickness of the feeder blocks has been gradually increased to about $\frac{3}{16}$ inch, or between 4 and 4.5 millimeters. This provides food for the fungus to establish itself in the bottle. The inoculum pieces are roughly 1 cm square, cut from Petri dish cultures that are 15 ± 1 days old. The planting routine is carefully scheduled so that even-aged soil-block cultures — 13–15 days — are ready to receive the treated blocks when the latter are ready to be placed in test. This principle of using even-aged cultures has been stressed by the Madison investigators, and it is considered to be a factor of major importance in the proper culture technique.

Standard Test Organisms

There have been continuous discussions since the beginning of laboratory tests in Europe, as well as in this country, about what test organisms should be used. Conforming to the experience and practice at Madison the following three numbered strains of wood-destroying fungi are recognized as the "standard" strains for the testing of oil type preservatives in coniferous wood:

Lentinus lepideus, Madison 534
Lenzites trabea, Madison 617
Poria monticola, Madison 698

All three are known to be associated with the decay of treated timber. *Lentinus lepideus* is particularly tolerant of creosote,[41, 66] and relatively susceptible to pentachlorophenol. It has frequently been isolated from decaying creosoted southern pine poles and other creosoted coniferous timber in contact with the ground. *Lenzites trabea* is generally an "above ground" fungus. It also has been isolated from decaying creosoted timber; and it is the principal cause of "shell rot" in the above ground sap-

wood of western red cedar poles. It is relatively susceptible to creosote and quite tolerant of pentachlorophenol in the block tests. *Poria monticola* is relatively tolerant of pentachlorophenol and of copper compounds under laboratory test conditions, and relatively susceptible to creosote. It is of special interest also because it may be identical with some of the fungi tested in Europe under the name of *Poria vaporaria*, and thus its use may facilitate comparisons of a sort with results obtained by other investigators. For instance, information has reached the Division of Forest Pathology at Madison, from Findlay at the Princes Risborough laboratory in England, that Harrow's *Poria vaporaria*[51] is the same as Liese's,[71] and that it has been identified as a strain of *Poria monticola* by Miss M. Nobles of Canada.

Within the last few years another fungus, characterized by the formation of conspicuous saffron yellow strands, has been found associated with decayed specimens of creosoted pine poles.[60] The writer has seen the tell-tale strands in old cull dumps only. It has been identified as *Poria radiculosa*. Whether it is truly a primary attacker or a secondary organism is not yet clear. Soil-block tests are under way at Madison to determine its significance as a possible species to supplement *Lentinus lepideus* in the evaluation of creosote.

In connection with the use of the three numbered "standard" strains listed above, there may always be some reasonable doubt as to whether the cultures employed in different laboratories have the same virulence. To answer this question precisely involves a lot of careful biological check testing, and such tests are already being made in the Division of Forest Pathology at the Plant Industry Station, Beltsville, Md. It is assumed for the time being that the numbered strains are virulent and satisfactory test organisms for such preservatives as creosotes and pentachlorophenol-petroleum solutions.

The Scope of the Soil-Block Evaluation Test

For a complete understanding of the scope of the soil-block evaluation test it is necessary to consider this test as having two functions. The first function involves the use of the soil-block test per se (without weathering) to measure the reaction of the test organisms to various quantities of a given preservative, and to compare these reactions against different preservatives. In this function the test has been used in lieu of the agar Petri dish test,[94] and is considered to be much more satisfactory as a screening test by workers at the Laboratories. It has been employed at Madison for testing the natural durability of wood, plywood, fiber board, etc.

The second — and more important — function of the soil-block evaluation is that incorporating a weathering or aging procedure. This puts the test in the more practical category of testing the wood preservative properties, viz., *toxicity and permanence*. In this respect it has something in common with the German Standard DIN DVM 2176[33] for short time mycological testing of wood preservatives by the block method and covers a broad concept from the treating through partial aging of the blocks. Separate German standards cover procedures for leaching[34] and volatility tests,[78] (p. 264 and Fig. 42 of Reference 78).

The Laboratories' concept of the scope of the soil-block test including the weathering procedure is definite. It must be appreciated that this method which employs manipulative procedures involving both toxicity and permanency yields significant data in a period of only a few months. The data derived must be correlated subsequently, of course, with the data covering the results of tests of ¾ inch stakes six to seven years later, with the data on test posts some ten to fifteen years later, and with data on poles in line some twenty-five years later.

It is only being realistic to say that the Bell System cannot afford to wait for physical life tests of new materials under natural conditions of exposure before recommending them where techniques and extensive experience permit acceptable estimates to be made from accelerated evaluation in relatively short periods of time.

Preparation of the Test Blocks — Manufacture

Southern pine sapwood, free from stain or decay, is used as a base material for the test blocks. The process of manufacture begins at the saw mill, where freshly cut logs selected for the purpose are carefully sawed into one inch boards. Straight grain material is most desirable. The boards are kiln-dried immediately and shipped as soon as practicable to the Laboratories. It has been the practice to store the boards in a steam heated basement where the humidity is low enough to hold the moisture content of the boards down to about 5 to 7 per cent. The sapwood only is used, which means that any small heartwood portions must be marked out for rejection. The blocks are accurately cut ¾-inch cubes. A ⅛-inch hole is drilled through the center of the tangential surfaces of each block. It has been found that drilling the hole through the transverse surface, which was the early practice with Waterman, Leutritz and Hill[126] is a difficult procedure; and sometimes it amounts to an impossibility because the harder summerwood layers deflect and break the drills. In any event, drilling through the tangential surface opens up more paths for longitudinal absorption and penetration, as well as

evaporation, of the preservative. The feeder blocks and the ¾-inch test blocks are usually made at the same time, from parts of the same boards. The blocks are kept clean, and reserve stocks are carefully stored in a dry room. The blocks in storage reach an approximate moisture equilibrium of 6 to 7 per cent, on an oven-dry weight basis.

Test Block Selection for Density

Random samples of the blocks are weighed and segregated into groups at 0.1-gram intervals, 4.10 to 4.19 grams and 4.20 to 4.29 grams, for example. Blocks of practically equal weight can be chosen for the comparison within any given series of different concentrations of a preservative. The weighed groups of blocks are kept in convenient lot sizes in a dry place. Since the blocks are accurately cut the segregation by weight amounts to a segregation by density.

It has not been found necessary or practicable to separate the blocks into groups with the same numbers of annual rings, although in some instances an approximation to this ideal has been attempted. Furthermore, it has not been found practicable to separate the blocks on the basis of the direction in which the rings run across their transverse faces. From experience to date it does not appear that either ring direction or ring count has any material effect upon the behavior of the blocks in the culture as far as determination of preservative thresholds are concerned; but experiments are under way at Madison to determine the effect of density on the relative degree of decay. Inasmuch as all of the blocks are placed in culture with the transverse surface down, so that alternate spring- and summerwood layers are exposed directly to the test organism, the latter can enter either springwood or summerwood in accordance with its ability to resist the concentration of the preservative present in these two parts of the annual ring.

Average Block Volume

The average volume of the oven-dried blocks, determined from random samples by a mercury displacement technique, was found to be 6.484 cc, with a standard deviation of 0.0831. This represents a coefficient of variability of 1.28 per cent. The minimum-maximum range of volumes ran from 5.93 cc to 6.87 cc. These extreme deviations are normally detected in handling the blocks and both high and low volume blocks are rejected. The variation in density and volume of the test blocks will be discussed separately in the paragraphs dealing with the treatment of the blocks.

Treatment of the Test Blocks

The blocks selected for any given treatment are numbered serially with India ink on the upper half of one of the radial faces. All blocks are then oven-dried for 24 hours at 105°C to an approximate constant weight. The blocks are removed from the oven, and placed in a desiccator over P_2O_5. Check tests of blocks held under these conditions show that they do not change weight by more than one hundredth gram within the period they are held for weighing. The cooled, oven-dried blocks are weighed to the nearest hundredth gram.

The weighed blocks are placed in beakers and arranged with a tangential face down so that the transverse surfaces do not touch and the holes are vertical. This refinement in placing the blocks may not be necessary to obtain satisfactory absorption, but the procedure has worked out well, and it has been followed consistently. For any given concentration a sufficient amount of creosote, for example, and toluene are combined by weight to leave in the blocks, after treatment, the desired retention of preservative. Experience has indicated the concentration required, which depends to a certain extent upon the type of vacuum equipment that is available as well as upon the density of the blocks to be treated and the nature of the treating solution. Actually the process of treatment is simple. The beaker containing a given lot of weighed oven-dried blocks is placed within a bell jar and subjected to a vacuum of 3 to 4 millimeters of mercury. When this vacuum has been reached the line to the vacuum pump is shut off, and the preservative is run into the beaker from a separatory funnel[126] fitted into a rubber stopper on the top of the vacuum chamber, the blocks being weighted down below the level of the preservative.

The absorption and distribution of the oil within the blocks seems to take place very rapidly. Generally speaking, the beaker containing the blocks and the preservative are removed from the vacuum chamber as soon as practicable to permit continuing the treatment of another group of blocks. However, the blocks are usually held in the preservative solution for an hour or two, which apparently is long enough to bring about essentially complete saturation. When all the treatments for a given group of concentrations have been finished (a) the treated blocks are wiped to remove the excess oil, and (b) they are weighed immediately to 0.01 gram. The retentions of creosote or pentachlorophenol, for example, are determined on a gain in weight basis by calculations from the amount of material picked up during the treatment and the concentration of the preservative in the treating solution.

Retention Gradients

In the hope of setting at rest some of the doubts and criticisms that have arisen about the accuracy of the treatments and the retention of preservative in the blocks, some results of the treatment process just described will be presented in rather elaborate detail.

The success of the treatments depends upon experience, as indicated previously, with the particular type of vacuum equipment available. However, once the level of performance to be expected from the vacuum equipment is learned, one has to take into account the variations that are introduced by the density of the blocks and by the specific gravity of the treating solution. It is the intent in all of the treatments at the Laboratories to arrive at a series of gradient retentions, on as accurate a line as possible, and as nearly as possible equal gradients, so that the fairest comparison can be made of the behavior of the different preservatives. Fig. 4 shows the gradient obtained by plotting the data shown in Table I for retention of creosote and retention of pentachlorophenol solution over the concentration of these preservatives in the treating solution. The analysis of the creosote — BTL 5340 — is shown in Table II. The slopes of the two gradients are considered to be about as close as the experimental procedure will permit. Fig. 5 shows the gradient

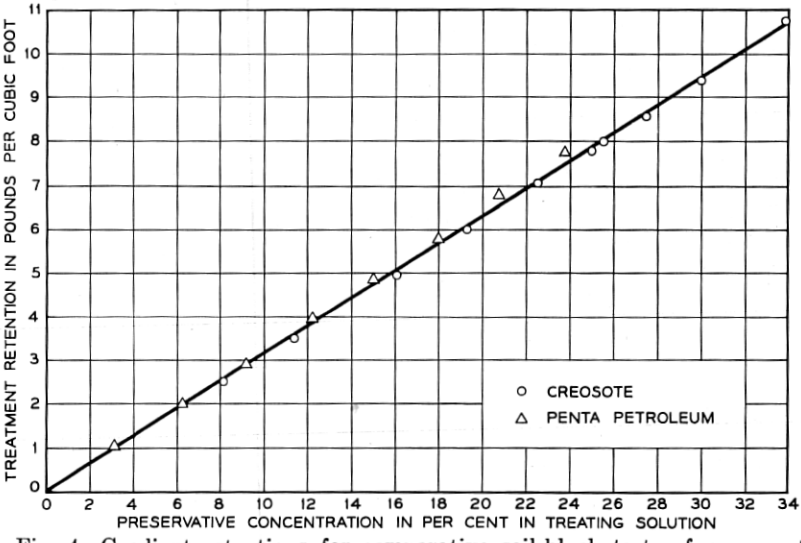

Fig. 4—Gradient retentions for comparative soil-block tests of a creosote (BTL No. 5340) and a penta-petroleum solution (4.92 per cent pentachlorophenol in Standard Oil Company of New Jersey No. 2105 Process Oil). The preservatives were used in toluene solution.

TABLE I — FULL-CELL TREATMENT

Soil block tests with creosote (No. 5340, see Table II) and with pentachlorophenol-petroleum (4.92 per cent in Standard of New Jersey No. 2105 Process oil) in toluene; absorption and retention of preservative data for parallel comparative tests.

Charge No.	Average Oven-dry		n	Average Absorption*		C†	Average retention						
							Whole preservative			Creosote		Penta	
							Creosote	Penta					
	Weight	Density		Total	Per cc			Sol.	Penta	Total	Per cc	Total	Per cc
	(gms)			(gms)		(per cent)	(lb/cu ft)			(gms)		(gms)	
9	3.77	.584	30	3.28	.509	8.18	2.49	—	—	.28	.043	—	—
10	3.78	.586	30	3.40	.527	11.45	3.48	—	—	.39	.061	—	—
6	3.80	.589	30	3.44	.533	16.11	4.96	—	—	.55	.085	—	—
5	3.83	.594	30	3.46	.536	19.33	5.99	—	—	.67	.104	—	—
8	3.80	.589	30	3.51	.544	22.55	7.08	—	—	.79	.123	—	—
1	3.74	.580	30	3.49	.541	25.00	7.81	—	—	.87	.135	—	—
4	3.77	.584	30	3.51	.544	25.55	8.03	—	—	.90	.140	—	—
3	3.80	.589	30	3.49	.541	27.50	8.58	—	—	.96	.149	—	—
2	3.78	.586	30	3.50	.543	30.00	9.40	—	—	1.05	.163	—	—
11	3.70	.574	30	3.33	.516	3.25		1.05	.052	—	—	.005	.0008
12	3.79	.588	30	3.33	.516	6.25		2.01	.099	—	—	.010	.0016
13	3.79	.588	30	3.29	.510	9.25		2.95	.145	—	—	.015	.0023
14	3.75	.581	30	3.31	.513	12.25		3.96	.193	—	—	.020	.0031
15	3.71	.575	30	3.34	.518	15.00		4.84	.238	—	—	.025	.0039
16	3.70	.574	30	3.34	.518	18.00		5.81	.286	—	—	.030	.0047
17	3.72	.577	30	3.38	.524	23.75		6.79	.334	—	—	.035	.0054
18	3.73	.578	30	3.38	.524	23.75		7.77	.382	—	—	.040	.0062

* Absorption is the total amount of the treating solution picked up at treatment, that is, the gain in weight, including both preservative and the toluene carrier.

† C is the concentration of the preservative, e.g., creosote or penta petroleum, in the treating solution, in grams per 100 ml.

for pentachlorophenol alone, without regard to the petroleum carrier, also plotted from data in Table I. The scale on the abscissa represents the concentration of either the creosote or the pentachlorophenol solution. The ordinate represents pounds per cubic foot retained by the blocks, calculated from the pickup during treatment and from the concentration of the creosote or pentachlorophenol in the treating solution.

The Amount of Preservative in the Blocks

The use of these gradient concentrations is a continuation of the procedure worked out in the earlier stages[39, 41] of the Madison tests.

TABLE II — ANALYSES OF CREOSOTE BTL No. 5340, WATER-FREE BASIS

	1. Fall, 1946	2. Spring, 1952*
Specific gravity 38/15.5°C	1.088	1.102
Distillation, per cent, cumulative to 210°C	0.00	0.00
210–235	0.80	0.00
235–270	12.87	13.59
270–300	42.12	
300–315	54.30	52.03
315–355	79.10	78.05
Residue above 355°C	20.90	21.64
Total	100.00	99.69
Sulph. res., gm/100 ml	0.51	0.59
Tar acids, gm/100 ml	4.10	4.44
Benzol insol., per cent	0.07	0.59
Specific gravity (38°C) 235–315°C		1.053
315–355°C		1.118

* Average of 2 analyses.

It should be noted that the retentions are calculated as averages for the respective charges. Attention is called to the small quantity of preservative material involved. Even in calculated retentions of creosote, for example, 9.40 pounds per cubic foot (Table I), the retention means 1.05 grams in the whole block, or 0.163 grams *in each cc of block volume*. In

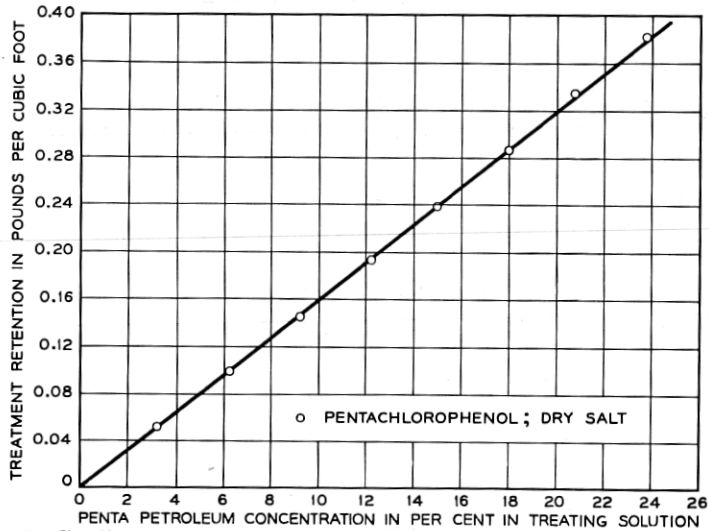

Fig. 5—Gradient retention of pentachlorophenol, calculated for the material alone, without the oil carrier. See Fig. 4.

the highest retention employed for the penta petroleum solution the calculated net retention averaged 7.77 pounds of the penta solution per cubic foot, or 0.382 pounds of pentachlorophenol per cubic foot; and these figures represent, respectively, 40 milligrams of pentachlorophenol in the average block, or 6.2 milligrams *per cc of block volume*. Exact data on treatment are discussed in the following paragraphs. The use of carefully calculated gradient retentions in each case makes it possible to detect any wide variation in the normal behavior of the blocks either with respect to pickup during treatment or in the reaction of the test fungus to the preservative.

Data are included in Table I on average oven dry weight of the blocks,

TABLE III — FULL-CELL TREATMENTS

Soil block tests; treating solution components, per cent by weight. (See Table I).

Charge No.	Penta-petroleum	Creosote	Toluene
19	4.65	4.65	90.70
20	10.50	10.50	79.00
21	15.00	15.00	70.00
71	—	23.00	77.00
72	—	23.00	77.00
73	—	24.75	75.25
74	—	25.50	74.50
75	—	30.00	70.00
76	—	32.00	68.00

average density on an oven dry weight and volume basis, average pickup of creosote or penta solution in pounds per cubic foot and in grams per block, the concentration of the preservative materials in the toluene preservative solution, and the average grams of preservative per cc of block volume. All of the blocks in these two groups of charges were chosen within a narrow density range.

Block Density and Preservative Absorption

It will be noted that in the charges in Table I there is a general trend upward in the grams absorbed at treatment per cc of block volume. as the specific gravity of the treating solution increases. This is, of course, one of the results of increasing the concentration of creosote, for example; and furthermore, as would be expected, the higher gravity solutions represented by the creosote treatments show a higher pickup in terms of total grams as well as in grams per cc. The make-up of the

TABLE IV — FULL-CELL TREATMENT WITH PENTA-PETROLEUM-CREOSOTE IN TOLUENE

Relation of variable block density to absorption of treating solution, grams per cc of block volume; density and volume on oven-dry basis. (See Tables V and VI).

Charge 19 Av. retention 2.99 lb/cu ft		Charge 20 Av. retention 6.69 lb/cu ft		Charge 21 Av. retention 9.59 lb/cu ft	
Density oven-dry	Absorption gms/cc of block vol.	Density oven-dry	Absorption gms/cc of block vol.	Density oven-dry	Absorption gms/cc of block vol.
.440	.599	.468	.589	.484	.581
.459	.583	.483	.584	.489	.576
.459	.586	.533	.549	.505	.564
.475	.575	.543	.542	.520	.550
.507	.561	.544	.527	.529	.555
.517	.545	.557	.537	.541	.541
.533	.544	.564	.529	.549	.538
.535	.525	.567	.530	.554	.532
.543	.530	.569	.569	.555	.526
.543	.537	.604	.510	.573	.538
.571	.508	.606	.511	.582	.528
.574	.524	.611	.504	.582	.530
.608	.488	.616	.499	.592	.517
.609	.495	.618	.491	.602	.512
.611	.500	.618	.497	.610	.497
.611	.533	.624	.503	.612	.507
.623	.483	.625	.493	.614	.502
.624	.493	.627	.492	.614	.508
.637	.476	.628	.488	.615	.540
.638	.477	.644	.481	.617	.495
.641	.474	.645	.488	.617	.504
.645	.478	.646	.482	.627	.495
.646	.473	.651	.482	.633	.497
.657	.462	.662	.433	.647	.488
.660	.453	.674	.461	.650	.487
.663	.466	.676	.465	.664	.416
.663	.468	.674	.452	.672	.473
.668	.462	.690	.456	.684	.470
.691	.447	.703	.445	.687	.464
.695	.450	.738	.427	.723	.442
Average					
.592	.507	.614	.501	.598	.512
Standard deviation					
.0726	.0435	.0616	.0405	.0591	.0370
Coefficient of variability—per cent					
12.26	8.58	10.03	8.08	9.88	7.23

treating solutions for Charges 19–21 and 71–76, inclusive, are shown in Table III. The relation of the density of the blocks to the pickup, i.e., the absorption at time of treatment, is illustrated in Tables IV, V and VI and in Fig. 6. The data have been split up to facilitate reference.

Table IV shows the complete data for oven dry density and for absorption in grams per cc of block volume for Charges 19, 20 and 21, with values for the average, for the standard deviation, and for the coefficient of variability. The pickup varies inversely as the density, which is to be expected when random blocks instead of selected density blocks are employed. The coefficient of variability in the density figures is evidently greater than it is in the pickup figures; and a lower figure for the latter is related to a lower figure for the former.

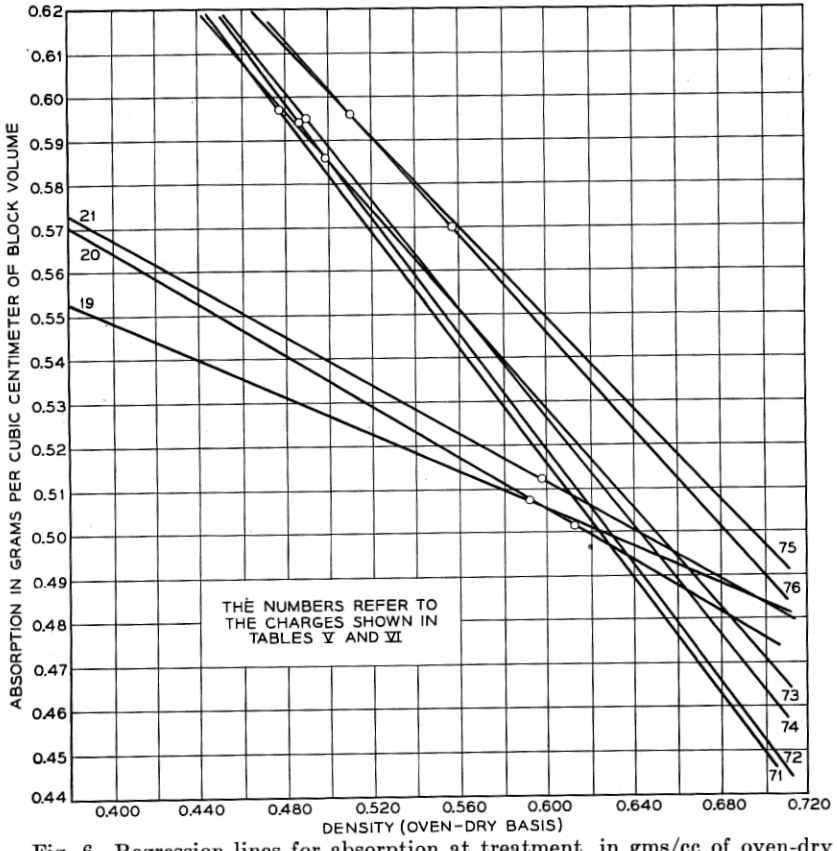

Fig. 6—Regression lines for absorption at treatment, in gms/cc of oven-dry block volume, on oven-dry density of the ¾-inch cube test blocks.

These same values for these three charges, 19, 20 and 21, and similar values for charges 71–76, inclusive, are incorporated along with average and range of retention data in Table V, and with statistical data for regression lines in Table VI. The data serve to illustrate the degree of variability in treatment results that may occur when random blocks are used. The best indices of these variations are in the columns showing

TABLE V — RETENTION DATA FOR FULL-CELL TREATMENT

Soil block tests; average and range of preservative retention, by charges, at treatment.

Charge No.	n	Penta petroleum lb/cu ft	Pentachlorophenol						Creosote					
			lb/cu ft			gms			lb/cu ft			gms		
			Av.	Min.	Max.	Av.	Min.	Max.	Av.	Min.	Max.	Av.	Min.	Max.
19	30	1.51	.093	.064	.097	.008	.007	.010	1.48	1.29	1.97	0.154	0.134	0.205
20	30	3.35	.163	.136	.219	.017	.014	.023	3.34	2.77	4.44	0.348	0.288	0.462
21	30	4.79	.237	.192	.269	.024	.020	.028	4.80	3.90	5.46	0.500	0.407	0.569
71	30	—	—	—	—	—	—	—	8.58	7.65	9.04	0.892	0.817	0.941
72	30	—	—	—	—	—	—	—	8.52	7.67	9.20	0.886	0.798	0.957
73	30	—	—	—	—	—	—	—	9.06	8.43	9.57	0.943	0.876	1.025
74	30	—	—	—	—	—	—	—	9.46	8.61	10.27	0.984	0.895	1.068
75	30	—	—	—	—	—	—	—	11.12	10.07	12.06	1.158	1.047	1.254
76	30	—	—	—	—	—	—	—	11.37	11.05	11.94	1.184	1.149	1.242

TABLE VI — FULL-CELL TREATMENT

Soil-block tests with (a) penta-petroleum creosote, and with (b) creosote, in toluene; relation of variable block density to absorption of treating solution.

Charge No.	n	Av. retention lb/cu ft		Density oven-dry			Absorption gms/cc vol.			Correl. coeff. r	Regression of absorption Y on density X
		Creosote	Penta-petro Creosote	Av.	σ*	c.v.†	Av.	σ*	c.v.†		
19	30	—	2.99	.592	.0726	12.26	.507	.0435	8.58	−.3444	.6286−.2066X
20	30	—	6.69	.614	.0616	10.03	.501	.0405	8.08	−.4282	.6736−.2820X
21	30	—	9.59	.598	.0591	9.88	.512	.0370	7.23	−.4718	.6893−.2957X
71	30	8.58	—	.477	.0428	8.97	.597	.0293	4.91	−.9589	.9109−.6580X
72	30	8.52	—	.486	.0457	9.40	.594	.0326	5.49	−.9233	.9143−.6589X
73	30	9.06	—	.499	.0456	9.14	.586	.0275	4.69	−.9426	.8701−.5692X
74	30	9.46	—	.489	.0402	8.22	.595	.0279	4.69	−.8916	.8969−.6181X
75	30	11.12	—	.510	.0538	10.55	.596	.0302	5.07	−.9206	.8595−.5170X
76	30	11.37	—	.557	.0095	1.71	.570	.0114	2.00	−.4583	.8781−.5543X

* σ = standard deviation.

† c.v. = coefficient of variability, per cent.

the average and total spread in grams of preservative absorbed. The effect of selection for density is shown clearly — within the particular treatment groups — by the figures for charges 75 and 76. In the latter the use of selected even density blocks reduced the spread to below half of that in charge 75, and reduced the coefficient of variability by two-thirds.

Regression lines for pickup in grams per cc of block volume on oven dry density are shown for the two groups of charges in Fig. 6. The flatter slope of the lines for charges 19, 20 and 21 seems to reflect the difference in specific gravity and viscosity of the treating solutions. Higher densities have greater effect on absorption of higher gravity solutions. The fact remains that there is considerable uncertainty as to the significance of strict selection of the blocks for density if one uses a series of closely spaced gradient retentions.

Data for 8-pound charges for comparative soil-block tests of two of the cooperative creosotes, Nos. 7 and 9, a low residue domestic oil, low in tar acids and naphthalene, and a British vertical retort tar creosote, respectively, are condensed in Table VII. The differences in the treatment results are not considered to be significant.

Weathering

The blocks remain on the racks on the laboratory tables for about one week, which is long enough to permit the evaporation of most if not all of the toluene. Experiments have shown that when blocks are treated with toluene alone the toluene is all lost, on a weight basis, within 24 hours. When treating solutions of creosote in toluene are used the evaporation rate, also determined by weight loss, is slower; but it is believed that all of the toluene is gone before the blocks are ready for test. In any event, after the above-mentioned preliminary drying period the blocks are handled in a manner that differs somewhat from the procedure that

TABLE VII — RETENTION FOR FULL-CELL 8-POUND TREATMENT DATA

For parallel comparative soil-block tests of cooperative creosotes No. 7 and 9 against *Lentinus lepideus*, Mad. 534: toluene-creosote treating solution.

Creosote No.	n	Density oven-dry	Creosote lb/cu ft				Creosote grams				Standard deviation	Coefficient of variability, per cent
			Av.	Min.	Max.	Spread	Av.	Min.	Max.	Spread		
7	30	.606	8.11	7.75	8.51	0.76	.848	.809	.889	.080	.0201	2.37
9	30	.601	7.90	7.49	8.56	1.09	.825	.780	.893	.113	.0237	2.87

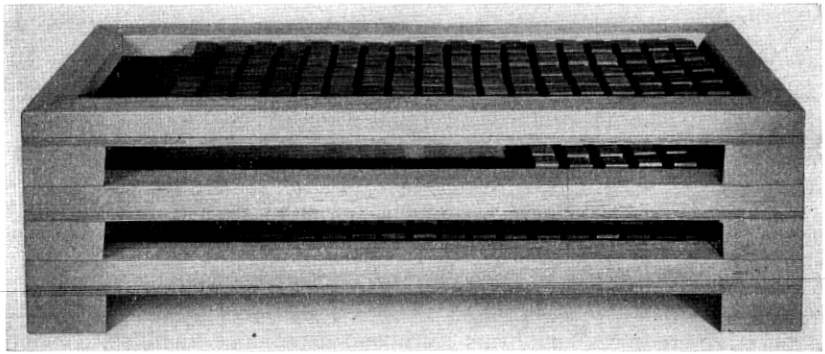

Fig. 7—Three unpainted stacked handling trays. The trays have plastic screen bottoms on which the blocks can be arranged with free air space all around, to promote even drying conditions.

has been followed up to this time at Madison. The principal difference is the omission of any tests of unweathered blocks. Instead, the emphasis is placed on the development of a weathering or aging cycle that will bring about total overall preservative losses like those that occur in ¾-inch stake specimens or in pole-diameter posts in the Gulfport test plot. The character and extent of such losses will be discussed later.

Two systems of weathering have been employed up to the time of this writing. The first consists in soaking the blocks over the week in water that is changed morning and night and drying them at room temperature over the weekend, in accordance with German standard for leaching;[34] and the second is the same method that is employed in the Madison tests[39, 41] in which the blocks are strung on nylon thread, separated by glass beads, and exposed to outdoor weathering under natural conditions for sixty days. The duration of this outdoor test has been limited to sixty days, regardless of the season or month of the year. The effectiveness of the climatic conditions at the Chester Field Station during the period from October, 1951, to April 1, 1952, compared with the roof weathering as conducted at Madison with the same creosote sample remains to be seen.

As for the German standard leaching procedure,[34] experience up to April 1, 1952, indicates that the method does not result in the removal of creosote, for example, in the same degree or manner as preservative materials of this type are removed by outdoor weathering conditions. It definitely is not comparable with the latter in its effect. The German leaching procedure simply uses too much water and not enough air and heat; and Bavendam[9] quotes Falck as saying that creosote is insoluble in water and that it cannot be *washed* out of wood. The failure of the

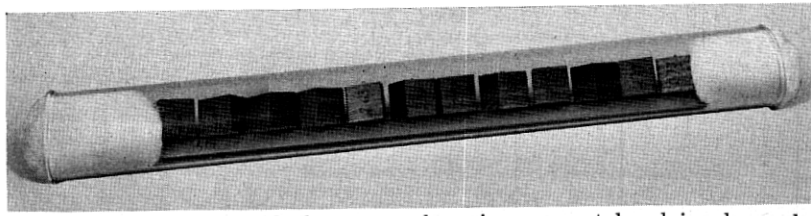

Fig. 8—Creosoted test blocks, arranged on pins on a metal rack in a large glass tube to facilitate handling during sterilization and the subsequent operation of planting in the soil cultures.

leached creosoted blocks to decay in the reported soil-block tests on the cooperative creosotes[39] confirms European experience. Schulze, Theden and Starfinger[56 (1)] (p. 15, Tab. 13 of Reference 54) indicate that there is little if any reduction in the preservative value of creosote as a result of their standard leaching tests. Therefore, in order to accelerate the weathering process new techniques are being worked out at the Laboratories in which the wet cycle is shortened (Cf. Rhodes et al,[50, 89]) and in which, without the use of a wheel, controlled artificial heat is used to speed up evaporation during the rest of the cycle.

Conditioning

Convenient unpainted wood trays (Fig. 7) with plastic screen bottoms are used for handling the blocks in groups at any time during their processing schedule. At the end of the weathering cycle, indoor or outdoor, the blocks are arranged on such trays and conditioned to an approximate constant weight and about 12 per cent moisture content on shelves in the 80°F and 70 per cent relative humidity of the incubation room. Weights before test, to the nearest 0.01 gram, are taken at the end of the conditioning period. The relative amounts of wood, water and creosote in the blocks are determined by weight from test blocks, and by weight and extraction from control blocks after sterilization.

Sterilizing

The test blocks are arranged for sterilization on metal racks in large glass tubes (Fig. 8). The autoclave temperature is held at 100°C for 15 minutes.

Flow Chart for the Bioassay Test

The various steps in the whole evaluation procedure are indicated in the flow chart shown in Fig. 9. Rhodes[89] used a similar idea to illustrate his procedure.

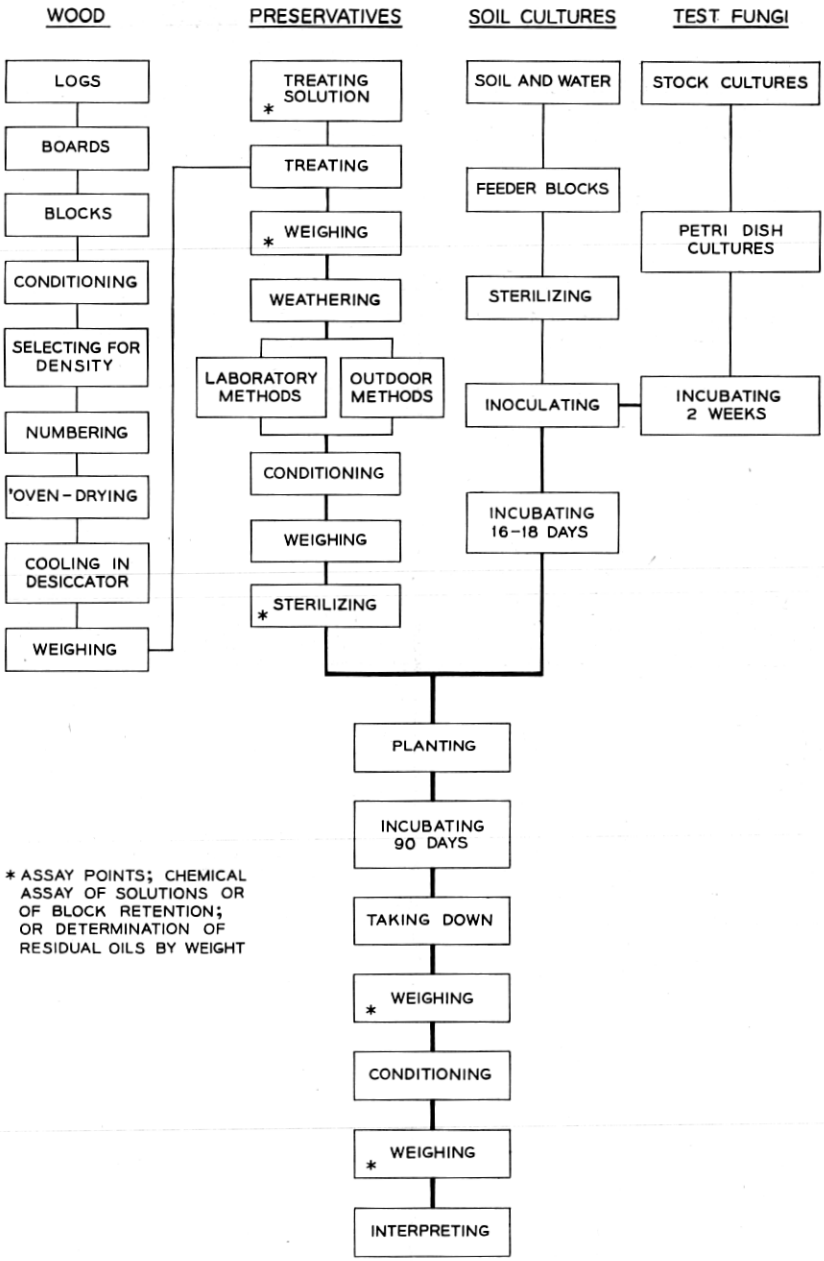

Fig. 9—Flow chart of the laboratory biossay test procedure. Steps in the overall processing must be carefully scheduled to provide adequate time for manipulation and to assure the requisite quantity of even-aged cultures at the time the treated and weathered test blocks are ready to go into test.

Some Madison Test Results

Inasmuch as results from recent Bell Laboratories' bioassay tests will not be immediately available, data from the Madison experiments[41] are used in order to illustrate the results that one may hope to secure from

TABLE VIII — BIOASSAY BY SOIL-BLOCK TESTS ON OUTDOOR WEATHERED BLOCKS

The relation of retention at treatment to block weight loss; Madison data.

Preservative†											
6			7			8			11		
n	R*	Weight loss per cent	n	R	Weight loss per cent	n	R	Weight loss per cent	n	R	Weight loss per cent
Test fungus, Lentinus lepideus, *Mad. 534*											
6	17.3	1.83	6	17.0	1.77	6	17.4	1.60	6	15.2	2.22
6	14.4	1.94	6	14.1	1.82	6	14.7	1.58	6	14.0	2.22
6	11.5	1.62	6	11.7	1.73	6	11.9	1.57	6	11.7	2.17
6	8.9	2.33	6	8.6	2.92	6	8.9	3.31	6	9.0	1.80
7	6.5	5.73	6	6.2	8.96	6	6.1	7.18	6	6.2	2.57
5	4.3	9.89	6	4.4	11.73	6	4.6	10.61	6	4.2	9.93
6	3.1	13.07	7	3.1	15.77	6	3.1	14.95	6	3.1	15.79
6	2.3	15.44	5	2.3	19.19	6	2.3	16.28	6	2.3	16.84

Preservative								
A			B			E		
n	R	Weight loss per cent	n	R	Weight loss per cent	n	R	Weight loss per cent
Test fungus, Lentinus lepideus, *Mad. 534*								
2	11.70	2.70	2	12.25	3.10	2	11.15	2.55
1	10.10	2.90	2	10.70	2.40	1	9.50	2.60
4	7.20	3.94	3	7.80	2.63	4	8.38	2.55
3	5.43	7.96	4	4.88	2.71	4	5.53	2.56
4	3.60	15.20	3	2.97	3.10	3	3.70	2.63
10	2.15	22.18	3	2.13	3.49	3	2.76	6.04
6	1.00	23.84	4	1.55	6.69	4	2.05	9.49
5	.58	17.35	3	.97	12.34	4	1.50	16.53
			3	.70	25.53	10	.77	29.70
			8	.40	35.43			

TABLE VIII—*Continued*

Test fungus, Lenzites trabea, *Mad. 617*

Preservative								
A			B			E		
3	9.14	2.57	2	12.35	2.80	3	10.93	2.70
4	7.58	2.42	2	10.30	2.60	3	9.17	2.40
3	5.53	2.73	3	7.80	2.80	2	6.95	2.60
2	3.70	2.81	4	4.95	3.24	3	5.30	2.49
3	3.20	3.97	3	3.33	14.21	4	3.95	3.14
6	2.17	8.57	11	1.51	35.76	3	2.47	8.00
7	1.14	38.96	10	.46	46.63	4	1.85	10.79
7	.60	55.44				6	1.08	52.90
						7	.64	55.06

* R = retention at treatment in lb/cu ft.
† Preservatives 6, 7, 8 and 11 are the numbered coop. creosotes (12).
A = BTL 5340 creosote,
B = 5 per cent penta in petroleum,
E = 50/50 by volume mixture of A and B.
All preservatives were applied in a toluene solution. The heavy lines represent approximate threshold levels.

carefully following the soil-block technique. The data, representing the writer's interpretation of the relation between average weight loss and average treatment retention, are shown in Table VIII and represented by the graphs in Figs. 10, 11 and 12. The preservatives in Figs. 11 and 12 labeled (A), (B) and (E) on the graphs, were respectively a domestic creosote (BTL No. 5340, Table II); a 5 per cent solution of pentachlorophenol in Standard Oil Company of New Jersey No. 2105 Process oil, and a 50/50 by volume mixture of these two.[41] Fig. 11 represents the results obtained with cooperative creosotes Nos. 6, 7, 8 and 11[39] and with BTL No. 5340.

In all three figures there are weight losses that can evidently be classed as operational losses, that is, losses by evaporation of some of the volatile materials still remaining in the blocks during the time they were in test and in the subsequent conditioning period.[41] The general areas in which the amount of preservative with which the blocks were treated failed to protect the wood against attack by the different fungi are shown by the rise in the weight loss lines. Perhaps the most interesting set of comparative results are revealed by Fig. 10. The test fungus was *Lentinus lepideus,* Mad. 534. From these graphs the threshold for creosote for this organism

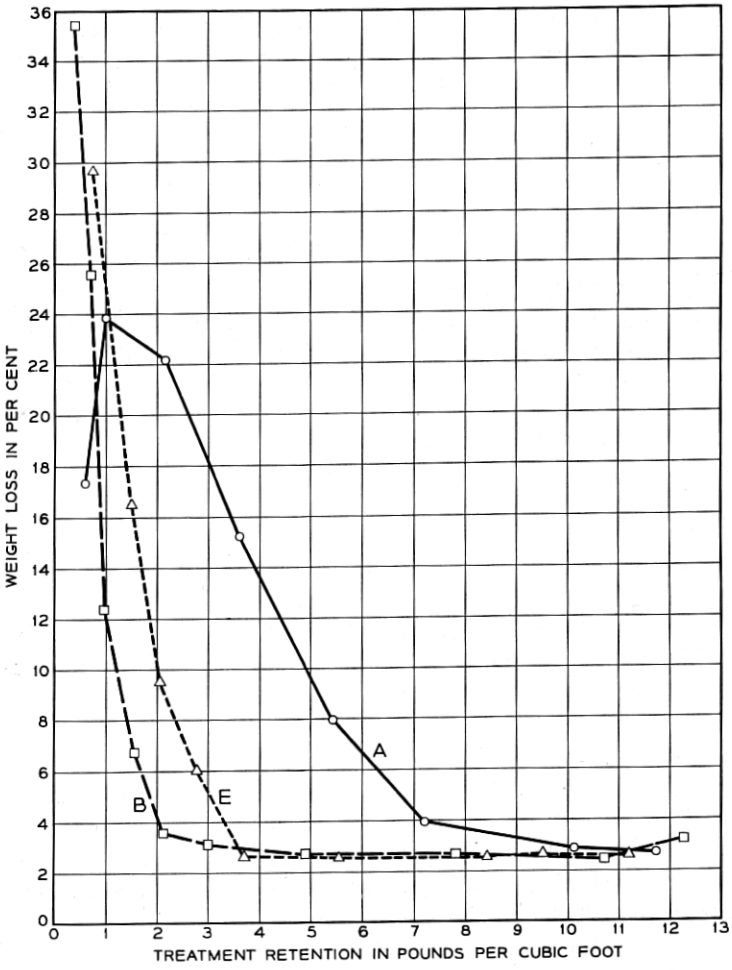

Fig. 10—Soil-block tests against *Lentinus lepideus*; weathered southern pine sapwood blocks; comparison of (A) creosote No. 5340, (B) a 5 per cent solution of pentachlorophenol in Standard Oil Company of New Jersey No. 2105 Process Oil and (E) a 50/50 by volume blend of the two; the relation of operational weight losses, losses by decay, and retention at treatment, lb/cu ft; based on Madison data. The Madison treatment thresholds for these 3 preservative solutions were set at 7.5, 2.4 and 3.4 lb/cu ft, respectively. See text, Table VIII, companion Figs. 11 & 12, Bibliography, Reference 41.

appears to be somewhat in excess of 7.5 pounds per cubic foot, whereas the thresholds for the pentachlorophenol solution are somewhere between 2 and 3 pounds, and the threshold for the mixtures of the creosote and the penta solution about 3.7 pounds per cubic foot. Duncan[41] gives these respective thresholds as 7.5, 2.4 and 3.4 pounds per cubic foot.

Fig. 12 shows that when decay does occur as a result of attack by *Lenzites trabea*, Mad. 617, the loss of weight in the wood is considerably greater than in the case of attack by *Lentinus lepideus*. In the case of *Lenzites trabea*, creosote appears as the best of the three preserva-

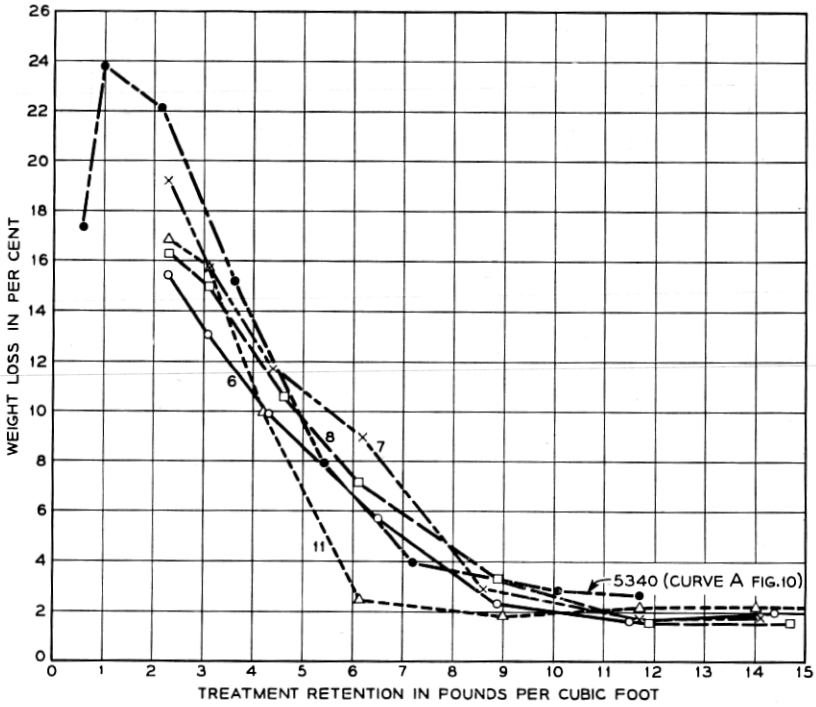

Fig. 11—Soil-block tests against *Lentinus lepideus*; weathered blocks; comparison of cooperative creosotes Nos. 6, 7, 8 and 11, and BTL No. 5340; based on Madison data. The Madison treatment thresholds for these five creosotes were set at 9.0, 9.0, 9.4, 6.5 and 7.5 lb/cu ft, respectively. See Table VIII and Bibliography, References 39 and 41.

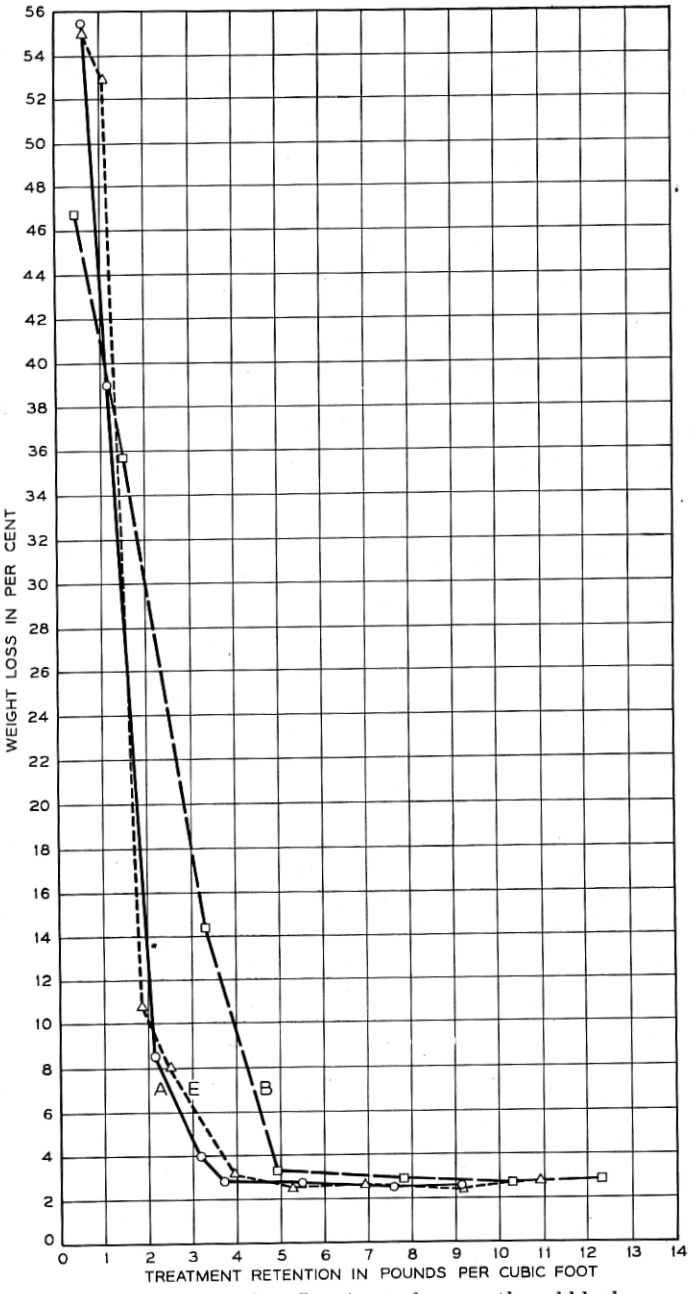

Fig. 12—Soil-block tests against *Lenzites trabea*; weathered blocks; comparison of preservatives A, B and E (Fig. 10); based on Madison data. The Madison treatment thresholds for these 3 preservatives were set at 3.2, 4.8 and 4.0 lb/cu ft, respectively. In the poorly protected blocks note the higher per cent weight losses caused by *Lenzites trabea* in comparison with weight losses caused by *Lentinus lepideus* (Figs. 10 & 11). See Table VIII and Bibliography, Reference 41.

tives; and the mixture of creosote and penta solution is somewhat better than the penta solution alone, although the difference is not great.

The results obtained in testing the different creosotes represented in Fig. 11 are similar in general character for the four domestic oils, but oil No. 11, the mixture of British vertical retort tar creosote and British coke oven tar creosote, appears to behave differently. The thresholds for all of these creosotes, as determined by the Madison investigators, are shown in Table XXXV. The figures in this table correspond very closely to thresholds determined by visual observation of the test blocks.

Check Tests at the Murray Hill Laboratories

It will be noted that in Fig. 11 the points used for locating the graphs are rather far apart in the general region of the estimated thresholds. The values shown in Table XXXV were obtained at Madison by the intersection of regression lines drawn through the points representing

TABLE XXXV — SUMMARY AND INTERPRETATION OF SOIL-BLOCK TESTS

Weathered, creosoted southern pine sapwood blocks; creosote losses; amounts and gross characteristics of residual oils at threshold retentions for *Lentinus lepideus*.

1	2	3	4	5	6	7	8	9	10	11
Item	Creosote No.*	Specific gravity 38/15.5°C	Residue above 355°C per cent	Threshold lb/cu ft	Per cent loss	Creosote loss lb/cu ft	Residual creosote lb/cu ft	Calculated residue above 355°C	Residual creosote	
									>355°C lb/cu ft	<355°C lb/cu ft
1	1	1.065	18.5	9.8	53.1	5.2	4.6	39.4	1.81	2.79
2	7	1.077	20.5	9.0	47.8	4.3	4.7	39.3	1.85	2.85
3	2	1.081	30.6	10.2	47.1	4.8	5.4	57.8	3.12	2.28
4	6	1.093	34.2	9.0	37.8	3.4	5.6	55.0	3.08	2.52
5	3	1.108	50.4	12.2	30.3	3.7	8.5	72.3	6.15	2.35
6	8	1.115	53.2	9.4	25.5	2.4	7.0	71.4	5.00	2.00
7	9a		21.2	5.7	40.4	2.3	3.4	35.6	1.21	2.19
8	9	1.001	20.0	5.8	43.1	2.5	3.3	35.1	1.16	2.14
9	10a		14.4	6.7	50.9	3.4	3.3	29.4	0.97	2.23
10	10	1.068	15.2	6.9	52.2	3.6	3.3	31.8	1.05	2.25
11	11	1.038	18.0	6.5	47.7	3.1	3.4	34.4	1.17	2.23
12	M1	1.107	41.9	8.0	33.8	2.7	5.3	63.3	3.35	1.95
13	M2	1.070	18.1	8.3	50.6	4.2	4.1	36.6	1.51	2.59
14	BTL 5340	1.088	20.9	7.5	46.6	3.5	4.0	39.1	1.57	2.43

* Creosotes 1, 2, 3, 6, 7, 8, 9, 10 and 11 are those in use in the Cooperative Creosote Tests (see Bibliography, References 12 and 39. Oils 9a and 10a are samples from the same lots as numbers 9 and 10. (See Bibliography, Reference 36.) For oils M1 and M2 see Bibliography, References 37 and 38. Creosote 5340 is shown in Table II.

operational losses and through the points representing weight losses. Data from a repetition of these tests is desirable in order to establish the thresholds more definitely from actual weight loss or observational data taken close to the assumed threshold points. At Bell Telephone Laboratories a check series of tests is now under way on cooperative creosotes 6, 7 and 8, domestic oils, and creosotes 9, 10 and 11, British oils; and comparison tests are also being run on creosote BTL-5340 and on 5 per cent pentachlorophenol in the 2105 process oil. The aim has been to treat the blocks to a series of retentions that vary narrowly around the thresholds set by the Madison investigators.

Across the Threshold

Fig. 13 is an illustration of representative blocks from the creosote series, line A (creosote BTL-5340) in Fig. 10, just at and below the threshold. Fig. 14 shows the character of the attack by *Lenzites trabea* on blocks treated with a 4.92 per cent solution of pentachlorophenol in Standard Oil Company of New Jersey's 2105 Process Oil in toluene. The blocks are represented at twice their original linear dimensions. The exact nature of the decay is difficult to show. The experimenter has to learn a system of diagnosis that involves both visual observation and the "feel" of the blocks for distortion and firmness that supplement weight loss data. For example, in Fig. 14, a threshold between 0.20 and 0.25 pound of penta per cubic foot (Blocks C and D) is indicated and this conforms closely to the results with the same penta-petroleum solution at Madison.[41]

The Significance of the Results of Laboratory Soil-Block Tests on Oil-Type Preservatives

The main conclusions from this discussion of the results of soil-block tests on weathered creosoted wood conducted at Madison are (a) that in general, under the test conditions, at least 8 and sometimes 9 pounds or more of creosote per cubic foot is a necessary treatment to prevent attack by *Lentinus lepideus* on ¾-inch cube blocks of southern pine sapwood; and (b) that a penta petroleum solution is much more effective than creosote against this same organism. As will be emphasized later, this general conclusion about *Lentinus lepideus* and creosote corresponds with the conclusions to be drawn from the interpretation of results of the small stake tests and from the test of pole-diameter posts in the Gulfport test plot.

The creosote tested is a better preservative against *Lenzites trabea* than the penta-petroleum, but the creosote threshold for this organism

is below what one would have to use commercially in order to insure protection against *Lentinus lepideus* and to preserve wood exposed to ground contact.

As far as *Lentinus lepideus* is concerned, the best overall preservative combination from the laboratory test would appear to be a 50/50 by volume blend of the creosote and the penta-petroleum solution, since such a blend appears to contain the best in both components. However, there are certain practical considerations, principally relating to the incompatibility of some creosotes and petroleums, which make the combined preservative a difficult one to operate with commercially.

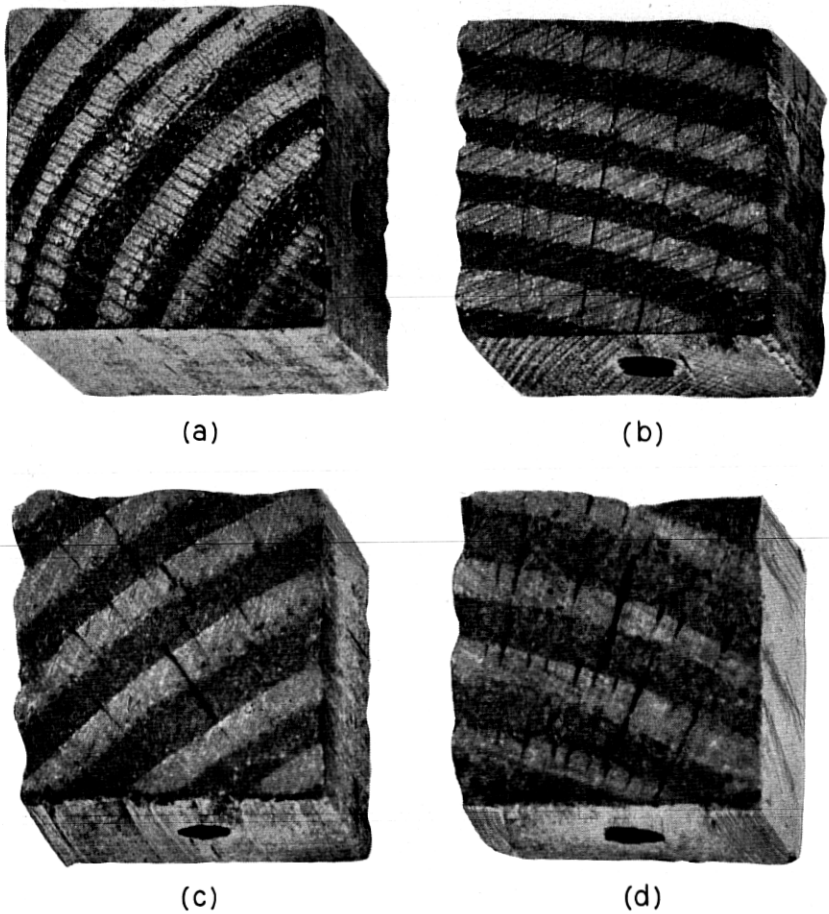

Fig. 13—Across the threshold—creosote. Test fungus, *Lentinus lepideus*; creosote concentration at treatment: (a) 11.70; (b) 6.92; (c) 5.45; and (d) 4.15 lb/cu ft.

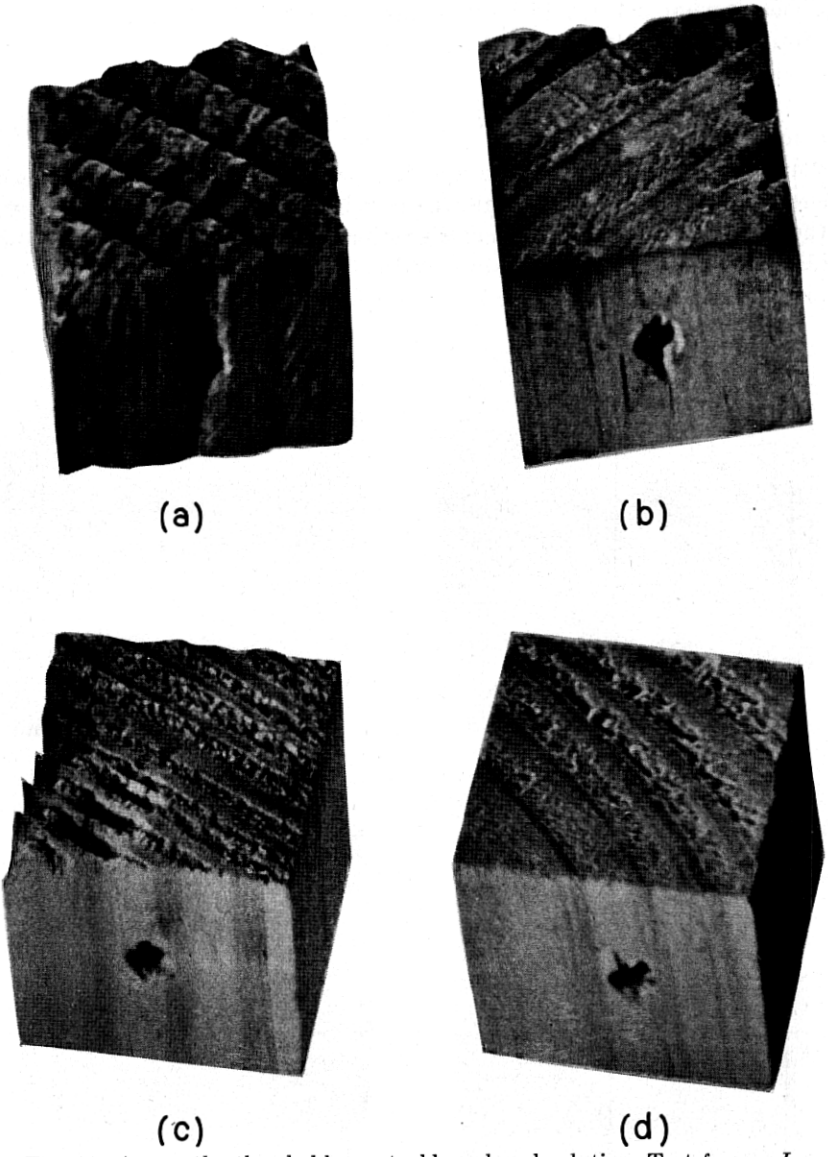

Fig. 14—Across the threshold—pentachlorophenol solution. Test fungus *Lenzites trabea*; dry penta concentration at treatment: (a) 0.052, (b) 0.099, (c) 0.193; and (d) 0.238 lb/cu ft. *Photo by A. H. Hearn.*

Relatively speaking the soil-block test procedure is much more rapid than the test plot experiments that are to be discussed next, but since the inferences with respect to retention requirements for creosote appear to be the same for the laboratory and the field tests, the former have a direct and immediate application in practical pole preservation.

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