

It is of interest in this connection to give the results of a commercial temperature-control test lasting about three weeks. The plant was of about 50 cords per day capacity, distilling wood composed of about three-quarters maple and one-quarter birch. Approximately 1100 cords were distilled during the test. The wood had not been seasoned for more than 8 months and could be considered very comparable with the wet wood used in the laboratory tests described in this paper. The yield of acetate of lime in the commercial test was increased about 12 per cent over former uncontrolled practice. As the plant had been using wood of the same quality prior to the test, the results of these experiments in the laboratory, comparing wet uncontrolled with wet controlled runs for birch and maple, indicate that the increase in acetate was due largely to the maple. The increases obtained in the plant and in these tests check surprisingly well.

WOOD ALCOHOL—The influence of moisture on the wood alcohol is almost as pronounced as on the acids. The beech, with the higher moisture, gave decidedly the highest yield of wood alcohol when controlled and even the uncontrolled wet beech gave a little more alcohol than either the controlled or uncontrolled dry beech. The alcohol results, therefore, also indicate that beech should not be seasoned too long and that the distillation should be carefully controlled.

In the case of maple, both uncontrolled and controlled wet wood gave higher yields of alcohol than the dry uncontrolled runs, showing that moisture also favors the alcohol in this species. However, decidedly the highest yield was obtained from the dry controlled distillation, verifying former experiments. Considering both alcohol and acetate, the data would indicate that for best results from maple the wood should be only moderately seasoned and the distillation carefully controlled.

The alcohol results for birch showed that without doubt this species should be well seasoned for the highest yields, as the wetter wood gave much smaller amounts of alcohol. The dry controlled birch gave the highest yield. Since the acetate yield for the dry controlled birch was so nearly the same as from the wood with higher moisture, although slightly lower, it would seem, considering both products, that birch should be well seasoned and the distillation carefully controlled for the best returns. Birch, then, seems to be different from beech and maple as regards the influence of moisture on the more valuable products, such as alcohol, acetic acid and charcoal.

SUMMARY

I—Semi-commercial laboratory destructive distillations were made with beech, birch and maple. One lot was seasoned for about 18 months and another lot about 6 months. The results showed that moisture had a decidedly favorable influence on the yields of acetic acid when the distillations were controlled after the exothermic reaction had begun. The data indicate that beech and maple should be distilled only after moderate seasoning in order to secure the highest yields of acetic acid, provided the distillations are carefully controlled. The yields of acetic acid from

birch which had been well seasoned were so nearly the same as from the wood which had been seasoned only about 6 months that there is no preference for this species, provided the distillations are controlled.

II—If the recovery of formic acid should become important in the distillation of hardwoods, the experiments showed that the highest yields were obtained from rapid (uncontrolled) distillations of wet wood, this being particularly true of beech.

III—A commercial temperature-control test using wood seasoned for only about 8 months gave practically the same increases in acetate as obtained in the laboratory tests.

IV—Former experiments showing the value of temperature control in increasing the yield of wood alcohol have been verified in these tests. Additional data on the influence of moisture shows that an excess tends to give still higher yields of alcohol in the case of beech and the same tendency is shown to a lesser degree for maple. With birch, however, the drier wood is preferred for the highest alcohol yields and although moisture favors the alcohol to a slight extent with maple as compared to uncontrolled dry distillation, the controlled dry maple runs gave decidedly the highest yields of alcohol for that species.

V—An excess of moisture in general gives lower tar and charcoal yields, but beech is the exception for tar and birch for charcoal.

FOREST PRODUCTS LABORATORY
MADISON, WISCONSIN

THE EFFECT OF CATALYZERS ON THE YIELD OF PRODUCTS IN THE DESTRUCTIVE DISTILLATION OF HARDWOODS

By R. C. PALMER¹

Received October 17, 1917

PURPOSE OF WORK

The purpose of the work was to study the influence of various reagents or catalyzers on the formation of wood alcohol, acetic acid, etc. (1) during the primary reaction occurring in the destructive distillation of wood and (2) during any secondary reactions that take place between the original products.

SCOPE OF WORK

The experiments conducted so far have been preliminary and include a study of the effect of hydrolyzing acid catalyzers in an attempt to induce the maximum splitting off of acetyl or formyl groups from the cellulose or ligno-cellulose and the hydrolysis of these groups to acetic and formic acids or the decomposition of intermediate products, such as carbohydrates, into these products. Any influence on the formation of other products was, of course, noted. Phosphoric acid was selected as a catalyzer in these preliminary tests as being the most adaptable. It could be readily injected into the wood in solution and was non-volatile, being transformed into the metaphosphoric acid at the maximum temperature of the destructive distillation of wood.

Maple and beech reduced to chips about 1 in. by

¹ Acknowledgment is made to Mr. H. Cloukey for making a number of the analyses.

1/2 in. by 2 in. in a pulpwood chipper were used. All tests were made in the autoclave, holding about 5 lbs. of wood, previously described in the report of tests on the effect of pressure on the yield of products, and the manner of making the tests was in general similar to that described in that paper.¹

Preliminary experiments were also made on the distillation of wood in the presence of, or saturated with, wood tar, in a study of the possibility of splitting off methyl groups from the tar to form methyl alcohol.

DETAILS OF METHOD

The catalyzer was brought into intimate contact with the wood by placing the chips in enough of a water solution of the acid to completely immerse them and the solution brought to the boiling point to drive out the air in the wood. The chips were then cooled in the solution, causing a penetration of weak acid into the wood. The excess liquid was drained off and the chips weighed immediately after no more liquid dripped from them. The amount of catalyzer present was then calculated from the weight of solution absorbed, knowing previously the strength of the solution. Titrations were made before and after treating the chips with catalyzer solution in enough of the tests to show that no appreciable selective absorption took place. In the case of acid solution the

MAPLE—The results with maple chips, using phosphoric acid as a catalyzer, are given in Table I.

Run 1 was made without any catalyzer at atmospheric pressure for comparison with catalyzer runs and the yields are about the same as previous tests in the same apparatus.

The experiments with maple showed no striking increase in the valuable products, although several of the other products showed decided differences when the wood was distilled in the presence of the acid. In Runs 3 and 4 the lower yields of acid are undoubtedly due to the effect of pressure, as noted in previous work. The catalyzer apparently seemed to have had a detrimental rather than a beneficial effect on the acid yield in this series.

The yields of wood alcohol are quite variable. Bearing in mind that in previous work with this apparatus the alcohol yields were somewhat lower than commercial yields and also lower than laboratory yields in a larger retort,¹ several of the runs showed decidedly more alcohol than the standard in the autoclave but not higher than controlled laboratory runs in the larger retort. The highest yield in the series was 2.18 per cent (Run 2) as compared with 1.37 per cent from the standard, an increase of 60 per cent. Whether the manner of distillation was responsible for the higher yields or the catalyzer was playing an

TABLE I—DISTILLATION OF MAPLE IN PRESENCE OF PHOSPHORIC ACID(a)

Run No.	Catalyzer Per cent	Max. Temp.		Pressure Lbs.	Pyro. Acid		Tar			Total Acid as Acetic	Acetic Acid	Formic Acid	Wood Alcohol	Moisture
		Retort	Bath		Less Moisture	Charcoal	Gas	Dissolved	Settled					
1.....	0	312	470	0	35.05	39.15	22.66	5.36	3.14	5.81	4.56	0.79	1.37	41.35
2.....	7.59	332	465	0	41.27	44.90	13.85	1.85	Neg.	5.05	4.52	0.40	2.18	45.35
3.....	7.92	323	470	60	40.57	46.20	13.40	1.17	Neg.	4.65	4.12	0.41	1.29	39.80
4.....	2.72	324	470	90	40.20	45.95	14.03	0.45	Neg.	4.55	3.98	0.43	1.46	36.00
5.....	23.75	320	470	60	40.80	44.05	15.15	0.72	Neg.	5.30	4.20	0.84	1.81	34.00

(a) Results are all in percentage of the dry charge.

chips were always apparently wet to the center. With the wood tar or creosote only a few of the largest chips were not completely penetrated by this method.

The chips, after treatment, were allowed to dry in some cases and in others were distilled immediately. Some runs were made putting the wood in a cold retort and others putting the wood in the retort previously heated to a temperature higher than the destructive distillation point.

The different control data were taken and analyses made as in previous work, including the (1) moisture content of the charge, (2) weight of distillate and charcoal, and (3) percentage of total acid, acetic acid, formic acid, settled and soluble tar, wood alcohol, and acetone, in the distillate.

RESULTS

The results in general are not very concordant, but several striking effects have been noted, indicating that when conditions are favorable the yields of primary products may be affected in a very marked degree by the presence of catalyzers. Not enough work has been done, however, to establish all the factors that influence the part the catalyzer plays in the decomposition reaction so that the results can be readily duplicated. The influence of various reagents that would poison the action of the catalyzer has not been worked out.

important part in the reaction cannot be determined, although the data would indicate the latter.

The distillation of wood in the presence of an acid catalyzer has a marked effect on the tar, on the charcoal, and on the pyroligneous acid (including only the water formed by destructive distillation). The tar which usually settled out of the pyroligneous acid is apparently practically destroyed and the tar normally dissolved in the acid liquor is reduced at least 50 per cent in most of the runs distilled at atmospheric pressure. Previous work showed that pressure alone decreased the tar very much, so it was to be expected that distillations under pressure in the presence of the acid catalyzer would give even smaller yields of tar. Part of the tar can, no doubt, be accounted for by the higher yields of charcoal which averaged about 44.5 per cent compared to a normal of about 40 per cent, although since no appreciable amount of tar coke was noted in the retort any coking of the tar must have taken place in the charcoal or, in other words, the tar was decomposed practically in the wood at the moment of formation. Part of the tar can also probably be accounted for in the pyroligneous acid which was increased from 35 to 40.5 per cent, as during the analyses of the distillates, especially after neutralization with alkali, much larger amounts of creosote oil than usual were

¹ THIS JOURNAL, 6 (1914), 890.

¹ See apparatus described in tests on temperature control, THIS JOURNAL, 7 (1915), 663.

noted in the distillate. No qualitative or quantitative study has been made of this oil, but the yields and properties compared to normal creosote will be thoroughly investigated.

The increase in charcoal and oils in the acid liquor does not, however, account for all the tar, especially since the gas yield is lower than normal, so it is apparent that some other factors are entering into the reaction.

BEECH—The results with beech chips using phosphoric acid as the catalyzer are given in Table II. In this series a more comprehensive study of variables was attempted.

Run 6 was made without a catalyzer at atmospheric pressure. Unfortunately, no previous work has been done with beech in the autoclave with which the yields from this run, which was made as a standard, can be compared. The results are quite different from previous work with beech in the large retort, which gave 1.87 per cent alcohol and 5.87 per cent total acid compared with the autoclave run of 0.99 per cent alcohol, 7.03 per cent total acid and 5.13 per cent acetic acid. We will assume an alcohol yield of 1.37 per cent instead of 0.99 per cent standard, since this was the yield obtained from maple chips in the autoclave. This assumption can be reasonably made be-

Runs 7, 8, 9 and 10 all showed decidedly more alcohol, the increases being 88, 80, 60 and 51 per cent, respectively, based on the assumed yield of 1.37 per cent. Apparently the alcohol yield increases with an increase of catalyzer up to 5 per cent catalyzer, the yield falling off slightly with higher percentages of catalyzer. This is shown in Table III.

The yields of alcohol from Runs 7, 8 and 9 are all appreciably higher than the highest yields obtained in the larger retort.

(b) *Pressure Higher than Atmospheric*—Runs 11 to 14, inclusive, were all made under constant pressure (110 lbs.) and moisture (about 60 per cent) conditions, but with variable amounts of catalyzer.

The yield of acids apparently bears no relation to the amount of catalyzer. In this series, however, the first marked effect of hydrolysis on the yield of acid is noted. Run 12 gave 16.05 per cent total acid and 13.82 per cent acetic acid, an increase of 128 per cent total acid and 169 per cent, or 2.7 times the acetic yield from Run 6, the standard. What conditions resulted in such a remarkable yield cannot be determined until further work is done. The yield of formic acid was also higher than normal but the proportional increase was less. This may be due to the fact that the maximum formation of formic acid takes place

TABLE II—DISTILLATION OF BEECH IN PRESENCE OF PHOSPHORIC ACID(a)

Run No.	Catalyzer Per cent	Max. Temp. Retort Bath Degrees C.		Pressure Lbs.	Pyro. Acid Less Moisture		Charcoal	Gas	Tar		Total Acid	Acetic Acid	Formic Acid	Wood Alcohol	Moisture
					Dis-solved	Settled									
6.....	0	320	470	0	35.80	40.30	19.05	7.70	4.79	7.03	5.13	1.47	0.99	39.15	
7(b)....	4.79	325	470	0	35.91	40.70	16.43	1.92	Neg.	6.76	5.20	1.20	2.58	138.90	
8(b)....	4.96	321	470	0	39.38	44.85	15.77	2.11	Neg.	6.60	6.45	0.11	2.20	84.70	
9(b)....	9.73	325	450	0	36.57	40.25	16.25	2.25	Neg.	6.96	6.70	0.19	2.47	135.90	
10(b)....	2.63	333	465	60	34.73	44.55	26.62	1.25	Neg.	6.68	5.12	1.19	2.07	143.70	
11.....	1.25	326	470	110	41.72	44.90	11.85	1.09	0.29	4.56	4.33	0.20	1.44	58.25	
12.....	2.45	331	465	110	39.77	46.45	13.75	1.52	Neg.	16.06	13.85	1.72	2.00	59.30	
13.....	4.85	327	465	110	41.26	44.20	14.54	0.70	0.20	3.58	2.43	0.88	1.33	58.50	
14.....	20.78	326	465	110	37.81	45.20	16.79	0.90	Neg.	6.55	5.33	0.93	2.37	58.10	

(a) Results are all in per cent of dry charge. (b) Charged from hot retort, carbon equals 5 per cent for Runs 7, 8 and 9.

cause beech and maple gave practically the same alcohol yields in the larger retort, and this yield is more plausible than the lower figure.

(a) *Atmospheric Pressure*—Runs 7 and 8 were made under the same conditions except moisture content, and in Runs 7 and 9 the only variable was the amount of catalyzer. In all of this group the total acid yields were slightly less than the standard, but the acetic acid yields were 25 and 30 per cent higher, respectively, for Runs 8 and 9. Run 7, however, gave practically the same acetic yield as the standard. Other work has shown that the presence of an excess of water in beechwood tends to give more formic acid, which may account for the fact that Run 7 with the higher moisture content gave more formic acid. The data also indicate that as the amount of phosphoric catalyzer is increased, as in Run 9 compared with Run 7, the tendency is to form less formic acid, the moisture content being the same in both cases. Run 10 practically belongs to this group, except that it was run at 60 lbs. pressure, all other conditions being the same, and it may be noted that this run with the higher moisture content showed the same tendency to give a high formic acid yield, as in Run 7, the yields of acetic and formic acids being practically the same for both of these runs.

more readily than the formation of acetic acid, the yield of formic acid, therefore, representing more nearly the maximum possible yield. In the hydrolyzation of hardwood with sulfuric acid for the production of sugars, the proportion of formic to acetic acid is often much higher than by ordinary destructive distillation, indicating that the decomposition of the sugars or whatever is the source of the formic acid takes place more readily than the formation of acetic acid. The yields of total acid are not higher than normal in any of the other runs of this group. In fact, Runs 11 and 13 gave exceptionally low yields of acid.

TABLE III

Run No.	Catalyzer Per cent of dry wood	Yield of alcohol Per cent of dry wood
6	0	0.99 (assumed 1.37)
10	2.63	2.07
7	4.79	2.58
9	9.73	2.47

The alcohol yields for this group are all higher than the actual normal, but only Run 12, which gave the high acid yields, and Run 14, with 20.87 per cent of catalyzer, gave appreciably more than the assumed normal of 1.37 per cent alcohol. Run 12 gave 46 per cent more and Run 14 about 73 per cent more alcohol than the assumed normal.

The same tendency as noted in the case of maple to give higher yields of charcoal and pyroligneous acid,

lower yields of soluble tar and negligible yields of settled tar, using H_3PO_4 as catalyzer, was also noted on all of these runs with beech, although the effect on the charcoal and pyroligneous acid was not so marked as with maple, especially when the distillations were made from a hot retort. Larger amounts of soluble creosote oil than normal were also noted during the analyses of the pyroligneous acid.

In the distillations from a hot retort the deposition of a very finely divided carbon in the outlet pipe was noted in several runs, amounting to as much as 5 per cent of the dry weight of the charge. This material was probably the result of decompositions of tar. No microscopic or chemical examination was made of the material, but this is now being done.

THE DISTILLATION OF WOOD AND TAR MIXTURES—A series of runs were made distilling beech chips which had previously been saturated with crude beechwood creosote. The results are given in Table IV. The runs were all made from a hot retort. In Runs 15, 16, 17 and 18 the conditions were practically constant, except for pressure.

The yield of total acid varied considerably in these runs but was not higher than normal in any case.

from that added to the charge it was necessary to make the assumptions given in the table, knowing in general the effect of pressure alone on the tar formed from the wood.

In distilling the wood and creosote under pressure difficulty was experienced in maintaining the pressure to the end of the distillation and it was always necessary to relieve the pressure when about two-thirds of the total distillate had been recovered, but a large volume of the distillate always came over during the blowing-off of the pressure. Coking always took place when the pressure was relieved, not before, and although the decreasing of the pressure should have lowered the temperature, the temperature in the retort did not fall until enough time had passed after the pressure was gone for coking to take place, showing the exothermic character of the coking reaction.

The fact that methoxy groups can be split off from the tar by distillation under pressure was indicated by a change in the physical properties of the oil. Fractional distillation of the tar before and after the runs showed that the tar from which methyl groups had been apparently split off contained high boiling fractions whose specific gravity decreased with in-

TABLE IV—DISTILLATION OF BEECH IN PRESENCE OF WOOD CREOSOTE AND MIXTURES OF CREOSOTE AND ACIDS

Run No.	Per cent Catalyzer		Maximum Temperature Retort Bath Degrees C.		Pressure Lbs.	Pyro. Acid Less Moisture	Wood Charcoal Estimated	Wood Gas	Per cent Tar Recovered as Oil ^(b)	Per Cent Tar as Coke ^(c)	Total Acid	WOOD ALCOHOL		Moisture	
	Tar	Acid	Retort	Bath								From Equal Parts Wood and Tar Based on Wood	Actual Per cent Dry Wood		Estimated from Tar Only
15...	103.0	None	327	475	0	25.80	44	24.20	72.70	7.07	6.72	1.37	1.37	0	7.60
16...	106.5	None	313	440	30	31.30	44	20.70	58.10	37.95	5.35	2.83	2.87	1.46	7.80
17...	120.2	None	321	440	60	32.10	44	21.90	44.10	39.70	5.36	3.12	3.40	1.75	7.80
18...	112.2	None	334	470	90	31.09	44	24.36	39.65	51.00	6.50	3.47	3.66	2.10	11.26
19...	99.2	H_3PO_4 2.55	321	470	60	32.23	44	21.77	62.95	49.20	6.87	1.03	1.03	10.12

(a) Results are all in per cent of dry charge. (b) Estimated from assumption that tar formed by distillation of wood = 15 per cent at 0 lb.; 8 per cent at 30 lbs.; 5 per cent at 60 lbs.; 3.5 per cent at 90 lbs. for Runs 15, 16, 17 and 18, respectively, and 1.25 per cent for Run 19. (c) Estimated from assumption that wood charcoal = 44 per cent.

The yield of alcohol, however, increased with pressure, the increase following a definite curve. The yield at 90 lbs. pressure for equal parts of wood and creosote figured in percentage of the dry weight of the wood was 153 per cent or $2\frac{1}{2}$ times as much as the yield obtained at atmospheric pressure with equal parts of wood and tar. Taking the yield from wood alone under these conditions as 1.37 per cent alcohol, as obtained in Run 15, the yield from tar alone was 2.1 per cent. Analyses of wood tar¹ have given 11.08 per cent methoxy group for crude creosote (195–255° C.). The results indicate then that the distillation at 90 lbs. pressure will split off about 20 per cent of these groups as methyl alcohol. Higher pressures would probably give better yields, but the use of higher pressures is restricted because of the loss of tar as coke. Since 70 per cent of the increase in alcohol is obtained at 30 lbs. pressure the lower pressures are more desirable because of the higher recovery of oil. The original tar contained $77\frac{1}{2}$ per cent oil and $22\frac{1}{2}$ per cent pitch, so the recovery of oil was 75 per cent for the run at 30 lbs. pressure. As it was not possible after distillation to differentiate between the tar formed by the distillation of the wood and the tar recovered

crease in boiling point, a most unusual property for wood tars. These fractions are being examined for acid phenols, which are likely to be present after splitting off the methyl groups from the phenol ethers.

Acetone determinations made in these studies showed that in no case was more than 0.5 to 1 per cent of the alcohol acetone and in Run 20, which gave the highest yield of alcohol, only 0.12 per cent of the alcohol was acetone. It should also be mentioned that in all of the tests with acid catalyzers the acetone yield was seldom over 1 per cent of the alcohol and generally less than 0.5 per cent of the alcohol.

WOOD TAR AND PHOSPHORIC ACID—The effect of the acid catalyzer on the tar formed in the normal distillation of wood has been noted above, the settled tar, which is the source of wood creosote, being practically destroyed. The effect of a combination of creosote and an acid catalyzer at once suggested itself as an additional means of splitting off methyl alcohol, and Run 19, using tar and phosphoric, was made to study that effect. The chips were first treated with acid and then allowed to dry out to about 10 per cent moisture and were then treated with creosote. The

¹ Pieper, Humphrey and Acree, THIS JOURNAL, 9 (1917), 566.

results anticipated were not obtained as the alcohol yields were no greater than normal. It would seem then that the action of phosphoric acid on the tar is probably much more severe than splitting off of methoxy groups, and is more likely the immediate formation of hydrocarbon gases and coke. The work along this line is being extended, making tar-phosphoric acid distillations at lower temperatures to see if the destructive effect can be lessened.

Several distillations were made using sulfuric acid as a catalyzer along the lines suggested by German Patent 185,934, but instead of getting increased yields of acid and alcohol it was found that, especially under pressure, the acids were decreased and no alcohol at all was formed. Indications were obtained that the SO₂ formed by the reduction of the sulfuric acid acted very detrimentally in the destructive distillation reaction.

CONCLUSIONS

The general conclusions to be drawn from these preliminary tests are:

I—Under the proper conditions a very high yield of acetic acid may be obtained by the destructive distillation of wood, by using phosphoric acid as a catalyzer. Two and seven-tenths times as much acid as normal was obtained in one run.

II—The distillation of wood in the presence of phosphoric acid showed a pronounced tendency to give more wood alcohol. Increases varying from 40 to 90 per cent were obtained.

III—The distillation of mixtures of wood and tar under pressure showed that the methoxy groups in the tar can be readily split off, forming wood alcohol. Nearly 20 per cent of a possible theoretical was obtained at 90 pounds pressure. The work is being extended to include a study of many other catalyzers and variables.

The recovery of the metaphosphoric acid residual in the charcoal, which is readily soluble in cold or hot water, could in all probability be made practically quantitative by simple leaching. Just what recovery can be made is being studied.

The experiments described are only preliminary laboratory investigations, and no attempts will be made to commercialize the ideas developed until further work is done. With the quantitative recovery of phosphoric acid its use as a catalyzer would be entirely practicable and the application of these ideas would seem to open up the possibility for the destructive distillation of small forms of wood waste. The commercial practicability of the distillation of wood and tar mixtures would be in the same direction and depend on the recovery of more valuable constituents of the oil as well as a high yield of wood alcohol. Patent application has been made by the author for the protection of these ideas, the patents being dedicated to the public.

FOREST PRODUCTS LABORATORY
MADISON, WISCONSIN

SOME EXPERIMENTS ON THE PULPING OF EXTRACTED YELLOW PINE CHIPS BY THE SULFATE PROCESS

By OTTO KRESS AND CLINTON K. TEXTOR

Received September 4, 1917

INTRODUCTION

Some time ago the Forest Products Laboratory was asked to determine whether longleaf pine chips, after the extraction of rosin and turpentine, would be suitable for the manufacture of kraft paper. This suggested itself inasmuch as commercial kraft is being made from longleaf pine and the removal of rosin should be an advantage provided there were no other factors entering. Extracted chips are only used as fuel under the boilers at the extraction plant and any excess over fuel requirements is a complete waste. If they can be converted into a by-product, the waste will be a source of income whereas its disposal is now an expense.

DESCRIPTION OF TEST MATERIAL

In preparation for extraction, the raw wood, hogged to the proper size, is placed in iron extractors, which, in this particular case, are not designed for pressure extraction, and steamed about 3 hrs. for recovery of crude turpentine. After this preliminary steaming treatment, the extractor is filled with hot gasoline, 58 to 60° Bé. and extracted for approximately 5 hrs. Two solvent drops are taken off and the chips are then washed with fresh solvent, this wash being used on the next extractor. The solvent solution containing the rosin and pine oil is evaporated first for recovery of solvent, then for pine oil, while the residue of rosin is run into barrels for shipment.

After the solution of gasoline and pine oil is all removed from the extractor and after draining for one hour to remove as much as possible of the above mixture, the extractor is steamed for 5 hrs. to recover solvent. The first part of the steaming occurs with wet steam, while the last half is with superheated steam.

The average yield per ton of wood extracted on a car weight basis is:

Rosin.....	250 pounds
Crude turpentine.....	8 gallons
Crude pine oil.....	2 gallons

The total amount of chips handled at the plant with which the Laboratory coöperated on these experiments is 400 tons per day, of which 300 tons are burned under the boilers, leaving 100 tons available per day.

The Laboratory received a shipment of 1,000 lbs. of "spent wood" chips, as they come from the extractor, and 500 lbs. of "fresh chips" which had not been extracted. We were informed that the original chips contained 12 to 13 per cent moisture, while the extracted chips contained 18 to 19 per cent moisture. These chips had been treated slightly differently from the ordinary run of chips, as in the usual practice saturated steam is admitted to the extractor for ten minutes preliminary to opening the digester. This treatment is given to avoid danger of fire.

PROCEDURE

PREPARATION OF THE WOOD—The chips from a superficial examination showed a large percentage of dust and