have a reducing action on platinum. Except with small flames it is impossible to mix sufficient air with the gas in the regular place at the tip of the burner without using so large an excess that the flame necks down and blows out. Mixing can be effected by placing a burner an inch or so from the lamp tip. The flame then starts mainly from the auxiliary burner, allowing the required air to be sucked into the intervening space. This serves for heating dishes, etc., but the flame is noisy and too poorly defined for glass-blowing.

Improvement was effected by admitting air to the gas current at a point back of the burner, as shown in Fig. I. Part of the air is diverted at A and mixed with gas at B. It proved essential at the pressures used, that the air follow the straight course at B with the gas entering at right angles. Fig. II shows the same arrangement incorporated in the lamp by

shifting of parts and addition of a few stock fittings. Cock C replaces the pinchcock of Fig. I. D is a three-way cock made by drilling an open two-way cock from surface to center of stopper and cutting away from the stopper the partition between the new hole and half the old one. This was added to permit escape of air cut off from the lamp, thus allowing better regulation and relieving rubber connections from the high pressure of our laboratory air pipes. For small flames the cross connection can be closed; for larger it can be opened until the flame reaches the desired diameter next the burner tip. In this way, with air at 20 lbs. per sq. in. and natural gas equivalent to 20 in. of water, there can be maintained quiet flames with sharp 3 in. inner cones or good diffuse flames of larger dimensions.

RESEARCH LABORATORY, NATIONAL CARBON COMPANY
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ADDRESSES

THE HARDWOOD DISTILLATION INDUSTRY IN AMERICA¹

By EDWARD H. FRENCH AND JAMES R. WITHROW

In view of the economic importance of the utilization of wood waste, and the increasing interest manifested in this utilization, it naturally surprises the inquirer that there is practically no literature upon this industry from the point of view of actual American practice. It may not be without value, therefore, to consider the progress made in hardwood distillation, and to trace its development from its inception in this country. We have gathered together consequently and classified much of the information which we have acquired in the plants of this industry during an experience in it totaling about 25 years.

This tracing of the development of an industry of the magnitude of this one is peculiarly interesting to the engineer, since it shows the necessarily slow evolution of the industry in the hands of the unscientific manipulator, and serves not only to emphasize the advantages possessed by the modern chemical engineer, but also to indicate how much more rapid the development might have been in the hands of experienced chemical engineers. It indicates also that in the future, under the direction of the competent engineer, important advances may be made much more rapidly than in the past. This latter statement is significant when it is remembered that progress thus far has been due either to accident or very often to workmen endeavoring to minimize the laboriousness of their work. As plants are operated in 12-hour shifts, this desire to economize energy may be readily understood.

For much of the historical information collected we desire to express our appreciation to Mr. Robert Mackay, who has been active in this industry for so many years. In fact during the past fifteen years our own continued intimate acquaintance with Mr. Mackay has been not only of unusual pleasure but of continuous instruction. A history of this industry would be incomplete without mention of the fact that, due largely to his personality and integrity, the firm with which Mr. Mackay is identified has had the unusual experience, in an extensive industry, of supplying engineering materials for every plant of this nature in the United States and Canada with perhaps two exceptions, for a period covering the past 25 years.

EARLY HISTORY

While charcoal burning was doubtless carried on in the earliest

days in this country, it was not until 1830 that James Ward¹ began the manufacture of pyroligneous acid, at North Adams, Mass. No claim is made that the pyroligneous acid was carried further though it may have been. We have, ourselves, no information on this matter other than the above citation. As far as we can ascertain, it was not until 1852 that works in the modern sense were inaugurated for the distillation of wood for the production of volatile products, and their semi-refined products. This does not appear in agreement with W. L. Rowland,² who is later cited by Monroe and Chatard and C. L. Campbell,³ all of whom state that the manufacture of acetate of lime and methyl alcohol was started in the United States about 1867 by James A. Emmons and A. S. Saxon, in Crawford Co., Pa. Our information in this case comes from various manufacturers who have been in this business from the earliest times or at least whose family connections with it go back to the beginning. One of these sources is Mr. Neil Patterson, whose father was in charge of the original "Scotch Works," built in 1852.⁴ In this year the Turnbull Company, of Glasgow, Scotland, who were engaged there in the copper and iron industry as well as that of wood distillation, built the first operation in this country, by way of expanding their "old country" business. The place chosen for this location was known as Millburn, New York, now Conklin's Station, on the D. L. and W. R. R., not far from Binghamton.

The Turnbull Company brought with them their apparatus as well as men experienced with their methods. On account of these Scotch workmen the works became known as the "Scotch Works," and this name has clung to it to the present time, although the original workmen gradually went with new operations which started shortly after this one. They had much to do with the subsequent expansion of the industry, all of them being later in charge of new works.

It is interesting to note in connection with this first plant, that it was a type that was copied in detail in the construction of those that followed, for many years afterward. All these plants were equipped with cast-iron cylinders 42 in. in diameter and 9 ft. long, set in pairs; thus each pair held about 1¼ cords of wood. The wood was cut 7 ft. long and the charge "run

¹ Monroe and Chatard, "Chemicals and Allied Products," 12th Census, United States, *Bull.* 210, 34.

² Special agent 10th U. S. Census on Chemical Products and Salt, p. 23.

³ *Met. and Chem. Eng.*, 1910, p. 155.

⁴ Mr. J. L. Stuart of Corbett and Stuart, one of the earliest firms in this business in this country, informs us that the date 1852 given by Mr. Patterson for the building of the Turnbull plant should be 1849.

¹ Read before the 7th Annual Meeting of the American Institute of Chemical Engineers, Philadelphia, December 4, 1914.

off" every 12 hours, and 6 days a week. The stills and pans were operated during these early days exclusively by direct firing. The fuel used for the retorts, stills and pans was the charcoal produced, over which was poured the residual tar to make a quick fire.

This original works was followed in 1865 by Emmons & Co., of Brookdale, N. Y. In 1868 Alonzo Smith started a plant at Sturroco, Pa. Keery Brothers, Cadonia; Mandaville, Emmons, Corbett and Mitchell, Livingston Manor, N. Y.; King and Co., Acidalia, N. Y.; Hilton & Co., Hiltonville, N. Y.; Brand & Schlayer, Frink & Ross, Tyler & Hall, Inderlied & Co., Bayless & Berkalue, and Wright & Co. were also early in the field.

During the early part of this period of development, the chief product desired was acetate of lime, for which from 12 to 15 cents per lb. was obtained. The alcohol demand was almost negligible; that made was of very inferior quality and was kept in barrels, and most of it was lost by evaporation or through some "illegal vent." The charcoal also had but little commercial value and was largely consumed as fuel at the works, although a small local demand gradually developed, starting with the neighboring tinsmith.

The change from the old charcoal heaps or pits to the kilns probably came quite early and perhaps before any of the improvements we have mentioned. It was not until 1876, after the retort system was well established and markets for its products had been developed, however, that Dr. H. N. Pierce's inventions made it possible to utilize partially the products wasted in the smoke from the charcoal kilns in Michigan, where great quantities of charcoal were prepared for blast furnace use.

The earliest attempt at refining alcohol, so that it might be of commercial value, was made by a Mr. Pollock, a chemist, living just out of New York City. In a short time a demand began to develop for this product, which steadily increased, and up until 1880 was at all times up to the dollar mark. It was during this period that the need of better refining was felt, as it was realized that a large loss was entailed in the refining operations. This necessity accounts for the coming of the Burcey pans in the early seventies. These are copper containers placed in the vapor line above the stills, with deflectors inside to project the vapor against the upper shell of the container, upon which a stream of water runs, causing fractional separation of the water from the alcohol vapor. The increase of alcohol product resulting from this improvement, together with the increasing market demand, made a central refinery necessary, and the producers united in building a refinery at Binghampton, N. Y., known as The Burcey Refining Company. This was in the middle seventies, and the plant is still in operation. It has served a good purpose, and to it was due perhaps more than to anything else the substitution of steam for direct fire distillation.

Before leaving this early history, it should be mentioned that the Turnbull plant, and in fact several succeeding plants, had but four pairs of retorts or 5 cords capacity to the charge; thus by obtaining two charges within 24 hours their total wood consumption was about 10 cords per day. Today there are single plants operating 160 cords daily. Thus we have an illuminating comparison, at least as far as size is concerned, of the development of the industry.

DESIGN OF APPARATUS

Even at the present time, there remain many plants using the old original retort system, with one very slight modification. Those first in service were of cast iron, while the later ones have shells of steel with riveted cast-iron necks and fronts. The purpose of this change was due to the fact that in the early operations, since the wood was then of much less value than later, and high yields were not so important, it was charred at a very high temperature, in order to increase as much as possible the capacity of the plant by shortening the time of firing.

As cast iron was much more durable for these high temperatures than steel, the use of cast-iron retorts was universally practiced. When, however, with the coincident rise on the cost of wood, it was found that lower temperatures gave an increase of all products, it was seen that steel could be employed to advantage, for its lower weight permitted the turning of the retort. This turning refers to the saving in apparatus universally practiced and consists merely in the removal of the brick setting when the side in contact with the fire becomes buckled, rolling over the retort and placing the uninjured part next the fire, thus materially increasing the life of the retort. This modern retort is of uniform size and design in all plants which still use this type of apparatus. The shell, as originally, is 42 in. in diameter and 9 ft. long, supported at the ends in a cast-iron cradle and having a cast-iron front, with door and lugs, for pinning the door shut. At the rear is a neck 10 in. in diameter, to which is attached the condenser.

The evolution of the condenser is especially interesting, in view of the fact that when the original condenser was installed each succeeding plant accepted it as being of proper size, and the new installation was made the same size. Then from time to time some one would discover that an addition would yield more product and at once the arbitrary sized unit was changed.

ANCIENT TYPE OF COPPER STILL—IN A NOW DISMANTLED RETORT PLANT. FORMERLY DIRECT FIRED, THEN CONVERTED TO STEAM HEATED

Originally the worm condenser was used, but it soon became apparent that the heavy carbon bodies caused clogging, and soon the log condenser was substituted and was used until the introduction of the upright tubular one. These log condensers were made by attaching, by a copper tee, a large-sized copper pipe, called the log, to each of a pair of retorts. Near the ends of this pipe were copper connections, joining the large log to a smaller one, and this in turn was similarly connected with others progressively decreasing in size. The whole log condenser lay in a plane at an angle of 45° and the whole was surrounded by a wooden box with water inlet at bottom and outlet at the top. The joints were simply slip joints and were slipped together and "sweeled;" that is, wrapped with a cloth liberally covered with red lead, which, after becoming dry or "set," offered a suitable and effective joint. At the ends of the logs were removable slip joint caps, for cleaning purposes, so that the cleaning from the heavy tarry and carbonaceous bodies was easily accomplished.

The first really important change in the general style of operation was that of replacing the old retort by the oven, which was done in the middle nineties and is claimed to have been first tried out at Straight Creek, Pa. This change was wholly mechanical, and consisted merely of substituting for the retort

a large rectangular oven, equipped with rails on the bottom for the purpose of carrying cars. These cars were loaded with wood and could be quickly pulled in and out mechanically and the charcoal unloaded outside the oven, greatly increasing the economy in this part of the operation, over the slow hand drawing of the charcoal of the retort system.

There appear to be, however, many more advantages in this style of operation than the mechanical advantages which were

TYPICAL PENNSYLVANIA WOOD YARD

at first anticipated, as an increased yield of products is obtained, due undoubtedly in part to more advantageous manipulation, but in our opinion also to the different chemical reactions that take place. In the old retort system complete carbonization of the wood took place in about 16 hours, and it required in the oven system 24 hours; yet the temperature of the gases at the neck are higher in the latter, running at a maximum under skilful manipulation to about 330 to 360° C., while in the former case the operation was consummated at not over 290° maximum. This maximum temperature is obtained about midway, or two-thirds, of the run, at which time a very decided exothermic reaction takes place, and care must be exercised that the liquor is not "burned;" *i. e.*, that the temperature is not permitted to rise. If this happens, undue formation of condensation products results, which is immediately indicated by an increased flow of gas and a darkening of the crude liquor. This exothermic reaction, which also takes place in coal distillation, probably accounts for the increase in temperature of the uncondensed gases in the oven system, since no greater proportionate radiation takes place in this system than in that of the retort construction. At this particular stage, on account of the nature of the source of heat (exothermic changes in the wood), a much more uniform temperature throughout the oven is maintained. This gives an excellent illustration of the possibility of considerable alteration, even in the chemistry involved in a process, when we change the mechanical manipulation.

An additional advantage obtained by this oven system is that the charge of residual charcoal may be drawn while the oven is still hot, thus economizing on the fuel necessary for reheating, while a partial cooling is necessary in the hand-drawn retort system. The time lost during this cooling was saved in one design at Dahoga, Pa., where the retort at the conclusion of firing was removed by a crane from its vertical setting and replaced at once by a previously charged retort. It is stated in the meager literature on this industry that "this type is probably most used."¹ This Dahoga installation, though it operated for many years, was the only plant of this type that ever was installed in this country, as far as we are aware, and certainly there is little likelihood of any existing now, for this one was recently converted into an oven plant.

There is still a third system, which should be at least men-

¹ *Met. and Chem. Eng.*, 1910, p. 155.

tioned, namely the kiln system. This was really the second in date of development, but the oven system was so closely allied in design and operation to the original retort system that the two were taken up together. The fact is, we would expect the logical development to have been: charcoal heap—charcoal pit—charcoal kiln—by-product charcoal kiln—retorts—ovens; but this does not appear to have been the case, in this country at least. As in the coke industry, we do not appear to have gone from bee-hive oven to by-product bee-hive oven, directly, but through the by-product oven, an outside and distinct invention. So in this industry, the charcoal kilns were converted into the by-product kiln system only after the development of the retort system.

When charcoal was produced in kilns, from 20 to 86 cords of wood were placed in a kiln constructed of bricks, and wood was used as fuel for carbonizing. The volatile products were lost through the opening provided at the top. With the advent of markets for charcoal and acetate, some charcoal manufacturers placed a pipe connecting this opening with a suction fan and a condenser and thus collected the volatile products. The amount of product thus recovered is but a fraction of that recovered in a retort or oven system. In Pierce's system the flat-topped circular kilns usually hold 50 cords. They are heated by gas burned in a furnace under the kiln. For the sake of brevity, a description of these interesting kiln plants will be omitted since their recovery of by-products is low and they are confined almost exclusively to the blast furnace plants of Michigan, where they are steadily and rapidly being displaced by oven plants.

YIELDS

The low yield from the kiln system, which runs about 100 lbs. acetate of lime and 4 gal. of alcohol per cord, together with the low yields obtained by *rapid or quick* firing, plainly show that there is a mean temperature which gives the maximum yield. Just what the curve of efficiency is, has never been definitely determined in a commercial plant, to our knowledge, though we have given it much experimental attention and others doubtless have worked on it also. In fact, there are so many factors to be considered in a comparison of experimental operations on a commercial scale that while the factors causing increased or lessened results are known, it is difficult to obtain

LOADED WOOD CARS AT OVENS

accurate definite knowledge as to just how much each factor contributes to the results. For example, we know that Michigan plants under approximately the same construction and operation obtain less product per cord than those of New York and Pennsylvania, without certain knowledge of the relative influence of the various contributing factors.

This lack of definite data on a commercial scale, so deplorable in most industries, results from the difficulty of obtaining suc-

cessive tests with all conditions alike, for we well know that any variation, for instance of temperature, has a peculiar relation to the results obtained. In addition, it goes without saying that the variety of species of the wood enters largely into the results obtained, and even the part of the tree used may cause variation. For example, a charge made up wholly of heart-wood will give better yields under the same conditions than a charge of slabs, making due allowance for the bark. Again we have found that, weight for weight, cord-wood obtained from the mature or virgin trees will yield higher than the small second growth; also we are convinced of a noticeable difference in results from wood of the same species grown on high and dry places, and that from low or marshy soil, the latter giving a slightly lower yield. Indeed it is interesting to hear the experienced operator maintain that wood cut in the winter months will give better results than that cut while the sap is running. We are, however, unable to verify this expression, and it is possible that some other factor may have been responsible for this often repeated statement. It is also held by many opera-

START OF OVEN PULLING

tors that, eliminating the consideration of extra fuel necessary for the charring of green or unseasoned wood, the results are not the same in finished product as from well seasoned wood. We can see from these statements that, with so many factors to be considered, it is extremely difficult to determine which enter most into the discrepancies between the various systems: retort, oven and kiln. However, from the purely practical standpoint we do know that there is a marked difference between yields obtained from the different systems as well as from the two general localities extensively operating.

If we compare the results obtained by the same system operated in the Allegheny Mountains with that of the Lake Superior region, we find that by taking the results on a yearly basis from a well operated plant and calculating the wood on a basis of a cord 8×4 by 52 in., there should be obtained conservatively 216 lbs. of 80 per cent acetate of lime, 11.3 gallons of 82 per cent crude wood alcohol, and 52 bushels of charcoal. The yield from the Lake district is slightly under this in alcohol

and decidedly so in the acetate yield, which is 175 to 180 lbs. on continued operation. However, here again we have at least two factors and it is difficult to determine their relative influence. First, in the eastern field, beech predominates while in the other hard maple is in larger proportion, and also slab wood is used much more largely. In fact, we have been unable to obtain data from a continued operation in which at least a small proportion of slabs was not used.

CONSTRUCTION

At this point it may be well to take up the question of construction, especially of the most successful and modern system, that of the oven, which is, with the exception of the mechanical manipulation, similar to the retort system.

The ovens are of uniform height and width, in most plants being 6 ft. 3 in. wide and 8 ft. 4 in. high, the length determining the capacity, running from 26 to 54 ft. and holding from five to ten cords. The steel used in the shell is usually $\frac{3}{8}$ in. while the bottom and back heads are of $\frac{1}{2}$ in. material. Ovens are built either with doors at both ends in order to withdraw the residual charcoal coincidentally with refilling the oven with the wood-loaded cars, or in many cases, with a door at one end, the oven being loaded after the charcoal has been run into the coolers. In any event, the door is hung upon a cast-iron frame riveted on the oven. This frame has a groove of about 1 in. for the use of asbestos packing and the cast-iron door is hung to the frame by riveted cast-iron hinges. The oven is stiffened by means of angle irons riveted perpendicularly on the sides, and on one side near the top are riveted 30 in. cast-iron nozzles, usually two in number, to which are attached the condensers.

The first ovens installed were placed upon piers in the setting, upon which were iron plates and rollers to accommodate expansion of the oven, but in the later settings the ovens are suspended from Tee rails laid across the brick setting at the top and supporting the oven by means of U bolts riveted thereto. The bottom of the oven is equipped with clips for holding the rails which carry the wood cars. These cars vary but slightly in length in different plants, running from 10 ft. 4 in. to 12 ft. 4 in. inside length and about 78 in. high; thus each car holds from 2 to $2\frac{1}{2}$ cords of 52 in. wood. Each has four 18 in. wheels with roller bearings running on $2\frac{7}{16}$ in. turned steel axles.

Immediately in front of the ovens are two charcoal coolers arranged in series and approximately of the size of the ovens. In the first the hot charcoal is drawn from the retort, where it stands for 24 hours. From this cooler it is drawn into the second one for another 24 hours of additional cooling. These coolers are made of very light iron with cast door frames riveted on each end, and light sheet iron doors. No bottoms are required, as the coolers are simply placed on the ground and banked along the sides and doors with loose dirt.

To the nozzles heretofore mentioned are connected the condensers. These are upright tubular condensers, made with copper hoods having a removable head, and are bolted to gun metal heads which are bored for the copper tubes, usually $1\frac{1}{4}$ in. O. D. seamless copper, and expanded into these gun-metal heads, the whole surrounded by a steel water-jacket with water inlet at the bottom and outlet at the top. Connected to a bottom pan bolted on the lower head is a goose-neck trap for trapping off the non-condensable gases, thus separating them from the pyroigneous liquor, which is run into a wooden tank. The gas from the charge is carried into a main gas line which runs to the boilers where the gas is injected under them by a small steam jet. This jet performs the additional and very important function of serving to reduce any back pressure on the ovens, thereby preventing leaks. In order to prevent this suction from becoming too pronounced and drawing over the lighter volatile products through the condenser into the gas line, a butterfly or clapper valve is arranged on the line and regulated

by a spring so set that any undue suction opens the valve, thus relieving the line.

PRIMARY DISTILLATION OPERATION

After this general outline of the first stage of the construction let us follow the operation to this point. After the loaded cars are run into the ovens the doors are pinned shut with taper pins driven into lugs which are parts of the castings of the door frames. The fires are started under the ovens and in about an

CHARCOAL COOLERS IN A NEW YORK PLANT

hour the water distillation and first dissociation begins, the distillate being of a very light yellow color and having a tannin-like odor, and running about $2\frac{1}{2}$ per cent of acid during the first hour when well seasoned wood is used. Gradually this acid content rises until about the fifteenth hour, when the content should be running from 12 to 14 per cent. After a few hours running, a small percentage of light tar begins to appear and steadily increases until at the latter end of the run tar is by far the larger constituent of the distillate. The acid content of the watery constituent gradually diminishes until it is almost negligible.

The product obtained under the usual practice, with average wood of at least a year's seasoning, is from 215 to 220 gallons of crude pyroligneous product giving an acid titration of 8 to $8\frac{1}{2}$ per cent and an alcohol content of 4 to $4\frac{1}{2}$ per cent. The tar yield is from 22 to 25 gallons to the cord, with a residual charcoal in the cars of about 52 bushels of 20 lbs. each. With

DUMPING CHARCOAL

green or unseasoned wood the total distillate is diluted and will run from 275 to 325 gallons of liquid.

In the early stages of the run the non-condensable gases are largely air and carbon dioxide. Therefore for the first few hours, and even after much of the hydrocarbons and carbon monoxide are accumulating in the gases, they have no fuel value. However, the percentage of carbon dioxide diminishes rapidly, and from the middle to the end of the run the oven gas

has an important fuel value. The total amount of gas will run from 11,000 to 12,000 cu. ft. per cord.

As we have already intimated, the influence of the temperature of dissociation is extremely important, and the yields are very dependent upon its regulation, *e. g.*, if, during the height of the run, the temperature at the neck of the oven materially exceeds 750° F. much greater volumes of non-condensable gases are being formed, together with an excessive amount of tar, and it is quite possible to lose from 5 to even 10 per cent of charcoal, together with a serious loss both of alcohol and acid content. Rapid firing, however, without reaching excessive limits, will materially increase the acid content at the expense of the alcohol, while slow even firing, operating the oven at a period in excess of the usual 24 hours, will increase the alcohol production somewhat and lessen that of the acid.

The interesting results obtained from excessive firing arise, as we hope to show later, both from a difference in the chemical dissociation and also from the subsequent formation of condensation products.

In drawing the hot charcoal from the ovens, quantities of combustible gas remaining in the ovens would cause explosion on the opening of the doors. This is obviated by the introduction of live steam into the front of the oven for a short time before the drawing is made.

HANDLING THE CRUDE DISTILLATE

The crude distillate or pyroligneous acid, called in the works "raw liquor," is first pumped into a series of wooden settling tubs, connected by brass piping. The first tub, where the greatest amount of tar settles, is connected near its top with an overflow pipe to the second tub. The second tub is connected with the third by a pipe somewhat lower, and this series of drops is continued so that the last tub is drawn from within about 18 in. from the bottom. The raw liquor thus settled free from tar is as a rule continuously run into the copper stills. These copper stills are for the purpose of distilling off the lighter volatile products away from the tars and oils which remained in solution in the settled liquor. They are from 7 to 10 feet in diameter with proportionate height, made of 12 to 14 gauge cold-rolled copper, and equipped with a steam coil, usually of 3 or 4 in. seamless drawn copper tubing starting from about 18 in. from the top and running helically around the walls and on the bottom to midway between the center and periphery. For a 2,000 gallon still, from 300 to 400 sq. ft. of heating surface should be used. In addition each still should be supplied with a live steam jet applied at the bottom and preferably through a copper ring having small steam openings.

After a copper still has run continuously for some time it gradually becomes filled with deposited tar and its flow of distillate diminishes. When this has reached a stage of very slow production, as it does every four days, the feed valve is closed and the live steam jet is opened, distilling the tar with live steam, thus obtaining considerable quantities of acid and light oils. This latter distillate is run into separate tubs, where the light oil on the top is withdrawn before the acid liquor is pumped into the original copper still distillate, which has been run into a wooden "mixing tub." The tar from the settling tubs is usually distilled in a separate tar still, sometimes made of wood, and distilled as has been that just described. This practice is followed as it permits the separation of the oils independently and without allowing them to come in contact with the original distillate, in which a portion would dissolve; thus the bulk of the distillate contains less impurities by this practice.

The mixing tub is usually from 12 to 14 ft. in diameter and 4 ft. high, having a very heavy wooden stirring arm fastened to the end of a heavy shaft, operated by a slow moving gear at the top. Slaked lime is slowly added to the liquor in the tub,

which contains its own indicator, the liquid changing at the neutral point to a pronounced wine color. For maximum yields, however, probably owing to the acetic esters present, the addition of lime should be continued until this color has been followed by a yellowish one and until a bead appears or bubbles form on the surface and disappear slowly. A very small further addition of lime at this point causes the acetate to stick to the pans when evaporated and prevents satisfactory crystallization. Under-neutralization also causes inferior quality in crystallization, and before final drying the acetate has a decided black color instead of a rich yellow-brown; also there is an accompanying loss in product owing to the esters lost.

From the mixing tub the neutralized liquor is jetted or run into an iron still known in practice as the "lime lee" still. In a few plants this is equipped with either Burcey pans, or in some more modern works with a fractionating column, although most plants do not fractionate at this point. The customary practice is simply ordinary distillation. The crude alcohol starts over at about 20 per cent on a Tralle scale but quickly rises to 35 and at times 40 per cent, gradually dropping in percentage to from 15 to 20, at which strength it continues for some time and then drops rapidly to 5 or 6 per cent, at which point it seems to hold stationary again for a long period. After all of the alcohol has been distilled over, a valve in the vapor neck is closed and the residual acetate solution is blown, by means of

SODA STILL—WOOD ALCOHOL REFINERY

live steam pressure applied in the still, to an iron settling pan above the top of the ovens, where the excess lime and insoluble impurities settle out in a few hours. The acetate liquor is then drawn into flat steam pans having a jacketed bottom for steam supply, the pans usually being about 16 ft. long by 8 ft. wide and 18 in. deep. Here the acetate liquor is boiled rapidly until crystals appear on the surface near the edges, when the steam is reduced so that the crystalline film which forms at once on the top does not break, which would cause it to fall to the bottom and form a hard crust. Care must be exercised in this "seeding" process as it is called, or a great quantity of this exceedingly hard crust or scale will be produced, which is unfit for the market. After this "seeding down" has continued until the whole mass in the pan has reached a very thick mushy consistency, it is shoveled out on the acetate or "kiln floor," which is a smooth brick or tile covered floor over the top of the ovens. Here the acetate is continually turned until dried and ready for sacking and shipment.

The "weak alcohol" from the "lime lee" still, containing from 9 to 12 per cent of acetone and alcohol, is accumulated and distilled in an iron still having either 5 or 6 Burcey pans or a column. This distillate is the final crude product produced by the works, and is usually shipped to a central refinery, although a few plants refine their own product. In this distillation all the product coming over above 60 per cent is run into

the storage tank, and the lower fraction is put back with the lime lee distillate for further distillation. The finished crude is sold on a basis of 82 per cent and contains about 16 per cent acetone.

Returning to the copper still residue, the tar from these stills, as well as that from the tar still, is run into a sump and from there jetted under the boilers for fuel. The two fuel products, gas and tar, furnish approximately 35 or 40 per cent of the necessary fuel for all operations.

Before leaving this subject of operation it might be said that many of the earlier plants omitted this copper still distillation, neutralizing the raw liquor directly after settling and thereby obtaining the "brown acetate" of commerce, which is today practically without market. This brown acetate runs from 60 to 65 per cent acetate.

PRODUCTION COSTS

The costs of producing the products above mentioned have been on a constantly increasing scale, owing in a large measure to the increasing cost of cord-wood and stumpage. During the past fifteen years, in the eastern district, stumpage in suitable location has been sold for as low as 15 cents per cord, while today 75 cents to \$1.00 is the approximate average. The cost of cutting varies from \$1.15 to \$1.35 per cord in fair timber and in scattered timber \$1.50 is sometimes paid. Today, including transportation, piling, etc., this will make the cost on board oven cars \$4.00 to \$5.00 per cord, though wood is being put in for as low as \$3.50 by users of mill-waste or by those having long standing holdings.

This gradual rise in the cost of wood is due to natural causes, among which are the increase in stumpage cost due to the ever-decreasing supply, and the natural rise in labor, together with increased length and cost of transportation. With the exception of one possibility, there appears to be no hope of any future lowering of costs. This one exception, a new cord-wood splitting machine, appears from our examination to have merit, and presents an entirely new system of cord-wood production. It is generally conceded that cutting and hauling the large log is less expensive than cutting and handling the cord-wood at the stump. The tree is therefore cut in logs and large limbs and transported to the machine, where a slide carries it first to a cross-cut saw, which cuts it into the proper lengths. The sawed pieces continue to a platform where they are either split in halves or quartered by one blow. The split wood automatically moves by a slide to the railroad car or wagon. It is claimed that under suitable conditions 50 cents per cord, or in some instances even more, may be thus saved. We have been unable to see continued operations of this machine as yet, and consequently can not verify this saving cost, but are inclined to believe that at least some reduction in wood cost is possible by this means.

The cost of fuel for a crude works, in addition to the tar and gas used, of course varies with the available supply. In the New York and Pennsylvania districts, when coal is used, this cost will amount on an average to \$1.15 per cord, and when natural gas at 15 cents per 1000 cu. ft. is used the cost is but slightly more. The labor at the plant is usually counted at \$1.25 per cord.

The following summation of production costs will be generally accepted as a fair basis in considering investment in new installations.

PRODUCTION COSTS PER CORD OF WOOD	
Wood (maximum) per cord.....	\$5.00
Fuel.....	1.15
Labor.....	1.25
Lime, 0.96 bu. at 19 cents.....	0.18
Bags (acetate).....	0.14
Freight (acetate) 16 cents per 100.....	0.35
Freight (alcohol).....	0.10
Selling commission acetate.....	0.11
Insurance 0.068 per cord	} 0.57
Taxes 0.112 per cord	
General expense	
Total.....	\$8.85

PRODUCTION VALUES

With the present slump in markets the values herein set down are low, especially that for acetate of lime. The past 10-year average would be about \$2.00 per hundred, while at present the market is slow at \$1.75 per hundred.

Alcohol (82%), 11 gal. at 25 cents.....	\$2.75
Acetate of lime, 216 lbs. at 1.75.....	3.78
Charcoal, 52 bu. net at 6 1/4 cents.....	3.38
Total crude value.....	\$9.91

This gives a production cost of \$8.85 and a sales value of \$9.91 per cord. It will be noted that no selling costs except in the case of acetate of lime have been included, because, in the works not refining or making the finished products, which are in number far in excess of those who do refine, the alcohol is sold under contract to the refiner, and the charcoal is either, as in the case of those plants in the Lake district, consumed at iron furnaces in connection with the plants, or as in the eastern field handled by a general sales agency controlled by the manufacturers, proportionately to their daily production capacity, yielding to the producer about the above-mentioned figure at present.

These production costs are based on the yields in the eastern field, which as pointed out were higher than those of the Lake region. This discrepancy is compensated, however, by the fuel and wood costs being lower in the Lake region, owing to the fact that in most instances mill-waste is used. The general relation between the two fields of operation, in this production cost and product value, is therefore approximately the same. It is undoubtedly true that many manufacturers can show much greater net profit than the above figures indicate, because of cheaper wood and fuel and in some instances because of plant design.

COSTS OF INSTALLATION

Eliminating the cost of wood supply, \$2000 per cord per day production is counted as a reasonable installation cost for a plant as described, for producing crude products. For instance, the cost of boilers, ovens, cars, coolers, pumps, etc., is substantially \$650. Copper work, including stills, condensers, piping, etc., approximates \$450. Brickwork for ovens and boiler settings, etc., will come to about \$190. These three general items are more or less about a fixed estimate, varying of course somewhat upon the price of materials at the time. The location, buildings and general costs vary with the design and the desire of the builder. However, the above stated general figure of \$2000 is a fairly accurate estimate, unless elaborate construction is undertaken.

LARGE SCALE EXPERIMENTATION

Realizing the possibilities and necessity of counterbalancing the increasing cost of production, several large operators have made extensive large scale experiments with a view to increasing yields and lowering production costs. Unfortunately the results obtained thus far have not justified the expenditures made, in spite of many optimistic claims,¹ owing in a measure to the many unlooked-for complications which arise between the laboratory and the works scale. Of the great amount of work which has been done along this line with which we happen to be familiar, space permits the citation of but a few illustrative instances.

In the fall of 1908 certain men largely interested in the business went to Germany to investigate developments there and returned with a process for eliminating the copper stills and making gray acetate of lime directly from raw liquor, a proposal which had already been made and studied in this country. This process had been reputed to be in successful operation in Germany. It was in fact merely the application of the cup column to the neck of the ovens. The cups and openings were of course of

larger size than in the customary column, but in effect this really was all that could be claimed for the new appliance. It was held that the pyroligneous vapors, passing through the condensed tarry liquor, were freed from their impurities by this means and that gray acetate could be produced from the resulting raw liquor by direct neutralization. Educated or impressed by the then incomplete American investigations mentioned, these men permitted themselves to be carried away by the assurances of the German engineers and, as has happened several times in our industrial chemical development in more or less identical circumstances, without any experimental investigation they equipped a large plant with this apparatus at an enormous expense, the copper work for which alone exceeded \$80,000. The plant did not prove satisfactory.

It will be at once seen from foregoing statements in this article, that there were two fundamental reasons which explain, in our opinion, the failure to obtain the desired results. *First*, as pointed out, mechanical clogging of the apparatus would occur, as was the case in the original worm condenser; and *second*, as we believe, the valuable products readily form condensation products with the unsaturated hydrocarbons and aldehydes present in the distillate. Certainly the passing of these vapors through the hot tarry liquid would tend to increase this chemical reaction. Moreover, the apparatus caused back pressure on the ovens and required a suction to overcome this objection. This increase of suction would tend to bring the vapors through the cups and the condensers at a more rapid rate, which of course would lessen their efficiency.

Later another process, also brought from Germany, was tried, which was in effect the same as the one above described, except that the vapors were forced through a body of hot tar for the purpose of washing them, but this also was not successful. In our opinion this failure was for the same fundamental chemical reason as mentioned above.

A process for accomplishing this result, but not as yet entirely developed, from which some very satisfactory results have been obtained, is based upon a totally different theory from those mentioned. This method places a fractional condenser on the neck of the oven, which is maintained at a temperature sufficient to liquefy the high boiling tars and oils. These, however, are largely mechanically carried along by the uncondensed and non-condensable gases, while a part of the tar is run off through a gooseneck on the bottom of the fractional condenser; the uncondensed vapors and the non-condensable gases with their suspended tar and oil liquids are carried into the bottom of a centrifugal basket or drum, installed in a copper container and having small perforations inside of the drum, which is supplied with wings to give a rapid motion to the gases. This centrifugal action throws the liquid particles or mist against the side of the container, on which are defectors set at an angle opposite to the direction of the moving drum. Thus the condensed bodies are forced to the bottom of the container and out of the zone of circulation, through a gooseneck into the tar receiver, while the vapors continue to a second condenser where they are condensed, the wood gas being trapped off in the usual manner.

This process has given uniformly increased yields of product but exact data as to saving, yields and cost of operation have not been determined as yet. A little over 1 H. P. is necessary for the operation of a 7-cord machine until speed is attained; then 3/4 H. P. is necessary.

The increase in product yield with this method is probably due to the fact that the vapor contact with the condensed and unsaturated bodies present is reduced to a minimum.

Another attempt at fuel conservation, which it seems should be possible of accomplishment, relates to the evaporation of the acetate liquor. As has been seen, much heat is necessary for this evaporation, and this method attempts to utilize the high

¹ *Met. and Chem. Eng.*, 1910, p. 434.

temperatures of the gases at the oven's neck before they enter the condensers. We are not aware of any installation of this nature although patents have been issued for accomplishing this end. Nevertheless, as it involves what appears to be a wholly mechanical manipulation there should be no great difficulty in saving at least a portion of this waste heat. Here again it can be seen that experimentation to accomplish this end is costly, since all experimental changes necessitate considerable financial outlay if tried on a large oven, and also involve interruption of regular operation which must from time to time inevitably occur.

Under this head of experimentation may properly come an elaborate attempt to utilize hardwood sawdust for alcohol manufacture. This was undertaken in 1903 by a well known lumber company in Cincinnati, who endeavored to destructively distil sawdust continuously by means of an endless belt which carried the sawdust through an oven for the purpose of carbonizing, tapping off the pyroigneous vapors and obtaining an uninterrupted production of charcoal, which was briquetted. The distillate was handled in the usual manner.

It can be readily seen that sawdust can not be destructively distilled in bulk, as is cordwood, for the insulating space between the particles prevents the heat from going through the mass. This explains the reason for the endless belt.

No expense was spared either in buildings or equipment, the buildings being of stone and the equipment of the best, the whole costing probably \$200,000. Notwithstanding this outlay, the operation was a complete failure, the plant being dismantled within a year and the buildings used for other manufacture. The high fuel cost in attempting to overcome the resistance to radiation in such a mass was an important item in the high production cost which resulted in the failure. In addition, sawdust charcoal is a more dangerous fire-risk than common charcoal, in the handling of which in the ordinary plant, special arrangements are necessary.

Within the past five years some plants have installed double and triple effect evaporators with a view to fuel economy, and reports as to the results are very contradictory, but the continued use indicates at least partial success. One Michigan plant is successfully drying its acetate with a drum and belt drier. Vacuum drying for acetate has been tried, but we are unaware of any success as the resultant product is a powder, while the market demands the crystal.

REFINING

From the time of the formation of the already mentioned Burcey Refining Company, the usual plan of manufacture contemplated only the production of the crude product as described, selling the products, those of the Eastern field, largely to a Buffalo refinery and the Michigan manufacturers to Detroit. Up until the passage of the Denatured Alcohol Bill by Congress they received a price usually ranging from 40 to 75 cents a gallon for the crude alcohol. After the passage of this act, and with the purchase of the Detroit plant by the one at Buffalo, the price of crude was at times as low as 15 cents per gallon, and during this period of adjustment intense depression prevailed among the crude producers, as it was feared that ethyl alcohol would largely supplant methyl. It was found, however, that the trade, particularly the paint trade, preferred wood alcohol even at a slightly higher price, and this fact virtually saved the industry. With the formation of a refining company among eastern crude operators representing somewhat over 1,000 cords daily production, and the erection of a refinery at Olean, N. Y., the monopolistic control of refining was immediately reduced. A very acceptable dead rental was paid to the crude producers for their uncompleted refinery, as well as a substantial increase for their crude product, which eventually reached and is maintained at about 25 cents per gallon of 82 per cent product.

The method first employed by refiners was merely that of fractional distillation, and we all perhaps remember the yellow, ill-smelling wood alcohol of commerce. Later, and for a long time, bleaching powder and sulfuric acid were used before the final distillation. This materially improved the product, but with the improvement of the columns used, treatment with sodium hydroxide and distillation is all that is necessary. The NaOH serves the purpose of polymerizing the aldehyde impurities and saponifying the tarry matter. The esters present are also affected. The formerly objectionable acetone present, which distilled with the heads, now has its own market value under the name of acetone-alcohol and is used in varnish removers, as a solvent in the rubber and other industries and also as a carrier for acetylene gas in lighting systems. Thus this 16 to 18 per cent of impurity is now in fact an added value to the refiner.

MARKETS

Prior to the present unfortunate European situation, the average production of acetate of lime from the manufacturers in this country amounted to approximately 7,000 tons monthly, and we are informed that the total European and Canadian production is almost equal to this, of which tonnage Austria-Hungary furnished about two-thirds. The plants in Europe, however, are usually on a very small scale in comparison with those in this country. Sweden alone has plants of a size in any way comparable with the larger ones here. Of the production of acetate in this country, from 50 to 60 per cent is exported, a considerable percentage to or through Belgium. Of course, it will be understood that these statements refer to the market situation before it became demoralized by the European war, although for a year prior to the declaration of war there had been a pronounced stagnation of the markets both at home and abroad.

Of the acetate of lime consumed in this country, by far the largest percentage goes into acetic acid production. The hardwood industry is therefore vitally connected with this acetic acid industry, and one of the most important developments in the last decade is the successful beginning made in combining the crude manufacture and this refined product manufacture in the same plant. Such combination plants have shown crude acetic acid yields in continuous operations running 92 to 96 per cent of theory using 5 per cent sulfuric acid in excess of that theoretically required, and giving on a continued daily production of 4,000 lbs., with a yield of 91 per cent of the possible theoretical yield, refined acetic acid of the finest double distilled quality.

The chief individual acetic acid market demands in this country are from the manufacture of white lead, paint colors, and the textile and leather industries, although in the aggregate the small consumers, such as the laundries, ink and drug trades, use great quantities. This country prohibits the manufacture of acetic acid vinegar but its production is permitted abroad.

The consumption of export acetone produced in this country is chiefly by England, and Italy is just now a large consumer. The industry is in a particularly thriving condition at the present time, owing to the use of acetone in production of explosives for the European war. There should be obtained 20 to 22 lbs. of acetone per 100 lbs. of acetate used, and in connection with the crude works acetone production is very profitable, as the general operating expenses are low. Of course its use in this country was very large for explosive manufacture and for the production of chloroform under the well-known Rump patents, regarding which there was so much patent litigation some years ago and which as you know were upheld. In comparatively late years, however, a large Michigan concern has produced chloroform commercially from carbon tetrachloride, which has probably influenced somewhat the acetate

consumption for this manufacture. There are 4 plants in the United States producing acetone for the market and within the past week ground has been broken in Pennsylvania for a 5th plant. There is but one plant operating in Canada to our knowledge.

Ten years ago the steel industry consumed by far the major percentage of charcoal produced. This percentage, however, has decreased considerably, owing to the improved methods for steel production. Many industries such as cutlery and car wheel manufacture demanded charcoal iron, but in these and many other instances coke iron has been substituted. This falling off in the proportionate use of charcoal in iron production has been made up in an increased domestic demand. Charcoal is now sold in small paper sacks in many cities for use both in hotels and private families.

The production of crude wood alcohol amounts to between 10,000,000 and 11,000,000 gallons per year, and of course it has innumerable uses, including those of the paint industry which is the largest single user.

Space will not permit, in a general article such as this, of going into details regarding production and markets of the other products such as formaldehyde, creosote, etc., which are directly or indirectly derived from hardwood distillation.

DISTRIBUTION OF PLANTS

Although not in agreement with any published figures, we place the distribution of distillation plants as follows:

LOCATION	No. OF PLANTS			CORDS DAILY			TOTAL No.	
	Oven	Kiln	Retort	Oven	Kiln	Retort	Plants	Cords
Michigan.....	8	..	..	1032	..	..	13	2082
Pennsylvania.....	33	..	..	1424	..	..	46	1639
New York.....	7	..	..	256	..	215	24	618
Wisconsin.....	2	..	..	96	..	362	3	346
	..	1 (oven-kiln)	..	..	250	..	1	20
Kentucky.....	1	..	..	20	..	..	1	49
West Virginia.....	1	..	..	49	..	..	1	32
Tennessee.....	1(a)	..	..	32	..	..	1	16
Vermont.....	..	..	1	..	..	16	..	..
TOTAL IN U. S.....	53	6	31	2909	1300	593	90	4802
Canada.....	9	..	..	424	..	..	10	472
TOTAL IN AMERICA...	62	6	32	3333	1300	641	100	5274

(a) A recently constructed 16-cord retort plant now being converted into an oven plant.

These figures will mean more if we remember that 5,000 cords of wood represent a pile 4 ft. high 52 in. wide and 7.5 miles long weighing when seasoned approximately 10,000 tons, thus showing the importance and size of the industry in this country.

CONCLUSION

It will be evident that the chief economic drawback in the present manufacture is the fact that most of the plants do not manufacture the finished products; thus in general it may be inferred that two sets of freight charges, commissions and handling expenses are required before the products reach the consumer. For instance, with acetate of lime at \$2.00 the net return to the producer is about \$1.75 per hundred. A large part of this difference could easily be saved by manufacturing the finished product at the crude plants.

Hitherto such combinations of crude and finished product manufacture have been thwarted in the majority of cases by an almost complete monopoly of the finished products by certain groups of chemical manufacturers, who by various manipulations maintained control of markets, or who were able to dominate the situation, as in the case of acetic acid, by their regional control of sulfuric acid manufacture, or even attempted control of pyrites importation. The violent market fights which have developed because of these facts have been carried through successfully in more than one instance by the crude manufacturer, because of his advantageous situation and it is

certain that these attempts, therefore, to thwart the natural economic development of this industry will sooner or later collapse to a great extent, or the refiner will be compelled to either go into the crude business himself or combine with the crude manufacturer.

We have attempted to show the difficulties met and the expensive experimentation that has been undertaken in the past for the improvement of this industry, and have pointed out some of the results thus obtained. When it is remembered that German statistics claim that but one of 15 serious trials of new chemical processes are successful and that this percentage amply pays for the losses entailed in the 14 unsuccessful ones, it can be seen that while these expenditures are individually unfortunate, in the aggregate this law of averages will probably hold good in the development of this industry, for its economic position demands the solution of the problems indicated.

COLUMBUS, OHIO

DISCUSSION

By H. O. CHUTE

I am sure the thanks of the Institute are due Messrs. French and Withrow for this contribution to the history and present status in the U. S. of wood distillation and the presentation of the large number of illustrations showing actual present working plants.

While much has been previously published on this subject it has in most cases been by those familiar only with the European situation or who had been consulting European works of the subject.

It is a singular fact that most of the places named as sites of the first works still have working plants and the people and firms then engaged are still in the business. This indicates that the location of a wood distilling plant is not a temporary affair but that the wood supply usually lasts for a generation.

While the statement is true that the carbonization is an exothermic reaction yet the phenomenon is not observed unless the wood is fairly dry, *i. e.*, with from 15 to 20 per cent water. With excess of water the heat disengaged is not sufficient to evaporate the water so there is no progressive action toward the center of the retort except as produced by the fire, and the transmission of heat to the center of the retort from the walls is difficult. This shows the necessity of having dry wood for successful work. Owing to this exothermic reaction the control of the temperature inside the retort by any method of heat insulation or surrounding thermal baths is foredoomed to failure.

While the first operators brought over the Scotch methods yet later arrivals including Burcey, brought over the French methods as then and now practiced. The Burcey pans are merely the French Pistorius pans used in the rectification of grain alcohol and the Otto plant of vertical retorts located at Sargeant near Dahoga, Pa., which has been described, was built by Burcey on the French system. These worked successfully for many years though they were never duplicated in this country.

It must be remembered that wood distillation in the U. S. has developed along two different lines. The charcoal iron industry was introduced at a very early period, in fact shortly after the settlement of the first colonists and this necessitated a supply of charcoal. No attempts to save any of the by-products were tried or accomplished till Dr. Pierce introduced his system of sucking off the smoke or vapors from the kilns with a fan and recovering the alcohol and acetate from them by condensation. With the large amount of charcoal necessary to supply the furnaces and with wood plentiful and labor scarce it became advisable to use kilns; these can use green wood and wood in the form of logs and quite rough, while the retorts required the wood to be carefully cut and split to a six-inch face and dried for about a year.