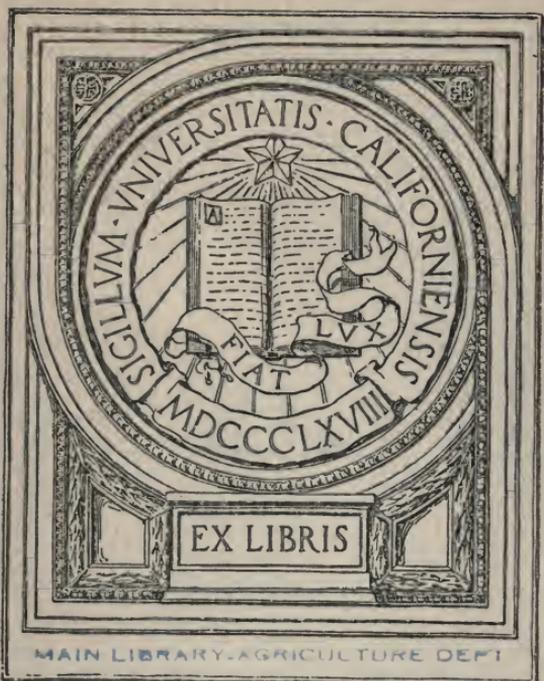


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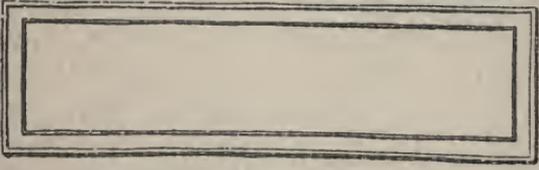


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U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY—BULLETIN No. 144.
H. W. WILEY, Chief of Bureau.

WOOD TURPENTINE:

ITS PRODUCTION, REFINING, PROPERTIES, AND USES.

BY

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1911.

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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., July 31, 1911.

SIR: I beg to submit for your inspection and approval the results obtained in an investigation made in this bureau by F. P. Veitch and M. G. Donk on the distillation of resinous woods, especially the product wood turpentine. The investigation has been in progress for several years, and although certain phases on which work is being continued are not complete, it is deemed advisable to publish at this time the results so far obtained. Many inquiries are received for information on the subject of resinous-wood distillation, and it is believed that the data herein given will be helpful, and also will further promote the profitable utilization of large quantities of waste wood.

I recommend that this report be published as Bulletin No. 144 of the Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

HON. JAMES WILSON,
Secretary of Agriculture.

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WOOD TURPENTINE:

ITS PRODUCTION, REFINING, PROPERTIES, AND USES.

INTRODUCTION.

Wood turpentine, "stump turpentine," or "wood spirits of turpentine," as the product is variously known, is turpentine oil made from cut pine, fir, or spruce, by distilling the wood in closed retorts. When properly refined it closely resembles gum spirits of turpentine, obtained by distilling the gum which oozes from the cut surface of the living tree. The turpentine made by distilling the wood with steam below a temperature of 150° C. more closely resembles gum spirits than that obtained by destructively distilling the wood. The latter is always contaminated with other oils derived from the breaking down of the wood and the resins which it contains. In some cases where the context shows plainly that wood turpentine is meant the term "turpentine" is used to avoid repetition; it should be noted, however, that commercially wood turpentine should always be designated as such and not as "turpentine."

Wood turpentine is used chiefly as a varnish and paint thinner as a substitute for, or in the place of, gum turpentine. Its production and use has increased slowly, partly because of its objectionable odor, its physiological effect, its lack of uniformity, and unsatisfactory working qualities due thereto, and partly because of the lack of knowledge regarding its ultimate effect on the color and durability of varnishes and paints.

The recovery of turpentine by distillation from the wood was attempted years ago, and the first patents relating to this subject were granted as early as 1841. The process was not developed, however, and it is only within recent years that any material success has attended its use. There are two primary causes for the numerous efforts which have been made during the past 10 or 15 years to recover turpentine from cut pine wood: First, the difficulty of supplying the increasing demand for turpentine, owing to the rapid decrease of the supply of live pine timber suitable for even the most advanced system of turpentine orcharding; second, the demand for a profitable method of utilizing the mill and forest wastes of the pine regions, not only that such wastes may be made valuable, but that

the damage arising from their nonuse may be prevented. Thus it happens that business men and inventors have been actively devising and exploiting apparatus and processes for the recovery of the turpentine from this waste wood, and to distinguish it from ordinary gum turpentine the product is quite properly spoken of as wood turpentine or stump turpentine.

Judging from statistics, the production of gum spirits of turpentine in this country is now on the decline, as the census reports show that in 1909, 28,941,000 gallons; in 1908, 36,589,000 gallons; in 1907, 34,180,800 gallons; in 1905, 30,170,499 gallons; and in 1900, 38,488,170 gallons were produced. On the other hand, the value of the turpentine was \$12,654,000 in 1909; \$14,112,400 in 1908, \$18,283,300 in 1907; \$15,170,999 in 1905, against \$14,960,235 in 1900. The pine timber available for turpentinizing is rapidly being exhausted, as is quite generally recognized among turpentine operators, and under present conditions no material permanent increase can be expected in the output of gum turpentine. This fact, as has been stated, is one of the chief reasons for the interest which has developed with regard to wood turpentine. If we accept the conclusion that the demand for turpentine can not be fully supplied from the present sources, it is reasonable to assume that if wood turpentine may be used for the same purposes, or for some of the purposes, for which gum spirits is used, it should find a ready market. The turpentine-using industries have been slow to accept wood turpentine, and although there is a market, it is still unfavorable, largely because of the nonuniformity of the product.

The owner of pine lands and the lumberman are directly interested in wood turpentine from another point of view, that of the profitable utilization of waste wood. In general, such methods of using wood are less profitable than to dispose of it through the usual channels, but the method utilizes material of little value for other purposes, which, when unused, is a source of danger from forest fires. Furthermore, the profitable utilization of waste timber and stumps makes it possible to clear cut-over lands at a minimum expense and prepare them for cultivation.

While it is well known that there are immense wastes of wood incident to the lumber industry, there are so few exact data that no accurate calculation can safely be made either as to the waste at the mill, including slabs and sawdust, or in the forest, including tops, laps, culls, dead-and-down timber, and stumps. Some figures on the subject, however, may not be without interest: The Census Bureau estimates that in 1908 approximately 11,000,000,000 board feet of southern yellow pine were cut; of Douglas fir, 4,000,000,000 board feet; and of western pine 1,225,000,000 board feet, a total of approximately 16,000,000,000 board feet. If the waste at the mill

and in the forest be placed at 50 per cent of this, a very conservative estimate, there are 8,000,000,000 board feet, or approximately 8,000,000 cords of wood (on a basis of 40 per cent unoccupied space in a cord), available for the recovery of turpentine and rosin and the manufacture of paper, acetate of lime, wood alcohol, oxalic acid, etc. From this class of material from 1 to 10 gallons of crude turpentine per cord are recovered under the present practice, the average recovery being 3 gallons per cord.

Pine wood also yields from 3 to 15 or 20 per cent of rosin, while exceptionally rich lightwood may yield from 30 to 35 per cent. The average rosin yield of good lightwood is probably between 12 and 18 per cent. Assuming low yields of turpentine and rosin, a calculation shows that it is possible to recover from the wastes of the yellow-pine lumber industry (including dead-and-down timber) as much or more turpentine, rosin, and rosin oils as are now produced by the ordinary methods of turpentine from the living tree. The profitable utilization of mill wastes in this way would add materially to the wealth of the South and help to conserve its timber resources.

METHODS OF PRODUCTION.

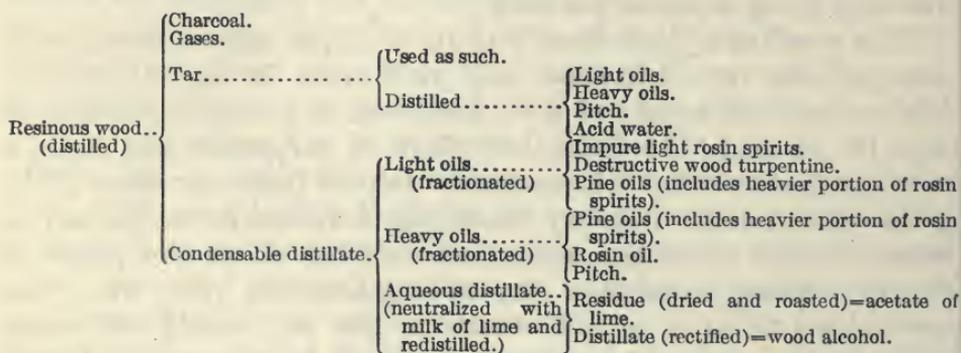
Wood turpentine can be recovered from any of the coniferous woods. Those which have been found to contain sufficient resin to justify working them are the long-leaf yellow pine, of the South, the Norway pine of the Central North, and the Douglas fir of the Northwest. The methods by which wood turpentine is recovered may be appropriately divided into three classes, (a) destructive distillation methods, (b) steam distillation methods, (c) extraction with solvents.

DESTRUCTIVE DISTILLATION.

In the destructive distillation method wood exceptionally rich in turpentine, and rosin (generally termed "lightwood" or stumps) is loaded into retorts holding one or more cords each, and slowly heated at a low temperature until all the turpentine and other low-boiling oils are driven off and condensed, when the oils are directed to other receiving vessels, and the distillation of the wood continued until nothing remains in the retort but charcoal. In conducting this process it is important that the temperature of the retort should not rise above 200° C. until all turpentine in the wood has been driven off, otherwise the wood is decidedly charred and the turpentine is contaminated with other materials from which it can not be subsequently purified. Industrially, it has been found impracticable until very recently to prevent such contamination, and for this reason destructively distilled turpentine has certain characteristic properties which distinguish it from turpentine prepared in other ways. The

time for distilling by the destructive process varies from 24 to 30 hours. Preliminary experiments conducted in the laboratory several years ago indicated that it is feasible to control the heating of the wood by using an oil jacketed retort, but no work on a commercial scale has been done with such apparatus as far as is known.

The following diagram shows the products of the destructive distillation of wood and the processes used:



STEAM DISTILLATION.

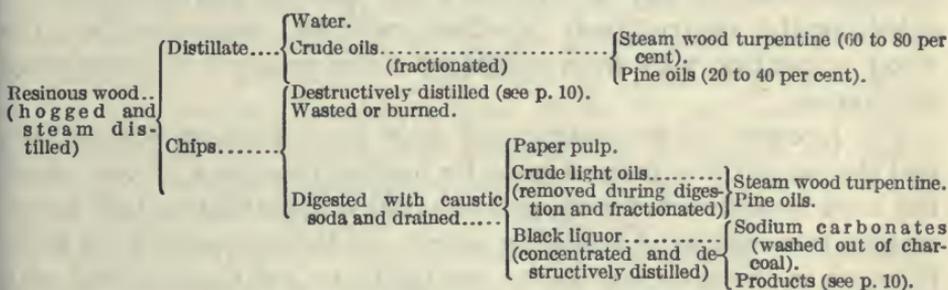
For distilling with steam the procedure and equipment are quite different from those used in destructive distillation. In the first place mill waste, including sawdust, can often be profitably utilized. Other wood must be chipped or cut into small pieces in order that the steam may penetrate it and remove the oils readily. The chipped wood, prepared by what is known as a "hog," is taken directly to the retorts by conveyers, or is placed in one of the many forms of inner containers which have been devised to facilitate the penetration of the chipped wood by the steam, and also the emptying of the retort at the conclusion of the distillation, both of which operations, even under the most favorable conditions, are quite difficult. Live steam is conducted into the retort until no more oils pass over, which requires in most plants from 3 to 24 hours. The distillate, consisting of a mixture of oils and water, is condensed, allowed to separate into two layers, and the crude oil is stored in large storage tanks, from which it is drawn to the refining still.

The live steam which is turned into the filled retorts may be superheated or saturated, and used at atmospheric pressure, under additional pressure, or at diminished pressure, as may be desired. All of these methods of handling the steam are used, but as a matter of fact, it can not be said that under the conditions at present usually obtaining in the industry, higher or better yields are secured by one procedure than by the other. As turpentine distills with steam at from 94 to 98° C., no advantage as to yield can logically be expected from the use of superheated or pressure steam, other than that which may arise from a quicker and more thorough penetration of the wood

by the steam (which, as has been said, is difficult to secure) coupled with the smaller quantity of vapor to be condensed. Further, the use of steam under pressure is questionable, as the terpenes are readily polymerized at high temperatures. On the other hand, it is relatively certain that owing to the difficulty of securing contact, the usual steam process in fixed retorts does not remove all turpentine from the wood, and an apparatus or process which will insure contact of the steam with all of the wood will give higher yields of turpentine. In this connection, the use of a rotating retort immediately suggests itself, and several plants claim to obtain larger yields with these. Distillation under reduced pressure, if found practicable, undoubtedly offers several important advantages, among which are the distillation of turpentine at low temperature, thus reducing the tendency to polymerization, penetration of the wood by steam, and distillation of the turpentine from the interior of the pieces of wood.

Prolonged heating is exceedingly costly, and with proper equipment is needless, as it has been demonstrated that turpentine may be removed in from two to three hours, when proper penetration of the wood by the steam is secured. The steam process is particularly applicable for the utilization of mill wastes, such as slabs and sawdust, and has found its greatest development in connection with the sawmills of the South.

The following diagram shows the products of the steam distillation of wood and the utilization of the extracted chips:

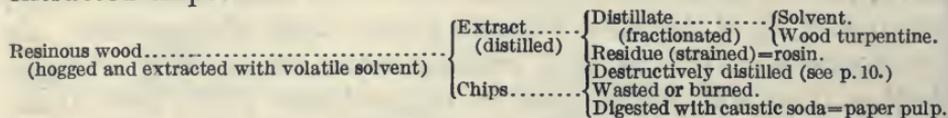


EXTRACTION WITH SOLVENTS.

Finally, turpentine may be recovered from wood by dissolving it in a volatile or nonvolatile solvent, or in an alkali. Extraction with volatile solvents is but little employed, and deserves more consideration than it has received. The economical working of solvent processes is dependent primarily on the possibility of a practically complete recovery, at a minimum cost, of the large quantities of solvent which are required to saturate the wood for extraction. Fortunately the solvent can be almost entirely recovered. The products obtained by the use of volatile solvents of low boiling point are of high quality; the rosin is clean and bright, while the turpentine is comparatively free from materials produced in the extraction.

This method has been studied by this bureau and by other investigators, and is perfectly simple and feasible, but for the reasons mentioned it remained undeveloped until very recently. It is now being placed on a commercial basis.

The following diagram shows the products obtained from resinous wood by extracting with volatile solvents and the utilization of the extracted chips:



Several processes have been developed which make use of a liquid having a higher boiling point than turpentine. Melted rosin, pitch, tar, pine oils, or crude gum turpentine are suitable in starting the plant, but as a matter of fact the bath finally consists of rosin, because of the fact that the volume is constantly increased by rosin removed from the wood. In such processes the liquid passes through a superheater, where its temperature is raised to about 200° C., and thence to a closed retort containing the wood so prepared as to insure rapid penetration by the liquid. In returning to the superheater the hot rosin passes through a still and the turpentine is distilled from it. The liquid is finally drained from the exhausted wood and used on subsequent charges. The advantages of this process over simple distillation with steam are that the treated wood is left in such condition that it may be sold for fuel, used as paving blocks, or subsequently destructively distilled without contaminating the wood turpentine with rosin spirits or other products of destructive distillation.

The recovery of turpentine and rosin by extraction with alkalis and the use of the extracted wood for making paper is a process which has been experimented with, and one mill is operating on mill waste, making turpentine and wrapping paper. In this process also the wood is cut up in the same way as for making paper pulp and treated with an excess of soda in solution. The turpentine may be removed by simple steaming previous to adding the soda solution, or it may be steamed off during the cooking with soda. The latter procedure has the advantage of requiring less time to complete the treatment of the wood. The cooking with soda may be conducted in several ways. The contents of the retort may be heated only sufficiently to dissolve the rosin without attacking the wood, after which the solution may be drawn off nearly free from ligneous materials, and the rosin or rosin oils recovered from it, or the wood may be cooked with the soda solution at a high temperature.

After the completion of the treatment the soap solution of soda and rosin, containing also the dissolved ligneous material, is drained

and washed out of the pulp, which, after suitable treatment, may be disposed of in several ways. The removal of the turpentine by this process is exceedingly rapid, and it is claimed that from the boiling alkali solution the turpentine may be removed by some processes in from 10 to 15 minutes.

REFINING WOOD TURPENTINE AND WOOD OILS.

The crude oil obtained by any distillation process is redistilled or refined before it is marketed. Crude oils from the destructive process are agitated with alkali whereby the greater part of the phenols, cresols, and related bodies are dissolved, and separated by settling from the turpentine, rosin spirits, and rosin oils, which are run off and separated by redistillation with steam as steam-distilled wood turpentine is. This is usually done in a copper-pot still of suitable size, live steam being conducted directly into the crude oil. Ordinarily this distillation is not conducted with care, and but little attempt is made to insure that the wood turpentine obtained does not contain considerable quantities of the heavier "pine oils." Distillation is usually continued until the oil passing over shows "bead" or until the specific gravity is about 0.8800 or 0.9000 at 15.5° C.

Recently at a number of plants more care has been exercised in refining and the product is distilled with steam several times from a pot still. Such careful refining is, of course, expensive and the yield is much reduced, but the product complies closely in specific gravity and behavior on distillation and evaporation with standard specifications for turpentine. The crude oil yields on redistillation, as this is commonly practiced, from 60 to 80 per cent of wood turpentine. As will be seen from the analyses (Table 1, p. 58), these oils contain notable quantities of heavier oils, and but few of them can be considered high-grade wood turpentine.

The pine oils from steam distillation are also redistilled with steam and are marketed in two grades, white and yellow or straw colored. Their specific gravity ranges from 0.8890 to 0.9600; refractive index from 1.470 to 1.500 and they begin to distill at from 165° to 180° C. Distillation is generally complete at from 215° to 240° C. and by far the larger quantity of most pine oils distills between 190° and 225°. The crude, destructively distilled oils, after being agitated with alkali and washed with water to remove phenols, cresols, etc., are separated into at least three fractions, preferably in a column still. The first fraction, distilling at from 80° to 150° C., consists essentially of the lighter constituents of rosin spirits with some turpentine and is contaminated with small portions of substances derived from the breaking up of the wood. The fraction boiling at from 150° to 180° C. is destructively distilled wood turpentine, that boiling at from 170° to 180° C. to 230° to 250° C. constitutes the pine oils with the heavier

constituents of rosin spirits, and the fractions boiling at from 250° to 400° C. consist of rosin oils contaminated with decomposition products from the wood substance.

The alkali solution after separation from the crude oils contains the wood creosote, tar, and other alkali-soluble bodies. The wood creosote and tar acids may be separated by adding sulphuric acid in excess, washing the separated material several times with water, and further refining, if desired, by distillation. (See Table 2, p. 60, for analyses of pine oils and still residues.)

EQUIPMENT FOR THE MANUFACTURE OF WOOD TURPENTINE.

The equipment of a plant for the destructive distillation of resinous woods would, of course, vary with its capacity, size and shape of retort, and arrangement of the plant. Small retorts of about 1 cord capacity are most commonly used, and an approximate idea of the kind and quantity of apparatus required for a plant distilling 12 cords per 24 hours is given by the following list:

- 12 retorts, 1 cord each.
- 12 condensers.
- 100 100-pound charcoal cans.
- 2 tar stills.
- 1 turpentine refining still.
- 2 or 3 settling vats for crude turpentine, crude pyroligneous acid, and free tar.
- 1 100-horsepower boiler.
- 1 20-horsepower engine.
- Pumps for supplying water to condenser and handling turpentine, etc.
- Storage tanks for crude and refined products.
- Conveyers and copper piping.
- Suitable buildings for housing equipment.

The yield of wood alcohol and acetic acid is less than half of that obtained from hard woods, or about 4 gallons of alcohol and from 50 to 90 pounds of acetate of lime, and practically no effort is made to recover either of these products. In case these are recovered, additional stills, tanks, and steam pans are required.

The cost of such a plant varies greatly in accordance with the quality and completeness of the equipment and with local labor and other conditions. Reliable builders of such plants quote from \$1,000 to \$2,000 per day cord on the basis of a 10-cord plant, with proportionately higher prices for smaller plants and lower prices for larger plants. For the steam distillation of wood where only turpentine and pine oils are recovered, a less elaborate equipment is required. For a plant of 10 units, the following list gives an idea of the equipment needed:

- 10 retorts, 1 cord each.
- 10 condensers.
- 2 150-horsepower boilers.
- 1 100-horsepower engine.

- 1 refining still and condenser.
- 2 hogs for chipping wood (1 in reserve).
- Storage tanks for crude and refined turpentine.
- Pumps and piping for water supply and turpentine.
- Conveyers.

The cost of such a plant will vary from \$10,000 to \$35,000, depending largely on the value placed on patent rights (many forms of retorts and other apparatus are patented, and as a rule the right to use is charged in the cost of the plant), and on the number of charges the plant will handle in 24 hours. The cost of a plant to take from four to six charges in 24 hours is, of course, greater than that of the same plant built to handle but one charge in the same time, though the cost on the basis of the number of cords of wood handled daily may be much less.

These lists of apparatus and estimates of cost of plant are simply intended to give a general idea of the equipment formerly furnished by builders and now in common use in the older plants, together with its approximate cost. No attempt is made to give details in regard to the machinery, arrangement, or housing. The boiler, engine, and condenser capacity of these older plants was rarely sufficient for maximum efficiency. Other special equipment now employed at several plants and considered preferable is described on page 54. The full equipment and its actual cost can be determined only when the location of the plant and the kind and quantity of work required are known.

COST OF PRODUCING WOOD TURPENTINE.

The cost of producing wood turpentine varies greatly in different plants and is dependent on the cost and quality of the wood obtainable for the distillation, the cost of operating and managing the plant, depreciation of the plant, and interest on the investment. The cost of wood, according to information collected by the bureau, varies from \$1.50 to \$3 per cord for what is known as "lightwood," delivered at the works. This charge is made up chiefly of the cost of gathering, hauling, and freight, the wood in the forest costing very little—from 15 cents to 75 cents per cord being a fair valuation. While it is the aim of the distiller to use only wood rich in turpentine and rosin, it is often quite difficult to get this, particularly when the wood is delivered at the works or at the railroad on contract, as the contractor does not as a rule exercise much care in selecting the best and richest wood. Better wood is undoubtedly obtained when it is selected and gathered by the distiller's own force, and, furthermore, the cost of hauling and handling is not so great as a rule as when it is done by contract. The cost of waste wood from a lumber mill averages much less, probably not more than \$1.50 per cord.

The cost of operating destructive distilling plants, including office expenses, labor, fuel, and packing products, is variously estimated by the owners and exploiters at from \$2.50 to \$6 per cord, while the charges for interest and the depreciation of plants are figured at from 60 cents to \$1.60 per cord of wood distilled. The cost of operating a steam distilling plant is estimated at from 47 cents to \$1.82 per cord and the charges for interest and depreciation are from 24 cents to 61 cents per cord. The cost of operating plants may be summarized as follows:

Relative cost of operating steam and destructive distillation plants.

Items.	Destructive distillation, per cord.	Steam distillation, per cord.
Cost of wood for distilling.....	\$1.50 to \$3.00	\$1.00 to \$3.00
Management, labor, fuel, packing.....	2.50 to 6.00	.47 to 1.82
Interest and depreciation.....	.60 to 1.60	.24 to .61
Total.....	4.60 to 10.60	1.71 to 5.43

It must be borne in mind that in destructive distillation other products, such as charcoal, pine oils, and rosin oils, are produced, the value of which, under proper conditions, should more than cover the greater cost of operating the destructive process.

The cost figures given vary so widely that it is quite evident that they may be greatly reduced by proper equipment and more rapid handling of the material. This statement applies particularly to the steam processes, in which this laboratory has found that the average time of distillation may be greatly reduced without lowering yields. Indeed, the greatest field for improving the wood-turpentine industry lies more in reduced cost of production than in increasing the yields per cord of wood, though there are, of course, great possibilities here also. In the examinations which have been made of wood-turpentine plants in the South, one is particularly impressed with the crudeness of equipment and the wasteful labor conditions which prevail in most cases. Even in the most modern plants decided improvements in arrangement and operation could be made. The possibilities of increasing profits by improvements in these particulars seem to be exceedingly inviting. Necessarily, improvement along these lines can only be made by competent management, and all such plants should be under the control of or have the advice of an able chemical engineer. The cost of production may also be largely reduced by a proper location and affiliation of the plant.

YIELDS OF VARIOUS PRODUCTS FROM THE DISTILLATION OF RESINOUS WOODS.

The yield of crude oil obtained is governed by two factors—the quantity of oil in the wood and the completeness with which this is removed by the method used. Thus, by steam processes from 2 to 15 gallons per cord are recovered from sawdust and slabs, from 8 to 20 gallons from lightwood, and as high as 30 gallons from very rich lightwood. By destructive processes from 10 to 20 gallons are recovered from ordinary lightwood, and larger quantities from exceptionally rich lightwood. By extracting with soda it is claimed that somewhat better results are obtained.

The operators claim that when using fixed retorts and the steam process a good yield from lightwood, under usual conditions, is approximately 15 gallons of crude oils per cord; with rotary retorts the production is increased to about 18 gallons, and experiments have shown that at least 3 gallons of crude oil, containing about 25 per cent of actual turpentine, are often left in the wood. The crude oil yields about 80 per cent of refined oil—a result which is only obtained by distilling much of the heavy oil with the turpentine. By the destructive process the yield of wood turpentine is several gallons per cord greater than by the steam process.

The data on yields of various products as summarized in the following tables were obtained from many sources, mostly from wood distillers, some from the literature, and some based on the experimental work of the bureau. The whole is harmonized as far as possible in accordance with the experience and judgment of the author. The data on yields of crude oils, refined wood turpentine, charcoal, methyl alcohol, acetate of lime, and unbleached wood pulp may safely be regarded as quite accurate. In fact, all the data on the steam and volatile solvent processes may be so regarded. The other data on pine oils, rosin spirits, rosin oils, and creosote, by the destructive and alkali extraction processes, are not exact and in general are to be regarded as careful estimates rather than as known results. This is due to the fact that less attention has been paid to the separation and utilization of these products, and what one producer terms "creosote" may in reality embrace all oils heavier than pine oil, or, in some cases, those heavier than turpentine. In other plants all oils heavier than pine oils may be combined with the tars and disposed of under this term. Further work will be necessary before more definite information can be given as to the quality and nature of the products variously termed "rosin spirits," "rosin oils," "creosote," "wood oils," "tars," etc., obtained in the destructive distillation of pine wood.

Data on the yield of various products from 1 cord (4,000 pounds) of long-leaf yellow pine ("lightwood").

Products.	Steam process.		Destructive process.		Alkali extraction.		Volatile solvents.	
	Dis-tills between—	Yield.	Dis-tills between—	Yield.	Dis-tills between—	Yield.	Dis-tills between—	Yield.
Crude oil.....	°C. 154-250	Gallons. 8-20	°C. 80-400	Gallons. 36-120	°C. 154-400	Gallons. 36-120	°C. 154-250	Gallons. 8-20
Refined wood turpentine.....	150-180	6-16	150-180	5-20	150-180	5-20	150-180	6-16
Pine oils.....	175-250	2-4	175-250	2-5	175-250	2-5	175-250	2-4
Rosin spirits.....			{ 80-150 180-250 }	2-10	80-250	5-15		
Rosin oil.....			250-400	20-65	250-400	20-60		
Creosote.....			100-400	8-20	100-400	8-20		
Rosin.....								1 300-800
Methyl alcohol.....				½-4		½-4		
Calcium acetate.....				1 40-80		1 5-15		
Charcoal.....				2 30-50		2 10-25		
Paper pulp, unbleached.....		1 1,000-1,600				1 1,000-1,600		1 1,000-1,600

¹ Pounds.

² Bushels.

Approximate yields of various products from 1 cord (4,000 pounds) of lean long-leaf yellow pine.

Products.	Steam process.	Destructive process.	Alkali extraction.	Volatile solvents.
	Gallons.	Gallons.	Gallons.	Gallons.
Crude oils.....	2-10	20-52	20-50	2-10
Refined turpentine.....	1½-8	2-10	2-8	1½-8
Pine oils.....	½-2	½-2	½-2	½-2
Rosin spirits.....		2-5	3-8	
Rosin oils.....		12-27	10-25	
Creosote.....		4-8	4-8	
Rosin.....				1 75-400
Methyl alcohol.....		½-5	½-5	
Calcium acetate.....		1 40-100	1 5-15	
Charcoal.....		2 35-50	2 10-25	
Paper pulp, unbleached.....	1 1,000-1,600		1 1,000-1,600	1 1,000-1,600

¹ Pounds.

² Bushels.

In these tables the distillation is assumed to be carried to completion in all cases. The tar which is formed is distilled, yielding rosin spirits, rosin oils, and creosote. Therefore tar is not given as one of the distillation products of wood, inasmuch as it is only an intermediate product. In practice the impure heavy oils which run out of the wood or result from the partial breaking up of the wood collect on the floor of the retorts and are withdrawn before they are completely distilled by the rising temperature of the retort. Tars from resinous woods consist, therefore, of the phenoloid bodies, cresols, and other related bodies from the incomplete distillation of the wood substance, mixed with pine oils, rosin spirits, rosin oils, rosin, and small quantities of organic acids. The quantity of tar per cord is somewhat less than the sum of the rosin spirits, rosin oil, and creosote. The maximum yield will rarely equal the highest figures given for those

products. The "rosin spirits" and "rosin oils" obtained by destructively distilling pine or its soda extract are necessarily contaminated, as is also the turpentine, with empyreumatic constituents derived from the distillation of the cellulose and lignin. They are not, therefore, strictly speaking, "rosin oils" and "rosin spirits," but may properly be termed "wood-rosin spirits" and "wood-rosin oils."

These facts should be borne in mind in considering the tabulated data on yields. It is hoped that more definite data as to the yield of various products from pine will soon be available. The operations in the past at wood turpentine plants have as a rule not been conducted with the care necessary to determine even average yields with exactness. The weight of a cord of wood varies greatly, and the amount of water in the wood may also vary. In order that exact data may be had, the wood at turpentine plants should be weighed, a sample analyzed, and all products measured or weighed. The wide values given for the several products are explained partly by variations in the composition of the wood; as has been stated, pine wood differs greatly in the quantity of resin it contains.

The following figures are taken from the census reports:

Statistics on the production of wood turpentine and other resin oils from wood (census reports).

Years.	Wood used.		Turpentine produced.			Oils produced.	
	Cords.	Value.	Gallons.	Gallons per cord.	Value.	Gallons.	Value.
1906.....	50,000	\$129,000	503,000	10.1	\$239,000	125,000	\$17,000
1907.....	62,000	211,000	655,000	10.5	305,000	392,000	69,000
1908.....	99,000	202,000	506,000	5.1	166,000	305,000	56,000
1909.....	116,000	234,000	683,000	5.9	243,000	323,000	70,000

Though the quantity of wood used in this industry has more than doubled in five years, the quantity of turpentine reported shows some fluctuation and a rather small increase. Owing to the fact that the products of both steam and destructive processes are included in the summary, the only conclusion warranted seems to be that the yield of turpentine per cord has greatly decreased, and this decrease is not balanced by an equivalent increase in the reported heavy oils. It seems probable that the decreased yield per cord reported in 1908 and 1909 is partly accounted for by greater care in refining, whereby the heavy oils are more completely separated. The decreasing yields indicate either that the wood used in late years is not so rich as that formerly employed, or that the distillation is less complete, or that a different system of reporting is employed.

PROPERTIES AND COMPOSITION OF WOOD TURPENTINE.

Ordinary gum turpentine recently prepared by distilling the crude gum may be defined as a mixture of terpenes having the same empirical chemical formula, $C_{10}H_{16}$, together with small quantities of certain derivatives of these terpenes, concerning which but little is known. These components differ in specific gravity, optical properties, boiling point, consistency, refractive index, odor, and probably in other physical and chemical properties. The peculiar suitability of gum turpentine as a diluent for paints and varnishes is due apparently to the fact that it evaporates neither too rapidly nor too slowly—not so fast as to leave the coated surface full of fine pores through which moisture may enter and destroy the paint or varnish, nor so slowly as to unduly prolong the drying. Further, its oxygen-carrying power hastens the oxidation of the linseed oil, and thus forms the binding coat which holds the paint bases and resins to the surface of the material.

Crude wood turpentine differs from gum spirits primarily in that it contains additional terpenes and terpene derivatives, together with other nonterpene derivatives. In that obtained by the destructive distillation of long-leaf yellow pine, dipentene, pentane, pentene, toluol, heptene, etc., have been reported, in addition to pinene.

After washing with soda to remove phenoloid bodies derived from the breaking up of the wood, fractional distillation, or fractionation alone in the case of the steamed distilled oils, will separate these mixtures into two or more portions, each of which will consist very largely of constituents whose boiling points are very close together. It is impossible, however, by distillation to separate those compounds having nearly the same boiling points, and as a matter of fact each fraction will also contain more or less of the heavier oils, depending on how carefully the fractionation has been conducted. Thus, from the destructively distilled oils, fractions distilling at from 80° to 155° C., from 155° to 180° , from 180° to 220° , from 220° to 250° , from 250° to 400° , or, as a matter of fact, between any desired limits, may be obtained.

The first fraction distilling between 80° and 154° C. closely resembles rosin spirits, and is in fact the lighter portion of rosin spirits contaminated with small quantities of materials from the nonresinous wood constituents. The portion passing over between 154° and 180° C. is destructively distilled wood turpentine. This fraction contains pinene and dipentene, together with smaller quantities of other compounds, at least some of which are normal constituents of rosin spirits. The heavier oils distilling from 180° C. up are pine and rosin oils in indefinite mixtures. They are used in the manufacture of lubricants, printing inks, solvents, etc. The crude oils

and tars of the destructive process are often used as disinfectants, cable coatings, wood preservatives, shingle stains, etc.

The crude oils obtained by the steam distillation of long-leaf yellow pine contain pinene, camphene, limonene, dipentene, terpineol, borneol, fenchyl alcohol, camphor, cineol or eucalyptol, etc. They are fractionated to two or three fractions, wood turpentine distilling at from 150°–160° to 175°–180° C., light pine oils between 170°–180° and 210°–225° C., and heavy pine oils at from 180°–190° to 230°–240° C.

Steam-distilled wood turpentine consists essentially of pinene, together with camphene, limonene, dipentene, cineol, and, depending on care of fractionating, more or less, terpineol, borneol, terpinene, etc.

The pine oils contain chiefly terpineol, borneol, and fenchyl alcohol, with small quantities of limonene, dipentene, terpinene, cineol, and even less pinene and camphene. Pine oils are used in medicine, in the manufacture of artificial camphor, terpineol, and terpine hydrate, as a solvent for pyroxylin in cheap varnishes and in other varnishes.

Many efforts have been made to remove from wood turpentine one of its distinguishing characteristics, namely, its peculiar odor, but so far with only partial success, as the odor often returns. Our own experiments indicate that this odor is due chiefly, if not entirely, to the heavy oils which wood turpentine usually contains. When these are removed by careful fractionation, wood turpentine consists almost wholly of the same constituents as gum spirits, which it also closely resembles in odor.

The first fractions obtained from crude steam-distilled turpentine resemble gum spirits very closely in odor and composition, but the yield of this portion is too small to justify the rejection of subsequent fractions. Hence, in separating from the crude oils all those that have approximately the same specific gravity and behave like turpentine when distilled, portions of the before-mentioned heavier oils distill and mix with the light oils and it is probably the presence of these heavy oils which accounts for the difference between gum spirits and wood turpentine. Destructively distilled turpentine with which the products of the distillation of rosin are mixed can not be freed entirely from rosin spirits and its accompanying odor. The following table shows the differences existing between commercial gum and wood turpentines in regard to those properties by which technical value is usually judged. The composition of wood turpentine is also shown by the analyses given in Table 1, page 58.

Constants of commercial gum and wood turpentines.

Determinations.	Gum spirits.	Steam distilled.	Destructively distilled.
Specific gravity at 20° C.	0. 8617 to 0. 8889	0. 859 to 0. 9150	0. 857 to 0. 898
Angular rotation at 20° C. in 100 mm tube.	-34. 8 to +29. 6	+16. 5 to +36. 14	+34. 4 to +77. 6
Index of refraction at 20° C.	1. 4684 to 1. 4818	1. 4673 to 1. 4755	1. 4666 to 1. 4810
Initial distilling point ¹	154 to 159° C.	153 to 177° C.	150 to 169° C.
Distilling below 170° C. per cent.	73 to 99	0 to 95	0 to 93
Distilling below 185° C. per cent.	88 to 99	20 to 98	61 to 97
Iodin absorption ²	350 to 400	300 to 362	300 to 398
Acid number	0. 140 to 0. 286	0. 080 to 0. 312	0. 028 to 0. 246
Saponification number	2. 44 to 8. 60	1. 06 to 8. 75	0. 65 to 4. 32
Color (Lovibond):			
Yellow	0. 7 to 2. 5	0. 5 to 10. 0	0. 4 to 4. 5
Red	0. 0 to 0. 5	0. 2 to 1. 4	0. 0 to 0. 8

¹ Emergent stem thermometer; uncorrected.

² Wjiss solution. Stood one-half hour; 200 per cent excess iodine.

From these figures it is quite evident that both steam-distilled and destructively distilled turpentines have many of the same general characteristics as gum spirits. A point of considerable interest, in view of the often-repeated complaint of painters that wood turpentine is strongly acid and because of this is very trying to the eyes, is the comparative acid numbers of the several classes of turpentine. There is no evidence to indicate that the acidity of wood turpentine is greater than that of gum spirits, though of course the acids may not be the same. The acid and saponification numbers are indicative of the amount of oxidation products which are present. These figures are not so striking, however, as is the iodine number discussed below. While it is true that the limits of specific gravity and of the quantity distilling below 170° C. do not differ widely among these three classes of turpentines, it is also true that the specific gravity will average higher and the quantity distilling below 170° C. lower in wood turpentine than in gum turpentine. In other words, the wood turpentine now on the market does contain too much pine oil distilling above 170° C., and to this fact, chiefly, is undoubtedly due the dissatisfaction which is expressed with reference to its drying properties.

The iodine number also throws light on the composition of the turpentines. It indicates that in certain cases the oil contains large quantities of constituents other than pinene or other terpenes of the same formula. In other words, the iodine number indicates practically what a distillation does—that is, the approximate quantity of heavy oils present. It will be seen from Table 2 that, compared with good gum spirits or with the light oils from crude wood turpentine, the heavy oils have a low iodine number; the iodine number therefore shows whether or not the sample contains considerable quantities of heavy oils, and also that these heavy oils are not of the same nature as the light oils. The same fact is shown in a general way by the saponification number. Those turpentines which have large residues that do not distill below 185° C. have high saponification num-

bers, or combine with much soda. This saponification is roughly proportional to the residue not distilling below 185° C.

The results on the crude oils (Table 3) differ from those on the refined products only in degree. The crude oils have somewhat higher specific gravities, absorb less iodine, and smaller proportions distill below 170° C., while the color is deeper than in the refined oils. The figures on crude oils show that as a rule they contain but little more heavy oils than the refined samples. In other words, but little has been accomplished in improving the product except by lightening the color. This is additional evidence that not enough care is exercised in refining.

PINE OILS.

"Pine oil" is the term applied to the heavier oils contained in crude wood turpentine. The term is more properly limited to the heavy oils of the steam-distilled crude oil. The heavy oils of crude destructively distilled wood turpentine are chiefly the heavier portions of rosin spirits and rosin oils.

Until very recently pine oil was almost entirely a waste product and the producer had some trouble in disposing of it. It is now finding more extensive use in paints, varnishes, and medicine, and in the manufacture of drugs and chemicals. Analyses of these oils, crude and refined, and also of refining still residues are given in Table 2, page 60. The samples which have been examined in this laboratory have specific gravities of from 0.889 to 0.944, a refractive index varying from 1.4765 to 1.4855; the initial distilling turpentine, when water is not present, ranges from 170° to 210° C. and the major portion generally distills between 200° and 215° C. The iodine values are much lower than those of the refined wood turpentine, while the saponification numbers vary about like those of gum spirits.

PRINCIPLES OF FRACTIONAL DISTILLATION.¹

When a mixture of liquids is distilled the vapors which arise always contain a larger proportion of the lighter or low boiling constituents and a smaller proportion of the higher boiling constituents than does the remaining liquid, but rarely, if ever, under commercial conditions, do they consist of these lower boiling constituents alone. The composition of the vapors which arise from the liquid bears a very definite relation to the composition of the liquid in the still. The greater the proportion of the heavy constituents in the liquid, the greater their proportion in the vapor. When an ordinary pot still, therefore, is employed even the first portion which passes over carries with it more or less of the higher boiling constituents. The proportion of these

¹ For extensive discussions of the subject of distillation see Hausbrand, "Verdampfen, Kondensieren, und Kühlen;" Rechenberg, "Gewinnung und Trennung der aetherischen Oele;" and Young, "Fractional Distillation."

heavier constituents thus carried over is primarily controlled by the proportion in the liquid. It is also greater the less the fractionating effect in the still. Fractionation, within limits, is decreased by increasing the velocity of the distilling vapors; that is, the more rapid the distillation, the smaller the space above the liquid in the still, and the smaller the opening to the condenser, the more of the heavy constituents the distillate contains. As distillation proceeds, the liquid in the still becomes richer in the heavier constituents and the vapors arising from this liquid also contain a larger proportion of these, although less than the remaining liquid, until finally all of the

lighter constituents are removed and the distillate consists wholly of the heavier ones. That is, in the distillation of a crude turpentine from a pot still, the first part of the distillate is a fairly light product, containing relatively small proportions of heavy oils. This first portion is followed sooner or later, depending on the proportion of light oils in the crude oil and the speed of distillation, by a product containing continually increasing proportions

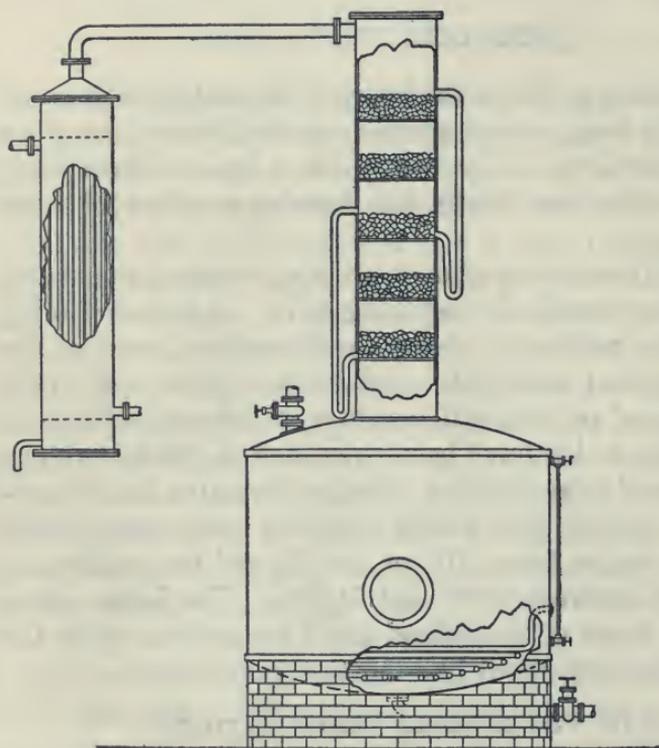


FIG. 1.—A simple periodic column still, with chambers containing broken rock.

portions of heavy oils, until at a certain point the product no longer contains light oils but consists entirely of what may be called the light pine oils mixed with small proportions of the heavy ones. As distillation progresses this product also gradually changes in composition and the proportion of heavy pine oils becomes greater until they form the entire distillate. For these reasons it is impossible to secure more than an approximate separation of crude wood turpentine into its several chief constituents by a single distillation from a pot still. It is necessary to make several redistillations of each of the intermediate fractions or portions which have been distilled between certain limits. Directions for doing this most satisfactorily are given on page 55.

The separation of the several constituents of a mixed liquid is most quickly, economically, and completely effected in a column still, several common forms of which are shown in figures 1, 2, 3, and 4. This consists essentially of a number of small, simple stills placed one above the other in the same column. The general statements made with reference to the liquid and vapor in a simple still are equally true of the contents of each still or chamber of the column still. The vapors above the liquid in the lowest chamber are richer in the

lighter constituents than is the liquid itself. These vapors pass upward through perforated plates or capped openings (see figs. 3 and 4) and, partially condensing, become the liquid in the second chamber, and the vapors in this chamber are in turn richer in the lighter constituents than is the liquid in the same chamber. This relation is repeated in each successive chamber until the vapors at the top of the column, in the earlier stages of the distillation, are practically pure or constitute a mixture of definite composition. If, when this point is reached, the heavier constituents of the liquid are not removed from the still, but the distillation is continued with rising temperature, these constituents pass upward into each successive chamber, obeying the same law as in distillation from the simple still. As

there are a number of successive stills, however, the mixed liquid when passed through a column still once is, as a rule, separated more completely into its several constituents than by several distillations in a pot still.

Several forms of column stills applicable to the refining of wood turpentine will be considered. Figure 1 shows one of the simplest forms. It consists of the still proper, surmounted by a fractionating column. The column consists of one or more chambers, containing broken quartz rock or other inert material, or of a number of chambers separated by plates, as shown in figure 2. The former is the

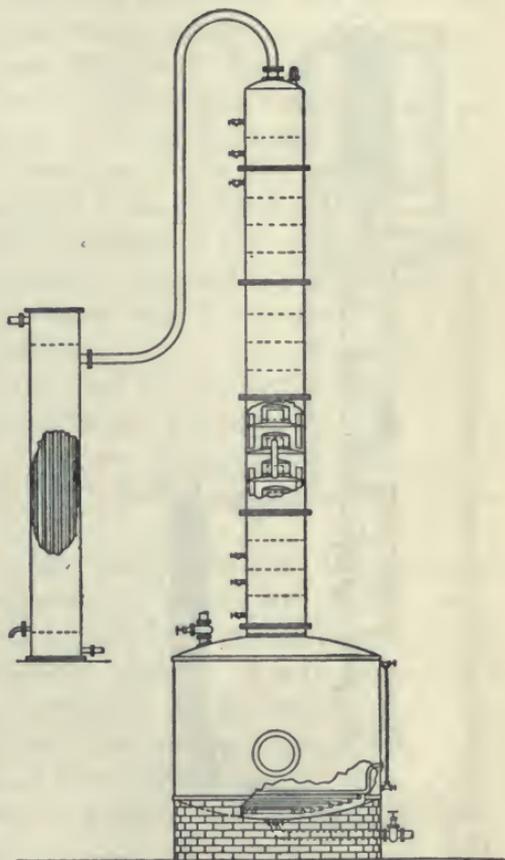


FIG. 2.—A periodic column still, with the chambers separated by hooded plates.

cheaper, but the latter is somewhat more efficient. Stills of the type shown in figures 1 and 2 are known as periodic; that is, they are not operated continuously, but distillation is discontinued from time to time, the residues emptied out, and the still recharged for another distillation. By careful and intelligent operation crude wood turpentine can be separated into three or four products of fairly definite proportions, each one passing in succession through the column and being condensed and stored in the proper receiving tank. It is

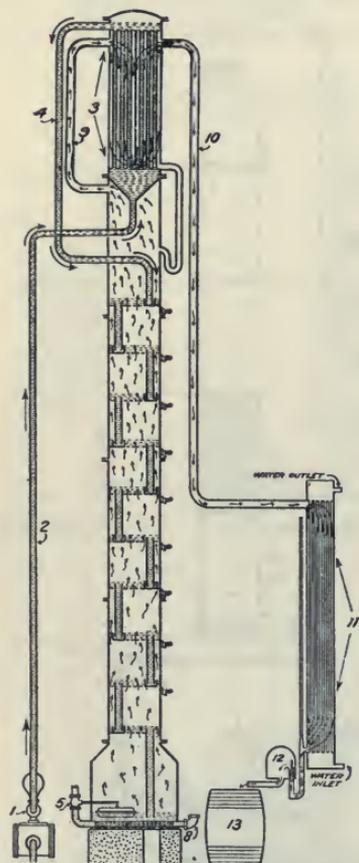


FIG. 3.—An ordinary continuous beer still used for turpentine distillation.

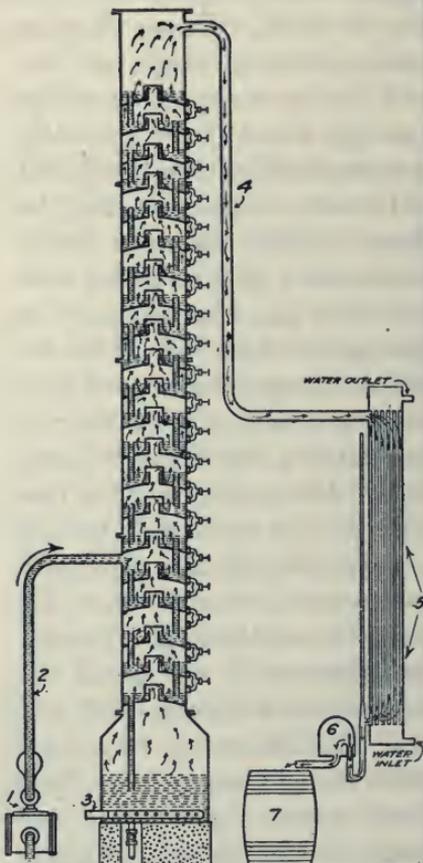


FIG. 4.—A continuous still with hooded plates.

preferable, however, to distill the wood turpentine through the column and the pine oils from a pot still, to which they are run from the bottom of the column still. Pine oils distill so slowly with steam that the use of a fractionating column in their case is not practicable. Periodic column stills are suitable only for small plants producing less than 600 or 800 gallons of crude oils per day, in which case the volume of the products does not warrant the installation of the more costly continuous still for the separation of three or more products simultaneously.

Two forms of the continuous still are shown in figures 3 and 4. These may be operated together to obtain three products, or individually to obtain two products. The crude turpentine enters the column through pipe 2 in a continuous stream. Here it comes in contact with live steam entering below and is carried upward where it is fractionated; the light oils of any desired composition, depending on the volume, speed, and temperature of the entering steam, pass continuously from the head of the column, are condensed, and run into storage tanks, while the heavier oils pass downward and out at the bottom of the still into a second and usually smaller column, or into a pot still from which they are again distilled. This distillate is pine oil of one or more grades and is heavier than that obtained from the first column. The condensed water and small quantities of other materials appear as waste at the bottom of the shorter column or remain in the pot still. The distillate from the first column should, under proper conditions of operation, contain all the wood turpentine and but little of the heavier constituents, such as the pine oils, etc., of the crude oils. These pine oils, if present, can be separated by a second distillation of the portion containing them. The composition of the first distillate may be largely controlled by the manipulation of the still, especially by the speed and the ratio between the steam and the crude oils entering the still. The pot still may be readily converted into a column periodic still by replacing the gooseneck with a suitable column.

It is highly important in turpentine distillation that the condensing surface be ample to fully condense the vapors and cool the condensed liquids to about 70° F. An insufficient condensing surface is too often a fault of wood distillation plants. It is to be borne in mind that in the South the water available for condensing and cooling has a higher temperature than that used farther north, and consequently larger condensers and more water are required. The consumption of cooling water may be greatly reduced by the use of large atmospheric condensers.

EXPERIMENTS IN REFINING WOOD TURPENTINE.

The purpose of refining crude wood oils is to separate them into their constituents or into fairly definite mixtures of commercial value. That portion known as "wood turpentine" should be similar to freshly distilled gum spirits as a paint and varnish thinner and be as free as possible from the very objectionable odor of the crude or imperfectly separated wood turpentine. Properly made wood turpentine should dissolve gums and resins as well as gum spirits, and when used in paints and varnishes they should be as durable and dry rapidly without pinholes and without crawling.

Further, it should be no more deleterious to the health of the workman than the gum spirits.

The refining experiments conducted in this laboratory have been controlled by determining the specific gravity, refractive index, initial boiling point, and behavior on distillation of samples drawn at stated times during the refining operations and of the several final products obtained from these operations.

USE OF THE PERIODIC COLUMN STILL.

The refining experiments on an industrial scale have been made with pot stills and two forms of periodic columns, which are shown in sections in figures 3 and 4. Figure 3 is an ordinary beer still of 10 chambers, such as is used in making alcohol, modified and adapted to the work. Both columns were used as periodic stills. The steam necessary to boil the crude turpentine is admitted to the bottom chamber of the column through the pipe 8. It boils the crude oils in this chamber and passes upward through one chamber after another, boiling the descending crude oil and carrying with it the turpentine vapors. The plates within the column are perforated so as to allow the steam to pass through them. The vapors rise to the upper part of this column and are conveyed through the pipe 9 into the heater 3, where they are circulated about the tubes containing the crude oil passing through the heater, and then are carried through the pipe 10 into the condenser 11 and thence to the separator 13 through the test box 12.

The crude turpentine is discharged from pump 1 and pipe 2 into the heater 3. This heater contains a series of tubes through which the turpentine is passed and around which the vapors coming from the boiling oils in the column on their way to the condenser are circulated. The heated turpentine leaves the heater through pipe 4 and reenters the column below the heater where it comes in contact with steam. The lower part of the column is divided by plates into a series of chambers, in each of which the turpentine is boiled and relieved of some of its light oils. The crude oil takes a downward course through the drop pipes and across each of the various plates. It should lose all of its light oils by the time it reaches the bottom chamber of the column, from which it is automatically discharged through valve 5, or subsequently distilled as pine oil, after the turpentine has been removed.

Figure 4 was also used as a periodic still in practically the same way. The crude oils should flow from a storage tank into the still through pipe 2. The column contains a series of chambers, upon each of which is carried about 3 inches of liquid. A constant level is maintained in each chamber by means of the drop pipes. The plates between the chambers are not perforated as in figure 3,

but the vapors ascend from one chamber to another through a pipe in the center of the plate, are deflected downward by a hood over the pipe, and forced to boil their way through the liquid on each plate by the steam which enters the still through the pipe 3. The arrows indicate the course of the vapor in the still. The vapors boil from chamber to chamber, becoming purer as they ascend until they reach the top chamber of the column, from which they are delivered through the pipe 4 into the cooler condenser 5, where they are reduced to a liquid. This cooling and condensing is effected by circulating cold water through the tubes around which the vapor passes.

The condensed vapor, turpentine, and water is drawn off at the bottom of the condenser and allowed to flow through the test box 6, where it can be examined. The steam pressure within the apparatus is registered by a pressure gauge not shown. The turpentine and water after passing through the test box is run into the separator 7, from which the turpentine may be run to storage tanks.

The general plan of the experiment was to draw off samples from time to time during the distillation, noting the temperature of the vapors escaping to the condenser and the relative volumes of turpentine and water in the condensed distillate, and making an examination of the samples, determining specific gravity, refractive index, initial distilling temperature, and behavior on distillation when 150 cc were distilled from an ordinary distilling flask, using an emergent stem thermometer. All of the turpentine obtained during a distillation was not run into the same tank, but at various predetermined periods it was directed into other tanks, and at the close of a distillation each fraction was measured, and the relative volume of water and turpentine determined. The residue of heavy oil was also measured, and samples from each tank were examined as just outlined. The results of the distillation and data obtained on the distilling oils from time to time during the process are given in Tables 4 and 5.

These data show the effectiveness of the separations at intervals during distillation and are in all respects similar to data obtained in the distillation of other closely related oils with steam in a column still. The first portions distilling consist almost entirely of light oil, but as distillation proceeds the proportion of light oil in the distilling oils decreases while the proportion of heavy oil increases until distillation is discontinued. The fact that the proportion of heavy oil begins to increase when the distillation is about half over is proof that large quantities of heavy oil were in the crude product. The first or A fractions of all distillations in the beer still were mixed together, and the A fractions of all distillations from the refining still were mixed together; each mixture was then again passed

through the still previously used. The data in Table 7 show that a second distillation gave a refined wood turpentine which complied with the most exacting requirements as to uniformity, specific gravity, and behavior on distillation; furthermore, the odor of this turpentine is much milder than that of the original crude oils, resembling quite closely that of gum spirits.

Table 6 contains all of the complete results obtained on the samples collected in the experiments in refining crude steam-distilled wood turpentine. The table is arranged on the basis of the specific gravity of the sample, that with the lowest gravity being entered first. From this table the successive samples of any distillation may be readily picked out. For instance 6A₁ and 6A₂ are the successive samples of the first fraction of the distillation of the sixth barrel and 9B₃ and 9B₄ are the successive samples from the second or B fraction of the distillation of the ninth barrel.

From the nature of the experiments, all of the data can not be strictly harmonious. Variations in atmospheric pressure occasion differences in distilling temperatures. Variations in steam pressure and volume occasion differences in distilling temperatures, and within limits in specific gravity and behavior on redistillation. On the whole, however, the data (see Tables 4 to 8) show that a low distilling temperature, specific gravity, and refractive index, together with a high percentage of turpentine in the steam distillate, are characteristic of turpentines of which 90 per cent distill below 170° C. It will be seen, furthermore, that when the percentage of oils in the distillate falls below 35 or 30 per cent these oils consist altogether of the heavier ones which distill above 170° C. Therefore, the conclusion drawn from Table 7 is that when the distillate contains 55 per cent or more of oils, these will comply with the requirements for a high-grade uniform wood turpentine, 90 per cent of which will distill below 170° C. When from 55 per cent down to 30 per cent of the distillate consists of oils, considerable quantities of light oils are present and the oils must be redistilled with steam, probably only once from a column still, to separate the light oils, which should, as before, constitute 55 per cent of the distillate. That portion of the distillate containing less than 30 per cent of oils contains only heavy oils, or pine oils, and need not be again distilled to separate oils boiling below 170° C. To this portion may be added the oils constituting less than 55 per cent of the distillate, of the redistilled middle fraction, or, if desired, this portion may be kept separate, as the oils are somewhat lighter than the heavy oils from the first distillation.

Table 8 contains the data concerning the several fractions obtained in the different distillations of the crude oils. These data are of the same general nature as those in Table 7. The temperature of distillation, proportion of turpentine in the distillate, specific gravity,

refractive index, and behavior on redistillation are in general harmony, as is to be expected. In general, the larger the percentage of the crude turpentine contained in the first or A fraction and the smaller the percentage of turpentine in the distillate, the higher the specific gravity and the smaller the quantity passing over below 170° C. on redistillation. The figures also indicate that those fractions of which fully 90 per cent passed over below 170° C. on redistillation constituted at least 54 per cent of the distillate from the refining still. Those fractions which constituted from 54 to 30 per cent of the distillate contained large quantities of heavy oils, and a second distillation was necessary to separate them and obtain a product 90 per cent of which passes over below 170° C.

The data under barrels 7 to 11A, 7 to 11 A+B, and 1 to 6 A (Tables 7 and 8) which were mixtures of first or A fractions, show that on a second distillation an entirely acceptable wood turpentine was obtained, and that approximately 92 per cent of the first fraction was light oil. Approximately 50 to 54 per cent of the crude oil was recovered in the first distillation as a high-grade wood turpentine. The highest figures, those on barrel 3, indicated that the crude turpentine employed did not contain more than 60 per cent of oils distilling below 170° C, while those on barrel 4, which are considered the most reliable, having been corrected for variations in pressure and temperature, indicate 54 per cent of light oil distilling below this temperature. This figure is probably correct within 2 per cent. That is, the crude oil contained from 52 to 56 per cent of light oils distilling below 170° C. The separation in a single distillation of a uniform commercial fraction containing 90 per cent of these $\left(\frac{90 \times 54}{100}\right)$ or 48.6 per cent instead of 54 per cent, may be considered a satisfactory separation, as it leaves in the heavy or pine oils 5 per cent of the original light oils. This is, as will be seen by consulting the tables, concentrated in a middle fraction constituting from 15 to 30 per cent of the original crude oil. Of this middle fraction 16 to 33 per cent is light oil, which can be almost completely separated by a second distillation with steam in a column still. This second separation will probably be sharper than the first, for the reason that by far the greater part of the heavy oils present in the crude oil has been removed, and of course the distillation is no longer affected by them.

USE OF THE POT STILL.

Similar, but less complete, experiments were conducted in Georgia using an ordinary gooseneck turpentine still for refining crude steam-distilled wood turpentine. These stills were charged with between 350 and 400 gallons of crude oils and refined with a slow current of live steam, from 10 to 12 hours being required to distill off the

wood turpentine from the pine oils. The distillate, consisting of water and turpentine, flowed from the condenser at a quite uniform rate of 1 gallon in from one minute to one and one-fourth minutes. As it was not feasible to measure the distillate, the refined wood turpentine, nor the pine oil recovered from the charge, these can only be calculated from the rate of distillation for the three experimental runs. These results are only approximate, but show roughly the quantity of heavy oils passing into the wood-turpentine storage tank.

The results obtained with the ordinary pot still having a goose-neck (Tables 9 and 10) agree quite well with those from the column still in indicating that the percentage of turpentine in the distillate must not fall below 54 if 90 per cent of the refined product is to distill below 170° C. Also that the portion of the distillate which contains between 54 and 30 per cent of oils is a mixture containing much pine or heavy oils, which should be again distilled to separate the turpentine from it; and, finally, that when the distillate contains less than 30 per cent of oils, these oils are not turpentine but pine oils.

In these experiments, the distillation of the so-called turpentine may be roughly divided into three periods; the first covering somewhat less than a third of the time, during which the light oils (90 per cent of which distill below 170° C.) are passing over; the second period covering from one-third to two-fifths of the total time, when a mixture containing larger and increasing quantities of heavy oils is distilling; and the last period, approximating one-fourth of the time, when heavy oils which should properly go to the pine-oil tanks are distilling.

Calculations based on the refining data indicate that the crude oils contained approximately 70 per cent of wood turpentine distilling below 170° C. Calculations from the factory records and the analysis of the refined wood turpentine showed 60 per cent of wood turpentine in the crude oils. The apparent discrepancy is doubtless due to the fact that no accurate measurements of volumes are available for calculation.

A comparison of the data in Tables 6 and 10 shows that the samples taken from the column still and from the pot still, when the percentage of turpentine in the distillate was the same, are much alike in composition. It must be remembered in comparing the data that the percentage of turpentine in the distillate can not be determined with great accuracy. The figures given may be in error 2 per cent. Further, the distillation of the samples from the column still were made with emergent thermometers and were not corrected for pressure.

On the whole, the data from the two forms of stills are very concordant. They agree in indicating that as long as the distillate from

The refining still consists of at least 54 per cent oils, 90 per cent of those oils will distill below 170° C.; that when the distillate contains from 54 to 30 per cent of oils, the oil is a mixture of light and heavy oils, which must be again distilled to separate the light oils distilling below 170° . When the distillate contains less than 30 per cent of oils, it contains practically no oil distilling below 170° . The specific gravity of the oils bears a fairly close relation to the percentage of oils in the steam distillate. When the percentage of oils in the distillate is 55 to 53, the specific gravity is 0.8634 to 0.8638; when the percentage of oils is 30 to 27 the specific gravity is 0.8811 to 0.8868 (omitting one doubtful result).

The data can not be employed to show definitely the comparative efficiency of the column and pot stills in refining wood turpentine. They do indicate, however, in a general way that the fractionation is sharper and that the intermediate fraction of the light and heavy oils is smaller from the column than from the pot still. It will be noted that in the earlier stages of the distillation from the column still, the distillate contained from 54 to 65 per cent of oils, while the distillate from the pot still contained only 54 per cent. This indicates, as has been said, a much sharper separation on the part of the column still during the first of the distillation. If the distillation were stopped at any given point, say when the percentage of turpentine in the distillate was 50, the separation in the column still would be more complete and the turpentine from it would contain less heavy oils than that from the pot still. It has been stated that from the column still a middle fraction containing about 5 per cent of the light oils that were in the crude oils is obtained. Calculations based on the rate of distilling indicate that the middle fraction from the pot still contains from 10 to 16 per cent of the light oils present in the crude oils, and that it is larger than the middle fraction from the column stills.

Experiments similar to the foregoing were also conducted with destructively distilled turpentine, and results of the same general nature as to the separation from the heavy oils, percentage of oils in distillate, etc., were obtained. The stills used in this work became unavailable at this time and therefore no effort was made to separate the light oils, distilling below 150° C., other than to make three fractions of the distillate. The first fraction should have contained the lighter oils with but small proportions of turpentine, the second fraction the wood turpentine, and the third the pine oils, or, more properly, the heavier constituents of rosin spirits. The separation between the light oils and the turpentine was not at all sharp, and a single fractionation in the column still did not give in any case a fraction which contained all the light oils, with but minor percentages of turpentine.

If distillation of the first fraction was continued sufficiently long to carry over all the light oils, it contained 50 per cent or more of turpentine, or if the distillation of the first fraction was discontinued when it had but a small proportion of turpentine, it did not contain all the light oils.

Between the turpentine proper and the heavy oils there was also a mixed portion, as in the case of the steam-distilled turpentine, and this contained notable percentages of light constituents. It is quite evident, therefore, that, when operating these stills in the manner described, it is necessary to make a second distillation of two middle fractions, one to separate rosin spirits from turpentine and one to separate turpentine from the heavy oils. Under the circumstances, it is not deemed advisable to include the detailed figures on the distillation of the destructive turpentine.

CONCLUSIONS IN REGARD TO REFINING.

From the data obtained in these experiments it is concluded that the percentage of oils in the distillate furnishes the most reliable and useful information as to the progress of distillation and the nature of the oils at different times, and this is also the simplest means of acquiring such information. Neither the specific gravity of the oils nor the temperature of the distilling vapors at the top of the still furnishes as reliable information, nor are they so conveniently determined. The temperature of the vapors is materially affected by the volume and temperature of the steam entering the still and by variations in atmospheric pressure. The former can of course be made practically constant by the use of reducing valves, but corrections for atmospheric pressure require more training, experience, and care than can probably be given at such plants.

The specific gravity of the samples taken during distillation often shows a decided conflict with the other data on the samples, but in the main it is a reliable indication of the progress of the distillation and the composition of the oil. But as this determination also requires more skill, and is in every way more difficult to make in the works, it is not as safe a guide as the volume relations of the distillate.

The column still will give sharper separations of wood turpentine from pine oils than can be obtained with the pot still, but the experiments here described do not indicate any great superiority of the column. The data available, however, are inadequate to permit the drawing of definite conclusions as to the economy or efficiency of the two forms of stills.

VARNISH AND PAINT EXPERIMENTS.

ANALYSES OF THE TURPENTINES AND VARNISHES EMPLOYED.

In order to have a strictly comparable series of experiments on the value of wood turpentine as a thinner, varnishes and paints were prepared and thinned with several different turpentines. That there might be no question as to the authenticity of the samples, the author personally collected them and was present when the paints and varnishes were made. Four turpentines, such as are usually found on the market, were used in these experiments—a gum spirits, a steam-distilled turpentine, and two samples of destructively distilled turpentine, both of which had been distilled after washing with soda. None of the wood turpentine was as good as can be prepared by careful refining; all except No. 3 contained much heavy oil which gave them a marked odor and undoubtedly made the varnishes dry more slowly. Results with these turpentines, therefore, would naturally prove more unfavorable to wood turpentine than if the properly refined article had been used. Analyses of the turpentines used in these experiments are given in the following table:

Analyses of the turpentine used in varnish experiments.

Determinations.	Gum spirits.		Steam distilled and refined.		Destructively distilled, washed with soda, and steam distilled.	
	No. 1.	No. 2.	No. 3.	No. 4.	No. 3.	No. 4.
Specific gravity at 20° C.	0.866	0.871	0.860	0.857		
Angular rotation at 20° C.	+4.20	+33.4	+11.5	+16.4		
Index of refraction at 20° C.	1.4698			1.4723		
Initial distilling temperature °C.	155	156	154	162		
Distillation tests:						
Distilling below 160° C. (per cent)	82	26	63	0		
Distilling between 160° and 170° C. (per cent)	13	48	29	65		
Distilling between 170° and 185° C. (per cent)	2	12	2	30		
Residue above 185° C. (per cent)	2	13	5	4		
Total iodine absorption	431	362	398	352		
Iodine absorption, addition	335	294	322	282		
Iodine absorption, substitution	48	34	38	35		
Acid number	0.347	0.270	0.110	0.040		
Saponification number	3.41	7.72	3.92	0.9		
Color in 200 mm columns:						
Yellow	0.74	5.0	1.60	0.84		
Red	0.2					
Polymerization residue (per cent)	<1	<1	<1	<1		

It will be noticed from these analyses that according to the distillation tests the gum spirits and one of the destructively distilled turpentines should be ranked as quick-drying turpentines, with the advantage in favor of the gum spirits; while the steam-distilled and the other destructively distilled turpentine are slow-drying, the latter being the slower in the initial stages, but the former requiring longer to dry hard. It should be noted that 74 per cent of the steam-distilled turpentine distills below 170° C., i. e., 26 per cent of the tur-

pentine is nonvolatile at that temperature; yet, as will be seen subsequently, the varnishing tests do not indicate that the varnish thinned with this turpentine took longer to dry than the others. Some observers reported that this varnish dried more slowly than the other samples, while others stated that it dried fully as fast. The comparatively low acid and saponification numbers of the turpentine refined from soda are to be expected. The steam-distilled sample is of deeper color than the others, but no difference can be detected in the color of the varnishes, which can be ascribed to the color of the turpentine used. While the analyses given indicate that there is a marked difference in the behavior on distillation between the several turpentines, this difference is no greater than that frequently observed between samples of gum spirits.

Two classes of varnishes were prepared and thinned with these turpentines; a coach finishing varnish to represent outdoor conditions, and a piano varnish to represent those used in interior work. These were prepared by two experienced makers under the writer's personal supervision, in accordance with their regular formulas for such varnishes. In preparing them the gums and oils were cooked in the usual way, and when sufficiently cooled portions of the batches were thinned to the desired consistency with each of the described turpentines. For comparison, in order that there might be no possible differences due to a variation in manufacture, a portion was also thinned with the turpentine regularly used by the firm. These varnishes were allowed to age for one year at an even temperature, when they were racked off and half-pint samples sent to prominent varnish makers, piano makers, and carriage builders, who had consented to make panel tests of the working qualities and to pass judgment on the merits of the different varnishes.¹ Small quantities of the turpentines were sent with the different varnishes that they might be thinned before using, if necessary, with the same turpentine used in its preparation. The firms which made these tests also prepared a panel with the varnish they regularly used for each class of work. There were thus standard varnishes thinned with gum spirits, two of which were identical, except as to the turpentine, with the three thinned with wood turpentine, while the sixth varnish was that with which the various varnishing workmen were familiar and with which they were securing satisfactory results. Very complete data were requested as to the working, behavior under the brush, tendency to check or peel, time of drying, difference in gloss, behavior when rubbed, disagreeableness to workmen, and other pertinent facts, all of which will be found in Table 11, page 69.

Extended analyses of the varnishes used in these tests has not been attempted because they would possess but little significance. It is in

¹ It was assumed that the finished varnishes made from the same batch of oil and gum would have practically the same consistency; this was not the case.

the preparation of the varnishes that the difference in odor between gum turpentine and wood turpentine is most strongly brought out, as it is much more pronounced at this stage than in the cold turpentine or in the completed varnish at ordinary temperatures, though here, too, it is quite easy to differentiate the several varnishes. The odor of the hot varnish thinned with the steam-distilled turpentine was much more objectionable to the writer than that of the varnishes thinned with the destructively distilled samples. In the former case the odor can be best described as resinous and nauseating, while in the latter case it was resinous, penetrating, and smoky.

The viscosity of the varnishes was determined by the Doolittle viscosimeter and found to be as follows, when expressed in angular degrees of retardation:

Viscosity of three kinds of varnish (expressed in angular degrees).

Varnish No.	Coach finishing.	Piano rubbing.	Piano flowing.
1.....	135.7	117.0	69.4
2.....	69.1	121.7	64.7
3.....	72.9	97.5	62.0
4.....	62.1	78.7	50.2
	61.5		
5.....	91.4	96.2	80.5
	92.2		

That is, the different samples of the same kind of varnish were not at all alike in consistency. This fact has but little significance, however, except that it made it necessary for each workman to thin the varnishes himself before using them. It is more than probable that this would have been done in any case and the final results do not indicate that the differences in consistency had any practical effect on the finished coat. It will be seen from the following table, and from Table 11, page 69, that the experimenters seldom thinned the varnishes in conformity with their determined viscosity. Neither is there any apparent connection between the turpentine content, the thinning required, and the time of drying of the different varnishes.

Order in which the varnishes should be thinned.

Coach finishing.	Piano flowing.	Piano rubbing.
No. 1	No. 5	No. 2
No. 5	No. 1	No. 1
No. 3	No. 2	No. 3
No. 2	No. 3	No. 5
No. 4	No. 4	No. 4

The percentage of volatile matter distilled from the varnishes is shown in the following table:

Percentage of volatile matter distilled from the various varnishes.

Various thinners used.	Coach varnish.	Piano rubbing varnish.	Piano flowing varnish.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
Gum spirits.....	59.4	64.3	53.
Steam-distilled wood turpentine.....	45.8	64.0	54.
Destructively distilled, soda-refined turpentine, 3.....	51.1	65.0	57.
Destructively distilled, soda-refined turpentine, 4.....	47.7	63.4	55.
Gum turpentine.....	51.6	66.9	53.

Perhaps the only point worthy of attention here is the large amount of volatile matter contained in the first coach varnish thinned with gum spirits. This is the varnish that has the highest viscosity and required the greatest amount of thinning.

The color of all the varnishes was determined, using the Lovibond colorimeter. Readings were made in the $\frac{1}{4}$ -inch cell. All varnishes were found to have the same amount of yellow, the only variation being in the red readings.

Color determinations on varnishes thinned in different ways (Lovibond readings).

Thinners used.	Coach varnish.		Piano rubbing.		Piano finishing.	
	Red.	Yellow.	Red.	Yellow.	Red.	Yellow.
Gum spirits.....	11.45	15	9.80	15	11.90	15
Steam distilled and refined.....	9.80	15	9.80	15	11.90	15
Destructively distilled and soda refined, 3.....	9.60	15	10.60	15	12.00	15
Destructively distilled and soda refined, 4.....	10.05	15	9.75	15	11.40	15
Gum spirits.....	10.15	15	9.90	15	10.60	15

No useful conclusion can be drawn from the color tests. The data furnished by the various experimenters are brought together in Table 11.

COMMENTS OF THOSE WHO TESTED THE VARNISHES.

Murphy Varnish Co.: We have reached the conclusion that the coach and piano finishing varnishes have been mixed. If it is so, our tests, so far as drying and rubbing properties are concerned, are valueless. Generally speaking, we are of the opinion that the samples you sent us are poor varnishes; poor as to materials and poor as to methods of manufacture.

A. B. Chase Co.: In our opinion these varnishes (piano) were prepared and thinned with a turpentine substitute. When these varnishes were polished they did not come up to our expectation, so we concluded they were gloss varnishes and simply gave the panels another coat and left them with a gloss finish.

The James & Meyer Buggy Co.: As far as we can judge without giving them one year's test in the open weather, No. 1 and No. 3 turpentines could be used by us about the same as regular turpentine. No. 2 and No. 4 could not be used at all, as they crawl apart under the brush.

Steinway & Sons: None of the varnishes is as good as that regularly employed by this firm.

Vose & Sons Piano Co.: We attempted to make the panel tests, following out your instructions, and are sorry to report that for all purposes the samples sent us were entirely impracticable. Some of the panels, in fact nearly all of them, still remain tacky after the considerable length of time since it was applied. It is entirely impossible to rub them.

DISCUSSION AND SUMMARY OF THE VARNISHING EXPERIMENTS.

COACH-FINISHING VARNISHES.

All experimenters thinned one of the varnishes made with gum spirits, three thinned all varnishes the same, and one thinned both gum-spirits varnishes. The other varnishes were not thinned.

The time that the varnishes remained tacky varied from 3 to 36 hours, and the results do not show that the wood turpentine behave materially differently from the gum spirits in this respect. The varnish thinned with the wood turpentine containing the most heavy oil (No. 4) does not remain tacky materially longer than the others.

The same statements apply to the time which is required for the varnish to dry hard; some found that all dried in 8 to 12 hours, others reported 12 hours, 24 hours, and 54 hours. Three experimenters reported the same drying for all the varnishes.

Three reported a difference in gloss; one, that the varnish thinned with the destructively distilled turpentine (No. 4) had the best gloss; another, that the gum was superior to the wood turpentine, and the third, that all the wood-turpentine-thinned and one gum-thinned varnish showed tears.

All observers detected the wood turpentine by the odor, and found the varnishes thinned with them objectionable because of this.

Only one observer raised any other objection to the varnishes; this one found the wood turpentine varnishes light in body.

Only one observer found any difficulty in working the varnishes; in this case the gum turpentine and the destructively distilled wood turpentine (No. 4) having the least residue were found objectionable.

In rubbing, one observer found all unsatisfactory; two found all satisfactory, and the others did not report specifically.

As to behavior under the brush, the results do not lead to any conclusion, as different observers express contradictory opinions as to the behavior of the same varnish. This is a working condition, however, which probably has but little to do with the nature of the varnish. Two observers find one of the gum-turpentine varnishes rather heavy in body; one finds the wood-turpentine varnishes thin and light in body; one finds that the varnish thinned with the destructively distilled turpentine (No. 3) loses luster, that all of them are silky in appearance, and that the one thinned with the destruc-

tively distilled turpentine (No. 4) leaving the least residue is the best. Another observer finds in striping that one destructively distilled turpentine (No. 3) works like regular turpentine, while the steam-distilled (No. 1) and the destructively distilled turpentine leaving the largest residue (No. 4) crack and crawl apart. This observation does not appear to have been made by the other experimenters and would seem to be connected in some way with the pigment present in the material used by this experimenter. It will be noted that these are the samples that contain large percentages of heavy oils not distilling below 170° C.

PIANO-RUBBING VARNISHES.

One experimenter did not thin the varnishes, one thinned one gum-turpentine varnish only, three thinned all varnishes alike, and two used different quantities of turpentine in thinning the varnishes.

In some instances the varnishes thinned with the wood turpentine remained tacky longer than the varnishes thinned with gum spirits; in others the reverse was true. In no case did the thinning agree with the viscosity tests of the varnishes. The time of drying varied but seemed to bear no relation to the kind of turpentine used in the varnish or to the viscosity of the original varnishes. In some instances the wood turpentines were much slower than the gum turpentines, while in others there was reported no practical difference in this respect.

With regard to luster, three found some difference among the varnishes. One reported that the steam-distilled wood turpentine (No. 2) had the best luster, the destructively distilled (No. 3) being second best. Another found one gum and one wood turpentine-thinned varnish had medium gloss, and one gum and one wood turpentine-thinned varnish had fair gloss. The other observers distinguished no difference in this particular. One found that the steam-distilled turpentine (No. 2) gave the best gloss, while one gum (No. 1) and one destructively distilled product (No. 3) showed tears.

Two found the odor of all the wood turpentine objectionable; one found the steam-distilled (No. 2) and one destructively distilled (No. 4) objectionable. One found both destructively distilled samples objectionable, while another said that the No. 1 gum-turpentine varnish had the odor of naphtha.

No difference was detected in working qualities, except that one observer found that the varnishes thinned with the destructively distilled turpentine (No. 3) worked unsatisfactorily.

All rubbed down well, one observer reporting in favor of the gum spirits. Three observers found no difference in behavior under the brush, while two others found some difference. One reports sweating.

As to body, there appears to be no difference that can be correlated with the turpentine used. One firm expresses the opinion that all the varnishes rub better than the varnish they regularly use; another, that only time and exposure make it possible to determine accurately the value of the varnishes; another, that they find it very difficult to form an opinion on the merits of the varnishes. The wood turpentines are said to contain very strong elements of resinous spirits, from which the best results can not be obtained.

PIANO-FINISHING VARNISHES.

One experimenter thinned both gum turpentine varnishes and that made with steam-distilled turpentine (No. 2); another thinned only one gum spirits sample. Two observers did not thin the varnishes, and two thinned all alike. In no case was the thinning done in the order of the viscosity of the several varnishes.

The varnishes remained tacky for from 4 to 55 hours, but there was no relation between this condition and the viscosity of the varnishes or the distillation tests of the turpentines.

The data do not indicate that there is any difference in the time required to dry hard. No difference in gloss was reported, except in one instance a gum turpentine varnish is stated to have the poorest gloss. One observer finds tears in Nos. 1 and 3.

In one case the odor of the destructively distilled, soda-refined sample was reported as objectionable; another objected to the odor of both destructively distilled samples Nos. 3 and 4, and a third observer detected all the wood turpentines, and stated that both of the gum spirits varnishes had an old fatty oil odor. The odor of the steam distilled and one gum turpentine was found objectionable in one case and in another both destructively distilled samples were so reported.

Only one observer found any difficulty in working the samples, the destructively distilled wood turpentines being difficult to handle.

None of the varnishes was found satisfactory in polishing, except by two observers.

Two observers found that all of the varnishes flowed freely from the brush; one that there was some difference. One observer found all the varnishes unsatisfactory, as they did not flow out well. The one thinned with gum spirits (No. 1) was best, the varnish thinned with destructively distilled turpentine (No. 3) was rated second, while the steam distilled (No. 2), the other destructively distilled (No. 4), and the other gum spirits (No. 5) varnishes were found to be poor. No choice could be made between the samples. The same observer remarks that in his opinion all these varnishes were prepared and thinned with turpentine substitutes. Another observer found all the varnishes unsatisfactory as polishing varnishes, the two destructively distilled samples being the worst. Another ob-

server finds the samples from fair to good in all respects, with those thinned with gum spirits the best in every respect.

All but one of the experimenters recognized that No. 1 coach varnish was the most viscous, and thinned it more than they did the other coach varnishes. One also thinned No. 5, which was the next most viscous varnish. None of the experimenters was able to detect working differences in the consistencies of the other samples of coach varnish. Three experimenters apparently attempted to thin the piano rubbing varnishes to approximately the same consistency; only one, however, approached the order indicated by the viscosity determination. Considering the results as a whole, it is not possible to state that any of these varnishes behaved materially differently from the others either in drying or under the brush. The observations of two experimenters are in harmony with the viscosity tests, but those of the others are not. Where a difference in luster was observed the wood turpentine were best.

None of the experimenters thinned the piano flowing varnishes in the order indicated by their viscosities. On these varnishes also the data do not warrant any conclusion as to the differences in their behavior on application, and no practical difference in gloss was observed. These, being flowing varnishes, were not intended to be rubbed, but all of the experimenters, through misunderstanding, rubbed the panels, usually with very unsatisfactory results.

In making a careful study of the mass of data obtained in this work one is at once impressed with the many different ways in which these varnishes were handled from the thinning to the finishing touches, and by the seemingly contradictory opinions which are drawn regarding them. There is no one point on which a decided majority agree except as to odor, and even here some do not object to the wood turpentine, while others complain of the gum spirits. So at variance are the reports that the conclusion is almost forced upon one that a workman's opinion of a varnish is largely influenced by whether or not it may be manipulated as he is used to handling varnishes under his working conditions. Other factors influencing the quality of varnish do not enter here because these samples are identical except as to the character and amount of thinner used. The above conclusion is not only supported by the contradictory reports, but also by the statements made by some that none of the varnishes was satisfactory.

It is rather surprising that no marked differences between the individual samples of the same class are definitely indicated in the reports of the several workmen. The determined viscosities of the several varnishes of a class indicate the order in which they should be thinned to bring them to the same consistency, or make them flow alike. The turpentine employed differed greatly, however, in their drying properties. The analyses indicate that Nos. 1 and 3

were quick-drying turpentine, while Nos. 2 and 4 were slow drying. In general, therefore, more of turpentine Nos. 2 and 4 would be required to thin a varnish to a desired consistency than of Nos. 1 and 3, but the resulting varnish should be slower in drying. It will be observed as a matter of fact that No. 4 varnish, in which destructively distilled turpentine was used, is the thinnest varnish of all and it was unnecessary to thin it further.

On the whole, no definite conclusion can be drawn from the data obtained by the experimenters on the handling and working of the varnishes. This is not a criticism of those who at considerable pains and inconvenience cooperated in the work. The unsatisfactory character of the data arose in part through the difficulty of explaining clearly in written directions exactly what experiments and what information were desired. Had it been feasible for a representative of the bureau to talk with and assist those actually doing the work, more valuable information could doubtless have been obtained. Of course the data could only be of value in determining the relative working qualities of the varnish thinned with the different turpentine; but little information as to the appearance and durability of the varnish was obtained. Opinions on this point must be based on a later careful examination and study of the finished panels. These panels were prepared four years ago. Since that time they have been exposed in one of the office rooms of the bureau for from 12 to 18 months, and during the rest of the time have been stored, protected from dirt and light. None has been exposed to the weather at any time.

Careful examination of these panels by a number of persons, including master painters and others familiar with the use of varnishes, discloses no marked deterioration of any of the coats nor any clear-cut difference between the appearance of the several varnishes of the same class, except that as a rule the varnish which the workman was in the habit of using regularly looked a little better than the others. This fact confirms the statement previously made that the familiarity of the workman with the varnish influences the success of his work, and, consequently, his opinion of the varnish.

In addition to these experiments the same turpentine were employed in thinning a white lead and linseed oil paint. No difference was observed in the working of the paint, and after drying all painted panels presented the same appearance. There was no difference in the color of the panels after one year. The interior woodwork of the new building of the Bureau of Chemistry was painted with zinc oxid and linseed oil thinned with other steam and destructively distilled turpentine. The workmen stated that the odor, particularly of the destructively distilled samples, was unpleasant, but it did not seriously inconvenience them or make them ill. Both

turpentines (also a light pine oil) behaved well under the brush and no objectionable features in working other than the odor were observed. After 18 months there is no apparent difference in the paint thinned with the different turpentines.

About five barrels of refined wood turpentine, which had been obtained in the refining experiments, and of which approximately 75 per cent distilled below 170° C. were turned over to the painter of the department and used by him on all classes of paint and varnish work from ordinary house painting to high-class furniture varnishing. No inferiority in the finished work or in behavior in applying was observed by the master painter. Only one workman made complaint regarding this material, claiming that it made him ill. The statement was not investigated.

COMMERCIAL OPINIONS AS TO THE VALUE OF WOOD TURPENTINE.¹

Before entering upon the research work on this subject the opinions of those having occasion to use turpentines in large quantities was asked as to the value of wood turpentine as compared with gum spirits.

OPINIONS OF WOOD TURPENTINE PRODUCERS.

Cheraw Naval Stores Co. (Inc.), Cheraw, S. C.: Our product is used largely by paint men in the South, * * * who claim that it is better paint spirits than the old process turpentine.

F. L. Huggins, Wilmington, N. C.: Wood turpentine dries better than ordinary turpentine. * * * The only difference is in the odor.

Morris Weslosky, Albany, Ga.: Its solvent power is as great, it dries quicker, and varnish made from it is no more difficult to work than that made from ordinary turpentine, but is more liable to check. Painters object to the odor and state that the acid that seems to be left in it hurts their eyes. Consumers object only to the odor.

Weed Distilling & Manufacturing Co., New York: We have heard no objection to our products which are taken by the trade at the market rates. The oils for varnish work are more readily sold every day.

Chesterfield Naval Stores Co., Cheraw, S. C.: No objection has been raised to the material.

Tyler Lumber Co., New York: We find that the solvent property of wood turpentine by the steam process is greater than that of gum turpentine. It will not dry as quickly as gum, taking about two hours longer. It has been claimed that for carriage varnish, etc., the varnish is not so flexible as when gum spirits are used. The only complaint of any moment is made by painters and other consumers on account of the odor which is objectionable for interior work where the building is closed.

Pensacola Tar & Turpentine Co., Pensacola, Fla.: The solvent power of wood turpentine is the same as the old fashioned article. It dries as well, and there is no difference in its working qualities. The only objection is that the odor is unpleasant. Its power of absorbing oxygen seems to be equal to any turpentine.

Naval Stores Supply Co., Biscoe, N. C.: We have been selling practically our entire output to users of paints, and we suppose that this turpentine was used exactly as the

¹ These statements are not in all cases quotations but are condensed from correspondence. The Bureau of Chemistry is not in any way responsible for these opinions.

turpentine made by the old process. The only objection that we have heard is with regard to the odor.

Spirittine Chemical Co., Wilmington, N. C.: The solvent power is the same. It dries as well and there is no more liability for varnishes thinned with it to check than those made with gum spirits. There is a difference in odor, a slight difference in color, but neither enough to injure its usefulness. We have as customers for this product many of the leading paint and varnish manufacturers and they have been buying to their complete satisfaction for many years.

OPINIONS OF PAINT AND VARNISH MAKERS.

Murphy Varnish Co., Newark, N. J.: Our tests of this material have shown that it is not so well adapted to varnish-making purposes as pure spirits of turpentine. It is objectionable on account of the odor and also because less of it is required in the manufacture of gum varnishes than the pure spirits of turpentine. As turpentine is the cheapest ingredient it is obvious that the cost of such varnishes is increased by its use.

The Glidden Varnish Co., Cleveland, Ohio: We are experimenting with wood turpentine continually, but up to the present time have been unable to find any product that was entirely satisfactory in odor. The proper deodorization of wood turpentine would no doubt make this product as valuable as pure gum spirits. In all of our experiments with a high-grade wood turpentine we find that the working properties of same are equal to those of gum spirits, but wood turpentine gives varnish or paint a very pungent odor which is very disagreeable to the user.

Harrison Bros. & Co. (Inc.), Philadelphia, Pa.: Properly distilled wood turpentine free from pyroligneous impurities has equal or better solvent properties than gum spirits. It dries as well, but possibly a little slower. Varnish made with it is in no way inferior to one made with gum spirits so far as working and physical properties are concerned. The principal objections raised by painters and consumers are the odor and irritating action of the vapors upon the eyes, while some painters claim that it also produces nausea.

Chicago Wood Finishing Co., Chicago, Ill.: The solvent power of wood turpentine is equivalent in every way to gum spirits. In drying it is not so much slower than there would be any great disadvantage. Mixed with varnishes we find that it is equivalent to gum spirits when mixed in any quantity of almost any kind of varnish in which a gum spirit would be used. It does not differ, as far as we have seen, in working qualities from gum spirits. The principal objection is its extremely pungent odor which, to most people, is very disagreeable, and in some cases causes violent headaches. Because of this odor we prefer the gum spirits.

Longman & Martinez, New York City: The best quality of wood turpentine evaporates much slower than gum turpentine, and were it not for the odor of wood turpentine it would probably be as well suited for use, to as large an extent, in paints as is gum turpentine. Neither is of any value whatever in respect to furnishing durability to paint. The odor of even a good quality of wood turpentine is not so often an objectionable feature.

Atlantic Drier & Varnish Co., Philadelphia, Pa.: The solvent power is equal. It dries as well and does not differ in manipulation from ordinary turpentine. It does not make an inferior varnish, and paint or varnishes made with it do not differ perceptibly in covering power from ordinary turpentine. Painters, consumers, and workmen all agree in objecting seriously to the strong odor and to its bad effects on the eyes. It is very much more offensive in every way than ordinary turpentine. We have found such serious objections because of the odor that we have abandoned the use of the material entirely.

Boston Varnish Co., Boston, Mass.: We have not gone into the flexibility or life of wood turpentine, but in our opinion from what we have used it is equally as good as

regular turpentine. In our opinion a varnish reduced with it would produce a product that would be absolutely impossible for any painter to use in any quantity in a close room. The principal objection to it for medium and high-grade varnishes is its odor. The above remarks apply only to the pure, properly made, and well-refined article.

C. A. Willey, New York City: From careful experiments in a practical way I am fully convinced that it will do everything that an ordinary turpentine will do, but the trouble is with its odor, of which the painters immediately complain so that we were obliged to discontinue its use on this account. Recently there is much improvement in the odor of some samples.

Charles H. Howell & Co., Philadelphia, Pa.: From some little experience we have come to the conclusion that barring the smell, the dark color of that which has simply been distilled, it is a good product, answering all purposes of gum turpentine, with only the objectionable odor against it. Our experience with the clear white article which we think is produced by treating with caustic is that it is utterly worthless, being very detrimental to paint, and especially to oil paint. Such treatment takes the greater part of the oil out and leaves an article no better than benzine. It contains a strong alkali which forms a soap of the oils in paints, and kills their durability.

Standard Varnish Works, New York, N. Y.: For our requirements we find this article to answer the same purposes as spirits of turpentine, the only noticeable difference being the odor, to which the trade seriously objects.

Berry Bros. (Ltd.), Detroit, Mich.: We do not and never have considered distilled turpentine as being equal in any respect to old process goods.

Emil Calman & Co., New York, N. Y.: We find that for many purposes it will replace the gum article. Its solvent power appears to be about the same and when properly manufactured it will dry as well as ordinary turpentine. We do not find any material difference in working qualities. There is objection from buyers of materials to the odor, which is stronger than that of gum turpentine. These remarks apply only to properly manufactured wood turpentine.

Boykin Manufacturing Co. (Inc.), Cheraw, S. C.: We have been marketing wood turpentine to the paint, furniture, and buggy manufacturers with success. We rarely have a kick on account of quality, and this class of trade claims that the wood turpentine does their work as well as the old process turpentine does.

The Institute of Industrial Research, Washington, D. C.: The solvent power of wood turpentine is high and is probably not less than that of gum spirits of the same specific gravity. When properly prepared and purified, it dries as well as gum spirits. From this point of view it is therefore a good varnish material and is said to work free and exhibit good covering power. The principal objections to wood turpentine are made on account of its unpleasant odor and lack of uniformity. It contains, if improperly purified, certain * * * products which are said to have a bad effect upon the kidneys of workmen who have to handle it. These properties when present also make it unfit for interior application.

John Lucas & Co., Philadelphia, Pa.: Perhaps definite or practical information on the subject of the commercial value of this product in comparison with the gum turpentine is not of a very reliable character. Experiments made or experience gained may not have been such as to fully determine the point. We have heard that varnish made from a wood product would, when applied over a varnish made from gum spirits, on account of its higher solvent nature, dissolve the surface of the previous coats and render the rubbing down almost impossible. In drying we have not noticed any particular difference. It has been our experience that a good grade of wood turpentine is equal in every respect to the gum spirits. A varnish properly made with wood turpentine of a satisfactory grade has just as good rubbing qualities as the gum-spirits varnish. The principal objection is its odor.

John W. Masury & Son, New York City: Aside from its objectionable odor there seems to be no reason why wood turpentine should not be used in paint as well as the gum turpentine. In the ripening or aging of varnish turpentine as well as linseed oil it absorbs some oxygen, * * * whereby the valuable properties of the varnish increase. * * * Here no solvent which does not have the capacity of such change can replace turpentine, but so far as we have been able to observe there is no great difference between wood and gum turpentine. We believe, therefore, that its pungent characteristic odor is the main obstacle to its universal substitution for the genuine in paints and varnishes.

As a problem apart by itself, however small may be the demonstrable scientific difference between wood and gum spirits, the conservative varnish maker will prefer the gum spirits. This very natural prejudice arises from his consciousness of unknown chemical relations in a process which for some time to come must remain largely empirical, and as his good reputation rests largely upon results so achieved he does not willingly take chances for the fraction of 1 per cent gain which the difference in price between the two turpentines has heretofore offered.

So far as paint is concerned the situation is clearer. Here turpentine is a volatile thinner, used to obtain a practical brushing consistency where it is desirable to keep the ratio of oil to pigment low.

Sherwin-Williams Co., Newark, N. J.: Our experience with the material is limited, but so far as we know the solvent power is not materially different. Careful tests have satisfied us that so-called wood turpentine does not dry as well as the gum spirits, but has a definite retarding action. In some instances this would not be objectionable; in others it would. So far as we know there is no other difference in varnish thinned with it nor in the manipulation or covering power of such paint and varnishes. We see no reason why the properly worked out process material should not be equal to ordinary turpentine.

F. W. Devoe & C. T. Reynolds Co., New York, N. Y.: The samples which we have examined have varied considerably, both in chemical and physical constitution. Some of them seemingly do contain a small proportion of the real turpentine, but most of them contain nothing other than what is probably known as pinene as distinguished from the terpenes of turpentine. For all practical purposes I believe that the solvent properties of the better grade would compare favorably with that of regular turpentine. Some of the better-made samples will evaporate with as little residue. The covering power of a paint or varnish made with wood turpentine should not differ in any way as far as I can see from that made with ordinary turpentine. The odor of the wood turpentine is so persistent that it can not be disguised. This odor is so disagreeable that we find painters and varnishers object to a varnish made with wood turpentine for the reason that it affects their eyes and also their throats, especially where the varnish is used in the interior of buildings where there is no draft to take away these odors.

Patton Paint Co., Milwaukee, Wis.: Considered as a paint thinner only, we believe a properly made wood turpentine is to all practical purposes just as good a thinner as gum turpentine. The principal objection that has been urged in the past against its use is that of its somewhat offensive and irritating odor. In the case of products of more recent refining processes these objections have been very largely if not wholly overcome; certainly reduced to such an extent that when present in the percentage found in ordinary paints no unpleasant results need be expected.

With regard to its utility as a varnish thinner we are not prepared to state as to whether in our opinion it would be safe to substitute wood products for gum turpentine on account of the turpentine being present in much greater quantities than in an ordinary paint. The objections previously urged are correspondingly more apparent. Moreover, any nonvolatile residue that might be contained in the wood turpentine would prejudice its use to a greater extent than in paint. When we refer to nonvolatile residue we consider the temperature at or about 70°.

National Paint, Oil and Varnish Association, New York City: The solvent power of wood turpentine is not less than that of gum spirits. If the gravity is the same it dries the same. It makes just as good varnish, it works just as free, its covering power is the same. The objections are purely a question of odor and uniformity. If it were made of uniform quality free from oil and poisonous acids it would be simply a question of educating the public to a different odor.

The Booth & Law Varnish Co., New Haven, Conn.: Wood turpentine has a decided value as a paint and varnish thinner and works as well as the gum turpentine in most cases. The principal objection to it in paint is the odor. To-day it is more uniform than formerly, almost equal to the best gum turpentine, and will answer as well in results.

OPINIONS OF USERS OF PAINTS AND VARNISHES.

Arnold Print Works, North Adams, Mass.: We have used wood turpentine in ordinary painting and find it gives entire satisfaction. It is much better for this work than many of the adulterated turpentines.

Magnus, Mabee & Reynard (Inc.), New York City: We find that wood turpentine in a number of instances will dissolve varnish gums at a lower temperature, and dries fully as well as the regular gum spirits of commerce. We have sold this product in carload lots to several large varnish manufacturers, who report to us that the results in using same are entirely satisfactory for the same uses that they have heretofore employed gum spirits of turpentine. When we first handled this product we occasionally had a few complaints because of its strong odor, but of late we have had no complaints at all, as our manufacturers are producing spirits of turpentine without the objectionable odor heretofore noted. We might add that there are various grades of wood turpentine, as the process of distilling same varies among different manufacturers. For instance, some use direct heat in the retort process, some use steam distillation, while others use a rosin bath. The last-mentioned process we consider produces the best product. Our customers are principally among the paint dealers and manufacturers of varnishes.

John A. Casey, New York City: The steam turpentine has a nice, sweet wood odor, and finds a more ready sale than the wood-destructive turpentine. It is our opinion, however, that as a solvent and drier the wood-destructive turpentine is to be preferred. We are still of the opinion that the wood turpentine will never really take the place of turpentine manufactured from the gum.

A good many varnish manufacturers are using, however, wood turpentine in connection with gum turpentine in large quantities, particularly within the last two or three years, as the price of turpentine manufactured from the gum is higher at the present moment than it has been any time since the Civil War. * * *

Of course we refer only to first-class goods and not to inferior goods. It seems that every wood-destructive plant makes a different article. When one process and only one will be employed and a uniform article can be put on this market, it will become a marketable article and of staple value.

Hartsville Furniture Co., Hartsville, S. C.: Have used a wood turpentine made by the destructive process, but abandoned it at once, as it made the eyes smart and burn. Odor not so objectionable.

W. W. Hines & Co., Petersburg, Va.: Can not sell on account of odor, particularly at present prices of gum turpentine (40 to 50 cents per gallon).

Hartsville Supply Co., Hartsville, S. C.: Do not have any difficulty in selling a destructive turpentine for painting purposes at 80 cents a gallon when gum spirits are 90 cents and \$1.

Tanner Paint & Oil Co., Richmond, Va.: We have tried to introduce same in a number of instances, but the average workman will not work same. In closely confined places, such as boat bottoms, inside of locomotive tanks such as used on tenders,

it is impossible for painters to use the wood turpentine on this class of work, as the odor from same is very nauseating in a closed place where there is no circulation of air.

Burchhardt Co., Cincinnati, Ohio: Our experience with wood turpentine has been very unsatisfactory. We find it almost unsalable on account of the penetrating odor. When a large surface is exposed freshly painted in a closed space, for example, any interior work, the fumes given off are exceedingly disagreeable, both as to odor and effect on the eyes. The natural gum turpentine is free from such objection.

A. B. Chase & Co., Norwalk, Ohio: Our experience with wood turpentine has been very limited. The only thing we noticed is that it curdled or precipitated the varnish, and that the odor was very offensive. We have decided that it is not to be used in thinning varnishes for use on high-grade pianos.

H. H. Franklin Manufacturing Co., Syracuse, N. Y.: We are of the opinion that wood turpentine would be of no use to us on account of the odor.

The Locomobile Co. of America, Bridgeport, Conn.: We have never been able to get satisfactory results in the paint department when using wood turpentine. Our painters state that it is very quick drying, has a tendency to thicken the paint after standing a short time, and on evaporation leaves the paint lifeless.

Brewster & Co., New York: We have only experimented with wood turpentine, but so far as we have gone we find that its solvent power seems to be less than ordinary turpentine, that it does not dry as well, and appears to leave a greasy residue on the surface of the article painted instead of drying flat and clear, as in the case where pure gum turpentine is used. It is also heavier to work. In covering power there is no appreciable difference. We have not found it satisfactory to use in our business where a very good finish is necessary.

Grand Rapids Chair Co., Grand Rapids, Mich.: Its solvent power is the same. It does not dry as well and makes a more inflexible varnish than ordinary turpentine. It is more difficult to work and appears to separate from the varnish. It deteriorates with age.

Newport News Shipbuilding & Dry Dock Co., Newport News, Va.: We beg to say that we are still using wood turpentine, or Florida spirits, and the only objection we find is that the odor is unpleasant, as before stated. If this bad odor could be eliminated, we would use it much more extensively.

Norfolk & Western Railway Co., Roanoke, Va.: We have found the wood turpentine to have as good solvent value as spirit turpentine, of slightly slower drying qualities, but would dry out in time giving good results. This time of drying has a practical value in many of our shop operations, and is oftentimes the determining factor. We would not consider it as good as spirit turpentine in varnish making, due to the presence in small quantities of creosote. We did not find wood turpentine difficult to manipulate, nor did we find it to give detrimental results so far as the covering value of the paint was concerned.

The odor is rather objectionable to painters, which is probably the only point so far determined on which we would not recommend its use.

Pullman Co., Pullman, Ill.: So far as we are able to see, the solvent power of wood turpentine is about the same as that of the ordinary turpentine, with the exception that it dries a little slower. The covering capacity is apparently the same as the ordinary turpentine, and we are unable to note any difference in its manipulation or working. No objection has been raised by any of our painters, and for all purposes it seems to be a good substitute for the gum turpentine.

Another prominent railroad company: During the past four or five months we have used samples of these products and there is considerable objection to them, principally on account of the odor, which is sickening. As to their solvent power we do not find there is any difference, nor do we find a difference in drying qualities. There is no difference in the work nor in manipulation. We notice in mixing these substitutes that they turn whitish as a foam which disappears when they are left to settle. From

our short experience we should say that they would crack or check in time. In the case where turpentine paints are used—that is, coach colors where varnish is applied afterwards—we notice that they change the color. We do not advocate the use of wood turpentine.

In the foregoing opinions there is perfect agreement as to the objectionable odor of wood turpentine, although it would appear from some of the statements that some samples differ but little from gum turpentine in this property, a fact which is confirmed by the experience and observation of this bureau. As a rule the manufacturers of paints and varnishes state that properly prepared wood turpentine is fully as satisfactory as ordinary turpentine, drying as well and working as well as gum spirits.

Dealers are generally of the opinion that it is of practically the same value as gum spirits as a diluent, but find that the odor seriously interferes with the sale of the material.

Manufacturers state that they have no difficulty in disposing of their entire product to paint and varnish makers, and at a price close to that of gum spirits.

There is considerable difference of opinion among the users of paints and varnishes, piano makers, carriage and motor builders, furniture makers, railroad companies, shipbuilders, and house painters. Thus a prominent builder of automobiles states that their painters know very little about it, but that it is very quick drying and evaporates so rapidly from the paint that it is left lifeless. They have never been able to get satisfactory results with it. Another company states that it could not be used indoors on account of the odor. A piano company states that it curdles or precipitates the varnish and has an offensive odor. A shipbuilding company finds that it works as well and appears to give the same results as ordinary turpentine, and the only objection is its unpleasant odor. A railroad company finds it satisfactory as a paint diluent, but objects to its odor.

It would appear that the material differences in the expressed opinions as to the drying properties and suitability in general as a paint and varnish thinner are due in large part at least to the well known lack of uniformity in wood turpentines as heretofore placed on the market. It is more than probable that in those cases where decided objection is raised the turpentine contained considerable percentage of pine oils, which not only affect drying properties, but greatly increase the objectionable odor.

CONCLUSIONS.

POSSIBLE PRODUCTION FROM WASTE MATERIAL.

The waste wood of the South and Northwest from the lumber industry—tops, stumps, slabs, and sawdust, and the dead and down timber from fires and storms—furnishes one of the great undeveloped

resources of this country. From this wood, by industrially developed chemical methods, the entire output of naval stores, embracing turpentine, rosin, tars, pitch, rosin spirits, and rosin oils, having an annual value of at least \$30,000,000, may be obtained without boxing or turpentineing a single live tree.

There is more than sufficient waste material to yield annually all the papers (except news), paper and box board, and building board required, for which wood is suitable.

More methyl alcohol, acetate of lime, and acetone can be produced from this wood than is now made in the country, and large quantities of ethyl alcohol may also be recovered. While it has not been proved that these four products can be made profitably from waste resinous wood, it is not at all improbable that the manufacture of one or more of them, in conjunction with other products, would be practicable.

There are millions of acres of cut-over land covered with stumps and dead and down timber, all of which, because of its resinous nature, decays very slowly, enduring for years. There is enough of such material to supply all demands for the above-mentioned products for a very long period. The processes, equipment, and technique for the utilization of this material are either in operation or may be readily devised.

LOCATION, MANAGEMENT, AND OPERATION OF PLANT.

The financial success of the production of wood turpentine and other materials from waste wood is not controlled by the yield of valuable products alone, but is also largely affected by the location of the works with reference to transportation of raw material, and the business ability and technical skill with which they are conducted. As labor and transportation charges are the chief elements in the cost of conducting these plants, they should be located as close to the supply of raw material as possible, in order that the cost of collecting and hauling the wood and disposing of the products may be reduced to a minimum. It is well to give the most careful attention to location; the plant should always command two independent avenues of transportation for raw material and finished products. Other things being equal, the cost of wood is least when the works are located close to a sawmill, as in this case much of the wood has already been collected for mill purposes, the wastes from which are available at the smallest possible cost. Mill waste, however, is seldom as rich in turpentine as what is known as "lightwood," so that if the mill is located at some distance from the lightwood supply it may prove more profitable to locate the plant at the lightwood supply and haul the mill waste. Each case must be decided after careful consideration of all the circumstances, and therefore

more definite general advice can not be given than to advise locating as close to the raw material as other factors of cost will permit. Generally speaking, the operation of wood utilization works will prove more profitable when conducted under the same ownership or in close cooperation with the lumber industry, because of the cheap raw material available under these conditions, and also of the possibility of using the equipment, waste steam, power, etc., of the saw mill in assembling the materials and in the works. Large quantities of water are required in these plants for producing steam, operating condensers, etc., hence such plants should be located where a sufficient supply can be obtained. For these reasons wood turpentine should be one of the by-products of the lumber industry—furnishing another means of minimizing the dangers from the wastes of the industry and turning them to profitable account. Turpentine should not be the only by-product, however. The greatest returns will be obtained when several of the following substances are made from the waste woods of the yellow-pine lumber industry, namely, paper wood turpentine, pine oils, rosin spirits and oils, alcohol, soap, etc. The total value of these products is greater than the value of the products of the destructive distillation of the wood.

The approximate comparative values of the products of the several chemical methods for the utilization of waste wood are indicated by the following table. The figures are values per cord of wood.

Good lightwood, destructively distilled.....	\$28
Good lightwood, steam distilled.....	8
Good lightwood, soda extracted, and turpentine, rosin oils, and soda pulp made.	44
Ordinary long-leaf pine, soda extracted, and soda pulp.....	38
Good lightwood, extracted with volatile solvent and turpentine, rosin and soda pulp made.....	40

That is, the values obtained from a cord of wood by making paper pulp are from \$10 to \$37 greater than when the wood is destructively distilled or steam distilled and the wood burned or thrown away.

INDUSTRIAL DEVELOPMENT.

It is believed that the utilization of waste woods for the recovery and manufacture of wood turpentine, wood oils, rosin, rosin spirits, and oils, tars, pitches, acetates, alcohol, and paper pulp can be made a profitable chemical industry.

This, of course, does not mean that any ill-considered scheme of utilizing this material will prove profitable. On the contrary, only the best and most thoroughly developed plans, embracing all those considerations which are the foundation of well-organized industries, can be expected to reap the greatest returns from the opportunities which as yet lie practically undeveloped. It is not proposed to discuss here the details of equipment, arrangement of plant, or opera-

tion and technique of the various processes employed. The chemical utilization of wood in the ways suggested requires the adaptation and coordination of the equipment and processes which have been employed successfully in other industries. This is a comparatively simple problem for the experienced chemical engineer, but is not within the scope of this publication.

There are a number of ways in which the machinery for the manufacture of pulp and other products may be assembled, and many forms of apparatus may be employed. Some general suggestions, however, may be made. The observations and experiments of the bureau fully warrant the conclusion that a rotary digester will give larger yields of crude oils at less expense than will fixed retorts. It is also best to use a rotary digester in pulp making. The greater yield of oils with the rotary digester is due to the more rapid and complete penetration of the chips by the steam. Hand labor should be reduced to a minimum by the use of machinery. The arrangement of the plant should be such that repeated and useless handling of material is avoided and the products should be carried to the ultimate form in which they find industrial application. Incoming wood should be unloaded directly into the chipper and only the excess stored. Indeed there should be as little storage of wood as possible. It is far better that the wood should be brought daily to the works as soon as it is cut and split in order to avoid the great loss of turpentine which occurs at the surface of freshly cut and split pine.

Such a plant should be managed by a man skilled in its technical control in order that an accurate knowledge may be had of the quality and quantity of the raw material used and of the products obtained therefrom. Undoubtedly much of the dissatisfaction which has existed with regard to wood turpentine has been occasioned by the ignorance of the producer of the properties which the material should possess, or of the methods of producing a turpentine uniform in composition and resembling closely gum turpentine.

The wood should be cut diagonally across the grain and chipped to a fairly uniform size. The chips should not be more than three-fourths of an inch through nor less than one-fourth of an inch and may be subsequently shredded with advantage. Great care should be exercised to avoid the production of dust on the one hand and large slivers or chunks on the other. Very fine material loses turpentine before it reaches the digester, and is overcooked, resulting in low yields of turpentine and of weak pulp. Penetration of the large chips by steam or alkali is very slow and often incomplete, and consequently the time of steaming and digestion is prolonged, the yield of turpentine is lowered and the pulp is insufficiently cooked, which results in inferior pulp and paper.

The bureau has found in laboratory experiments that when the wood is prepared to pass a $\frac{1}{4}$ -inch screen the oils are removed completely in less than two hours steaming and no more is obtained on cooking. When prepared to pass an inch screen, but not a $\frac{1}{4}$ -inch screen, the oils are removed in from two to two and one-fourth hours steaming, and no more is obtained on further cooking. If the chips will pass a 2-inch screen, but not an inch screen, it requires three hours steaming and 12 per cent of the total oils is obtained in subsequent cooking. With properly prepared wood, the time for removal of turpentine is reduced at least one-third. The advantages of reducing the wood to a proper size and using a rotary have been demonstrated in the laboratory; 0.25 to 0.88 per cent of oils were recovered from the chips which had been steamed in a fixed retort. The smallest recovery was from chips passing a $\frac{1}{4}$ -inch screen and the largest amounts from chips not passing a 1-inch screen. The chips should, therefore, be screened before they are placed in the digester and those which are too large should be made smaller by regrinding, or, preferably, by shredding.

Under proper conditions the turpentine can be steamed out of the chips in about three hours. So far as the removal of the turpentine is concerned, only a small pressure need be carried on the rotary until the crude oils are off; 40 pounds pressure applied intermittently is ample. The pressure may then be raised for the purpose of cooking the wood. The wood should not be reduced to chips and stored as such for any length of time before steaming, as cut-up wood loses turpentine quite rapidly, especially in warm weather. (Hogged wood has been known to lose half the turpentine in one week.) If the grinding plant can not deliver the chips to the digester with sufficient rapidity, chipping may be begun before the digester is ready to receive the charge, and the chips held for a short time in an overhead bin, from which they can be spouted to the digester. Large amounts of chips should not be so stored, however, and losses due to volatilization or fire must be guarded against.

Two digesters, each holding 8 cords (12 to 16 tons) of chips, can handle at least 50 cords (75 to 100 tons) of wood per 24 hours, three cooks being made in each digester. If lightwood is used, from 400 to 1,000 gallons of crude oils should be obtained by steaming the chips. From the black liquor by destructive distillation, from 1,500 to 5,000 gallons of oils, consisting of rosin spirits and rosin oils mixed with oils (chiefly phenols and cresols) derived from the ligneous material removed by the soda, are obtained. The full amount of methyl alcohol usually obtained by destructive distillation of pine wood, together with traces of acetic acid, are also contained in the aqueous distillate from the black liquor. From the residue of the distillation of the black liquor, sodium carbonate is dissolved and recausticised in the usual way.

Preferably a globe-shaped digester revolved on hollow trunnions should be used. The cooking liquor and steam are fed through one trunnion while the steam carrying the volatile oils is removed through the other. When the oils practically cease to come over the valve in the take-off trunnion is closed and the wood cooked at the proper pressure.

Suitable equipment for a plant making steam wood turpentine and using 50 cords of wood per day consists of two 8-cord rotary digesters, a total boiler capacity of 450 horsepower, engine capacity of 250 horsepower, a hog, a shredder, conveyers, condensers, refining stills, storage tanks, pumps, piping, and building. The whole will cost between \$35,000 and \$50,000.

REFINING.

Steam and crude oil vapors should not be condensed together before refining, as this calls for unnecessary apparatus and the increased steam consumption makes the operating expenses larger. The vapors should be so handled as to separate the light oil with some heavy oil from most of the heavy oils and the water. For refining 1,000 or more gallons of crude oils per day a continuous column still should be used, large enough to take care of the volume of steam entering it, and so constructed that two, and possibly three, distinct products may be obtained. Each column should be so arranged that its contents can not be carried forward to the column containing the lighter oils, but the residues from the fractionation in one column may pass to the next lower one from which heavier oils are obtained and so on until the condensed water is practically freed from oils and goes to waste from the bottom of the last column. Stills of this character can be built, and while their first cost is high, they insure, when properly constructed and operated, the separation of fairly uniform products.

The experiments made in this laboratory also show that in order to obtain a wood turpentine, which will comply in specific gravity, refractive index, evaporation, and behavior on distillation with gum spirits, it is necessary that the heavier oils be removed from contact with live steam as rapidly and as completely as possible. To this end the fractionating plates should carry the minimum depth of liquid and the steam should not be allowed to continue to pass through the heavy oils after they have been freed from the light oils. The hot heavy oils pass downward to other fractionating chambers, each of which may be operated to yield a fairly definite product, or they may be refined in a pot still. The general character of the products obtained with the column still is shown in Tables 4 to 8, pages 62 to 66.

In these plants when smaller quantities of crude oils are to be refined, the first cost of the columns and difficulties of control make the use of two simpler stills advisable, a periodic column still for the turpentine and a simple gooseneck still for the refining of the heavy or pine oils. The same apparatus is suitable for the refining of the oils obtained by the destructive processes. It is necessary to wash these products with soda or lime, as has been pointed out, and also to refractionate once, or even several times, the portions obtained from the first or preliminary fractionation.

As long as the distillate contains only water and such oils as may properly be termed wood turpentine the percentage of wood turpentine in the distillate will be from 62 to 54 per cent and throughout nearly the whole distillation this relation may exist. When large quantities of pine oils begin to distill, however, the ratio of oil to water in the distillate drops rapidly and the changing quantity of oil in the distillate, together with the speed with which the ratio of oil to water changes, furnishes the best evidence of the nature of the distilling oils. It has been observed in general that when a column still is employed and the percentage of oils in the distillate decreases rapidly, the oils may be mixed with the turpentine until the percentage of oils in the final distillate falls to about 45 per cent. If a pot still is employed the indications are that when the percentage of oil in the distillate falls below 50 it should no longer be mixed with the turpentine, since distillates containing less than these amounts no longer meet the requirements for turpentine and should be run to another tank until the percentage of oil in the distillate falls below 30 per cent, when the distillate should be run into a third tank. The oils in the third tank will consist of pine oils alone, while those in the second tank will consist of a mixture of turpentine and pine oils and should be again distilled, and the distillate run into tank 1 until the oil in the distillate falls below 50 per cent, when the remainder is run into tank 3 without further distillation.

STANDARDS OF QUALITY.

Since wood turpentine is used as a substitute for gum spirits of turpentine, it should comply with the customary tests applied to gum spirits to determine its suitability for this purpose and also with specifications for gum spirits.

Inasmuch as the highest grade of wood turpentine need not be employed in certain kinds of work, several grades, each suitable for a specific purpose, may be made. In this way users will be enabled to buy economically such wood turpentine as is suitable for their various purposes, the utilization of the heavier oils will be promoted, and the less perfectly equipped and operated plants will be able to find a market for their products.

Three grades have been suggested by this bureau for gum spirits,¹ and there appears to be no reason why wood turpentine should not be graded in the same way and comply with the same specifications. These specifications are:

Standard or No. 1 turpentine should have a specific gravity at 20° C. of from 0.862 to 0.870; a refractive index at 20° C. of from 1.468 to 1.476; 95 per cent should distill below 170° C., and a layer of not less than 200 mm should be required to equal in color the Lovibond yellow glass No. 1. On polymerization with 38 normal sulphuric acid the residue should not exceed 1 per cent, should be reddish in color and viscous, and its refractive index at 20° C. should be from 1.500 to 1.520. An unadulterated turpentine which does not agree with these requirements may properly be regarded as not of standard or No. 1 quality.

Second quality or No. 2 turpentine should have a specific gravity at 20° C. of from 0.862 to 0.875; a refractive index at 20° C. of from 1.468 to 1.480; 90 per cent should distill below 170° C. and a depth of not less than 100 mm should be required to equal the Lovibond yellow glass No. 1. The polymerization residue must not exceed 1 per cent and must have a refractive index of not less than 1.50.

Third quality or No. 3 turpentine should have a specific gravity at 20° C. of from 0.865 to 0.880; a refractive index at 20° C. of from 1.468 to 1.485; 60 per cent should distill below 170° C. and a depth of not less than 50 mm should be required to equal the Lovibond yellow glass No. 1. The polymerization residue must not exceed 1 per cent and must have a refractive index of not less than 1.500.

Wood turpentine that complies with the specifications for No. 1 and No. 2 turpentines will have but little objectionable odor, and it is believed will meet all reasonable technical requirements for paint and varnish thinners, except possibly those for varnishes of the highest grade.

VALUE OF WOOD TURPENTINES AS PAINT AND VARNISH THINNERS.

The experimental work which has been done and the information that has been collected leave no doubt that wood turpentine, both destructive and steam distilled, can, by careful refining, be made to correspond closely with gum spirits in all those properties by which the suitability of turpentine, as a paint and varnish thinner, is judged. The wood turpentine that contains no products of destructive distillation and is free from heavy oils differs very slightly in odor from gum spirits, and but little, if any, objection can be raised to the technical use of well-refined wood turpentine on this account. It may be used in well-ventilated places without serious inconvenience to the workmen. Well-refined wood turpentine is a suitable paint and varnish material, except possibly for the highest grade varnishes; and it may be employed for these purposes without detriment to the paint or varnish.

¹ U. S. Dept. Agr., Bureau of Chemistry Bul. 135.

DESTRUCTIVELY DISTILLED.

59	Crichton, Ala.	Dec., 1904.	Mallonee.	Manufacturer.	0. 8690	1. 4688	157	47	79	89	11	+18.1	354.8301	441.7	9.4	0.0093
62	Mobile, Ala.	Jan., 1905.	do.	do.	8650	1.4696	157	11	50	70	30	+13.7	380.5271	554.5	4.9	.0031
1175	Fayetteville, N. C.	Nov., 1905.	Clerk	F. P. Veitch	8600	1.4000	154	64	92	94	6	+6.1	0.0398	3322.238.1	3.9	.0110
1176	Savannah, Ga.	do.	Mallonee	do.	8570	1.4723	162	52	66	96	4	+9.6	0.359	282.935.5	0.9	.0040
1184	Jacksonville, Fla.	do.	McKeithan	Manufacturer.	8660	1.4676	154	52	88	94	6	+21.8	0.414	9310.952.0	1.9	.2330
1195	Philadelphia, Pa.	do.	McKeithan	C. Schramk & Co.	8630	1.4691	154	42	88	94	6	+19.9	(9)	(9)	(9)	(9)
1229	Jacksonville, Fla.	do.	McKeithan	F. P. Veitch	8740	1.4718	150	21	76	89	11	+17.2	8.369	2275.446.9	6.1	.0346
1237	Vancouver, British Columbia.	1906.	do.	Bray	8640	1.4695	153	30	52	68	32	-50.4	1.5343	8247.443.2	7.5	.0277
1238	do.	do.	do.	Manager	8620	1.4666	156	70	93	97	3	-44.9	0.436	1323.355.9	2.1	.0143
1317	Savannah, Ga.	Dec., 1906.	Mallonee.	Manufacturer.	8380	1.4746	+6.5	273.5183	545.0	17.3	.0533
1318	New York.	1906.	do.	do.	9230	1.4790	158	7	49	65	35	+16.6	340.4259	240.6
1319	Kipling, Ala.	do.	Billing	F. P. Veitch	8930	1.4692	154	51	71	83	17	+16.9	322.2247	037.6	1.6	.0026
1542	Albany, Ga.	Feb., 1906.	do.	do.	8940	1.4791	120	41	68	87	13	(9)	3.0375	5258.758.4	19.0	.2343
1543	do.	do.	Clark	Dealer.	8760	1.4680	152	79	88	93	7	+19.7	292.7159	963.5	(7)	(7)
1561	Fayetteville, N. C.	do.	do.	do.	8678	1.4510	169	0.418	6312.053.3	7.9	.0128
10951	New York.	Jan., 1910.	do.	do.

1 Below 165° C. 2 Below 180° C. 3 Above 180° C. 4 Above 175° C. 5 Missing. 6 Too dark. 7 No end point.

TABLE 2.—*Pine oils and still residues.*
STEAM DISTILLED.

Laboratory No.	Description of sample.	Date of sampling.	Source.	Specific gravity at 20° C.	Refractive index at 20° C.	Initial distilling temperature, ° C.	Per cent distilled below—					Color.		Iodin value.		Saponification number.	Acidity num-ber.	
							185° C.	200° C.	215° C.	225° C.	240° C.	250° C.	Yellow.	Red.	Addition.			Substitu- tion.
1181	First heavy oil dis- tillate.	Nov., 1905.	Waycross, Ga.	0.889	1.4783	170	26	64	92	97	1.7	0	178.9	48.2	28.0	0.0073	
1182	Second heavy oil dis- tillate.	do.	do.	.932	1.4844	1 102	1 2	6	64	95	2.5	0	92.6	53.3	2.7	.0042	
1363	Refining still residue after No. 1182.	do.	do.	.927	1 99	0	0	1 1	45	45	70	96.4	1.5	(*)	
1366	No. 2 white oil.	do.	Lyons, Ga.	.928	102	1 2	29	93	96	2.6	0	129.3	62.2	3.9	.0339	
1367	Refining still residue.	do.	do.	.960	98	0	1 1	8 18	28	119.3	67.2	14.6	(*)	
1540	Light oil.	Feb., 1906.	Live Oak, Fla.	.937	1.4817	100	0	1 6	89	30.0	5.0	121.7	49.5	6.5*	.0449	
1541	Heavy oil after light oil.	do.	do.	.951	1.4894	190	1 1	56	71	85	122.5	58.4	(*)	(*)	
2657	Light oil tailings from refining still.	Aug., 1907.	Orange, Tex.	.944	1.4880	100	1 4	22	90	126.0	45.0	4.5	.0386	
2658	Heavy oil tailings from refining still.	do.	do.	.958	1.4975	205	52	68	91.3	77.4	(*)	.0177	
1565	Refined pine oil.	Apr., 1907.	Wilmington, N. C.	.937	1.4844	±100	1 2	31	86	90	20.0	2.8	118.0	52.0	11.5	.2071	
1814	Refined pine oil (soda extracted).	May, 1907.	Lynchburg, Va.	.944	1.4855	100	1 3	20	94	148.0	52.0	9.2	.1783	
1815	Crude pine oil (soda extracted).	do.	do.	.931	1.4852	180	4	32	76	88	6 93	6 181	156.1	55.8	(*)	.0392
14944	Water-white pine oil.	1910.	Bradford, Fla.	.8942	1.4765	173	7 51	8 130
14945	Straw-colored pine oil.	do.	do.	.9434	1.4819	210	38	9 117	1034.0

DESTRUCTIVELY DISTILLED.

44	Pine oil.	Nov., 1905.	Jacksonville, Fla.	0.910	1.4864	153	61	72	78	84	(*)
1183	do.	do.	Savannah, Ga.	0.907	1.4844	162	23	(*)
1550	Crude heavy oil.	Feb., 1906.	Collins, Ga.	1.085	1.5520	152	10	10	24	(*)
1544	do.	do.	Albany, Ga.	.958	1.5028	140	48	58	62	68	75	(*)

1 Contained water.

2 Too dark.

3 Below 230°.

4 No end point.

5 Temperature fell from 130°.

6 Below 130°.

7 Below 180° = 32 per cent.

8 Below 205°.

9 Below 220°.

10 Depth in millimeters to equal Lovibond glass No. 1 yellow.

TABLE 3.—Crude wood turpentine.

STEAM DISTILLED.

Laboratory No.	Process.	Place of sampling.	Date of sampling.	Sampled by—	Remarks.	Specific gravity at 20° C.	Refractive index at 20° C.	Initial distilling temperature.	Per cent distilling below—			Per cent residue above 185° C.	Specific rotation as 20° C.	Color.		Iodin absorption.			Saponification number.	Acidity number.			
									160° C.	170° C.	185° C.			Yellow.	Red.	Total.	Addition.	Substitution.					
1177	Bennor	Tallahassee, Fla.	Nov., 1905	Manufacturer	0.8680	1.4683	155	50	89	97	3	+19.5	10.0	1.4	417.4	313.4	52.0	3.2	0.0359	3.2	0.0359	
1184	do.	Macon, Ga.	do.	do.8790	1.4719	155	21	74	85	15	+15.2	35.0	9.8	8.9	0.1610	8.9	0.1610	
1227	do.	do.	do.	do.8680	1.4691	158	36	87	95	5	+15.3	1.5	0	408.5	309.5	49.5	3.0	0.0248	3.0	0.0248	
1228	do.	Waycross, Ga.	do.	F. P. Veltch8840	1.4733	156	20	64	80	20	+15.0	431.3	322.3	54.5	2.6	0.0255	2.6	0.0255	
1360	Bennor	Macon, Ga.	Feb., 1906	do.8710	1.4721	157	12	75	89	11	+22.2	13.5	1.8	407.2	285.4	60.9	3.2	0.0177	3.2	0.0177	
1365	Bethune	Lyons, Ga.	do.	do.8740	1.4720	152	13	71	88	12	+12.5	1.1	0	398.3	281.5	58.4	2.6	0.0154	2.6	0.0154	
1538	do.	Live Oak, Fla.	do.	do.8760	1.4932	153	66	85	91	9	+17.9	24.0	5.0	412.3	313.3	49.5	3.0	0.0177	3.0	0.0177	
1552	do.	Macon, Ga.	do.	do.8660	1.4686	151	77	89	95	5	+17.9	24.0	5.0	425.9	313.4	55.8	2.6	0.0424	2.6	0.0424	
1553	do.	do.	do.	do.8730	1.4612	152	67	81	89	11	+23.2	12.0	15.0	408.6	284.2	62.2	7.3	0.1157	7.3	0.1157	
1555	do.	do.	do.	do.	Late running; kept under pressure one-half hour.	.8920	1.4713	+10.0	386.3	273.3	56.5	10.8	0.0314	10.8	0.0314	
1556	do.	Live Oak, Fla.	do.	do.8850	1.4741	+9.3	95.0	20.5	390.7	279.1	55.6	3.9	0.1268	3.9	0.1268	
1558	do.	Wilmington, N. C.	do.	do.9370	1.5059	160	0	6	24	76	(1)	21.6	6.0	386.3	273.3	56.5	8.8	0.2155	8.8	0.2155	
2654	Weed	Orange, Tex.	Aug., 1907	Manufacturer8760	1.4733	154	47	74	84	16	+20.4	386.9	280.3	53.3	2.5	0.0053	2.5	0.0053	
16051	Steam and H ₂ SO ₄ .	Georgetown, S. C.	Jan., 1911	do.8724	1.4748	165	20	78	22
16052	Steam and soda.	De Soto, Miss.	Apr., 1909	do.8900	1.4728	160	* 15	40	72	28
20534	do.	Branford, Fla., 1911	do.8967	1.4730	32	68	32

DESTRUCTIVELY DISTILLED.

1546	Mallonee	Collins, Ga.	Feb., 1906	F. P. Veltch	0.9180	1.4915	150	21	46	67	33	(1)	315.0	215.0	50.0	(2)	(2)
1549	do.	do.	do.	do.9630	1.5107	145	10	19	34	66	(1)	286.6	104.2	91.2	(3)	(3)
1560	do.	Fayetteville, N. C.	do.	do.8680	1.3319	152	76	89	91	9	+19.8	10.0	0.8	421.2	304.4	58.4	3.4	0.0323	3.4	0.0323

1 Too dark.

* Below 165°.

* No end point.

TABLE 4.—Distillation data on refining crude steam-distilled wood turpentine with steam in a beer still.

[Figures are on subsamples and total fractions.]

Barrel number and fraction.	Sample No.	Distillation in progress.	Temperature at top of still uncorrected.	Turpentine in fraction.	Turpentine in distillate.	Specific gravity at 20°C.	Refractive index at 20°C.	Initial distilling temperature.	Per cent distilled below—										
									160° C.	165° C.	170° C.	175° C.	185° C.	200° C.	215° C.	230° C.			
Crude turpentine.						0.8964	1.4741	100											
Crude turpentine.						.8900	1.4728	158											
Crude turpentine.								160											
6A.	1	0	96.4		63	.8618	1.4668	154	82	94	97	99	99	166	272	377	479		
6A.	2	48	97.0		60	.8619	1.4682	156	56	85	93	97	97	160	264	369	472		
6A.	3	62	97.5		54	.8635	1.4687	157	38	73	86	90	94	160	264	369	472		
6A.	4	71	98.0		47	.8659	1.4699	158	4	59	76	86	94	160	264	369	472		
6A.	5	75	98.2		46	.8669	1.4703	159	4	45	70	82	92	160	264	369	472		
6A.	6	81	98.4		43	.8692	1.4717	160		31	57	70	83	160	264	369	472		
6A.	7	84	98.7			.8734	1.4728	162		17	49	60	84	160	264	369	472		
6A.	8	84	99.0			.8764	1.4738	163		5	40	52	79	160	264	369	472		
6B.	9	91	99.5		35	.8904	1.4765	±170			2	2	52	160	264	369	472		
6B.	10	120	100.0		27	.9236	1.4809	±180					52	160	264	369	472		
6B.	1 to 7	84	96.4 to 98.7	32½	52	.8627	1.4698	±156	49	83	92	97	97	160	264	369	472		
6B.	8 to 10	35	98.7 to 100.0	5	23	.8907	1.4770	±167					12	42	69	85			
						.9004	1.4775	165											

1 Below 180°.

2 Below 185°.

3 Below 190°.

4 Below 195°.

FROM BEER STILL.

Barrel number and fraction.	Sample No.	Distillation in progress.	Temperature at top of still uncorrected.	Turpentine in fraction.	Turpentine in distillate.	Specific gravity at 20° C.	Refractive index at 20° C.	Initial distilling temperature.	Per cent distilling below—						
									160° C.	165° C.	170° C.	175° C.	185° C.	200° C.	215° C.
Mixed first fraction from beer still		Minutes.	° C.	Gallons.	Per cent.	0.8610	1.4678	{	50	79	91	96	98		
1 to 6A	1	0	96.0		65	.8614	1.4667	156	67	85	92	96	98		
1 to 6A	2	2	96.0		61	.8601	1.4672	155	87	96	98				
1 to 6A	3	103	98.0		40	.8686	1.4737	153	95	98					
1 to 6B	4	122	100.0		9	.8626	1.4790	163	9	69	89			97	99
1 to 6A	1 to 3	103	96.0 to 98.0	46.5		.8607	1.4682	185	77	92	97	98		17	85
1 to 6B	4	19	98.0 to 100.0	4.0		.9137	1.4784	159	14	77	91		10	43	83

FROM REFINING STILL.

7 to 10A	1	2	95.8		58	0.8611	1.4665									
7 to 10A	2	6	95.6		59	.8601	1.4661									
7 to 10A	3	27	96.0		61	.8698	1.4662									
7 to 10A	4	61	95.4		58	.8601	1.4666									
7 to 10A	5	108	95.6		58	.8603	1.4668									
7 to 10A	6	181	96.5		54	.8599	1.4688									
7 to 10A	7	190	96.9		53	.8609	1.4698									
7 to 10A	8	215	98.2		39	.8669	1.4728									
A	1 to 6	181	95.4 to 96.5	40½	59	.8600	1.4670	155½	81	96	98					
B+C	7 to 8	34	96.5 to 98.2	5½	47	.8622	1.4703	158		34	73	87	97			
B+C	7 to 8	25	96.9 to 98.2	3½	40	.8724	1.4705	163		27	66	80	88			
C	7 to 8	25	96.9 to 98.2	3½	46	.8631	1.4710	161		13	63	82	95	89		
Residue						.9174	1.4787	172					19	53		75

1 Manipulating steam valve, cutting off steam all the time, and temperature falling.

TABLE 8.—Summary of refining experiments with column stills showing the relation between the percentage of turpentine in the distillate and the composition of the turpentine.

Sample.	Fractionated in—	Total time of distillation.	Time elapsed from beginning of distillation.	Temperatures at top of column.	Turpentine in distillate.	Specific gravity at 20° C.	Refractive index at 20° C.	Initial distilling temperature. ¹	Per cent distilled below—		
									160° C.	165° C.	170° C.
1 to 6A 2	Beer still	122	2	96.0	61	0.8601	1.4672	153	98	99	99
1 to 6A 1	do.	122	0	96.0	65	.8614	1.4667	153	96	96	96
9A 2	Refining still	175	21	97.8	61	1.4673	1.4673	155 ¹	66	90	96
9A 1	do.	175	19	97.0	61	.8617	1.4685	155	64	88	94
7A 1	do.	76	2	96.8	62	1.4669	1.4669	155	74	92	96
6A 1	Beer still	129	0	96.4	63	.8618	1.4668	154	82	94	94
6A 2	do.	129	48	97.0	60	.8619	1.4682	156	56	85	93
7A 2	Refining still	76	14	97.2	59	.8620	1.4671	156	52	94	94
8A 1	do.	130	1	96.6	64	.8623	1.4669	155	59	88	97
6A 3	do.	76	35	97.8	53	.8634	1.4679	157	28	75	87
5A 3	Beer still	129	62	97.5	54	.8635	1.4687	157	38	73	86
7A 4	do.	166	56	97.6	52	1.4693	1.4693	157	29	72	87
7A 4	Refining still	76	52	98.4	51	.8654	1.4691	158	1	51	76
6A 4	Beer still	129	71	98.0	47	1.4699	1.4699	158	4	59	70
6A 5	do.	129	75	98.2	46	.8669	1.4703	159	4	45	70
8A 2	Refining still	130	101	98.8	49	.8670	1.4702	159	0	37	64
7A 5	do.	76	60	98.8	45	.8679	1.4700	159	0	19	57
1 to 6A 3	Beer still	122	103	98.0	40	.8686	1.4737	163	0	9	49
6A 6	do.	129	81	98.4	43	.8682	1.4717	160	0	31	57
8B 3	Refining still	130	111	99.2	45	.8705	1.4717	161	0	4	38
9B 3	do.	175	152	98.8	43	.8715	1.4715	164	0	2	38
5B 7	Beer still	166	76	98.5	42	162	0	31	55
6A 7	do.	129	84	98.7	42	162	0	17	49
6B 8	do.	129	91	99.0	35	.8734	1.4728	162	0	5	40
6B 6	Refining still	76	76	99.8	31	.8764	1.4738	±163	0	0	6
8B 4	do.	130	130	100.0	32	.8815	1.4745	170	0	0	0
6B 4	do.	129	101	99.5	27	.8844	1.4717	170	0	0	0
6B 9	Beer still	129	175	99.8	25	.8904	1.4765	±170	0	0	0
9B 4	Refining still	175	175	100.08944	1.4786	±175	0	0	0
6B 10	Beer still	129	129	100.09236	1.4809	±189	0	0	0
1 to 6B 4	do.	122	122	100.09326	1.4790	±185	0	0	0

¹ Emergent stem thermometer uncorrected.

TABLE 10.—Summary of Table 9 showing constants of turpentine samples taken during the progress of distillation, and the percentage of turpentine in the distillate.

[Total time for distillation of turpentine No. 1, 900 minutes; No. 2, 800 minutes; No. 3, 835 minutes.]

Sample number.	Distillation number.	Time elapsed from beginning of distillation.	Turpentine in distillate.	Specific gravity at 20° C.	Refractive index at 20° C.	Initial distilling temperature.	Per cent distilling below—						
							160° C.	165° C.	170° C.	175° C.	180° C.	185° C.	190° C.
		<i>Minutes.</i>	<i>P. ct.</i>			<i>° C.</i>							
19923.....	1	4	54	0.8634	1.4660	153	30	79	90	92	93	
19924.....	1	45	53-54	.8638	1.4662	157	21	77	87	91	94	96	
19925.....	1	495	45	.8665	1.4693	160	0	28	62	77	83	87	
19914.....	3	565	44	.8683	1.4690	162	0	16	59	75	82	86	
19926.....	1	555	43	.8686	1.4707	163	0	8	53	71	81	86	
19927.....	2	620	40	.8693	1.4710	161	0	5	51	69	76	85	
19915.....	3	540	38-39	.8718	1.4715	163½	0	4	36	61	74	80	
19916.....	3	630	33	.8763	1.4740	167	0	0	7	36	62	70	
19920.....	2	720	30	.8811	1.4750	166	0	0	3	10	41	60	
19917.....	3	685	27	.8830	1.4750	166	0	0	3	12	40	55	
19921.....	2	745	27	.8868	1.4753	173	0	0	0	9	20	45	
19918.....	3	720	25	.8889	1.4770	171	0	0	0	6	11	44	
19919.....	3	765	22	.9005	1.4790	176	0	0	0	0	1	4	
19922.....	3	810	18	.9140	1.4792	183	0	0	0	0	0	4	

TABLE 11.—Detailed opinions of the workmen on the varnishes used—Continued.

PIANO FINISHING OR FLOWING VARNISHES—Continued.

Data.	Varnish.	A. B. Chase Co.	Wm. Knabe & Co.	Murphy Varnish Co.	Devoe & Reynolds Co.	Steinway & Co.	American Paint Manufacturing Association.
Gloss of varnish surface.	1. Gum spirits distilled spirits.	All alike.....	All alike.....	Poorest gloss.....	All good (1 and 3 show tears).
	2. Steam distilled filled spirits.						
	3. Destructively distilled spirits.						
	4. Destructively distilled spirits.						
	5. Gum spirits (probably).						
Odor is objectionable.	1. Gum spirits distilled spirits.	Not objectionable.....do.....	Old fatty oil odor..... Odor like turpentine substitute.....do.....do.....	Not objectionable..... Objectionable..... Not objectionable.....do..... Objectionable.....	Not objectionable..... Objectionable..... Not objectionable.....do..... Objectionable.....
	2. Steam distilled filled spirits.						
	3. Destructively distilled spirits.						
	4. Destructively distilled spirits.						
	5. Gum spirits (probably).						
Objectionable to work with.	1. Gum spirits distilled spirits.	Not objectionable.....do.....	Old fatty oil smell.....	Not objectionable.....do.....
	2. Steam distilled filled spirits.						
	3. Destructively distilled spirits.						
	4. Destructively distilled spirits.						
	5. Gum spirits (probably).						
Are difficult to work with.	1. Gum spirits distilled spirits.	None are.....	None are.....	None are.....	Not difficult..... Difficult.....do.....do..... Not difficult.....	None are.....	All work nicely.
	2. Steam distilled filled spirits.						
	3. Destructively distilled spirits.						
	4. Destructively distilled spirits.						
	5. Gum spirits (probably).						

Varnishes rub down.	1. Gum spirits.....	None are satisfactory from a polisher's standpoint.	All too slow and too elastic to rub; can not be rubbed.	Rubs out well, good surface.	Medium in rubbing.	All rub well.
	2. Steam distilled spirits.					
Behavior under the brush.	3. Destructively distilled spirits.	Flows freely.....	(All flow too freely for the class of varnishes.	Rubs fairly well, fair surface.	This flows heavily.....	
	4. Destructively distilled spirits.					
	5. Gum spirits (probably).					
General remarks.....	1. Gum spirits.....	All unsatisfactory; did not flow out well. The first was best, third second best, and the other three equally poor.		Rubs good quickly, fair surface.	We consider all of them very medium varnishes.	
	2. Steam distilled spirits.					
	3. Destructively distilled spirits.					

PIANO RUBBING VARNISHES.

Varnishes were thinned with—	1. Gum spirits.....	1 part thinner to 4 parts varnish.	2 parts thinner to 1 part varnish.	All thinned alike.
	2. Steam distilled spirits.	1 part thinner to 5 parts varnish.	Not thinned.....	1st coat 1 to 1.....
	3. Destructively distilled spirits.	do.....	do.....	2d coat 1 to 1.4.....
	4. Destructively distilled spirits.	do.....	do.....	3d coat 1 to 1.4.....
	5. Gum spirits (probably).	do.....	do.....	4th coat 1 to 5.....
Varnishes remained tacky.	1. Gum spirits.....	1 part thinner to 21 parts varnish.	do.....	5th coat 1 to 5.....
	2. Steam distilled spirits.	Not thinned.....	do.....	6th coat 1 to 5.....
	3. Destructively distilled spirits.	do.....	do.....	do.....
	4. Destructively distilled spirits.	do.....	do.....	do.....
	5. Gum spirits (probably).	do.....	do.....	do.....

TABLE 11.—Detailed opinions of the workmen on the varnishes used—Continued.
PIANO RUBBING VARNISHES—Continued.

Data.	Varnish.	A. B. Chase Co.	Wm. Knabe & Co.	Murphy Varnish Co.	Devoe & Reynolds Co.	Steinway & Co.	American Paint Manufacturing Association.
Varnishes hardened in—	1. Gum spirits.....	Allowed one week between coats on all.	Appeared hard in 5 days. Allowed 10 days.	18 hours.....	48 hours.....	8 to 12 days.....	40 hours.
	2. Steam distilled spirits.			22 hours.....	72 hours.....		36 hours.
	3. Destructively distilled spirits.			20 hours.....	do.....		Do.
	4. Destructively distilled spirits.			18 hours.....	do.....		40 hours.
	5. Gum spirits (probably).			20 hours.....	48 hours.....		36 hours.
Varnish was applied in—	1. Gum spirits.....	3 coats.....	3 coats, 3 days between coats.	3 coats.....	4 coats.....	6 coats.....	4 coats.
	2. Steam distilled spirits.			Luster fair.....	Luster full and rich, best.		Medium gloss.
	3. Destructively distilled spirits.			Good and full, second best.	Good and full, second best.		do.....
	4. Destructively distilled spirits.			Luster fair.....	Luster fair.....		Fair gloss.....
	5. Gum spirits (probably).			Good and full, third best.	Good and full, third best.		do.....
Gloss of varnish surface.	1. Gum spirits.....	No perceptible difference in gloss.	Can not distinguish any difference.	Smells of naphtha.	Strong resinous odor.	Objectable.....	All good. No. 2 best, Nos. 1 and 3 show tears.
	2. Steam distilled spirits.			Smells of York spirits.	do.....		
	3. Destructively distilled spirits.			Worst of any.....	do.....		
	4. Destructively distilled spirits.			O. K.....	do.....		
	5. Gum spirits (probably).			Not objectionable.....	do.....		
Odor is objectionable.	1. Gum spirits.....	Samples not large enough to say definitely.	On account of odor.....	Not objectionable.....	Not objectionable.....	Not objectionable.....	All work nicely. Can work with all. No. 3 most objectionable and can not work any length of time in closed room with No. 4.
	2. Steam distilled spirits.			do.....	do.....		
	3. Destructively distilled spirits.			do.....	do.....		
	4. Destructively distilled spirits.			do.....	do.....		
	5. Gum spirits (probably).			do.....	do.....		

<p>Are difficult to work with.</p>	<p>1. Gum spirits. 2. Steam distilled spirits. 3. Destructively distilled spirits. 4. Destructively distilled spirits. 5. Gum spirits (probably). 1. Gum spirits. 2. Steam distilled spirits. 3. Destructively distilled spirits. 4. Destructively distilled spirits. 5. Gum spirits (probably). 1. Gum spirits. 2. Steam distilled spirits. 3. Destructively distilled spirits. 4. Destructively distilled spirits. 5. Gum spirits (probably). 1. Gum spirits. 2. Steam distilled spirits. 3. Destructively distilled spirits. 4. Destructively distilled spirits. 5. Gum spirits (probably).</p>	<p>Not difficult.</p>	<p>On large surface</p>	<p>Difficult.</p>	<p>Not difficult.</p>
<p>The varnishes rub down.</p>	<p>Satisfactorily.</p>	<p>Satisfactorily</p>	<p>{All rub well but sweat soon after. No. 3 is toughest.</p>	<p>{Rubs out well, good surface. Rubs out fair, fair surface. Rubs out quickly, fair surface.do Rubs out well, good surface. Works a little stiff. Very free.....do</p>	<p>Satisfactory</p>
<p>Behavior under brush.</p>	<p>Does not flow freely.</p>	<p>Flows well.</p>	<p>{All good on the small surfaces used.</p>	<p>{Pair working Works free.</p>	<p>All flow freely from brush.</p>
<p>Character of body.</p>	<p>Body not so good.</p>	<p>Heaviest body</p>	<p></p>	<p></p>	<p>No difficulty in flowing with any.</p>
<p>General remarks.</p>	<p>These samples rubbed better than the regular varnish that we use.</p>	<p>Time and exposure can only determine accurately the value of the varnishes.</p>	<p></p>	<p>We find it difficult to form an opinion as to the merits of these goods. The best working results can not be obtained with Nos. 2, 3, and 4.</p>	<p></p>

TABLE 11.—Detailed opinions of the workmen on the varnishes used—Continued.

COACH FINISHING VARNISHES.

Data.	Varnish.	Devoe & Reynolds Co.	Murphy Varnish Co	H. H. Babcock Co.	Fay & Bowen Engine Co.	James & Meyer Buggy Co.	Paint Manufacturing Association of the United States.
Varnishes were thinned with—	1. Gum spirits.....	1 part thinner to 8 parts varnish. Not thinned.....	1 part thinner to 3 parts varnish. Not thinned.....	1 part thinner to 12 parts varnish.	1 part thinner to 8 parts varnish. Not thinned.....	1 part thinner to 20 parts varnish.	{ All thinned alike 120 cc. varnish thinned with 30 cc. of the proper turpentine.
	2. Steam distilled spirits.do.....do.....do.....do.....do.....	
	3. Destructively distilled spirits.do.....do.....do.....do.....do.....	
	4. Destructively distilled spirits.do.....do.....do.....do.....do.....	
	5. Gum spirits (probably).do.....do.....do.....do.....do.....	
Varnishes remained tacky.	1. Gum spirits.....do.....do.....	About 5 hours.	About 3 hours.	7 hours.	3 hours 5 minutes.
	2. Steam distilled spirits.do.....do.....	About 6 hours.	About 4 hours.	4½ hours.	2 hours 40 minutes.
	3. Destructively distilled spirits.do.....do.....do.....do.....	4 hours.	Do.
	4. Destructively distilled spirits.	Average 36 hours.....do.....do.....do.....	3½ hours.	2 hours 20 minutes.
	5. Gum spirits (probably).do.....do.....do.....	About 3 hours.do.....	2 hours 15 minutes.
Varnishes harden in—	1. Gum spirits.....do.....do.....do.....do.....do.....	54 hours.
	2. Steam distilled spirits.do.....do.....do.....do.....do.....	47 hours.
	3. Destructively distilled spirits.do.....do.....do.....do.....do.....	51 hours.
	4. Destructively distilled spirits.	Average 48 hours.....do.....	About 24 hours.....do.....do.....	49 hours.
	5. Gum spirits (probably).do.....do.....do.....do.....do.....	Do.
Varnish was applied in—	1. Gum spirits.....do.....do.....do.....do.....do.....	4 after filler.
	2. Steam distilled spirits.do.....do.....do.....do.....do.....	
	3. Destructively distilled spirits.do.....do.....do.....do.....do.....	
	4. Destructively distilled spirits.	2 coats.....	1 coat.....	1 coat.....	3 coats.....	2 coats.....	
	5. Gum spirits (probably).do.....do.....do.....do.....do.....	

Gloss of varnished surface.	1. Gum spirits.....	Very little difference. All look more like piano varnishes than coach varnishes.	Good	No difference in gloss.	All good. Nos. 3 and 5 best. Nos. 1 and 3 show tears.
	2. Steam distilled spirits.				
	3. Destructively distilled spirits.				
	4. Destructively distilled spirits.				
	5. Gum spirits (probably).				
Odor is objectionable.	1. Gum spirits.....	This is worst.	Good	None objectionable.	Do.
	2. Steam distilled spirits.				
	3. Destructively distilled spirits.				
	4. Destructively distilled spirits.				
	5. Gum spirits (probably).				
Objectionable to work with.	1. Gum spirits.....	Only one that smells of straight turpentine.	In odor and tight body.	None objectionable.	Can work with all, but No. 3 is most obnoxious. No. 4 can not be worked any length of time in closed room.
	2. Steam distilled spirits.				
	3. Destructively distilled spirits.				
	4. Destructively distilled spirits.				
	5. Gum spirits (probably).				
Are difficult to work with.	1. Gum spirits.....	None seriously.	None are.	None are.	They all work nicely.
	2. Steam distilled spirits.				
	3. Destructively distilled spirits.				
	4. Destructively distilled spirits.				
	5. Gum spirits (probably).				
The varnishes rub down.	1. Gum spirits.....	This is.	None are.	Satisfactory.....	All rub well.
	2. Steam distilled spirits.				
	3. Destructively distilled spirits.				
	4. Destructively distilled spirits.				
	5. Gum spirits (probably).				

! The regular varnish used in striping buggy gears was thinned with the four turpentine.

TABLE 11.—Detailed opinions of the workmen on the varnishes used—Continued.

COACH FINISHING VARNISHES—Continued.

Data.	Varnish.	Devoe & Raynolds Co.	Murphy Varnish Co.	H. H. Babcock Co.	Fay & Bowen Engine Co.	James & Meyer Buggy Co.	Paint Manufacturing Association of the United States.
Behavior under the brush.	1. Gum spirits.....	Not free; stiff.	Works full and well.		Does not flow very freely.	Best flowing.	
	2. Steam distilled spirits.	Flows fair.	do.		Flows very freely		
	3. Destructively distilled spirits.	Flows free.	Works too full.	Flows fairly well.			No difficulty in flowing.
	4. Destructively distilled spirits.	do.	Works too thin.		Not as free as Nos. 2 and 3.		
	5. Gum spirits (probably).	Flows very free.	Works full and well.		do.		
1. Gum spirits.	Heavy body.			Little heavy.			
2. Steam distilled spirits.	Medium body.			Very light body.			
Character of body.	3. Destructively distilled spirits.	do.			do.		
	4. Destructively distilled spirits.	do.			do.		
	5. Gum spirits (probably).	do.					
	1. Gum spirits.			Silky appearance.			Works like regular turpentine. Cracks and crawls apart in striping.
	2. Steam distilled spirits.			do.			
3. Destructively distilled spirits.			Silky appearance; loses luster.	Think that the first and the last are the best varnishes.			
4. Destructively distilled spirits.			Best of lot.				
5. Gum spirits (probably).			Silky appearance.				
General remarks.							

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