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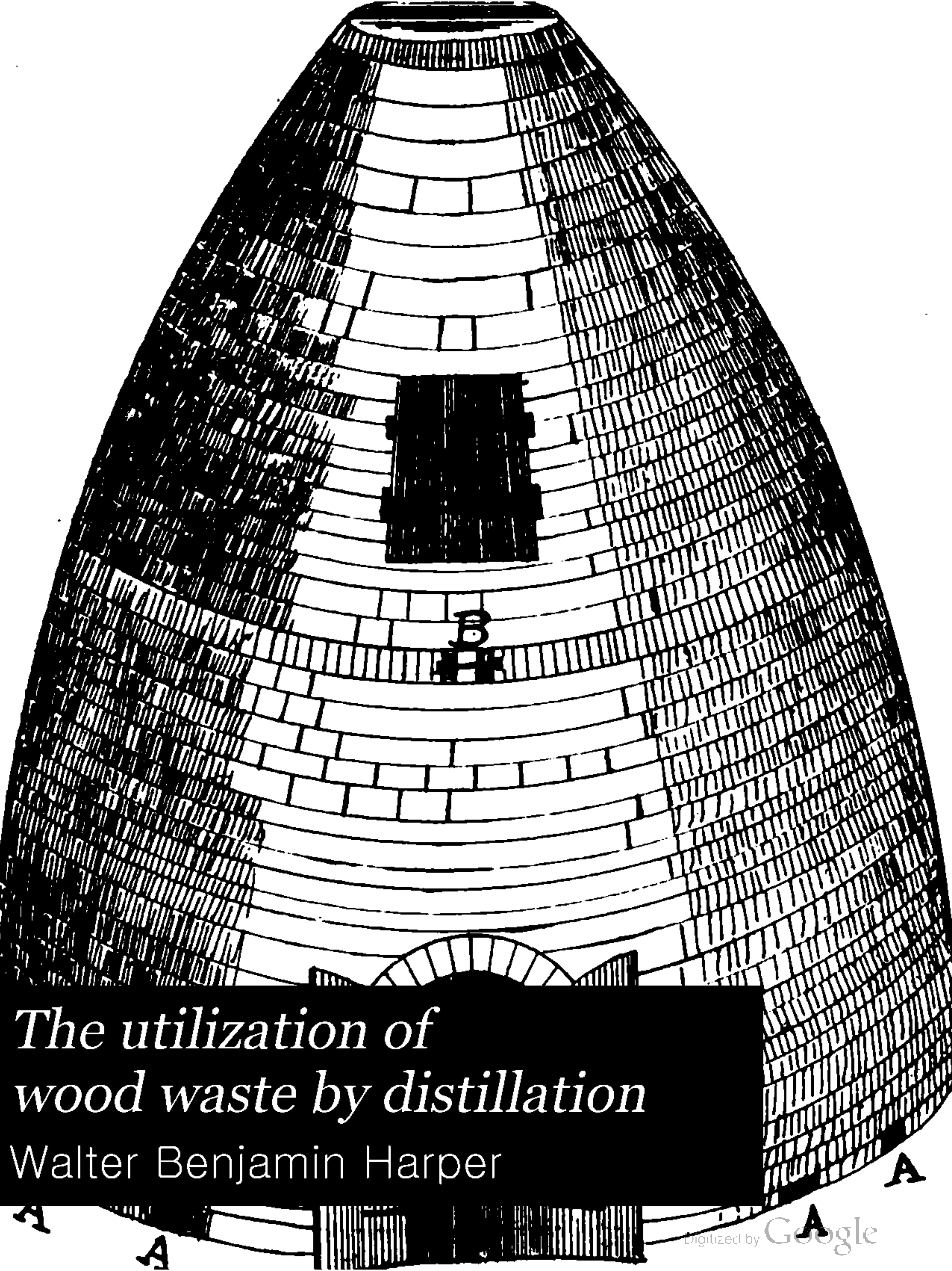

The utilization of wood waste by distillation

Walter Benjamin Harper

A GENERAL CONSIDERATION

. . . OF THE . . .

Utilization of Wood Waste by Distillation

The Utilization of Wood Waste by Distillation

A General Consideration of the Industry of Wood Distilling, including a description of the apparatus used and the principles involved

...ALSO...

METHODS OF CHEMICAL CONTROL
AND DISPOSAL OF THE PRODUCTS

...BY...

WALTER B. HARPER, M. S.

First Edition
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CONTENTS

CHAPTER I.

CHAPTER II.

CHAPTER III.

CHAPTER IV.

CHAPTER V.

CHAPTER VI.

CHAPTER VII.

CHAPTER VIII.

CONTENTS

CHAPTER IX. Page

CHAPTER X.

CHAPTER XI.

CHAPTER XII.

CHAPTER XIII.

ILLUSTRATIONS

ILLUSTRATIONS

PREFACE

The lack of literature on the subject of wood distillation, particularly that which relates to the treatment of resinous woods, led the author to believe that a description of the various processes that have been used or suggested to accomplish this purpose might be interesting and acceptable to many.

In the pine wood industry so much money has been wasted trying to carry out successfully the plans of over-enthusiastic promoters that it is well to direct along sane lines of investment any further capital that may be advanced for distillation processes. Great and wonderful results have been promised by promoters and in most cases the processes tried did not even yield a small profit. This bad result has been caused chiefly by the fact that those who have experimented with the various processes on the small scale have made erroneous deductions from the results obtained. Usually some feature that is essential to success has been overlooked and when the process is started on a large scale this feature is brought out so prominently that it cannot be successfully overcome and the plant fails. The greatest mistake is usually the estimation of the cost and quantity of the particular grade of wood with which the experiment was made. It usually develops that a sufficient quantity of wood of the right quality cannot be obtained. Many of these essential conditions for success are pointed out in the text.

The lumbermen should be the most interested as they control the forests. The great inducement to lumbermen to enter the business is to dispose of the vast quantity of refuse incident to the milling operations. The advisability of treating refuse will depend upon the quantity and its use for fuel. With the use of band mills and lath mills, the amount of refuse is getting to be so small that it is hardly sufficient to supply enough fuel to furnish the power for the mill. All distilling processes take considerable fuel, consequently it is only at those mills where the refuse has no value as fuel that it would pay to install a distilling apparatus. The discussion of these features is brought out in the book.

In the forest no successful method has been discovered to utilize pine or fir wood, taking the wood as it comes without selection and only such a process could be called a success economically. It is to be hoped that rotary processes may achieve that end, but the outlook is not promising.

It has been the author's intention to write this book in such a manner that the different phases of the subject might be touched upon and easily comprehended by an unscientific person. With the information herein obtained it might be possible for him, if suitably located, to establish a distilling plant on a sound basis. On the other hand, this information may save some parties from a loss in a contemplated investment. Furthermore the book ought to act as a stimulus to those who are already engaged in the industry.

Most of the credit for the success of the book is due to Mr. W. E. Barns on account of his interest in the matter and the expense incurred.

A great deal of information on the subject has been taken from the articles contained in the columns of the periodicals given in the appended list. The purpose of the author has been to give the opinions of others prominence, when in accord with facts. More explicit information relating to these articles would probably be acceptable, but in many cases the author had only clippings without date.

The author expresses his thanks to Dr. C. E. Coates of the Louisiana State University, for the use of his library, from which a great deal of the chemical information herein contained has been derived.

The arrangement of the contents might have been improved by combining all the information concerning a certain product under one head. However, the information required can in most cases be easily found by consulting the index and table of contents.

WALTER B. HARPER.

Laboratory of the La. State University.

BATON ROUGE, April, 1907.

THE UTILIZATION OF WOOD WASTE BY DISTILLATION.

CHAPTER I.

INTRODUCTION.

In the discussion of this subject the chief wood that will be considered is the long leaf yellow pine or Georgia pine, although some reference will be made to other kinds of wood.

The pine contains so much more gum and resin that it offers more opportunity for successful distillation than those kinds of woods which contain few of these substances. On account of the abundance of raw material any successful method of using the vast quantity of waste pine would be of great importance as an aid to the material welfare of the nation, and particularly to the South. The importance of this problem has now been partly realized by a large number of individuals. Some lumber companies in the South have already made investigation of the problem, but as yet none have decided fully what is the best method of utilization.

Distillation of the wood in closed vessels, although of very ancient origin, has been brought forward as a new means of practically disposing of this waste product. The increasing demand and consequently increasing price of spirits of turpentine have led manufacturers to seek some substitute. As an oil closely resembling turpentine can be obtained from pine wood, and even fallen and dead pine by distillation by heating with steam or direct heat, this method has the double advantage to the country of supplying a substitute for a commodity, the supply of which is evidently failing and at the same time utilizing a material that has been almost worthless heretofore. We give a description then of some of the attempts that have been made to produce a successful process of distillation. A great deal has been done, but there are many points to be worked up before a complete utilization can be obtained by this means. It is almost impossible to calculate the amount of raw material obtainable for this purpose, but in the South there are fully 1,000,000 cords that are annually going to waste either as saw dust or as fallen timber.

CHAPTER II.

HISTORICAL CONNECTION.

Probably the ancients noticed the fact that is so apparent to all who have made wood fires that wood chars on heating to a black coal. There then was noticed that a slow fire left more coal when it went out; after this in attempts to smother fires with dirt larger quantities of charcoal were produced, thus leading to the method that is even now prevalent of making charcoal by smothering lighted piles of wood with earth. We have charcoal produced in the South today by stacking wood and covering it with earth and igniting the wood at the bottom of the pile. This method of treating wood sufficed to attain the object intended—that of producing charcoal. That the vapors and gases produced were of any particular value, except perhaps to smoke meat, was not known until a much later date. Glauber in Miraculum Mundi, 1658, noticed the presence of pyroligneous acid in the cooled vapors, and Thènard in 1802 showed that this acetic acid, or pyroligneous acid, was the same as that made from alcohol; but it was not until the discovery of the presence of methyl alcohol by Taylor, 1812, that much attention was given to the recovery of the vapors. We find, though, that M. Philip Lebon in 1799 made use of the gas, and in 1801 he lighted his house with gas from wood. The gas was of low candle power as then produced, but Pettenkofer in 1849 showed that by rapid heating of the wood a much more luminous gas was formed, and this gas was used for a time in some of the old German cities.

A great many researches on the destructive distillation of wood have been made by Violette, Vincent, Stolze and others. As a result of these investigations it has been generally observed that for the production of gas and charcoal chiefly the distillation should be performed quickly in small apparatus as this affords means of quickly heating the wood; but for the production of oils and acetic acid large retorts heated slowly are better. That the method of firing influenced the quantity and nature of the distillate is shown by the following table:

Wood 100 Parts.	Total Distillates.	Tar.	Hydrated Wood Vinegar (Raw)	Per Cent Pure Acetic Acid.	Charcoal Dry.	Uncondensed Gases.
Hornbeam—						
Slow	52.40	4.75	47.68	6.43	25.37	22.23
Fast	48.52	5.55	42.97	5.23	20.47	31.01
Birch—						
Slow	51.05	5.46	45.59	5.63	29.64	19.71
Fast	42.98	3.24	39.74	4.43	21.46	35.56
Beech—						
Slow	51.65	5.85	45.80	6.21	26.69	21.66
Fast	44.36	4.90	39.46	3.86	21.90	33.75
Oak—						
Slow	48.15	3.70	44.45	4.08	34.68	17.17
Fast	45.24	3.20	42.04	3.44	27.73	27.03
Larch—						
Slow	51.61	9.30	42.31	2.69	26.74	21.65
Fast	43.77	5.58	38.19	2.06	24.06	32.17
Spruce—						
Slow	46.92	5.93	40.99	2.30	34.30	18.78
Fast	46.35	6.20	40.16	1.78	24.24	29.41

The above is from the tables given by Prof. Fisher in his Chemical Technology. The slow distillation represents starting with wood in a cold retort and heating slowly for six hours, and the fast distillation represents results obtained by placing the wood in glowing retorts and maintaining the temperature for three hours. This table will be of some service to a distiller wishing to know how he has been firing, as it is only necessary to compare the results obtained to get a working idea.

Stolze's table shows the following results obtained by careful carbonization:

100 Lbs.	Wood Vinegar Pounds.	Containing Acetic Anhydride Pounds.	Tar, Pounds.	Charcoal, Pounds.	Gases, Cubic Metres.
Birch	44.9	8.9	8.6	24.4	9.8
Beech	44	8.6	9.6	24.6	10.8
Hornbeam	42.5	7.6	11.1	23.9	10
Oak	43	7.7	9.1	26.1	10
Fir	42.3	4.2	11.9	26.6	12.5

And Assmus on a manufacturing scale shows as follows:

100 Lbs.	Wood Vinegar, Lbs.	Sufficient to make Calcium Acetic, Pounds.	Acetic Anhydride, lbs.	Tar, lbs.	Charcoal, Pounds.	Crude light oil, lbs.	Crude heavy oil, lbs.
Birch	46	5.2	3.9	8	23.5	1.2	4.6
Birch bark—1st extract.	22	0.6	0,4	30	18.6	21,6	3
Birch bark—2nd extract.	20	0.7	0.5	20	22	12	4.7
Oak	42	6.0	4.5	8.8	27.5	0.8	3.3
Fir	42	3.2	2.4	10.5	22	1.3	5.7
Pine	44.5	3.0	2.3	9.5	22.6	0.6	3.5

A noticeable feature is the small percentage of acetic anhydride in the wood vinegar yielded by the pine and fir; also in Stolze's table the large amount of gas yielded by fir. These features of the distillation of these woods explain why distillers of yellow pine do not try to save the pyroligneous acid or wood vinegar.

These are some of the results obtained in European experiments and practice. We are interested to know of early American attempts to utilize wood so that we may obtain the benefit of American experience. The manufacture of pyroligneous acid was begun in the United States by James Ward in 1830 at North Adams, Mass. The manufacture of acetate of lime and methyl alcohol was started in the United States by James A. Emmons and A. S. Saxon in Crawford county, Pa., and in 1874 George C. Edwards established the Burcey Chemical Works at Binghamton, New York, to refine the crude wood spirit produced by the various acetate manufacturers. In 1876 Dr. H. M. Pierce obtained a series of United States patents for inventions which he was to apply to the recovery of the smoke from charcoal kilns in Michigan. According to the census of 1900 in the Digest of Patents Relating to Chemical Industries, the name of M. A. LeBrun Virloy is found as having obtained a patent in 1863 for a special furnace for carbonizing organic matter. The next one, granted to A. H. Emory in 1865, shows that turpentine had been extracted from pine wood before this time. Several other patents after this mention the distillation of wood. In 1872 a patent was granted to J. D. Stanley, which is of especial interest, as he established a plant at Wilmington, N. C., which, although it proved a failure, owing to lack of sufficient financial backing and probably other causes, was transferred in 1878 to the Spiritine Chemical Co., who have continued it with more or less success until the present time. Since then numerous processes have been promoted, mostly copied from German or French methods, and each year adds new patents to the already large list. A description of some of these will be given later as a great many have some one little point either overlooked or not mentioned by the others: but first we must take up the general consideration of distillation in order to understand more fully the principles upon which they are based.

CHAPTER III.

PRINCIPLES OF DISTILLATION.

Distillation comprises that process which consists in heating substances in closed vessels with the intention of converting the substance into vapors and of condensing these vapors. Sometimes an indestructible substance is left as a residue which is not further acted upon by the degree of heat used. Sometimes the vapors are composed of different substances than the original, and at other times when the original substance is composed of a mixture of materials, the vapors of each separate material have a tendency to come off by themselves or with other bodies of similar physical properties.

In all cases of distillation we find that the vapors formed occupy a great deal more space than when the substance is in the solid or liquid state. This is well exemplified in the case of water that is turned into steam or water vapor; the space being nearly seventeen hundred times as great when not compressed. Generally vapors are compared with the solid or liquids in the ratio of 1,000 to 1. In designing distilling apparatus this point must be taken into consideration.

The object of distillation is to separate one substance from another; consequently it is a method of purification. Generally in the case of liquids the substances distill without decomposition, although not always so. Sometimes to prevent decomposition it is necessary to distill under a vacuum. Whenever the material distilling is overheated decomposition generally ensues. In the case of solid substances great difficulty is experienced in distilling them without decomposition. It is only those substances that are easily melted, or are of an elementary or mineral nature that can be so distilled. With organic substances decomposition generally occurs to a more or less extent.

Whenever this decomposition takes place it is usually called destructive distillation. The resulting condensed vapors are called products. When the resulting condensed vapor exists in the same form in the original substance it is called an educt. When a substance is easily converted into a vapor it is spoken of as being volatile. Some substances cannot be volatilized, such a substance is wood. This would be called non-volatile. Heat usually causes a substance to change to vapor and some substances become vapors at lower temperatures than others. Thus in a mixture containing two or more substances of widely different boiling points one would expect that the one with the lowest boiling point would vaporize or distill first. However, with substances whose boiling points are near together one may give off a heavy vapor which will retard its distillation. Owing to this a general rule is given by Wanklyn that "the quantity of each ingredient which distills will be found by multiplying its tension at the boiling point of the mixture by its vapor density."

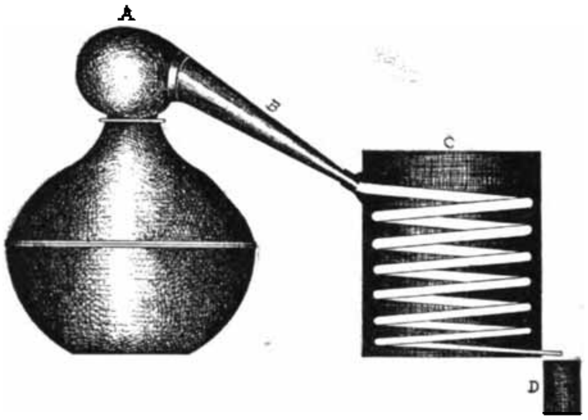


FIG 1—WATER STILL.
A—Head.
B—Arm.
C—Tank and worm.
D—Receiver.

To carry out the process of distillation the apparatus consists primarily of five principal parts: the retort or still; the still head, sometimes very complicated in construction in fractionating stills; the condenser, comprising tank containing cooling liquid, if not air-cooled, and the worm, pipe or other device through which the vapors pass to be cooled; the receiver, comprising any form of receptacle for catching the products of condensation, and the connections which are generally pipes connecting the various parts.

In Fig. 1 the various parts of a common water still are shown. This could be used for distilling other substances as well as water, as it has all the necessary parts. All that is necessary is to put the water in the still, place a fire of some kind under it, and add the cooling water. Generally there should be an overflow pipe in the tank so that the water as it gets hot can be forced over with cold water admitted at the bottom to take its place.

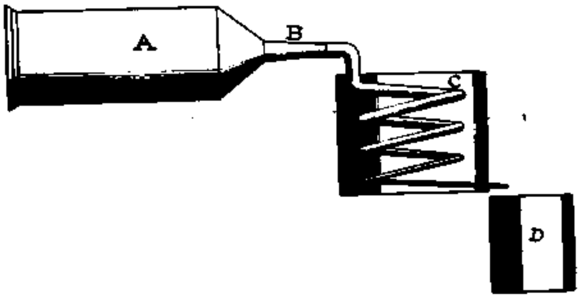


FIG 2—RETORT AND WORM.
A—Retort.
B—Arm.
C—Tank and Worm.
D—Receiver.

For destructive distillation a distilling apparatus would be more of the form shown in Fig. 2. This form usually has no still-head.

Our object being to distill wood, it is necessary to proceed by destructive distillation. In the early treatment of wood no apparatus was used to collect the vapors, but they were allowed to escape into the air; the residue left after all the volatile matter formed had been distilled was the only valuable part saved. The earliest arrangement was to cover over the wood with earth and set fire to it near the middle of the pile and allow only a limited supply of air to reach it. In this way the heat from that part of the wood which is burned distills the other part, leaving charcoal, as not enough air is admitted to allow this to burn.

As this method of utilizing the wood may be the best in some localities, a view of a kiln is herein presented as illustrated in Wagner's Chemical Technology. This is called an Italian kiln, as this form is much used in Italy. It consists of three or more poles stuck in the ground and separated from each other by wedges, N Fig. 3. The wood is stacked on end, as shown, the upper part being filled in with pieces lying horizontally. The whole mass is then covered with earth and ignited.

Another form of kiln is the Slavonian, similar to the Italian kiln, but instead of several poles at the axis there is usually but one. Also a passage way is made from the outer edge of the kiln at the bottom to the middle of the pile, thus making it easy to light the pile in the middle.

A form used in Norway called the Schwarten kiln is suitable for making charcoal from slabs. For an axis several planks are tied together and driven into the ground; around the axis blocks are built up to form a cone-shaped mound. Upon this mound the planks or slabs are placed, leaning on the edges instead of lying flat, thus enabling the heat to penetrate better into the mass. A kindling passage is left at the bottom and the whole covered with earth and ignited.

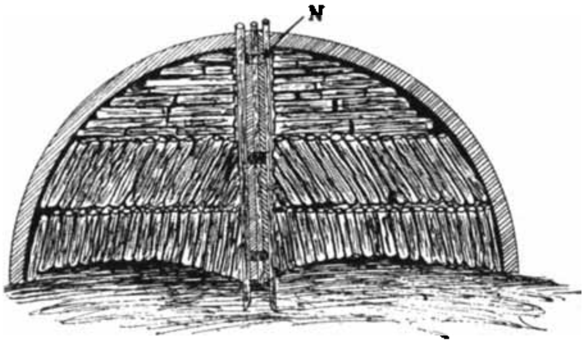


FIG 3—ITALIAN CHARCOAL KILN.
N—Wedge.

Considerable experience is necessary to burn a kiln so as to obtain the most charcoal and to obtain it free from brands or not thoroughly charred pieces. The first thing to do is to drive off the water with as little heat as possible. Considerable air must be let in at first in order to give the fire a good start and cause it to spread rapidly. The escape of steam and wood vapors make considerable noise as they escape through the earth covering and cause the earth to crack and sometimes explode, making the heap fall in. All exposed places are quickly covered over. The nature of the vapors emitted from the cracks determines the progress of the distillation, so as soon as the watery vapor and the heavy smoke of the next stage changes to a lighter color there is not much decomposable matter left. To remove this without burning the charcoal the air supply is diminished and the heat made as far as possible to travel from the top to the bottom, and from the middle to the circumference. When the smoke becomes pale and blue no tarry matter is left except near the edges of the kiln, the blue smoke seen coming from the combustion of the charcoal itself. To stop this all the airholes are closed as tightly as possible, and the kiln allowed to cool off. When sufficiently cooled the earth is taken off and any glowing charcoal quenched with water. A kiln requires continuous attention night and day until finished. The time varies with the size of the kilns, some taking as much as two weeks.

On account of the wood in the bottom layers being stood on end these kilns are called standing kilns and on account of their mound shape, meilers. In a horizontal kiln, where the wood is simply corded, we find attempts made to support it by outside means. A kiln of this type is shown in Fig. 4. Here the wood is stacked up and a frame made of posts surrounding it. The posts are connected with slabs, shingles, boards or even logs, thus forming an enclosure for the wood. A space is left between the stacked wood and support and this space filled with earth, the top also being covered with earth. One end is usually higher than the other, as is shown in the illustration. To ignite the heap a door is left in the smaller end, as shown at C. The fire once started requires only the attention needed to stop cracks and to fire evenly. As part of the wood becomes charred it is taken out through the small end. This form of kiln is much used in Central Europe.

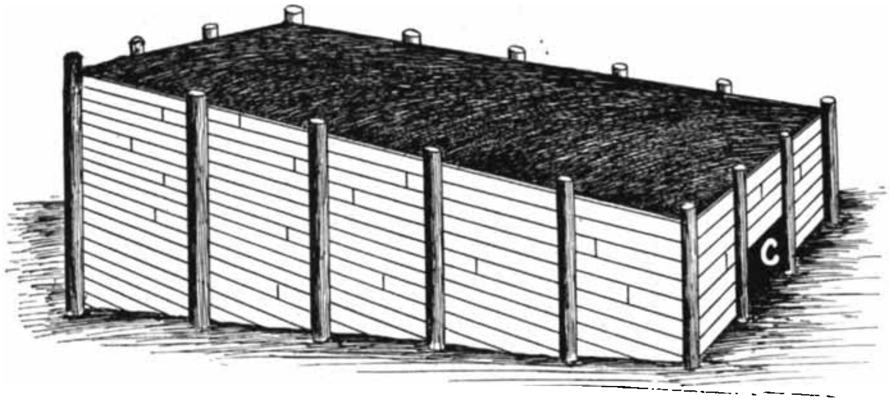


FIG 4—HORIZONTAL CHARCOAL KILN.

In this country charcoal making in kilns is not a very remunerative occupation as now carried on. In the South it is carried on mostly by negroes who succeed in making enough to keep them from starving. A kiln, though, such as the Italian form, is so easily constructed and can be built so near the raw material that this form has continued to be popular through the ages. There is no outlay of capital for apparatus and the covering material is always at hand. The drawbacks are that a large amount of wood is burned, the dirt gets into the charcoal, a great many brands are left and the charcoal being quenched with water easily breaks up.

A further form of kiln is used in Russia and the Carolinas for making pine tar. In this kiln the fat or rich wood is split into small pieces and stacked in layers. As the mass heats the tar runs to the bottom and is led by a trough into a barrel or pit in the ground. This is still a definite industry in the South and the tar thus produced has a ready sale. It is a great deal cheaper than it should be because of its being produced largely by turpentine hands during the idle season.

Fig. 5 shows two views of such a kiln. A mound of earth is made and the bottom made V shaped, forming a sort of trough which inclines toward the outlet pipe. The bottom is covered with clay and sometimes shingles, so the tar may be as free as possible from dirt. The fire progresses from the outside to the middle, being lighted at the bottom and the vapors escaping at the top. Most of the charcoal is consumed, as the chief object is to obtain the tar. The tar collects at the bottom of the kiln and is taken out at regular intervals, generally every morning. It is generally several days before it commences to run and it is usually hot. To keep it from igniting it is led at least three feet from any flame. This tar was usually put in barrels containing about 320 pounds and made on the spot; now the tendency is to sell in old oil barrels containing fifty gallons and quotations are now largely made on that basis. Notwithstanding the ease of construction in the earthen kilns, the supported kilns are to be found in many localities. Instead of making a supported wall of boards and earth, as in Fig. 4, brick is used without the addition of earth as a covering. One of the earliest forms was hemisperical, like a brick kiln with the brick left out here and there near the top for air holes. The wood was put in at the top and through a door at the bottom where the pile was ignited. The charge was fired in the usual way and when finished the charcoal was allowed to cool and then was taken out through the door at the bottom.

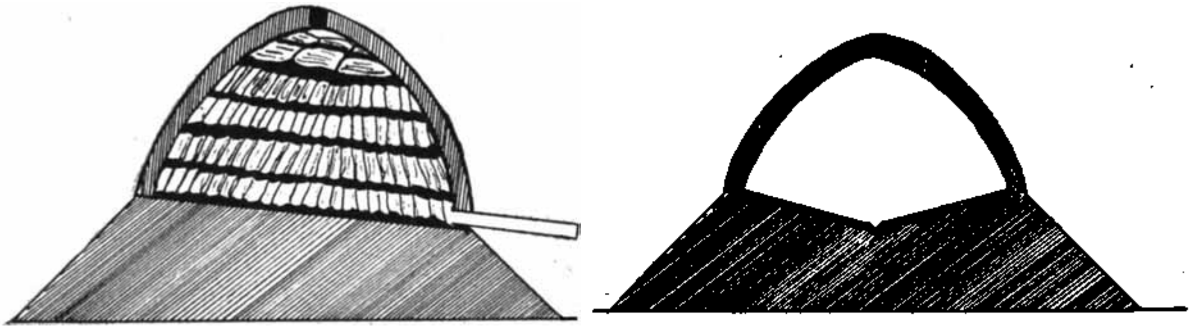


FIG 5—TAR KILN.

A form of kiln now in use is the bee hive or cone-shaped kiln, Fig. 6. These kilns are made of brick, usually 24 feet in diameter and 24 feet high, holding about forty cords of wood. The bricks are laid in a circle two courses thick, one of fire brick and one of red brick until about half way up when the remainder is finished with one course of red brick. To strengthen the walls bands of iron are placed at intervals and tightened with a bolt, as shown at B. It is claimed by some that brick kilns are not as good as the earth-covered mound or meiler, but experiments and practice show a yield of 45 bushels of charcoal against 35 bushels from the meiler, and at some iron furnaces the charcoal is said to work better. The difference in the yield is sufficient to pay for the extra expense for the brick.

In this kiln the two doors shown are of iron and on hinges. The upper one serves for putting in wood and the lower one for putting in wood and

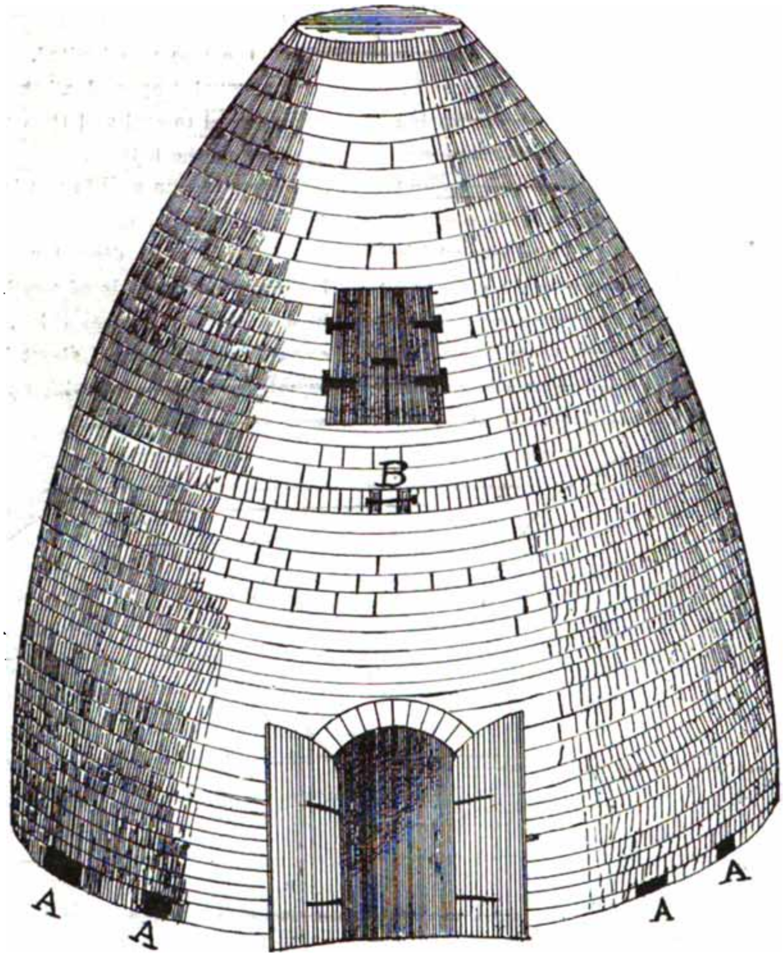


FIG 6—BEE HIVE OVEN.
A—Draft holes.
B—Tightening bolts.

taking out the charcoal. At the bottom, spaces are left for the admission of air; these can be stopped up with loose brick when necessary.

Another form of brick kiln is shown in Fig. 7. This is square in front with an arched top. It is usually built 16 feet wide by 16 feet high and runs back about 40 feet, outside measure. This will hold about 80 cords. This size is usually built a brick and a half thick and supported with 10x10 frame work timber. The cost of such a kiln in any locality can be readily calculated. The working of the kiln is similar to the bee hive, the doors being for charging and discharging and the holes at the bottom being for the admission of air.

In all these kilns no attempt was made until recently to save the vapors. In Michigan connecting pipes have been added for some time, but the practice did not become universal. It is not difficult to save the vapors from the brick kilns, all that is necessary being to lead the vapors through a condenser by means of a suitable pipe. Different methods of distilling with the object of saving the vapors will be taken up in the next chapter.

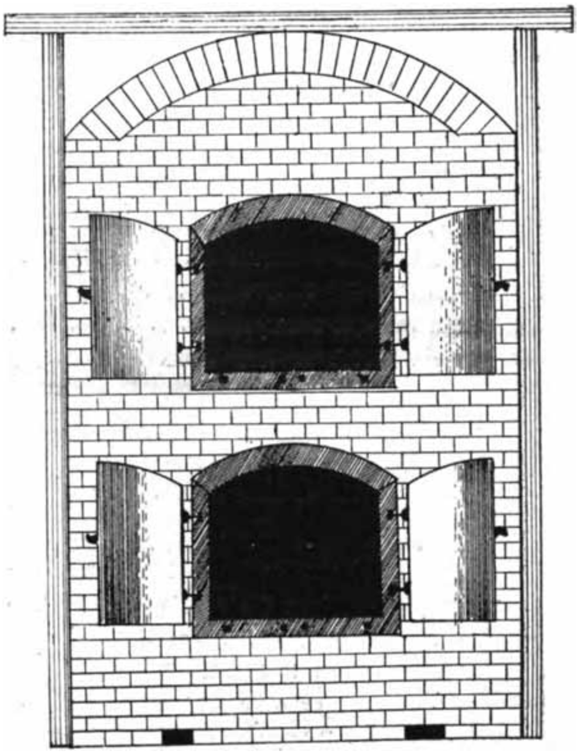


FIG 7—RECTANGULAR BRICK KILN.

CHAPTER IV.

APPARATUS NECESSARY FOR DESTRUCTIVE DISTILLATION.

As has been previously stated, the necessary apparatus for destructive distillation consists of a retort, arm or connecting pipe, condenser and receiver. The earliest forms of retorts were probably the brick kiln. This form is to be distinguished from the iron retort in that air has limited access to it, whereas an iron retort is usually a closed vessel, being as air-tight as possible.

In Sweden the brick kiln was modified from those previously described, when the advisability of saving the vapors became of sufficient importance. The connecting pipe made a definite outlet for the products of combustion and a means was soon devised to regulate the supply of air. The Swedish oven, so-called, which was thus evolved, took the form as shown in Fig. 8. This is a hemispherical shaped kiln or oven with an opening at the top A with cover B by means of which the wood can be dropped in. At C is a door serving the double purpose of allowing the wood to pass in and as an opening for drawing out the charcoal. At D is another door controlling the air supply which passes along the passageway E to the grate F upon which the wood is placed. The heat for charring is supplied by the combustion of a part of the wood in the oven, the vapors and gases from the combustion passing through the pipe G to the condenser. The size of the grate can be varied to suit circumstances. As shown, this oven takes a great many bricks, but it can be made of less thickness; the extended doorway at C can be omitted and the door placed directly in the wall.

In this country in the Pierce process ovens the shape of a brick kiln are employed. The uncondensable gases formed are led under the kiln and burned with just enough air for combustion and the heated gas is passed directly through the spaces between the wood in the kiln. This process will be described later.

An attempt was made by Hahnemann to heat the wood from the top of a brick kiln with a cylindrical shaft in the middle with openings at the bottom so that the gases of combustion could pass down through the wood, and by following the shaft escape at the top into the air. A pipe at the bottom was supposed to carry off the vapors. It can be readily seen that the light vapors from the distillation would also escape at the top.

A modification of the bee-hive form was also constructed. This had an inner cone for the wood and the space between the two walls was used as a furnace to heat the inner wall. The products of the distillation passed out at the bottom. The idea was a good one, but the furnace is not of the best form to get satisfactory results from the fuel.

To heat the wood in a retort, and at the same time not have the flame from the fire to touch it, Reichenbach devised an oven consisting of a rectangular brick chamber made as air-tight as possible, and mounted with suitable doors for the admission of the wood. The furnace gases were then passed through the chamber by means of large closed pipes. These pipes becoming red hot, the heat was communicated to the wood and the contents of the oven distilled. The vapors and tar formed were taken off at the bottom.

It is evident that ovens have many disadvantages as a means of carbonizing wood for the recovery of the liquid products of distillation. As the desirability of excluding the furnace gases became apparent, apparatus was devised that would more readily transmit heat. Cast-iron was resorted to, then clay, and then wrought iron and steel. At the present time most retorts are made of boiler plate, although some use cast-iron and a few clay. A clay retort does not readily burn through, but it is difficult to keep them tight, as they are so apt to crack, thus causing them to leak when under pressure. Cast-iron retorts are made thick in order to have strength and have an advantage over wrought-iron in that they do not readily burn through. They are also liable to crack, and when the crack is large a disastrous explosion will ensue; also they cannot be repaired easily.

A boiler plate retort offers many advantages in that being thinner, the heat is more readily transmitted, and although they may sometimes crack from unequal expansion and contraction, and are sure to burn through sooner or later, still they can be easily repaired by patching either with a bolt patch with gasket or preferably by riveting.

Clay retorts are generally of one form, the ⌓ shape of the coal gas retort, and are used in a similar manner. Cast-iron retorts are usually simple in shape and are like some forms of vertical and horizontal steel retorts. Wrought iron or steel retorts assume various shapes; some are rectangular boxes, some are boiler shaped cylinders and some are of irregular shapes.

Ovens or boxes are not used in the pine wood distillation, but could be. These ovens are set in pairs in brickwork and are provided with large doors at one end and three or more delivery pipes at the side of each oven. They are usually 27 feet long, 6 feet wide and 7 feet high inside, and rails are laid upon the floor of the oven by which steel cars loaded with cord wood can be run in. These cars hold 2½ cords of wood, and an oven of this size will hold two cars. Some ovens are 48 to 50 feet long and capable of receiving four cars at one charge. These ovens are much used in localities where there is natural gas for fuel.

The pine wood distiller has had more experience

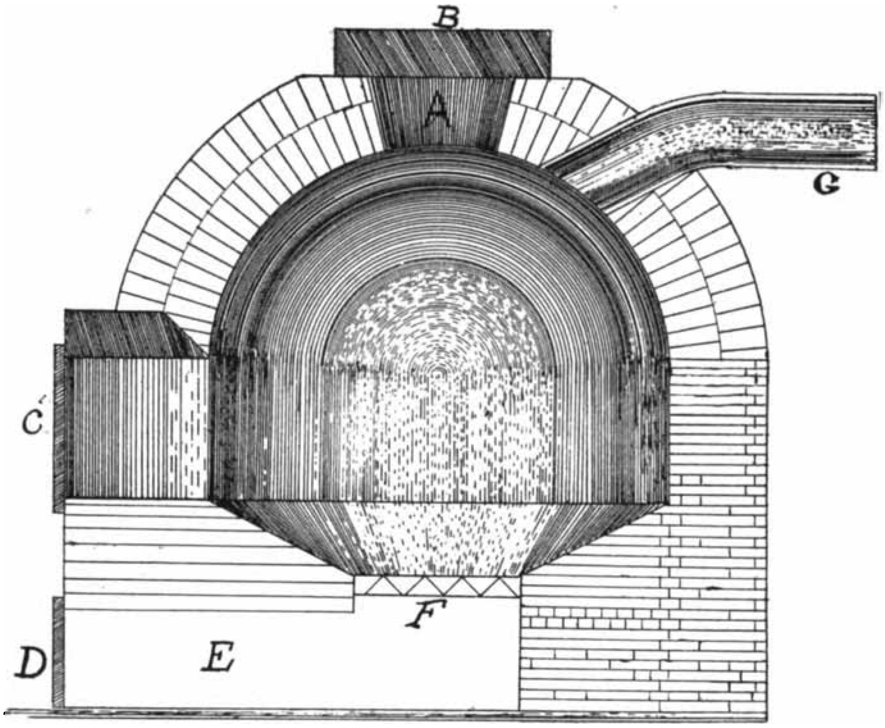


FIG 8—SWEDISH OVEN.
A—Air opening.
B—Cover.
C—Door.
D—Air supply door.
E—Passage way.
F—Grate.
G—Pipe to condenser.

with cylindrical iron retorts. These are made of all shapes and sizes, varying more to obtain patent than for practical utility.

The size of retort for turpentine distillation seems to be an unsettled controversy. Some claim small retorts are more efficient and some claim that large ones are better. In reality much depends upon the method of working and the products sought. One thing is certain, and that is that a large retort cannot be heated in the middle as easily as a small one when the heat is applied by external firing. Wood is a non-conductor of heat, and if the retort is well filled, as it should be to save space, the wood near the shell of the retort is bound to be burned before that in the middle has been thoroughly charred. This was one reason why Reichenbach put pipes in his kiln as mentioned before, so that the heat could be more evenly distributed. To offset this apparent disadvantage, users of large retorts try to make use of the radiating power of heated brick. This is exemplified by the working of a baker's oven, in making bread, the radiated heat from the vaulted arch concentrating at a common center makes the heat in the middle greater than it otherwise would be, thus the inner part of the loaf is cooked without burning the outside.

With ordinary firing of a retort only portions of a shell are heated to the same degree, but with radiated heat the temperature is kept more even. Although a large retort sems to be a disadvantage when fired by a direct heat, such cannot be the case in the steam process for extracting turpentine where steam alone is used. Here the steam can come in direct contact with all of the wood alike; in fact, it can be placed in the middle if need be. In this process, then, the only limit to the size of the retort need be the mechanical drawback incident to the rapidly filling and discharging when intermittent processes are used.

In early practice with retorts they were made of such a size as to allow the contents to be thoroughly charred in twelve hours with the necessary conditions for yielding the most products. Thus we find retorts 5½ to 6½ feet long with a diameter of 2¼ to 3¼ feet for horizontal retorts and for vertical ones a diameter of 4 to 5 feet and a height of 7½ feet.

Gradually the size has been increased until now we have them of such a size that they can be charred and emptied in 24 hours.

Retorts used in the hardwood industry, and now used with some success with pine, are made about 9 feet long and about 50 inches in diameter and are provided with tightly fitting doors and an outlet pipe of about 15 inches for the vapors. They are sometimes set in pairs, sometimes single and sometimes not protected from the direct flame, but should be, as they will not burn out so readily nor buckle so easily. These retorts hold less than a cord, so in the pine wood distillation it is better to increase the diameter about 6 inches so that they will hold a full cord. Cars are seldom used on retorts of this size, as they cut down the space. The thickness of the retort varies with the size, a one cord retort being ⅜ to ½ inches thick. Vertical retorts are built about this same size.

Other sizes are to be found, though, in the pine wood distilleries, varying from 3 to 9 feet in diameter and 5 to 30 feet long.

In designing a retort there are certain things to be taken into consideration when external heat is applied. There is no perfect retort for wood distillation, nor can there be, for such a retort would require that every part of the wood should be heated to the same degree of heat at the same time and for the same length of time. If such a one were constructed it would not be possible to charge it. One would think that a large cylindrical retort of very small diameter would be the best. Such would be the best as far as heating the middle is concerned, but we find, unfortunately, that the vapors evolved are themselves decomposed when kept in contact with the hot sides of a retort, as they would be in passing from one end to the other. This might be obviated by having several openings for vapors to escape, but this would necessarily increase the cost of construction. To get the capacity and at the same time to avoid having the length too long and the diameter too great, we find that the compromise is effected by having the retorts of such a length that but little decomposition takes place and of such diameter as to enable the charge to be finished in a given time.

A horizontal retort is shown in Fig. 9. This shows a larger size fitted with rails for a car of wood. Retorts of this class when the wood is to be destructively distilled, should be made of as good steel as possible, with a high tensile strength and with as few sections as possible. The size should be arranged so as to allow of its being made of commercial sizes of steel. The various sections cannot be rivetted too carefully, as a defective rivet is very troublesome, particularly if in a position that is difficult to get at. If possible, the various pipes should be so connected that the retort might be turned when it begins to burn. Outlet pipes should be as large as possible, so as to allow of the rapid escape of the vapors. Arrangements should be made to allow the placing of a pyrometer and any necessary gauges. To make the heads tight cast-iron rings are usually placed in one end of the retort which fit with corresponding rings in the doors. By the use of bolts or wedges the doors can thus be tightly fastened. If the doors are small they are hung on hinges, but if large it is necessary to have a wheel on the bottom to support the weight when the door is opened. Sometimes the doors are counterbalanced by weights similar to a dry kiln door, and sometimes hoisted by suitable hoisting machinery, such as engine, winch, crane or crab.

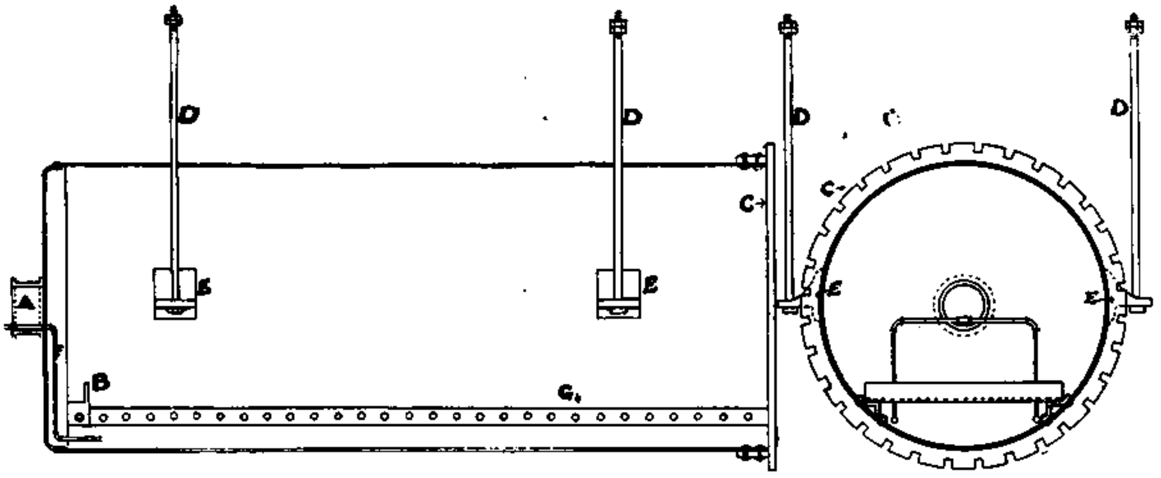


FIG. 9.

A—Exit pipe to condenser.
B—Car stop.
C—Cast iron ring.
D—Supporting rod.
E—Lug.
F—Steam pipe.
G—Track.

To support a retort in a furnace it is preferable to keep all the weight away from the brickwork. This can be done conveniently either by hanging by means of rods from I beams or by using lugs similar to those found on boilers, and placing iron pipes or posts under the lugs, in all cases allowing for the expansion of the retort. Whatever means of support used, any part of the support exposed to the direct action of the fire should be made of cast-iron or some material not easily burned.

A retort furnace should be made of brick and well lined with fire brick. A furnace suffers greatly from the continual heating and cooling incident to distilling. Only the best work of the masons is suitable for the purpose. The joints should be made thin and the fire brick should be laid with a coating only of the best fireclay, that kind from which brick was made being preferable.

To support the furnace walls a boiler front is very satisfactory and adds greatly to the appearance. The brickwork in horizontal retort furnaces that is just above the retort has a tendency to crack, so the boiler front should extend around the top of the retort. The walls through and through should be suitably tied with long rods, anchor rods not being as good. A buckstay placed horizontally above the rear end of the retort and connected with the boiler front with a long bolt will help the back wall. Furnaces made with these precautions are found to give but little trouble. Arrangements can be made, and one form is the subject of a patent by means of which small rollers can be placed in the cooler parts of the brickwork.

The retort can be lowered on these rollers and turned, thus exposing a new surface to the hottest part of the fire when one part is burned.

A vertical retort and setting is shown in Fig. 10. The same rule applies with these as with horizontal retorts. As the vapors rise they are apt to be decomposed at the top, as the heat of the furnace naturally rises. By keeping the bottom cool they serve as a ready means of extracting tar, although a great deal of retort capacity is lost, as the wood ought not to be placed where the heat cannot thoroughly char it. These vertical retorts are much used in France and have the advantage that they can be lifted out of the furnace by means of a crane and a new retort filled with wood placed in while the other is cooling. Modifications of this idea are found in the South and West. One plant at Georgetown, S. C., uses a brick retort and runs in a basket. On the Pacific coast an iron retort is used instead of the brick. It is fitted with an open work basket and this is lowered into the retort, filled with wood, the retort cover bolted on and the wood distilled and the charcoal immediately lifted into an iron cooler. This saves the wear and tear on the retort and furnace. The distilled products go out at the bottom. In some vertical retorts they are made slanting to let the charcoal slide out at the bottom.

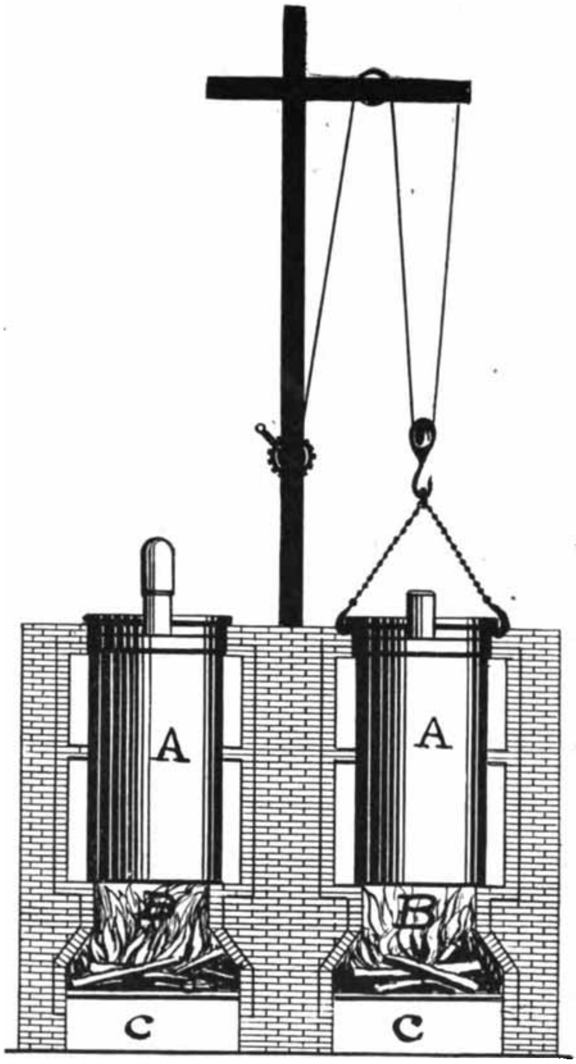


FIG. 10—VERTICAL RETORT.
A—Retort.
B—Combustion chamber.
C—Ash pit.

There are several kinds of special retorts, but most of them deal with sawdust or hogged wood. There are but three that claim to be continuous in operation, the remainder being intermittent. One form, the Bowles patent, is said to be in successful operation with hardwood sawdust. The object of all seems to be to stir up the wood so that the steam can play more readily upon the surfaces of the pieces and also to prevent the formation of channels. These forms will be described under PROCESSES.

Charcoal Coolers.—These consist of sheet iron boxes made for use with those retorts where the charcoal is taken out hot. They have a form corresponding to the use to which they are to be put. Where cars are used they are pulled out hot and run into a box of a suitable shape. Where baskets are used they are drawn into cylinders of similar size and shaped to the retort. In another form sheet iron cars are run up to the door of the retort, the door opened and the charcoal quickly raked out into the car, a cover put on and the edges luted with clay and the whole wheeled away to cool.

Connections.—The connecting pipes between retort and condenser should be as short as possible where direct connection is made between each retort and a condenser. In some plants several retorts are connected with one condenser. In these cases a long main pipe is necessary and some way must be devised to keep the vapors from one retort getting to the other. This can be done by means of valves or by a hydraulic seal, such as is used in the hydraulic main of gas works. The latter method is much to be preferred, as valves stick and if large and made out of iron are gradually eaten by the acetic acid vapors and are not tight. On account of the destructive action of these vapors upon iron it is advisable to use copper which is little attacked. Cast iron is better than wrought iron, which should not be used at all. These pipes should all be large and so arranged as to be easily cleaned; it is better to get cooling water on them in some way, as this will prevent the formation of a deposit of tarry matters that would otherwise adhere strongly to the pipe. This cooling is very important when working with wood rich in resin and tar. The size of these pipes can be determined by calculating the amount of vapors evolved (see yields) from the wood in a given time, the vapors occupying at least 1,000 times more space than the liquid products. To be safe make it 1,700 times, which, with the cooling effect of the air or water on the connecting pipes, will leave room for the

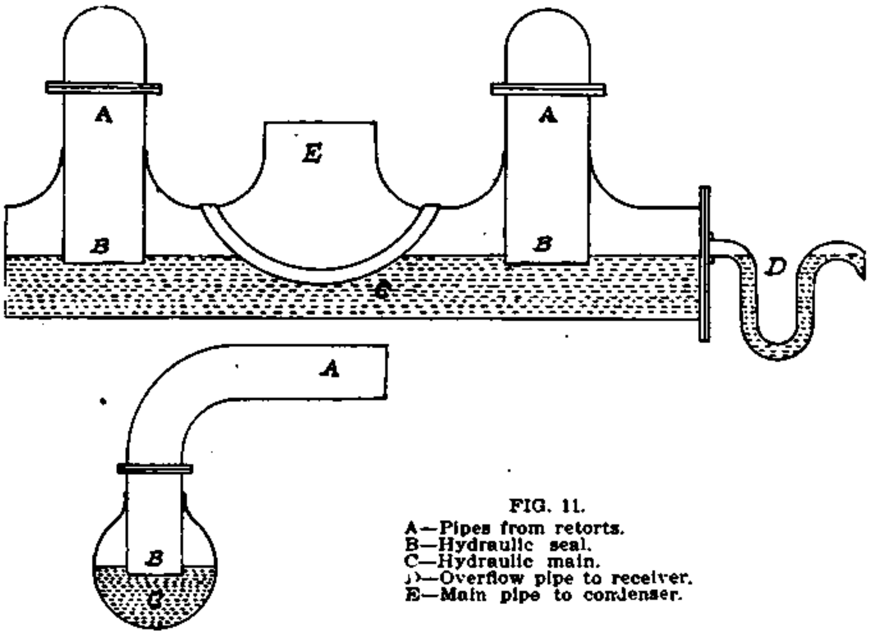


FIG. 11.
A—Pipes from retorts.
B—Hydraulic seal.
C—Hydraulic main.
D—Overflow pipe to receiver.
E—Main pipe to condenser.

rapid exit of the gases. There should not be over one pound pressure. The pressure being small, the thickness is only great enough to make the pipe of sufficient rigidity to stand outside strains. Of course, in steam pressure turpentine retorts the thickness must be large enough to stand the strain from the inside. See Fig. 11 for illustration hydraulic main. In addition to the connections between retorts and condensers it is necessary to pipe the gas either to a holder or to the furnace. The gas is separated from the condensed products in the condenser by means of several devices, the most simple of which is shown in Fig. 12 A. This consists of nothing but a brass T, the liquid falling and the gas rising being kept from following the liquid by means of the goose neck, as shown. If the pressure became too high the liquor in the bend might blow out and the gas escape. Gas valves are sometimes carelessly left closed, and this blowing out would at once give warning. The pipe carrying the gas should be made of copper, but as most of the acid is taken out of the gas other materials are often used, as copper is very expensive. A wooden pipe would be a very suitable pipe. By giving the pipe a backward slope liquid products that might be mechanically carried along with the gas would fall back. By placing a box with lime in connection with the pipe the acid would be caught and the pipe would be free. This box would have to be cleaned and fresh lime added from time to time, the acetic acid being saved where acetate was made regularly. Another form of separator is shown at B, Fig. 12, and another at C, Fig. 12. The one at B is used at several places in the South. In this form the gas escapes from the top of the cone-shaped head and the liquor is drawn off by means of the cocks, through the funnels shown, connected with pipes leading to tanks made to receive the several products of distillation, such as turpentine, tar-oil and tar. In the one at C, used at Lake Charles, La., the end of the condenser is led under the liquid, thus forming a hydraulic seal to prevent the gases from re-

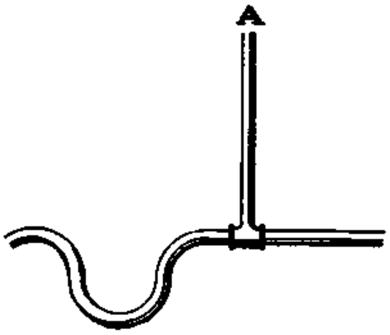


Fig. 12-A.

A—Gas to air.

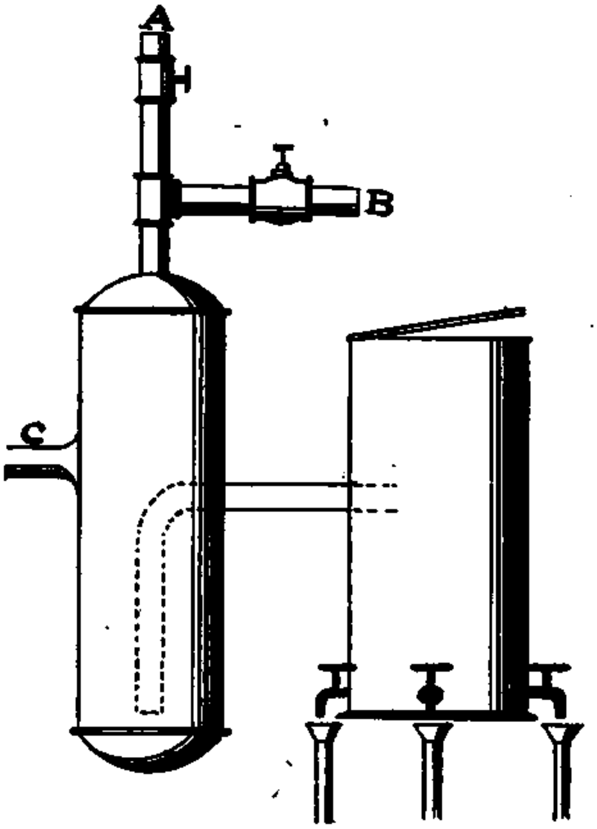


Fig. 12-B.

A—Gas to air.
B—Gas to furnace.
C—From condenser.

turning to the retort, if for any reason the gas should explode or the retort cool off suddenly. This causes only a slight back pressure, as the end of the condenser only dips under the liquid about three-fourths of an inch. This contrivance also causes a steady flow of condensed products from the goose-neck outlet.

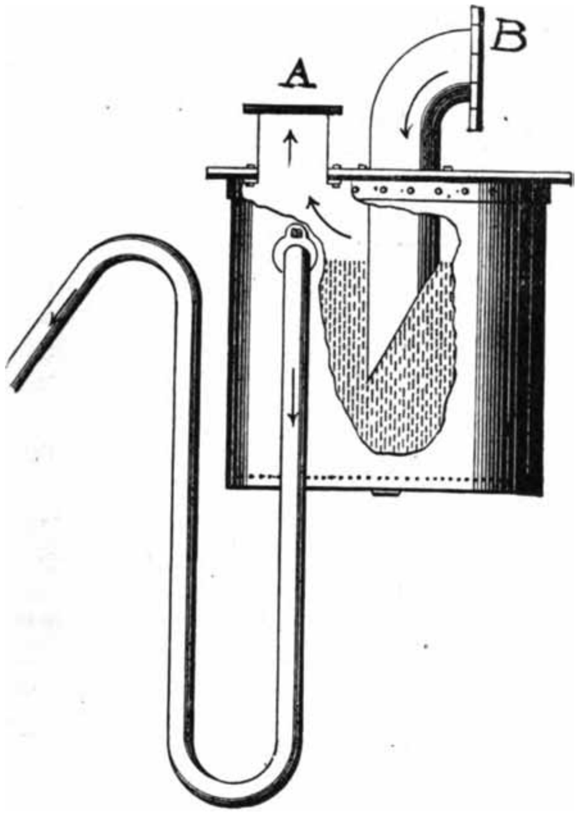


FIG. 12-C.
A—Gas outlet.
B—From condenser.

Sometimes other contrivances are placed in the connecting pipes, such as tar separators, vapor absorbers and milk of lime receptacles, but the best practice seems to be to get the product out of the wood as soon as possible and do the refining afterwards in more suitably constructed refining apparatus. When the condensed products are not caught directly in the receivers, a pipe or trough is used to convey them to the receivers. As a general rule, whenever the vapor contains acid, all pipes and vessels should be made of copper or wood. We find wooden troughs very extensively used for conveying the liquors.

Condensers.—One of the most important parts of a wood distilling plant is the condenser. There are three general forms used, namely, the worm, tubular and box condensers. In addition to these there are modifications of the three. The efficiency of a condenser depends upon the amount of cooling element and the length of time each particle of the vapors is allowed in contact with the cooling surface. They should be made of copper where there is acid.

Small tubes offer more surface in proportion to their cubic contents than large ones, consequently we find that tubular condensers are made with numerous small tubes rather than with a few large ones. However, they must not be too small in the larger sizes of condensers. A worm condenser must be made large, in order to take the large volume of vapor; as the vapor condenses the pipe can be made smaller toward the end. These large pipes increase the cooling surface in this form of a condenser, for they must be made just as long as a small pipe having enough cooling surface, because the gases and vapors would pass through a short condenser before the middle current in a large pipe would have time to strike the cooling walls and be condensed. It seems to be a rule with gases and vapors that they cannot be heated or cooled very rapidly unless they come in contact with some solid substance, in the above case the walls of the pipe. For this reason the box form of a condenser is not very efficient, owing to the large volume of vapor present in the chamber at one time in comparison to the cooling surface. The most efficient condenser costs the most, hence we find that the other forms are used in preference on account of original cost only.

The vapors from the distillation of wood are of such a nature that it is absolutely necessary to cool them thoroughly, otherwise some of the vapors will get into the gas pipe and not only tend to destroy it, but will themselves be lost. Furthermore, it is advisable to cool them quickly so as to relieve any pressure that might form in the retort. The more pressure in the retort the more tar. etc., will be decomposed, forming permanent gases. In reality, some form of exhauster ought to be used in destructive distillation plants, so as to remove the products of distillation rapidly and thus prevent the decomposition in the retorts. Many plants are now introducing such apparatus. Any form of rotary exhauster, such as are used in coal gas works, would be especially adapted for this purpose.

A condenser for wood distilling comprises in addition to the worm, etc., a tank of some kind to contain the condensing water. These tanks can be made of wood or of tank steel and should be provided with an inlet pipe at the bottom for cold water under pressure and with an outlet pipe at the top to remove the hot water forced over by the cold water coming from below. In this way the nearly cooled vapors coming in contact with the walls of the pipe cooled by this entering cold water become completely cooled and at the same time the hot water at the top being much cooler than the incoming vapors, also has a cooling effect, and thus the water is used very effectively. An iron tank is better, as it is not so apt to leak.

On account of the desirability of cooling the vapors quickly those plants using a main line for all the retorts should make provision for cooling this pipe with water. The surface of the pipe would in this way act very effectively in cooling off the vapors. Furthermore, these main pipes get very hot and sometimes the heat becomes so great as to carbonize some of the vapors and a heavy deposit is formed that is sometimes difficult to remove and might at times block the pipe. With water-cooled pipes this would not happen. The heavy tar vapors would condense in this main and flow out the overflow pipe shown in Fig. 11, and thus reach the receiver. The first part then of the condensing apparatus should be the hydraulic main when such is used.

In some plants making wood turpentine by the destructive distillation method in order to avoid the bad odor that is so difficult to remove from the turpentine produced, two sets of condensers are used. In such cases the only way to direct the vapors is by means of valves. The connecting pipe must be cooled and the water surround the valve or a great deal of trouble will be caused by the valve sticking generally from the tar, etc., getting on to the threads of the stem. Where the turpentine is distilled (generally through a small pipe) a valve is turned off and a larger one leading to the other condenser is opened and the distillation is continued. Except for a very particular grade of goods this method is not to be commended as it entails an extra expense for a second condenser and also for a very large valve which if made out of brass, as it ought to be, would be very expensive, and if made of cast iron the threads of the seat would eventually be eaten out by the vapor and the valve practically destroyed. If a separation of the distilled products is to be made it should be made at the end of the condenser similar to the method pursued in distilling petroleum; any small amount of tarry matter in the condenser remaining after the distillation could be removed, either by washing or draining, or if mixed with the first runnings of the next turpentine distillate it can be sufficiently removed by the refining process used.

Box Condenser.—This form of a condenser is simply a rectangular receptacle made of copper for receiving the vapors. It has the necessary inlet pipe for the vapors and an outlet pipe for the condensed product. It is placed in a wooden or iron tank containing the condensing water. In the Bilfinger process this inner chamber is lined with wood. A reversal of this system might be better by putting the cooling water inside and the vapors in an outer chamber; it would have the advantage of being cooled some by the outside air, and at the same time have a little more cooling surface on the water side. It might be a little more difficult to construct, but would need less material to get the same effect if iron were used in its construction. With copper the cost would be much increased as there would be four extra walls.

Worm.—The next simplest condenser is the worm.

As before stated, one end must be very large in order to receive the large volume of vapors, also it occasions them to move more slowly, thus causing them to remain longer in contact with the cooling surface. The worm condenser is shown in Fig. 1 and Fig. 2. To receive wood distillates all that is necessary is to attach some form of gas trap or separator, such as shown in Fig. 12, so as to separate the gas. The tank shown is for the containing water and should have an inlet pipe at the bottom for cold water and an overflow pipe at the top for the hot water to escape. Several forms of pipe coolers are used in the various industries. One form is seen in artificial ice factories where several rows of pipes are connected with return bends and a perforated pipe above all from which a stream of water, the entire length of the pipe, falls upon each pipe in its descent. This water is cold at the top layer of pipes, but as it reaches the bottom it becomes quite warm, finally falling to the floor or ground and running off. In such an apparatus the liquid or vapors should preferably come in at the bottom and come out at the top. Such an apparatus is not good for vapors, although suitable for cooling liquids. With vapors the condensed matter would cause back pressure unless the vapors came in at the top, in which latter case they would not be thoroughly cooled unless a great amount of water was used.

In the gas works we find the pipes placed vertically instead of horizontally and cooled only by air. Provision is made for the collecting of the condensed products by means of a partitioned box at the bottom. In this way the condensed product falls into the box and the gas passes on to the next chamber following the pipe, and any other further condensed product is separated in like manner until the gas escapes from the condenser.

Another form similar to what was used at a wood distilling plant at New Orleans was of the style shown in Fig. 13. This is sometimes called a box cooler because of its outward shape, but should be distinguished from the box condenser before described. In the distillation of wood various tar-

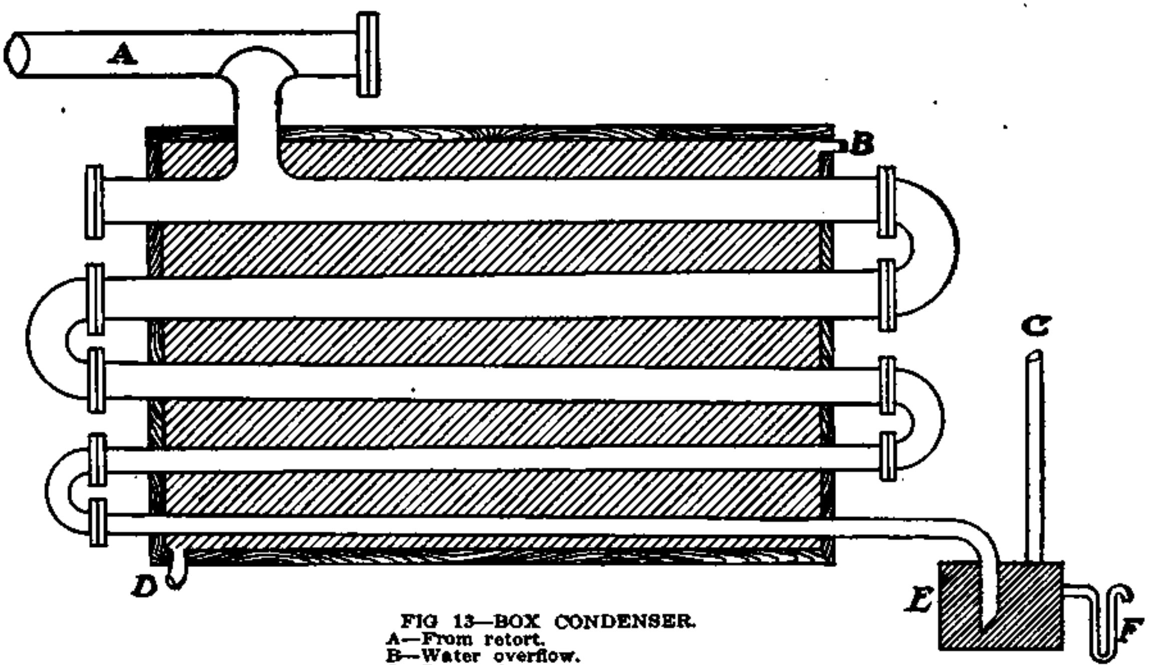


FIG 13—BOX CONDENSER.
A—From retort.
B—Water overflow.
C—Gas to furnace.
D—Water inlet.
E—Gas trap.
F—Goose-neck discharge.

ry matters form which sometimes adhere to the pipe and cannot be washed out. One can easily understand that an ordinary worm condenser would be troublesome if it should have occasion to block. To avoid this the arrangement shown in Fig. 13 is made. First, the pipe leading from the retort is connected with a T instead of a direct elbow and one end fitted with a cap or flanged head that can be removed for cleaning purposes. Then each return bend is on the outside of the box and being flanged to the ends of the pipe protruding through the box they can be readily taken off for cleaning purposes. Of course, proper stuffing boxes should be made for the ends of the pipe, so that the pipes can expand and at the same time no water escape where the pipe goes through the box. As in the worm condenser the pipes can be gradually made smaller toward the discharge end. Sometimes the first pipe is subdivided into two connecting pipes so as to give the condenser more cooling surface where the vapors first come in contact with it. At the outlet the usual gas trap should be placed. Instead of having the pipes directly under one another they can be placed with alternate pipes to one side and only a little lower, thus making the box wider and not so deep. It is best to give each pipe a rather sharp decline so that the condensed products will readily flow out.

Counter Current Pipe Cooler.—Instead of the pipes being placed in a box as in the above form, each pipe may be surrounded with a larger size pipe containing the cooling water. These water pipes are connected with one anotner at alternate ends. The return ends are not covered, but remain exposed as in the box cooler so that they can be

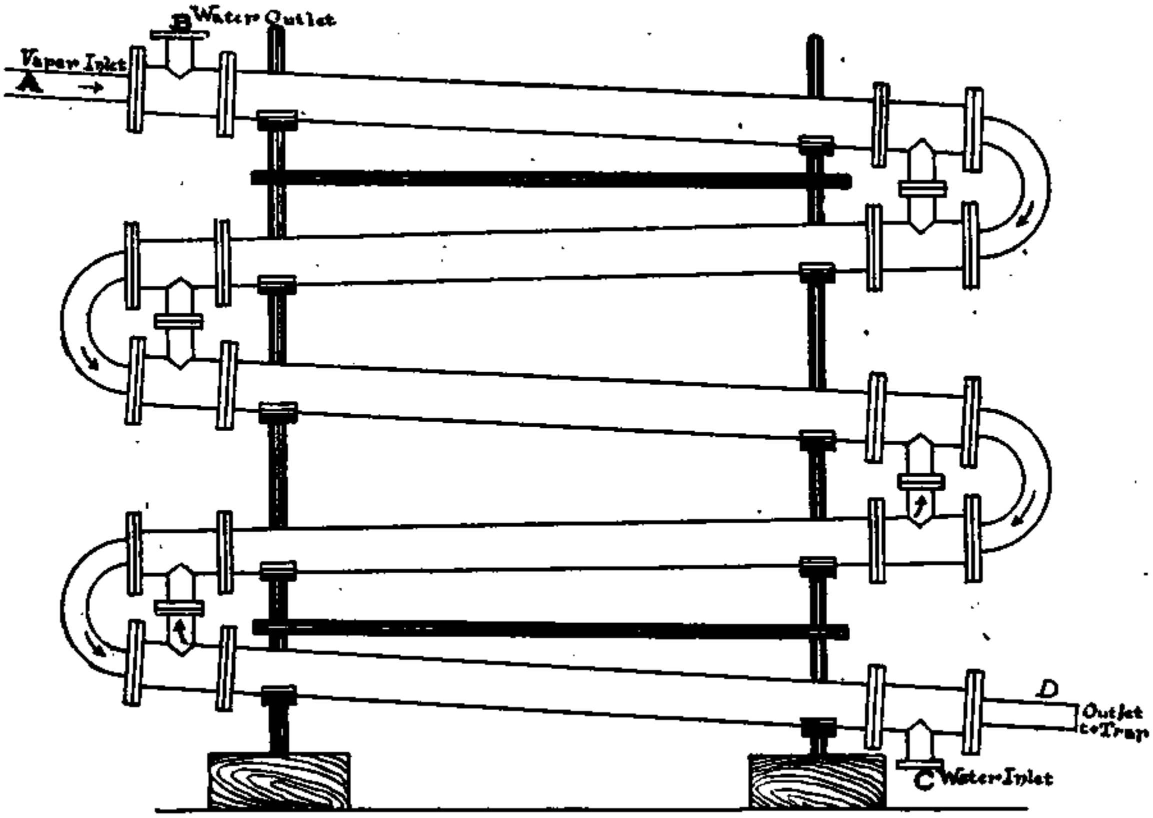


FIG. 14—DOUBLE PIPE COUNTER CURRENT CONDENSER.

readily taken off and the pipes cleaned. Where acid vapors are condensed the inner pipe should be of copper, but the outer pipe containing the water is usually of iron. Such a cooler is shown in Fig. 14 and the connection in Fig. 15.

Tubular Condensers.—It has been noticed that small tubes are more efficient as cooling agents than large ones in proportion to their carrying capacity. This is due to the fact that the cooling surface is actually greater, and also the rapidity with which the vapors can be brought into contact with the cool surface. A tubular condenser is more complicated, has more chances to leak and has the further disadvantage that as usually constructed a large part of the cooling surface is not brought in contact with the cooling water.

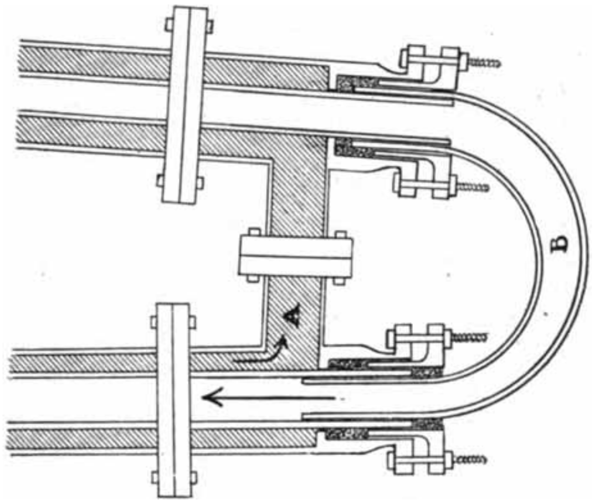


FIG 15.
A—Water.
B—Condenser pipe.

This latter condition is purposely made so that the tops can be easily removed, but it is easy enough to cover them with water if need be.

A tubular condenser as used in a steam process plant is shown in Fig. 16. This form has been in use in the hardwood distillation for some time. Other forms are used, modified only to enable them to be cleaned out better. Interested parties should obtain the views of the makers.

In making this form of condenser the outer shell should be made of tank steel and fitted with suitable inlet and outlet pipes. The inner part should be of copper and should be well braced in the tank and rest upon a small support in order to allow the water to get under it and cool the bottom chamber. The top is in the form of a dome and is fitted with a head that can be removed and tightened on by means of the yoke and wheel shown. In larger sizes wing nuts could be placed around the circumference and thus draw the edges together. The tubes should be well beaded into a bronze or brass plate on both ends, these ends forming the top and bottom respectively of the lower and upper chambers. The upper chamber or dome should be fitted with a large opening for the inlet of the vapors and the bottom chamber fitted with an outlet for the condensed vapors and a hand hole (not shown) for cleaning out purposes. These connections should pass through suitable stuffing boxes to prevent the water from coming out of the tank. A gas separator should follow the condenser. The upper dome should be of sufficient size to distribute the vapors as they come in without causing back pressure and the tubes should be of sufficient size and number for the same reason. In fact, it would be better to have them of greater capacity so that when the vapors enter if they should be under pressure they would naturally have a tendency to expand and thus reduce the pressure and help relieve the retort. Other forms of condensers could be used and different positions might be given those described, a horizontal tubular condenser being better for condensing engines, but usually to cool the vapors from the wood it is better to have them enter at the top and discharge at the bottom as is the case with vertical condensers.

In regard to the size of the various condensers as a general rule 150 square feet of cooling surface is sufficient for each cord with water at the usual average temperature found in the South and the present rate of distilling.

The following points are to be considered in estimating the size: 1st, the volume of vapors

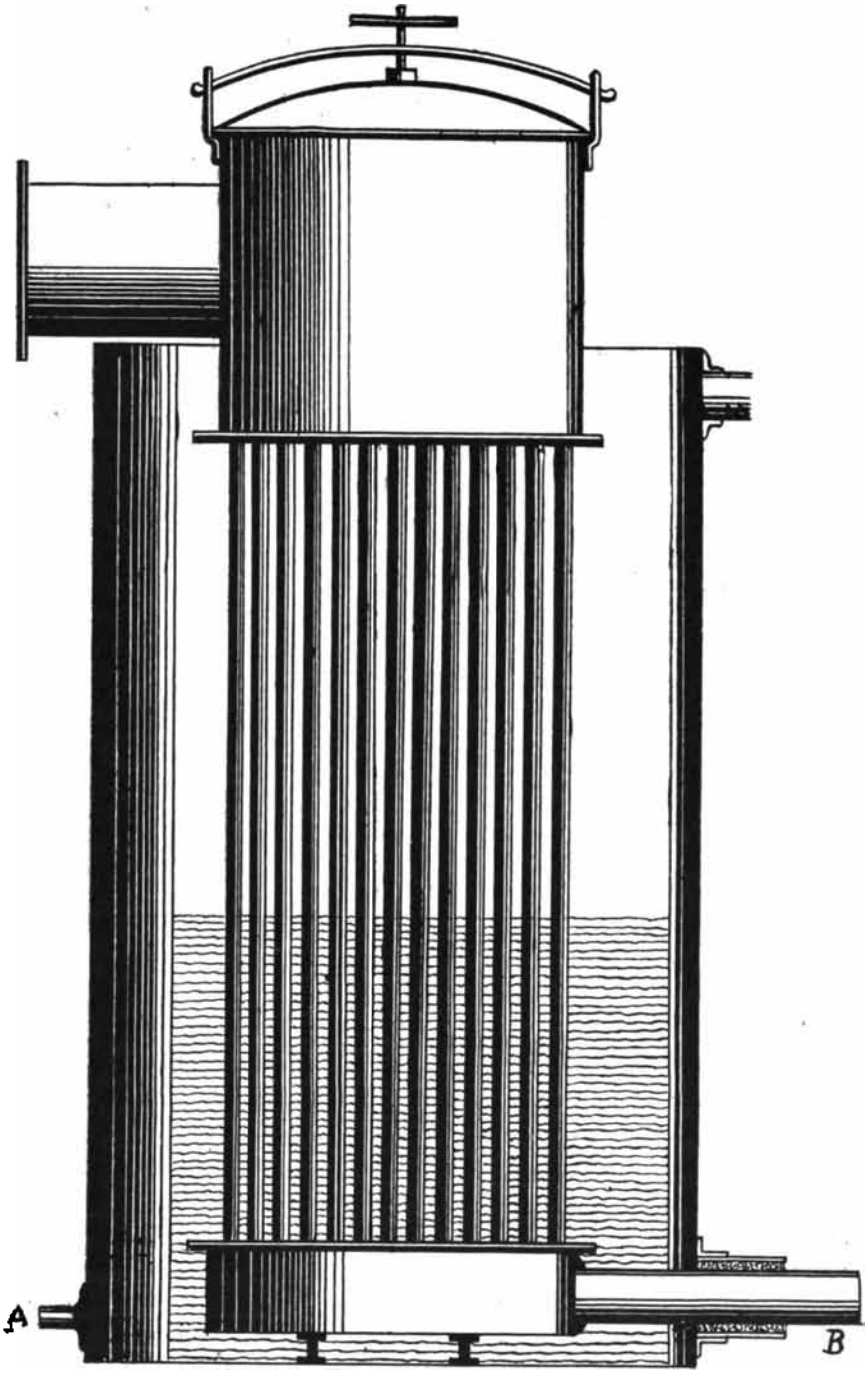


FIG 16—TUBULAR CONDENSER.

A—Water inlet.
B—To gas trap.

to be condensed in a given time. 2nd, the rate of speed at which they are flowing. 3rd, the temperature of the cooling water and the kind of material of which the cooling surface is made. The volume of the vapors can be determined by multiplying the number of cubic feet of products expected in a given time by 1700. If 30 cubic feet of material were distilled in 24 hours then 1 1-4 cubic feet would be the average distilled in one hour; this multiplied by 1700 equals 212,500 cubic feet of gases or vapors. In addition to this there would be not over 20,000 cubic feet of gas at 212° Fah., if the destructive process is used. With this latter process the temperature reaches 900°, consequently the gas which is produced at that temperature is equal to over twice the volume at 212°, so the entire volume to be disposed of would be at least 232,000 multiplied by two, or 930,000 feet.

To cool this product no general rule can be made that would be more than a rough approximate. With the steam process it is simply a matter of condensing so much steam at a given temperature and pressure. The calculation is based on a mathematical basis and is estimated by the number of heat units absorbed by copper surfaces under given conditions. The temperature, specific heat, specific gravity, volume, etc., of the vapors must be known, the temperature of the cooling water at its entrance and exit are also necessary factors. I might be considered that it would follow the same rules of cooling water with water, but such is not the case.

Kent gives it that "whilst 400 to 600 units of heat are transmitted from water to water through iron plates per degree of difference of temperature per hour, air or other dry gas transmits only about 2 to 5 units according as the surrounding air is at rest or in movement. In a locomotive boiler where radiant heat was brought into play 17 units of heat were transmitted through the plates of the fire box per degree of difference of temperature per square foot per hour.

A more detailed discussion of the heating and cooling of vapors and liquids will be given under the description of the heating of steam stills and the cooling of vapors.

The rate of speed of the vapors in reality regulates the volume of the vapors impinging upon a given surface per hour, so might mean the same as the first item mentioned. However, as it is necessary for the vapors to touch the surface before being condensed it can be conceived how in a large cooling pipe a large amount of vapor could be in the middle of the pipe without being cooled and any short sudden pressure would force this out uncondensed. For this reason it might be advisable to make them just a little longer than otherwise.

The temperature and amount of cooling water used also affects the size of the condenser needed. Where the water is cold the condenser would be smaller, and where plenty of water is to be had cheaply it could be used to help a small condenser by rapid circulation. Where there is such a vast difference between the vapors and the cooling water, water at 212° acts as a cooling agent at the top of the condenser. It is better to have the condenser of such a length that at the required rate of distillation the water will overflow at about 200° Fah. It is cheaper to have a large condenser than to have to pump water.

The thickness and nature of the metal influences the efficiency of the condenser. M. Peclet found that with perfectly clean metal the quantity of heat transmitted is inversely proportional to the thickness. Many say that it makes no difference what the thickness is as far as the transmitting of heat is concerned, but that it is only necessary to make the coils or tubes thick enough for strength and rigidity. Copper pipe 1-16 inch thick is much used for the purpose in pipes and tubes, but for the large condensers greater thickness is required and for tube plates 3-8 inch or more.

In tubular condensers the tubes are about 1½ inches to 2 inches in diameter, and 6 to 10 feet in length, and from 3-32 to 5-32 thick. In arranging them in the plates it is necessary to have them far enough apart so as not to weaken the plate itself and also to facilitate construction.

Receivers and Storage Tanks.—In the destructive distillation and in the other processes for obtaining turpentine and tar from wood some method must be used to separate the oily matter from the watery portion. The condensers discharge their products either directly into a tank of some description, or into a pipe leading to such a receptacle. When the first distillate from pine wood comes over the product consists of water and oil when steam is used and slightly acid water and oil when destructively distilled. Upon standing this mass separates in two layers, the oily matter on top and the water below. All that is necessary to separate them is to draw off the water from below or to let the oil overflow. The same rule applies to the tarry products also, only we find the acid water often on top. This liquor usually has to stand a long time before it can be completely separated so it is desirable to have several tanks for receiving, allowing the others to settle while one is filling up.

In the South wooden tanks are not of much service for collecting turpentine in any form. As the oil from the retort contains water it would soften any glue that might be used to keep them from leaking. An iron or earthenware tank is the only suitable receptacle for the crude turpentine.

For tar and acid iron is not good and wood will leak, but as the acid products are of comparatively little value wooden vessels can be used, provided they are furnished with suitable bolts for tightening the bands on the tank. For discharge cocks large wooden beer taps are very suitable. Sometimes large pits are made in the ground and the sides boarded up and pitched. These are all called settling vats or pits.

All that is necessary in these receptacles is to have suitable pipes and valves to draw off the separated liquors or to pump or blow them out into the refining apparatus.

It is advisable to have collecting tanks for each crude product for at least a week's run. This is especially true with regard to the tarry liquors, as the longer they are allowed to settle the better the separation. In this case seven tanks sufficient to hold a day's run should be used, or better, seven tanks each twice the capacity of the tar still would be suitable. By this means the liquor in the first tank would have settled for seven distilling periods and the water and acid (both together equal to about one-half) could be drawn off and the remainder put into the still.

CHAPTER V.

REFINING METHODS.

The crude materials or distillates having been collected in the receptacles just described, it is necessary to treat them in some manner in order to make them merchantable.

The crude turpentine consists largely of turpentine oil, resin oil, resin and more or less of the gummy and extractive matters to be found in wood. As generally produced, one or two distillations will sufficiently refine the turpentine and leave it white and pleasant smelling. However, in some cases it is better to treat with some kind of alkali, which will remove the gums before distilling. Some operators put caustic soda solution directly into the still, but this is not to be recommended, as a resinate of the alkali is found when resin oil is present, and the steam used in refining will decompose it toward the latter stages of the distillation and distill it over into the refined oil, and thus make it gummy and slow drying. If the distillation is stopped at this point all the turpentine is not distilled and the yield is less. Lime, when used, should be put into the still, as it forms a compound not easily separated from the oil. Although lime will take out resin and is cheap, it is not very suitable as a refining agent. It takes a long time to act and when put in the still direct it causes a coating on the walls and steam pipes, which is not only difficult to remove, but makes it necessary

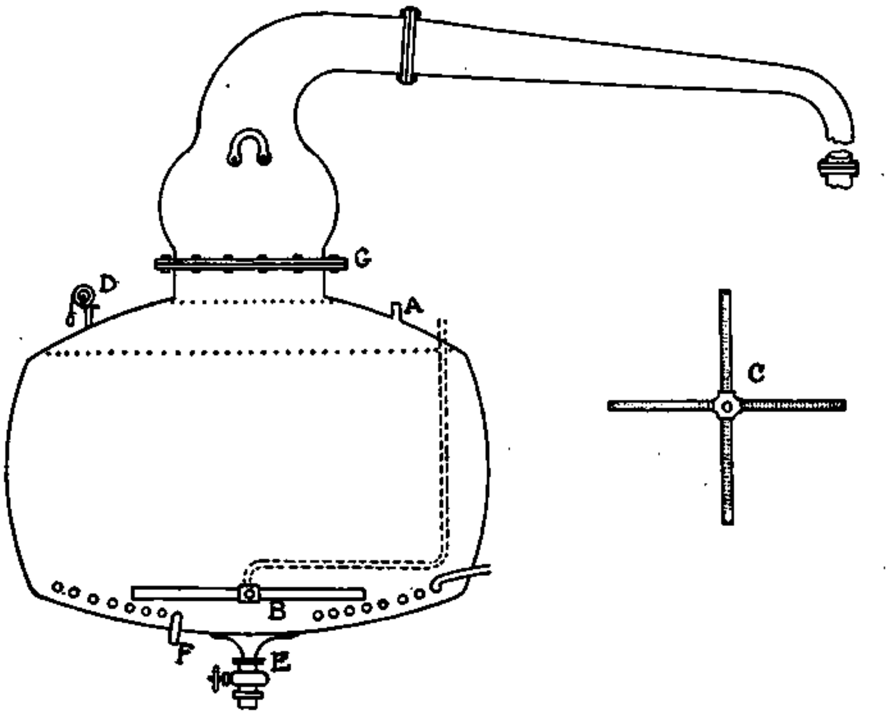


FIG. 17—750 GALLON STEAM HEATED TURPENTINE REFINING STILL.

A—Charging bung. B—Steam jet.
C—Detail of jet. D—Vacuum valve.
E—Discharge valve. F—Exhaust from coil.
G—Boiler connection between head and still.

to use more steam in the closed coil, as the heat does not penetrate well through the coating. Caustic soda seems to be the best chemical for purifying. A weak solution answers the purpose and should be thoroughly mixed with the crude oil. This is best done by stirring with cold compressed air or by means of any mechanical stirrer used in the arts. This mixture should be allowed to settle and the lye drawn off at the bottom as closely as possible, then water added and the lye washed out by mixing and settling with the water. The residual oil should then be pumped into a copper or iron still and distilled by means of steam. Such a steam still is shown in Fig. 17. This still is so arranged that the crude oil can enter at A. A steam scroll composed of a closed copper pipe is shown on the bottom; close to it at B is a perforated cross made of copper pipe and shown in detail at C. This allows sufficient live steam to enter to thoroughly stir the contents of the still. Water is admitted through A at the same opening as the crude oil. A vacuum valve is placed at D to prevent any collapse by a sudden cooling. At the bottom are two outlets, the one at E for the residue of the distillation to be discharged, and the one at F to allow the condensed steam to escape by connection to a steam trap, or otherwise. To make the cap or head tight it is bolted onto the still as shown at G. This is connected with an ordinary worm condenser or tubular condenser, or the other forms mentioned. The still is an ordinary 750-gallon steam still.

In designing a still it can be readily seen that the greater the diameter the more evaporating surface, consequently we find that some makers of stills make them of very large diameter and not very high. On the other hand, with steam stills some prefer to have the body of the still of the same height as the diameter. The thickness varies with the maker; the McMillan Bros. Co., of Mobile, Ala., make the top of a 2,000-gallon still of No. 12 Stubb's gauge copper, the bottom of No. 10 and the sides of No. 13. A still of this size furnished with 100 lineal feet of 2-inch tubing, No. 13 gauge, with the brass cross shown in Fig. 17, and all the necessary connections will be supplied for $800 to $900, according to the number of attachments and extra work.

When heating stills by steam coils to get the best effect, the coil must be drained. This is done by means of a steam trap, an apparatus which holds the steam back and at the same time allows the water to escape. As condensed water is forming along the entire length of the coil the diameter of the coil should be rather large, so as to be able to carry the water to the steam trap without letting the water fill up part of the coil, thus preventing the full action of the steam. Be sure to have a steam trap large enough to carry off the water. In working with a steam trap the water condensed in the coil under pressure when it escapes, will boil and cause steam when exposed to the atmospheric pressure, thus causing one to think that the trap is leaking steam. The only thing necessary with a good steam trap is to have it large enough.

The size of the coil and trap can be determined beforehand by considering the contents of the still to be nothing but water. This we can do because the temperature of a mixture of water and turpentine, such as is usually distilled, does not have a higher boiling point than does water. Also, the specific heat of turpentine of a density of .872 sp. gr. is .472, water being 1, so being less than water, one would be on the safe side to consider it as water.

We will not go into the scientific details of the causes and principles of heating and cooling, but confine ourselves to a brief consideration of the subject. The effectiveness of heating liquids by means of steam in a closed coil depends upon many conditions. The coil should be drained as well as possible so as to get the full effect of the steam at the temperature and pressure used. The circulation of the liquid, the thickness of the coil and the presence or absence of a deposit on the coil affect the efficiency. The difference in temperature between steam in the coil and the liquid to be heated also affects the time of transmission to a large extent, the greater the difference the more heat units per degree difference of temperature being transmitted.

The element of time enters into the consideration of the subject, for no matter how hot the steam may be it will take some time to heat the liquid. The time required depends upon the supply of steam and the rate of transmission is usually expressed by the number of British thermal units (B. T. U.) transmitted from the hot side to the cool side per degree Fahrenheit of temperature difference per square foot per hour. A B. T. unit is the heat required to raise one pound of water 1 degree Fah., the standard being the degree from 39.1 degree Fah. to 40.1 degrees Fah.

Experimental work indicates that copper coils are more effective than those of other metals in common use, and for these certain experimenters found 250 B. T. units were transmitted per degree Fah. difference in temperature per square foot per hour when heating cold water, and M. Peclet found that to evaporate at 212 degrees Fah. as many as 935 B. T. units were transmitted per degree of difference, due probably to the more rapid circulation of the water. Other experimenters find different amounts, one testing steam feed water heaters observing that 368 B. T. U. were transmitted up to 212 degrees Fah., and 660 B. T. U. at 212 degrees Fah., probably with a steel coil, although not stated. Our problem in a still is confined to heating water (water and oil) by means of steam, hot gases acting differently. If we take a high rate of transmission our coils would be made small and steam trap large, and when we came to condensers the worm or tubes would be small and our water supply large. If for any reason the coil did not work perfectly there would be a bad condition of affairs, as one could not change the coils readily. By taking a low rate of transmission and one was disappointed in the results of transmission and it was higher, then all that would be necessary to do would be to increase the size of the steam trap and to increase the supply of cooling water to the condensers, something which can be readily done.

The above experiments on the transmission of heat were made with clean coils. Those who are obliged to use foul coils, as in the case when distilling resinous oils, find that about one-third of the rate of transmission given above is the proper working rate. In evaporating sugar solutions the water from which would be more difficult to distill than in the case under consideration, from 265 to 376 B. T. U. are transmitted.

One would be safe in averaging 300 B. T. units per degree Fah. difference in temperature per square foot per hour, both for heating the liquor to the boiling point and distilling. In addition to heating the water to be distilled the cooling effect of the air on the surface of the still itself must be overcome by the heat of the coil in order to force the vapors high enough in the pipe to where it bends to the condenser. The influence of the air is equal to about 1.7327 B. T. units per degree of difference in temperature (Fah.) between the contents of the still and the outside air, (the extremes being taken for each square foot surface per hour when the still is of copper, and about 1.438 B. T. units for iron). This includes all the surface of the still and vapor pipe to the point at which the pipe turns to the condenser.

On an average one gallon of water per square foot per hour should be evaporated by an iron coil and about three gallons by a copper coil, and often more.

The size of the steam trap is calculated in a reverse manner, the cooling effect of the water evaporated causes a certain amount of steam to be condensed, this being based on the B. T. units. The steam trap should be more than large enough to carry away this water.

A concrete example of this calculation can be made with the 750-gallon still illustrated. Suppose one wishes to distill 500 gallons in ten hours, what would be the size of the coil and steam trap, the steam being supplied at 300 degrees Fah. temperature and 53 pounds pressure? Take the weight of a gallon of water at 8.34@62 degrees, or better, perhaps, 8 1-3 pounds. We then have about 4,167 pounds of water to distill. If it takes 1 B. T. U. to raise one pound of water 1 degree Fah., it would take 4,167 B. T. units to raise the 4,167 pounds 1 degree. If the water was at 60 degrees to bring it to 212 degrees Fah., or the boiling point, there would be 152 degrees to raise the 4,167 pounds of water with 4,167 B. T. units for each degree. This would equal 4167x152, or 633,384 B. T. units expended to bring the water to the boiling point. Considering no pressure (although there may be a little), it is necessary to heat further only sufficiently to evaporate the water. When we evaporate water the latent heat must be considered and instead of 1 B. T. U. for each pound to raise it one degree it will be found that each pound of water absorbs about 966 B. T. units and the temperature doesn't change. Then to evaporate the water we must supply in addition to the heat necessary to bring it to the boiling point 4167x966, or 4,025,322 B. T. units, which, together with the 633,384 used in heating the water makes a total of 4,658,706 B. T. units to be supplied in ten hours, or 465,871 B. T. units per hour. (Plus allowance for air cooling.)

The temperature in the coil is 300 degrees Fah. and the water 60 degrees, the average water 136 degrees, a difference of 164 degrees. For each degree, according to previous decision, 300 B. T. units are transmitted per square foot per hour, or 164x300, or 49,200 B. T. units per square foot per hour under the given conditions. The total to be supplied in one hour, not counting the cooling effect of the air, is 465,871 B. T. units, so 465,871 divided by 49,200 equals about 9.48 or 9½ feet, nearly. To find the size of steam trap it will be found that only the latent heat of the steam is used in heating with a coil using a trap. The latent heat of steam at 53 pounds or 300 degrees Fah. is about 904 B. T. units, and to lose 465,871 B. T. units in one hour it would take 465,871÷904, or about 515 pounds, of condensed steam per hour. The weight of a gallon of water at that temperature is about 7.7 pounds, so the steam trap should have a carrying capacity of about 67 gallons per hour. It will be noticed that this amount is almost seven times the number of square feet in the coil. The heat in the condensed water passing through the steam trap should be utilized, but it never is. To use this heat in the still the coil would have to be large enough to condense all the steam to 212 degrees Fah., and not use the steam trap, a practice that would not succeed well. It is better to make the trap a size larger. An important point to consider is the size of the steam pipe. This should be large enough to supply sufficient steam to heat the coil sufficiently. There is no use in having plenty of heating surface and not enough steam.

Further discussion of this matter can not be given here. It is a question of steam practice familiar to engineers. Other vapors than steam act differently, generally the rate of transmission being much less. Fire gases act slowly, but their action is also quite well known.

Tar Stills.—At this place will be considered only the stills necessary for removing the light oils present in the tar without distilling the tar itself.

The crude liquors containing acids and tannin matters are very injurious to iron, so copper ought to be used. Some remove the organic compounds present, which become dark when acted upon by iron, by treatment before distilling.

Another distinction is made as to whether they are to be heated by direct fire or not. In the former case they are made of two shapes, one like a coal tar or paraffine still, with a diameter about as great as the height and the bottom concave extending toward the middle about one-sixth to one-third of its height, so that the fire will have more surface; a man-hole is usually provided at the top and it is also supplied with suitable mountings for steam pipe and thermometer. The other form is similar to a horizontal boiler. In both forms the vapor is carried off at the top similar to the vapors from a turpentine still and condensed in a similar manner.

The steam still is the better as it provides a surer way of controlling the temperature and prevents the tar from being burnt. This should be made in all particulars like the turpentine still already described, and if made of copper will prove to be very efficient. The size, of course, must be proportioned to the product, as there is much more tar formed than turpentine. The heating coil is only used for heating the water and driving off the oil, the tar not being distilled. These stills should be placed in such a position that the tar can be readily drawn off into a tank. A continuous still for removing the oils from the tar has not been used, but a simple one could be very easily devised for use at a plant where there was sufficient crude material produced.

Wood Oil Still.—The oil from the tar still can be redistilled in a regular turpentine still. By making it of iron with a copper head and worm this oil can be treated with caustic soda or other chemical, the caustic solution drawn off and the oil redistilled, making it of a very bright color. When all the turpentine has not already been removed from the tar products the first distillate of the redistillation of this oil will be practically white. Real wood oil should not contain turp, but should be a mixture of ordinary wood, red oil and various distillates of rosin or resin found in the wood.

Still Heads.—Several attempts have been made to rectify spirits of turpentine produced from wood. As ordinarily produced wood turpentine contains some light oils that it is desirable to remove to prevent its drying too rapidly; also a heavy oil is to be found which is often carried over with the steam when the distillation is too rapidly conducted. To insure steady working of the stills several devices are in use. Two of these are shown in Fig. 18. In A, instead of a still head like the one shown in Fig. 17, a small pipe is carried up to a considerable height before turning to the condenser. This pipe offers considerable cooling surface to the air, consequently quite a little fractionating can be done with it. The lighter vapors can be condensed first; then the turpentine. In B the long pipe is branched and pipes placed on each, the lighter vapors passing through the upper one, and when they are all distilled the valve is closed

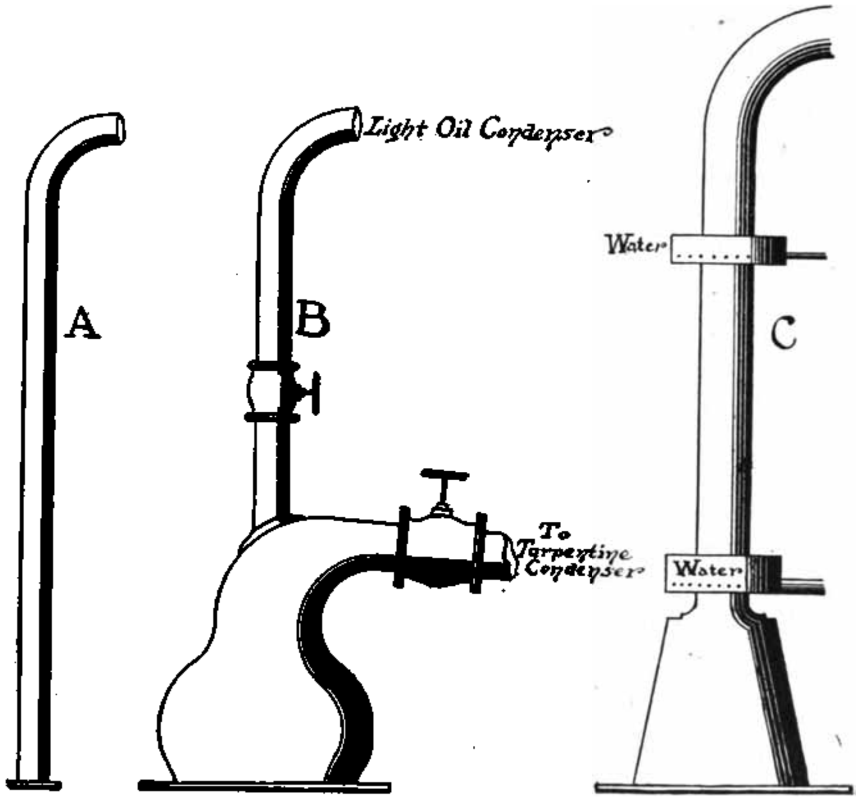


FIG. 18—STILL HEADS.

and the vapors pass through the branch to the other condenser. At C is practically the same thing as at B, except water is placed around the pipe to insure its being sufficiently cooled. These methods are all crude.

In Fig. 19 is a more elaborate head designed to be placed on a retort condenser; the proper place for it, however, would be on a still.

It consists of a tank A filled with water by means of pipe K overflowing through pipe R. The water is allowed to enter between the plates C. B. on both sides, H being merely a support. Each section is cone-shaped and T is a band running around the sides of each section so as to enclose the top and bottom of the vessel and forms a chamber for the vapors, thus enabling them to come in contact with the cool walls, B and C. Between the walls B and C is a plate D, held at one side by the support shown, and this prevents the vapors from rushing directly out through P, thus acting as a baffle plate to the vapors.

The operation is as follows: The vapors enter

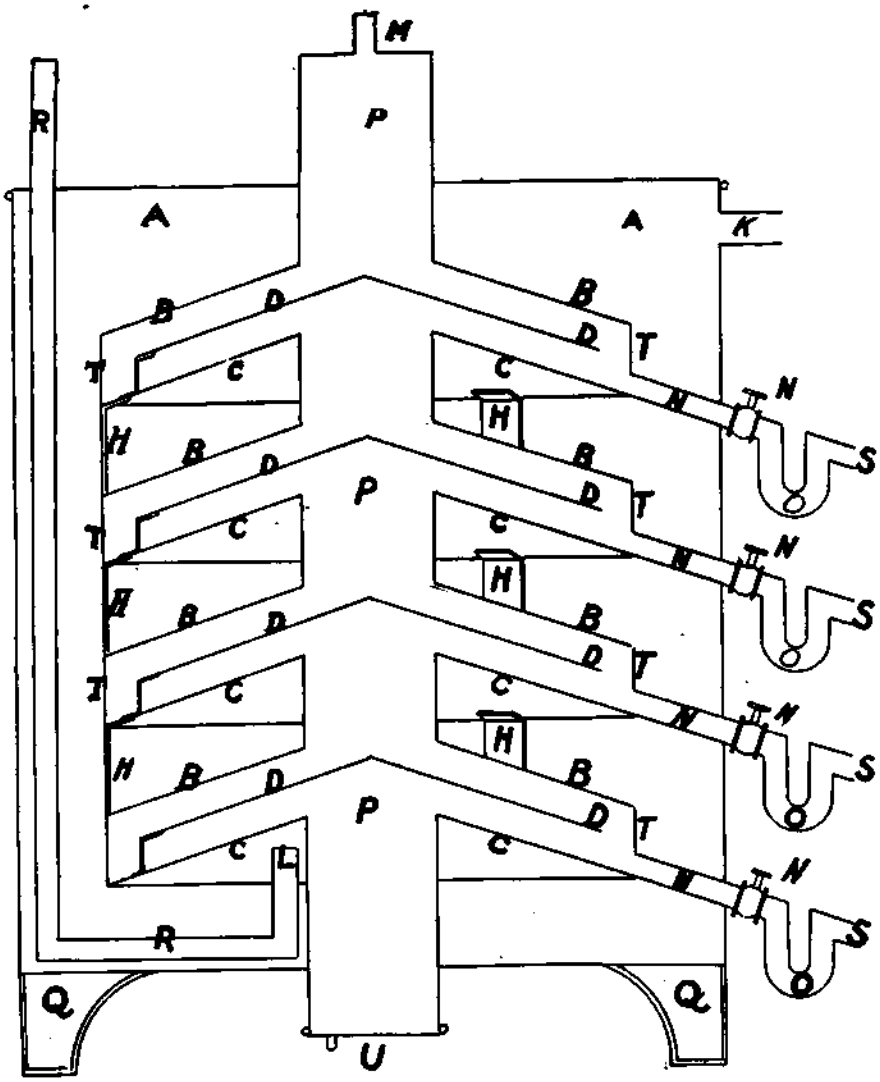


FIG. 19—HEGE'S PATENT HEAD.

at U, pass up P, strike the baffle D and roll back under the edges of D, which are open, and then continue above D until they hit the next P, and so on. C being on an incline any vapors condensed on surfaces C, D and B, cannot fall back through pipe P, into the still, but follow down N. As originally designed no valve was shown at N, but there should be one there and it should remain closed until that portion of the apparatus reaches the proper temperature. In this way any light oils condensed when still is first started instead of running out at S in first section to tank intended for heavy oils, the increasing heat would again vaporize this condensed matter and eventually put it into the section wherein it belongs.

To determine the proper temperatures a thermometer should be placed before the valve N in each section. Any vapors that do not condense are carried away at M. This apparatus has one very bad feature in that the cold water comes in at the top and the hot water is forced out at the bottom. It might be better to invert the condenser and have U connected with the still just the same and have a ∩ on the connecting pipe.

A form similar to the above described condenser is used, which permits the condensed vapors to fall back into the still. No water is used and temperature regulated so that only the light oils pass through the head and are condensed. After the light oils pass over a valve leading to this still head is closed, another valve opened leading to a worm condenser and the distillation carried on in the usual manner.

If, by the use of a still head, the bad odor in the turpentine when it is mixed with tar can be removed, as it is done to a large extent by one company, then the regular hardwood method of distillation is the best way of handling the distillation of wood. However, even the best refiners using that process leave a trace of disagreeable odor and a light cast to the oil.

Alcohol Stills and Acetate Pans.—The wood alcohol from pine wood is seldom recovered, as the yield is too small. The yield of acetic acid in the condensed liquor is also much less than with hardwood. In Germany alcohol is made. Generally three stills are required to make the refined alcohol. The first one separates the alcohol and the wood vinegar, the second for the treatment with lime, and the third for rectifying. As carried on in the wood alcohol plants in the North this is as far as the distillation goes, the further rectification being done at refining plants in special column stills.

The acetate pans are usually flat sheet iron pans, like salt pans, and heated with the waste gases and by steam. The mixture is stirred to keep from burning the acetate and thus decomposing it into the carbonate. A more detailed description of this process will be given in describing a German process.

Condensers.—Under this head there is nothing different to describe than from those connected to the retort. In refining it is not so necessary to make arrangements for cleaning as the oil has a tendency to act as a cleansing agent itself, and as no tar should be allowed in the condenser, it is an easy matter to keep it clean.

But the sizes of condensers has been left for consideration at this place. With surface condensers on condensing engines one square foot of heating surface is allowed for every 10.6 lbs. of steam condensed. This leaves the water at about 120° Fah., the temperature of the hot well. In distilling turpentine it is necessary to bring the condensed material to as low a temperature as possible, in the summer time to at least 100° Fah. Then in the case of the engine condenser the exhausts enter under some pressure, whereas in distilling very little pressure is developed. Again, it is known that hot gases, such as come from a retort, do not transmit heat to water as readily as does steam, so a suitable formula must be found to suit the case. In the condenser the cooling surface should be as great, or greater, than the surface of the coil in the still and should be able to condense all the vapors that the still distills. No allowance can be made for the coating on the coil in the still, as a coating also forms in time on the condenser surface.

It can be considered then that the copper surface of the condenser can transmit the same number of B. T. units as the coil in the still per square foot. This was taken at 300 for each degree Fah. difference in temperature per hour.

Continuing the example given under stills, how many square feet of condenser surface will be necessary to condense 500 gallons of water in ten hours with the cooling water at 60°? The difference in temperature between the vapors and the mean of the water is 212-130 or 82 and the number of units transmitted per degree 300, and for 82 degrees 24,600 per square foot per hour. The number of pounds of vapor in ten hours was 4,167. To cool it the latent heat would be 966 B. T. units, and the cooling from 212 to 60° equals 152 B. T. units, or a total of 1,118 B. T. units per lb. and for the whole amount 4,167x1,118=4,658,706 B. T. U. to be cooled in ten hours or 465,871 B. T. U. in one hour, as we found under the first case with stills. We have then 465,871 B. T. U. per hour and an absorption of 24,600 per square foot per hour by the cooling surface so to absorb it all it would take 465,871 divided by 24,600, or about 19 square feet. This is about twice the heating surface of the coil in the still, but this ratio varies with the initial pressure and temperature of the stéam, the higher the temperature the greater the ratio between the heating surface and the cooling surface.

The whole problem shows that it depends upon the amount of heat transmitted by the copper in a given time. In a water heater this rate was only 68 B. T. U., using steel tubes, and in condensing benzol vapors only 10 B. T. U. were transmitted per hour per degree difference in temperature for each square foot of cooling surface. With a tar still giving final vapors at 637° Fah. only 812 B. T. U. per hour per square foot of surface were transmitted not for each degree, but altogether.

In a retort condenser where there is such a large amount of gases other than steam an allowance should be made of a total transmission of only 1,000 B. T. U. per square foot per hour.

Condensing Water.—Knowing the square feet of condenser surface required, multiply by the number of heat units transmitted per square foot per hour and this will be the total number of heat units to be carried away by the water. The number of heat units has been calculated all along on the mean temperature of the water and as the experiments on transmission were made that way it is advisable to use the mean temperature of the water in calculating the results on the condensers. With the cooling water it is different. If the water comes in at 60° and goes out at 200° it is evident that 140 B. T. U. are carried out with each pound of water, irrespective of the time it takes to get hot. The total heat represented in B. T. units per hour divided by this difference in temperature equals the number of pounds of cooling water required per hour. In the above case there were 465,871÷140, or 3,328 lbs. per hour, and at 8 1-3 lbs. per gallon equals about 399 gallons per hour.

Storage Tanks.—By making the first receivers of condensed retort vapors of considerable size they help out the storage capacity of a plant. These receiving tanks have been spoken of before. In addition to these tanks others are needed for the refined product. In some plants arrangements are made to have the condensers on one floor of a building and the storage tanks beneath so that no pumping is necessary. A pump, though, particularly if brass-lined, is usually cheaper than the additional floor. A common iron tank is not suitable for turpentine, but a good galvanized tank or an enameled tank are very suitable and hold well. An enamel made of white lead is sometimes used with success, but the best is a glass enamel baked on.

Galvanized tanks are made upright with a convex bottom—they are to be preferred. Glass enamel steel tanks are made in horizontal and vertical positions, the former are better to save height, and the latter easier to measure. They are usually made in sections composed of flanged rings and these flanged rings bolted together form the tank. Tanks for tar can be made of ordinary iron. The first tar tank is one of about the capacity or twice the capacity of the tar still. This is used for receiving and cooling the hot tar from the still. When cool the tar can be pumped to a large storage tank. This large storage tank should be set on piers high enough to permit barreling of the tar. There should be plenty of storage capacity, as the product is likely to be slow moving.

Shipping and Packages.—For turpentine eight-hooped white oak barrels, holding 52 gallons, are of the right kind. Some use six hooped oil barrels, but they are more difficult to keep tight. The hoops should be well driven and the barrels glued on the inside. This is done by putting about four or five gallons of hot glue in the bung hole, inserting a plug and rolling the barrel so that the glue will touch all the inner surfaces. The plug is then taken out and the barrel set on a run to drain. Sometimes it is necessary to glue two or three times, using about a pound or so of glue per barrel. If no water is put in with the turp the barrel will remain tight for some time if not exposed to the sun.

A tank car is better for shipping purposes, as it will not usually leak, but it is not always as clean. By coating the cleaned surface with shellac it keeps the oil in good condition. For tar second hand oil barrels are much used. These should be thoroughly repaired and coopered and preferably soaked before putting in the tar. A tar barrel must be kept out of the sun or it will surely leak in summer weather. A picture is here shown (Fig. 20) of a steam and destructive distillation plant, showing tar barrels ready for shipment and also tank car. Tar could be shipped in tank cars to great advantage. Freight on wood distillates is extremely high in proportion to their value to the regular naval stores.

FIG. 20—SHIPPING TURPENTINE AND TAR AT A STEAM AND DESTRUCTIVE DISTILLATION PLANT.

CHAPTER VI.

SPECIAL COMBINATIONS OF APPARATUS AS USED IN MODERN PLANTS.

The different pieces of apparatus just described are used in various combinations according to the process used in distilling.

These processes may be divided under several heads, but the best division seems to be into Steam Processes, Steam and Destructive Distillation and Destructive Distillation. A fourth division of Special Processes might be used to designate those processes used in destructively distilling saw dust and those using rotating retorts, also those retorts which are to be moved from place to place and those comprised of conveying machinery suitably enclosed.

No attempt will be made to assign any relative value to the patents. It has been the custom for one man to advance an idea and for another to patent it, so the patent itself will be discussed only.

In considering these patents, it is advisable to understand something of the composition of pine wood and the products of distillation. These will be found in detail in a special chapter under that head. Briefly stated, however, the main object of the steam process is to extract the turpentine, while the object of the other processes is to obtain all the products possible so that the wood may be completely utilized.

Fat wood contains the woody fibre and resins with other substances. The resin content is more stable at a high temperature than the woody fibre, so it is necessary to consider only the temperature that first attacks the wood itself. Cellulose is affected at 320° Fah., but the temperature could be raised slightly above that without doing any serious harm to the products of decomposition and distillation. To remove the resin and leave the woody fibre intact, three or four methods could be employed. The rosin could be dissolved out by suitable solvents and the solvent evaporated. This might possibly be a good way now that denatured alcohol is promised to be cheap, but requires considerable capital locked up in the solvent. Another way would be to melt it out by dry heat, another by steam heat, another by heating and squeezing. Extraction and steaming would probably be the methods that produce the greater yields, as a residue would be left in the other cases that could not be removed by the method employed.

In the steam process for removing the oils, the first plants worked with large pieces of wood which were afterwards destructively distilled. The steaming process thus employed was re-discovered (?) in this century and extraction by this means continued. However, a more important discovery which influenced the industry more than any process of extraction was a means of cheaply comminuting the wood. This machine is known as the "hog." The advent of this machine in the business has made possible the extraction of turpentine from chipped wood in only a small portion of the time previously required.

At first high pressure with steam was used, but the tendency has been to gradually diminish the pressure until now ten or fifteen pounds is considered the proper amount. The author believes a vacuum is better, accompanied with sufficient steam heat to start the oils from the wood. Under atmospheric pressure the oil contained in the resin will distill in presence of steam at 212° Fah. It seems to be more a question of volume of steam rather than pressure. Sufficient volume of steam should be admitted to rapidly carry over the oil.

An experiment to determine the proper temperature of steam admitted for distillation when the operation is carried on at 40 lbs. pressure was made at the Massachusetts Institute of Technology by Messrs. Wiggins, Smith and Walker, with the result "that the optimum conditions for both turpentine and rosin are an initial temperature of 175° C, followed by steam superheated to 400°

C." By this method there is a larger amount of both resin and turpentine produced. "Below 170° C the oil comes over slowly and will ultimately cease to distill unless the temperature be raised. If the temperature is run above 200° the yield is not improved; the turpentine is discolored and has a burnt odor. The yield of resin appears to be decreased with the higher heat, due to the fact that it decomposes and distills over with the turpentine."

The above applies, of course, when working at 40 lbs. pressure. It shows, though, that a certain amount of heat is better than either a higher or lower temperature, and this amount of heat should be determined for different pressures. The loss of resin at high temperatures may be due to decomposition or to actual distillation. Resin under atmospheric pressure can be distilled with but little decomposition when in vacuo or by means of superheated steam. By using steam alone on the chipped wood turpentine generally distills and the resin remains in the retort except that which is carried over with the steam.

When the destructive process is used with or without steam a process should be selected that readily removes the turpentine from the wood without decomposing the fibre, or some process that has a special method of refining mixed distillates in a suitable manner. With those drawing out the turpentine first, the apparatus used should be one that easily removes the turpentine and then decomposes the remaining wood at the least expense for fuel and at the same time with the least damage to the retort. When the turpentine is removed the wood is of the same bulk and usually dry and in good condition for destructive distillation. As the distillation progresses the bulk of the residue becomes gradually smaller, so those processes that heat from the top only are at a disadvantage, for when the heat should be greatest the material distilling is falling away from the heat instead of towards it as it should. In most cases this disadvantage could be overcome by having flues with suitable dampers for directing the fire gases.

It takes more fuel where the retorts are protected, also where cars are used. More damage is caused without protection also with cars as the shells must be made hotter in the latter case. A small retort takes more labor than a large one, but has proportionately more heating surface. The use of steam is recommended with these processes under the same conditions as with chipped wood, only for a longer time.

Steam Processes.—Under this head will be comprised those processes using steam with or without pressure for extracting the turpentine. In this process nothing is obtained but the turpentine, although some utilization of the residue is attempted. The steam process has several distinct advantages over the others. Some of them are that the wood being in a fine state of division the process does not take but from one to six hours against 24 to 48 by the destructive method. The turpentine produced is of a more uniform quality, the apparatus is not destroyed and the residue left after distilling is sufficient for the fuel necessary to furnish the steam and cooling water. On the other hand, it has the distinct disadvantage in localities where charcoal is in demand of not being able to utilize a poor quality of wood except where such wood has but little value, such as sawdust. Where wood is relatively expensive the steam process will not draw out enough turpentine to pay for the wood itself. This must be taken into consideration in deciding about building a plant.

In reviewing some of the patents on the subject one finds that the use of steam, superheated and saturated, with and without pressure, has been patented since 1865 by Hall and Emery, and the same was probably in use before that time. Since then numerous patents have been obtained for practically the same principle, but with the retort slightly modified in some particular. As all these steam methods were followed with destructive distillation they will be found under that head. Of those processes which stopped the distillation when the turpentine was distilled, the one that first attracted much attention was Krug's patent, exploited by the Standard Turpentine Company, who located their first plant at Waycross, Ga. An ordinary hardwood distillation plant was erected with retorts having a bumped head to stand the pressure. Each retort was mounted with a steam gauge and two valves, one valve leading to a condenser for turpentine, and the other leading to a condenser for tar products. The turpentine valve was simply a relief valve opening at a definite pressure.

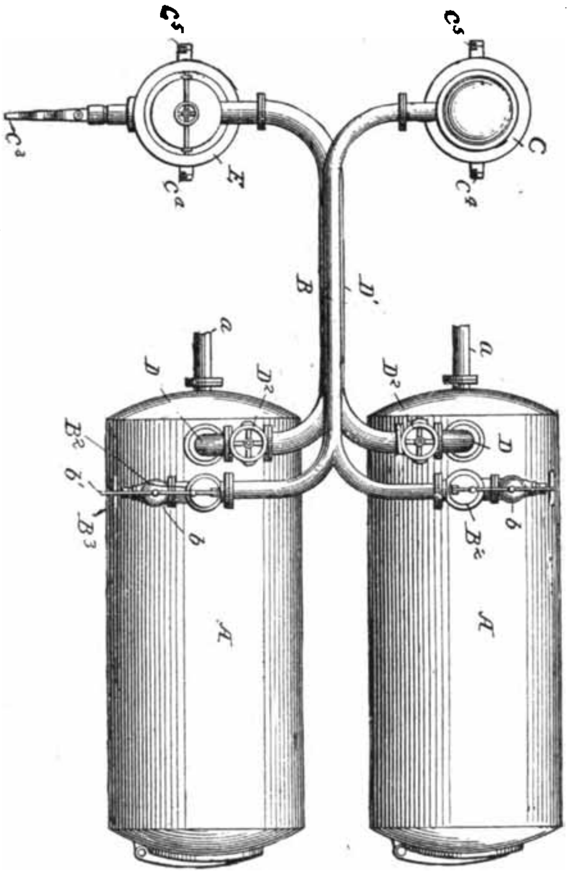


FIG. 21.—KRUG'S PATENT (PLAN).

The object of the process seemed to be to steam off the oil and then to destructively distill the residue if required. Upon trial the troubles of the earlier investigators were encountered, one great difficulty arose in the inability of steam even under pressure to remove all the oil from a block of wood in the time of distillation. To obviate this the wood was ground up in a hog and then distilled. Under these conditions much success was claimed in extracting the turpentine. Of course, owing to the fine conditions of the wood, destructive distillation was not to be thought of. Another trouble with the plant was the difficulty in charging and discharging the retort with the fine wood. The retorts being horizontally placed, it was necessary to throw the material in by hand. At another plant constructed by the main company the retorts were set vertically, thus enabling them to be charged by means of a conveyor. This process was interesting and great credit is due to the promoters. The idea has been copied in many forms, some using pressure and some not, the most improvement being made in the mechanical handling of the raw material. As to the validity of any of these patents there must be some doubt.

Fig. 21 shows the retort used in this process and the mountings. A is the retort with bumped heads; a is an inlet for steam which, however, is not usually used, the steam being taken in from the top at the front end and a perforated coil placed in the retort. The retort being set in a brick furnace external heat can be applied with the steam if necessary. The turpentine valve B3 is an ordinary valve which can be closed when necessary, while at B2 is the automatic valve to regulate the pressure. At D the tar vapors can arise and pass through D2 to the condenser E and discharge at C3. The valve D2 probably wouldn't work but once without choking with pitch or coke. The turpentine vapors after passing B2 pass to the turpentine condenser C where they would be condensed and discharged. Two retorts are connected with one condenser, as shown. The illustration represents a plan, the upper parts of the apparatus being shown.

Another patent along the same lines as the foregoing is that of Hoskins, shown in Fig. 22. One of the methods suggested of utilizing hogged pine wood and saw dust has been to make it into paper. This patent endeavors to show a method by means of which the turpentine can be distilled and the hogged wood remaining in the retort or digester can be treated with caustic soda to form wood pulp. In the illustration A represents the digester made very strong in order to withstand heavy pressure. The digester is surrounded with a steam jacket (a) provided with an inlet pipe (a1) and outlet pipe (a2), a steam tight charging door (b) near its upper end and a steam tight discharging or clean-out door (c) at the lower end. In the digester chamber is an outer steam coil (d), having a valved inlet pipe (d1) and outlet pipe (d2) and an inner perforated steam coil (e) having a valved inlet pipe (e1). Extending through the cover (c) at the lower end of the digester is an outlet pipe (f) provided with a valve (f1) and extending from the top of the digester is an outlet pipe (g) provided with a valve (g1). Interposed in the pipe (g) is a pressure gauge (h); the pipe (g) leads to the upper end of the coil (i) of the condenser (B). In the outlet pipe (i1) of the coil (i) is a valve (i2) and the condenser is provided with a valved cold-water-inlet pipe (k) and a water-outlet pipe (k1).

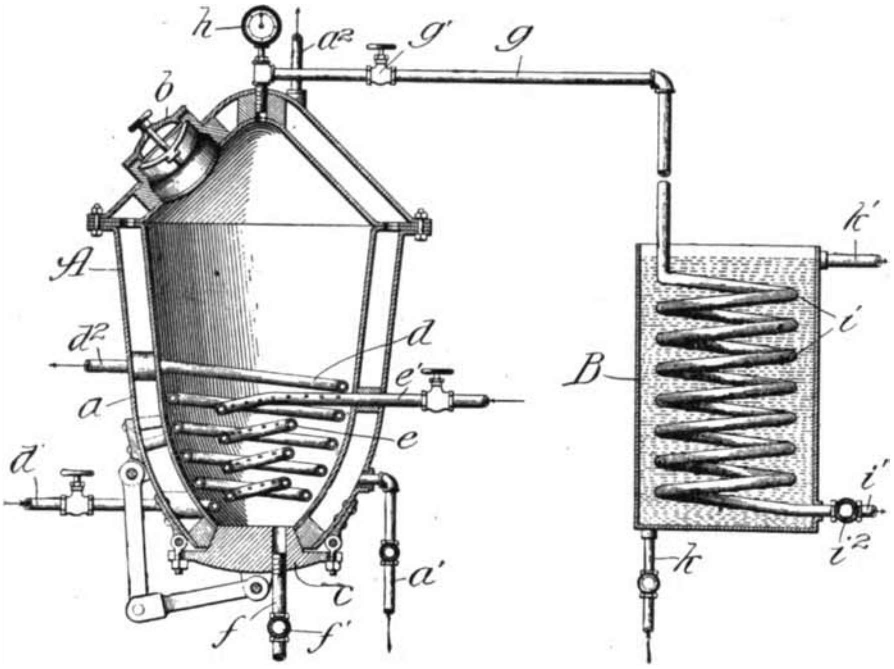


FIG. 22.—HOSKINS PATENT.

The turpentine is steamed off and condensed in the condenser shown. During the operation the resin collects on the bottom and can be drawn off at f. Care is taken not to get the wood too hot and spoil the pulp. After the turpentine has been extracted, caustic soda of about 1.20 sp. gr. is put into the digester and the whole heated and the pressure raised to 75 or 90 lbs. for six to twelve hours, according to the quality and quantity of the wood treated. The alkaline liquor is drawn off and treated as in paper mills or destructively distilled. The wood pulp is blown out and is ready for further treatment in the paper mill. The quality of paper made by pine will be spoken of later.

In this process one operation could be made to do the work of two if the turpentine was taken off when the wood was being treated with caustic soda instead of before.

The next steam process to be considered is that of Mallonee, different in one important respect from Krug's, in that it provides a means of stirring the chipped wood. The process is evidently simply a modification of Krug's. From the illustration in Fig. 23 the essential parts are readily to be seen. The retort is at 1 Fig 1, and is fitted with a mechanical stirrer operated by means of gear wheels. At 17 is a manhole for the entrance of the chipped wood and for cleaning out purposes. Live steam is introduced through the perforated shaft of the stirrer and for heating the resin a closed coil is placed on the bottom. At 16 is a pipe and valve for drawing off the hot resin and at 14 a pipe for leading the turpentine to the condenser. The second still shown in Fig. 2 is used to extract the remaining turpentine and resin from the wood. This latter is simply a retort with bumped head furnished with relief valve and gauge and draw-off pipe similar in nearly all respects to the Krug retort before described.

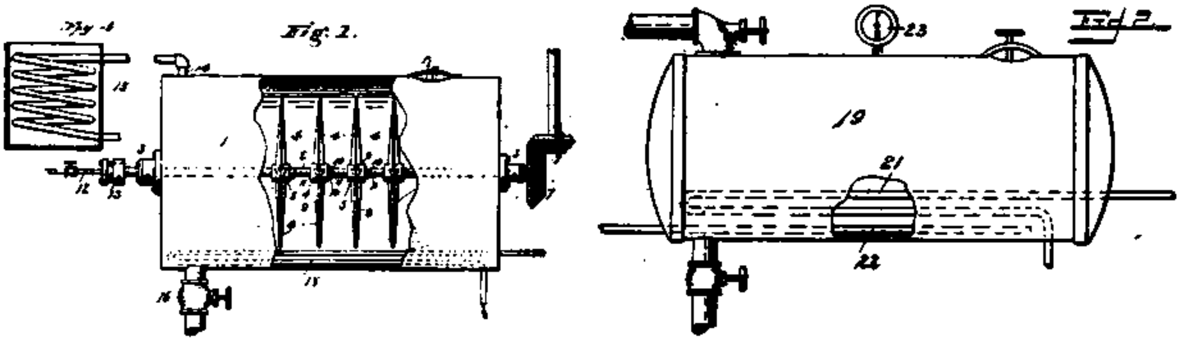


FIG. 23—MALLONEE'S PROCESS.

To use the apparatus the wood mixed with water is charged into the retort or still through the manhead shown in Fig. 1; at the same time the stirrer is put in motion. When sufficiently full the contents are heated by means of the closed coil until the water begins to distill (known by the pipe 14 becoming warm), then live steam is turned on under low pressure, and the turpentine carried over and condensed. When the oil ceases to distill the live steam is shut off and the heating continued by means of the closed coil for a short while until the resin settles, when this is drawn off. The pulp is then run into the still 19 and heated under pressure of 60 to 100 lbs. by means of steam or otherwise, a weak solution of caustic soda being added to extract the resin. The relief valve opens with an excess of pressure, allowing the turpentine to escape to the condenser. After the oil has ceased flowing the pressure is gradually removed and the contents discharged.

As shown in the illustration, this process does not present many merits. The stirrer would be very inefficient if the retort were nearly full of wood and at one plant where a similar stirrer was used they were found to be liable to be destroyed by the arms being broken off. Using water in charging the arms can work more freely, but unless there was a lot of water in the retort (in which case much more heat would be required), channels would form where the arms turned and the stirring would not be very effective. Also, the necessity of charging and discharging two retorts or stills in the manner shown would prove a great drawback. As is the case with the previous process, the necessity of the first part of the operation does not seem apparent.

Another process for the removal of the turpentine vapors from ground wood is that of Hirsch, shown in Fig. 24. In this process the difficulty of discharging the horizontal retorts in the Krug and Mallonee process is done away with. Here the retort or still is set vertical, the wood entering the door 14, fills the interior and is ready for distillation. Steam is turned on by means of a perforated pipe and the pressure allowed to rise to 60 lbs., which is indicated by the relief valve 28, and gauge 29. When the pressure reaches 60 lbs., the large valve 32, is opened and the turpentine sent to the condenser. When the distillate contains but a small amount of turpentine, the steam is shut off and the refuse wood drawn out through the bottom doors. The valve 32, might be a relief valve the same as in the Krug and Mallonee processes. The advantages and disadvantages of the apparatus are apparent from the illustration.

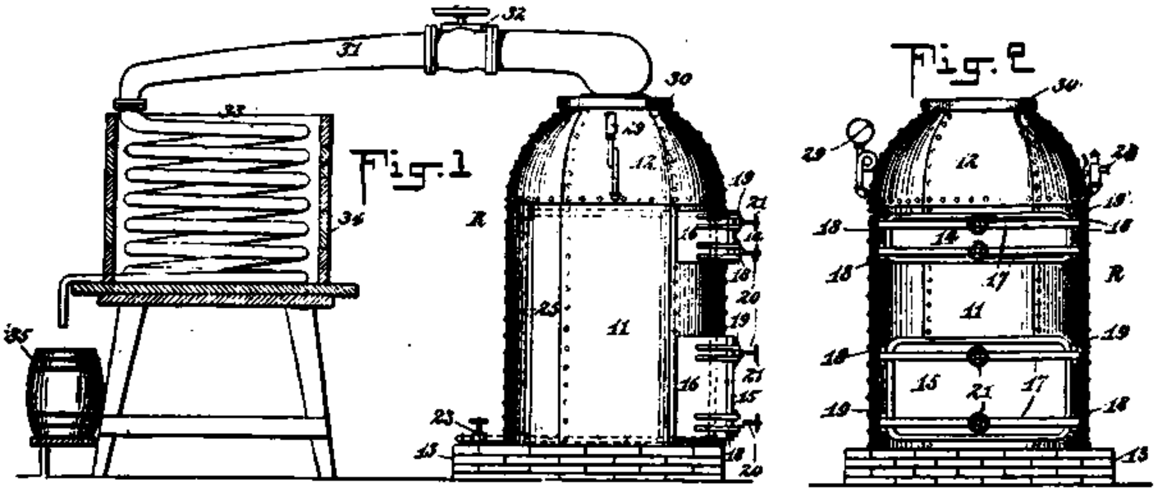


FIG. 24—HIRSCH'S PROCESS.

There are a great many companies offering apparatus for sale under patents all described similar to the process which we will now consider.

In the Gardner process we find the best arrangement of any steam process. The whole success of the steam processes depends upon the mechanical arrangements for handling the wood. In the above processes, it has been noticed that the cost of operating with saw dust or other material giving light yields, would be too great on account of the disadvantageous methods of handling the raw material. We will see from the illustration how this difficulty is removed in this process. Unless some form of rotating retort is discovered which will give sufficiently greater yields in a given time to pay for its extra initial cost, this process, or the various modifications of it, will prove to be the best for all-round work. Mallonee in his process claims as one reason for stirring the material that the yield is greatly increased and the time of operation lessened. This can be done better with a stirrer with vertical shaft than with a horizontal one, but none are entirely satisfactory, as saw dust is not easily stirred.

Gardner's process is illustrated in Fig. 25. In practice only one retort is used, the upper one being found unnecessary. Often a simple bin is used in its stead.

To operate the conveyor 6, brings the hogged wood and saw dust and deposits it into the bin, from whence it falls into the retort 2. All the openings are then closed and fastened, except the vapor pipe 11. A false bottom 10, serves to keep the saw dust from falling into the vapor pipe. Steam is turned in by means of pipes 14 and 15, the distilled turpentine passing out at the bottom. The stirrers 12, are turned to keep the saw dust from packing. After the oil is extracted the bottom is dropped, as shown, and the material falls out in bulk. The residue dries quickly and can be used for fuel.

The next steam process to be considered is that of W. W. and T. L. James. The illustration, Fig. 26, shows a rectangular box retort standing on edge, supplied with steam coils and also perforated pipes for live steam. The turpentine is extracted by means of steam under pressure, the pressure being controlled by relief valve 32.

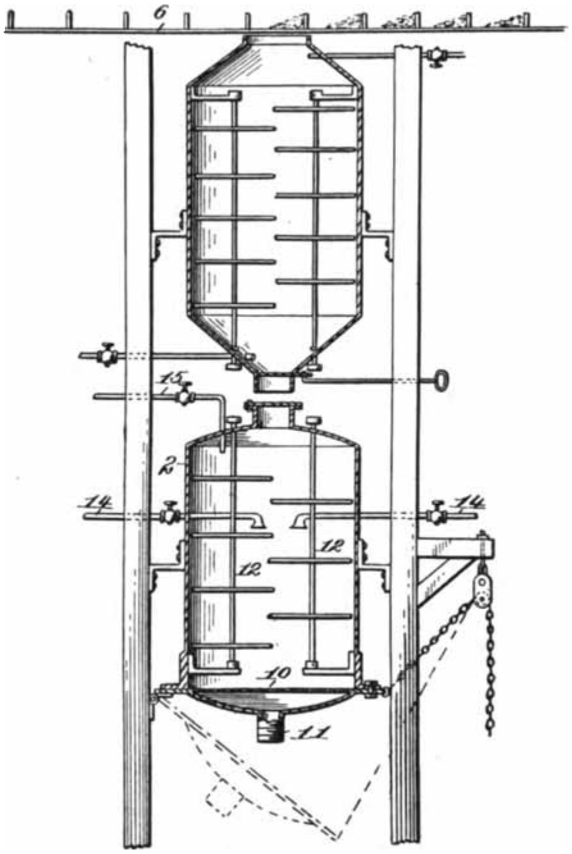


FIG. 25—GARDNER'S PROCESS.

This form has a bad shape for pressure, and attempts are made to keep the sides from bulging by means of bolts. It has nearly every conceivable disadvantage as compared with other steam processes, and but one slight advantage in that it takes less cooling water, the condensing being done by mixing the combined vapors with water by uniting water pipe 13, with vapor pipe 33. Even then, an ordinary jet condenser would be a better arrangement.

The McMillan process was devised to supply a means of rapidly discharging the retort after the ground wood has been distilled. The arrangement consists of an iron retort set vertically with an inner device J, Fig. 27, composed of several sections, each one of which is separate from the others and capable of being forced together or opened by means of the bolts L.

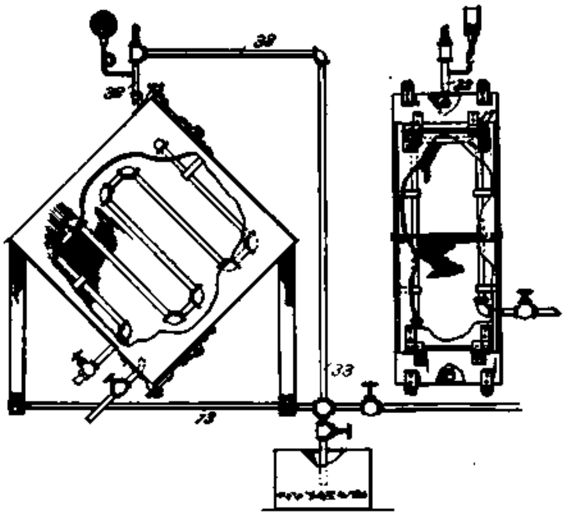


FIG. 26—JAMES' PROCESS.

The wood enters through the large valve D, and drops into the device J, where it is distilled by means of steam entering at A, the vapors passing out at E. At F is a safety valve and at R is a discharge pipe for rosin, tar, etc. After the turpentine has been extracted, the valves are all closed and the sections of the inner device J, loosened by means of the bolts L, thus allowing the distilled residue to fall to the bottom or the retort. The discharged gate B is then opened and steam or air added through A—A1, and the residue blown out.

It can be readily seen that as wood swells when steamed and is difficult to remove from the retort on that account, such a device would overcome this

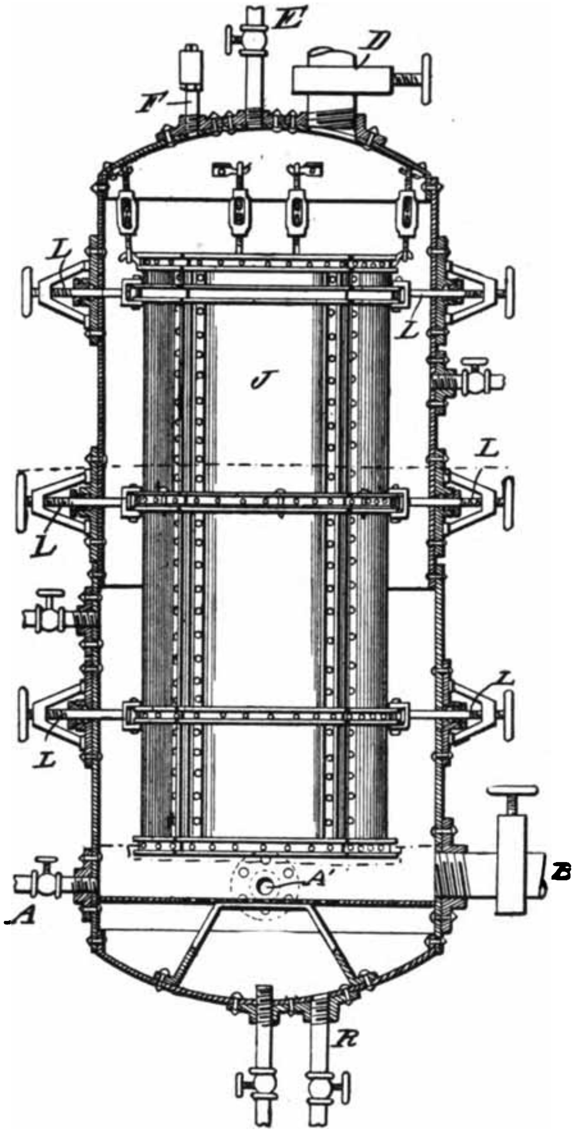


FIG. 27—McMILLAN'S PROCESS.

difficulty nicely. However, stirrers should be used in order to obtain the best results, and with these the material ought to be so loosened as to easily fall out of an ordinary retort such as Gardner's. On this account, such a device is too expensive for the results obtained.

STEAM AND DESTRUCTIVE DISTILLATION AND DESTRUCTIVE DISTILLATION PLANTS.

There are two methods of destructively distilling wood, one using steam to take off the turpentine and the other not using any. Plants designed for the latter method can be easily changed to the first form by simply adding a steam pipe, so in considering these plants they will be classed together. The use of steam is considered by some to be injurious to the oil produced and also to increase the time of distilling. However, most plants get better oil and better results with the use of steam.

The use of steam followed by destructive distillation, would form an ideal process if it were not for the great drawback that steam will not take out all the turpentine from a block or wood without decomposing the woody fiber, except by prolonged heating. That it will do so if heated long enough, has been satisfactorily proven, particularly in the case of short pieces, such as sawed knots. If the wood were hogged then steam would take out the oil readily, as is observed in the steam process, but this spoils it for charcoal, without which product the destructive process cannot hope to pay. Furthermore, saw dust has been found very difficult to distill destructively until recently. The fine wood chars near the shell of the retort, but the heat cannot penetrate to the middle very easily, even in retorts of small diameter. Special retorts to be described later overcome the difficulty of distilling, but the fine charcoal must be disposed of at a good price in order to make the process pay. Briquetting may help if the right kind of bond can be found.

Unless such a process as above described can be discovered, those plants now to be considered must be located near a supply of rich wood and have a ready market for their charcoal. Bad location has been the cause of many a failure. When well located, though, these processes are much more valuable than the steam process alone, because all the valuable products are obtained from wood and only cheap refuse is used for fuel. A process that can utilize the wood without having to use part of the prepared material to fire the furnaces of the plant itself, is the process that would be the most acceptable. Whether such a process will ever be discovered it is difficult to determine. In the steam process the utilization of the pulp for the manufacture of alcohol, oxalic acid or possibly cellulose might prove a satisfactory solution of the problem.

As before stated, wood distilling plants operating on pine wood were in use before 1865. The patent records mention the devices of Hull and Emery about that time. They distilled without steam, and with steam at ordinary pressure and heavy pressure.

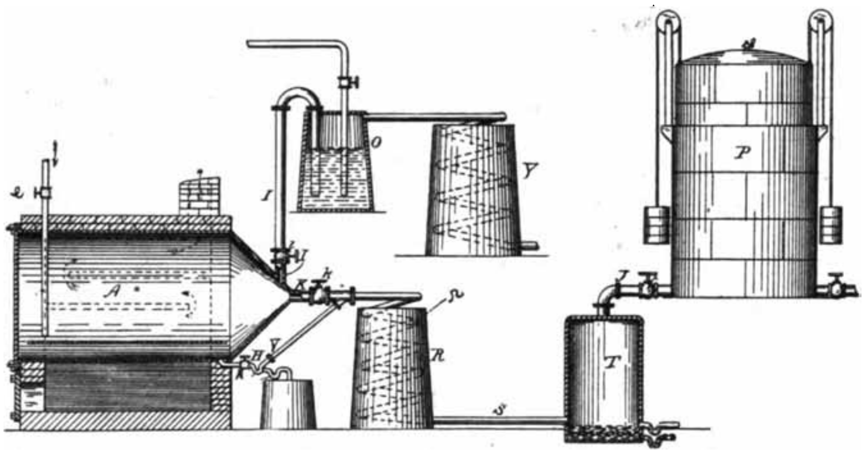


FIG. 28—WHEELER'S PROCESS.

A division of the later processes will be made into those using horizontal retorts and those using vertical retorts.

Horizontal Retorts.

Wheeler's Process.—A process for the extraction of oils from pine wood was patented in 1870 by Wheeler, and improvements added, as shown in Fig. 28.

In this process, the valves i, k and h, were added to his original idea and the pipe I, changed to its present position from the middle of the top of the retort. The retort is shown at A, supplied with steam through valve e. The turpentine vapors pass through pipe I, through tank O, containing hot milk of lime, and cooled in the condenser Y. The creosote vapors go through K and condense in R; any heavy tar condensed in pipe K, flowing down pipe V. When I and K are closed, the uncondensed gas and heavy vapors formed coming from pipe H, can pass up this pipe to the worm r, where the vapors will be condensed while the gas passes through S, to separator T, to gas main P.

For about three or four hours the retort is heated at a very low temperature by means of a low fire and live steam and the turpentine taken off through pipe I. The valve i, and the steam valve are then closed and the retort heated to 230 degrees Fahrenheit, for about two hours, the vapors now passing through valve k, and the gas separating from the condensed liquors at T, going to the holder P. The receiver being changed, the vapors formed at 300 degrees to 400 degrees Fah., pass through the same exit K, and this heating continues for six hours. The valve h, being opened and the valve k, closed, the tar and gas flow out through pipe H, for about an hour or so. As the retort holds but a cord, this severe heating does not affect it so much as it would if it were larger. This separation of the vapors will be noticed in later patents.

Several other patents followed Emery's before Wheeler's, but they are not of sufficient importance to describe. Mention has been made of Stanley's patent. This process was patented later than Wheeler's and comprised an ordinary distilling apparatus consisting of retort and condenser, with necessary arrangements for heating. This apparatus was sold to the Spiritine Chemical Company, who found it convenient to improve according to Hansen's patent, which will be described later.

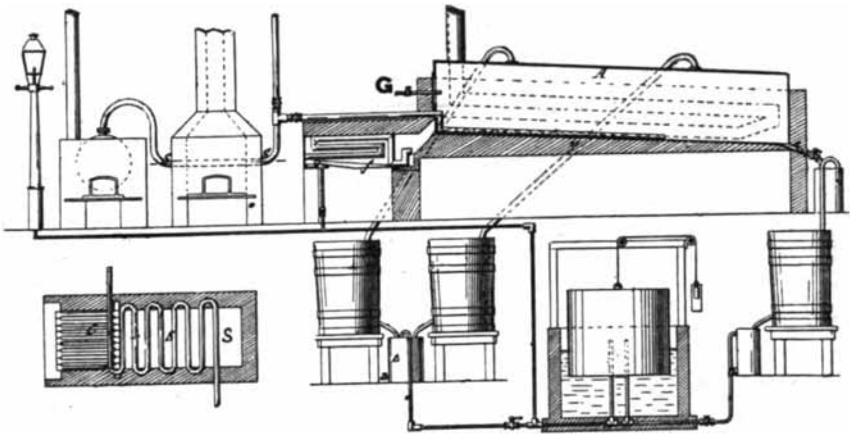


FIG. 29—MESSAU'S PROCESS.

Messau process, illustrated in Fig. 29, is of interest, as two features are brought in that may affect later conditions; first a method of superheating is shown, then a method of adding air to the charge.

In the illustration, A is the retort and S the superheater. The retort is of boiler iron and inclined so that the liquid products can escape at the bottom pipe on the right. The fire does not touch the bottom; the heat passing by means of suitable flues so as to heat the contents. To assist the heating, air is admitted at G. The operation is similar to others; the turpentine being taken off at a low temperature, the tar oil next and the tar running out the back pipe. A modification of this process consists in substituting a bee-hive brick retort in place of an iron one.

The next process is that of Hansen & Smith. The retort is known in the hardwood industry as a double ender, on account of there being a fireplace at each end. The retort B, Fig. 30, is made about 25 or 26 feet long and holds about six cords of wood. The wood is run in on cars, as at F, Fig. 1.

To distill the wood a fire is started at both ends of the retort, the flame passing to the partition A, through the opening g, then winding around the retort, being guided by the partitions b1 and b2, and finally escaping by means of the stack b3, to the air. The bottom of the retort is protected from the direct flame by means of the arch a1. The vapors pass out through pipe f, to a suitable condenser. After the wood is charred the fires are put out and the furnace cooled by means of a ventilating fan, shown at H, connected with h of the furnace, Fig. 1. This apparatus was intended to produce wood creosote, which was then used for creosoting lumber.

There are several Koch processes that are in use, plants being built similar to each one. These plants have nearly all proven failures for some reason or other. The principal reason seems to be small yields from the wood used and a poor market.

They have two bad features, one is a brick arch for protecting the retort and the other is the use of a closed car. Both of these features mean an increased cost for fuel. Too much of the heat goes up the chimney during the latter part of the operation. The arch does protect the retort and is of value where large retorts are used and fuel is cheap. The car is a rapid way of withdrawing the charcoal, but those plants using these cars find that the wood is not evenly heated, the wood in one part being charred before the turpentine is extracted from the wood in another part. It may be that the furnace flues could be proportioned better and the cars be made of openwork structure, so that they can be more readily heated. In this case, a cooler should be used into which the charcoal could be drawn.

Parts of some of these processes are shown in Fig. 31. The retort A, can be made of any size and rails laid near the bottom. The car K, is filled with wood and rolled into the retort, the door of which is then closed. A fire is started in the furnace at D and the flame follows the flue d, until it reaches the baffle at the end of the retort then returns by means of arched brick flues E, E1, Fig. 8, to the front of the furnace, then instead of uniting under the retort and burning a hole therein, the flames enter the side walls at g and then rush back along the sides of the retort to the back flue h and then down to i, which communicates with the stack. This furnace seems to answer the purpose very well when properly constructed, but with oil fuel, the back wall at E has a bad habit of dropping when the furnace gets hot. This trouble is not experienced with wood.

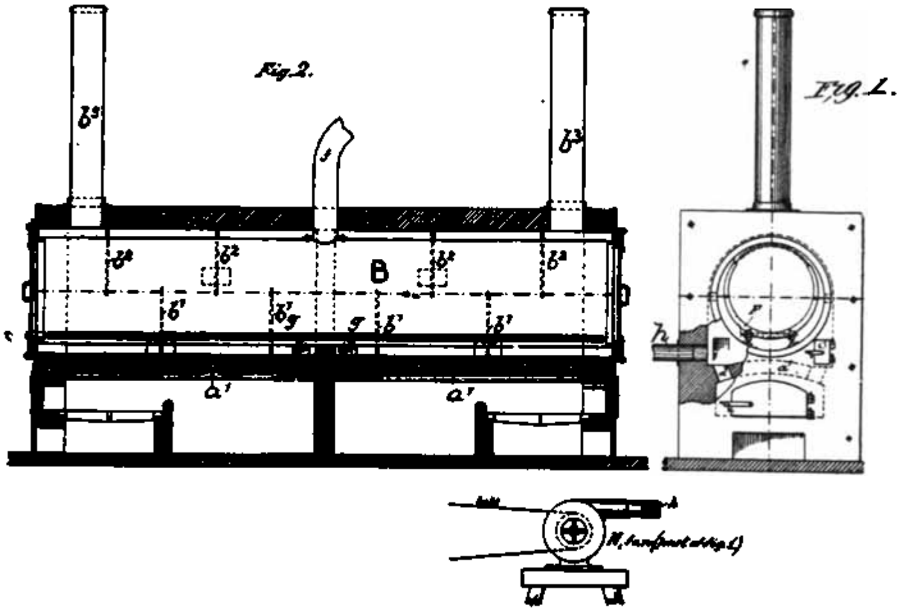


FIG. 30—HANSEN & SMITH PROCESS.

As the heating continues, superheated steam is let in from pipe b, until the turpentine is distilled, when the steam is shut off and the distilling finished by the heat of the fire. The vapors leave the retort at r and follow the pipe to w. This valve is closed when making turpentine and the valve V open and the oily vapors thus escape through pipe P, to a condenser. After the turpentine is taken off, the valve w, is opened and valve V, closed, the heavy vapors going to p, where part of them are condensed and the remainder and uncondensable gases pass through u to a condenser and gas separator. To prevent a deposit of tarry matter in the pipe r, it is surrounded by water contained in the trough W.

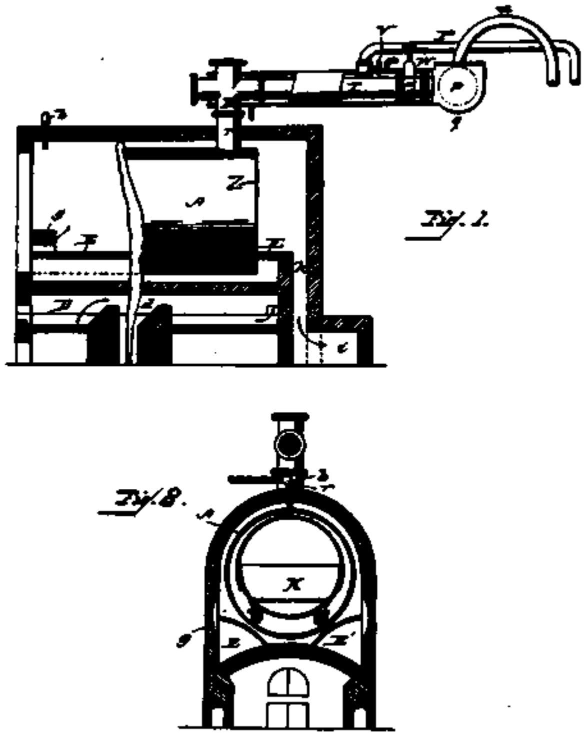


FIG. 31—KOCH'S PROCESS.

Instead of using the pipe as herein shown, the pipe in practice is lowered to Z, the cross R being left out. The water jacket is then brought into connection with the back end of the retort. By this arrangement the pipe r, is found to remain free from a heavy deposit of carbon. Often a tar pipe is placed at the bottom of the front end of the retort, by means of which the hot tar can be removed without vaporizing it.

Under proper conditions, with well proportioned furnaces, this process ought to distil wood without much trouble. As before stated, it takes considerable fuel as now carried on. The use of cars was practiced in the hardwood industry before this process was patented, and their use will be noticed in Hansen's & Smith's process, previously described. The subject will be brought up again later.

It has been stated that in the hardwood industry small retorts are used very successfully for carbonizing wood. By connecting these retorts with suitable condensers, they can be used for distilling pine. One of the most successful destructive distillation plants in the South is comprised of a series of such retorts.

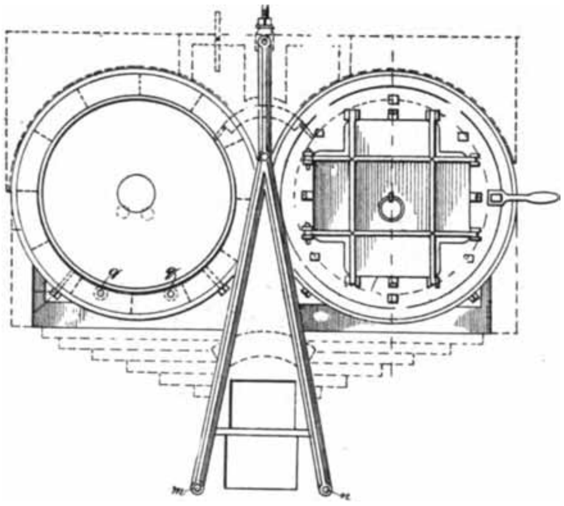


FIG. 32—BADGLEY'S PROCESS.

To protect the brickwork, when two of such retorts are set in one furnace, Badgley devised the apparatus shown in Fig. 32. It consists of a Λ shaped bar placed between the retorts and held in position by bolts m, n, etc., passing through the furnace to the rear wall. Rollers are placed at q, q, upon which the retort can be turned if necessary. Arrangements are also made so that the swollen retort can be easily withdrawn when burned through. The other features of the process are not of particular interest to the pine wood distiller.

Another arrangement for turning the retorts is shown in Inderlied's patent, Fig. 33. The rollers at a and the balls at b b facilitate the turning of the retort. An arrangement is also made to control the fire gases so that they will not accumulate at one place on the retort. No brick protection is shown, the fire gases arising enveloping the retort and pass out through the ports e e e, etc., into the flue f and thence to the chimney. Each of these port holes can be closed by means of a damper if necessary.

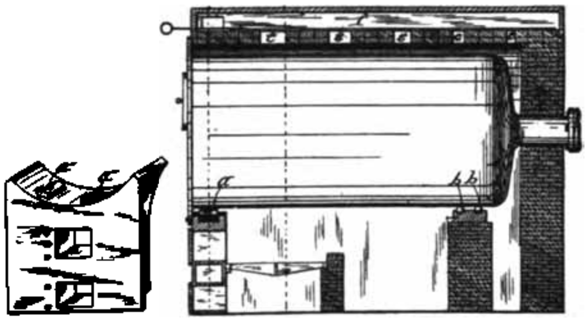


FIG. 33—INDERLIED'S PROCESS.

The flange on the vapor pipe is arranged so that the retort can be given a sixth of a turn or more at a time. Instead of putting two retorts in a furnace, as in Badgley's, this method only allows but one. However, although the furnace construction costs more with only one retort in a setting, it is so much more easy to control the operation when one is used that perhaps this arrangement might be best with pine wood.

If large retorts are to be used in pine wood distillation, the oven form having given satisfaction in the hardwood industry ought to be satisfactory when distilling pine. Usually these are heated by natural gas or oil, but Chapman has designed a furnace shown in Fig. 34 that is intended for saw dust and wood firing. Whether such are in use or not the author does not know.

The oven is at A set in a double-ended furnace. The bottom is protected by means of the tile arch E, in which are port holes to allow some of the fire gases to pass through, the remainder turning back under the overhanging arch F and thus enter the space beneath the retort. In this manner the heat is to be uniformly distributed. It is more than likely that in practice the flames coming through e, striking the bottom of the oven, will burn a hole therein.

The firebox is so constructed that air can be

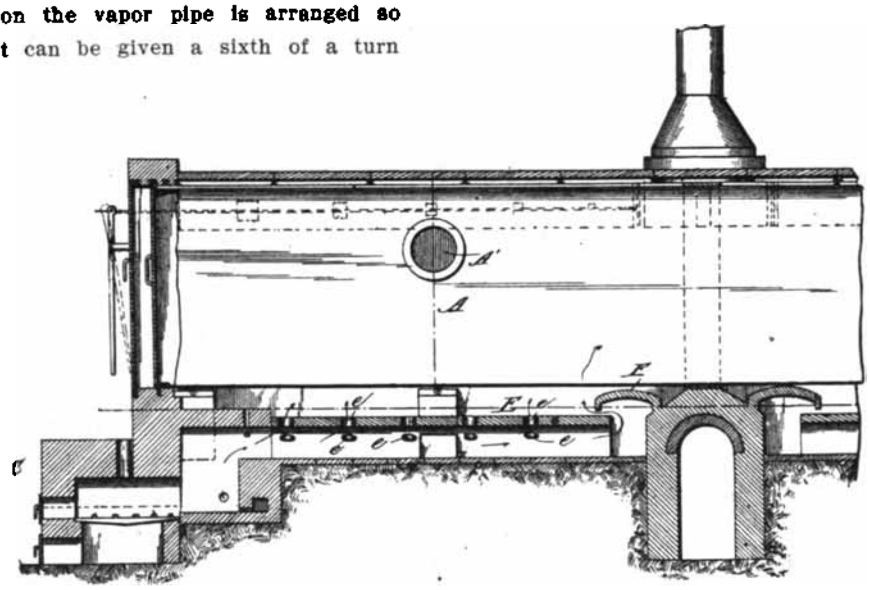


FIG. 34—CHAPMAN'S PROCESS.

supplied through c, so as to burn the saw dust fuel from the top and also to act as a feed hole.

These retorts can be made 50 feet long and should be supplied with rails upon the bottom on which cars carrying the wood can be rolled. For pine wood distillation, suitable steam pipes could be added and the charge distilled in the usual way. The vapors would pass out through vapor pipe A1 to a suitable condenser. Arrangements should be made so that the charcoal can be drawn out hot.

The next process is the device of one who has had experience in both hardwood and pine wood distillation. A plant is now in operation comprising twenty of such retorts in batteries of five with a boiler in between. Ten retorts are in line on one side and ten on the other with the charging ends on the outside of the square formed. The vapor pipes are thus between the two series of retorts. One condenser is used for a series of retorts for the various grades of oil distilled, the vapors from each retort being controlled by a series of valves.

We have in Fig. 35 the illustration of the setting of one of these retorts. This apparatus was devised after several years' use of the Koch processes, so a resemblance to them in many respects may be expected.

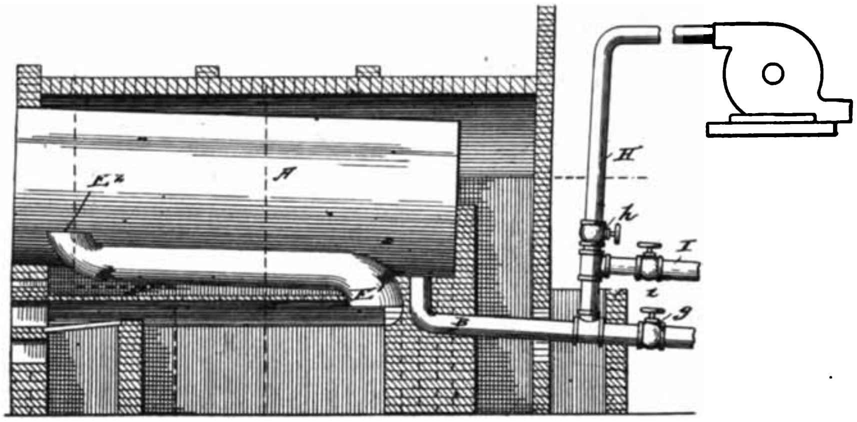


FIG. 35—GILMER'S PROCESS.

The retort is at A and inclined so that the products of distillation may pass out at B at the bottom. This pipe B is set in brickwork and thus protected from the direct contact of the flame, but at the same time is kept warm enough so that the tar can flow. The furnace construction differs from the Koch process in that the flame on its return from the back at E1 is turned up at E2 instead of into the wall, as shown in Fig. 31g. The object sought is to heat the top of the retort more than the bottom so that the distillation might begin at the top and the products of distillation pass out at the bottom. The idea of taking off the products of distillation at the bottom is a good one and is practiced largely with vertical retorts, particularly on the Pacific coast. One would think, though, that the vapors of turpentine being so light, they would be better taken off at the top. Such might be the case, but the proportion of other products is so much greater and the vapors so much heavier that it would be easier to take them all off at the bottom. Furthermore, it would be necessary to vaporize the tar to drive it through a high pipe, whereas with a pipe at the bottom it can be drawn off as a liquid, thus saving fuel. When the object is to separate the vapors into fractions as shown, it is not advisable to heat any portion of the retort too strongly, as otherwise the wood in one part of the retort would be yielding tar before the wood in another part had lost all of its turpentine and thus the turpentine be contaminated with tarry vapors. Other disadvantages of heating a retort at the top would be that it is more difficult to heat the top than the bottom; a fact well known; and also that in the later stages, when the most heat is required, the wood as it decomposes settles to the bottom, thus getting further and further away from the direct heat, and at the last stage, when the most heat is required, the wood is nearly half the diameter of the retort, away from the top. It would seem that a definite uniform heat, progressing slowly greater in intensity until the wood is charred, is better than to heat either the top or the bottom to a different degree; then by taking off the products of distillation at the bottom the best conditions would be fulfilled with little decomposition of the resin or vapors.

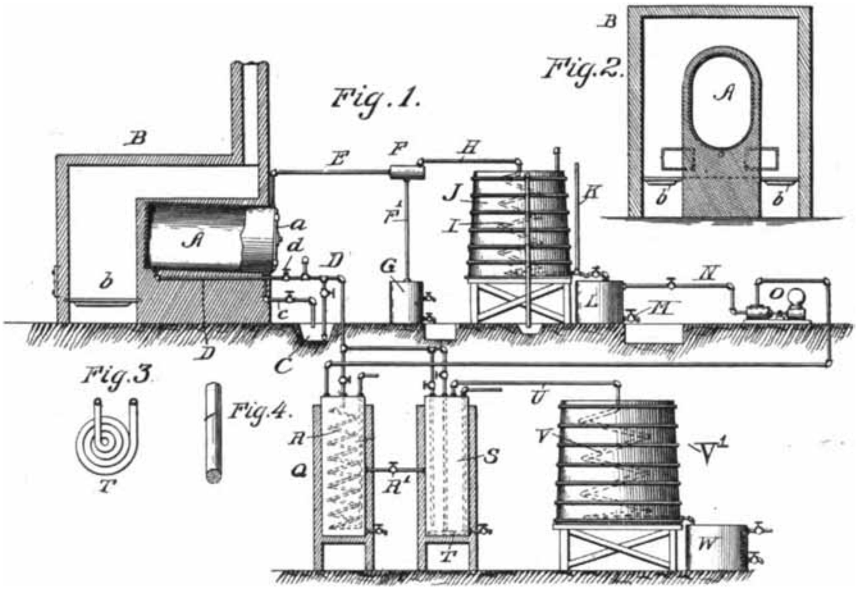


FIG. 36—BROUGHTON'S PROCESS.

As the products come over, the light oils pass up the vapor pipe H and connect with a main pipe from the other retorts of the same series. In a similar manner when the proper temperature is reached, valve i is opened and valve h is closed, and the vapors pass through I to another main pipe. Toward the latter end of the distillation, the tar flows through valve g. In all cases, the uncondensable gas is separated at the end of the condenser.

The collected crude products are sent to a refining house, where they are specially treated according to the kind of product. For this purpose copper stills are used, having about the same height and diameter and furnished with suitable steam coils, vapor connections, etc., for distillation. The turpentine is generally once distilled, then treated with lime and aerated, then redistilled very slowly so as not to carry over any coloring matter. The vapor pipes are small and carried to a considerable height to a condenser, under which is set a galvanized tank to receive the distillate. The water is drawn off from time to time, leaving the oil, which is placed in barrels for shipping.

In Broughton's process, Fig. 36, steam is used, followed by destructive distillation. Here an attempt is made to fractionate the oils direct from the retort. In many respects it resembles Bilfinger's process, except that a horizontal retort is used instead of a vertical one. In the illustration the process looks rather complicated, but in reality it is very similar to others and can be operated as readily. The refining apparatus is also shown.

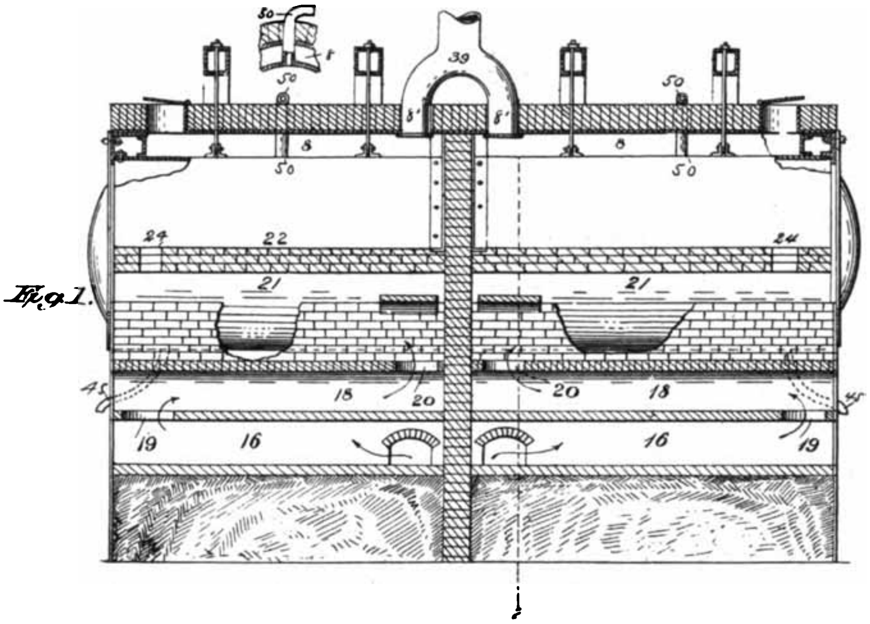


FIG. 37—MALLONEE'S PROCESS—Fig. 1.

The retort A is set at an incline to the front. At a is the charging door. The grates are in the rear of the furnace, B, one each side at b1 Fig. 2. By the arrangement of the furnace, the direct flame from the fire cannot affect the retort. But it is a very poor device for heating, the arrangement shown in Koch's process, Fig. 31, being far better. The vapor pipe for the light oils, E, connects with F, a contrivance similar to the barrel shown in Fig. 41 at 25 and serves the same purpose, that of separating, partially, light oils from heavy oils. J is the condensing tank, K the gas pipe, L the receiver. O is a pump for pumping the liquor to the heating tank Q, from whence it goes to the still S through the worm V to receiver W.

To operate, the wood is charged at a, the door fastened and steam turned on at about 300 degrees Fah. to carry off the major portion of turpentine. This continues about six hours, when a fire is started and the retort heated to a temperature of 450 to 500 degrees Fah., this part of the operation also taking about six hours. The steam is then shut off and the retort gradually heated to about 800 degrees Fah., or until the wood is thoroughly carbonized. The turpentine which comes over during the first six hours goes through E. F. H and J, to the receiver L, the heavier oils going down F1 to G, where they are drawn off. The turpentine and some creosote that comes off during the next six hours follow the same course, the tar oils and some creosote dropping down F1 to G and the turpentine and some creosote going to L. The tar formed flows out of the bottom pipe c into the trough C. This comprises the regular operation for obtaining the crude materials. Only the turpentine is refined. The oil is separated from the water in the receiver L and then forced by the pump O to the heater Q, which acts as a sort of reservoir for the still S. The oil in Q is heated by a steam coil to about the boiling point of turpentine and when sufficient collects in the heater it is drawn through R1 into the still S, which is heated by a closed coil, T, at the bottom. The shape of the still as shown is not of an approved pattern. As the temperature is raised the turpentine distills, aided by a jet of steam, through U to the condenser V1 to the receiver W, where it is ready for shipment.

This process offers nothing specially new, but it brings back the idea of refining in a current of steam something which is being neglected at plants of this kind. If by this method of procedure all the turpentine in the wood can be recovered without color or odor, a great advance has been made over the older processes. It is doubtful, though, if turpentine containing creosote can be refined in the manner described and creosote not be found in the distillate.

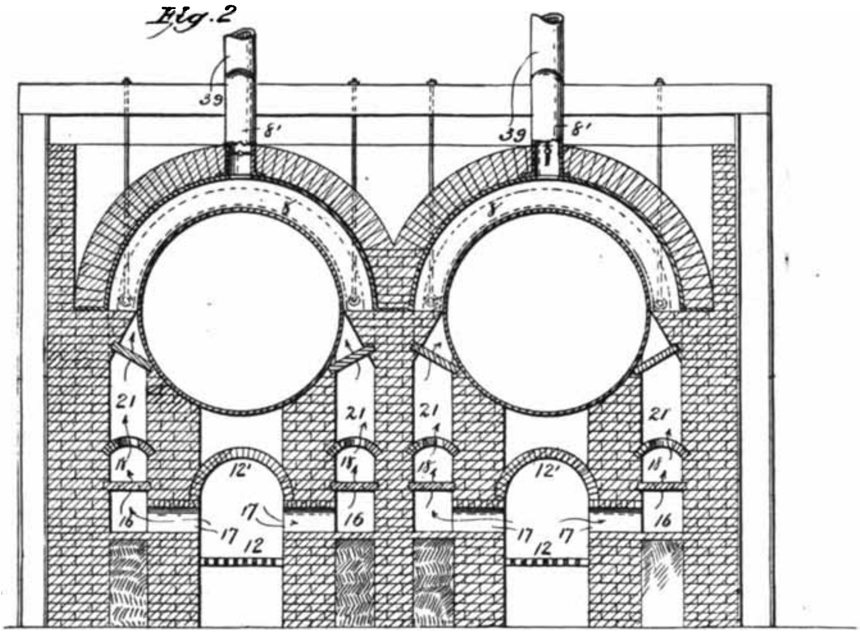


FIG. 37—MALLONEE'S PROCESS—Fig. 2.

Mallonee's process sometimes uses steam to extract the turpentine and sometimes not, according to the operator. There are several processes suggested by the same inventor, the steam process being already described. The illustration Fig. 37 shows part of two arrangements. Fig. 1 shows a double-ended retort set in a special furnace so arranged that the bottom of the retort is not heated to a high degree.

Starting at 12, Fig. 2, the flame goes back under the arch 12-1 to the connecting flue 17 by means of which it passes to 16. Following 16 to Fig. 1 it passes upwards through 19, then rearwardly through 18, then upwards through 20 into chamber 21. Here it passes along the sides of the retort and going through 24 enters the chamber 8 above the retort, from which it passes through 8-1 into the stack 39. The vapors and the tar fall to the bottom and flow out the pipe 45, while the gases pass out at 50.

A previous patent is shown in Fig. 3 of the same illustration. The patent claims state "that heretofore the method commonly used consisted in reducing the wood by means of fire acting on the closed retort containing the wood and then catching and condensing all the distillate in one bulk and running it into one receptacle. The contents of the receptacle were then refined by the use of an ordinary still." As the Bilfinger process using vertical retorts was being exploited before this patent was applied for, it is surprising that it was not known that the distillates were divided into several parts by other parties. Koch and Wheeler did the same thing sixteen years before.

However, the method as used in this process is worth considering, for a distinct method is described which is more definite than some others; the refining operations being much more distinctive.

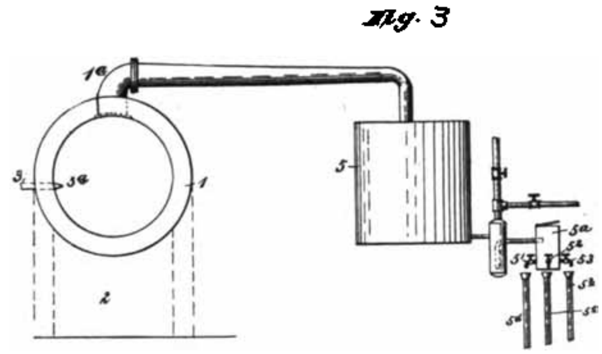


FIG. 37—MALLONEE'S PROCESS—Fig. 3.

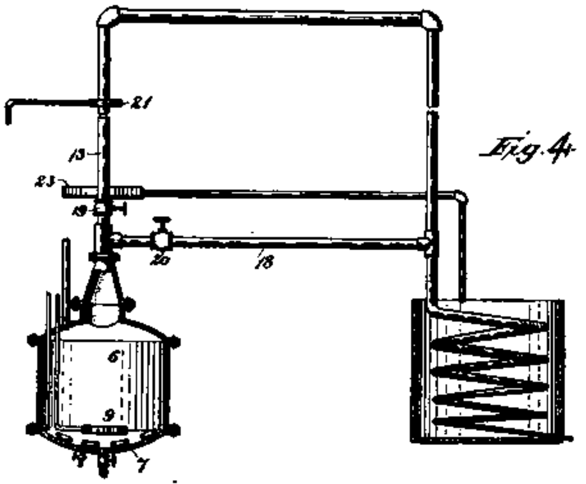


FIG. 37—MALLONEE'S PROCESS—Fig. 4.

In the illustration 1 represents the retort set in the furnace 2, steam when used enters at 3a. The vapors pass through 1a to the condenser 5 and thence through the gas trap to the liquor separator 5a. The refining apparatus is shown in Fig. 4 of the same illustration. The still 6 is of ordinary construction furnished with steam closed coils 7 and steam jet 9. The light oils pass through valve 19 up pipe 13 about twenty to twenty-five feet, then turn to the condenser. The turpentine which distills later is allowed to pass through valve 20, the condenser valve 19 being closed.

To operate, the wood being in the retort and the head tightened, fire is started in the furnace and steam turned into the retort to help carry the vapors to the condenser. The distillate increases in gravity as the heating progresses and the condensed matter is allowed to flow through the cocks, down pipe 5d (Fig. 3), to a receiver until the sp. gr. reaches 0.92. This cock is turned off and the distillate allowed to flow through cock 5-2 down 5c until the sp. gr. reaches 0.96, then the cock 5-3 is opened and the remainder of the distillate allowed to flow through 5-3 down pipe 5b until the wood is thoroughly charred.

The second part of the operation is briefly described. The first two fractions are treated separately in stills of the construction shown. The idea is to fractionate the oils. To do this the first portion of the distillate is allowed to go up pipe 13 (Fig. 4), where the pipe is cooled by water spray near the top at 21, the excess water being caught in pan at 23. This has a tendency to condense the heavy oils and allow only the light naphtha-like oils to pass over. After these are taken off caustic soda of about 1.20, amounting to 5 or 10 per cent of the charge, is allowed to gradually enter the still. This causes frothing and when it subsides more naphtha-like oil is distilled over, then as the turpentine starts to distill it is turned directly into the condenser through pipe 18, the valve 19 being closed. More details will be given under refining methods.

The third fraction is treated similarly to the others, except that caustic soda is not added. When the light oils from this third fraction cease coming from pipe 19, part of the residue in the still will pass through pipe 18 to the condenser when valve 19 is closed. After this oil ceases to distill, instead of heating with fire heat to thicken the tar, the residue is distilled in a current of steam which takes over the remaining light oils, leaving the tar in the still in good condition for the market.

In Palmer's process the object is to obtain turpentine only from short pieces of wood by means of steam under pressure. Here resort is again had to the use of cars to transport the wood with facility. Three rail or two rail tracks are used in the retort upon which the specially constructed cars are run in. The patent relates particularly to the use and construction of the cars. Money could have been saved by investigating other processes in use at the time application was made for the patent. The same idea, better in some respects, had been in use several years before this patent was issued. An illustration of such a plant using cars of this kind for extracting the turpentine by passing steam through a perforated pipe, extending through the middle of a perforated car will be found elsewhere. At this plant the steam treatment was followed by destructive distillation.

As the perforated car is perhaps the best form of car for use for both purposes, a description of it may as well be given here.

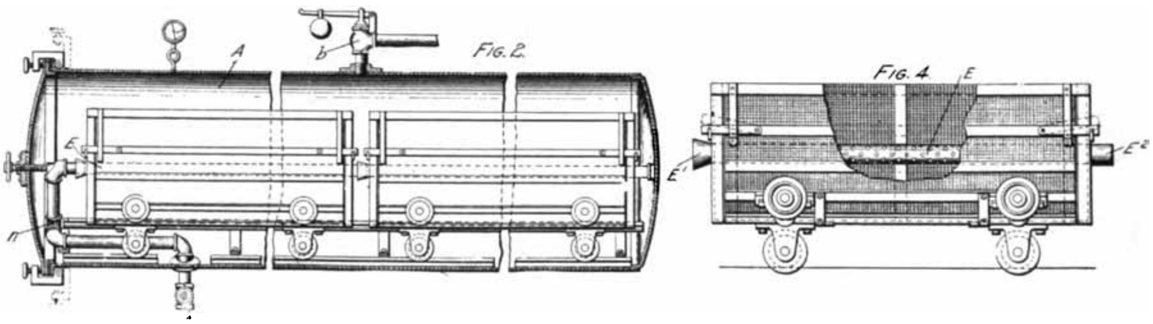


FIG. 38—PALMER'S PROCESS.

In the illustration, Fig. 38, Fig. 2 represents a retort, A, fitted with a three rail track upon which rests two cars. A steam pipe, E, sends steam through the middle of each car. This pipe is renewed by means of the revolvable joint, n, when the cars are to be taken out. Any excess pressure of steam escapes through the lever safety valve, b, taking with it the turpentine vapor as described in Krug's steam process. By setting the retort in a proper furnace the wood can be destructively distilled and the charcoal be drawn out in the cars and rolled into a suitable air tight cooler, the volatile product escaping in the usual manner.

The special construction of the car is shown in detail at Fig. 4. At E is the perforated pipe fitted at one end with a funnel shaped arrangement, E1, and the other end without the same. By this arrangement when two or more cars are placed in one retort the end E2 fits in the funnel shaped end of E1 of the other car, thus making a loosely connected pipe extending through all the cars. The framework of the car is made of slats of iron and the whole covered with wire netting.

It has been found in practice at the steam and destructive plant, before spoken of, where similar cars were first used that there are certain features of construction necessary to make the cars satisfactory.

They must be made sufficiently rigid that the wheels will not get out of line, two rails are better than three, the perforations in the steam pipe and in the netting must be very large to keep from filling up with tar and gum, and in making tar should be fitted with a device for scraping the bottom of the retort.

In Adams' process an oven with a V-shaped bottom is surrounded on the sides and top with an iron shell. At one end of the shell is a fireplace and on the other a brick flue. The space between the oven and outer shell is for the fire gases to circulate, and by means of partitions these gases are led rearwardly in a zig-zag manner to the flue without touching the bottom of the oven. The oven is set horizontally and from various points in the bottom are tar pipes. At the top of the oven are several vapor pipes leading to a condenser, and one safety pipe connected with the flue for the escape of gas when there is much pressure in the oven.

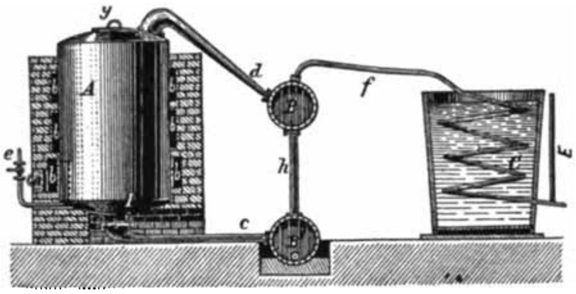


FIG. 39—HESSEL'S PROCESS.

Like all retorts where the heat is not applied to the bottom, it is difficult to thoroughly char the wood, so when the distillation is about finished air is admitted through suitable openings in the oven, which burns the remaining tar left in the wood, the products of combustion being carried through the safety pipe to the flue. The only merit it possesses is that it is easy to get at the tar pipes.

Vertical Retorts.—Although a horizontally placed retort is more easily heated, there are many processes patented which require a vertical retort. A vertical retort with a diameter much less than its height ought to make better tar than a horizontal retort, for the reason that it is not so necessary to keep the bottom hot in order to completely char the wood. When tar is vaporized it usually breaks up into thinner products and leaves a deposit of coke. By keeping the bottom of the retort cool this vaporizing of the tar can be prevented to a large extent. In vertical retorts of small diameter only a comparatively small surface comprises the bottom, so for this reason, in spite of the difficulty of heating, a vertical retort is of value.

Several forms of vertical retorts have been in use in the hardwood industry in Europe for some time. All these forms seem to have given way to the form shown in Fig. 39, known as Hessel's Thermo-Boiler, or Swedish Thermo-Boiler. This illustration is taken from the Consular reports of 1901, given by Consul-General Mason at Berlin.

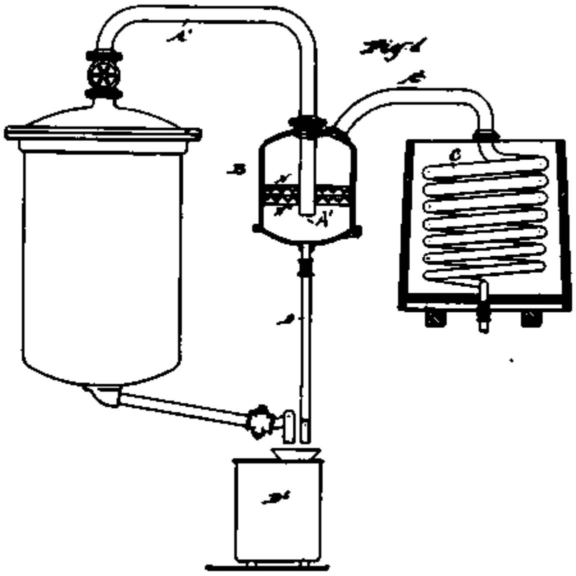


FIG. 40—ROAKE'S PROCESS.

A close resemblance to this apparatus will be seen in American processes patented since that time. As this form can be readily used for distilling pine wood, a description will be here given.

The wood is dropped in at Y into the retort A and the cover placed on. To make turpentine a fire is started at a and superheated steam turned in from pipe e. The turpentine and light oils pass over through to B, where the creosote falls to B1, the light oil following f to the condenser C, and thence into separate receivers; the gas escaping through E into the air or led to the furnace. The tar formed falls to the bottom of the retort and flows out at c into B1, which acts as a tar pipe for a series of retorts. The charcoal formed is drawn out at y or drawn out at a door near the bottom, b.

The first American process for distilling pine or other wood by means of vertical retorts that will be considered is that of Roake, Fig. 40. The process has to do entirely with the removal of the heavy vapors from the lighter ones by condensation in a special vessel, B, corresponding to B, Fig. 39. This vessel is made very large, so that the vapors entering at the bottom from pipe A1 move very slowly, thus giving more time for the heavy vapors to condense and flow down D into D3, while the light vapors pass through A2 to the condenser c. The trays n n1 are added so as to present more cooling surface to the vapors. It can be readily understood that this apparatus could be used with horizontal retorts.

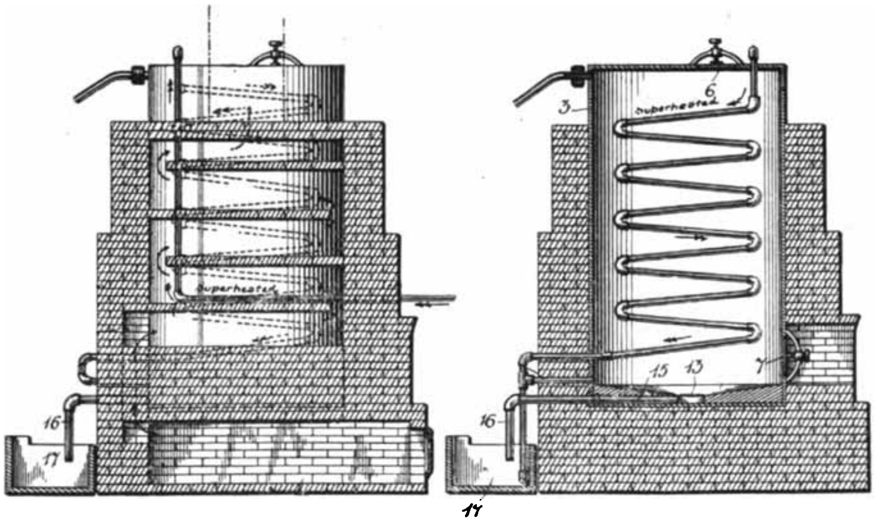


FIG. 41—BILFINGER'S PROCESS.

The next process, that of Bilfinger's, Fig. 41, is perhaps the most exploited of any of those using vertical retorts.

The illustration shows the general construction, 3 being the retort, 22 a box condenser, 31 a goose-neck trap for the gases which escape above the roof through pipe 34. The products of condensation are divided into three grades and each grade carried into a separate pipe to a storage tank. The draw-off valves from the condensers leading to these pipes are shown at 32. The barrel 25 serves the purpose of Roake's device just described, that of separating some of the heavier vapors from the lighter ones. In practice a pipe was placed at the middle of the shell of the retort connecting with the tar pipe, 16, thus making another separation of the vapors in the retort itself.

The furnace construction and retort with heating coil are shown also. The retort instead of being round is oval, and two are set in one furnace, with the fire door between. No grates are used, as the fires can be better regulated without them. At the bottom of each retort at 7 is a man-hole for withdrawing the charred wood. At 13 is a screen to hold back the chips and broken wood, while the tar flows on through pipe 15 to the trough 17. In practice conveyors carried sawed wood to the top of the furnace, from whence it is put into the retort through manhole 6. After charging the retort the manhead is replaced and tightened and a fire started in the furnace. In some plants steam is not used. The temperature is kept quite low and very clear, white oil distills over and it is allowed to flow into its proper receiver. As the temperature rises a more and more yellowish product is obtained and in the latter stages a bad odor and considerable gas, the latter escaping into the air. By closing and opening the proper valves at 32 the different grades of oil go to their respective receivers. In the meantime, most of the resin and tar is discharged at 16 and flows from the trough into a well. The wood is not thoroughly charred, but the process is stopped with the making of red wood, or torrified charcoal. The tar formed by this process is of very good quality, as it is not contaminated with the black tar formed during the later stages when wood is completely charred.

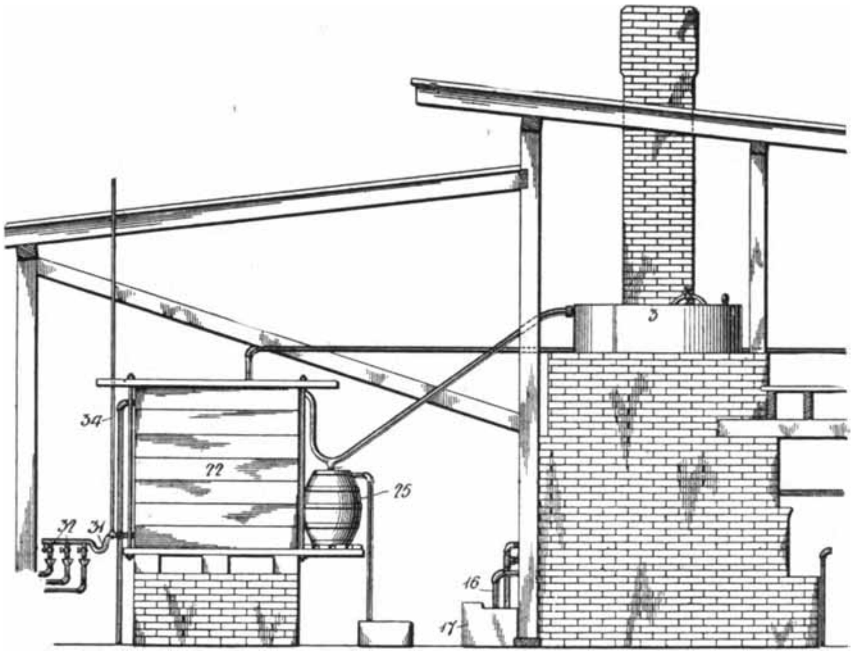


FIG. 41—BILFINGER'S PROCESS.

A plant of this kind generally consists of ten or more retorts set in a row and hold about one cord each. A distillation usually takes 36 hours, thus allowing each retort to be charged and distilled four times per week, starting Sunday night at 12 M. N., and ending Saturday night at 12 M. N. The different grades of oil thus produced are redistilled and are then ready for the market.

Of the many plants erected to use this process all, or nearly all, have failed. Although like all other processes, the different by-products can be obtained from the wood and of very good quality, it is not to be expected that a plant making only turpentine and tar and taking 36 hours to complete the operation, would be as successful as a steam process which takes only from one to six hours. As with all destructive distillation processes, the retorts are damaged to a great extent by the heat and are found to leak. Furthermore, a destructive distillation process that does not make salable charcoal cannot succeed in competition with a steam process, as the tar produced by this method is not of sufficient value to warrant the expense of obtaining it. With charcoal at a high price, a destructive plant might pay where a steam plant would not. This would be particularly true in those cases where wood costs more than the value of the turpentine produced from it.

The failure of the different Bilfinger processes has had a very depressing influence upon the wood turpentine business generally, which never has been very encouraging, anyway, notwithstanding the booming it has always received each time a new patent is granted. The owners of the plants have themselves to blame, for in most cases they had their choice of better and longer tried processes.

Processes taking 24 hours or less are now demanded, and those processes that cannot fulfill the conditions should not be considered. One party who used the Bilfinger process has now a prejudice against all destructive processes, considering them to be total failures. He is an adherent of the steam process, which has proven satisfactory to him.

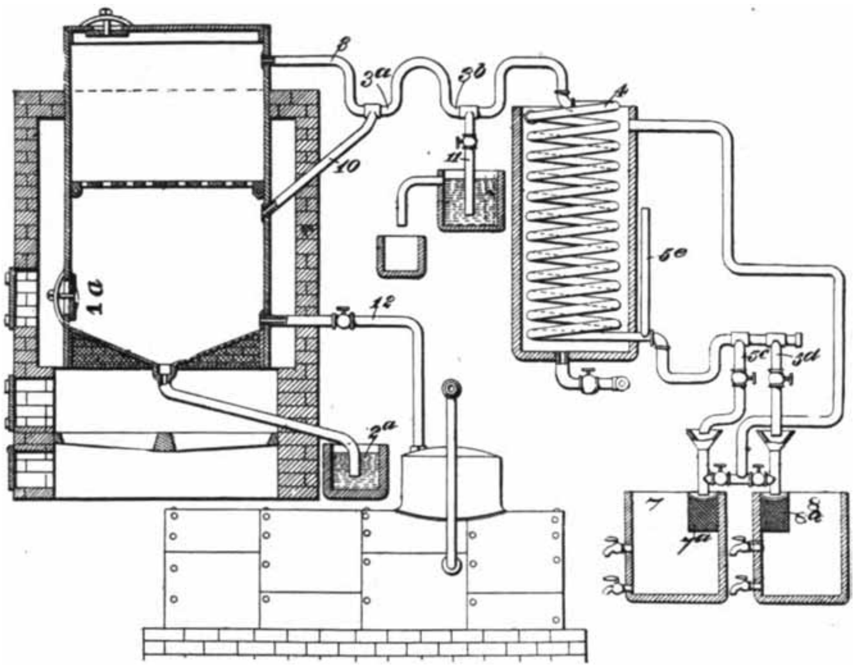


FIG. 42—PALMER'S PROCESS.

There are two Palmer processes using vertical retorts, the latest one being shown in Fig. 42. The working of the apparatus can be easily understood. Starting with the wood in the retort and the vapor pipe 5d or 5c at the end of the condenser open, a medium fire is started and steam turned in from pipe 12. The vapors of oil and water rise and pass out through pipe 3, where the tar oils are supposed to condense at 3a and flow down pipe 10 and the creosote to condense at 3b and flow down pipe 11 and the remaining vapor passes through condenser 4, where part of it is converted into liquid form and runs down 5c or 5d to the receivers, 7 or 8, the uncondensed gases escaping through 5e. One of the receivers, 7 or 8, is used for the light turpentine oil, which comes over first, and the other for the heavier oil during the later stage. Each receiver is supplied with a filter, 7a and 8a, and also with connections for a supply of warm water. This warm water is used as a refining agent to remove some of the impurities. The tar flows out at the bottom after the steam has been cut off and the heat raised, and finds its way to a sealed trough, 2a. The charcoal is taken out at 1a.

This process, although similar to the Bilfinger, is not as good. The bottom not being well protected, the tar is overheated, and what is worse, the tar pipe, 2a, will choke up and burn off if set as shown. The same drawbacks of the Bilfinger process relative to the time of distilling apply equally as well here.

Another process, evidently a modification of Bilfinger's, is that of Douglas, shown in Fig. 43. Two retorts are set in one furnace and connected with the same series of condensers. Instead of passing all the vapors through one condenser and separating them into three portions at the tail pipe, a separate condenser is used for each light product. In operating the wood is placed in the retort and a fire started in the grate. No steam is used, the vapors rise on account of the heat and the light vapors pass out near the top, down pipe 18, over barrel 25-1, which receives any condensed creosote flowing down pipe 24. The light vapors pass on through the condensing coil 19, where they are condensed. As the heat progresses the heavier vapors formed do not rise as high, but are allowed to flow through pipe 25 to where it joins pipes 33, 26 and 27. Any condensed matter can thus flow down either pipe 33 or 26 into the barrel, while the uncondensed vapors follow pipe 27 to condenser 28, where they are partially condensed and flow into a receiver, any uncondensed gases going up through pipe 31. Pipe 32 carries out any light tar oil direct to the barrel, any light vapor and uncondensed gas going up pipe 33 through 27 to the condenser 28, where they are separated and condensed in the usual manner. The tar flows out 35 into the trough 36. This process also has the drawbacks of the Bilfinger process, as previously mentioned.

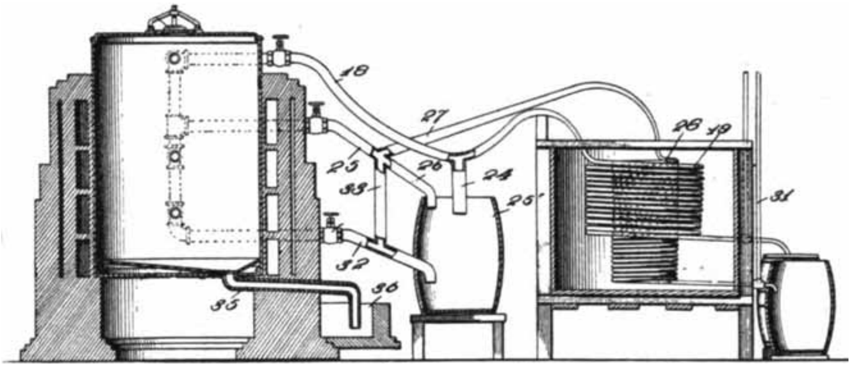


FIG. 43—DOUGLAS' PROCESS.

A more elaborate process for the refining of the vapors coming from the retort is that of Clark & Harris. Chemicals are used to fix the impurities.

The object of the patent seems to be to protect the inventors in a process for the extraction of pine oil. The claims state that pine wood yields up a small quantity of turpentine as an educt, whereas the bulk of the light oil is a product of decomposition coming over when the temperature reached 240 degrees to 300 degrees Fah. Furthermore, the claims state that the pine oils will not distil over with a low temperature steam. If this were true, the modern steam plant using steam at 5 to 10 lbs. pressure would yield only a small quantity of turpentine when used with fat pine.

However, actual practice shows 15 to 16 gallons of oil to the cord under these conditions, which is as good a yield as when the temperature is higher. The discussion of what the products and educts from pine wood are will be left for another chapter.

This process is illustrated in Fig. 44. To carry out the operation the retort a is filled with pine or fir wood and heated gradually in any suitable manner. During the distillation tar is formed and is best drawn off from the bottom of the retort as fast as it is formed, the tar pipe, a2, being always open.

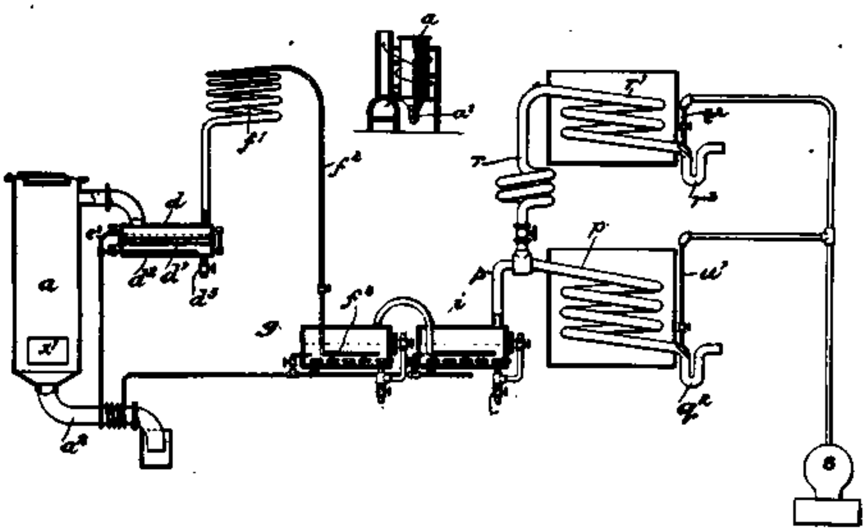


FIG. 44—CLARK & HARRIS PROCESS.

The vaporized products of the distillation pass into the air condenser d, into the bottom of which the heavy oils, acetic acid, water, etc., precipitate and from here they can be drawn off through passage d3. It is advisable, however, to let them stay a short time in the bottom of the condenser, in order that the heat of the vapors may evaporate any pine oils contained therein, which would otherwise be carried off with the heavy products in case they were withdrawn immediately. However, the best way to guard against possible loss of pine oil through condensation at this point consists in providing open and closed steam pipes e1 and d2, within the condenser d, whereby the temperature therein can be regulated to prevent condensation of the pine oil vapors. The open steam pipe e1, also serves to help along the vapors and to clean out the apparatus when necessary. The condensate in the condenser d will in actual operation preferably be kept at such level that the live steam from pipe e1 will play over its surface. From the condenser d the vapors pass through the pipe to condenser f1, where more condensation of heavy vapors take place. These flow back to d, while the lighter vapors pass down f2 through the perforated pipe f3 into the box g containing milk of lime or other forms of alkali, which absorbs the acetic acid, etc. Any carbonates formed would, of course, be decomposed by any excess of acetic acid to form acetates. The unabsorbed vapors then pass to a similar box or tank i, containing a solution of caustic soda of preferably 1.21 sp. gr. This alkali absorbs the heavy oils, forming a soluble disinfectant. The vapors pass up p into the air condenser r, where the pine oils are condensed, while the light, bad-smelling oils pass to the condenser r1 and are liquified. A condenser p1, is used to further condense any pipe oil vapor not going beyond r. The gases are tapped by the

U, r3 and q2, and the pump S draws the gases away from the apparatus by means of pipes, u1 and t2. The charcoal is drawn out at the door X1.

There is not much doubt that this process can produce a clear, white oil during part of the distilling process, but it would seem that the process would require a great deal of care in order to make all parts of the apparatus work together, and also it appears that it would be necessary to distil slowly. The industry requires retorts that will extract the oils rapidly like the steam process does, so that there will be but little expense for plant equipment. One retort with the steam process will do as much in one hour as with this process in twelve. It is more economical to have refining stills, which are very small in comparison to the amount of oil treated, than to have refining retorts.

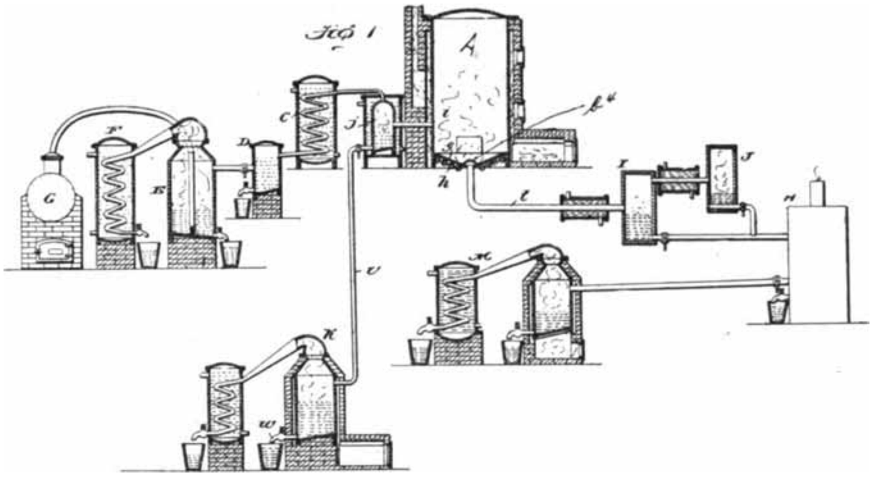


FIG. 45—SIBBITT & McLEAN.

Another process using vertical retorts is that of Sibbitt & McLean. In addition to distilling the wood, an apparatus is added to distil the tar.

In the illustration, Fig. 45, A is a retort in which the wood is distilled. The furnace construction is of the usual type. At the bottom of the retort are water pipes, h, for cooling the tar, and a strainer b4, to keep the dirt and chips out of the tar pipe.

The operation of the device is as follows: The wood is placed in the retort and heat applied. The vapors pass through i into the creosote chamber j, where the creosote separates. The light vapors pass to the condenser C, and the oil and water formed collect in D. The oil rises to the top of the water and overflows into the refining still, E. Here the usual refining process takes place by distilling with steam coming from the boiler, G. The steam and oil vapor liquify in the condenser, F, and are collected and separated in the usual manner.

The creosote that separated in j is allowed to flow down pipe v into the creosote refining still, K. Here the creosote is distilled by means of fire heat and condensed in the usual manner, the heavy tarry products remaining in the still and being drawn into w when necessary.

The tar flows down pipe t to a series of condensing tanks, I and J, from which the tar can be run into the tar still, H. Here the light oils are driven off. Any water remaining in the tar can be separated and tar sent to the still, M, for further treatment. Here the tar itself is distilled, light oil of tar vaporizing while the pitch and heavy oil remain in the still from whence they can be withdrawn.

The operation of the process can be understood from the description of the more simple ones already given. This treatment of the tar would be found to be very unsatisfactory.

One hears so much about European methods of distilling resinous woods, that the following Russian method is described to show that American processes are as good as European.

The illustration, Fig. 46, shows Friis's process. The retort is made of iron, with a V-shaped bottom, surrounded with a brick chamber which contains the flue gases. The grates are at c at both ends and the fire gases are made to travel around the top and sides by means of partitions. To help heat the interior of the retort, flues d pass through the retort at several places.

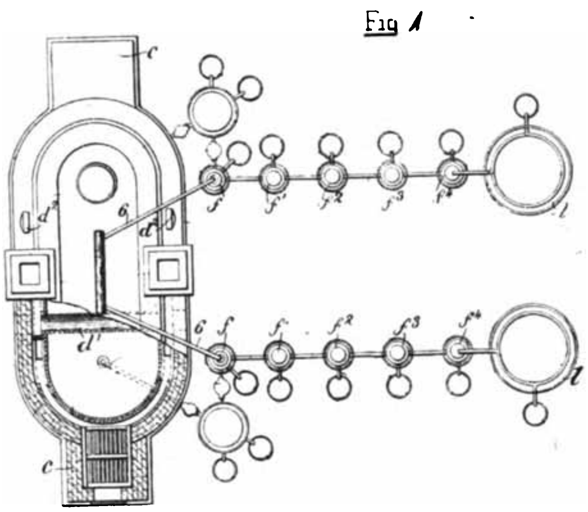


FIG. 46—FRIIS PROCESS.

The tar and resin formed flow through pipes at the bottom, while the light oils pass through pipe 6 to a series of box condensers, f, f1, etc., surrounded by water, the very light vapors being condensed by the worm condenser l. The liquid collected in the various box condensers is drawn into suitable tanks for storage. The charcoal is drawn out through suitable openings at the bottom of the retort. The operation of this process is readily understood by pine wood distillers.

In Ross & Edwards' process is found a combination of the old principles of the Swedish oven previously illustrated, Fig. 8, and the more modern ideas of fractional condensation of the vapors to remove the creosote.

In the illustration, Fig. 47, the retort is represented at 1. The wood is set on the grate, 4, and ignited and the carbonization carried on by internal combustion the same as with an ordinary charcoal kiln. The vapors escape through pipe 6 Fig. 2, down pipe 7 Fig. 3, where they enter the hydraulic main, 5. This main is sprinkled with water by means of perforated pipe, 8a. This cooling causes the heavy oils to separate while the light vapors pass up pipe 9 through 10 Fig. 2, to chamber 11, where they strike the baffle plate, 13, and pass under and up the condenser pipe, 15. The chamber 11 is used to further purify the vapors. The gas formed passes out of pipe 19. The tar is drawn off at 25 and the charcoal removed at 3. With a kiln this latter would probably be of poor quality.

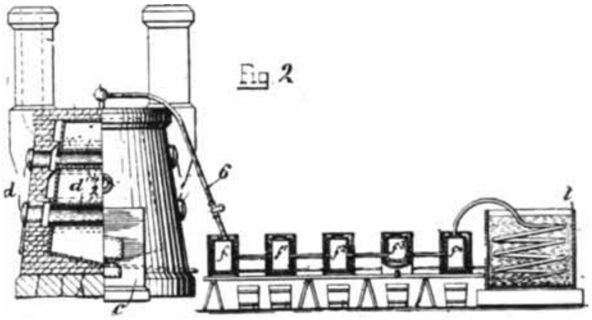


FIG. 46—FRIIS PROCESS.

The writer would suggest that if anyone wishes to use a kiln heated internally that instead of using fat wood for fuel, which is actually done in the above process, it would be better to place a similar kiln to one side of the one holding the fat wood. By placing cheap, low yielding wood in this kiln and connecting it with the one containing the fat wood the hot gases formed by burning the wood in the first kiln could be led through the wood in the second kiln, thus causing this wood to distill without much loss of valuable products. Of course, this increases the cost of a plant, but the saving in yield of products would warrant it. One great objection to the kiln form is that both the fire gases and vapors from the wood must pass through the condenser, which must necessarily be made larger and require more cooling water. When heated from the outside the fire gases go up the stack.

As a refining method this process can be compared easily with the others previously considered. The general principles are the same and are carried out in a similar manner. None of these retort refining processes, except that of Clark and Harris, seem to take into consideration that when the oil first begins to distill the creosote separators being cold, condense considerable turpentine which usually finds its way to the creosote tank.

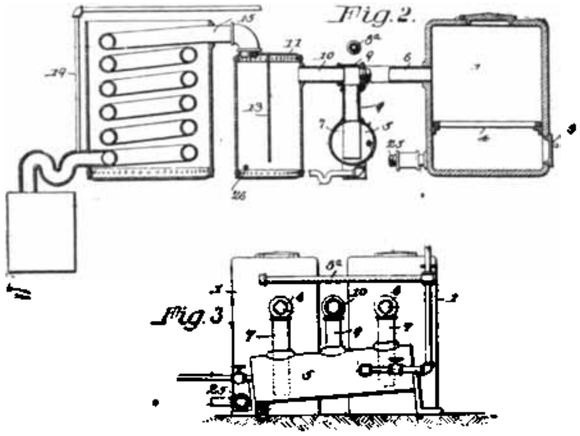


FIG. 47—ROSS & EDWARDS PROCESS.

Probably the best of the vertical retort processes is that of Mathieu, Fig. 48. It was devised by a man of considerable experience in the industry in this country and in France. A resemblance is noted to the French process shown at Fig. 10, Chapter IV.

In this method a process is used of quickly withdrawing the charcoal without waiting for the furnaces to cool down. At AA, Fig. 48, is represented a series of retorts made of fire clay or iron. Covering the top of the retort is the head, B.

The operation is simple. The basket D is filled with wood and carried over the retort in the manner shown and lowered into the retort. The cover being placed on, the distillation is proceeded with in the ordinary manner, the light oils passing out at b and the tar flowing out at b2.

The illustration shows how, after the wood is charred, the basket containing the charcoal is drawn up into the cooler, J, where the air is excluded. This operation with one cord retorts occupies less than five minutes. The cooler and its contents are then placed to one side after the bottom cover has been put on to exclude the air, and a spray of water from pipe P allowed to flow over the surface of the cooler, escaping at the overflow pipe m at the bottom.

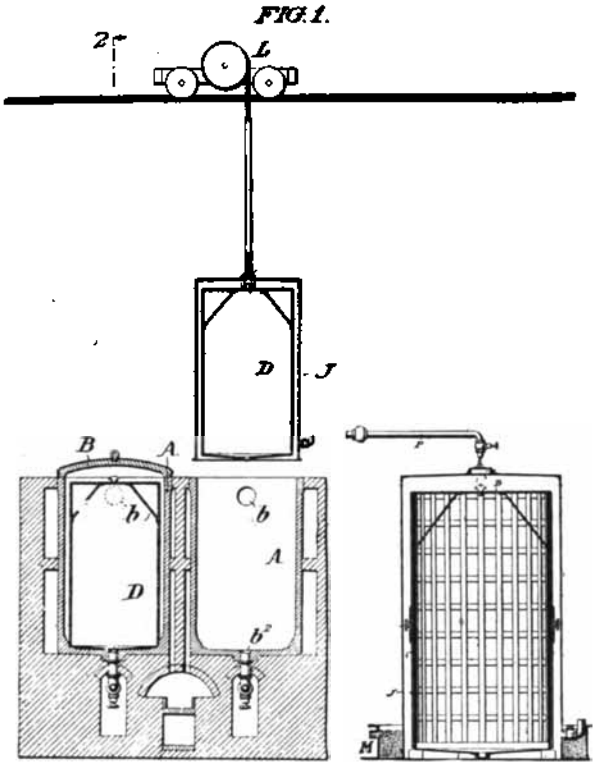


FIG. 48—MATHIEU'S PROCESS.

This process is readily understood and is comparable with those processes using cars in a horizontal retort. Both processes offer equal advantages. The vertical retort ought to make better tar, while the horizontal retort ought to be more easily heated. A horizontal retort has the advantage that its sections are not entirely dependent upon the rivets to hold them in position, whereas in a vertical retort when hot there is a tendency to sheer off the rivets, owing to the weight of the superimposed sections.

A process used on the Pacific coast to a limited extent is that of Jewett, Fig. 49. The principle is very similar to Mathieu's, but is more complicated. It consists of a vertical retort, 16, protected at the bottom with fire brick, 9, and a metallic shield, 13, surrounding the upper portions. A basket is used as in Mathieu's process, but it is a complicated affair arranged in sections so as to stand the wood on end. Under each section is a solid sheet iron pan, 28, to receive the products of the distillation, and to direct them to a hole in the center of the pan, through which they fall to the bottom of the retort. Through this circular space, which extends through the middle of the basket, a water pipe is led branching below each section as at 30-30. These branches are perforated and serve as a means of quenching the charcoal after the distillation is finished. All the products of the distillation go out at the bottom and are led to condensers and tanks as required.

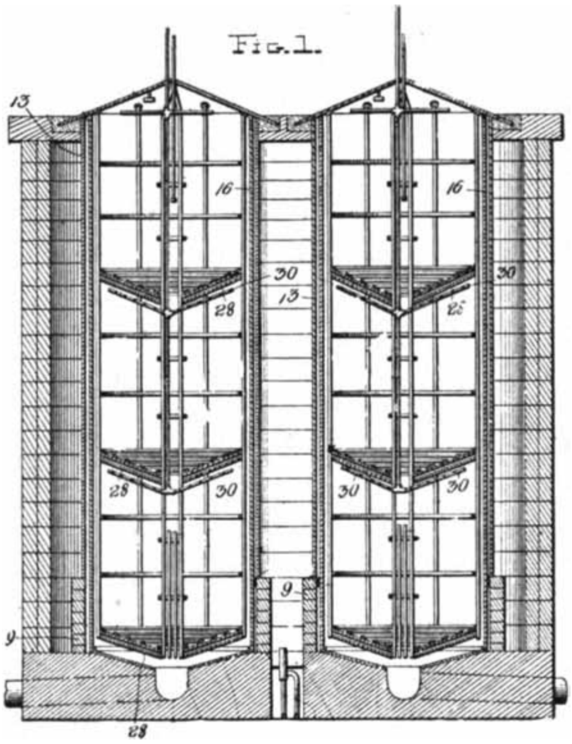


FIG. 49—JEWETT PROCESS.

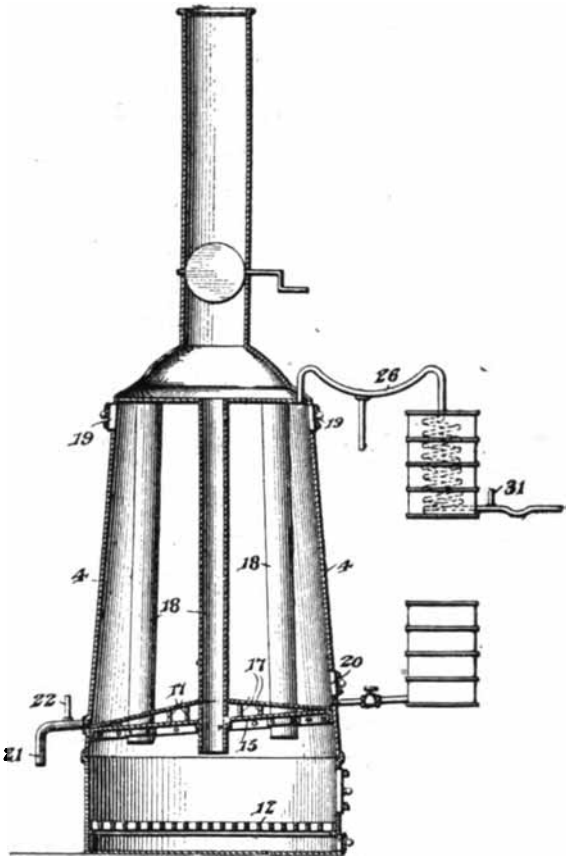


FIG. 50—FIVEASH PROCESS.

This process, although patented a couple of years after Mathieu's, is not as good. It is bad practice to wet charcoal, as it causes it to powder easily. In practice it will probably be found that the metallic shield will warp and burn out and probably change the retort so much as to prevent the entrance of the basket. Then the basket itself is not as good as the simple one used in Mathieu's process. This latter form answers all the requirements necessary, and with the cooler forms a very satisfactory combination. The process offers an advantage in that it takes off all the products of distillation from the bottom, thus preventing in a measure any possible overheating.

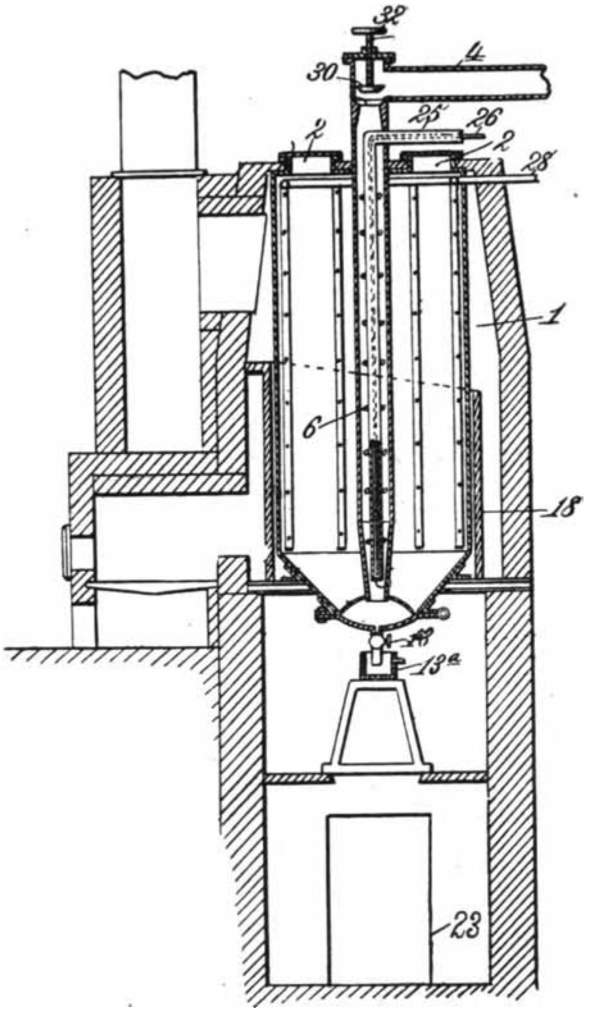


FIG. 51—WILLIAMS PROCESS.

In the Fiveash process, Fig. 50, an attempt is again made of heating the contents of the retort by means of flues. The writer does not believe that this apparatus is in use, but it may be.

It is a self-contained form made out of steel. The retort is at 4 and is heated by burning fuel placed on the grates, 12. The products of combustion pass upward through the flues, 18. To cool the bottom, the chamber, 15, is filled with water, the steam formed passing through 17 into the retort. At the top of the retort is a charging door, 19, for the wood and a discharging door, 20, for the charcoal. The volatile matters pass out through 26 to the condenser, the tar flows out at the bottom at 21, while the gas escapes through 22 and 31.

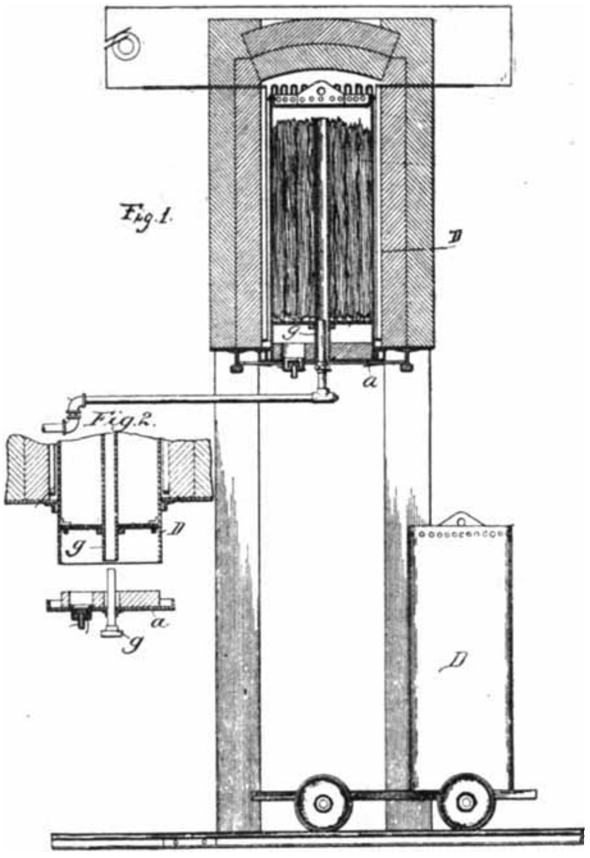


FIG. 52—SNYDER'S PROCESS.

Apparatus of this kind has been tried since Reichenbach's time, but for some reason does not give the satisfaction that it seems it ought. The action of dry heat on flues might be compared to the heating of boiler flues when there is no water in the boiler. In this process one end is protected to some extent by the water at the lower joints, but not at the top. It is difficult, also, to draw the charcoal and fill with wood. If it were not for the bad effect of the heat on the flues this idea of heating the middle of the retort would be a good one.

Another Pacific coast process is that of Williams, Fig. 51. Steam under pressure is used to carry off the light oils, and the residue distilled by fire heat.

In the illustration 1 is a steel retort set vertically in a furnace. The bottom is protected by a shield, 18. At the bottom of the retort is a door for removing the charcoal.

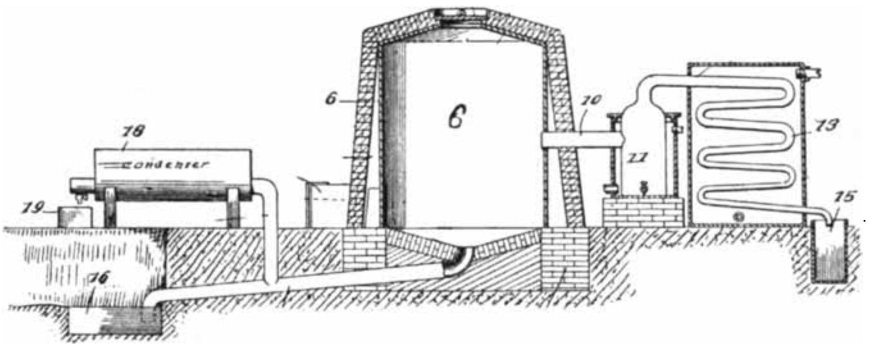


FIG. 53—COPILOVICH.

The operation of the process is simple. The wood in short lengths is dropped through openings 2, 2, at the top, and the openings covered. Steam is turned in through pipe 28 until the desired pressure is reached and the excess steam allowed to escape through valve 32 at the top. The steam carrying with it the oil vapors passes through the perforations at 6 before it reaches the exit valve. After passing the valve the vapors follow pipe 4 and are condensed in the usual manner. Any resin formed is drawn off from time to time through the bottom valve, 13. After the light oils are distilled the steam is turned off and the wood heated by means of the fire in the furnace. The vapor valve 30 is opened wide and the creosote and tar oil vapors pass through, while the tar is drawn off into a receptacle, 13a, at the bottom. To regulate the temperature means are provided for admitting cooling water through pipe 26, the hot water returning through pipe 25. By opening the door the charcoal is allowed to fall into cooler, 23, which is covered over to exclude the air.

This process seems to have been well devised, although the necessity of so many perforated steam and vapor pipes is not so apparent. The arrangement for getting at the bottom of the retort is especially to be commended, as the tar often blocks the pipes. The wood, though, must not be allowed to get down too far or it will not thoroughly char. In taking the charcoal out from a large retort it makes it necessary to place the retort and furnaces rather high in order to allow room for the cooler underneath.

Another process using vertical retorts supplying means for withdrawing the tar without heating the bottom is that of Snyder, Fig. 52.

The wood is put in a container, D, enclosed in a brick furnace. This furnace is heated by electricity or other means, no heat being applied to the bottom of the retort. The container, or retort, D, has a perforated bottom so that when the bottom of the furnace, a, is closed the vapors can pass through pipe, g, to a condenser. The charcoal is removed by lowering D on to a suitable car without waiting for the furnace to cool down.

This method doesn't show any advantages over

Williams' process, as in the latter all the vapors can escape at the bottom if desired. The method of raising and lowering seems to be much more awkward than putting it in at the top. As to the effectiveness of electric heaters definite statements will not be made here. They are rather expensive in other lines of industry, and it is to be expected that such would be the case here. However, as with all the different pine distilling processes, great claims are made. The statement is made that a current of a thousand amperes having a potential of two hundred volts gives satisfactory results for a retort holding one-half a cord of wood. Of course, with larger retorts heated from the outside, it would take relatively more.

Another process devised to allow the tar to escape without undue heating is that of Copilovich, exploited for use with Norway pine in Minnesota, Fig. 53.

The retort 6 is set in a brick furnace. The wood is put in at the top and a fire started in the furnace. The light vapors pass through 10 to a creosote condenser, 11, and the uncondensed vapors pass to the condenser 13 and thence to the receiver, 15. The tar and heavy vapors pass out at the bottom, the tar going to the tank, 16, and the vapors and gases to condenser, 18. No arrangement seems to be made for the separation of the gas, so this would come out with the vapors and enter tanks, 15 and 19, and would be apt to explode if brought in contact with a flame. The charcoal is withdrawn through a door at the bottom on the side.

A Swedish process using such a form of retort was experimented with for a time in Mississippi, but for some reason a larger plant was not built.

Denny's process provides a vertical retort protected on the sides with sheet asbestos and on the bottom with a double bottom containing sand.

In the illustration, Fig. 54, A represents the retort fitted with the usual exits for vapors. The wood enters through the top door, 5, and the light vapors pass out through pipe 21 to a suitable condenser and the heavy vapors and tar flow out through the pipe, 20.

The furnace is heated with wood or other fuel and the flames strike against the asbestos lagging, 5-1, which protects the retort from blistering. The flues are so arranged that the flames are deflected from a straight course by means of a baffle, 11, thus causing the retort to be heated more effectively. To protect the bottom sand is placed in the space, 14, between plates 12 and 13. The charcoal produced is drawn out at 18.

There are doubtless many patents now being applied for that will be issued shortly. Probably all of them will resemble more or less closely some of those herein described, hence readily understood.

It will be seen that some of the essential requirements of processes of these kinds must be, first, a good furnace constructed to obtain the greatest heat from the smallest amount of fuel; second, proper distribution of the flame so as not to burn the retort at one place; third, proper position of the retort so that all the wood contained therein can be thoroughly charged; fourth, accessibility of parts for repairs; fifth, good arrangements for drawing off the tar without choking the pipe or burning the tar; sixth, rapid removal of the charcoal produced so as to save the heat of the furnace; seventh, rapid charging of the retort and quick distilling methods for obtaining good products, and, above all, eighth—an absolutely essen-

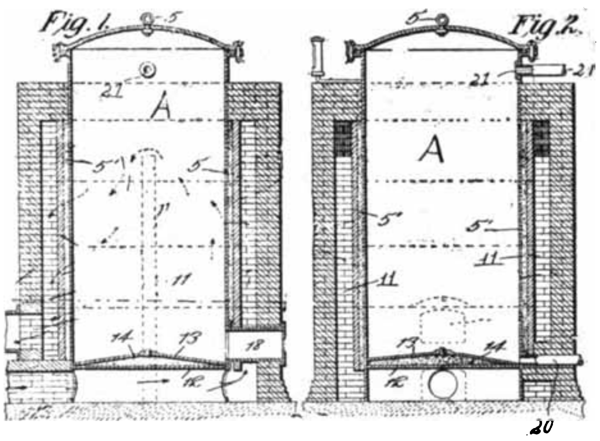


FIG. 54—DENNY'S PROCESS.

FIG. 55A—THE KRUG STEAM PROCESS—SHOWING RETORTS.

FIG. 55B—THE KRUG STEAM PROCESS, SHOWING CONDENSERS tial condition—the production of salable products.

An illustration of a steam process in actual operation is shown at Fig. 55. The retorts are enclosed in brickwork so that the residue could be distilled if required. At Fig. 56 is shown a steam and destructive distillation plant, using cars in large retorts.

Any form of retort, if it be nothing but a piece of water or gas pipe closed at both ends with an exit for vapors, will make oil from resinous woods, when heated in the proper manner. This is the reason that there are so many processes. Some form of retort is designed and erected and distilled products are obtained in quantity, and the problem is supposed to be solved. But some of these processes must be more economical in operation than others, and an attempt is expected to be made by the Forest Service of the relative value of each, and also which if any are sufficiently economical. When proper tests are applied a great many of the processes now being exploited will disappear, and the sooner the better for interested parties who might wish to invest.

Special Retorts and Processes.

Some of the processes which are to be described under the above heading might have been described under steam or destructive distillation processes, but certain features connected with them make it advisable to consider them separate-

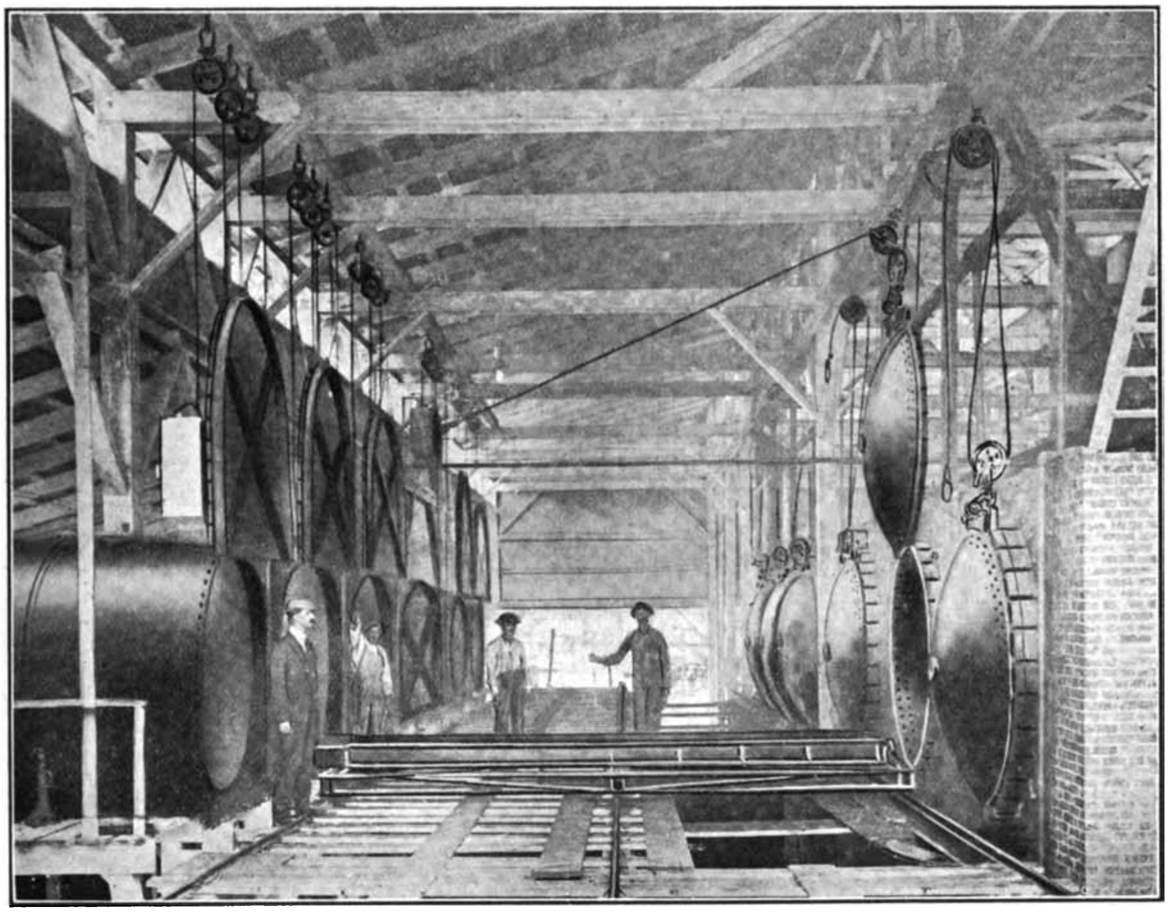


FIG. 56—STEAM AND DESTRUCTIVE PROCESS, USING COOLERS AND CARS.

ly. This is especially true of rotary and portable apparatus.

Rotary Processes.—As has been stated, wood, being a non-conductor of heat, it is difficult to heat the middle of an ordinary retort to the same degree of temperature as the shell. This feature is more apparent when the wood is in a finely divided state such as saw dust. Consequently when the turpentine is extracted in the steam process, the residue cannot be destructively distilled in an ordinary retort, as it seems to be extremely difficult to heat the saw dust sufficiently to distill it, except at the edges of the retort.

With hardwood saw dust, attempts were made to distill it with superheated steam, but it took so much steam to carbonize that the condensed water diluted the distillate of pyroligneous acid and wood alcohol so much that the extra evaporation necessary to make acetate, made the process unprofitable. Steam has the advantage over direct heat as applied to a large closed retort, in that it can be applied to the inside while direct heat is usually limited to the outside, except in a few cases. The feature of diluting the pyroligneous acid, which is noticed with the distillation of hardwood, with steam is not objectionable when distilling pine wood, as this portion of the distillate is usually not saved anyway, and most of the oils and the tar are nearly insoluble in water and can thus be separated by gravity. Large quantities of steam, though, are costly.

A stationary retort ought to cost less than a rotary one and when working for turpentine alone, unless the difference in yield in a given time would pay for the difference in the initial cost, the rotary retort will find no practical use. In the case of steaming pine wood for turpentine, it is claimed by those interested that as much as 25 per cent more turpentine can be obtained in a given time, and if such proves to be the case, rotary retorts will probably supersede the stationary ones. At the present time stationary ones have the field, although one or two rotary retorts are in operation and giving, as is claimed, better satisfaction.

In destructive distillation, the rotary retort is one of the most feasible ways of distilling saw dust, either hardwood or pine, if such be necessary. With the latter not much would be gained, as pine saw dust makes very poor tar and charcoal.

By using rotary retorts in a proper manner, the author believes a better utilization of waste wood can be made, particularly of medium fat knots such as cannot be used in the ordinary steam process on account of the cost of gathering being more than the amount received for the turpentine produced. This is one case where a destructive process may be better than steam.

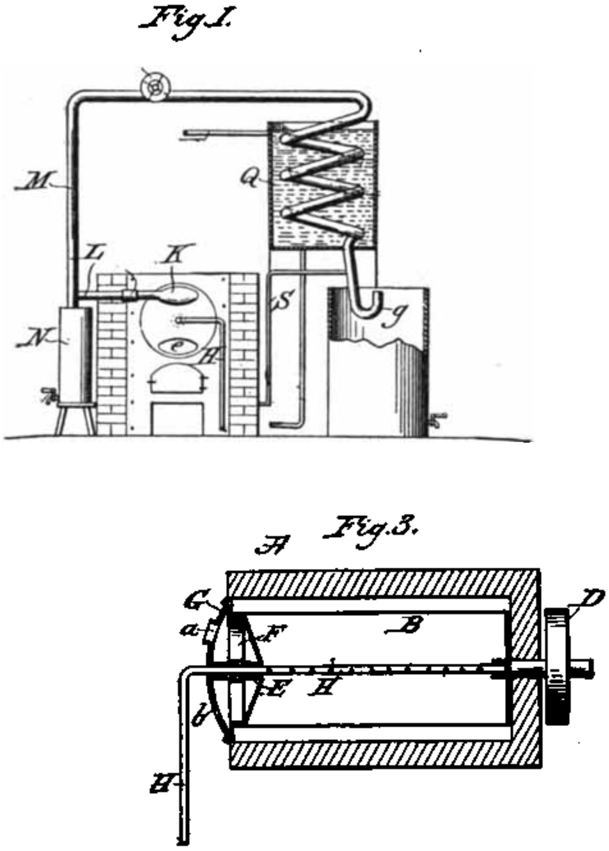


FIG. 57—BERRYS' PROCESS.

As grinding the wood caused such a great saving of time in the steam process, so it may be expected that a great saving of time will be occasioned when ground wood is destructively distilled, provided the heat for this purpose can be as effectively applied as it is by using steam in the steam process. This is the result expected with the special saw dust distilling apparatus herein described, especially the rotary retorts. All manner of forms of rotary retorts have been designed so as to cover all possible modifications, so that although many patents will be applied for when the success of this form of apparatus is demonstrated, only a few of them can possibly be valid.

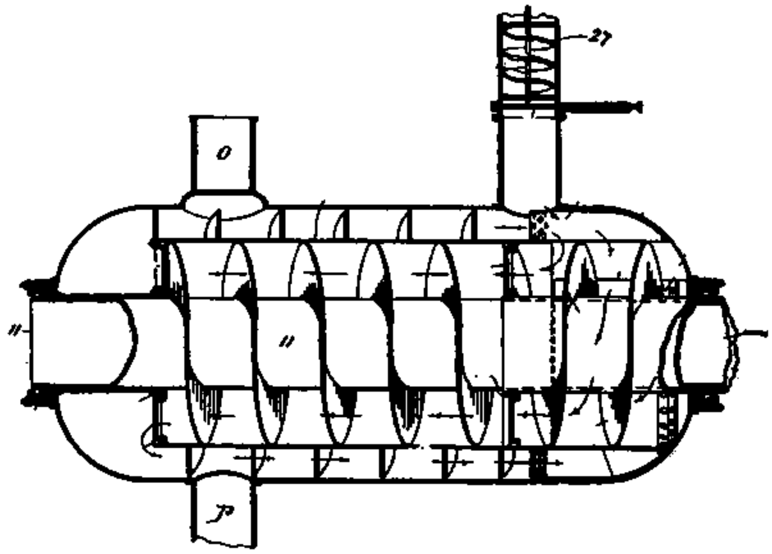


FIG. 58—SPURRIER'S PROCESS.
27—Feed screw.
11—Flue.
O—Exit for vapors.
P—Exit for charcoal.

The rotary retort is supposed to be the product of the past few years, but it will be noticed by what follows that several attempts have been made in this country as well as in Europe to use such in distilling hardwood saw dust.

The first patent noticed by the author is that of Berry, Figure 57. This contrivance was devised to utilize shells of cocoanuts and seeds, etc., and for wood. The exact arrangement needs description, as the illustration is not so easily understood. Observing Fig. 1, we have apparently an ordinary retort with an arrangement at K for leading off the vapors to pipe L, the tar formed dropping to N and the light oils passing up M to the condenser Q. A trap at g holds back the gas which escapes through S to the furnace. The retort is heated in the ordinary manner as shown. In Fig. 3 is shown a cross section. Here the retort is represented at B; and at one end it is connected with a shaft leading to the pulley D. The other end is supported by the head G from which a tubular shaft extends into the retort. Turning on the end of this shaft is the spider frame F connected with the retort. The head G being stationary, it is advisable to make a good joint between the retort and head, otherwise air might enter or gas escape according to the pressure. An exhaust is usually used, consequently any gas drawn in would probably be fire gases, containing little oxygen, so little damage would be done if the head is not absolutely tight. A stationary perforated pipe H is added in order to quench with steam or water the charcoal formed.

It will be noticed that K is removable and is placed over a and that e is also removable and is placed over the opening b. The opening a is for filling the retort and b for withdrawing charcoal. The regular operation is the same as for a stationary retort, except that the retort is slowly turned, thus causing each particle to be evenly heated.

A more elaborate retort is used in Spurrier's process, Fig. 58, for distilling hardwood sawdust. This consists of two helices, one working inside of the other so as to send the sawdust in opposite directions. The retort is heated by fire gases led by suitable flues around the retort with a large flue leading through the middle of the retort. Although such a retort might be suitable in distilling turpentine from pine saw dust, it is much too expensive in comparison with other processes. Another process using rotary retorts is that of Larsen, also used on saw dust. Owing to the ease with which a slight modification may be made on an invention and a patent obtained, the inventor of this process shows six different modifications of the same principle. Considerable ingenuity is shown in each modification and a complete understanding of the conditions of distilling are implied. Two different forms are indicated; one in which the retorts is encased in brick work and the other a self-contained form. Only the two types will be described. Fig. 1 in Fig. 59 represents the retort set in masonry. The retort is at A and rotates on a a.

The fire gases starting from the grate pass to

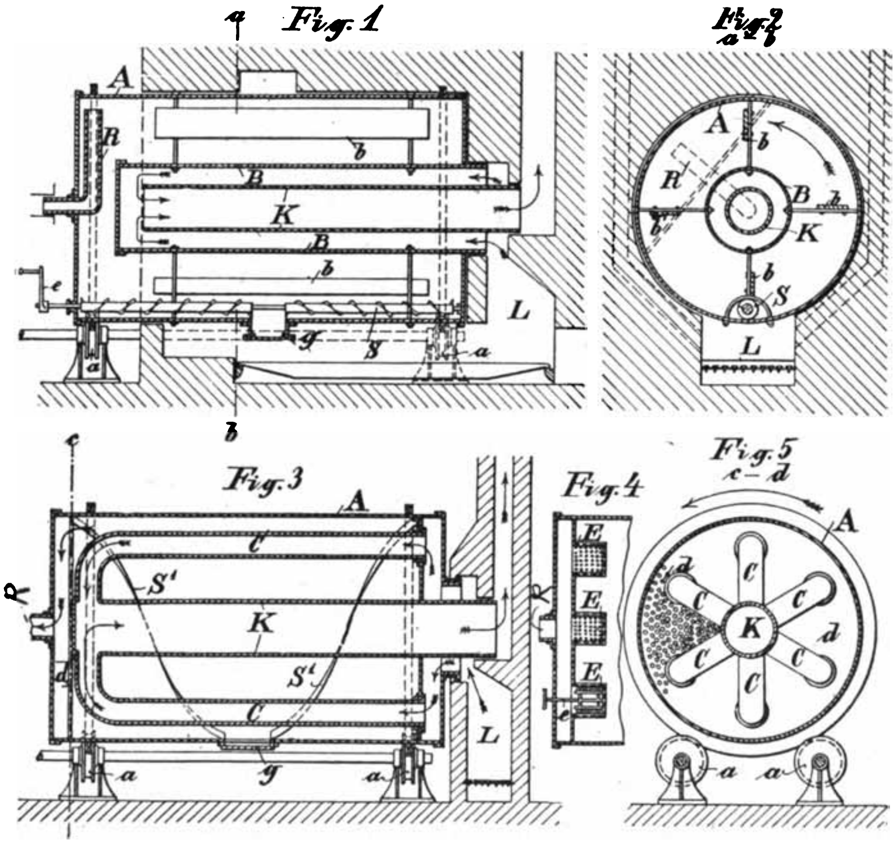


FIG 59—LARSEN'S PROCESS.

the chamber L, thence follow the course of the arrows through B, then back through K to the chimney. The distilled vapors escape through R, which is turned up so as to be above the material in the retort. The material is dropped through the brick work above into manhole g (shown at the bottom) and when charred is drawn out at the bottom through the manhole g by the action of the right and left hand screw conveyer S, operated from the outside at e. The vanes b rotate with the retort and help spread the material.

The other form shown at Fig. 3, 4 and 5, Fig. 59, is self-contained. The one above could be made so also by leaving out the surrounding brick. The action of this is similar in most respects to the other. The fire gases instead of being in two pipes, one surrounding the other, are in numerous pipes. The flame as it arises from the grate is prevented from entering K, so it goes through the flues C to a central chamber at the back, from which it is drawn through pipe K to the chimney. The vapors pass through screens set in the back plate (shown in detail at Fig. 4) and thence through pipe R. To keep the screens clean a wire brush e Fig. 4 is used and worked from the outside when necessary.

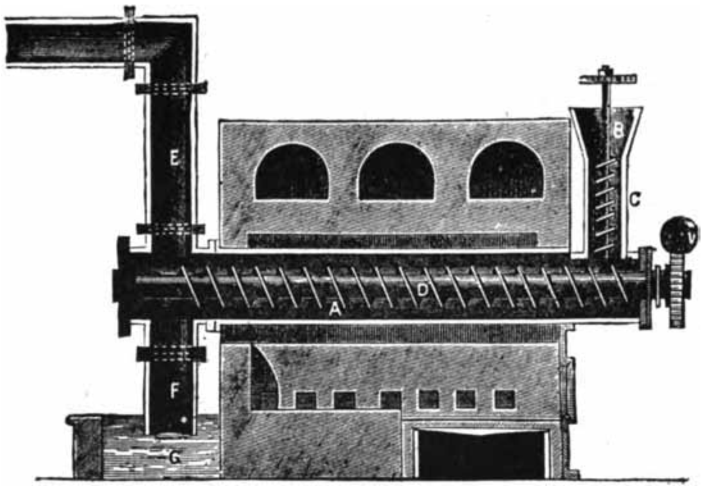


FIG. 60—HALLIDAY'S APPARATUS.

These devices are well designed to overcome the difficulty of heating the retort. As arranged, the whole retort revolves, flues and all.

One feature of machine design is the accessibility of parts for repairs. Unless the above retorts are very large, one who is acquainted with the destructive action of heat when wood is distilled can readily understand that great trouble might arise from the use of such retorts. Another difficulty would probably be the warping and bending of the flues under the effect of the heat. A further disadvantage is that in destructive distillation, every rivet used is apt to give trouble. Instead of there being fewer in this apparatus, there seems to be need of many. If, in the working of the apparatus, these difficulties do not manifest themselves, we have in these processes, perhaps, one of the best for the destructive distillation of saw dust and the like.

An apparatus much used in England and Europe for distilling hardwood saw dust is the device constructed by Halliday.

According to the description of it given by Hubbard in his "Distillation of Waste Wood," this process is a continuous one, and consists of a cylinder with feeding screw; according to the speed with which the screw is driven, the wood can be exposed for a longer or shorter time to the action of the heat, and thus a larger yield of acetic acid is obtained than is possible from the charcoal mounds. The fact is, however, not to be ascribed to an especially favorable construction of the apparatus, but exclusively to the form of the raw material. From small fragments of wood, the distillate is much more rapidly evolved than from large billets, and the distillate undergoes much less decomposition in the apparatus. The saw dust is thrown in the hopper B (Fig. 60).

In this hopper a revolving screw C delivers the material at an appropriate rate into a horizontal cylinder. The latter is heated by the furnace A. A second screw D keeps the material in the retort in constant motion, and at the same time conveys it gradually to the other end of the cylinder. The wood becomes carbonized as it traverses the cylinder so that by the time it reaches the further end it has parted with its volatile products. Two tubes are connected with this end of the cylinder. One of these, F, descends into an air-tight closed cast-iron receiver, or else into a cistern, G, filled with water; the other, E, carries off the products of distillation to the condenser, which consists of tubes surrounded with water. It can be readily seen that this apparatus with a perforated shaft for steam could be easily used for distilling pine. This apparatus has been in use for a long time.

In Viola's apparatus an arrangement is made to rotate the outer shell of the retort in addition to the use of a screw conveyor.

In the illustration Fig. 61 is shown one form

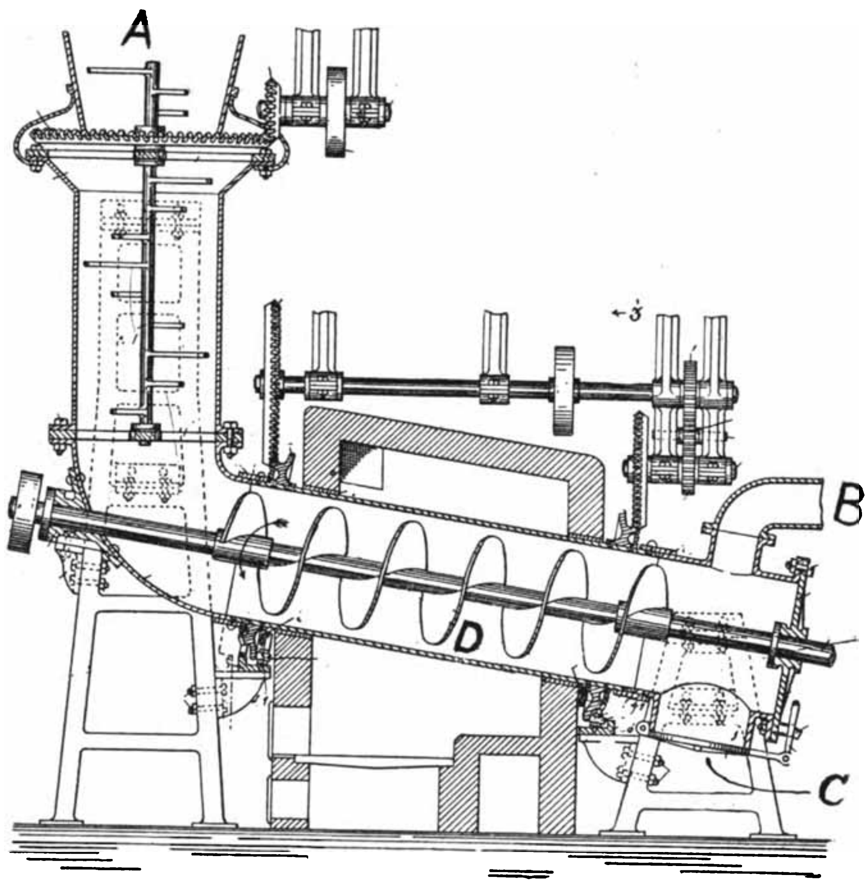


FIG. 61—VIOLA PROCESS.

of this apparatus. Its method of operation is readily understood on account of its similarity to the Halliday apparatus just described. The raw material enters at A, which portion of apparatus is provided with a stirrer to keep the material from packing. As the wood drops to chamber D it is moved along by the screw conveyor to the bottom end, by which time it should be distilled. The retort shell of this part is rotated in order to help stir the material. The vapors pass out through B to a suitable condenser, while the charcoal is drawn out from time to time by opening the cover C. This apparatus is continuous.

Another form is used intermittently. The feed end is omitted and also the screw conveyor. One end is furnished with a door for the admission of the wood, which is held in position by a frame, while it is being distilled. The vapors escape through B, as is shown. Charcoal is pulled by means of the frames to C. This opening is arranged to dip under water like in the Halliday apparatus. This process seems to be very complicated and does not appear to be as efficient as the Halliday apparatus, which is far more simple.

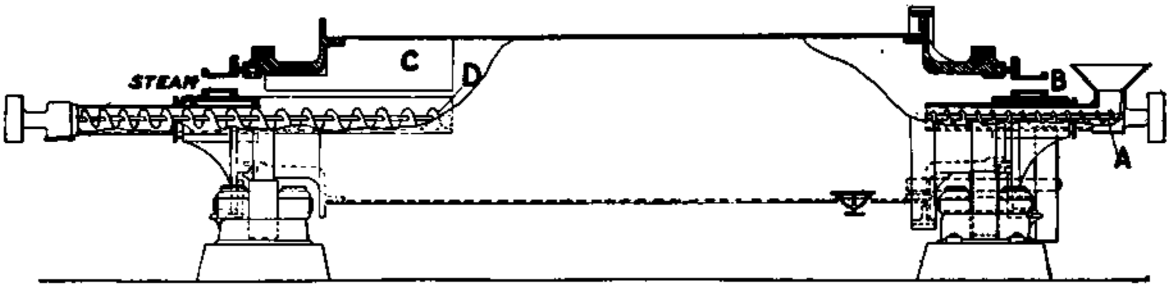


FIG. 62.—HARPER'S PROCESS.

A steam process continuous in action connected with the saw mill would be very convenient if it worked automatically and at the same rate as the saw dust, etc., was supplied. This suggested in the year 1900 the Harper process, one modification of which is shown in Fig. 62. Many other modifications can be made, such as inclining the retort, extending the heads, changing the location of the rotating parts, etc., all of which have been considered in connection with the process as carried out. The illustration shows the form which is operated under slight pressure, no pressure or vacuum, according to the nature of the substances to be distilled. It can be readily seen that such an apparatus would be of service in other lines of chemical industry, where distillation, roasting or drying processes are used.

In the distillation of wood, it could be surrounded by a brick furnace and saw dust and hogged wood distilled by its means. Steam can be admitted to the conveyor shaft as well as at the place shown. In distilling rich wood an arrangement is added which takes off the resin formed. In distilling under pressure the screw conveyors are so arranged that no steam can blow through while feeding and the rotating part properly packed.

The retort as used in accordance with illustration is filled partly full by the feed screw A and the steam either saturated or superheated turned in. The material moves along the bottom of the retort on account of the settling action of the mass. The steam being under low pressure cannot escape through the conveyor on account of the mass of material in the feed box and conveyor trough. A ready outlet for the vapors is furnished at B, preferably screened to prevent saw dust from blowing through the pipe.

The heads of the retort are stationary and only the shell and the lifting vanes C rotate. A steel tire is provided for the retort at both ends so that it will stand the wear of rotating. No weight is allowed on the driving machinery, the entire load being carried by the rolls.

By the time the material reaches the rear end, all the volatile matter is distilled and the vanes catching the material lift it and deposit it upon the trough of the screw conveyor B, and thus it is carried out and dropped on to a conveyor leading to the furnaces.

At those plants where turpentine and tar are mixed and then separated one of these retorts would answer the purpose. For those who prefer to distill the turpentine separate from the tar, two would be necessary, one for the turpentine and one to destructively distill for the tar. As used above, the apparatus would be used for saw dust, which would not pay to distill destructively, so only the turpentine would be taken off. In this case the shell should be covered with suitable lagging.

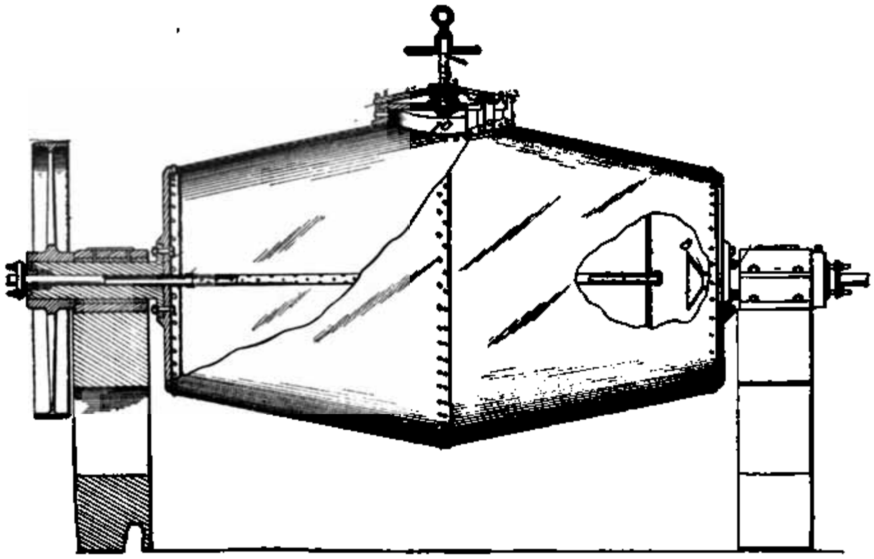


FIG. 63—FLEMING'S PROCESS.

The form of screw conveyor shown in these forms of apparatus is not very suitable for moving saw dust, but other forms can be used which are suitable. With very fat wood the above apparatus is supplied with a special chain for scraping the bottom.

For destructive distillation in such a retort instead of heating the shell, a self-contained form can be used by putting in flues similar to those used in the Larsen process or the wood can be charred in the retort, as illustrated, by means of superheated steam, thus requiring no brick furnace.

If required, this apparatus can be used stationary or intermittently. In the latter case, after the retort was charged all that would be necessary would be to stop the feed and discharge screws until the distillation was ended.

By using hot gases containing no oxygen the oils can be extracted and the gases passed through a reheater and used over again. Gas takes up heat and loses heat rather slowly. Hardwood saw dust can be distilled with this apparatus.

All rotary and continuous processes are more expensive to construct, cannot be worked well on variable material, and are difficult to make tight under heavy pressure. However, much pressure is not now used. They have the advantage of being made in any length when supported properly and can thus be made to hold 100 tons or more, the size being determined by the rate of distillation.

The next is Fleming's process, shown in Fig. 63. Not much need be said concerning this process, as it is so simple in construction that it is apparent from the illustration.

It consists of a rotary retort working intermittently, the ground wood being fed in the manhole 10 at the top and discharged at the bottom through the same orifice, when the retort is turned.

To operate, steam is turned on through the perforated pipe shown and the steam and turpentine vapors pass through the funnel-shaped mouth of the pipe 8 to the condenser, the retort being slowly turned during the operation.

Another form of retort constructed on exactly the same principles as that above is that of Coe. It consists of a large steel ball, instead of being elongated as in Fleming's process. The supports, manhead, steam and vapor pipes, etc., are adjusted in a similar manner to Fleming's.

A sphere will stand pressure better than a cylinder and would be more rigid, but the elongated form would be heated to better advantage.

One feature noticeable about the process is that Coe claims a yield of 25 per cent more by his process than by the stationary steam process, and attributes it to the stirring of the material in the retort and the breaking up of the ground wood as it rubs together while rotating.

It can be seen that either of those forms would not cost much more than a stationary one and a 25 per cent extra yield would more than pay the initial cost. They must be made thicker than the others in order to stand the strain of suspension and the weight of the wood.

A later process than Fleming's is that of Jackson, Fig. 64. As can be readily seen the difference lies chiefly in turning the retort around so that it rotates from the sides instead of the ends. This makes it necessary to change the arrangement of the steam pipes so as to make them longer.

To operate, the wood is dropped in at B, the head bolted on and steam turned in. The steam supply enters the shaft at G, then leaves again at C and follows a pipe outside the retort and then enters the retort at J 1. The vapors follow a similar course, leaving the retort at a point opposite to J 1 and coming back again to the shaft at D and passing to a condenser. This arrangement of steam pipes and vapor pipes is entirely unnecessary. There is no reason why the steam could not continue in the shaft until it entered

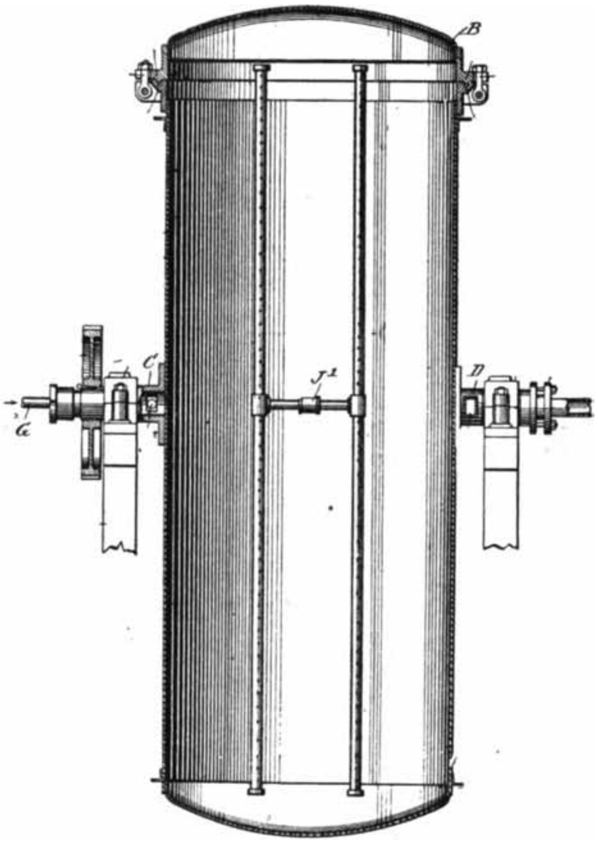


FIG. 64—JACKSON'S PROCESS.

the retort, as in Fleming's process, and then pipe to J 1, if necessary.

A patent should have been granted to but one of these three parties, as the principle of the one is the same as that of the others.

A great many patents have suggested the advisability of using pine wood pulp for paper-making after the turpentine is extracted.

The only one ever tried is that of Handford's, Fig. 65. A plant using this process was built near a paper mill using yellow pine for making pulp by the soda process. It proved unsuccessful for two reasons chiefly; first, because in using the lean wood the operation of extracting the turpentine did not pay for itself, and second, the residue after treatment made a very poor quality of paper.

As will be found later, under Chapter XI, processes for the preliminary extracting of the turpentine from pine wood when making paper pulp are not necessary.

Fig. 65 represents a plan of the process. At A is hog through which the chunks of wood pass to A1, where the chips are torn into shreds by a machine called a fiberizer. The material thus treated falls upon the conveyor H by means of which it is taken to the top of the rotary digester B and drops therein through the manhole K. A stirrer with paddles is on the inside of the digester which takes the material from the entering end K to the opposite end, where underneath in a similar position to K is another manhole which allows the material to be dropped out of the digester. The steam enters and the vapors leave the digester in the same manner as in Fleming's process; the steam going in through the shaft from pipe L at one end and going to the condenser through pipe E at the other end. A closed conveyor is under the digester B and it takes the discharged material above D D and it is permitted to fall and pass through heavy steam heated rolls, which press out the liquid material in the residue; any vapors rising and entering pipe E1 and thence going to the condenser. The material that is discharged from the rolls is then used for paper making.

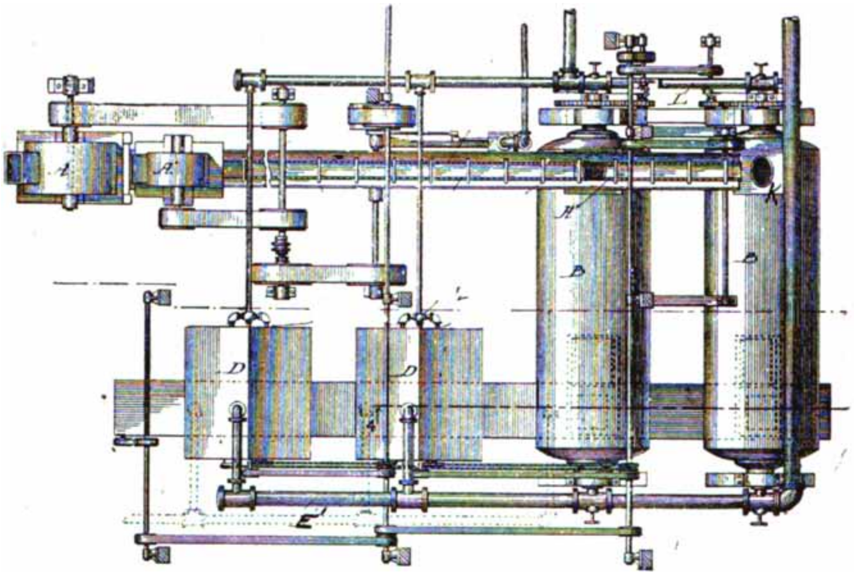


FIG. 65.—HANDFORD'S PROCESS.

On account of the non-success in making paper from the residue an attempt is now being made to manufacture straw board from the pulp. There is no use making turpentine, though, if the apparatus does not extract it cheaply enough; it would be better to make straw board or pulp direct.

Movable Retorts.—Under this head come those retorts that are so made that they can be easily taken down and transported from place to place, thus bringing the retort to the wood rather than the wood to the retort.

The first process to be mentioned is that of Dromart, used in France. Here retorts holding 14 cords are made in sections each section weighing about 110 pounds. This was used way back in 1830. The iron sections were made to fit each other so as to make a dome shaped kiln and the joints luted with clay. There was a chimney at the top with arrangements for regulating the draft. The heat was supplied by the carbonizing of the wood inside, as in an ordinary brick kiln.

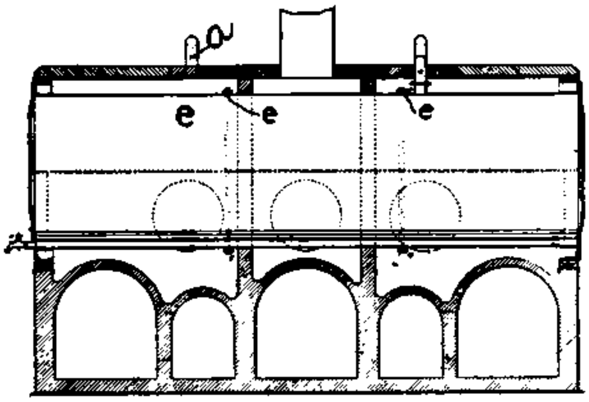


FIG. 66.—SMITH'S PROCESS—Fig. 3.

The only other process of the kind to be discussed is that of Smith. Its object was to provide a suitable portable apparatus for distilling wood and at the same time be used for creosoting lumber. This was devised at the time that wood creosote was used in preference to coal tar creosote in creosoting lumber, and had a bearing more on the production of creosoting oil than on turpentine, although pine wood was used.

The illustration shows the process sufficiently to set forth the idea. In Fig. 3, Fig. 66, is represented a retort set in a furnace for heating. It is divided into three sections, as shown at e e, the vapors escape by a, Fig. 5, to a condenser. The wood is taken in on trucks, as shown, and the distillation proceeded with in the ordinary manner. One retort is used for distilling and the other for creosoting.

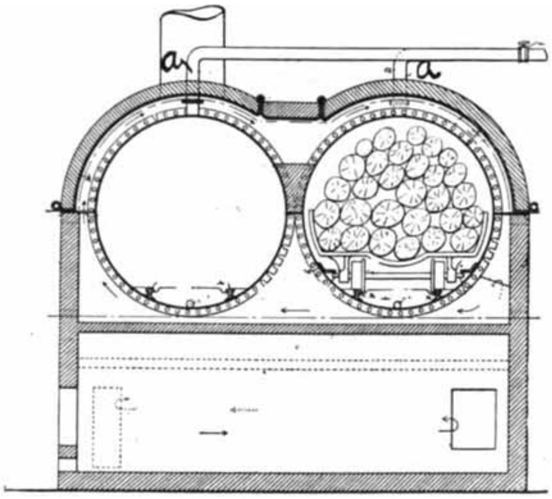


FIG. 66.—SMITH'S PROCESS—Fig. 5.

A later process combining wood distilling and creosoting is that of Davis, Fig. 67.

To operate, wood is placed on trucks on track 3 and the whole rolled into retort B B. The retorts are made of brick or iron surrounded with an envelope of brick or other suitable material, the space between being filled with sand so as to stop up any leaks that might occur in the retorts. Hot rosin or other preserving agent is pumped from the heating vat 6 into the retort, this fluid not being hot enough to decompose the woody fibre, but at the same time being sufficiently hot to distill the turpentine. Instead of applying heat to the retort, the rosin when it becomes cool is pumped through the heater 24 and is reheated, a continuous circulatory system being kept up by means of pump 20.

These retorts are preferably arranged in pairs, as shown and provided with connections so arranged that one retort may be cleaned and recharged while the other is being operated, thus effecting a great saving in time and obviating the need of repumping to and from the main source of supply of the distilling and preserving fluid.

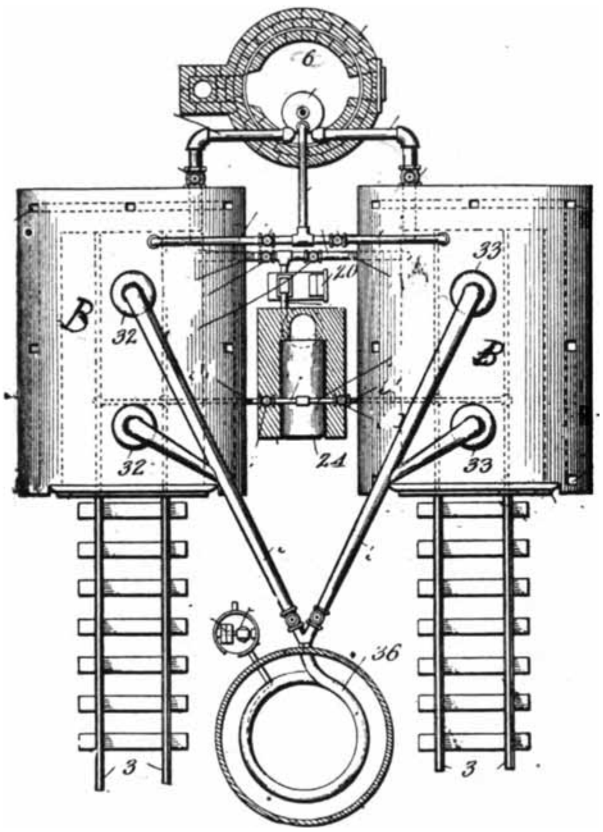


FIG. 67.—DAVIS' PROCESS.

The vapors from the distillation pass out the openings 32, 33, to the condenser 36, where they are condensed and collected.

There are two processes devised by Weed, using a bath of rosin, one for distilling terpenes only and the other for distilling terpenes and also the wood itself. The latest process only is illustrated. Fig. 68 represents the arrangement of the apparatus.

To operate, wood is placed in the retort a on the netting g, situated above the perforated steam pipe d. Rosin is melted in the boiler p and pumped out by pump o directly into the retort. As it cools it is drawn through pipe i to the branch s, then again to the pump o. This time it passes through coil 1, which is heated by furnace m and then into the retort through perforated pipe j. As it issues from these perforations, it comes in contact with the steam coming from pipe d carrying with it the vapors of turpentine which pass out through c to a condenser.

When the distillation is finished, the hot rosin is allowed to flow back through valve z to the boiler p, where it is kept hot for the next charge. Of course, it could be pumped into another retort if desired. The wood is then withdrawn.

In the other form only the retort is used. The rosin and wood are put into the retort and a fire started under the bottom and the heat raised to the required point, the volatilized products passing to a condenser.

Neither form is as good as Davis', although they have been used.

In the Craighill and Kerr process, the wood is treated with a dilute solution of caustic soda in order to hold back the resins and acids, and then steamed in the usual way at a temperature of 110 degrees C. The vapors can be passed through a bone-black filter if desired, before condensing. To remove the rosin, etc., water is added to submerge the wood, steam is applied and the wood is digested at a temperature equal to the boiling point of the alkaline solution with which the mass was saturated and the digestion continued until the rosin has completely entered into combination by saponification with the alkaline solution. Thereupon the solution is drawn off and the wood well drained.

To make fibre, the wood is digested under pressure with caustic soda solution of 1.075 to 1.10 sp.

gr. As this solution attacks the fibre if the heating is prolonged, only a portion of the coloring matter is taken out and the solution withdrawn. A solution of sodium carbonate is then added and the heating continued with or without pressure until more of the coloring matter is extracted, leaving the fibre about the color of light manilla wrapping paper. The fibre is then bleached by chlorinated soda.

This process will need close watching with various kinds of wood on account of the different degrees of fatness. The rosin can be recovered only with difficulty.

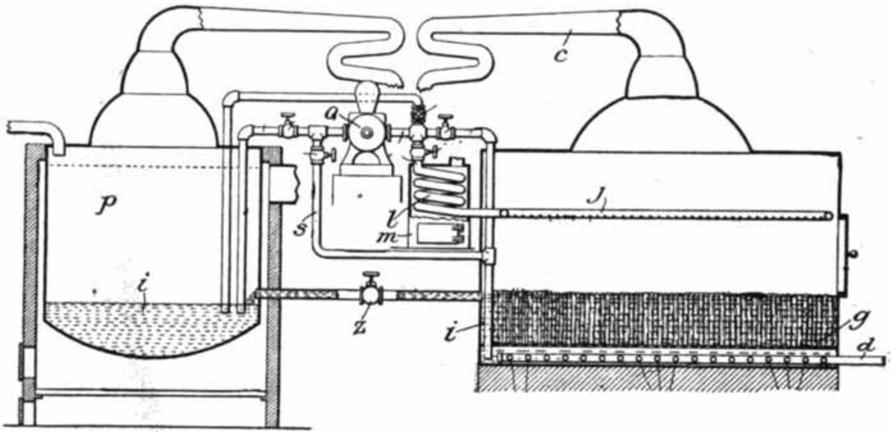


FIG. 68.—WEED'S PROCESS.

Hale & Kursteiner don't use any caustic soda or steam in their process, shown in Fig. 69. This invention is based upon the discovery "that when wood is subjected to the action of a bath of water maintained at a temperature just below the boiling point or approximately 212 degrees Fah., the terebinthinate or gum will separate from the wood and retaining its turpentine or its volatile or more buoyant constituents will rise to the surface of the bath, whence it may be removed or caused to flow over to a suitable still in which it may be subjected to a distilling operation for separation into its constituent parts, turpentine, rosin-oil and rosin. The wood from which the terebinthinate has been so removed may be subjected to destructive distillation in the ordinary way and for ordinary purposes." It is claimed all of the gums, etc., are eliminated from the wood.

The operation of the process is simple. The wood in blocks enters at A3. Water heated to 130 degrees Fah. by means of steam coil B2 is added, and the temperature raised by means of the gas burner C to about 210 degrees or 211 degrees Fah. As the gums exude and float, they overflow through pipe F into the still G, passing through a glass observation bulb F2 on the way. By this contrivance it can be seen whether any gum is coming over with the water. As it is necessary to let water overflow with the gum, this water is drawn off through cocks 1, 2, 3, 4, 5, wherever a layer of it is found. Some water is left in the still with the gum and when the still is sufficiently full it is heated by vapor burner G1 to about 215 degrees Fah., when the turpentine distills over clear and white. After the turpentine and water are distilled, the rosin remaining can be distilled for rosin-oil or drawn off as rosin.

Claims are made that from 128 pounds of rich long-leaf straw pine wood during a period of from three to five hours, one gallon of high-grade turpentine and about twenty-five pounds of rosin of fine quality are obtained. This is about the same yield as from other processes.

Unfortunately, others who have tried the same process several years ago, using very rich pine coming from a yellow pine saw mill, do not seem to be able to get any satisfactory results. This shows that the process requires too much skill in order that the ordinary operator might succeed.

It is possible that in some cases that an extractive process might prove of service under some conditions, particularly when working with very fat wood. To supply this possible want the author has devised an apparatus which consists in chipping the wood to the size required for paper stock or the like and then bringing it into a chamber in a continuous manner, where it is acted upon by a suitable solvent, such as ether, carbon bisulphide, carbon tetra chloride, alcohol, etc., this solvent being continually reused and kept in circulation in a suitable manner; the extracted matter being removed from time to time and the solvent evaporated and condensed. The fibre left is white and soft. The residue left after evaporating the solvent is distilled with steam, which removes the turpentine, the rosin remaining in the still from whence it can be withdrawn hot. The color of the rosin depends upon the color and age of the wood, dead wood giving a light red, which can be bleached, if necessary.

Conveyor Processes.—Under this head will be described those processes which use conveyors of some kind to make the wood pass through a heated zone. The first of these was probably Halliday's, which has been mentioned under rotary retorts. Another retort said to be better than Halliday's is that of Bowers. In this process a long rectangu-

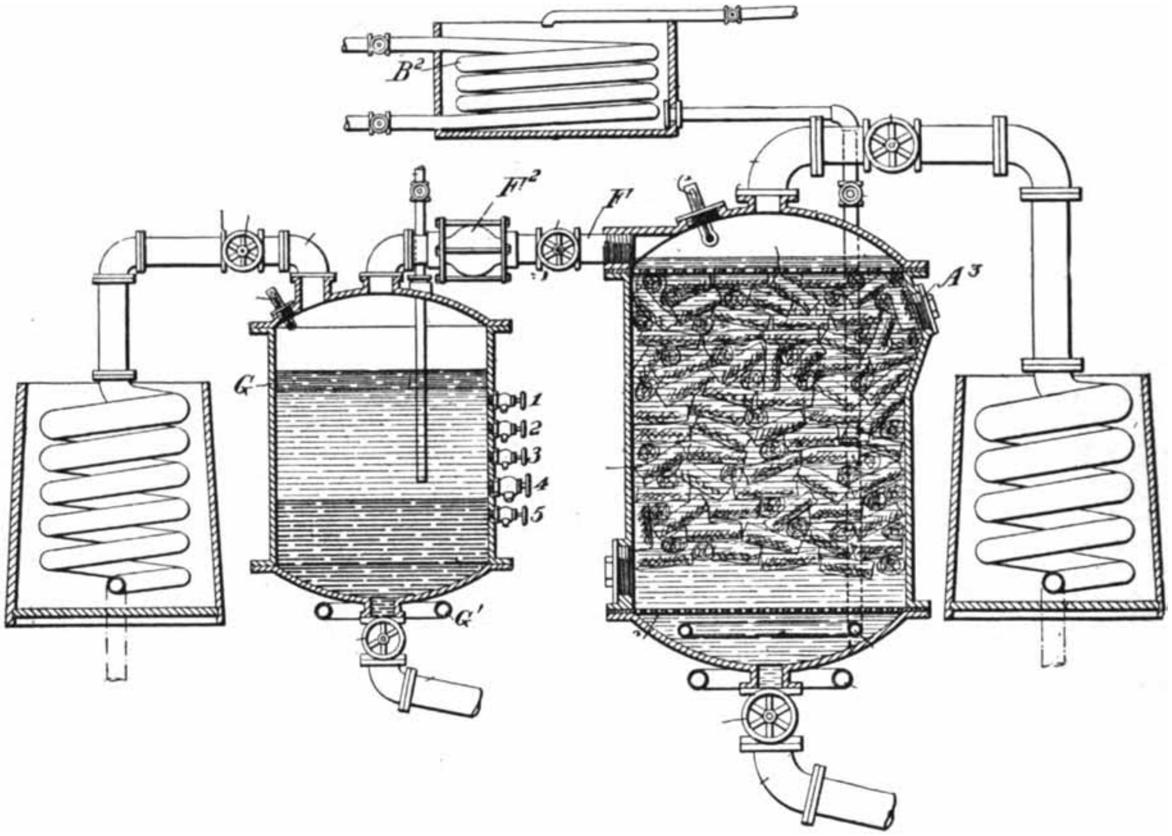


FIG. 69.—HALE & KURSTEINER PROCESS.

lar box, not over a foot or two high, is divided into parallel segments by means of partitions. At the end of each partition is set a pulley or sprocket wheel on a vertical axis. A continuous chain conveyor works in and out of each partition. Arrangements are made for tightening the chain, when necessary, while the distillation is in progress. To make a bend in the conveyor, the flights of the chain are hung from the side of the chain and thus pass under the sprocket wheel while the sprockets engage the chain above the flights.

The sawdust enters at one end of the apparatus and as it winds back and forth in advance of the conveyor flights it is completely distilled. The same apparatus, when gently heated, is used as a dryer. It is said that much success is encountered using these retorts for distilling hardwood chips, a brick furnace being used for heating purposes. Generally two are used; one for drying and the other for distilling. Such a system might be of service in distilling fat pine; the first retort taking off the turp and the second one the tar.

The next patent, that of Dobson, agitated the lumber trade for a while. It is a very slight modification of a Russian process, which never materialized. The type is so distinctly different from the others that were exploited at the time that it is interesting to know wherein its usefulness lies.

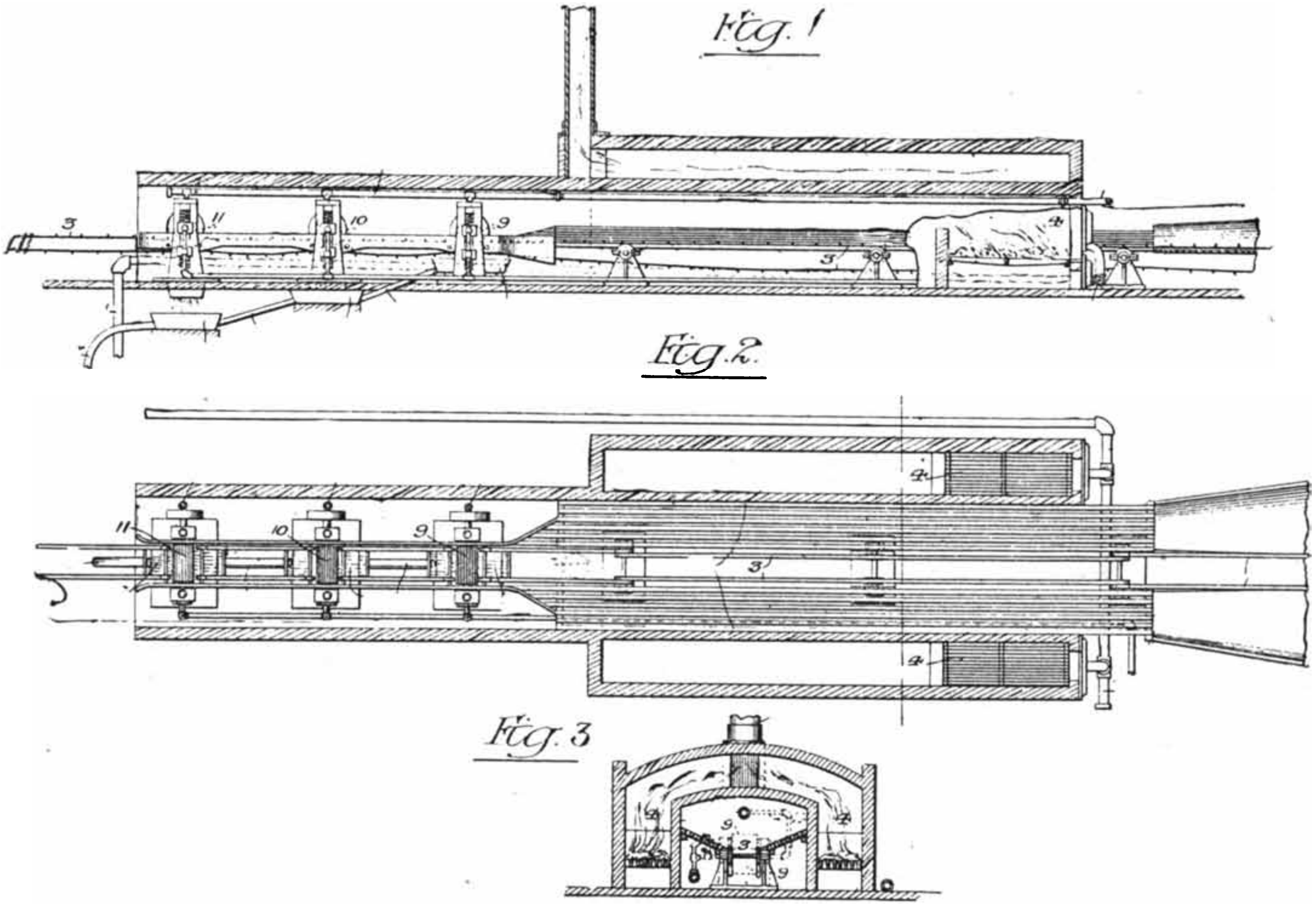


FIG. 70.—DOBSON'S PROCESS.

It is a definite project to advance ground-up wood by means of suitable conveyors, directly through a brick furnace, and while passing through the cooler parts of the furnace, the wood is to be pressed and steamed, thus expressing the tar and distilling the turpentine; the residual wood to be used for paper making.

In the illustration, Fig. 70, a brick furnace is shown at Fig. 3 with grates on both sides, 4. The conveyor space is shown at 3 and is arranged so that the furnace gases are excluded.

The operation of the process would be simple enough if it would work at all, but the inventor fails to make any arrangement for the collection of the turpentine vapors. The material passes through the furnace on the conveyor, where it is heated sufficiently to draw the resin, steam being added to facilitate the operation (turpentine would escape and an outlet to a condenser should have been made). When the material reaches the rear end of the furnace it is passed through a series of squeezing rolls, 9, 10, 11, Fig. 1, and thence out at the rear end. Such an operation could never work with sawdust, as was intended, as there is scarcely any resin in it, and only under careful operating with fat pine. By heating the hogged wood without using live steam, it might be possible to sufficiently melt the rosin so that most of it could be squeezed out. It would seem, though, that some turpentine must be lost unless some provision were made for collecting it.

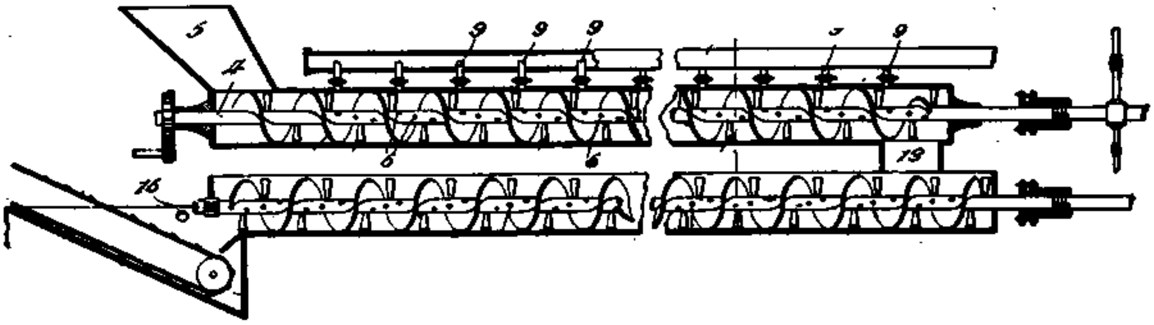


FIG. 71.—KERR'S PROCESS.

In the Kerr process is a device which ought to work well, provided the form of screw conveyor shown is suitable for moving chipped wood. The illustration, Fig. 71, shows the arrangement very clearly.

The ground wood enters the hopper 5 and falls upon the screw conveyor 4. Steam enters through the shaft by means of port holes 6, the vapors from the wood passing through 9, 9, 9, etc., to a condenser. With high pressure, the steam would also blow out through the hopper, so only light pressure steam should be used.

When the wood gets to the end of this conveyor, it drops down 13 to another conveyor, which does not fit the trough so closely as the first. Here the wood is treated with the alkaline solution mentioned in Craighill and Kerr's process, the level being kept by means of the overflow pipe 16. Steam is admitted to the trough in a similar manner as it is in the first one, but for the purpose only of keeping the alkaline solution hot.

This form of continuous process is very effective for the removal of turpentine. One trouble would be that the chips would blow through pipe 9 into the condenser pipe. This could be prevented by using screens. Another trouble is the expense of the construction. A screw conveyor 4 inches in diameter will deliver 100 bushels (equal to 118 cubic feet approximately) in one hour. The usual time of distillation with sawdust, as ordinarily practiced, is one hour for each particle of wood.

With a 4-inch conveyor with a distilling period of one hour the trough should be large enough to hold a cord of wood in order that all the wood passing through might be acted upon for one hour. This would require a trough over 1,500 feet long. With a screw 3 feet in diameter, the trough would have to be nearly 20 feet in length. It would be better to use a rotary retort with a small conveyor at each end. But with a distilling period of only 15 minutes or less, then the trough would be made much smaller and this apparatus would be very efficient.

An apparatus working on the same principle has been in use in another industry for some time. Instead of using one long conveyor several are placed one under the other and the whole encased with a suitable covering.

There are several patents based on this same principle, each with a screw conveyor in a closed trough, some using steam and some hot gases. It is difficult to distinguish any material difference in most of them.

The Heidenstam process is a device by means of which an attempt is made to briquette sawdust and then distill the briquettes to make charcoal and by-products from the wood contained therein.

Very elaborate plants have been designed for carrying out this process, but it is doubtful if any of them will pay, and it is certain that they will not in this country, where charcoal, the only product now of much value that is produced by this method, can be cheaply obtained. Formerly the production of wood alcohol by this method might have made the process useful in the hardwood region, but now that the tax has been removed from grain alcohol, this feature is lost. The process is very ingenious and has one point that is very important, and that is the pressing of the briquettes while distilling.

The great objection to the process is the difficulty in compressing sawdust. Many attempts have been made to do this by engineers so as to make a more compact fuel, but they have all signally failed unless a very expensive and uneconomical binding agent is used. By the above method a very poor briquette is formed, and it is only by the action of a poor compressing apparatus in the retort that any hard charcoal briquettes are formed at all, and these only few in number as compared with the total carbonized.

For pine wood this process is scarcely suitable, for it is easier to take the turpentine out before briquetting and thus the only good briquetting would do would be to make the sawdust or groundwood suitable for charcoal when sawdust is used, or charcoal and tar when fat wood is used. Charcoal and tar can be made from fat wood, if necessary, without first hogging it, and to treat sawdust in this manner simply for charcoal could not possibly pay. The by-products from pine wood, such as wood alcohol and acetic acid, are not of sufficient importance to count on, and at most works, when incidentally produced, are not saved.

If it should be advisable to make charcoal from sawdust or hogged wood, which might possibly be the case when located near a blast furnace, it would be better to carbonize in a rotary retort, such as one of those described, which could be done in one-fourth the time needed when the wood is in solid form, and then briquette the charcoal, using the tar formed as a binding agent. With sawdust the tar formed would be of such poor quality that this would be a good way of utilizing it.

A method of distilling by superheated steam has lately come into use in Sweden. Although the use of superheated steam for charring wood has long been known, hardwood distillers did not use it much, because it diluted the acid liquors too much. It has the further disadvantage of requiring large condensers and stronger retorts.

A series of retorts are used, each holding 200 to 1,000 cu. ft. The hottest steam is introduced into the one most nearly charred and the coolest steam into the one just charged. Ten retorts are used in a series and carbonization is completed in 12 to 20 hours and drawing and charging in four to five hours, so that each retort can be worked off in 24 hours. The greater part of the heat is required for evaporating the moisture contained in the wood, but cooler steam can be used for this purpose and hotter steam for charring. The steam together with the gases evolved from the wood pass from one retort to the next, and so on. When carbonization is completed in one retort saturated steam is passed in for one hour and this becomes superheated to some extent and after passing through the superheater is used for completing the charring of the next retort. After the steam, water is introduced into the retort in small quantities and afterwards for one hour a fine spray of water is showered upon the charcoal, which is then ready to be drawn.

The heavy oils are condensed and fall into a covered tank containing boiling water. The steam and gases from the finished retort are led through this tank and serve to heat the water where the heavy oils are condensed. These oils have a boiling point of 200 to 250 degrees C., and by using superheated steam the yield is said to be increased 17 per cent and the oil to be of much better quality. In the further description of this process, the wonderful statement is found that only 15 per cent of the weight of the charcoal produced is needed for fuel.

To avoid diluting the distillate with water when superheated steam is used for carbonizing, Wasbein used hot gases direct from a gas producer to distill the wood. The charcoal produced is used in the gas producer. This requires careful manipulation and large condensers, as not only the distilled products from the wood, but also the hot gases must be cooled. Processes of this kind are used in Germany.

Pierce Process.—In this process as described by Landreth Proc. A. A. A. S., 1888, the charring of the wood is effected in circular, flat-top, brick kilns holding 50 cords each. The wood is charred by heat produced by gas burned in a brick furnace under the kiln, and into and through which the products of combustion pass. The gaseous products of the dry distillation of the wood pass from the kiln to the condensers, where the tarry and liquid products are condensed and the gas sent back to the kiln. Thus, none of the charcoal produced is burned to carbonize other wood, as in the common pits or ovens. The gas which elsewhere is wasted is here not only sufficient to effect the carbonization of the wood, but furnishes fuel for the boilers required about the works.

In another description of the process it is stated that there is a large amount of gas left over. The gas burned under the furnace passes through the red-hot charcoal in the kiln, thus causing the carbon dioxide to be reduced to the monoxide at the expense of the charcoal.

Such a process, although largely used in distilling hardwood, cannot be very well applied to pine wood. The gas produced by pine wood distillation is not sufficient to effect carbonization, besides pine wood should never be heated to such a degree that carbon dioxide would be reduced to carbon monoxide.

Although all these processes that have been described relate chiefly to pine wood, yet all the destructive distillation processes could be applied to hardwood by simply omitting the part that relates to the turpentine. Instead of working up the oils, the pyroligneous acid is saved and wood alcohol and acetates made in customary manner. A description of a German process for carbonizing pine will be given later, which utilizes the acid in this way. All the pyroligneous acid that is made by distilling pine by the foregoing processes can be treated in this manner. The yield is so small, however, that it hardly pays.

The patents herein described are given below in the order of their application.

Wheeler, 7-5-1870
Messau, 7-3-1872
Stanley, 1872
Hansen & Smith, 10-10-1885
Berry, 12-15-1885
Wheeler, 8-26-1886
E. Koch, 3-1-1887
Smith, 12-13-1887
A. Koch, 8-13-1889
Koch & Danner, 8-26-1889
Badgley, 4-8-1890
Inderleid, 7-1-1892
Spurrier, 9-10-1898
Spurrier, 10-11-1898
Krug, 10-1-1903
Matthieu, 10-12-1903
Palmer, 12-9-1903
Dobson, 12-23-1903
Mallonee, 12-23-1903
Hoskins, 2-1-1904
Broughton, 6-13-1904
Fiveash, 6-24-1904
Davis, 7-16-1904
Harper, 7-22-1904
Fleming, 7-28-1904
Hirsch, 8-1-1904
Palmer, 8-20-1904
Ross & Edwards, 11-11-1904

Larsen, 10-7-1899
Weed, 1-6-1900
Weed, 2-15-1901
Roake, 6-21-1901
Gilmer, 6-25-1901
Adams, 12-27-1901
Chapman, 3-17-1902
Viola, 1-15-1903
Handford, 12-27-1904
Copilovich, 1-12-1905
Williams, 1-16-1905
Gardner, 3-11-1905
Sibbett & McLean, 6-20-1905
Jewett, 6-29-1905
James & James, 7-6-1905
Friis, 9-26-1905
Viola, 1-28-1903
Palmer, 5-11-1903
Bilfinger & Halleck, 4-28-1903
Clark & Harris, 6-10-1903
Bilfinger, 7-11-1903
Mallonee, 7-18-1903
Douglas, 9-1-1903
Kerr, 9-26-1905
Craighill & Kerr, 10-3-1905
Jackson, 10-11-1905
Davis, 10-23-1905
Snyder, 12-1-1905
McMillan, 3-24-1906
Craighill & Kerr, 4-17-1906
Hale & Kursteiner, 8-14-1906

CHAPTER VII.

THE EXECUTION OF THE PROCESSES OF WOOD DISTILLATION.

A description of each individual process cannot be given in detail here, but one of each general kind can be given as a type. In distilling it is absolutely necessary to work for certain definite products so as to obtain large yields of these. When working for large quantities of turpentine more time needs to be taken and the heat kept low so as not to draw out tar. In the same way when working for large quantities of tar the distillation must take place slowly or large quantities of gas will be found at the expense of the tar and charcoal.

The Steam Process.—In this process it will be considered that a series of vertical retorts are to be used set in a row. Above each retort should be a bin, unless each retort is worked alternately. Underneath the retorts should be troughs of sufficient size to hold the residue dropped from the retorts. In this trough should run a conveyor to the boilers, or elsewhere. Above the bins and retorts should be a conveyor from which the ground wood from the hog drops by means of suitable chutes to the bin or retort.

To start the plant the first retort should be filled with wood and steam turned in until the pressure reaches not over 5 or 10 pounds, or such pressure as is determined upon. At first it is advisable to allow the steam to rush in rather rapidly in order to quickly heat the retort. The stirrers should be started and the distillation continued until all the oil has been distilled over, or rather until only such a small quantity is present in the distillate as to no longer pay to continue. The steam as it warms the retort soon finds its way to the condenser, and there with the oil is condensed and flows out to the receiver. The proportion of oil and water varies with the richness of the wood, most of the oil coming over in the first part of the operation. This oil is not pure, but consists of oil and resinous matters, with a small proportion of ethers, aldehydes and ketones, which gives it a decided odor. Oil is only slightly soluble in water, and it is only necessary to allow the mixture coming from the condenser to settle when the water goes to the bottom. By using two or three tanks the oil from the first one, containing water in suspension, overflows into the second one, where more water separates, and from thence to the third, by which time enough water is taken out to furnish an oil suitable for simple redistilling in order to make it ready for the market.

It is claimed by some that in distilling sawdust only the gum turpentine found in the sap is distilled by the steam, but such is not the case, for not only is the gum oil distilled, but also that contained in the heart wood. In addition to this, resin comes over mechanically, thus coloring the crude product, but not giving it the bad odor that would come if the tar was started.

In the meantime, the other retorts are filled and started, and the material still coming in the conveyor is allowed to fall into the first bin. In the case of sawdust the first retort would probably be distilled by the time the other retorts were filled, and the bin would not be necessary. However, if the first retort is not finished, the material should be collected in the bin and the bin fitted with a large opening at the bottom to allow the material to drop out quickly into the retort when required. The charge being worked off, the steam valve is closed, the bolts taken off the door at the bottom is opened, and the residual material allowed to drop out. The door is again fastened on and a new charge dropped in from the bin above. The residue is conveyed to the boiler.

A modified working of the process is to use simply two retorts, or steamers, and have them of a size sufficient to handle all the raw material. By filling one while the other is distilling the use of bins is not necessary. It can be seen, though, that this means two retorts against a bin and a retort, and, as a bin ought to be the cheaper, this modification means greater initial cost for retort. In addition, with two retorts each would have to have special valves and connecting pipes, which further increases the initial cost. The gain would be in the saving of time in dropping the distilled material out and filling the retort from the bin, a process which ought not to take much time. Even if stirrers could be used satisfactorily, it will be found that the sawdust will pack and scaffold, or arch, thus making sawdust expensive to distill and to discharge.

It will be seen that the execution of the steam process is not a very difficult matter, and all that it needs in the way of labor is a man of sufficient intelligence to take care of a boiler when a hog is not used and an ordinary stationary engineer when a hog and engine are used. Of course, on a large scale where considerable product is made, good superintendence is necessary in order to obtain the most economical workings.

Steam and Destructive Distillation.—In this process we have a more difficult proposition than in the plain steam process. Neither this process nor the one to follow have any especial advantage, unless charcoal is to be made, consequently the process should be made to yield as much turpentine in the first stages and as much tar and charcoal in the later stages as possible, and but little gas. Although the gas formed has a high heating value, it takes more fuel to make it and at the same time the yield of the more valuable by-products of the wood is less, particularly of charcoal.

This is the process the writer would recommend when the three primary products, turpentine, tar and charcoal, are wanted, but for turpentine and tar without charcoal a special process, to be described later, seems to be the more suitable.

In the preparation of the wood a general idea must be obtained from experiment as to the yield in turpentine from a cord of four-foot wood, two-foot wood and one-foot wood in fifteen hours' heating. From this data can be obtained the increased value, if any, that is occasioned by sawing the wood into short pieces. The wood prepared in the proper manner is then charged into the retort, either by means of cars or by hand, and the doors fastened, gauges, etc., adjusted, a fire started in the furnace, and the heat raised as quickly as possible to 212 degrees Fah., without injuring the brickwork and retort seams. This takes from one to three hours, according to the size of the retort, then superheated steam is allowed to enter the retort in considerable quantity until the contents thereof are brought to approximately 325 degrees Fah., when the supply of steam should be cut down so that a considerable portion of the condensed matter is oil, say not less than one-twenty-fourth. The heat is maintained at 325 degrees Fah., not because the oil will not distill at a temperature less than this, but because it is necessary for the heat to penetrate to the inside of the block of wood and thus draw out all the oil and resin. Care must be taken not to go above this temperature or the wood would begin to decompose and empyreumatic vapors come over. Cellulose begins to decompose at 320 degrees Fah., but only slightly. Wood is a poor conductor of heat, and it is not definitely known how large a block may be, and at the same time be small enough to allow the heat to draw the resin from the middle of the block when the outside temperature is 325 degrees Fah., nor is it definitely known how long it would take for the heat to penetrate to the middle when the temperature outside is held at that degree. There is no reason why this cannot be determined, and it probably will be soon.

The amount of distillate which comes over by this treatment need not be large, but should be a very decided quantity. It is better, though, to use furnace heat as much as possible, so as to have the furnace hot, ready for the second part of the operation, so only enough steam should be used to carry over the vapors. This saves condensing water, also. When the condenser hasn't been used for some time, a green colored oil is obtained at first, due to the dissolving by the oil of the copper acetate left in the pipe from a previous distillation. Often the oil is water-white at first, quickly changing to yellow, and then to amber as more and more resin is distilled. When the amount of oil falls off and the temperature in the retort still remains the same, it is an indication that most of the light oils have been distilled, and that to obtain heavier oil with a higher boiling point the heat must be increased. The appearance of gas about the fifteenth hour after starting when the retort has been gradually heated indicates a changing point. The oils coming over can be tested with a hydrometer, if desired, but with proper firing the receiver should be changed and any gas apparatus coupled up if not already in position. Steam can now be left on or closed off, according to the notion of the operator. The author prefers to use steam throughout the entire operation, even when an exhauster is used, as a certain effect is produced that might not be otherwise. As no acetic acid is usually recovered, the excess of water in the distillate is not much to be feared, unless the proportion is such that the tar and acid will not properly separate.

From now on the progress of the distillation can be determined in various ways by experienced men. The heating should not be great enough to cause back pressure from the gas, and at the same time a steady stream should flow from the end of the condenser with weak force. The gases formed are at first blue when ignited, from the combustion of the carbon monoxide, and as the distillation proceeds this flame becomes gradually yellow, and toward the end of the distillation the heavy white yellow flame of the heavy hydrocarbons makes its appearance and continues until the distillation is finished.

Of course, the distillation can still be carried on by means of an ordinary thermometer until the last stages are reached, when it is necessary to use a pyrometer of some kind, or a special 1000 degree Fah. mercurial thermometer. When the temperature reaches 500 and in some cases 600 degrees Fah., some operators change the receiver again and then continue the distillation to obtain the thicker tar. Others collect the entire tarry distillate in one vessel and then redistill. Ordinary distillation would be finished when the temperature reached a few degrees over 800 degrees Fah., but in some cases, owing to the formation of paraffins of high boiling points it is better to run to about 900 degrees Fah. The author has sometimes been obliged to go even higher than that. With separate condensers for each retort the end point is readily ascertained by the slacking off in the quantity of the distillate. When this point is reached the fires can be withdrawn and the gas turned into another fireplace. The heat of the furnace walls will be found to be sufficient to complete the distillation. By having the furnaces hot at the time the wood is ready to decompose the destructive distillation itself requires but eight hours in a three-cord retort. If lined with brick the retort will not be specially injured. When the distillation is ended the retort will be found to be red hot on the bottom. It is allowed to cool until the iron becomes black, when the charcoal is drawn. The residual gas remaining in the retort should be touched off like when making coal gas, and the admittance of gas from other retorts prevented. The heads are then removed and the charcoal immediately withdrawn. Using cars, the products can be easily pulled into coolers without much loss from burning. Others simply draw the charcoal into a pit and wet it with water or cover it with wet charcoal dust. Some drop the charcoal into cars and lute on a sheet-iron cover.

Destructive Distillation.—This process is carried on exactly the same as the foregoing in the latter part of the operation and very similarly in the first part, except no steam is used, consequently the first distillates are smaller and darker. The first portion is usually caught in separate receivers, but this is not always the case.

A rather complete plant for the utilization of pine wood as carried out in Germany is shown in

Fig. 72. This is a plan of a plant for distilling ten cords of pine wood in twenty-four hours.

The wood is brought in on cars on the track A fitted with suitable turntables, B, B, B, B. To use the labor employed properly the retorts are charged one after the other, instead of all at once. The retorts (c, c, etc.) on the left would be charged first, and those on the right last. The distillation proceeds in the usual manner, the vapor being condensed in the condensers D, D, etc., two pipes being in one cooling box. The distillates from all the retorts go down pipe E to the tank F, F, F, where the oil and acid separate. The oil goes to the tank G, and the acid containing the wood alcohol is either sent to one of the stills J, J, or to the tank H to be first neutralized with lime. In making brown acetate the lime is added to the liquor in H and the excess of lime and sediment removed by the filter press I. The liquor is then transferred to an iron still J and the alcohol distilled. To make grey acetate several methods are employed. In one the acid and alcohol are distilled in the copper still J, leaving the tar as a residue. This tar is forced in a spray under the boilers and used for fuel unless the market price warrants its sale. The alcohol and acid are then brought to the neutralizing tank H, where the liquor is exactly neutralized with lime and filter-pressed. The alcohol is then removed by distillation in the iron still J and collected at K. Another method is to distill the crude pyroligenous acid and collect the alcohol until the sp. gr. of the distillates is 1 and then to change the receiver and collect acetic acid separately. This acid is then neutralized and evaporated. Another method is to distill the pyroligneous acid in a copper still and to make the vapors pass through

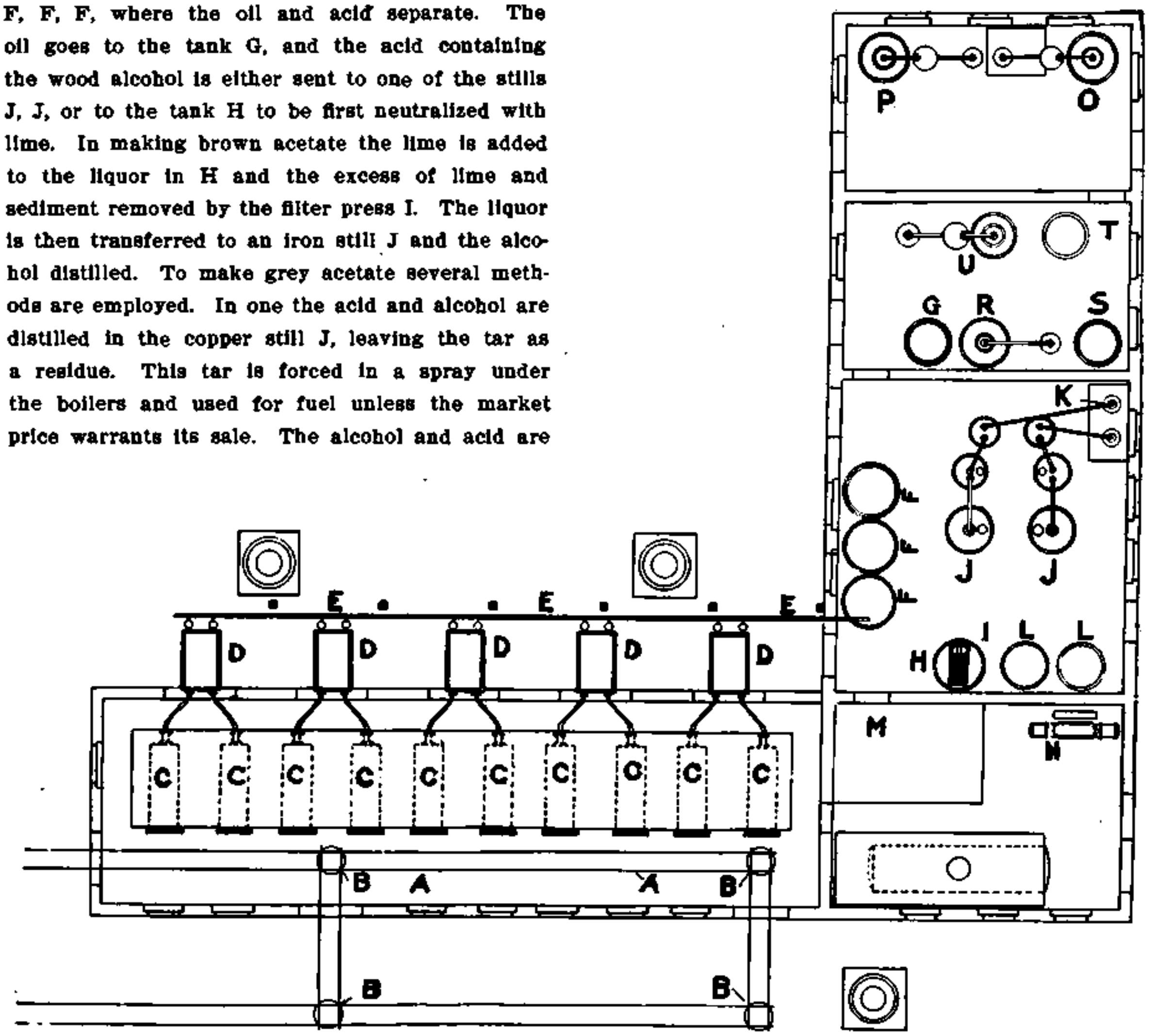


FIG. 72—GERMAN DESTRUCTIVE DISTILLATION PLANT.

milk of lime, which absorbs the acetic acid and permits the alcohol to pass on to the condenser. This latter method would take less fuel, as the acetate liquor would be hot. It would be difficult to regulate the supply of lime for neutralizing.

In the North a method is used whereby the lime is added directly into the iron still from a lime box situated above it. The still is furnished with a stirrer to thoroughly mix the liquid, otherwise the pyroligneous acid is treated by the first method both for grey and brown acetate of lime.

The acetate or neutralized liquor thus produced is evaporated to a thick paste in the steam-jacketed pans, L, L, furnished with stirrers. This paste is then spread out on the acetate pans, M, to dry, care being taken not to char. At some plants the drying takes place in rooms heated with waste furnace or retort gases. (See Calcium Acetate, Chapter X.) With this particular process the acetate pans are located in the boiler room, where the water pump N is also situated.

The alcohol coming from the still J is collected until the gravity is about 1. This weak alcohol is then sent to the still O and distilled. In this still alcohol of .965 sp. gr. is converted into portions, some as light as .816 sp. gr., containing 95 per cent alcohol. The general average of this still is considered to be 82 per cent. Care must be taken not to let the wood oil mix with the high proof alcohol or it will render it non-miscible with water. This is usually avoided by collecting the different portions separately and returning some of the liquor to be redistilled. With the German method the first runnings consisting of more or less colored liquor is caught separately until the middle fraction begins to distill over. After the middle portion distills, products with a higher boiling point come over, their presence being first noticeable by the turbidity of the distillate produced when water is added to it. Subsequently the distillate itself is rendered turbid and eventually it comes over in two layers, oil and water. Finally only water comes over, impregnated with empyreumatic substances. The alcohol rendered turbid by water can be treated in two ways; it can be added to the turbid distillate and the mixture added to the next charge of crude in the same still, or it can be diluted until it shows a specific gravity of 0.934 and allowed to rest for a few days, when the greater portion of the hydrocarbons separate as an oily layer on the top, and can be drawn off. The alcoholic fluid left is redistilled over lime and makes strong alcohol that does not become turbid upon the addition of water. The oily fractions are mixed together and redistilled separately, when a further quantity of middle fraction is obtained. The wood oil obtained is known as red oil, and is usually burnt. By distilling the strong alcohol obtained from the above still after adding a little sulphuric acid in the still P a very strong highly refined alcohol is produced.

At some plants the crude wood alcohol is passed through towers containing wood charcoal, which serves to remove some of the ketones, aldehydes and tarry matters.

None of these processes serve to remove acetone. To do this several methods are used; one is to form a compound of wood alcohol and calcium chloride, which is stable at 100 degrees C. By gently heating the acetone is driven off, and then by adding water and raising the temperature to 100 degrees C. the calcium chloride compound decomposes and the methyl alcohol distills. Others add caustic potash and iodine until the yellow color disappears, then distill. (Regnault & Villejean.) The watery alcohol is repeatedly rectified over lime, and finally over sodium or phosphoric anhydride to remove the last traces of water.

The crude turpentine oil containing the tar, resin, etc., is taken to the tank G, from whence it enters still R, where the light oils are removed and are collected in tank S. From S the oil goes to the washer T, where it is washed with alkali water, acid, and again with water, and then rectified in the fractionating still U, and is then settled ready for shipment. The tar is removed from the still R and is immediately ready for shipment. The residues from the other stills are mixed with crude oil and again distilled until they begin to accumulate, when they are otherwise disposed of. The charcoal left in the retort is taken out by means of a chain fastened to a scraper, placed in the rear of the retort, and dropped into an iron bogie running on the small track. The bogie is covered with sheet iron and the edges luted with clay to exclude air and the whole rolled away to cool in a storehouse. It is necessary to have one car for each retort, and about three for bringing in the wood.

Special Process.—The use of a rotary retort should be described here, as the author believes that this method will eventually be the one used to utilize all kinds of waste wood, particularly the average dead pine found in the woods. Wherever such pine can be found yielding four gallons of turpentine to the cord and not costing over $1.50 per cord, delivered, this process can be successfully employed. Under such disadvantageous conditions the closest economy in working is necessary.

The combination of apparatus suggested would be two rotary retorts, a boiler, a superheater, a blower, an exhauster, two condensers, two stills and condensers, one water pump and a hog. Those who prefer mixing the distillates need have but one retort and condenser and extra still. A conveyor from the hog to the retort would be needed and one from the end of the retort to the boiler. The wood should be hogged in the usual manner and brought to the retort for distillation. In the first retort the turpentine should be worked off and condensed and the chips discharged into the second retort, where they are thoroughly charred by means of superheated steam, hot inert gases, or by means of fire gases passing through special flues or through the wood itself.

With fat wood each operation should be performed in six hours and the charred wood used for fuel in addition to whatever other fuel is needed. The tar carried over by the steam is condensed in the usual manner, and then redistilled to recover any light oils, and by passing a current of superheated steam through the mass while hot sufficient of the heavier oils can be carried over and the tar left of the proper consistency. By this method of operating the tar obtained would be of sufficient value to pay for the fuel and wood, and the turpentine to pay for the labor and leave a small profit in addition. Charcoal is of no value in most communities except in small quantities, so this method would probably be the best that could be devised to treat the average wood found in any locality. It offers further attractiveness in that when the supply of wood is exhausted the entire outfit could be easily removed to another place. For those who prefer to use but one retort it is best to have it long so that the end farthest from the feed can be heated to the charring point, and the hot gases led out through a pipe at the feed end, the heat from these gases thus being utilized to partially distill the incoming wood with which they come in contact on the passage through the retort. The products can then be refined. In all cases it is better to coat the shells of the retort with asbestos, or other covering, so as to prevent radiation as much as possible.

In those cases where very poor wood is used it might be that the tar made became too dark, owing to the lack of resin. This might be mixed with the fine charcoal and briquetted and sent to an iron furnace, or again distilled to form special charcoal bricks such as are used in foot-warmers. It can be readily understood that in these cases only a small plant could pay, as the demand for charcoal in the latter form would be limited, indeed. With blast furnaces the results would be more encouraging and a large plant would be needed. This would be an easy way of solving the problem in such districts. It is claimed by manufacturers of briquette machinery that briquetting can be done for 50 cents per ton. By the ordinary method of distilling large wood it takes from twelve to twenty-four hours to char, whereas by this method only six hours is necessary, so this difference in time would more than pay for the cost of briquetting.

Wood Gas Making.—So far it has only been intimated that wood gas is yielded in the destructive distillation of wood in sufficient quantities to pay for working for gas alone. However, the yield of gas ranges from 20 to 50 per cent of the weight of the wood. By ordinary distillation 20 to 30 per cent is the usual yield, but by rapid heating the yield is greatly increased. The weight of 1,000 cubic feet of wood gas is about 50 pounds at 62 degrees Fah., so a ton of sawdust might, under certain conditions, be made to yield 1,000 pounds of gas, or about 20,000 cubic feet, whereas one ton of the best gas coal from Grahamite, W. Va., gives but 15,000 cubic feet of coal gas and most coals only about 10,000 to 12,000. Wood gas must be purified with lime to remove the carbon dioxide, and then it has an illuminating value, according to Liebig, of 6 to 5 compared with coal gas. (See analysis of wood gas under Chapter XII.)

To carry out the process of making gas, several methods are used. One consists in heating the wood in ordinary retorts and then passing the vapors through a superheater, which decomposes them and forms uncondensable gases. One method is to throw the wood as quickly as possible into a glowing retort and to collect the gases in the ordinary manner. Another utilizes the principles of a water gas apparatus, and treats the wood in a similar manner. Another method is to collect the gas separately, then by using the charcoal in a water gas system the charcoal can be glowed by the air blast and the wood gas passed through it to decompose as far as possible the carbon dioxide contained therein, then steam added until the temperature became too low for decomposition. If the charcoal was too expensive, coal or coke could be used. Then the distilling method with hot gases, or preferably producer-gas, could be used.

An attempt will not be made to enter into the details of operation of these various methods, as wood gas manufacture is not apt to be a flourishing industry, owing to the fact that the proper place to make wood gas economically would be in the woods, because wood is too bulky, as compared with coal, for transport; and it is not to be expected that it would pay to transport the gas even by pipe line, except in those cases where the gas could be made near the city.

The author would suggest the possibility, however, of utilizing sawdust for gas making in those States where lumber mills are located in comparatively large cities. To do this it would be necessary to use those forms of apparatus spoken of for distilling sawdust destructively. Of these, Larsen's and Harper's rotary retorts, Halliday's screw retort, and Bower's chain retorts might be used. The last two are usually enclosed in brick so no further modification would be necessary. With pine or fir distillation the turpentine could be taken off in a previous operation and the residue destructively distilled for gas. Larger vapor pipes and condensers would be necessary, owing to the sudden formation of gas when the wood is brought in contact with the hot retort. In all cases it is presupposed that the wood has been dried. To use the gas under a pressure of 1-12 to ⅛ inch of water, bat wing tips having a width of about 0.0394 inch give the best results. A Welsbach mantle is also very serviceable.

A method used in France endeavors to utilize wood in generators that are designed to supply gas for motors. The consumption of ordinary wood for such purpose is about five pounds per horsepower hour. The heating is done from top to bottom. The wood that first enters is charred and falls to the bottom as red-hot charcoal. The air blast having been heated by the pipe through which the gas is escaping, enters the top of the generator and, coming in contact with fresh wood, partially distills it, and the products of distillation pass through the red-hot charcoal at the bottom, thus decomposing the tar to form more gas and the combined vapors and gases pass out at a pipe at the bottom through a filter and then to a separator. It can be seen that unless carefully regulated the supply of oxygen in the air would be exhausted before reaching the charcoal at the bottom of the generator and the charcoal remain unburned. Arrangement is made for drawing this residual charcoal into water, where the ashes will sink and the charcoal float. This charcoal could be dried and returned to the top of the apparatus.

Wood gas has some advantages over coal gas when used in gas engines. For this reason attempts may be made to utilize wood waste by converting it into gas.

Any of the processes herein described that are based on destructive distillation are suitable for making gas. Those that arrange to protect the retort from the effects of severe heating and those that treat small pieces of wood effectively are to be preferred.

Two forms of gas generators can be formed from these classes of apparatus; one using closed retorts and the other being open for the admission of air similar to a gas producer. As a gas producer the apparatus would necessarily be of the suction type, although in a few instances the type using a blast could be employed.

Those familiar with coal gas generators can readily add the necessary modification to wood distilling apparatus to bring satisfactory results.

CHAPTER VIII.

REFINING PROCESSES.

General methods of refining have already been given and some special methods of refining have been given under the different processes. The patents now to be described are considered chiefly because they show a definite method of carrying out principles already in use or known, rather than because they relate to any new methods or principles.

Crude turpentine as it comes from the retort is invariably more or less colored at some stage of the operation, no matter what the process is that may be used, consequently refining is necessary to remove objectionable impurities. This latter is especially true where all the products of distillation are mixed together and then refined.

In all methods of refining the same general object is to be attained. The crude turpentine may contain light and heavy oils and tar. The object of refining then is to separate the turpentine from these other ingredients when they are present. Light oils in most processes are produced from the rosin by local action, while in some destructive distillation processes the presence of light oils is always to be expected. In the steam process, the objectionable impurity is a heavy oil and is easily separated by ordinary distillation, the light oil coming first and the heavy oil remaining in the still or collected separately.

Chemicals are often used, such as lime and caustic soda and even acid; the use of mineral acid is not to be commended as it has a tendency to change pinene into dipentene. All these chemicals have been in use for a long time, coupled with steam distillation.

By noticing the methods described under the following patents, a general idea can be obtained of the methods used in refining the crude turpentine.

Mallonee's Apparatus—The method is shown in Fig. 73. It consists of a series of similar units, working separately in a similar manner. The

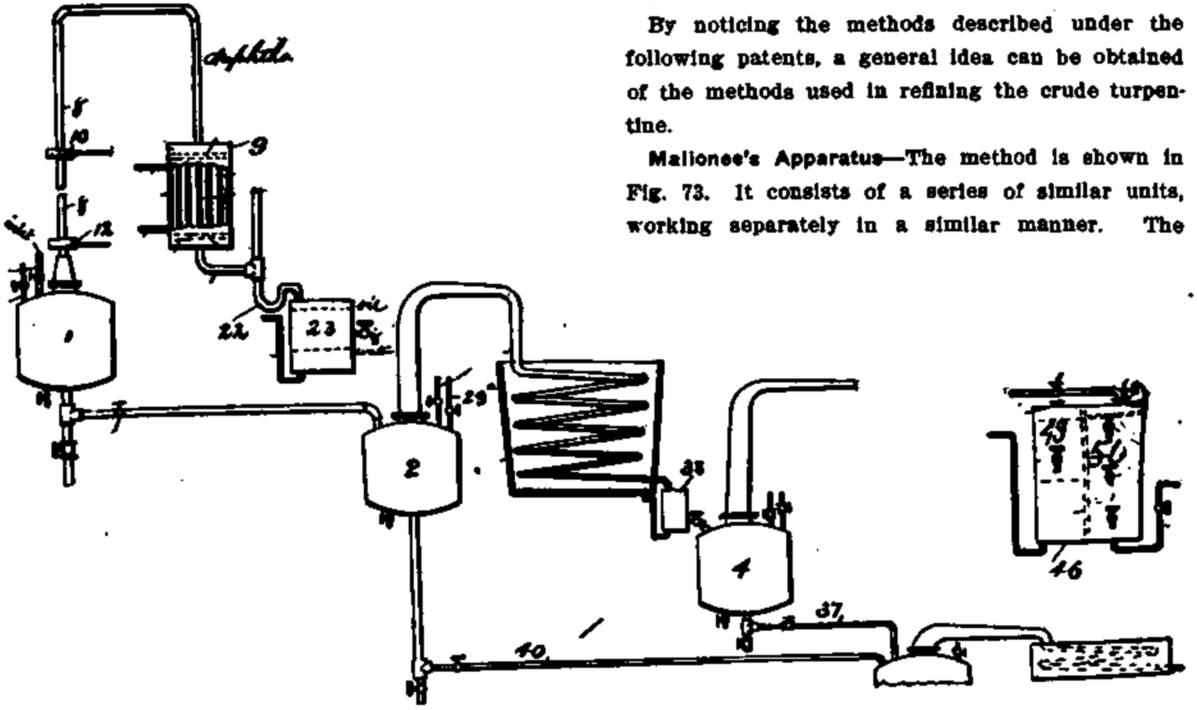


FIG. 73—MALLONEE'S PROCESS.

operation of the stills shown, have been described in part under Mallonee's process, Fig. 37. The distillate from the retort is caught in three separate fractions according to the specific gravity; the first fraction being caught from 0.855 sp. gr. to 0.920; the second fraction from 0.92 to 0.96 sp. gr., and the third fraction from 0.96 sp. gr. to the end of the distillation. The refining apparatus shown in Fig. 73 deals entirely with the first two fractions and the operation is carried on in a similar manner with both fractions, the only difference being in the proportion of the different products which distill over.

The crude oil is placed in still 1 and heated in the ordinary manner by means of steam (see Fig. 17, Steam Still). The light oil vapors pass up pipe 8, which is about twenty feet in length, and are condensed in the tubular condenser 9 and pass into the receiver 23. To prevent the turpentine vapors passing to the condenser with the light oil, a spray of water is applied at 10 to cool the vapor pipe, the water passing down the pipe to the pan 12, from whence it is carried away. A gas trap is shown at 22 and an oil and water separator and receiver at 23.

After distilling the light oils, the remaining oil in the still is allowed to flow into still 2 through pipe 27. In this still the turpentine is distilled in the usual manner by means of steam. The distilled turpentine is separated from the water at 33 and allowed to flow into still 4, where it is redistilled in order to make it clear. The residue in stills 2 and 4 is permitted to flow into still 3 by means of pipes 40 and 37, respectively. From this still heavy oils are recovered by distillation with steam, as before, and collected in two fractions in the separator 46. This separator is divided into two compartments and the first fraction of the distillate, comprising oils lighter than water, passes from the pipe 44 into the compartment 49, and the second fraction, consisting of oils heavier than water, is collected in compartment 50. The water is separated by gravity from the oils and the oils sent to storage tanks or barrelled.

Gilmer's Refining Process—The crude material used in this apparatus is the oil obtained by distilling pine wood at a low temperature, and is only impregnated to a slight extent with creosote or tar vapors.

A combination of the apparatus used is shown in Fig. 74. At 1 is a receiving tank for the crude oil and acid water coming from the retort. The acid water being drawn off, the crude oil is allowed to flow into still 5, where it is mixed with about 50 per cent of pure water. The mixture is

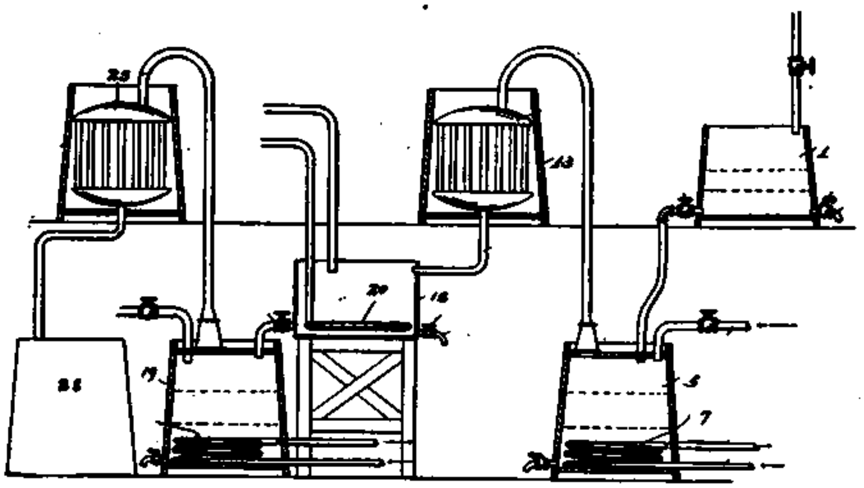


FIG. 74—GILMER'S REFINING PROCESS.

heated by means of the steam coil 7, which is kept under the water so as not to be in contact with the oil. The distilled oil and water are condensed in the tubular condenser 13 and are collected in the tank 20. The residue in the still 5 is thick tar and is sold as such. The mixture in tank 15 settles by gravity and the water is drawn off. The oil is then treated with lime water of about 3 or 4 degrees Baume, in the proportion of one part of lime water to two of turpentine. The mixture is then subjected to a thorough agitation and aeration by blowing air therethrough for about one hour. The mixture is again allowed to settle and decanted to separate the lime water. The turpentine is then again distilled in still 19 in a similar manner as before. To avoid injuring the product the distillation is performed slowly, about four barrels being distilled in eight hours. The steam in the close coil should be at a temperature of about 335 degrees Fah. The vapors from the second distillation are condensed by means of the tubular condenser 25 and are collected in a storage tank 28.

Heber's Process—The two processes just given require a specially prepared crude product. In the Heber process the idea is to purify the bad-smelling oils produced by destructive distillation processes by a chemical treatment consisting of treatment with oxidizing compounds. The process requires considerable care, as turpentine is also affected by the chemicals used, and an excess must therefore be avoided. Before starting the process the oil should first be removed from the bulk of the tar by ordinary distillation with steam. This crude oil containing tarry impurities is then distilled over lime to remove the remainder of the tar. A mixture of one to two per cent of lime with water is used and the distillation carried on by means of steam. The oil still contains some coloring and odoriferous matter. To the oil is added a sufficient quantity of a ten per cent soap solution to thoroughly dissolve or emulsify the oil, the whole being mixed in a still provided with a suitable agitator. When emulsified, a five per cent solution of permanganate solution holding from three to five pounds of potassium permanganate and four to six pounds of sulphuric acid of 66 degrees Baume in solution is slowly added, the mixture being constantly agitated or stirred. This agitation is continued until the permanganate solution introduced into the still has completely lost its color, after which by the addition of calcium chloride or zinc sulphate, the soap solution is precipitated as insoluble calcium or zinc soap. The turpentine is then distilled in the usual manner by the use of steam.

The oxidizing of the soap emulsion can be accomplished by using chromic acid and sulphuric acid in the same strength and proportions as are used with the permanganate solution. When salts of chromium are used, about six to nine pounds of potassium bichromate (or an equivalent quantity of sodium bichromate) should be used for each one hundred pounds of oil which has been treated with soap solution. These six to nine pounds of bichromate are converted into a five per cent aqueous solution and four to seven pounds of concentrated sulphuric acid of 66 degrees Baume slowly added to the same. The treatment is carried on further as with permanganate.

It can be easily understood that if the quantity of impurities present varied much, there would be danger of losing turpentine in considerable quantity.

CHAPTER IX.

GENERAL CONSIDERATIONS FOR THE ESTABLISHMENT OF A PLANT.

To those contemplating the erection of a plant for wood distillation the several conditions and requirements herein contained are essential to success, and in special cases other considerations would be necessary.

The first essential is a supply of raw material of the proper quality. There must not be any guess-work about this, but the amount and the quality of the material should be definitely known in order to regulate the size of the apparatus. It is best to own the necessary raw material, but if not owned it is not best to buy a lot of wood until the success of the plant is assured. On the other hand, owners of raw material will be found to quickly raise the price unless some provision is made in advance for a sufficient supply. For this reason, it is better in such cases to obtain an option at a definite price for a certain period.

To estimate the quantity of material to be found a simple riding over the land will not suffice. The wood should be gathered from a certain tract, cut up and separated into the various qualities, and these separate portions tested for the amount of oil. Having determined this, further calculations can be made as to the expected success of the venture. Manufacturers of machines claim anything from a yield of five gallons for sawdust to fifty gallons for fat wood. Generally these tests, if made at all, are made upon one stick, or one lot of wood, and are not safe estimates.

After having satisfactorily determined the amount of raw material, the next point to be considered is its location. Some of the best wood for distillation is located in such inaccessible places that it is not to be considered at all, while, on the other hand, inferior wood can be obtained so much more cheaply that it would pay better to use it. If possible, it is best to have the supply where it can be reached by both rail and water. The water route would be cheaper, both for transporting and loading, but it has the disadvantage in that the wood near the bank has generally been culled for steamboat purposes and is consequently inferior. With railroad transportation, the loading is more expensive, although somewhat compensated for in the unloading. With tram roads the wood is also apt to be culled. One advantage in this previous gathering of the wood is the fact that laborers can be more readily obtained who are used to that sort of work, and probably have teams ready and can haul by contract, thus saving one of the greatest troubles in the business. A difference of 50 cents per cord in the cost of raw material will often ruin the prospects of a plant.

Another important feature is the location of the plant itself. A steam plant requiring but few repairs and not occupying much space, nor employing much labor, might be placed directly in the woods itself, other conditions not being considered. With a destructive distillation plant, where the retorts are often out of order, close proximity to a repair shop would seem advisable. However, the difficulty of obtaining a few laborers to live in the woods and stay would seem to indicate that the proper location of a plant should be sufficiently near a town of some size so that in an emergency laborers could be obtained more easily. Another point to be considered in point of location is the disposition of the product. In this industry, wagon-hauling won't do like the custom in many instances practiced in making gum spirits; the plant must be located on a railroad, and, if possible, connected with some water route. This is particularly true with those plants making tar and charcoal, both of which are bulky articles as compared with their intrinsic value. Of course, a junction of two opposing railroads, coupled with water communication, would be an ideal combination seldom realized. Furthermore, in making charcoal a market for the same must be found near at hand.

After deciding upon the location, the treatment of the wood before distillation should be considered. Wood for this industry should be bought by weight, dry, and the diameter limited to 6 inches. This is especially true when using knots, as one cannot tell what the yield will be by the cord, as wood is of different lengths and sizes and is generally very crooked; also, the fatter the wood the heavier it is, and thus more inducement is offered to gatherers to supply the rich wood in preference. After the wood is delivered, further treatment is generally necessary. In the case of steam distillation yielding turpentine only, the finer the material is hogged the better will be the results. The exact degree of fineness that would be the most economical can be determined by experience, and would be dependent upon the difference in yield in a given time as compared to the cost of labor and time expended to comminute it.

With the destructive process it is an open question whether it is better to use the wood long or to saw it into short lengths.

The loss in sawdust must be considered, as well as the labor cost of sawing. The comparison would have to be made between the yields in a given time, and the cost of preparation. It is generally supposed that the resins and products of distillation exude from the ends of the piece, so the more pieces there are, there will be twice as many ends. This is, to a large extent, true, and it would be supposed that to hog it would make it infinitely better for distillation. While this is true as regards the removal of the turpentine, yet when destructive distillation sets in, the process not only does not work smoothly, but the material next to the shell of the retort can be thoroughly charred and the interior not charred at all. However, the author finds that, although more products of decomposition come out at the ends, yet a considerable portion comes out over the entire surface. It would be necessary, probably, to make a definite test at each plant to ascertain the best conditions.

It may be doubtful sometimes what to use for fuel, some recommending using the charcoal produced when a destructive distillation process is used; others crude oil; some sawdust, and some wood. Not that which is the cheapest, but that which is of the least value to the plant should be used. For instance, charcoal might be made for $5 per ton, and be cheaper than coal at $8 per ton, but if charcoal could be sold for $10 per ton it would be of more value to the plant to sell it than to burn it in preference to coal. In the same way, it might cost $1 per ton to make the residue from the steam process, and this would be cheaper than wood as fuel at $2 per ton; but if the residue could be sold for $3 per ton for making oxalic acid, then the residue would be of more value to the plant if sold than if burnt for fuel. This method of calculation is, of course, very familiar.

The quantity of fuel used by the different processes per cord of wood varies with the dryness and the amount of pitch. Information concerning this is not easily ascertained. In the hardwood industry certain facts are definitely known relative to the fuel used per cord, and for pine wood it is considerably more. The fuel proposition at most wood plants is a serious one unless favorably located.

In the steam process distilling sawdust with a yield of one to three gallons per ton with a distillation period of one hour, it is claimed by those making such distillations, that it requires only about one-quarter of the residue for fuel. This looks rather low. Using fat wood yielding fifteen to eighteen gallons per cord, with a distilling period of three hours, it takes about all the residue as fuel to furnish steam for the operation.

In the steam and distilling process it is claimed that only one cord of wood is required as fuel for each cord of wood distilled. One plant, however, using arches to protect the retort, distilling wood yielding ten gallons of turpentine to the cord, with a distilling period of twenty-four hours, took 2½ cords of slabs to use as fuel to distill the wood, pump the condensing water and furnish lights, etc., and when oil was submitted as fuel it required 6.4 barrels per cord to furnish the necessary heat for the above purposes. This plant used cars for drawing out the charcoal.

In the hardwood industry small retorts use about 500 pounds of soft coal, plus the tar produced per cord of wood distilled, the coal equal to about one-half a cord of wood. The large ovens require from 5,000 to 8,000 cubic feet of natural gas, 30,000 cubic feet of this being equal, approximately, to a ton of coal.

From this, it seems that results are apt to vary, and only a general rule can be given. For the steam process about one quarter to three-quarters of a cord of fuel per cord of wood distilled, and for the destructive process on pine wood from one to two cords of wood, or its equivalent, according to the construction of the furnace and the process used.

The next consideration is the kind and quantity of apparatus needed, and this is determined by the process used and the yield per cord; fat wood taking longer to distill, thus requiring more apparatus for a given capacity.

Using the steam process for sawdust and slabs, a plant independent of a saw mill would need boilers of sufficient capacity to furnish steam for distilling, pumping water and running the engines for the conveyors and hog, as well as any stirring apparatus used in the retort. The size of this apparatus is determined by the quantity of raw material to be utilized per day.

With some processes it is best to build in the form of units, having the units as large as possible to make economical working. Take, for example, a vertical steam retort with an opening at the top for charging and a door at the bottom for discharging; it can be readily seen that if all the work was to be done in one of these, that cases might arise in which it would take special machinery to raise and lower the doors, whereas if made in smaller units they could be easily operated by hand, or by means of simple contrivances. Under certain conditions in using a very large retort, by letting in only a small amount of steam, the whole might condense without doing any work. Except in those cases where the labor cannot be used to advantage, it would seem advisable to use as large retorts and condensers as possible, consistent with continuous working. Where the work can be performed better in alternate retorts, then half the size would be better, two units being used. The steam process, taking from only one to six hours, has the advantage over the others, in that a night crew is not necessary.

All steam processes ought to extract the oil equally well with the same conditions of steaming. The difference would be in the time of steaming, and it is to be expected that those processes which stir the wood would give the quickest and best yield. For this reason, rotary retorts are in use, and if the saving in time or increase in yield warrants the additional initial cost, they will be much used. But the essential difference in the steam process must be in the method of handling the raw material, and an examination of the plans of the proposed process will enable one to judge of its merits in this direction. In making estimates on all plants, plenty of margin should be allowed for unforeseen contingencies, particularly as to the yield, the amount of fuel used and the condition of the market.

If it has been decided that charcoal should be made, then choice should be made of a steam and destructive process or a destructive process without steam. Perhaps the most important thing to consider in connection with these processes would be the method of handling the charcoal produced. Those processes that arrange for the removal of the charcoal while the retort is hot are to be preferred. There is a great saving in fuel, and also in time.

Ordinary destructive distillation in one cord retorts ought to take from twenty-one to twenty-two hours, thus enabling the contents of each retort to be distilled once in twenty-four hours. Some attempt to complete the distillation in less time than that, but the damage to the retort is very severe. The use of cars is practiced in ovens and large retorts, but in order to make them of sufficient rigidity, it is necessary to make them of such a thickness that they affect the heating value of the retort. This can be readily observed in those cars open at the top and less open at the bottom. In this case the wood is often found to be charred at the top of the car and only partially so at the bottom, and this when the heat is applied to the bottom.

But when the wood is placed directly in contact with the walls of the retort, the quality of the turpentine is impaired, so some means should be devised to keep the wood from coming in contact with the walls of the retort and at the same time serve to remove the charcoal. Perhaps more openwork cars provided with a means of scraping the trash and fine charcoal from the bottom of the retort upon their exit, may prove satisfactory. On account of the deposit of coke on the bottom of the retort, which is the residue from the distilled tar which drops from the bottom of these openwork cars, a long retort is difficult to clean out while hot, and as they must be cleaned to prevent burning of the bottom, the process used must provide a means for the removal of this material. The use of large or small retorts is a question at issue with pine wood distillers. If small retorts were used cars would not be advisable, as they occupy relatively too much space, and full advantage of the capacity of the retort cannot be obtained. However, other things being equal, the author, although preferring small retorts in most instances, sees no reason why a large retort constructed upon the same principles as the long ovens used in the hardwood industry, and fired by crude oil or natural gas, would not give as good satisfaction with pine wood as with hardwood. Exactly the same principles would govern the firing, and it would be only necessary to use a correspondingly large quantity of superheated steam during the first part of the operation to carry off the turpentine vapors. It would take about 8,000 cubic feet of natural gas per cord to distil the charge in such an oven. In these cases cars could be used as found in the hardwood industry.

The use of an oven brings forth the question of furnace construction and the cost of fuel. There is no doubt that the cost of charring in cars is greater than without their use, but the question of using an arch to protect the retorts from the injurious action of the fire gases is much agitated. It has been found at a steam and destructive distillation plant using retorts seventeen feet long, set in a furnace with return flues, that if properly made the furnace arches stood very well, but if they became out of order for any reason they would fall in at the most critical periods of the distillation and allow the flame to severely injure the retort. Furthermore, the fuel used was over twice as much as is usually used when no arch is present. Firing with crude oil over six barrels was necessary to distill one cord of medium rich wood, when without an arch one cord of wood would be sufficient. It would seem that with short retorts the use of the arches below the retort causes the heat to go up the chimney, whereas in a long oven the heat of the fuel would be better absorbed by the brickwork, owing to the longer time of contact. It is on this account, probably, that arches are found under some of the 50-foot ovens used in the hardwood industry, and none under the 9-foot cylindrical retorts. It would seem that in the latter case the retorts would rapidly burn through, but this seems to depend upon the firing, as a Florida company has used some for three years and a Northern hardwood company used some continuously for five years, and only one out of seven had needed to be even turned in that time. On the other hand, at the same place a whole set had been burned out in less than a year. As an oven is so difficult to replace, it is advisable to protect them to some extent. An arch as long as it stands and is not in direct contact with the iron of a retort will undoubtedly protect the retort, and in those cases where the fuel is cheap enough to allow it, the arch could be advantageously used.

The next feature to consider in judging the process for the production of turpentine, tar and charcoal is the quality of the turpentine produced. Although the refining of the crude product is perhaps the most essential thing, connected with the quality of the oil produced, nevertheless those processes that do not take off the turpentine before destructive distillation begins and collects it in a separate receiver, are obviously at a disadvantage as far as quality is concerned. When rosin in distilled rosin spirit is produced, and this is usually found in the turpentine produced when the receiver is not changed, consequently this turpentine is not as good as it would be if it did not contain this substance. If this kind of oil can be sold at the same price as better qualities, and it has been so far, then, of course, it is not desirable to separate the rosin spirit; but the time is coming when only the pure terpene can be sold at a high price, while the mixtures must be sold for less.

At the present time it is necessary to have a colorless, agreeable smelling oil, and this should be sought for.

Special processes can be judged by their claims, and these are given in connection with the process.

Extraction processes might be made to pay after the market is established, but their final success will depend largely upon the demand for the rosin and rosin oil.

Market Conditions.—The most important problem connected with the distillation of pine and fir wood is the disposal of the products. If interested parties would spend their money advertising wood turpentine, instead of augmenting the patent office receipts by obtaining useless patents, much more could be done toward solving the problem at hand. There are plenty of good processes, and they will yield a product of the finest quality if handled right.

A great deal has been written about the ignorance of the men who produce wood turpentine, but far more could be written of the ignorance of consumers and buyers. A sample of nearly chemically pure pinene was sent to a buyer of a leading varnish company, and the sample returned as not satisfactory. Instead of testing the oil, the cork was removed and judgment passed on the odor. This oil could not be duplicated at less than twice the price of ordinary turpentine.

Complaints are made concerning the variation in quality, but there is also a variation in the quality of gum spirits. By having storage tanks of sufficient size, a standard grade from each plant can be readily obtained. But what is the use of making a very good quality of turpentine when it does not command any better price than an inferior quality? Thousands of gallons of slightly yellow oil have been sold which would have been refined if the price had warranted it. Only so much could be obtained for it, good or bad. With a standard grade made by some of the methods herein given, the large varnish and paint houses can be easily convinced of the merits of this turpentine. The painter is the most difficult to persuade; the least variation in the odor at once makes him suspicious. One painter using wood turpentine painted a house with white lead and added a Japan dryer containing some sulphuric acid so that the paint would dry quickly. The dirty looking house produced was laid to the turpentine, instead of the wrong kind of dryer.

The consideration of the market for turpentine needs no further illustrating than the fact that Chicago paint manufacturers are using thousands of gallons of it, and claim that it works better. However, there is no indication of their being willing to pay the market price for it. On the other hand, varnish makers have not found a wood turpentine but what it varies too much to suit their requirements.

What applies to the turpentine market also applies to the tar market. Here there is more excuse for complaint, for the retort tar is not always as good as it might be. With a little care, so that the resinous and oily products will be left in the tar, this product could be made of a quality satisfactory to cordage manufacturers. It should be sold more by viscosity than it is, as some tar of the required specific gravity is really too thin.

The market for tar is limited, but with the prominence now being given to steam processes, the supply is apt to be lessened, thus causing a prospect of a better price.

With charcoal the market is entirely local, although some sell to blast furnaces at a distance. The price of charcoal is easily ascertained, but the consumption cannot be easily determined.

A comparison of the cost of operating by the various processes will be given below. Different values can be substituted to suit the special conditions, the ones given below being only approximate. In the steam process using sawdust a value must be placed on the residue used for fuel, as in many cases this could be sold. A steam plant uses so much more wood in a given time that a given supply would be much sooner exhausted than with a destructive distillation plant. It is not necessary to operate a steam plant at night, as a distillation can be completed in three hours, but it is necessary to operate at night with a destructive distillation process, as it takes about twenty-two hours. In twenty-four hours a steam plant ought to distill eight times as much wood.

Steam Plant.

Plant and equipment		$25,000
Depreciation on retorts only 10%	$1,200	
Interest at 6%	1,500	
Operating expense—		
Depreciation and interest, per month	$ 233	
2080 cords wood at $3	6,240	
Labor and superintendence, $1 per cord	2,080	
Total expense per month	$8,553	

Expense per cord, $4.112.

Twelve Hour Basis.

Operating expense—	
Depreciation and interest, per month	$ 233
1040 cords wood at $3	3,120
Labor and superintendence at $1	1,040
Total per month	$4,393

Expenses per cord, $4.224.

Destructive Process.

Plant and equipment		$25,000
Depreciation on retorts 20%	$2,400	
Interest at 6%	1,500	
Operating expense—		
Depreciation and interest, per month	$ 325	
260 cords wood at $3	780	
Labor, etc.	450	
260 cords fuel at $2	520	
	$2,075	

Expense per cord, $7.98.

Yields per Cord.

RICH WOOD.		LEAN WOOD.	
15 gal. turpentine at 50c	$ 7.50	5 gal. turpentine at 50c	$ 2.50
10 gal. wood oil at 20c	2.00	3 gal. wood oil at 20c	.60
90 gal. tar at 6c	5.40	50 gal. tar at 6c	3.00
46 bu. charcoal at 10c	4.60	46 bu. charcoal at 10c	4.60
	$19.50		$10.70
Expense destructive	7.98		7.98
Profit per cord	$11.52		2.72

The steam process obtains turpentine only, so the figures would be:

	Rich Wood.	Lean Wood.
Yield	$7.50	$2.50
Expense 24 hours	4.112	4.112

Profit per cord......$3.388 Loss per cord......$1.612

On a yearly basis of 250 days.

Steam Process—24 Hours.

Fat Wood. Profit.		Lean Wood. Loss.	
20,000 cords at $3.388	$67,760	20,000 cords at $1.612	$32,240
Profit on $25,000	271%	Loss on $25,000	128.96%

12 Hours.

10,000 cords at $3.276	$32,760	10,000 cords at $1.50	$15,000
Profit on $25,000	131%	Loss on $25,000	60%

There would be considerable difficulty in obtaining 20,000 cords of wood at $3 per cord.

Destructive Process.

Fat Wood. Profit.		Lean Wood. Profit.	
2500 cords at $11.52	$28,800	2500 cords at $2.72	$6,800
Profit on $25,000	114.2%	Profit on $25,000	27.2%

Figures for a rotary retort will not be put down here, as they have not been even aproximated with this apparatus.

From the above, it can be seen that the steam process gives greater returns on fat wood than the destructive process in the same length of time. With lean wood the reverse is apparently true, and an actual loss might be expected in some cases. The apparently wonderful returns from the destructive process is due to setting the market value on products of a similar nature. However, although prospectuses relative to these plants put down these values, unfortunately, the market conditions are such that often these products cannot be disposed of at any price. This has been particularly true of wood oil until recently, when it has been disposed of to some extent in creosote paints. The charcoal is often not sold at all, or for only half the values assigned to it. At one plant, however, the prices above given have been obtained in limited quantities.

It can be seen that this problem of wood distillation is not so simple as it appears at first sight. This accounts probably for the number of patent processes that apparently solve the problem. Upon further investigation and the expenditure of considerable money, these processes, which start out so fairly, prove, in most cases, to be dismal failures. Instead of building plants on a commercial basis at the start, more experimenting should be done on a small scale until all the particular points in a given locality have been considered, then a plant should be built in units, making one unit pay before building the next, and so on until the limit is reached.

It has been found that the profits of this industry, as shown on paper before the plant is built, are far greater than the actual working results, and in many cases the loss is greater than the figured profit was expected to be.

Owing to the wide difference in proportion between the percentages of resinous products in different trees and in different localities many instances will be found where it is not possible to operate plants of this kind successfully. The variations in the yields from a cord of different pines and firs will be found in the next chapter.

Of the two processes, steam and destructive distillation, the former is probably better for fat wood and for cheap wood, such as sawdust. With wood yielding small amounts of turpentine, the steam process would not yield enough product to pay for the gathering of the wood, unless some use is found for the residue. Using the destructive process, with a good demand for tar and charcoal, it might be possible to use such a class of wood successfully. The greatest advantage of a successful destructive distillation process would be that it takes so much less wood to obtain products of the same value. With the steam process a given supply of wood is too soon exhausted.

CHAPTER X.

COMPOSITION OF WOOD AND PRODUCTS OF DISTILLATION.

Pine wood has a structure very similar to other kinds of wood, but substances peculiar to the pine family are also to be found.

Generally considered, pine wood consists of cellulose* or woody tissue, containing gums, resins, salts, sap, etc., and the outer bark. When wood is spoken of, it immediately suggests lumber or fuel, but the uses of wood in other lines will be found to be very many.

The woody fibre consists primarily of two substances, cellulose and lignin, the first having the formula n (C_4 H_{10} O_5); 100 parts containing 44.45 parts carbon, 6.17 parts of hydrogen and 49.38 of oxygen. There is also in wood recently cut a large percentage of water, amounting in some cases to as much as 45 per cent. Air-dried wood generally has about 20 per cent moisture. All this can be evaporated from the wood by heating it at 105 degrees to 110 degrees C. for a sufficient length of time, but it will reabsorb practically the same amount when again exposed to the air. Very fat pine contains less than 10 per cent moisture.

Pine wood has a specific gravity of from 0.55 to about 1.15, being in the latter case very fat or pitchy. Usually the fat wood is harder than the other and does not decay so rapidly.

The composition of nearly all kinds of wood is relatively the same, provided they be dry and contain no large amount of resins and gums. This average is 49.70 per cent carbon, 6.06 per cent hydrogen, 41.30 per cent oxygen, 1.05 per cent nitrogen and 1.80 per cent ash. This, of course, would not apply to fat pine. By extracting the resin, the fibre left would be nearer to the above composition than the original wood.

The most important part of pine wood to a distiller is the resin. This contains the turpentine and the more resin, the more oil and tar. In the pines the resin fills in the space between the cells. In other woods water is found in the porous part, but in the pine the resin takes the place of this water to a large extent. It is difficult to determine the amount of water in fat pine on account of the turpentine evaporating with the water when the wood is heated.

The resin seems to be of two kinds, that contained in the heart wood and that contained in the sap. Although the exact physiology of the formation of resin in a tree is not known, it is generally supposed to be due to the infiltration of the sap resin into the heart wood of the tree. This resin loses its fluidity and is not drawn out by tapping the tree. The difference in the odor of the turpentine produced from the tapped tree and that produced by distilling the wood itself is probably due to the odors of the different impurities in these oils, coming from the different resins. Special investigations by the Forest Service have shown that the distribution of resin throughout the tree from top to bottom follows no law, the larger amounts being found as often in the top or middle portions as in the butt-logs. Nevertheless the impression prevails that there is more resin in the stump and this impression seems to have sufficient basis to be a fact.

The cause of the formation of resin in the pine is not easy to explain; resin passages arise from the shrinking away from each other of the walls of neighboring rows of cells, an intercellular space being thus formed and gradually filling up with products of decomposition and secretion which is called resin. These resin passages are found even during germination and continue to form until the tree dies. It is claimed that this formation continues after the death of the tree, an example being given of the change of old stumps into light wood. It may be that after the tree is cut down, the sap rises each year and remains in the wood and finally when the roots die, this resinification ceases. It takes so long for a stump to decay that the evidence of the presence of additional resin after death could not be well proven unless the stump was specially marked at the time the tree was cut.

According to the evidence of Prof. Tschirch, of Switzerland, who has made recent investigations of the causes of resin secretion, the seat of resin secretion is in a mucilaginous layer lining the inner walls of the resin ducts. These ducts are present in the untapped tree, but many more of a secondary nature are formed when the tree is tapped. The latter effect seems to be an effort of nature to heal the wound. This evidently shows that the production of resin during the life of the tree is not a product of resolution.

If the resin is formed by the decomposition of the cellulose and starch while the tree is growing, the production of hydrocarbons from carbohydrates by a change in cell growth is demonstrated, as well as the production of the compounds found in the tar, by means of the destructive action of heat.

On the application of a moderate heat to the wood, this resin exudes. Considerable heat is necessary to draw all the resin on account of the capillary attraction of the woody fibre. If the heat is sufficiently increased or steam added, the turpentine will distill from the resin, and if the heat be further increased, the resin itself will distill. When not very hot, the resin will hold the turpentine, which can be removed by a subsequent distillation with steam or direct heat.

This resin contains turpentine, some pine oil, resin oil and rosin. It is soluble in ether, alcohol, carbon bisulphide, etc. Alkalies unite with it to form partially soluble compounds. Strong acids decompose it. Upon destructive distillation the turpentine and other oils distill and the rosin is decomposed into rosin spirit, rosin oil, gas and pitch. The specific gravity varies from .8 to 1.15.

After the resin is removed from wood, what is left is mostly woody fibre, consisting chiefly of cellulose.

To obtain cellulose pure from the woody fibre, it is necessary to treat the wood with various solvents such as ether, alcohol, dilute acid and alkali and finally wash with water. Cellulose is a carbohydrate and thus in this respect is similar to starch and sugar and although it cannot be converted into starch it may be changed into sugar. This feature is interesting as it is possible to make ethyl alcohol from the sugar produced.

Acids and alkalies affect cellulose; hydrochloric acid forms hydro-cellulose and wood sugar, nitric acid produces nitro-cellulose and other products of oxidation according to the strength of the acid used. Strong solutions of alkalies affect it; caustic soda being used to mercerize cotton, while melted caustic alkalies change it into oxalic acid, this being another important reaction of particular value in treating sawdust. A mixture of cupric oxide and ammonia known as Schweitzer's reagent, dissolves cellulose, and the addition of acid to this solution causes a flaky precipitate.

Viscose is a compound of cellulose formed by treating calico (cotton) with strong soda or potash and washing with alcohol. These compounds treated with carbon bisulphide $C S_2$ form thiocarbonates which are soluble in water. The viscous solution of these thiocarbonates is called viscose.

To the distiller, the most interesting feature connected with the treatment of wood is its decomposition by means of heat. The main products produced are pyroligneous acid, gas and resinous products, wood alcohol, wood oil or red oil, tar, and charcoal.

Below is given a list of the various substances that may be expected from destructive distillation of pine wood containing resin. This list will explain why refining some of the products might be expected to be difficult. Charcoal is the residue.

Gases.

Carbondioxide, carbon monoxide, hydrogen, methane, acetylene, ethylene, propylene, butylene, pentine, benzol.

Wood Oil and Tar.

Benzol, toluol, xylol, styrolene, naphthalene, retene, paraffine, dimethyl ethers of pyrogallic acid, methyl pyrogallic acid and propylpyrogallic acid, phenol, the three creosols, the xylenols (1, 3, 4 and 1, 3, 5) phloral, pyrocatechin, orthoethyl phenol, guaiacol, pyrogenous resins, pinene, sylvestrene, dipentene, amylene, hexylene, pentane, toluene hexahydride, toluene tetrahydride, xylene hexahydride, xylene tetrahydride, xylene, cumene hexahydride, cumene tetrahydride, cumene, terebenthene, cymene hexahydride, metiso-cymene, metapropylethyl benzene, dioctene, diterebentyl, diterebentylene, didecene, propionic aldehyde, furfural and methyl, furfural, methylfurfural, dimethylfurfurane, trimethyl furfurane, pyroxanthine.

Wood Vinegar and Wood Alcohol.

Furfural, formic acid, acetic acid, propionic acid, butyric, valerianic, capronic, crotonic, angelicic and caproic acids, valerolactone, pyrocatechol, methyl alcohol, methyl acetate, acetone, methyl formate, methylethylketone, allyl alcohol, dimethyl acetate, acetic aldehyde, methylamine, ethyl alcohol, hydrocoerulignon, allyl alcohol, isobutyl alcohol, isoamyl alcohol, methylpropylketone, ketopentamethylene or cyclopentanone or odipic ketone, ketohexamethylene or pimelic ketone. Alphamethyl-Beta-ketopentamethenylene, pyridine, methyl pyridine, valeraldehyde, allyl alcohol and water.

Residue.

Charcoal, containing carbon, hydrogen and oxygen and mineral matter.

When wood is first heated in a retort, only water and a little furfural are driven off until the temperature reaches 150 degrees C. With fat pine, some oil also comes over. As the temperature approaches 160 degrees C. decomposition sets in, the entire loss in weight of the wood from 150 degrees to 160 degrees being only about 2 per cent, most of which is water. This latter temperature corresponds to about 320 degrees Fah.

The following table shows, according to Violette's tests, the relative percentage loss at the different temperatures:

Temp. C.	150°	150-160°	160-170°	170-180°	150-280°	280-350°	150-430°	430-1500°
Loss per cent.	water only	2	5.5	11.4	63.8	6.5	81.	1.7

At first only a small amount of acetic acid comes over, but the percentage increases gradually until the temperature reaches about 280 degrees C., when the proportion of acetic acid diminishes. At a temperature of about 325 degrees C. (617 degrees Fah.) a sudden formation of gas takes place and the temperature increases rapidly to 375 degrees C. (707 degrees Fah.), the extra heat being caused by the decomposition of the wood.

When the temperature reaches about 430 degrees C (806 degrees Fah.) most of the volatile matter is distilled and only charcoal remains in the retort. With some woods containing large amounts of paraffines, they are not distilled under 535 degrees C. (998 degrees Fah.).

Why cellulose breaks into so many different products when distilled, it is difficult to state. The molecule is very complex, and it is probably on account of the large number of atoms.

An explanation of the process is given by Mills, in his use of the term cumulative resolution. Instances of this are very common in inorganic chemistry, one example of which he gives in the caso of manganese dioxide splitting up when heated into trimanganic tetroxide and oxygen according to the following:

$$3\,MnO_2 = Mn_3O_4 + O_2.$$

With woody fibre, water instead of oxygen is lost, and new products formed. As the temperature increases more water leaves. The following is Mill's illustration:

Cellulose Alcoholoids.	Extreme Accumulation.
$C_6H_{10}O_5$	$C_6H_8O_4$
$C_6H_8O_4$	$C_6H_6O_3$
$C_6H_6O_3$	$C_6H_4O_2$
$C_6H_4O_2$	C_6H_2O
C_6H_2O	C_6

It will be noticed that each product is formed by the loss of one molecule of water, $H_2 O$, from the compound above it.

Although this may be the mode of decomposition, the final results would be better expressed approximately as occurring as follows:

$C_6 H_{10} O_5 = 3C + 4 H_2 O + C_3 H_2 O$

Wood	Carbon	Water	Gas and Tar
100	22.2	44.5	33.3

From the gas formed something can be determined in regard to the progress of the distillation. When large proportions of oxygen are present, as in the early stages, an abundance of carbon dioxide $C O_2$ is found in the gas; later, carbon monoxide C O is found, and finally the heavier hydrocarbons and hydrogen. The presence of hydrogen is probably due to the degrading of the hydrocarbons while hot.

Methane, $C H_4$ loses hydrogen and becomes by equation

$$2 C H_4 = C_2 H_2 + H_6$$

and when very greatly heated

$$C H_4 = C + H_4$$

In a similar manner the formation of some of the other hydrocarbons found in the gaseous products may have been produced according to the following reactions:

$3 C_2 H_4 = 2 C_2 H_2 + 2 C H_4$

Ethylene Acetylene Methane

$4 C_2 H_4 = 2 C_2 H_2 + 3 C H_4 + C.$

Ethylene Acetylene Methane Carbon

$2 C H_4 + CO = C_3 H_6 + H_2O$

Ethylene Carbon Oxide Propylene Water

$10 CH_4 = C_{10} H_8 + H_{32}$

Methane Naphthalin Hydrogen

The presence of acetic acid in the distillate is supposed to be due chiefly, and the methyl alcohol wholly, to the decomposition of the vascular matter, and not of the cellulose. The properties of the many products of distillation cannot be given here, as only a few are of sufficient importance. A description of only the most valuable will be given.

Turpentine.

Oil of turpentine obtained from the gum of live trees and spoken of as gum turpentine or orchard turpentine, has a general formula of $C_{10} H_{16}$. It is comprised of a mixture of two or more terpenes, all having the same empirical formula, but varying in their constitutional or graphic formula, according to the method of bonding between the carbon atoms.

This oil should be a water white, light refracting liquid of 0.8620 to 0.8720 sp. gr. and distilling between 156 degrees and 170 degrees C. It is very soluble in ether, absolute alcohol, carbon bisulphide, essential oils, fatty oils, benzine, acetic acid, gasoline, chloroform, etc. It is only slightly soluble in water and glycerine. It oxidizes very readily to form a thick oil and becomes "fat" and has an acid reaction.

Some grades upon vaporizing increase in volume 193 times, absorbing as latent heat 74 cal. per gram. The vapor density air=1 is 5.0130. Flash point 89—94 degrees F. Sp. heat .472 boiling point 155—160 degrees C.

The uses of turpentine are quite well known. In addition to its use in medicine, it is used in paints, varnish, sealing wax, shoe blacking, etc. The three kinds of turpentine usually considered are American, French and Russian, the French oil being levo rotary and the other two dextrorotary.

Terpenes are classified according to their power of absorbing bromine. Some absorb two and some four atoms of bromine. Some do not combine with bromine at all. This variation is supposed to be due to the different ethylenic linkings. There are not as many different terpenes as was formerly supposed, but there are a large number of hemiterpenes, sesquiterpenes and polyterpenes.

In the oil from the gum the chief constituent seems to be pinene. There are three modifications of this, varying according to their action on polarized light. In American and English turpentine these rays are deflected to the right and the oil is said to be dextrorotary. In the French oil it is laevorotary. An inactive form is also found. American oil of turpentine contains both, the dextro-rotary being in excess.

This specific rotary power is determined by means of an instrument called a polariscope, the action of which can be learned by referring to treatises on chemistry and light.

Another feature of the terpenes is their power to refract light. An example of refraction is given when one end of a stick is inserted in water. The apparent bending of the stick is due to the different degrees to which the light from the stick is affected by water and air. The refraction is measured by the trigonometrical relationship of the refracting angles. This is done by means of some form of refractometer. It is spoken of as the refractive index. The working of this instrument (the refractometer) can be found described in works on oils and fats and on light.

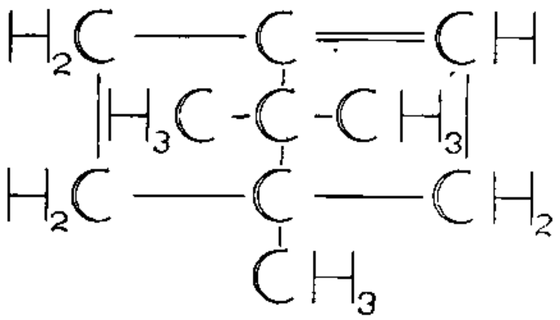
The specific rotatory power of orchard turpentine is given as being anywhere from $[\alpha]_D = -3$ to $+ 20$, and the index of refraction $N_D = 1.4682$ to 1.4737 at 20 degrees C.

Pinene.

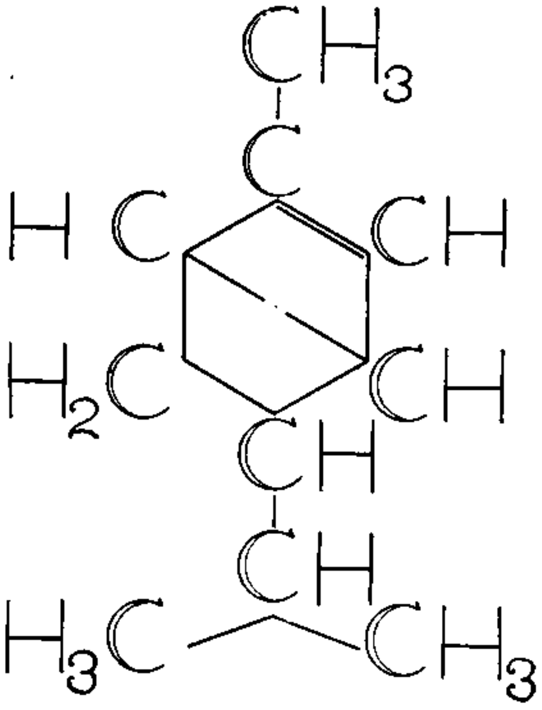
On account of the difficulty of separating the different kinds of pinene, some of the constants given are uncertain.

The boiling point is 155 degrees to 156 degrees C. and the sp. gr. at 20 degrees C. 0.858 to 0.860. Kannonikow gives as the specific rotatory power of pinene as $[\alpha]_D = + 32$ degrees for the dextro and —43.4 degrees for the laevo at 21 degrees C. Rolfe gives $[\alpha]_D = + 45.04$ and —44.95. The index of refraction at 21 degrees is $N_D = 1.46553$.

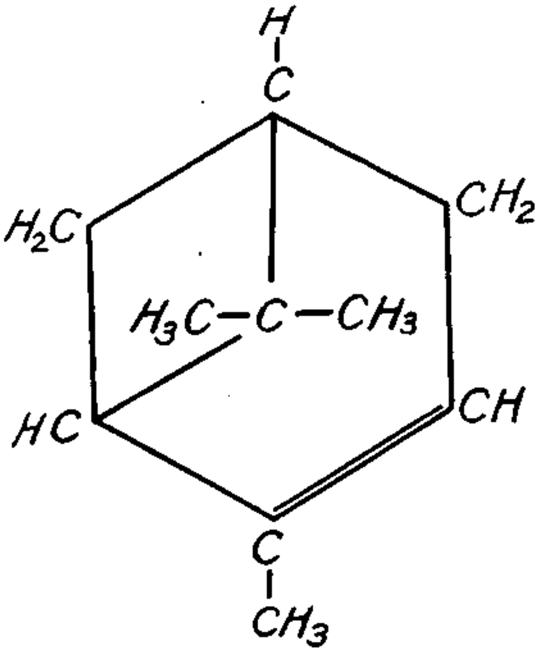
And Bredt the following:



Wallach considers that pinene has an intercalary linking and ascribes to it the formula below, which equals $C_{10} H_{16}$.



And Wagner:



Dipentene.

When pinene is heated to 250 degrees to 270 degrees C., it is converted into dipentene. This substance is inactive to polarized light. Its boiling point is 175—8 degrees C. The sp. gr. at 20 degrees C. is 0.845 and the refractive index $N_D = 1.47308$. One form prepared from caoutchouc boils at 175 degrees to 176 degrees C., and sp. gr. 0.844, and the refractive index $N_D = 1.47194$ at 20 degrees. This is a very common form of terpene and is found in many oils.

Sylvestrene.

This is found in Russian turpentine oil. It has a very agreeable odor similar to lemons and also to the oil of bergamot. It boils at 175 degrees to 176 degrees C., has the sp. gr. of 0.8480 at 20 degrees C., and the refractive index $N_D = 1.47573$, with a specific rotatory power $[\alpha]_D = + 66.32$.

The characteristic reaction of sylvestrene is shown by adding one drop of concentrated sulphuric acid to a drop of sylvestrene in acetic anhydride, when a blue color is produced. Another terpene, carvestrene, shows the same reaction. This may be inactive sylvestrene.

The other terpenes of importance will be found in the following table. The temperatures given are in degrees Celsius:

The question is, what oil is obtained when pine wood is distilled with steam or direct heat? There seems to be but little doubt that it is a terpene of the formula $C_{10}H_{16}$. The oil produced by steaming and by destructive distillation is different in some respects, and is due to the fact that the latter contains oils coming from the decomposition of the rosin. When wood is destructively distilled and the products of decomposition are all collected in the same receiver this rosin spirit and also wood oil are to be found in the distillate and cannot be satisfactorily separated.

The oil produced without decomposition of the wood gives the tests for pinene, forming a solid hydrochloride with dry hydrochloric acid gas, and having similar constants. In many cases, samples of wood turpentine show more pinene than orchard turpentine. On the other hand, bad smelling colored wood turpentine shows tests for other oils.

Tests of fir terpene produced by distillation of the Douglas fir by means of steam and direct heat are as follows, determined at the University of Minnesota:

Temp. 20 degrees C.	Steam.	Destructive.
Specific Gravity	.8621	.8662
Boiling point (degrees) C....	153.5-154	157-160
Index of refraction..........	1.47299	1.47246
Spec. Rot. Power...........	—47.2	—29.4

Terpene.	Solid or Liquid.	Modifications.	Boiling Point.	Specific Gravity.	Temp.	Specific Rotatory Power $[\alpha]_D$	Temp.	Index of Refraction.	Temp.
Pinene	......	3	155-156	0.858-0.860	20	+45.08 and — 44.95	21	1.46553	21
Camphene	S	3	160-161	0.842-0.850	54-48	..... and — 80.61	54	1.46140	54
Fenchene	......	3	155-156	0.867	20	..... and — 6.46	20	1.46900	20
Limonene	......	2	175-176	0.846	20	+106.8 and —105.0	20	1.47459	20
Dipentene	......	1	175-176	0.844	20	Inactive		1.47194	20
Sylvestrene	......	1	175-176	0.848	20	+66.32		1.47573	20
Carvestrene	......	1	178	....	..	Inactive		....	..
Terpinolene	......	1	185-190	....	..	Inactive		....	..
Phellandrene	......	2	170	0.847	19	+60.33 and —17.64	19	1.48800	19
Terpinene	......	1	179-181	0.847	..	....		1.48458	20
Thujene	......	2	170-172	0.836	22	....		1.47145	22
Synthetical Terpene...	......	1	173	0.823	18	....		1.46010	19
Fenchelene	......	1	175-178	0.842	..	....		1.47439	20
Euterpene	......	1	161-165	....	..	....		....	..
Tricyclene	S	1	153	....	..	....		....	..
Bornylene	S	1	149-150	....	..	....		....	..
Sabinene	......	1	162-170	0.840	..	....		1.46600	..

Another terpene from the Norway pine investigated at the same university gave

Temp. 20 degrees C.	Steam.	Destructive.
Specific Gravity	.5636	.8666
Boiling point (degrees) C...	153-154	158-160
Index of refraction..........	1.47127	1.47160
Specific Rot. Power.........	+17.39	+7.56

Wood terpene from different samples of oil from yellow pine:

	1	2	3	4	5
Specific Gravity...	0.865-0.867	0.862	0.862	0.863	0.864
Boiling point C...	155-157	159	156	158	156

The author made the following tests on samples of oil. One marked white oil is a wood turp, the two off-color oils were later distillates from the same charge from which the white oil was obtained. The other two were made from sawdust and fat wood. These two oils were very clear and white and apparently well refined, and had a strong odor of sawdust.

			Off-Color.		Sawdust	
Temperature.	White Oil.	Orchard Turp.	1	2	1	2
Specific Gravity 20/20......	0.8654	0.8668	0.871	0.888	0.8762 21/21	0.890
Boiling point (degrees) C..	156.5	158.25	159	157	160	167
Index of refraction........	1.4721	1.4732	1.4715	1.4748	1.4748	1.47820
Spec. Rot. Power..........	+17.91	+17.53	+17.77	17.15	+16.83	+8.99
Flash Point C.............	32	32	32	32½	38	....
Distilling under 165½......	88.50	91.00	85.00	....	32.78	....

Although the tests here given do not prove that this oil is pinene, the production of a solid hydrochloride of the formula $C_{10}H_{16}HCl$ may be considered to be a partial proof, and this has been done. Also terpin hydrate has been made from this oil.

The preparation of pinennitroso chloride and pinennitrol piperidin from this oil, as well as other compounds, ought to be as confirmatory a test as would apply to pinene from gum spirits.

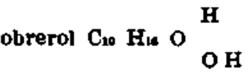
As has been noticed in many instances, the oil from destructive distillation varies greatly from that of the steam process, and is probably due to the presence of rosin spirit and wood oil.

For fuller description of the terpenes see some special work under that head.

Pine Oil.

This name is applied to wood turpentine and also to a compound to which is given the formula $C_{20}H_{16}$. This latter is supposed to be formed when pine wood is distilled at about 400 degrees C. (Pat. L. Pradon, May 1, 1883). Another so-called pine oil is produced, as stated in Clark's patent, previously described, at 240 degrees to 300 degrees Fah., and is a product of the destructive distillation of the wood. The term pine oil is also applied to all the oily products of the pine collectively. Rosin spirit is sometimes called pine oil.

Resin Oil.

It has been stated that the resin contains turpentine, pine oil (?) resin oil and rosin. After the turpentine is removed from the resinous crude turpentine obtained by distilling the wood, a thick, slightly yellow oil comes over. Although the oil is well known, no distinctive name is given to it, nor is its chemical composition known. It may be the intermediate compound between turpentine and rosin and consequently be found to contain oxygen. It might be supposed that this compound would be pinole hydrate mixed with oily matter, this pinole hydrate being the same as sobrerol  $C_{10}H_{14}O\genfrac{}{}{0pt}{}{H}{OH}$ produced by exposing oil of turpentine to the action of moist oxygen in the sunlight.

However, an investigation made at the Massachusetts Institute of Technology seemed to indicate that it was more of the nature of turpineol. Turpineol seems to be an alcohol $C_{10}H_{17}OH$, and several different compounds go by that name. One form boils at 215 degrees to 218 degrees C., and if it contained a small amount of turpentine it might be expected to boil at a lower temperature.

In the investigation above mentioned, the yellow oil was submitted to a fractional distillation. Almost the entire amount boiled between 200—214 degrees C. The fraction 209—211 constituted fully 60 per cent of the whole, and was apparently a homogeneous substance. When the oil was diluted with alcohol, saturated with dry HCl gas, and cooled, it solidified to a mass of white crystals, melting at 50 degrees C.—The indications are that it is terpineol.

Rosin.

The substance left after distilling the turpentine oil from crude gum turpentine is called rosin or colophony. This substance is also produced by carefully boiling down the resin drawn from heartwood by heat. This latter is not as good as the gum rosin, as it is colored too deeply.

Rosin is very brittle, melts at 100 degrees to 140 degrees C., and has a specific gravity of about 1.075. Alkalies convert it into a deliquescent and soluble soap called "rosin soap." It consists of abietic anhydride and abietic acid. Some consider it a mixture of hydric pinate and sylvate and consider that rosin may be an oxidation product of turpentine, as follows:

$$4C_{10}H_{16} + 3O_2 = 2C_{20}H_{30}O_2 + 2H_2O$$

Whether the oil is formed from the rosin or the rosin from the oil is not definitely known.

The oleo-resin from which ordinary rosin is produced is considered by Tschirch & Koritzschoner to consist of the following ingredients:

Palabienic acid $C_{13}H_{20}O_2$	5%
Palabietic acid $C_{20}H_{30}O_2$	6%
A and B Palabietiolic acid $C_{16}H_{24}O_2$	56%
Spirits of turpentine	20%
Paloresene	10%
Impurities, bitter principles and water	3%

Upon distillation only the oils pass over, and it would be expected the other products would remain behind practically unchanged. It doesn't seem advisable to give any definite composition to pine products as now produced, for not even the turpentine itself is of stable composition.

The distillation of the gum turpentine is performed in copper stills heated by a direct fire, hot water being added to the still from time to time in small quantities. Steam would probably be better. The temperature of the distillation is much reduced, but does not follow exactly the laws governing the distillation of two immiscible liquids. The temperature of distillation of oil of turpentine with steam when both vapors are saturated is less than 100 degrees C. After the oil is removed, the cap is taken off the still and the excess water boiled off and hot rosin run off through a cotton filter into a trough, from which it is dipped into barrels.

Rosin is stable at 150 degrees C., distillation taking place at a higher temperature (250-300 degrees). Rosin spirit or pinoline, rosin oil, gas and coke or pitch are the products of decomposition. When distilled in a vacuo or by means of superheated steam, very little decomposition takes place. Rosin can be separated from mineral oils by treating with acetone; the rosin being soluble and the mineral oils not.

Rosin Spirit.

As this substance is to be found in oil of turpentine produced by destructive distillation, some of its properties will be described here.

Rosin spirit is a very complex body produced by the destructive distillation of rosin. It boils below 250 degrees C. (78 to 250 degrees), and resembles oil of turpentine, for which it is sometimes substituted. It is now often called naphtha and amounts to about 3 per cent of the rosin charge. The spirit is found to contain a mixture of hydrocarbons and oxygenated bodies.

Professor Mills has made an examination of rosin spirit. He states that "a fraction from the spirit boiling pretty constantly at 154-156 degrees had the sp. gr. .852 at 14.4 degrees C., and almost exactly the composition of turpinol $(C_{10} H_{10})_2 H_2 O$. The turpinol of Wiggers and List is said to have the sp. gr. .852 and boil at 168 degrees C. Their product gives a crystalline hydrochloride $C_{10}H_{16}2HCl$, but rosin turpinol does not appear to do so, and is certainly not identical with ordinary turpinol. When rosin turpinol is treated with strong oil of vitriol, it yields a liquid having the odor of terebene. When treated with bromide, it furnishes an oily product, containing from 31 to 43 per cent of the reagent; chlorine is similarly taken up to the extent of 50 per cent; hydric chloride to the extent of 18 to 19 per cent. Another fraction, boiling at 188 degrees to 193 degrees and dried over sodium, agreed in composition very closely with turpentine, but it could not be made to yield a solid hydrochloride."

Renard gives a list of light hydrocarbons with low boiling point that are to be found in rosin spirit and rosin oil. Rosin spirit is water-white in color, smelling of terpene. It is generally heavier than turpentine. The sp. gr. may vary from 0.852 to 0.883 and the flash point from 96 degrees to 102 degrees F. in closed tester. It has no rotary power, one sample showing only $[\alpha]_D = +0.2$; the refractive index of same sample being 1.4780. It should not contain rosin oil. The bromide test is 184 to 213.

Rosin spirit is said to contain pentine $C_5 H_8$ (b. pt. 50 degrees), isobutylaldehyde, isobutyric, caproic and other fatty acids, methyl alcohol (50 gr. from 150 kilos.), a hydrocarbon $C_9 H_{12}$ (b. pt. about 160 degrees C.), a homolog of benzene, ordinary cymene and a new cymene (metapropyltoluene), metaisobutyltoluene (186-188 degrees), parabutyl toluene, dipentene, a large portion of a heptine $C_7 H_{12}$ (103 to 104 degrees), probably methyl propyl allene $CH_3. CH: C: CH. C_2 H_5$. This liquid is characterized by giving a succession of colors (yellow, red, green, deep blue) when agitated with strong sulphuric or hydrochloric acid. In presence of air and water, it forms a gylcol $C_7 H_{12} (OH)_2$, which crystallizes with one molecule of water in long, slender prisms, seen in old samples of resin spirit.

Rosin Oil.

This oil is produced by the destructive distillation of rosin and comprises the bulk of the distillate. The specific gravity of rosin oils ranges between 0.975 and 0.995. That ordinarily used is between 0.982 and 0.988. The iodine value averages 112 to 115. B. pt., 300 to 400 degrees C.

As much as 4 to 10 per cent unaltered rosin often distills over, and this gives an acid reaction to the oil of from .05 per cent to 5 per cent.

Rosin oils are soluble in ethyl alcohol and also in a mixture of phenol and glycerine; also in phenol alone, but not in glycerol. Alcohol with phenol dissolves it, as do carbon bisulphide and turpentine. A mixture of equal parts of phenol, alcohol and rosin oils forms a good mixture.

The action of nitric acid on rosin oil varies; some grades it attacks readily, while other grades are not affected unless heated.

Rosin oil is not truly saponified by alkalies, but unites with them to form greasy bodies. A mixture with lime solidifies soon; one with caustic soda in a few days, and with caustic potash in a longer period. A formula of $13C_{10} H_{16} Ca (OH)_2$ is ascribed to the commercial "Rosin grease."

Renard considers that about 80 per cent of rosin oil consists of diterebentyl $C_{20} H_{30}$ (b. pt. 343-346 degrees C.), 10 per cent of diterebentylene $C_{20} H_{28}$ and 10 per cent of didecene $C_{20} H_{36}$ (b. pt. 332 degrees C.).

Some consider that rosin consists of a mixture of abietic acid $C_{44} H_{64} O_5$ (m. pt. 165 degrees C) and small quantities of phenols, with a mixture of hydrocarbons $(C_{10} H_{16})_n$ (b. pt. above 360 degrees C.). Rosin oil is used as an adulterant for olive and boiled linseed oils and other oils and as a lubricant on iron bearings.

Wood Oil.

This term is applied to the first oil distilled from tar and also the oil dissolved in the pyroligneous acid.

Refined wood oil is the oil distilled from this crude oil by means of steam. There are a great many different substances found in the crude oil, a list of which has been already given. The wood oil from hardwood has been investigated by G. S. Fraps and described in the American Chemical Journal, Vol. 25, No. 1. The light oil from fir wood, which would resemble pine wood oil, has been investigated by the chemists at the University of Washington at Seattle. This can be found in Journal American Chem. Society, Vol. 25, Part II., p. 764.

The wood oil will vary according to the method of production, but it can be expected to contain the light rosin oils and light tar oils. The interesting feature to the distiller is that an oil can be produced that is very light and equal in quantity to about two-thirds the amount of the turpentine produced. This oil is generally yellow and turns darker upon exposure to the air, due probably to the presence of a tar product. The oil when first distilled from the tar, contains a large amount of creosol and some carbolic acid, both of which can be removed with soda. This oil, when oxidized sufficiently to destroy the coloring matter and then redistilled, can be made almost water white in color.

The refined oil has a distinctive odor, is a powerful solvent, a quick dryer, and can be used for outside painting. It is often used as a creosote paint when mixed with suitable pigments. In this case it is better not to remove the creosols and phenols.

It has a different composition from oil of tar produced by destructive distillation of the tar, but it contains some oil of tar, that is formed by the decomposition of the tar in the retort while the wood is distilling.

Tar.

Tar obtained by destructive distillation of pine wood in closed vessels seems to be somewhat different from the pine tar coming from a tar kiln. There are several reasons to be ascribed for this; one is that the turpentine is removed, another that the tar itself is decomposed into lighter oils and depositing coke, and another reason is that the tannin in the wood acts on the iron of the retorts and stills and causes a dark color.

In making kiln tar only extremely fat wood is used, while in retorts a poorer quality is often used. Lean woods give dark tars, sawdust tar being nearly black.

The following comparison between fir tar from a retort and Stockholm and pine tar is given below:

	Fir Tar.	Stockholm.	Pine Tar.
Color.	Black almost.	Brownish black.	Brown.
Odor.	Smoky but Characteristic.	Smoky.	Resinous.
Consistency.	Syrupy.	Syrupy.	Syrupy.
Specific gravity....	1.10	1.09	1.11
	Per cent.	Per cent.	Per cent.
Light oil	3	3	3
Creosote oil.......	34	30	40
Pyroligneous acid..	4	5	2
Pitch	59	62	53
Hardness of Pitch.	Brittle.	Less Brittle.	Soft.
Color of Pitch.	Black.	Black.	Brown.
Light oil sp. gr.	0.945.		
Color.	Amber.		

Norwegian tar, according to Knut Ström, has the following characteristics: Strongly acid, soluble in alcohol, acetic acid, ether chloroform, and benzene. Sp. gr. at 15 degrees C., 1.068. Composition 4.78 per cent volatile acids (as acetic acids), 11 per cent phenols and 61 per cent hydrocarbons. Volatile acids (85 to 90 per cent) formic and acetic; proprionic acid, normal butyric acid, normal valeric acid, (the normal valeric acid discovered by Renard in pine resin M. pt. 175.5 degrees C.), methyl propylacetic acid, normal caproic acid, oenanthylic acid and normal caprylic acid. No unsaturated acids discovered. Of the phenols, were found phenol, guaiacol, cresol, creosol, ethylguaiacol and two phenols $C_{11} H_{16} O_2$ — $C_{12} H_{14} O_2$, respectively. Of the hydrocarbons about 14 per cent solid (containing retene $C_{18} H_{18}$), and 86 per cent liquids. J. So. Chem. Ind., 1900.

The specifications required for good pine tar are given below:

	Deg. C.	Per ct.
Distilling under.....................	150	9.70
Distilling between	150-350	42.61
Distilling between	350-363	26.62
Coke		21.07

Some of the products of distillation of different tars will be found in Chapter XII.

Pitch.

There are several kinds of pitch coming from different sources, such as coal tar, wood tar and rosin pitch.

Stockholm pitch is made from pine wood tar either by boiling it down or by destructively distilling it. This pitch is brilliant black, having a concholdal fracture, but brittle, crumbling between the fingers. Its specific gravity is 1.105 and melting point 82 degrees C. It is slightly adhesive, and becomes sticky on warming; at 40 degrees C. it twists easily. When heated, 88 to 88½ per cent volatilizes, leaving a soft, friable coke containing .7 to .84 per cent ash. The odor when boiling down is very distinctive.

Benzol dissolves it, and also pyridine bases. Petroleum spirit dissolves only 91 to 92 per cent. Sulphur present, only .01 per cent. The solution in petroleum shows no bloom or fluorescence, the spectroscope cuts out only the violet spectrum, no bands visible, nor is there any indication of chrysene said to exist in this particular tar or pitch.

This pitch dissolves almost completely in alcoholic potash. This neutralized and boiled yields volatile fatty acids. (Thorpe's Dict. of Applied Chem.).

Pyroligneous Acid.

This acid comprises chiefly acetic acid, wood alcohol, acetone and dissolved oils. Other products are found and are contained in the list previously given. The amount of acetic acid from pine wood is equal to about 2.5 to 5.5 per cent of the weight of the pyroligneous acid.

Acetic Acid $C_2 H_4 O_2 = C H_3 C O_2 H$.

This acid is produced chiefly from table vinegar made from alcohol and from wood vinegar or pyroligneous acid. It is also a product of the decomposition of cellulose by alkalies and acids.

The strongest acetic acid is known as "glacial acetic" acid, from its crystallizing in icy leaflets at about 40 degrees F. Above 60 degrees the crystals fuse to a thin, colorless liquid of an exceedingly pungent and well-known odor.

The gravity of pure acetic acid is given as 1.055 to 1.060 at 59 degrees F. The sp. gr. of a solution of acetic acid in water is no indication of the amount of acid. Common acetic acid of commerce is a slightly colored liquid of about 1.04 sp. gr., and containing appromixately 30 per cent anhydrous acid.

The boiling point of pure acetic is 118 degrees C. It gives off a vapor which burns with a flame like alcohol.

The action of heat is interesting, as it is sometimes affected in a hot retort. When its vapor is passed through a red-hot tube, it yields several products, among which marsh gas and acetone are conspicuous. This action is much more marked in the presence of glowing carbon. Acetic acid is very corroding. It strongly attacks iron; wrought iron is eaten out very quickly and cast iron becomes so soft that it can be whittled with a knife. It has been found that real hot vapors affect wrought iron less than colder ones, hence we find that the hottest parts of a retort are made of wrought iron, while the connecting pipes and cooler parts of the retort, such as the head, are made of cast iron. Acetic acid attacks copper slowly to form acetate or verdigris.

Acetic acid is the strongest organic acid and is not easily oxidized. With alkalies and metallic bases it forms acetates which will not be described, except calcium acetate.

Commercial acetic acid is prepared from gray or brown acetate of lime by distilling with concentrated hydrochloric acid in copper stills, care being taken to have an excess of lime salt in the still. The acid formed is colored and contains about 50 per cent anhydrous acid. With dilute acid in the still, the acid is purer and contains only 30 per cent anhydrous acid. Often the acid is distilled in Marx vessels and filtered in towers through freshly burned charcoal.

Wood Alcohol $C H_4 O = C H_3 O H$.

This substance in a pure state is a colorless, mobile liquid, known as Columbian and Colonial spirits. It boils at 149 degrees Fah. It is very inflammable, burning with a pale flame. The pure alcohol is difficult to distinguish from grain or ethyl alcohol, as the odor and color are alike. Methyl and ethyl alcohol can be distinguished from each other by distilling them with dilute sulphuric acid and potassium dichromate, when the former yields formic acid and the latter acetic acid. The formic acid produced may be distinguished from the acetic acid by its property of reducing silver ammonio-nitrate to the metallic state when warmed with it.

Methyl alcohol forms a crystalline compound with calcium chloride ($Ca\ Cl_2$) the formula being $Ca\ Cl_2\ (C\ H_4\ O)_4$. This compound is stable at 100 degrees C., so by heating to 100 degrees C. the acetone and methyl acetate distill over from crude wood spirit, leaving this compound. By adding an equal weight of hot water this compound is decomposed, and by continuing the distillation, pure methyl alcohol distills over, accompanied by some water, which can be removed by contact with quicklime and distillation. It can also be obtained pure from wood spirit by heating with anhydrous oxalic acid in a flask connected with an inverted condenser, until the methyl alcohol is converted into methyl oxalate $(C\ O.\ O\ C\ H_3)_2$, which separates in crystals on cooling. The crystals are collected, washed with water and distilled with potash.

Commercial methyl alcohol is often slightly yellowish in color and has a disagreeable odor. It is largely used as a solvent in varnish making, the acetone contained therein being an advantage. The turbidity noticed when crude wood spirit is mixed with water is due to the separation of hydrocarbons, which were contained in the alcohol.

Wood alcohol is used for the preparation of methylated spirit or, as it is called, "denatured" alcohol, which is a mixture of grain alcohol with a small percentage of wood alcohol or other denaturizing agent.

Its production in the United States will probably be somewhat curtailed on account of the lack of demand, due to the use of "denatured" alcohol in its stead.

Acetone.

Acetone is found in wood spirit. It boils at 56.3 degrees C., and hence cannot well be removed from the alcohol by distillation over lime. To remove it, the alcohol is fixed by the calcium chloride, as described under wood alcohol, and the mixture distilled under 100 degrees C. until the acetone is driven out.

Regnault and Villejean dissolve in the wood spirit, previously purified as much as possible, 10 per cent of its weight of iodine, add concentrated solution of potassium hydroxide in small portions until decoloration is complete, and distill the mixture at a very moderate heat. The iodine and caustic unite with the acetone to form iodoform. Sometimes the alcohol is treated with chlorine and chlor-acetones are formed showing high boiling points and from which the alcohol is separated by distillation.

On a commercial scale, acetone is made by the dry distillation of gray acetate of lime at 290 degrees C. in retorts which are connected with a cooling apparatus. In Chute's process the pulverulent material is continuously conveyed in a thin film or layer over a heated surface maintained at the proper temperature, and the acetone is removed by a current of oxygen-free gas moving in an opposite direction, under a partial vacuum, the gas being reheated and reused.

A commercial method for the production of acetone devised by Dr. E. R. Squib consists of passing acetic acid vapor through a rotating iron cylinder, heated to about 500-600 degrees C., and containing pumice stone with precipitated barium carbonate. On leaving the still the vapors pass through a fractional condensation apparatus, to remove water and acetic acid; the dilute acetone condenses in a second condenser. The barium carbonate acts merely as a contact body, since the temperature is always above that at which barium acetate decomposes.

Acetone produced from acetate of lime by distillation is impure and needs refining. Sodium bisulphite is added to form a double salt with the acetone, which is readily purified by crystallization from aqueous solution. By heating this salt with sodium carbonate solution, acetone is set free and may be distilled off in a pure state. The water is removed by fused calcium chloride.

This action of sodium bisulphite can be utilized to remove acetone or other ketones from wood oils. When acetone loses the elements of water by the action of dehydrating agents such as $H_2 S O_4$ HCl and Ca O, condensation products are formed, such as mesityl oxide, a liquid smelling of peppermint and boiling at 130 degrees C. ($2(CH_3)_2 CO - H_2O$), phorone and mesitylene, all of which are found in the products of distillation of pine wood.

The sp. gr. of acetone is 0.80. It is inflammable, burning with a luminous flame. It mixes with water, alcohol and ether. When oxidized it yields acetic acid and carbon dioxide.

Acetone can be separated from a comparatively strong aqueous solution by adding concentrated calcium chloride. The acetone will rise to the top.

Calcium Acetate.

This substance is found in trade as the gray and brown acetate of lime. There is also, of course, a chemically pure salt.

The difference lies in the amount of tarry matter contained therein. Generally the yield of brown acetate of lime is one-third greater than that of the gray, but, of course, it means more impurities rather than more acetic acid.

To make acetate all the waste heat is utilized as far as possible, some plants even putting their acetate pans on top of the retorts.

It is cheaper to make brown acetate of lime, as it takes less fuel. This form of acetate is made by directly neutralizing the pyroligneous acid with lime and then distilling off the alcohol. The residual liquor is then evaporated to dryness and partially charred to destroy tarry matters. The gray acetate is made by distilling the pyroligneous acid to remove both the acetic acid and alcohol. The acetic acid vapor is passed through lime before it reaches the condenser, thus fixing it, while the alcohol vapor passes on and is condensed.

Brown acetate is made at some plants from the pyroligneous acid obtained from pine wood by simply evaporating without the recovery of the alcohol. This product does not give as much satisfaction as the hardwood variety.

As stated under destructive distillation methods, acetate is made by neutralizing the settled pyroligneous acid exactly with lime or limestone and filter-pressing to remove tarry products and impurities in the lime, etc. The fluid is then acidulated with crude hydrochloric acid and allowed to rest, whereby a deposit is formed. The clear liquor is drawn off and evaporated in copper pans heated by steam and provided with a set of stirrers to prevent the acetate from burning to the bottom. The tarry matter rising to the surface is removed through a sliding door. When the sp. gr. (measured hot) reaches 1.116, the separation of acetate begins, and gradually the mass forms a thick paste which is removed and spread on flat iron pans to be dried. During this last operation, the material should be constantly stirred with iron shovels. Some finish the drying in rooms heated with waste furnace or retort gas.

Sometimes gray acetate is made by distilling the alcohol and then after changing the receiver the acetic acid is distilled over and afterwards neutralized with lime and evaporated in the usual manner.

In the North, the acetic acid and alcohol are removed by distillation, the tar remaining in the still. The distillate goes to another still, where lime is added and the alcohol distilled off. The acetate in the still is removed and evaporated in the usual manner, forming gray acetate.

Charcoal.

The residue in the retort left after distilling wood is charcoal, and it is very important that it contains but little tar, as this will cause it to smoke.

The amount of carbon in charcoal produced at various temperatures is shown as follows:

Temperature of carbonization.		
Cent.	Fahr.	Carbon per cent
150	302	47.51
200	392	51.82
250	482	65.59
300	592	73.24
350	662	76.64
432	810	81.64
1023	1873	81.97

It will be noticed that there is little to be gained by heating above 810 degrees Fah.

Charcoal exposed to the air absorbs moisture in variable quantity, according to the temperature at which it was burned. Thus, at 150 degrees it absorbs 21 per cent of its weight; at 250 degrees, 7 per cent; at 350 degrees, 6 per cent; at 450 degrees, 4 per cent, and at about 1500 degrees C., about 2 per cent.

The kindling temperature in the open air is higher where the heat has been greater. Thus coals burned at 260 degrees to 280 degrees take fire at 340 degrees to 360 dgrees C.; those burned at 290 degrees to 350 degrees take fire at 360 degrees to 370 degrees; those at 400 degrees kindle at 400 degrees, and those at 1000 to 1500 degrees, at 600 degrees to 800 degrees, and these latter burn with difficulty.

Charcoal has great powers of absorption. It will absorb coloring matter from liquids and also absorb large quantities of gases, those gases that are most soluble in water being the most absorbed. This property is much more marked when recently charred and the air excluded.

In a box or case containing one cubic foot of charcoal may be stored a little over nine cubic feet of oxygen, representing a mechanical pressure of 126 pounds to the square inch. From the store thus preserved, the oxygen can be drawn by a small hand pump.

The composition of charcoal made at the same temperature varies with the different woods. One species of wood burned at various temperatures gave the following:

Temperature of charring.	Carbon.	Hydrogen.	Oxygen.	Ash.
270 Deg. C.	71.0	4.60	23.00	1.40
363	80.1	3.71	14.55	1.64
476	85.8	3.13	9.47	1.60
519	86.2	3.11	9.11	1.58

The specific heat of charcoal at different temperatures is given below:

0-23 Deg. C.	0-90 Deg.	0-223 Deg.
.1653	.1935	.2385

CHAPTER XI.

YIELDS AND DISPOSALS OF PRODUCTS.

The question arises, what is the yield of different products per cord of wood when distilled? It is quite apparent that the amount of different products will vary greatly, according to the different conditions. The unit is generally the cord, although the amounts and percentages are sometimes given by weight.

A cord of wood means 128 cu. ft., but it never amounts to that much; knots, crooked sticks, short ends, etc., all cause the actual quantity to be less. The amounts should be determined by weights, and even then differences occur on account of the varying content of water.

The actual per cent of wood as given by Marcus Bull, is 56 per cent solid wood and 44 per cent interstitial spaces.

The official determination in Prussia, according to B. E. Fernow, is:

	Timber cords.	Firewood cords. (over 6" diam.)	Billet cords. (over 3" diam.)
Cu. ft.	80	,75	60

	Brushwoods (less than 3" diam.)	Roots.
Cu. ft.	23.70	47.36

The amount of turpentine in the pine may be stated to vary from 0.27 to 3.50 per cent of the weight of the wood.

A complete test of light wood estimated at 6,000 lbs. to the cord was made at the Massachusetts Institute of Technology, with the following results. The turpentine was taken off with steam at 40 lbs. pressure, and the residue destructively distilled:

From steam distillation:

Product.	Amount.	Yield per cent.
Turpentine	24.9 gals.	3.00
Yellow oil	4.4 gals.	0.56
Rosin	318 lbs.	5.30

From dry distillation of the wood:

Grey acetate of lime.............	46.2 lbs.	
Light oil	18.4 gals.	2.34
Charcoal	1050 lbs.	17.50
Gas	3750 cu. ft. (?)	2.00
Wood tar	1217 lbs.	20.23

From distillation of rosin:

Rosin spirit	2.5 gals.	0.3
Rosin oil	10.9 gals.	1.5
Blue oil	7.25 gals.	1.0
Green oil	5.6 gals.	0.8
Pitch	12 lbs.	0.2

From distillation of wood tar:

Creosote oil (15 per cent)........	306 lbs.	5.1
Wood pitch	516 lbs.	8.6

From creosote oil:

Creosote	45.9 lbs.	0.76

Specific gravity of the wood, 1.075.

A test on 600 lbs. of dry light wood gave the following:

	Pounds.
Spirits of turpentine	21¼
Pyroligneous acid	95
Heavy oils and tar	150
Charcoal	127
Water and gas	206¾
Total	600

This is supposed to equal a yield by the cord of 24 gallons spirits of turpentine, 88 gallons of pyroligneous acid, 120 gallons tarry and heavier oily products and 56 bushels of charcoal.

One of the earlier plants gives the following from a cord: Spirits of turpentine 5 to 18 gallons, of heavier oils and tarry products known as dead oil or creosote from 60 to 100 gallons, and of stronger acid (of a specific gravity 1.02) 60 gallons, or of weaker acid 120 gallons.

An analysis given by Prof. Cox is as follows: First quality, turpentine, 16 gallons; second quality, turpentine, 10 gallons; alcohol, 6 gallons; acetate of lime, 160 lbs.; tar, 1 barrel.

Another from a cord weighing 5,000 pounds, 8½ to 9 gallons turpentine, a barrel to a barrel and a quarter of tar, about a gallon and a half of alcohol and about 170 gallons pyroligneous acid.

Another more detailed, the results of investigations made by Mr. J. D. Lacey, of New Orleans:

Turpentine	22 gals.
Pine Tar	75 gals.
Wood alcohol	2 gals.
Lime acetate	40 lbs.
Charcoal	48 bu.

Another:

Russian turpentine	16 gals.
Rosin oil	42 gals., kidney.
Rosin oil	8 gals., heavy.
Creosote	10 gals.
Wood alcohol	4 gals.
Acetate of lime	100 lbs.
Charcoal	500 lbs.

No one has experimented with wood of varying fatness, using large quantities.

The author made a test on some long leaf yellow pine cut into short lengths. These short lengths, after cording carefully, measured 2.36 cords and weighed 8,614 lbs., or about 3,647 lbs. per cord.

The yield per cord, air dry, was as follows:

		Weight, lbs.	%
Clear white turpentine	18.64 gals.	134.20	3.679
Wood oil	11.09 gals.	86.50	2.371
Tar	96. gals.	846.72	23.216
Acid	95.9 gals.	830.49	22.771
Coke		14.74	.404
Charcoal		796.00	21.826
Yellow oil and pitch	6.78 gals.	57.02	1.563
Gas and loss		881.33	24.166
Total		3647.00	99.996

The turpentine, after distilling, was a clear white oil, testing as follows: Sp. gr. at 20 degrees C., 0.8654-B. Pt. 156.5 degrees C. Index of refraction, 1.47210, sp. rot. power plus 17.91 at 20 degrees C., flash point 32 degrees C. (closed tester).

The turpentine was taken off by means of superheated steam under a pressure of only a few ounces, then redistilled, yielding the above quantity of oil. The residue was then destructively distilled and the wood oil steamed from the tar. The wood oil contained no turpentine, but did contain large quantities of rosin oil and creosote oil.

In Minnesota, with Norway pines, the yields are said to be:

Turpentine	16 gals.
Tar	30 gals.
Charcoal	30 bu.

On the Pacific coast, with fir, the yields are about the same as in Minnesota on the same class of wood.

With sawdust the yield varies with the amount of rosin in the wood, from ½ gallon to 5 gallons per ton, air dry. With fat slabs the yield would be about 24 gallons, or less, according to richness.

The yield depends entirely upon the resin, as nearly all processes will extract the oils if given time and heat enough. In destructive distillation processes it can be readily understood that too rapid heating causes more gas at the expense of the other products.

In the hardwood industry one plant using 70 per cent maple averaged per cord as follows during the year 1906, from wood four foot long:

Wood alcohol	11.32 gals., 82%
Acetate of lime	172.56 lbs.
Charcoal	54.18 bu.

In the United States, by weight, for hardwood, the following are given as the average results in per cent:

Wood alcohol	1.434	
Acetate of lime	6.250	82%
Charcoal	31.2	

In Germany the results from a cord of pine are as follows:

Turpentine	12.25 gals.
Brown acetate	88.82 lbs.
Tar	40.4 gals.
Wood alcohol 82%	4.1 gals.
Charcoal	808.0 lbs.

The following table of yields is given by the Bureau of Chemistry and gives an idea of the variableness of the yields from different woods.

AMOUNT OF PRODUCTS YIELDED PER CORD OF WOOD.

Classes of woods.	Charcoal. Bushels.	Alcohol (crude) containing acetone. Gallons.	Acetate of lime. Pounds.	Tar. Gallons.	Wood oils. Gallons.	Turpentine. Gallons.	Gas.
Hard woods	40 to 50	8 to 12	150 to 200	8 to 20	....	....	....
Resinous woods	25 to 40	2 to 4	50 to 100	30 to 60	30 to 60	a12 to 25 b2 to 10	....
Sawdust (hardwood)	25 to 35	2 to 4	45 to 75	....	....	....	....

a Lightwood. *b* Sawdust.

With pine, the yield in turpentine by steam distillation is, with ordinary pine 2 to 5 gallons per cord, while good light wood yields from 10 to 20 gallons and averages about 15 gallons per cord, and very rich light wood from 20 to 30 gallons per cord.

Unfortunately, the greater amount of the available pine is of the low yielding variety and the supply of good light wood at most plants is soon exhausted, thus causing failure.

RESULTS FROM DIFFERENT METHODS OF CHARCOAL MAKING.

Coaling Methods.	Character of woods used.	In volume per cent.	In weight per cent.	Bushels of Charcoal per cord of wood.	Weight in lbs. per bushel of charcoal.
Odelstjerna's experiments: birch dried at 230 F.		..	35.9	....	....
Mathieu's retorts, fuel excluded, (air dried, average good yellow pine, weighing about 28 lbs. per cu. ft.)		77	28.3	63.4	15.7
Mathieu's retorts, fuel included, (air dried, average good yellow pine, weighing about 28 lbs. per cu. ft.)		65.8	24.2	54.2	15.7
Swedish ovens, average results, (good dry fir and pine mixed)		81.0	27.7	66.7	13.3
Swedish ovens, average results, (poor wood mixed fir and pine)		70.0	25.8	62	13.3
Swedish meilers, exceptional (fir and white pine wood mixed, average 25 lbs. per cu. ft.)		72.2	24.7	59.5	13.3
Swedish meilers, average results		52.5	18.3	43.9	13.3
American kilns, average results, (average good yellow pine weighing about 25 lbs. per cu. ft.)		54.7	22.0	45	17.5
American meilers, average results, (average good yellow pine, weighing about 25 lbs. per cu. ft.)		42.9	17.1	35	17.5

DISPOSAL OF THE PRODUCTS.

With the demand for turpentine increasing at a rapid rate, it would seem an easy proposition to dispose of some of the products of distillation. However, the prejudice against wood turpentine and retort tar is still very great.

Turpentine.—Turpentine ought to be sold as such upon its merits. It is necessary to make a refined article of stable quality in order to command a ready sale, even at a less price than the market price of orchard turpentine.

Any surplus might be disposed of by converting into turpentine derivatives, such as terpine hydrate, terpineol (used for making lilac perfume), camphor, artificial camphor and similar compounds.

Yellow Oil.—This compound, if it proves to be terpineol, may be changed into numerous bodies. In the present state of knowledge regarding this product it may be used in combination with caustic as a disinfectant, for stack paint or for mixing with the residue for kindling.

Wood Oil.—The refined wood oil can be mixed with suitable colors to form shingle stains. The crude wood oil can be used for dark colored shingle stains and for creosote paints.

The pyroligneous acid is sometimes used as a sheep dip, disinfectant, spray on trees, etc. It can be converted into acetate of lime and black liquor (iron acetate), which latter is used as a mordant for dyeing, and can also be used for a black stain on wood.

Tar oil, the name applied to the crude tar and oil coming from the retort, is used in various ways, as for soap, etc., also for creosoting lumber.

Tar.—This is generally sold as such, but may be regularly distilled to produce oil of tar, creosote oil and pitch. Black tar and pitch might be used in road-making and for making products where coal tar is now used.

Rosin and Resin.—These are best sold as such, or mixed with the tar. A separate plant might be added to destructively distill to obtain rosin spirit, rosin oil and pitch. The rosin itself might be sold to ship chandlers, sealing wax manufacturers, etc., or when the residue from the steam process is used for making gas, this can be added to the wood to make rosin gas.

Charcoal.—The two chief methods of disposing of this are domestic use and for blast furnace consumption. In the latter case the advisability of encouraging the use of brands or half-charred pieces is to be commended. In this way destructive processes do not take so long, the tar is better and the weight of the brands is greater than that of the charcoal.

The coke on the bottom of the retort contains considerable ash. Probably the best use of this is for fuel, although at many plants it is thought that this material won't burn. It has been suggested to grind up this material and use it for similar purposes as gas carbon.

The production of this coke should be avoided as far as possible by careful operation.

Residue from the Steam Process.—The proper disposal of this residue at satisfactory prices will help solve the problem of the utilization of waste pine wood. The following are suggestions: Sidewalks in small towns (used as such in Germany); fuel, wall-plaster, kindling (by mixing with rosin or yellow oil); destructive distillation in special retorts for tar products and gas-making; oxalic acid, ethyl alcohol and cellulose products.

Many attempts are being made to find a process that will enable the residue to be converted into paper. So far everything designed is the reverse of what it should be. The paper industry is more important than the pine wood distillation industry can ever hope to be. Instead of devising a process for making the residue from a turpentine process into paper, a process for making paper which will incidentally extract the turpentine is the process that is required.

In making paper the resin, which is of importance to the distiller, is a detriment to the paper-maker. In making turpentine from pine wood only fat wood is used, except in a few instances. The bulk of the waste wood is not fat, consequently only in those cases where the waste is cheap, such as sawdust, can any distillation process extract enough turpentine to pay for gathering the wood. Unless then, the extraction of the turpentine pays for itself, there is nothing to be gained in this manner, for the paper manufacturer can obtain his stock as cheaply without the turpentine being extracted.

To utilize waste wood by converting it into paper, the proper place to extract the turpentine would be at the digester of the paper mill, instead of at the digester or retort of a turpentine apparatus.

The author visited a paper mill using yellow pine and found that all that was necessary to recover the turpentine was the placing of a condenser and a valved connecting pipe at the top of the digester. Using the soda process of paper-making, the reaction in the digester is carried out at a pressure of 90 to 110 lbs., the heating being done by means of steam. This action thoroughly extracts the turpentine, more so than many of the processes herein described, as the heat is contiued longer. The action of the caustic is not necessarily detrimental, as it combines with any tarry matters.

With a digester fitted with a condenser, all that is necessary is to open the valve leading to the condenser, and the oil, mingled with water, will flow out from the end of the condenser. The oil is found to be slightly yellow, the same as ordinary crude oil produced from steam pressure processes. By a simple redistillation this can be made white.

If all classes of waste pine wood could be made into a quality of paper that it would pay to make, then the above method of extracting the turpentine while making paper solves the problem of the utilization of the waste wood from pine and fir wood. The question is up to the paper manufacturer, and not to the wood distiller.

An exception may be made to the above method in the case of resinous wood, but only a small quantity of the waste is fat. However, fat wood could pass through the above process and be as good as it would be if it passed through any of the regular distilling processes, except those that extract the resin by other means than caustic. With fat wood more caustic would be needed in the digester in order to remove the excess of resin, and this alkali could be recovered in the usual way, or with very fat wood, might be neutralized and the resin recovered. The turpentine would come off as usual. The quality of the paper would be the same, whether the oil was recovered in a regular distilling apparatus or in the digester of the paper mill. It can be readily understood that the pressure in the digester can be low while the turpentine is being taken off, if desired. Attempts are being made in New York State to recover turpentine from spruce, and the above method, if tried, will be found the most suitable.

CHAPTER XII.

CHEMICAL TESTS AND COMBINATIONS.

In this chapter will be recorded some of the tests and analyses of the different products as made by various investigators. Some have already been given, and they will not be repeated. In addition will be given methods of combining the different products in order to produce some of the various derivatives.

Turpentine.—The results of the test applied to the turpentine produced at the Massachusetts Institute of Technology by distilling fat wood with steam are given below:

Specific gravity	0.865—0.807
Color some samples	Water white
Spec. rot. power	+ 28.7
Acid values	.0077 to .0079%
Esters as bormyl acetate	7%
Distilling below 163 degrees C.	80%
Distilling below 175 degrees C.	90%
Residue upon evaporation	0.71 to 1.02%

(For others see Chapter X.)

Tar.—Several statements have already been made relative to the tests that have been made on tar. The following are distillation tests on various tars:

PARTS.

	Acid	Light Oil	Heavy Oil	Pitch	Gas and loss
Meiler tar from So. Austrian Black fir sp. gr. 1.075	10	10	15	50	5
Specific gravity		(.966)	(1.014)		
Meiler tar from Bohemian pine sp. gr. 1.116	10	5	15	65	5
Specific gravity		(.977)	(1.021)		
Retort tar from Salzburg sp. gr. 1.18	10	10	15	55	10
Specific gravity		(1.012)	(1.022)		
Tar from distillation process by superheated steam	5	20	25	30	5
Specific gravity		(.920)	(.978)		

Hardwoods give on an average a tar which by distillation yields according to Vincent:

Watery distillate (wood spirit, acetic acid)	10 to 20%
Oleaginous light distillate sp. gr. 0.966 to 0.977	10 to 15%
Oleaginous heavy distillate sp. gr. 1.014 to 1.021	10 to 15%
Pitch	50 to 65%

ANALYSIS OF CHARCOAL.

Product.	Heat °C.	Carbon,	Hydrogen.	Oxygen & Loss	Ash
Dry wood	150	45.71	6.12	46.29	.08
Charred wood	260	67.85	5.04	26.49	.56
Red charcoal	280	72.64	4.70	22.10	.57
Brown charcoal	320	73.57	4.83	21.09	.52
Dull black	340	75.20	4.41	19.96	.48
Lustre black	432	81.64	1.96	15.25	1.16
Extreme white heat	1500	96.52	0.62	0.94	1.95

GAS.

Analyses of wood gas made by Pettenkofer show:

	Carbonic Acid	Carbonic Oxide	Methane	Hydrogen	Heavy Hydrocarbons
Per cent	18 to 25	40 to 50	8 to 12	14 to 17	6 to 7

Some gas purified gave:

	Carbonic Oxide	Hydrogen	Marsh gas	Heavy Hydrocarbons
Per cent	25 to 40	29 to 49	24 to 35	7 to 9

An analysis of the gas from distilling fir made at the University of Washington, Seattle, Wash., gave the following results:

	Carbon Dioxide	Carbon Monoxide	Hydrogen	Methane	Light hydrocarbon	Nitrogen
Per cent	12.3	28.8	30.6	7.3	6.3	14.7

From analyses of gas generator gases:

Carbonic acid	10.00	10.70	15.20
Carbonic oxide	18.50	17.90	15.40
Methane	0.70	3.10	7.20
Hydrogen	17.40	17.60	12.60
Nitrogen	53.40	50.70	49.60

The gas from yellow pine contains all the above ingredients and also some that are characteristic of rosin gas. A gas made from Virginia pine by

Pettenkofer's process, which consists in charring the wood and then pushing the charcoal formed to the back of the retort and letting the water from fresh wood pass through it while hot, analyzed as follows: Hydrogen, 44 per cent; marsh gas, 5.4 per cent; carbon monoxide, 33.7 per cent; carbon dioxide, 10.5 per cent; nitrogen, 6 per cent; oxygen, 0.25 per cent. A ton of wood (2,240 lbs.) is said to produce 36,500 cu. ft. of such gas.

Ordinary hardwood gas has tested 26 per cent CO_2 40 per cent CO and 11 per cent marsh gas, the remainder being hydrogen and hydrocarbons. A purified gas showed as follows: 25 per cent marsh gas, 30 per cent hydrogen, 30 per cent carbon monoxide, 8 per cent hydrocarbons and 7 per cent carbon dioxide and air. It is difficult to purify over lime.

Tests of Pyroligneous Acid, Rosin, Oils, Etc.— The composition of these compounds has been stated under the description of the same, and it is not necessary to repeat them at this place. It is well to again call attention to the fact that those woods which are hard containing relatively large amounts of lignin and incrusting substances give larger yields of acetic acid and methyl alcohol than those woods containing but small amounts of this harder material and consequently called soft wood.

Combinations or Derivatives.

Turpentine Derivatives.

Camphor $C_{10}H_{16}O$.

The most important derivative of turpentine is camphor. To prepare two general methods are pursued: one is to treat the turpentine direct with suitable reagents, and the other is to first change the pinene into pinene hydrochloride from which camphor can be made.

The first method was tried unsuccessfully in this country, but it is of interest nevertheless. The process used was that of Thurlow, which consists in heating the turpentine with anhydrous oxalic acid at a temperature below 120 degrees C.

According to Collins, the process is carried out on a small scale, as follows : In a steam-jacketed reaction tank, oil of turpentine and anhydrous oxalic acid are placed, the results of the reaction being pinyl oxalate and pinyl formate. The liquid mass formed is pumped into a set of stills for treatment. Here it is distilled with live steam in the presence of an alkali, the resultant formation occurring as ordinary camphor and borneol camphor dissolved in the oily products of the reaction. These oils are fractionally distilled to extract the camphor and borneol further. After the pleasant smelling oils have passed over, the camphor and borneol distill in the steam and are precipitated in the condenser in a white mass somewhat resembling boiled rice. The crude product is then forced by compressed air through a filter press and thoroughly washed to free it from all traces of oil, when it is dropped into an oxidizing tank, where the borneol oxidizes into the ordinary camphor.

The mass is transferred to a rapidly revolving centrifugal machine, where the oxidizing liquors are thrown out and the camphor, being heavier, remains behind comparatively pure, but stained from the oxidizing compound, so that it resembles light brown sugar. After removal from the separator it is placed in a large steam jacketed sublimer. In this vessel a slow heat frees it from any water it may contain, and the temperature is then raised to the boiling point of camphor and a rapid current of air projected over the surface of the pan, blowing the camphor into a condensing chamber, where it settles in the form of snowflake-like crystals.

The yield of camphor by this process is from 25 to 30 per cent of the weight of the turpentine used. In addition to camphor, there are a number of light oils produced in the process, which are also found in nature, namely, dipentene, oil of lemon, oil of lime and a number of other natural terpenes and essential oils. This process of synthetically producing camphor takes about fifteen hours.

In most of the other processes pinene hydrochloride is formed by passing dry hydrochloric acid gas into dry turpentine oil, both being well cooled. If a rise in temperature is prevented during the reaction the oil solidifies almost completely after saturation with the gas to a camphorlike mass. Some pass the gas into a mixture of turpentine and chloroform and distill off the chloroform and then the hydrochloride.

This product is called artificial camphor $C_{10} H_{16}$. H Cl. It melts at 125 degrees C. and boils at 208 degrees, suffering almost no decomposition.

The conversion of this product into camphor is based on the fact that when the hydrochloric acid is removed by a feeble alkali, such as aniline, or by means of sodium acetate and acetic acid, the compound formed is not pinene, as would be expected, but camphene, a terpene closely allied to camphor, which, by oxidation, can be converted into camphor.

A process patented in this country uses lime to remove the chlorine and then oxidizes the camphene with nitric acid. It is doubtful if much camphor would be produced when lime is used.

A general method for the production of camphene is to heat pinene hydrobromide or hydrochloride with sodium acetate and glacial acetic acid at 200 degrees C.

The oxidation of the camphene to camphor may be performed in the following manner, using isoborneol as an intermediary:

Two hundred and fifty parts by weight of glacial acetic acid and ten parts by weight of 50 per cent sulphuric acid are mixed with 100 parts by weight of camphene and the whole heated to 50 degrees to 60 degrees C. for a few hours and the mixture frequently agitated. Two layers are formed at first, but after a short time a perfectly clear slightly colored or colorless solution results. The reaction is complete in two or three hours and the product is diluted with water, the resultant isoborneol acetate separating as an oil. This is washed a few times to remove the free acid, and without further purification it is boiled for a short time with a solution of 60 parts by weight of potassium hydroxide in 250 parts by weight of ethyl alcohol in a still connected with a reversed condenser. The greater part of the alcohol is then distilled off and the residue poured into a large quantity of water; isoborneol is precipitated as a solid mass, which can be separated pure by filtering and recrystallizing from petroleum ether. The isoborneol is then oxidized with just enough nitric acid, or with a solution of chromic anhydride in glacial acetic acid, the resulting product being camphor.

Instead of using isoborneol itself, a German process starts with isoborneol acetate or benzoate. The oxidation may be performed, for instance, by means of chromic acid, nitric acid, permanganate, manganese and sulphuric acid, Caro's acid, etc., working either in solution or in suspension. The following formulas are given:

Ex. 1.—One hundred and twenty-seven parts by weight of isoborneol acetate are dissolved in 2,000 parts by weight of glacial acetic acid or other suitable acid, which is not affected by the oxidizing agent, and then oxidized with 78 parts by weight of chromic acid. The reaction being completed, the excess of solvent is distilled off, the residue washed out with water and purified in the usual manner.

Ex. 2.—One hundred and twentyseven parts by weight of isobornyl acetate are well mixed with 78 parts by weight of chromic acid in 2,000 parts by weight of water, at about 90 degrees C., until no more free chromic acid is present. After cooling the raw camphor crystallizes out and is then purified in the usual way.

Ex. 3.—One hundred and seventy parts by weight of isobornyl benzoate are well mixed with 78 parts by weight of chromic acid in 2,000 parts by weight of water at a temperature of about 90 degrees centigrade for so long as no more free chromic acid can be identified. After cooling, the former raw camphor is separated from the adhering benzoic acid by boiling with alkalies, and is further purified in the usual way.

There are a great many patented processes using the hydrochloride as a basis. One decomposes the hydrochloride with phenols, etc., and oxidizes the camphene in the usual manner.

Another dries the turpentine with calcium carbid and then treats slowly with dry H Cl gas at 30 degrees C. The compound thus formed is heated to 180 degrees C. with a metal and oxidizing agent, such as zinc and barium peroxide and sodium and peroxide. It is stated that when manganese dioxide is used alone no metal is necessary.

Also from camphene when treated with chromic acid mixture at 180 degrees, ozonide ($C_{10} H_{16} O_3$) is formed, and this treated with water loses oxygen to form a lactone, camphenolide, and by heating this compound in presence of water, camphor is formed.

Terpine Hydrate $C_{10} H_{18} (OH)_2 + H_2 O$.—The following is Hempel's method of production: A mixture of eight parts of turpentine oil, two parts of alcohol and two parts of nitric acid of sp. gr. 1.255 is well mixed and placed in flat basins. After standing for a few days the mother liquor is poured off from the crystals of terpine hydrate, and neutralized with an alkali, after which a second crop of crystals separate. Terpine hydrate can be found in any pharmacy.

Cineole $C_{10} H_{18} O$.—This is found in the oil of eucalyptus and other oils. In making the above compound (terpine hydrate) the mother liquor is found to contain this product. Its formation is probably due to the action of the dilute acid, on terpine hydrate and terpineol. By distilling the mother liquor from the manufacture of terpine hydrate with steam and cooling to a low temperature the oil found in the distillate, the cineole will separate, or by treating the distilled oil with concentrated phosphoric acid and neutralizing the resulting compound with an alkali cineole will be produced. Cineole is a liquid with an odor resembling camphor. It has a specific gravity 0.930 at 15 degrees C. and a refractive index, $N_D = 1.45961$ at 17 degrees.

Terpineol $C_{10} H_{17} O H$.—It has been mentioned that the yellow oil left after distilling the turpentine from the crude oil from the steam distillation of pine wood may be a terpineol. There are three of these compounds recognized and one form is solid. Terpineol is produced from terpine hydrate by the action of dilute acids. It may also be produced by the action of formic acid on geraniol at a temperature of 15 degrees to 20 degrees. The terpinyl formate formed is changed to terpineol by hydrolysis. Terpineol is found in many oils.

A more extended description of these derivatives cannot be given here. They are given to illustrate the possibilities of utilizing the turpentine if the market prejudice continues to exist.

Rosin Derivatives.—It has been stated that the rosin can be employed in many ways, one of which is by distilling it for other products. The action of heat on rosin should be known by a pine wood distiller, so the following methods are given:

A still or retort may be a vertically placed wrought iron cylinder, or the cylinder may be placed horizontally. Some use a regular still shaped like a coal tar still. Whatever shape is used is connected with a suitable copper condenser and heated by means of a direct fire. The size of the still varies, but usually is large enough to contain fifty to seventy barrels of rosin.

The flow commences in about one to one and a half hours after firing, and continues until there only remains in the still a charry mass resembling coal. The distillation period covers a space of about twenty-four hours.

The best oil is obtained between the fifth and twenty-second hours running, and is of a pale yellowish brown color. It then begins to darken, and after an hour or an hour and a half the result is a very black gumming oil. The firing is generally stopped before this time on account of the difficulty in removing the residuum from the bottom of the still.

According to Renard, that portion of the distillate boiling under 360 degrees C. is called rosin spirit and that which boils above 360 degrees C. is called rosin oil. This point is also marked by the falling off in the quantity of the distillate and by the specific gravity of the distillate showing about .951. The residuum when not completely charred is called pitch and the last product coke. The gas produced is very heavy and a powerful anaesthetic, containing carbonic oxides, ethylene, butylene and pentine.

The products from a seventy-barrel still are given below:

Name of product.	Amounts	Yield %
Rosin spirit	60-70 gals.	3.1
Rosin oil	1600 gals.	85.1
Coke	600-700 gals.	3.9
Acid and water	40-50 gals.	2.5
Gas and loss		5.4

The yield from an actual run made at a plant in Pennsylvania were as follows:

Amount of rosin		5660 lbs.		Per ct.
Naphtha or spirit	about	100 lbs.	approx.	1.75
Raw oil or rosin oil	about	4960 lbs.	approx.	87.60
Water	about	47 lbs.	approx.	0.80
Pitch	about	210 lbs.	approx.	3.70
Gases, loss, etc.	about	350 lbs.	approx.	6.20

Apparently, this run shows more oil and less spirit than the seventy-barrel charge, but the raw oil upon boiling gave off about 6.2 per cent of water and naphtha.

In Russian plants about 1½ per cent of lime is added to the rosin in the still.

The pine oil or rosin spirit is refined in a manner very similar to turpentine, by simple washing with water and redistilling once or twice. Sometimes caustic soda, or other alkali is added before distilling, in order to remove rosin oils and acid. Most of the rosin oil on the market is obtained by one distillation. It is improved by redistilling in the same manner as it was obtained. Sometimes the oil is distilled three times after coming from the first still, each grade being known as first (from the rosin), second, third and fourth run.

Formerly gas of high quality was made from the rosin, 100 lbs. furnishing 1,300 cu. ft. Rosin spirit is used as a substitute for the oil of turpentine and is known as pinolene and pine oil.

Rosin oil is about one-fourth soluble in soda solution, the other three-quarters being comprised largely of hydrocarbons (above 360 degrees C.).

To make rosin grease, a smooth cream of slacked lime and water is first prepared and a small portion of the oil is mixed with this in the proportion of about four parts of oil to three parts of slacked lime. Oil is then added to the greasy semi-solid mass until of the required consistency. The finished grease is often composed of about 1 part of lime to 20 to 25 of oil. Various terms are applied to crude oil, such as blue, green, red, kidney and heavy, according to the characteristics thereof.

The pitch formed by the destructive distillation of rosin, as well as that made by boiling down tar, is the ordinary pitch of commerce. Rosin pitch is different from tar pitch in color, properties and composition, yet it is near enough like it for most practical purposes. It is yellowish brown, brittle, compact, easily crumbling between the fingers. Its specific gravity is 1.09 and it melts at 68 degrees C. It loses 82½ per cent on heating, leaving a spongy, soft coke. It has an odor of rosin when heated. It is soluble in benzine and pyridin.

Wood Residues.

Oxalic Acid.—The action of strong alkalies upon wood is to convert the cellulose into oxalic acid, which combines with the alkali to form an oxalate. There are several methods of making oxalic acid by this general process, varying in the proportion and kind of alkali used and the method of converting the resulting oxalate into oxalic acid. A mixture of caustic potash and caustic soda, 40 parts K O H and 60 parts of Na O H, seems to be the cheapest proportion of alkali. Soft woods give the larger yield, thin layers are better than thick layers. It would seem to be better to use those proportions of wood and alkali as would give the largest percentage of oxalic acid as compared with the amount of alkali used, but difficulties in the working of the process allow of not more than 50 parts of sawdust to 100 of alkali. The oxalic acid is formed chiefly from the cellulose of the wood and not from the lignin.

A detailed description of the making of oxalic acid can be found elsewhere. A general description of one process only will be given here.

Fine sawdust is gradually added to a strong solution of 1½ parts caustic potash and one part caustic soda, contained in iron pans. The mixture is then evaporated with constant stirring so as to obtain a moist, powdery residue. At first only water is removed but gradually the mass turns darker and the wood begins to decompose and emit a pungent odor. At a temperature of about 180 C. the mass becomes a greenish yellow. The temperature should be gradually raised to about 240 C. and held there until the wood is dissolved, the total time being about six hours. The resulting material consists of a mixture of sodium and potassium oxalates and carbonates with some impurities from the decomposition of the wood, which gives it a distinct color.

To extract the oxalate the material is thrown into iron filter boxes with wire gauge false bottoms, and the potassium washed out with water drawn through the mass by means of a vacuum pump. The residue consists of sodium oxalate, which is decomposed by heating with milk of lime in an iron pan supplied with a horizontal stirrer, calcium oxalate and caustic soda being formed. By evaporating the soda lye it can be used again. The calcium oxalate after it has been washed with water is decomposed by sulphuric acid in wooden vats lined with lead.

The potash salts washed out from the crude oxalate in the first operation are also boiled with lime to recover the caustic potash, which is also used again. The oxalic acid formed by decomposing the calcium oxalate with sulphuric acid is evaporated and crystallized repeatedly in lead pans until sufficiently free from sulphuric acid. The mother liquor mixed with more sulphuric acid is used to decompose more calcium oxalate.

Ethyl Alcohol.—The preparation of grain alcohol from wood has long been attempted. When cellulose is treated with dilute acid it is converted into a fermentable sugar. Soft woods are the best, as they contain relatively more cellulose. So far, a great many difficulties have been encountered in the practical manufacture of spirits which have not been entirely overcome. The acid used may be suitable for the production of the sugar, but when the acid is neutralized with lime the resulting product interferes with the fermenting; when sulphuric acid is used it seems to prevent, in a measure, the conversion of the cellulose into alcohol, although the calcium sulphate formed does not interfere with the fermentation. The following conditions seem to be necessary to obtain the best results with ordinary mineral acids and soft woods:

	Wood Cellulose (bisulphite)	Pine Wood.
Proportion of total liquid	6 times wt. of cellulose	5 times wt. of wood
Concentration of acid	0.5 per cent H_2SO_4	0.6% H_2SO_4
Pressure	10 atmospheres	9 atmospheres
Duration of digestion	1½ hours	15 minutes
Yield sugar (Fehling's test)	41 per cent	20% of wood
Fermentation	Free	Variable
Yield of alcohol from sugar	70% of theoretical	60% highest

Under properly controlled conditions, one long ton of wood should yield a little over 17 gallons of absolute alcohol.

The variation in the fermentation of sugars is due to the presence of pentoses, which are not fermentable, the hexoses only being decomposed by the yeast.

One experimenter using European pine sawdust made only seven gallons of absolute alcohol from a short ton.

Another, working on a large scale, succeeded in obtaining about 19 gallons of absolute alcohol from a long ton of sawdust containing 20 per cent moisture, equal to nearly 24 gallons to the ton of dry sawdust. The quality of the sawdust is said to be most satisfactory, there being no turpentine flavor or odor.

A process for making alcohol on this principle proposes to use sulphurous acid instead of sulphuric acid, or other mineral acid previously used. This process is known as Classen's process. Sulphurous acid (formed by passing sulphuric dioxide into water) easily loses this sulphur dioxide when heated, consequently when wood is treated with this acid and sugar formed the acid can be decomposed by heat and the sugar solution left ready for fermentation and containing nothing to seriously interfere with the fermentation process. The sulphur dioxide formed by the decomposition of the sulphurous acid can be recovered by passing it through water.

A company with a large capital is attempting to utilize pine wood sawdust in this manner at Hattiesburg, Miss. The author has not attempted to obtain the details of the process as carried out by this company, and has no information as to the success of the venture.

The general principles of the Classen process are known, and can be briefly stated.

The steps in the process are as follows:

1. The manufacture of the sulphurous acid, which is done by simply passing sulphur dioxide gas into water. This gas can be made by burning sulphur in suitable receptacles or from pyrites.
2. The treatment of the wood with the weak sulphurous acid, under pressure in a steam jacketed rotary digester.
3. The blowing off of the gas and steam from the digester to recover the acid.
4. The removal of the treated sawdust into leaching or exhausting vats, where the sugar is washed out with water.
5. The neutralizing of the sugar solution thus produced by means of carbonate of lime or other alkali.
6. The fermenting of the sugar solution.
7. The distilling of the alcohol as ordinarily carried out in distilleries.

The conditions of working should be carried out very closely. An acid solution of about one-third the weight of sawdust is used in the digester. The digester is slowly turned and the steam in the outside jacket heats the contents of the digester to approximately 295 degrees Fah. The pressure rises to 100 lbs., or more, to the square inch, and is maintained for about three hours. By the action of the acid some of the cellulose is changed to sugar. By blowing out the steam and acid into absorbing tanks 75 to 80 per cent of the acid is recovered.

To extract the sugar from the treated sawdust, the material is removed from the digester and put into a series of tanks similar to a diffusion battery. Here fresh water enters the tank containing residue with a small amount of sugar, and then passes to another containing residue with a larger proportion of sugar, and so on until the tank containing the material fresh from the digester is reached, from whence it passes to the neutralizing vats. In this manner the sugar is extracted from the treated sawdust with the use of but little water, and makes a much stronger sugar solution than if each tank was washed separately. Generally, the contents of each tank is washed ten times before removing the residue.

A yield of 450 to 500 lbs. of sugar, about 70 to 90 per cent of which is fermentable, is claimed as the yield from a long ton of dry sawdust. This sugar is in a dilute acid solution, which must be nearly neutralized so that the acid will not affect the action of the yeast which is added to the solution to cause alcoholic fermentation. This neutralizing and fermenting is done in suitable tanks and vats. The fermenting must be done at the proper temperature and when finished the "mash" is distilled in column stills. The yield is claimed to be 25 gallons of absolute alcohol per long ton of dry sawdust.

The residue, consisting chiefly of lignin and other matter not acted upon by the acid, and amounting from two-thirds to three-quarters of the volume of the original wood, can be used for fuel, or otherwise treated. When sawdust is acted upon by acids it loses its elasticity and consequently can be easily moulded into briquettes, which can be used for fuel or destructively distilled.

Whether the process will be successful or not remains to be seen. It requires careful attention to details to obtain the best results.

Tar and Derivatives.—The chief derivatives from tar would be the products of distillation. The products of the destructive distillation of tar have been mentioned, and in the first part of this chapter a table is given showing the amount of the various products obtained by distillation of various kinds of tar.

The distilling operation is smilar in all cases, the yield of the different products varying according to the quality of the tar. In distilling pine tar from retorts the light oils should first be removed by steam and the tar remaining treated before distillation by washing with lime water to remove acid. Of course. this acid can be distilled off if desired, thus making the use of lime unnecessary.

Starting with neutral and water free tar, the tar is placed in a still made of wrought or cast iron, the latter being preferred for small stills, as the tar can be easily coked in them if required. These stills are generally made a little over two-thirds as high as the diameter and furnished with suitable inlet valves at the side near the top and an outlet pipe at the bottom for withdrawing the hot pitch. To keep the tar from boiling over, and to help heat the mass more evenly, a stirrer is provided. Sometimes the bottom is made concave, so that the fire can come nearer the middle of the still. On the top of the still is a spherical head, from which comes a pipe leading to the condenser. Usually the still is entirely bricked in to prevent radiation.

The still is heated very slowly for the first five or six hours. The best tar contains some water, and this causes a noise in the still. This water distills first and is followed by light oil, or oil of tar, as it is called at the pharmacies. This oil quickly turns brown on exposure to the air. The receiver is changed when the sp. gr. of the oil reaches about 0.98. Following the light oils a heavy oil comes over, having a sp. gr. of upwards of 1.01 and a yellowish green color. The distillations can be continued until nothing but coke remains in the still, but this is so difficult to remove that it is best to stop with the production of pitch, which can be drawn out hot from the still, if proper care is taken to prevent it from igniting. This pitch can be run out on iron plates and broken up for fuel, or used for similar purposes as coal tar pitch.

Some make the distillation according to temperature, the oil collected under 150 degrees C. being called light oil, and that collected above 150 degrees C. is known as heavy oil. Some collect the light oils up to 240 degrees C. and the heavy oils between 240 degrees C. and 290 degrees C., this latter method not being common.

The oils are often washed with caustic soda and redistilled, the light oils being used as a substitute for turpentine. The heavy oil contains most of the creosote, amounting to about 15-25 per cent in pine tar oil and about 17 per cent in the creosote oil of the fir, being in the latter case 5 per cent of the tar. The usual method of obtaining creosote is to treat the heavy oil with strong lye of about 1.20 sp. gr. Usually a small sample is treated first, in order to determine about how much soda is needed. This alkaline solution is drawn off from the remaining oil. Often both oils are mixed after neutralizing with the soda, and then rectified by distillation. The receiver is changed as soon as the temperature rises above 302 degrees F., and is changed again when the temperature rises above 482 degrees F. Some take the fraction between 150 degrees and 250 degrees.

The rectification and treatment with soda is repeated many times in some cases, but finally the light oil is collected separately from the heavier. The light oils thus produced contain mostly xylol, but also eupion and kapnomar; the heavy oil contains the paraffin.

The alkaline liquors have absorbed the creosote. These liquors are then boiled in an open pan to expel hydrocarbons, and when cooled saturated with sulphuric acid and allowed to repose. The fluid separated thereby is creosote. This is sometimes again dissolved in alkali and reprecipitated with sulphuric acid until entirely soluble in caustic soda. In either case it should afterwards be distilled and the middle portions, collected between 200 degrees and and 220 degrees C., are called commercial wood creosote. To further purify it, it is treated with ¼ to ½ per cent of potassium bichromate and ½ to 1 per cent of sulphuric acid, allowed to repose twenty-four hours and again distilled. The distillation is generally carried on in glass vessels, the portions between 205 degrees and 220 degrees C. being collected separately.

Wood creosote is a colorless, highly refracting oil with a sp. gr. of 1.03 to 1.087, and a boiling point from 205 degrees to 222 degrees C. Stockholm creosote from pine tar consists chiefly of creosol $C_6H_3(CH_3)\begin{cases}OCH_3\\OH\end{cases}$ Wood tar creosote does not solidify with moderate cold; it is a powerful disinfectant, but does not disintegrate like phenol is apt to do. It is insoluble in water, but readily so in ether, alcohol, glacial acetic acid, chloroform, benzine and carbon bisulphide. Fifteen parts of wood creosote with ten of collodion dissolve to a clear solution, whereas under the same conditions coal tar creosote forms a gelatinous mass. Crude wood creosote contains, in addition to creosol, phloral, guaiacol, etc., eupion, kapnomar, picamar, cedriret, pyrene, pittacal, etc. and these can be recovered from wood tar. Such are not commercially important.

The pitch formed in the still by distilling the tar comprises a large bulk of the original tar. There is still left about 88 per cent of volatile matter, which can be removed by heating, leaving 12 per cent of soft friable coke.

This pitch is soluble to a large extent in alcohol, potash, benzine, etc. It contains some volatile fatty acids and hydrocarbons. See Pitch, Chapter X.

CHAPTER XIII.

CHEMICAL CONTROL OF A PLANT FOR THE DISTILLATION OF WOOD.

The destructive distillation of wood is as much a chemical operation as the distillation of petroleum. Very few manufacturing industries using destructive distillation processes employ chemists. One of the best known of these industries is the coal gas manufacture and it is only recently that chemists have been used at these plants even in large cities. One reason is that the field has been occupied more by engineers rather than chemists.

With the steam process for obtaining turpentine, the services of a chemist are not so essential unless derivatives are to be made, but with large destructive distillation plants, one chemist or several could be of great service in the operation of the plant. At present very few chemists are acquainted with the details of the operation of wood distilling plants and this lack of familiarity on their part has been pointed out to their disadvantage, by the men who have been in control of these plants. But put an experienced chemist in a position to learn the details of the business, and his general knowledge will soon place him far in advance of the men engaged in the business who are now familiar with it. This superiority has been shown markedly in other lines of chemical industry and it is not to be wondered at, as this is what chemists have been trained for. The pine wood distilling business is not attractive to chemists as it has not proved to be a very paying industry.

The following scheme of chemical control is not considered to be anything but suggestive. If plants of any size should get on a paying basis, and a laboratory established, this plan could be modified to suit conditions or another used in its place. The arrangement naturally falls under the given headings.

Measurements.

Wood.—This should be weighed in the car or wagon or, if desired, measured, but weighing is to be preferred. When sawdust or ground wood is used, it should be measured by the capacity of the retorts and if weight is desired, the approximate density can be taken by sample. The trash in a load should be weighed once in a while to determine the per cent in this form, particularly when long wood is used.

Crude Turpentine.—This should be weighed also to get accurate results, but for technical purposes it can be measured in tanks of known capacity, which are accurately gauged. When sent to the still the temperature should be taken, the specific gravity and a sample of each still charge.

Water Separated from the Crude Turpentine.—It is better to measure this through a meter, the specific gravity and temperature being taken at suitable intervals. A sample should be taken also so as to be able to determine the amount of oil that may escape.

Caustic Liquor.—This should be kept in a special air-tight tank of known capacity. The caustic should be weighed and the water measured before mixing. In using, the strength having been previously determined by testing a sample, the temperature being known, the quantity of liquor used should be noted by the gauge.

Refined Turps.—As these come from the still, the water settling out can be measured and the temperature taken. A regular sample should be taken to determine any loss in oil. The oils can be measured from each run in the separating tanks when barreled or when pumped, the temperature being taken.

Condensing Water.—The water from each condenser can be measured when the exact working of each one is to be ascertained. The entire condensing water used may be measured by the work of the pumps or by sending through meters.

Tarry Products and Pyroligneous Acid.—These products from the retort can be determined by volume, a large sample being taken and the temperature noted. This should be done before any great settling takes place. The workings of each separate retort should be gauged as far as possible.

After settling, if acetate is to be made, the pyroligneous acid can be measured and sampled, the temperature taken and the acid sent to the neutralized tank. If acetate is not made, the acid should be sampled and sample kept to test the tar content and the acid run off. The tar oil can be treated similarly, being sure in all cases to note the temperature.

Stills.—Measure all liquors going into stills and sample same and take temperature. The condensed water and condensing water should be measured as used.

Tar.—Should be weighed when barreled or when shipped in tank cars. At other times measure and sample, noting the temperature.

Other Products.—The same general rule applies to them. Weigh the solids and measure the liquids, in all cases sampling and noting the temperature.

Sampling.

Wood.—The sampling of wood is a very difficult operation. For long wood, perhaps the best way would be to take about three sticks of what appeared to be average wood and pass them through a hog and thoroughly mix the sample thus produced. It should be quickly covered and kept in an air-tight vessel. Hogged wood or sawdust can be sampled as it enters the retort.

Crude Turpentine.—In bulk a sample can be obtained by simply filling a suitable bottle and corking it. To sample a single retort which is not supplied with a separate receiver, it can be arranged so that a small part of the distillate can flow into a suitable bottle. A sample should be taken of each charge entering the still.

Other Products.—All the other liquid products can be sampled like the crude turpentine.

Liquors in Settling Vats.—When the liquors are of such a nature that a marked separation will not take place, a special device is necessary for sampling. A good way is to get a long glass tube about one inch or more in diameter and of sufficient length to reach the bottom of the tank. One end should be fitted with a piece of rubber. By inserting this tube into the liquid at different levels, a number of samples can be drawn out and collected in one bottle and shaken together.

In very large tanks valves can be placed at intervals on the tanks and the samples drawn from each and mixed. In this case, it is necessary to catch a large sample and return it to the tank before a suitable sample can be obtained from each valve.

Gas.—Samples can be drawn as often as desired by the regular methods. The flue gases should be watched in starting new retorts so as to enable the fireman to learn their peculiarities, if any.

Standardizing Apparatus.

Balances and Weights.—Two balances should be used, one sensitive to five milligrams and the other very sensitive, turning when loaded with one or two-tenths of a milligram.

Test the balance arms by balancing weights against each other and then changing them to the opposite pans, where they should again balance each other. The weights, of course, should be standard and have a correct relation between them and the balance and between each other.

Polariscopes.—A good polariscope should be used showing left and right-handed scales. The zero point should be set properly and all errors on the scale noted. The scale can be checked by standard quartz plates, and these latter themselves checked once in a while.

Refractometer.—This instrument as well as the polariscope should be standardized at 20 degrees C. Probably for technical use one with a standard at 30 degrees C. would be better on account of the heat in summer. Generally a form similar to Abbe's would be suitable. The basis used is distilled water with its reading at the standardizing temperature.

Thermometers.—These should be compared with a standard thermometer which has been officially corrected.

Hydrometers or other form of spindles. These can be checked by means of the pycnometer.

Flasks.—These can be standardized to the polariscope temperature. This temperature should be used for everything. By marking a 100 c. c. pipette so as it will deliver exactly 100 c. c. at the given temperature, all the flasks can be standardized by means of this. Otherwise, weigh with the proper amount of water, using 1 gram in vacuo at 4 degrees C. equal to 1 c. c.

Water Meters.—These should be standardized by weighing a quantity of water passed through.

Tanks.—The contents of these can be most accurately determined by filling from a barrel placed on a platform scale, weighing all water admitted and making correction for temperature. Otherwise, calculate the cubic contents from measurements and allow for the temperature.

Burettes and other Volumetric Apparatus.—The same temperature should be used as for flasks and contents accurately determined by weighing the water delivered. When one piece is properly standardized it can be used to standardize the others.

ANALYSIS.

Turpentine.

For the analysis of turpentine the following method is given by the Bureau of Supplies and Accounts, Navy Department, May, 1903.

1. The turpentine must be properly prepared distillate of the proper kinds of pitch or pitch pine, unmixed with any other substances; it must be pure, sweet, clear and water white.

2. A single drop allowed to fall on white paper must completely evaporate at a temperature of 70 degrees Fah. without leaving a stain.

3. The specific gravity must not be less than 0.862 or greater than 0.872 at a temperature of 60 degrees Fah.

4. When subjected to distillation, not less than 95 per cent of the liquid should pass over between the temperatures of 308 degrees Fah. and 330 degrees Fah., and the residue should show nothing but the heavier ingredients of pure spirits of turpentine.

5. A definite quantity of the turpentine is to be put in an open dish to evaporate, and the temperature of the dish maintained at 212 degrees Fah.; if a residue greater than 2 per cent of the quantity remains on the dish it will constitute a cause for rejection.

6. **Flash Tests.**—An open tester is to be filled within one-fourth of an inch of its rim with the turpentine, which may be drawn at will from any one can of the lot offered under the proposal. The tester thus filled will be floated on water contained in a metal receptacle. The temperature of the water will be gradually and steadily raised from its normal temperature of about 60 degrees Fah. by applying a gas or spirit flame under the receptacle; the temperature of the water is to be increased at the uniform rate of 2 degrees Fah. per minute. The taper should consist of a fine linen or cotton twine (which burns with a steady flame) unsaturated with any substance. When lighted it is to be used at every increase of 1 degree temperature, beginning at 100 degrees Fah. It is to be drawn horizontally over the surface of the turpentine and on a level with the rim of the tester. The temperature will be determined by placing a thermometer in the turpentine contained in the tester so that the bulb will be wholly immersed in the liquid. The turpentine must not flash below 105 degrees Fah.

7. **Sulphuric Acid Test.**—Into a 30 cubic centimeter tube graduated to tenths, put 6 cubic centimeters of the spirits of turpentine to be examined. Hold the tube under the spigot and then slowly fill it nearly to the top of the graduation with concentrated oil of vitriol. Allow the whole mass to become cool and then cork the tube and mix by shaking the tube well, cooling with water during the operation, if necessary. Set the tube vertical and allow it to stand at the ordinary temperature of the room not less than half an hour. The amount of clear layer above the mass shows whether the material passes test or not. If more

than 6 per cent of the material remains undissolved in the acid this will constitute cause for rejection.

A test of turpentine for petroleum oils formerly used was to agitate the oil with sulphuric acid (two parts to one of water) then distill in a current of steam. Treat the distillate of oil with sulphuric acid (four parts acid and one of water) and again distill; any oil coming over being petroleum.

The chief difference between wood turpentine and gum turpentine, as now produced, is in the distillation test, but both oils vary much in their properties. Whether it is advisable to adhere to the standard set for orchard turpentine in testing wood turpentine is doubtful. Perhaps it would be advisable to make a new standard of requirements for wood spirits of turpentine and make all manufacturers adhere to it.

The chief concern of dealers is that oil of turpentine contains no cheap adulterations. The series of tests given above will locate any petroleum admixtures.

Of the various special tests, only a brief outline will be herein given. Utz observes the refractive index and treats with iodine water and observes the color as compared with a known sample similarly treated. Hersfeld treats with concentrated sulphuric acid, then with fuming sulphuric acid, only a small definite portion of oil remains; any excess representing adulteration. The determination of the refractive index is made on small fractions of the distilled oil, before treatment with sulphuric acid. McCandless makes three successive polymerizations with concentrated sulphuric acid once and fuming acid twice, distilling after each treatment in a current of steam and testing the distilled oil in the refractometer. Worstall treats with iodine under exact conditions. Hinsdale evaporates a weighed quantity of known turpentine in one watch glass and the same amount of unknown sample in another and places them in a water bath at 170 degrees Fah. until the known sample evaporates, the residue remaining in the other being adulterants. Hall treats with sulphuric acid under exact conditions, and observes the rise in temperature, similarly to the Maumene test for vegetable oils.

Hydrochloric acid is stated to turn wood turpentine black, but such is not the case with well-refined samples.

A test for rosin spirit in wood turpentine is given by Valenta as follows: Mix one to two parts of a six per cent solution of iodine in carbon bisulphide or carbon tetrachloride and heat on a water bath. A green to olive green is produced by pinoline or rosin spirit and none by wood spirit or turpentine. For a mixture of oil of turpentine, wood turpentine and rosin spirit take equal volumes of the mixture and a one per cent solution of auric chloride in a test tube and shake, heat one minute in water bath and shake again. Pure oil of turpentine shows separation of gold in oily layer only but when the other substances are present the aqueous solution is completely decolorized.

Wood Oil.—No satisfactory tests are given for this oil, as it is of such a varying composition as now produced.

At those plants which wish to make a standard certain tests might be applied. These would have to be determined by the chemist in charge and a standard set.

The following plan might be of service. Test for specific gravity. Take the iodine value, the specific rotary power and refractive index (if too dark dissolve in water white petroleum), take the flash test and fire test, determine boiling point, try tests given under turpentine, and for creosote oils, determine the amount of creosote by any of the regular methods. From these data a definite grade may be established at each plant.

A method of investigation to determine the constitution of wood oil is to be found in the American Chemical Journal, Vol. 25, No. 1. This cannot be given here, as it cannot very well be carried out in a technical laboratory.

Tar Oil.—The only test that is necessary to make on this oil would be for the presence of pyroligneous acid. It has been found that the pyroligneous acid and tar do not always separate distinctly into two layers. The sample of the mixture is taken as it comes over, and if it does not separate into distinct layers, two methods may be used to give approximate results as to the amount of oil and acid in the mixture.

The first would be to thoroughly mix the sample and then take out a definite amount, say 25 c. c., put in a graduating cylinder and dilute to 500 c. c. (or more, according to the rate of separation). When thoroughly separated the amount of tar oil can be read off and the pyroligneous acid considered to be the difference between the amount of tar oil and the original amount. For greater accuracy the bottom part of the graduated cylinder could be made of small bore and mounted on a flat base.

The second method consists in placing a sample in a graduated flask of a centrifugal tester, and reading the percentage of tar or acid from the scale after revolving. In most cases a special flask would need be used, as over 50 per cent of the distillate is sometimes acid; the centrifugal tester, though, would be the best device to use for the separation.

In taking stock, it would be necessary to know the amount of tar in the tar oil.

To do this a sample of the mixed liquor or the tar oil itself after separating the acid should be placed in a distilling flask and the light wood oils distilled by means of a current of steam. The residual tar can then be weighed and the light oils measured. The distillation should continue no longer than the degree to which it would be carried out at the plant. It might also be distilled with water and the light oils measured and the difference considered as tar or the tar dried and weighed.

Measuring the oils is not a very safe way, as they are soluble in water to a considerable extent.

Tar.—When the tar contains water a quick technical way would be to separate it in a centrifugal tester and read off the amount.

One test for tar proper has already been given in part. It consists in fractionally distilling the tar in a distilling flask with a short head. Care should be used at first, as any water contained therein causes severe bumping. The specifications are:

Distilling under 150 degrees C., 9.70 per cent.
Distilling between 150-350 degrees, 42.61 per cent.
Distilling between 350-363 degrees, 26.62 per cent.
Coke, 21.07 per cent.

Tar varies so much that certain definite tests like the above could be only approximated. The chief methods for judging tar are in regard to the color, weight and viscosity. The color can be determined by placing a drop on a sheet of white paper; the spot should be light brown. The specific gravity varies from 1.05 to 1.12. The thickness or viscosity is generally judged by the eye. No standard can be set for this property, but arrangements could be made to sell tar within certain limits as determined by a viscosimeter. Another test, to distinguish between some grades of retort tar is to pour a drop on the surface of a piece of smooth white poplar or deal wood and note the relative length of time it takes for the sample to darken on exposure to the air, as compared with a sample of known quality.

Acetates and Pyroligneous Acid.

To determine the acid, reckoned as acetic acid, in pyroligneous acid, several methods are given. One method is to take 25 c. c. and dilute with 1,000 c. c. or more of distilled water, titrate with normal alkali, using phenolphthalein as an indicator, and calculate the percentage. Often with pine wood acid, the end point is too indistinct and another method must be used.

C. Mohr gives the following method: Weigh off 10 grams of wood vinegar, heat in a beaker with about 3 grams of pure barium carbonate (test the carbonate) until effervescence ceases, and filter. The solution of barium acetate is strongly colored, but the carbonate remaining undissolved very little. The residue after washing is dried and weighed and the quantity of acetic acid present calculated; each gram of dissolved carbonate corresponding to 0.809 gram of acetic acid or 10 grams of wood vinegar contains 6.09 per cent.

It is quicker to treat the undissolved carbonate with an excess of normal nitric acid and titrate back with normal soda, using litmus as an indicator.

Instead of barium carbonate, Mohr also uses precipitated moist calcium carbonate, the alkalinity of which is determined. This is added to the wood acid in excess and the mixture boiled to expel CO_2, then filtered and treated as before.

Acetates.

Acetate of lime, such as the brown and gray, are tested for acetic acid in various ways. Two methods will be given.

Stillwell and Gladding's method is as follows:

A 100 to 120 c. c. retort, the tubulure of which carries a small funnel fitted in with a rubber stopper, and the neck of the funnel stopped tightly with a glass rod shod with elastic tube, is supported upon a stand in such a way that its neck (the retort neck) inclines upwards at about 45 degrees; the end of the neck is drawn out and bent so as to fit into the condenser by help of an elastic tube. The greater part of the retort neck is coated with flannel, so as to prevent too much condensation.

One gram of the sample being placed in the retort, 10 c. c. of a 40 per cent solution of P_2O_5 are added, together with as much water as will make about 50 c. c. A small naked flame is used and if carefully manipulated, the distillation may be carried on to near dryness without endangering the retort. After the first operation the retort is allowed to cool somewhat, then 50 c. c. of hot water added through the funnel, another distillation made as before, and the same repeated a third time, which will suffice to carry over all the acetic acid. The distillate is then titrated with alkali and phenolphthalein.

The following is the method of Grimshaw, taken from Allen's Organic Analysis, as given by Sutton:

Method of Procedure: 10 grams of the sample are treated with water and an excess of sodium bisulphate ($NaHSO_4$) the mixture diluted to definite volume, filtered and a measured portion of the filtrate titrated with standard alkali; a similar portion meanwhile is evaporated to dryness with repeated moistenings with water, to drive off all free acetic acid. The residue is dissolved and titrated with standard alkali, when the difference between the volume now required and that used in the original solution will correspond to the acetic acid in the sample. Litmus paper is the proper indicator.

Wood.

The only tests necessary to be made on wood are those which determine its content of water and resin.

Sampling is the most difficult part. If possible, a few average sticks from a cord lot should be passed through a suitable disintegrating machine. In the absence of this, each stick should be cut into in several places, and rasped a little at each place, with a wood rasp. In both cases the finely divided product should be bottled, so as to prevent the evaporation of moisture.

Moisture.

Determine this by heating a sample of two grams or more in an air bath kept at 105-110 degrees C. Cool in a desiccator and weigh and the loss in weight indicates the moisture. This method is not so very accurate, as the wood is apt to oxidize slightly, and in fat woods a great deal of turpentine would escape with the water.

The author knows of no method for testing this class of wood, but would suggest the following:

Take 2 to 5 grs. of the finely divided wood and place in a weighing bottle fitted with a tight ground glass stopper. Make a hole in the stopper and weld a piece of glass tubing around the opening, then bend the tubing so as to form a U. In the tube, put pieces of lime or soda lime. Weigh the stopper and the tube containing the lime, also weigh the bottle and its contents. Insert the stopper in the weighing bottle and place the whole apparatus in an oven heated to about 105-110 degrees C. Heat for one hour, then withdraw the apparatus and allow to cool either in a desiccator or by closing the end of the U tube.

Weigh each part separately. Repeat until the loss in weight of the weighing bottle is relatively constant. Then heat the tube containing the lime to about 200 degrees C. in an air bath or otherwise; cool and weigh, repeating until the loss in weight is constant.

The method is based on the following apparent principles: The first heating drives the water and any oil over into the tube. The lime fixes the water. Any oil condensed is driven off by the second heating. Slaked lime holds its water of combination even when heated to 250 to 300 degrees C.

The loss in weight of the weighing bottle represents moisture plus volatile oil. The weight of the lime tube after the second heating minus its original weight represents the moisture. The moisture found subtracted from the moisture and oil lost from the weighing gives the amount of volatile oil. If any volatile oil is found to have passed over, it should be considered as turpentine and added to the amount found by distilling the ether extract.

A method used in determining moisture in explosives containing volatile oils might also be used. It consists in treating the ground sample with calcium carbide in a tube, taking precaution not to mix the two until the tube is connected with a gas measuring apparatus. The acetylene gas formed is measured over salt water and corrections made for temperature and pressure. An allowance is also to be made for the amount of moisture retained by the lime formed during the reaction, as this moisture does not act on the calcium carbide. On this account 1 c. c. acetylene equals .001725 gr. moisture, instead of .00162 gr.

In making the test the reaction can be hastened by heating in a water bath to 100 degrees C.

The wood residue from the moisture determination is placed in a filter cone of a Soxhlet fat extraction apparatus and the cone inserted in the tube. The tube should then be connected with the flask. It will be found that in determining the moisture of fat wood that some of the resin has exuded from the wood on account of the heat and flowed to the bottom of the crucible or other container used in making the determination. After removing the wood this resin can be dissolved in ether and poured upon the cone in the tube. The apparatus is used as in making an ordinary fat analysis, the resin being extracted from the wood and collected in a small flask. The ether is evaporated and the flask cooled and weighed. The difference between this weight and the weight of the empty flask equals the weight of the extracted matter. By connecting the flask with a suitable condenser and carefuly heating it, the turpentine will distill over. It is better, though, to make this distillation by simply passing a current of steam through the extracted resin, and the turpentine will distil without danger of decomposition of the resin.

The turpentine can be separated from the water and weighed or measured. The flask containing the resin should then be placed in an air bath until the water is driven off, and then weighed. The difference between this weight and the weight of the flask equals the weight of resin. The weight also serves as a check on the turpentine.

The woody fiber left after extracting with ether can be burned in a crucible and the residue weighed as ash. If desired it can be distilled by placing it in a glass retort. The retort should be placed in an air bath, the tube of the retort extending through an opening in the side and connecting with a condenser. The condensed products can then be collected and separated and each weighed separately. The charcoal should also be weighed. The gas can be collected and measured.

The acetic acid in the pyroligneous acid can be determined by Mohr's method and the wood alcohol by the phosphorous diiodide method, given under pyroligneous acid and wood alcohol, respectively.

Determination of real methyl alcohol in wood spirit (Allen's Org. Analysis, Vol. 1, p. 73).

A dry flask is furnished with a cork fitted with a tapped funnel or pipette and connected with an inverted condenser; 15 grams of phosphorous diiodide are placed in the flask and 5 c. c. of the sample of wood spirit (measured at 15 degrees C.) added slowly, drop by drop, by means of a pipette; 5 c. c. measure hydriodic acid of 1.7 sp. gr. containing in solution 8.5 grams of free iodine is next added through the pipette. The flask is then heated to 80 to 90 degrees C. by immersion in hot water for a few minutes, after which the condenser is placed in the ordinary position and the contents of the flask are distilled and collected in a graduated tube. The distillate is shaken with water and the volume of methyl iodide read off. Corrections of 8 volumes per 1,000 must be made for the solubility of the methyl iodide in water, and for the loss due to the vapor which fills the apparatus. This error, which is constant for the same apparatus, is determined by distilling a known measure of iodide of methyl, measuring the distillate and thus ascertaining the loss. Krell prefers to pass a current of air into the apparatus, through the pipette, and thus drive out the vapor of methyl iodide. Under these conditions, 5 c. c. of pure anhydrous methyl alcohol yields 7.45 c. c. of the iodide.

By the iodine process any methyl acetate present in the sample is converted into iodide and hence increases the apparent percentage of methyl alcohol. For most purposes the error thus introduced can be neglected. If desired the quantity present can be previously determined approximately by heating a known quantity of the wood spirit with standard soda and titrating the excess with standard acid. Forty parts of Na O H neutralized corresponds to 74 of methyl of acetate, or 32 of methyl alcohol. The amount of methyl alcohol so found should be subtracted from the total amount corresponding to the iodide in order to ascertain the real amount of methyl alcohol existing as such in the sample.

When acetone is present it distills over with the methyl iodide, and it is only by repeated washing that the distillate can be wholly freed from it. Bardy and Bordet have constructed a table showing the diminution in volume undergone by methyl iodide containing various percentages of acetone by washing with water. In the absence of the table, the error caused by the presence of acetone might be avoided by saponifying the washed distillate with alcoholic potash evaporating to dryness, dissolving the residue in water, acidulating an aliquot part of the solution with nitric acid, and then precipitating the iodide by silver nitrate. 235 parts of iodide of silver represent 32 of methyl alcohol.

Dimethyl acetal also comes over, 5 c. c. of which yield 5.3 c c.. of methyl iodide. The others are either soluble in water or are converted into resinous bodies.

For the preparation of the iodide of phosphorous, 15.5 grm. of phosphorous are dissolved in 350 c. c. of carbon disulphide, and 127 grm. of iodine are gradually added, the vessel being kept well cooled. The diiodide separates in crystals, which are dried in a slightly warm current of air and preserved in a well-stoppered bottle. A qualitative test for methyl alcohol given by Mulliken & Scudder consists in plunging a hot copper spiral into the liquid to be examined and adding one drop of a solution of one part resorcin in 200 parts of water, then pour the solution carefully upon concentrated sulphuric acid so as not to mix. After three minutes with slight mixing methyl alcohol causes rose red flocks.

Creosote.—There are many substances that go by the name of creosote. Coal tar creosote and wood tar creosote are separated by Hagar's method. The following is an abstract of Allen's description of Hagar's method:

Three measures of absolute glycerol are mixed with one measure of water, and the solution used as a solvent. Treat one measure of the sample in a Mohr burette with three measures of the diluted glycerol, and allow the liquid to stand until separation has occurred. If the creosote be pure, the volume will remain unchanged. If reduced the glycerol layer is tapped off and the remaining creosote again shaken with three times its measure of diluted glycerol and the measure again observed, This second treatment will always suffice for the removal of the coal tar acids, unless their proportion is very large, and hence the volume of the residual layer will indicate the proportion of real wood creosote in the quality of sample taken. The nature of the residual creosote can be verified by the collodion test (should form a clear solution) while the coal tar acids can be recovered from the glycerol solution by filtering it to remove suspended traces of wood creosote, diluting with water and agitating with chloroform. On spontaneous evaporation of the separated chloroform the coal tar acids are obtained in a condition of sufficient purity to allow of their positive recognition.

Acetone.—The gravimetric determination of this substance is by means of caustic and iodine. A volumetric method that can be used in presence of ethyl alcohol is given by Sutton, as follows, modified by Squibb and Kebler. The solutions required are as follows:

(1) A 6 per cent solution of hydrochloric acid.

(2) A decinomal solution of sodium thiosulphate.

(3) Alkaline potassium iodide solution prepared by dissolving 250 gm. of potassium, 257 gm. of sodium hydroxide (by alcohol) in water, likewise made up to a litre. After allowing the latter to stand, 800 c. c. of the clear solution are added to the liter of potassium iodide.

(4) Sodium hypochlorite solution: 100 gm. of bleaching powder, (35 per cent) are mixed with 400 c. c. of water; to this is added a hot solution of 120 gm. of crystalized sodium carbonate in 400 c. c. of water. After cooling, the clear liquor is decanted, the remainder filtered and the filtrate made up to a litre; to each litre is added 26 c. c. of sodium hydroxide solution (sp. gr. 1.29).

(5) An aqueous acetone solution containing 1 or 2 per cent of acetone as pure as may be had, say, 99.7 per cent.

(6) Starch solution, prepared by treating 0.125 gr. of starch with 5 c. c. of cold water, then adding 20 c. c. of boiling water, boiling a few minutes, cooling and adding 2 gm. of sodium bicarbonate. This starch solution will keep for some weeks.

To 20 c. c. of the potassium iodide solution are added 10 c. c. of the diluted aqueous acetone, an excess of the sodium hypochlorite solution is then run in from a burette and well shaken for a minute. The mixture is then acidified with the hydrochloric acid solution, and while agitated an excess of sodium thiosulphate solution is run in the mixture, being afterwards allowed to stand a few minutes. The starch indicator is then added and the excess of thiosulphate retitrated. The relation of the sodium hypochlorite solution to the sodium thiosulphate being known, the percentage of acetone can be readily calculated.

In the above reaction 1 molecule of acetone requires 3 mol. of iodine to four 1 mol. of iodoform. One atom of available chlorine will liberate one atom of iodine from the K I in the alkaline solution, or 1 c. c. will liberate just enough I to make 1 c. c. of the same normal strength as the hypochlorite solution originally was; therefore, by reading the number of c. c. of hypochlorite consumed as so many c. c. of iodine solution of the same normal strength, the calculation is reduced to the basis of iodine. Expressing it as a proportion and letting y equal the amount of combined I and x that of acetone, we have (taking I as 126.5)

$$759:58::y:x \text{ or } x=y\,\frac{58}{759} \text{ or } x=y\ 0.07641.$$

Example of calculation—10 c. c. of the acetone solution containing 1 gm. of the liquid to be analyzed required 14.57 c. c. of iodine solution of same strength, or combining we have

$$\frac{14.57\text{x}0.806\text{x}0.1260\text{x}.07641}{\text{1 gm of solution of acetone}}=11.31 \text{ per cent.}$$

Many other methods of analysis of the various products should be reviewed in order to get a systematic routine for a wood distilling plant. Of the methods given, all are recognized as standards for the particular conditions and products to which they apply. It is beyond the scope of this work to give more methods than are herein contained.

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