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# THE MANUFACTURE OF ETHYL ALCOHOL FROM WOOD WASTE

#### By

# F. W. KRESSMANN **Formerly Chemist in Forest Products**

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### PREFATORY NOTE.

This report is based primarily on experiments conducted by the author, Mr. F. W. Kressmann, at the Forest Products Laboratory, prior to December, 1916. The information thus obtained has been supplemented by the results obtained in succeeding years by Mr. Kressmann while in private employment as manager successively of the Standard Lessee Corporation and the International Alcohol Corporation and as director of the development department of the latter corporation. Acknowledgments are due these companies for their consent to the utilization of information obtained by Mr. Kressmann in connection with his work for them and to Mr. Kressmann himself for his assistance in the completion of the investigation and report.

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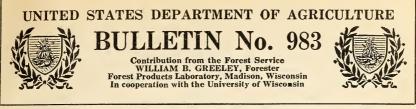
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# THE MANUFACTURE OF ETHYL ALCOHOL FROM WOOD WASTE.<sup>1</sup>

By F. W. KRESSMANN, formerly chemist in Forest Preducts.<sup>1</sup>

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#### SOURCES OF ETHYL ALCOHOL AND COMPARATIVE COSTS OF **PRODUCTION.**

The production of fermentable sugars and ethyl alcohol from cellulosic materials, such as straw, linen, cotton, peat, wood, and in fact, all plant fibers, has engaged the attention of chemists and technologists for nearly a century. It is only within the last two decades, however, that serious attempts have been made to utilize wood waste for this purpose. The principal sources of fermentable sugars from which alcohol is at present derived are the hydrolytic products of starch and the sugars obtained from fruits and such sugar-factory residues as molasses.

Corn yields about 2.4 gallons of 188-proof spirit a bushel; and, although the price of corn and other grains used varies with the

<sup>&</sup>lt;sup>1</sup> The author acknowledges with pleasure his indebtedness to Messrs. Homer Cloukey and H. N. Calderwood, of the Forest Products Laboratory, whose aid in making the hundreds of analyses necessary to the research was invaluable. Acknowledgment is made also to Drs. S. F. Acree and E. C. Sherrard for helpful criticism and review. For help rendered by men from outside the Forest Service, the author acknowledges his obligations to Messrs. Boyt and Groves, of Georgetown, S. C., to Dr. R. C. Gravenburg, distillery superintendent, at Fullerton, La., and to Dr. T. B. Wagner, of New York. Many others have assisted in the production of this work, and to all of them the author is grateful.

<sup>54976°-21-</sup>Bull, 983-1

season and from year to year, before the war the average cost of the materials for making grain alcohol, fuel excluded, was about  $27\frac{1}{2}$  cents a 188-proof gallon. Manufacturing costs, including coal, interest, repairs, depreciation, taxes, labor, etc., range from 10 to 17 cents a gallon of 188-proof alcohol, depending upon the location and efficiency of the plant.

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One gallon of molasses yields from 0.45 to 0.48 of a gallon of 188proof spirit. The price of molasses before the war averaged from 5 to 7.5 cents a gallon, and, therefore, the approximate cost of raw material in a gallon of molasses spirit was from 10 to 15 cents. The cost of production of molasses spirit is slightly less than that of grain spirit, but in either case the cost of raw material is comparatively high.

One ton of dry sawdust or other wood waste (or its equivalent on an air-dry or green basis) will yield from 12 to 20 gallons of 188-proof spirit. The disposal of this waste in the vicinity of a sawmill or other large woodworking plant is specifically an item of loss, because most sawmills produce waste in excess of their own power requirements. Sometimes the waste is not worth more than 30 to 50 cents a ton, and this makes the cost of raw material in a gallon of ethyl alcohol from sawdust about 2 cents. This includes also the fuel charge, for the residue after conversion and extraction is available for fuel, whereas in grain distilleries about 7 tons of coal and in molasses distilleries about 4 tons are required in producing 1,000 gallons of 188-proof spirit.

If the manufacturing cost of producing ethyl alcohol from wood can be reduced to the same figure or nearly the same as that for making it from grain or molasses, there will be a large margin in favor of producing the alcohol from wood waste. Of course, with a yield of 12 to 20 gallons from a ton of wood and 80 gallons from a ton of corn, the amount of material handled in certain parts of the plant producing alcohol from wood will be four or five times as great as in a grain distillery of equal producing capacity, and this will require a larger-sized plant and an increased operating cost.

In recent years the production of ethyl alcohol from sawdust has received a great deal of attention, and a large amount of money has been spent in the technical development of the process. A number of plants have been built in this country, but only two have been considered commercial successes.

Because of the importance to the lumber industry of the problem of waste disposal, and because this process is practically the only one applicable to the disposal of wood waste, the Forest Service has investigated the different processes and, so far as possible, the plants that have been built, in order to learn the causes of former failures and to aid in the commercial development and success of the processes.

#### AMOUNT OF WOOD WASTE AVAILABLE.

The amount of wood waste produced by converting a tree or a sawlog into lumber is, of course, variable and depends upon the diameter of the tree, the quality of the timber, and the efficiency and equipment of the sawmill. The following tabulation shows that more than half of the cubic contents of the tree is wasted:

P	er cent.	Per	cent.
Entire tree	. 100	Slabs	8
		Edgings	8
Stump	. 2	Shavings.	4
Тор	. 18		
Sawdust.	12		62
Bark	. 10		

This includes limbs, top, and stump-the parts of the tree left in the woods-in addition to the waste at the mill or factory. The mill waste, particularly the part available without extra transportation charges, is of great interest in connection with the manufacture of ethyl alcohol.

The annual cut of lumber in the United States for the five or six years preceding the World War was approximately 40,000,000,000 feet board measure.<sup>2</sup> The mill waste from this cut has been estimated by Margolin <sup>3</sup> as follows:

P Sawlog	er cent.
sawlog	100
Bark	13
Sawdust	13.5
Edgings and trimmings	8.7
Slabs	8.7
Careless manufacturing and accidents.	3.5
Loss in cutting to standard widths and lengths	1.7
-	
Total waste	49.1
Lumber	50.9

For each thousand board feet of lumber produced from sawlogs (which is equivalent to 83.3 cubic feet of solid wood) there is, according to the above table, 80 cubic feet of waste, distributed as follows:

Bark	. 21.3
Sawdust	
Edgings and trimmings	. 14.2
Slabs	
Careless manufacturing and accidents	
Loss in cutting to standards	. 2.7
Total	80

<sup>2</sup> The Lumber Industry, Part IV, Bureau of Corporations, U. S. Dept. of Commerce; The Production of Lumber in 1913, Bulletin No. 232, U.S. Dept. of Agriculture.

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After the amount of the bark is deducted there remains in the form of waste 58 cubic feet of solid wood to 1,000 board feet of lumber cut, or 2,320,000,000 cubic feet annually. As the average weight of a cubic foot of air-dry wood is about 35 pounds,<sup>4</sup> this is a total annual waste of 40,000,000 tons. Some of this total annual waste is used for fuel at the mills, or may have a fair market value if the mills are located in centers of population; but it is estimated that over onehalf, or 20,000,000 tons, is absolute waste, and that about 15,000,000 tons of this is from coniferous woods.

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According to data for 1907 submitted to the Forest Service by the lumber manufacturers, 650 mills were reported to be cutting between 10,000,000 and 25,000,000 feet a year; 161 mills between 25,000,000 and 50,000,000 feet; 39 mills between 50,000,000 and 100,000,000 feet; and 2 mills over 100,000,000 feet annually. This is a total of 852 mills, each of which cuts 10,000,000 or more board feet a year. For 1913, each of 974 mills was reported as cutting this amount. This shows that the number of large operators is increasing instead of decreasing, and that the supply of raw materials is so concentrated as to be available for any use to which it can be put.

# THE PRESENT VALUE OF WOOD WASTE.

Most of the wood waste produced to-day is valuable only for fuel for the production of power at the mill. In some places methods of closer utilization have been worked out; but, compared with the total amount of wood waste produced, the quantity of material so utilized is negligible unless the mills are located in or very near large cities.

Most mills produce waste in excess of their own power requirements, and in large mills equipped with especially efficient power plants this excess is from 50 to 65 per cent of the total amount produced. A waste burner, therefore, is almost invariably necessary, and its use involves not only a loss of large quantities of wood, but also a fixed charge for its operation. The cost of burning waste varies widely with the size and efficiency of the mill, but figures gathered by the Forest Products Laboratory indicate that it ranges from 30 to 66 cents a cord. Assuming that 37 cubic feet are burned for each thousand feet board measure of lumber cut, this is a charge of from 11 to 22 cents a thousand feet on all of the lumber cut, and means that the present cost of waste disposal amounts to about \$6,000,000 annually, in addition to the value of the wood.

The wood waste available has great potential value, but its utilization has not as yet reached the stage where the waste has much more than a nominal value.

<sup>&</sup>lt;sup>4</sup> The green weight, log scale, of yellow pine will range from 9,000 to 10,000 pounds to the thousand feet, and the green weight of the lumber produced will range from 4,200 to 4,600 pounds. Allowing for the usual 15 to 20 per cent overrun, we ordinarily get about 4,500 pounds of waste to 1,000 feet of yellow pine cut. The waste from other species will vary in the proportion of their respective weights to the cubic feet or to the thousand feet, log scale.

#### ETHYL ALCOHOL FROM WOOD WASTE.

As will be shown, the production of ethyl alcohol is so far the only process that is applicable to the utilization of average mill-run refuse from the coniferous woods and from a few of the hardwoods. It affords a means of converting waste material into marketable commodities at a profit, provided sufficient quantities of waste material are available in the proper locality and at the proper price. The process is not applicable to the commercial conversion into alcohol of lumber, merchantable timber, or other expensive forms of wood. It, therefore, rests largely with the lumbermen themselves whether they will accept a price for their waste which will make it commercially available, or whether they will demand a price that will make its utilization prohibitive. Cooperation between the sawmill and the distiller is absolutely necessary, for the distiller is dependent upon the mill for his raw material, and the lumberman is dependent upon the distiller for the successful disposal of his waste. During the war, the return of 50 cents a cord for waste was not attactive to many of the lumbermen, with the exception of some in Mississippi, where, however, State laws prohibit the manufacture of ethyl alcohol.

Some sawmill owners have not been willing to tie up their waste on a 10-year contract at the price mentioned, but have preferred to continue for a time to burn it in the speculative hope that some better method of disposal might be found. Such action is, of course, perfectly legitimate and may possibly be the proper one. The alcohol plant costs about as much as the sawmill and its auxiliaries; so that a considerable supply of raw material—enough for at least 10 years is necessary to justify the initial investment. As the life of the saw mill is continually decreasing, each year that passes reduces materially the prospect of utilizing the sawmill waste.

# LIMITATIONS TO THE UTILIZATION OF WOOD WASTE.

The utilization of wood waste, particularly sawmill waste, is limited in a number of ways. The bulkiness of the waste material makes a minimum amount of handling imperative and practically prohibits its transportation, except for short distances and by means of mechanical conveyors, such as belts and fans. The form of the waste is one of the greatest difficulties in the way of its more complete utilization. In sawdust and shavings not only has the length of the wood fiber been reduced, but the fibers have been lacerated to such an extent as to destroy their value for pulp and paper production.

The destructive distillation of sawdust and shavings has not so far been found practicable. There have been two reasons for this: First, the small size of the material makes it so poor a conductor of heat that it can not be charred completely in the forms of retort and kiln in ordinary use; and, second, the charcoal produced is so finely divided that it is difficult to cool and handle and there is no ready

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market for it. In addition, the waste as it comes from the mill is usually a mixture of all forms, and any attempt at separation, except perhaps a simple blowing or screening to remove the very fine stuff, will increase the cost of the raw material to a prohibitive figure. Therefore, in any satisfactory process for the utilization of mill waste, it must be possible to handle any and all forms of waste as it comes from the mill.

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Except in factories using only one or two species of wood, or in mills cutting only a few similar species, such as the "yellow pine" (longleaf, shortleaf, and loblolly) of the South, the differences in quality and form of the waste have operated against its efficient utilization. This is because many processes, such as pulp and paper making or destructive distillation, require a particular species in order to give a yield and quality of product that will make the processes commercially feasible.

Woods of all species and forms, however, have one point in common—they all contain more or less cellulose, which makes up the fibers of the wood, along with an incrusting substance called lignin. A chemical utilization of this cellulose would overcome the objections stated above as to the form of the material, length of the fiber, and species, provided the amount of cellulose present was sufficient to give a yield of alcohol that could be handled profitably on a commercial scale.

#### PROCESSES FOR THE MANUFACTURE OF ALCOHOL FROM WOOD.

The processes used for the production of ethyl alcohol from wood may be grouped into two general classes: Hydrolysis of wood into fermentable sugars by the use of dilute acid (preferably mineral acid) as a catalyzer, and solution processes, in which the wood is dissolved in concentrated acid and the diluted solution is then subjected to hydrolysis.

The first process consists, in general, of digesting sawdust or hogged and shredded wood with a dilute mineral acid under 60 pounds or more of steam pressure. This converts part of the wood into a mixture of pentose and hexose sugars. The latter are then fermented into ethyl alcohol.

Processes of the second class, involving the use of concentrated sulphuric acid and in which the wood is actually dissolved by the acid, as in the Ekstrom <sup>5</sup> process, have not received commercial attention, notwithstanding the fact that Flechsig<sup>6</sup> many years ago showed that cotton cellulose could thereby be converted into dextrose and alcohol almost quantitatively. The more recent work of

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<sup>&</sup>lt;sup>5</sup> French Patent No. 380358; German Patents Nos. 193112 and 207354.

<sup>&</sup>lt;sup>6</sup> Zeit. für Physiol. chemie., 1882.

Willstatter and Feichmeister <sup>7</sup> with fuming hydrochloric acid on cotton and wood has confirmed these results; but in all those experiments the amounts of acid required have been so large that the initial and recovery costs for acid have prevented commercial development.

Whether the source of the fermentable sugars is the cellulose or the lignin of the wood has long been a subject for debate and has also been the occasion of considerable investigation; but the fact remains that a wood cellulose like soda or sulphite pulp has been found to produce about twice as much fermentable sugar and alcohol as the same amount of the original wood, the yields being in proportion to the cellulose content.<sup>*a*</sup>

#### HISTORY OF THE PROCESSES.

The first recorded attempts to produce sugars and alcohol from vegetable fiber were those of Braconnot<sup>8</sup> in 1819. From that time until the publication of Simonsen's <sup>9</sup> paper in 1898 little work of value was done.<sup>10</sup> Simonsen's review of the problem is well worth quoting here, because it tersely describes the situation at that time:

The literature of this problem is imperfect and faulty to a high degree. It contains many inaccurate and impossible statements and contradictions. There is no record of any systematic investigation as to the effect of a variation of the different factors, such as amount of water, pressure, amount of acid, and time in high-pressure inversions. Parallel and comparative experiments on cellulose and wood are also lacking, so no information on the relation of the incrusting substances to the inversion processes is at hand. That these investigations may have been made and their results kept secret is not impossible, since factories have been established. Such researches could hardly have dealt with high-pressure inversion, which has only been carried out practically on a large scale for the last 20 years. Yet the manufacture of spirit from cellulose material by means of inversion under such unfavorable conditions as that over 100 per cent of sulphuric acid was required for the dry wood and the corresponding quantity of calcium carbonate or lime (and taking into account the high price of the material at that time and the length of time required for the process) seems to point to the fact that the inversion of wood will be the method of the future if only a satisfactory process can be found.

Simonsen carried out a long and painstaking research on the subject, in which he investigated both cellulose (sulphite cellulose) and sawdust in a systematic way. As an inverting agent he used sulphuric acid, and from his results concluded that the best conditions for the inversion of sawdust were as follows:

Time of inversion	15 minutes.
Acidity	0.5 per cent H <sub>2</sub> SO <sub>4</sub> .
Proportion of wood to liquid	
Pressure about	
Pressure about	9 atmospheres.

<sup>7</sup> Berichte, 1913, 2401.

a Koerner, Zeit. Ang. Chem., 1908, 2353.

<sup>&</sup>lt;sup>8</sup> Gilbert's Annalen der Physik, 1819, 63, 348.

<sup>&</sup>lt;sup>9</sup> Zeit. für ang. Chemie, 1898, 195, 962, 1007.

<sup>&</sup>lt;sup>10</sup> The references to the original literature from 1819 to 1898 will be found in the bibliography at the end of this bulletin.

These conditions gave him a yield of alcohol equal to about 6 per cent of the dry weight of sawdust used, although few fermentation experiments were made.

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As was shown later by Neumann,<sup>11</sup> Simonsen's work is contradictory in some respects, because of the fact that only a single experiment was made under each set of conditions. In his work on a large scale,<sup>12</sup> he was generally unable to duplicate the results obtained in the small autoclave cooks. The yields of alcohol varied considerably, although under the most favorable conditions and in a few exceptional cases he obtained yields which were slightly higher than those secured on the small scale. Simonsen's process was patented July 12, 1898.<sup>13</sup> Korner<sup>14</sup> later substantiated Simonsen's yield of 6 per cent and showed further that the yield of alcohol and sugar was in proportion to the cellulose content of the sawdust, straw, and sulphite cellulose used as raw material.

A. C. Classen developed a new process in which sulphur dioxide was the inverting agent, although his first patent<sup>15</sup> covered a mixture of sawdust and concentrated sulphuric acid (50° to 60° B.) in which the mixture was subjected to great pressure in a hydraulic press. In the original Classen process<sup>16</sup> an aqueous solution of sulphurous acid was used, though later Classen obtained three patents<sup>17</sup> in which chlorine, air, or oxygen were used as the oxidizing agents to convert the sulphurous acid to sulphuric acid. Still later he obtained a patent<sup>18</sup> covering the process of treating the wood with sulphuric anhydride, and an additional patent<sup>19</sup> covering the process of heating this mixture to 123° to 135° C.

He also patented <sup>20</sup> the use of a smaller amount of a more concentrated solution of sulphurous acid, claiming that the acid recovery was more efficient when the sawdust in the digester was only slightly moist. In 1914 he patented <sup>21</sup> the use of platinum, ferric oxide, etc., as catalytic agents to convert the sulphurous acid into sulphuric acid in the digester.

The French rights to the Classen process were sold to a M. Taffin, who had experimented for several months at Tolques before the purchase. After satisfying himself that the process was practical, he organized in August, 1904, the Compagnie Industrielle des Alcools

19 German Patent No. 123911.

<sup>&</sup>lt;sup>11</sup> Neumann, Dissertation, Dresden, 1910.

<sup>&</sup>lt;sup>12</sup> Zeit. für ang. Chemie, 1898, 962.

<sup>&</sup>lt;sup>13</sup> United States Patent No. 607091. It was also patented in Norway, Austria, England, France, Canada, Hungary, and Germany.

<sup>14</sup> Zeit. für ang. Chemie, 1908, 2353.

<sup>&</sup>lt;sup>15</sup> German Patent No. 111868.

<sup>&</sup>lt;sup>16</sup> German Patent No. 118540.

<sup>&</sup>lt;sup>17</sup> German patents Nos. 118542, 118543, and 118544.

<sup>&</sup>lt;sup>18</sup> German Patent No. 121869.

<sup>20</sup> German Patent No. 130980.

<sup>&</sup>lt;sup>21</sup> United States Patent No. 1101061.

de l'Ardèche, and built an experimental plant at d'Aubervilliers, in which he planned to determine the most suitable forms of apparatus. After some time the company interested M. André Bernhard, of Lille, one of the largest distillers in France. The capital stock of the company was materially increased, M. Bernhard became manager and director, and the company decided to increase the capacity of the old plant and erect a still larger one in the Vosges. The chemists of the company were meanwhile perfecting a process whereby the acetic acid formed during the cooking of the wood with the sulphurous acid could be recovered along with the major part of the sulphurous acid. In addition, a special type of digester known as a "saccaraficateur"<sup>22</sup> was developed. This consisted of a steel cylinder 2<sup>1</sup>/<sub>2</sub> meters in internal diameter by 21 meters long, through which were spaced 22 tubes 160 mm. in diameter. Outside of each end of the tube heads were flanged boiler-steel jackets, one to receive the live steam from the boiler and the other to take off the condensed steam, the heating being indirect. This type of apparatus will be discussed further in connection with the plant built at Port Hadlock, Wash. Instead of the sugars being extracted in diffusion batteries, water and calcium carbonate were added to the digested sawdust, the whole mass was fermented directly, and afterwards was distilled in the usual type of beer still. Higher yields were claimed for this method than for the extraction method. This plant operated intermittently for a time, a number of runs being made in 1908 on American woods, primarily for the purpose of interesting American capital; but apparently no continued commercial operation resulted on French material.

In 1903, Classen sold the patent rights for America to the Classen Lignum Co. of Chicago, a corporation organized under the laws of the State of New Jersey. This company erected an experimental plant at Highland Park, Chicago, which had a capacity of about 2 tons of dry sawdust a day of 24 hours. Later the company erected a plant at Hattiesburg, Miss., at a cost of about \$250,000, to operate on sawmill waste of longleaf pine. A number of mechanical and technical reasons for the failure of this plant have been outlined by Ruttan.<sup>23</sup> The disadvantages of this process were as follows: (1) The great length of time (from 4 to 6 hours) necessary to convert from  $1\frac{1}{2}$  to 2 tons of wood; (2) the large quantity of acid required; (3) the prolonged action of so much acid and water in the rotating digester reduced the wood to a very fine powder and formed much sulphuric acid, which, acting on the sugars and other substances present, produced gums and caramels and made the complete extraction of the sugars from the residue very tedious and expensive; (4) the digester was lead lined, and the repair of the buckling and breaking of the

<sup>&</sup>lt;sup>22</sup> This apparatus is described in detail in French Patent No. 358696.

<sup>23</sup> Jour. of the Soc. of Chem. Ind. 1909, 1290.

lining after every two or three operations proved a source of great delay and expense.

Ewen and Tomlinson, who were associated with the Classen process, began experimenting along new lines to overcome the difficulties that prevented the old process from becoming a commercial success. The results of their researches (United States Patent No. 763472) were: (1) The time of hydrolysis was shortened from 6 hours to 45 minutes; (2) the treated wood waste was obtained in a form which could be quickly and efficiently extracted; (3) a digester was devised which was not affected by the process; (4) the quantity of acid employed was reduced; (5) a large and uniform yield of fermentable sugars was obtained from the wood.

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In general, these results were accomplished in the following way: Instead of adding an aqueous solution of sulphur dioxide to the sawdust and afterwards heating this large volume of water, steam was used as a source of both heat and moisture, and the sulphur dioxide was introduced into the digester in a gaseous form. This method shortened the heating period and also decreased the amount of wood that was reduced to a powdered condition, thereby permitting a more complete extraction.

On October 26, 1909, Ewen and Tomlinson were granted a patent protecting the process of producing fermentable sugars from lignocellulose (United States Patent No. 938308). This patent shows that they had given up the use of sulphur dioxide, both gaseous and in solution, and were employing sulphuric acid as the inverting or catalytic agent. A study of the patent reveals the fact that the ratio of water and acid to dry wood which they used was practically the same as in the method patented by Simonsen and referred to above. Ewen and Tomlinson, who were then the engineers and technical advisers of the Standard Alcohol Co., erected a plant at Georgetown, S. C.,<sup>24</sup> for the production of ethyl alcohol from sawmill waste. This plant was later acquired by the E. I. du Pont de Nemours Powder Co., which operated it intermittently until the early part of 1913. A fire then destroyed the main sawmill of the Atlantic Coast Lumber Corporation, and the alcohol plant was not operated until the summer of 1914, when the sawmill had been rebuilt. The alcohol plant has been operated successfully since that time under the Ewen and Tomlinson patents.

Several years ago the Classen Chemical Co. interested western capital in the erection of a plant at Port Hadlock, Wash., on Puget Sound, for the production of ethyl alcohol and cattle food from sawdust obtained from mills at Seattle, Tacoma, Everett, Anacortes, and Port Blakely. The plant was equipped with six digesters of the

<sup>&</sup>lt;sup>24</sup> For a description, see R. von Demuth, Zeit. für ang. Chemie, 26, 786; also G. Foth, Chemiker Zeitung, 37, 1221, 1297.

same size and shape as those used in the French plant, the idea being to save steam by means of indirect heating. Sawdust and enough water were added through a manhole into the space between the tubes to raise the moisture content to about 45 per cent. Anhydrous sulphur dioxide was then added, and the mixture was cooked at 75 to 100 pounds pressure. The cost of conversion was excessively high, because the corrosion of the digesters was very rapid; the time necessary to heat them by indirect heat was very long; and it was necessary to replace the low-pressure steam with high-pressure steam in the outside jacket in order to prevent the sulphurous-acid gas from leaking out of the digester into the jacket. The extraction equipment was very inefficient, as the modern type of diffusion battery was not used. The plant was very well built, and much of the equipment was imported from France at a high cost. The extracted sawdust was mixed with Hawaiian molasses and was put on the market as a cattle food.<sup>25</sup> It was necessary to dry the extracted material to about 12 per cent moisture, in order to prevent decay, and this gave great difficulty because of explosions of dust in the driers. In addition, the plant was located 80 miles from a railroad, and this distance greatly increased all transportation charges both to and from the plant. This and the very poor design of the digester and extraction equipment were, no doubt, the chief reasons for the failure of the plant.

In the process covered by United States Patents Nos. 985725 and 985728, granted to W. P. Cohoe, of Toronto, Canada, hydrochloric acid is used and preferred as a catalytic agent because of its volatility. A yield of 25 to 28 per cent of fermentable sugars is claimed. It is also stated that the acid can be completely removed from the wood by blowing it out with steam. If this is true, the cost of neutralization is removed. It is also claimed that 1 to 2 per cent of acetic acid can be obtained from the preliminary steaming of hardwood sawdust. In addition, the preliminary steaming is claimed to be of value, because, after the blowing off, the sawdust is of a constant moisture content, irrespective of its initial moisture content. In a later paper Cohoe <sup>26</sup> further describes his work in which hydrochloric acid is used. The following quotations are of particular inter-

<sup>&</sup>lt;sup>25</sup> The production of cattle food from sawdust has been attempted at another plant in this country, situated at Marinette, Wis: This plant, however, was unsuccessful in marketing the product obtained, since the chief value of hydrolyzed sawdust as a cattle food lies in the carbohydrates that it contains. Carbohydrate foods, as a rule, are the cheapest that the farmer can grow for himself, and usually the only foods purchased are nitrogenous concentrates. The Marinette company was finally forced to add oil cake and similar materials to its product. In addition, the Port Hadlock plant had a great deal of difficulty from spoilage due to the absorption of water and the consequent growth of mold in their product. The material was put on the market under the trade name of "Bastol." A similar material has recently been produced in London by Zimmermann (see article in Jour. Soc. of Arts, 1912, p. 68).

<sup>&</sup>lt;sup>26</sup> Jour. of the Soc. of Chem. Ind., 1912, 513.

est in the light of more recent work of the Forest Products Laboratory.

1. In view of the fact that the reducing sugars produced by this reaction do not all ferment and also by the fact that the total amount of conversion was not by any means represented by reducing sugar, it occurred to the author that the presence of the hydrolyzing agent during the heating stage might exert a harmful effect upon the final result. To overcome this the digester was filled and heated to the reacting temperatures and then the reacting agent was introduced. This method is the one finally adopted.

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2. Given proper preparation of the materials by the preheating and a proper adjustment of phases in the digester, all the time necessary for a successful reaction is that required by a proper mixing of materials. In other words, with proper preparation the reaction itself is practically instantaneous.

3. Throughout all runs the observation made of results on the laboratory scale with regard to the fact that this reaction runs to an equilibrium was confirmed. It has been found by repeated experiment that by a proper adjustment of the phases the concentration at which the equilibrium occurs may be varied.

The yields given in Cohoe's paper, however, do not bear out the claims in his patents, for the paper reports a maximum of 20 per cent of sugars, and the patents claim 25 to 29 per cent of sugars.

After the Georgetown plant was disposed of to the Du Pont Co., the Standard Alcohol Co. underwent a reorganization, some foreign capital was introduced, and a plant designed to produce 5,000 gallons of 188-proof alcohol a day was erected at Fullerton, La. This plant was never operated successfully by the Standard Alcohol Co. because of certain internal financial difficulties caused by the war. Since the introduction of additional foreign capital was out of the question, new American interests acquired a lease of the plant to demonstrate to their own satisfaction the commercial feasibility of the process. These interests, under the name of the Standard Lessee Corporation, operated the plant from July, 1916, until June, 1917. They then purchased the plant and patents under the name of the International Alcohol Corporation. The plant has been operated successfully since the latter part of 1916 up to the present time (December, 1918).

The most recent series of patents by Tomlinson, assigned to the Standard Alcohol Co. (United States Patents Nos. 1032440 to 1032450, inclusive) cover the forms of digester, the various processes for feeding the material to the digester, the methods of mixing the acid and wood, and the processes of digestion. The chief points of these patents may be summed up as follows: Patents Nos. 1032441 and 1032442 cover the thorough mixture of the sawdust and dilute acid as they are being fed to the digesters. Patent No. 1032440 is a process patent relating to the method by which the acid liquid is introduced into the digester with the steam after the temperature has been brought to 212° F. but before it has reached 235° F. Patent No. 1032443 covers the recovery of turpentine as well as sugar, and Patent No. 1032444 the apparatus used in the process. Patent No. 1032445 covers the apparatus for mixing the sawdust and liquid acid (acid in a liquid form) as it is being fed to the digester. Patent No. 1032446 covers other apparatus for this purpose that employs acid in a gaseous form, and Patent No. 1032447 covers the process for the same. Patent No. 1032448 is a continuation of No. 938308. Patents Nos. 1032449 and 1032450 cover the apparatus and process whereby the acid liquor is introduced after the charge has first been steamed, and whereby, it is claimed, a more thorough mixing and greater yields are obtained. This idea of introducing the acid after the steaming is one of the features of Cohoe's earlier patents.

Recently another series of United States patents was taken out by Gallagher and Mork and assigned to the Standard Alcohol Co. Patent No. 1037185 covers the relief of pressure during cooking and claims thereby to eliminate products that inhibit fermentation. Patent No. 1056161 covers the process of cooking at high pressureat 135 pounds for 15 minutes, then at 70 pounds for 30 minuteswhereby, it is stated, the wood dextrins are converted into dextrose. The claim is made that wood dextrin is converted into dextrose faster than the wood dextrin is produced from lignocellulose under the ordinary conditions of cooking. This is along the same line as the work published three years previously by Neumann, who called the wood dextrins hydrocellulose. Patent No. 1056162 covers the use of waste sulphite liquors as diluting agents for the sulphuric acid used as the hydrolyzing agent. Patent No. 1056163 relates to the use of chlorine either alone or in conjunction with sulphuric acid. The chlorine must be removed before fermentation because of its inhibiting action on yeast. Patent No. 1091327 relates to the use of "beer slop," the residue from the beer still, as a material for diluting the acid used as the hydrolyzing agent. Patent No. 1096030 covers the use of sulphuric acid and hydrochloric acid, or chloride salts with sulphuric acid, as the hydrolyzing agent.

As has been mentioned before, the work of Neumann confirmed that of Simonsen, involving sulphuric acid as a catalytic agent; but Neumann's main work was with gaseous hydrochloric acid. Girard<sup>27</sup> has shown that hydrocellulose is produced from cellulose by the action of gaseous hydrochloric acid, and Neumann has found that sawdust yields some dextrose in addition to the hydrocellulose. Some of the residue of hydrocellulose and sawdust left after the extraction of the dextrose can be converted further into dextrose. Neumann's yields are comparable to those of Simonsen, although by repeated inversions of the residue Neumann obtains a decided increase of total sugars. The individual inversions of the residue, however, do not yield sufficient sugar or alcohol to be of technical

<sup>&</sup>lt;sup>27</sup> Annales de Chimie et Physique, I, 24, 5 ser., 344.

value. The work of Korner <sup>28</sup> and of Reiferscheidt <sup>29</sup> also substantiates in the main that of Simonsen. Korner attempted to duplicate Classen's work with sulphur dioxide, but could not. Reiferscheidt obtained Classen's yields by using a longer cooking period than that specified by Classen. In addition, several other investigations have been made in which hydrofluoric <sup>30</sup> and other similar acids were used. These investigations are of scientific rather than technical interest, and reference to them may be found in the bibliography.

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Different from the processes mentioned above, in which the inversion of the wood is brought about only by heating with mineral acids, is that class of processes in which the cellulose or wood substance is changed to oxycellulose, acid cellulose, cellose,<sup>31</sup> soluble cellulose, or hydrocellulose before the real inversion takes place. Mention has already been made of Girard's work and Neumann's adaptation thereof. Gentzen and Roth <sup>32</sup> patented the use of ozone <sup>33</sup> as an oxidizing agent in conjunction with sulphuric acid. A yield of 34 per cent of dextrose is claimed in the patent, but Korner, in repeating the work, could not obtain any increase in yield over that obtained by sulphuric acid alone.

Korner further investigated the effects of hydrogen peroxide, potassium dichromate, and potassium persulphate in conjunction with sulphuric acid. Hydrogen peroxide increased the yields about 50 per cent above those obtained with sulphuric acid alone, whereas potassium dichromate, potassium persulphate, and ozone all decrease the yields. The use of salts, such as the dichromate and persulphate, may be criticized in that their presence may facilitate the production of secondary compounds.

By prolonged treatment of spruce with concentrated nitric acid, Lindsey and Tollens<sup>34</sup> prepared an oxycellulose which could not be hydrolyzed to a sugar under any condition. If we consider that the salts present in Kroner's experiments exerted no deleterious action and were inert during the inversion, then the results of Lindsey and Tollens seem to be in accord with those of Kroner, in that the oxycellulose prepared by them was the final product of oxidation, and the product obtained by Kroner with potassium dichromate or persulphate was an intermediate product, some of which could be hydrolyzed. Perhaps only a part of the wood was completely oxidized, and the yields of sugar obtained were on the remaining material which had not been acted upon by the oxidizing agent.

<sup>28</sup> Zeit.für ang. Chemie, 1908, 2353.

<sup>29</sup> Ibid., 1905, 44.

<sup>&</sup>lt;sup>30</sup> J. J. D'Orlowski, French Patent No. 405187; also L. Spassky, French Patent No. 451268.

<sup>&</sup>lt;sup>31</sup> G. Ekstrom, United States Patents Nos. 1087743 and 1087744.

<sup>82</sup> United States Patent No. 745676.

<sup>&</sup>lt;sup>38</sup> See also Charles Doree and M. Cunningham, "The action of ozone on cellulose III action on beech wood (Lignocellulose)"; Jour. Chem. Soc. 103, 677-686; Jour. Chem. Soc. 101, 497-512.

<sup>&</sup>lt;sup>24</sup> Liebig's Annalen, vol. 267, 341.

The action of hydrogen peroxide would then necessarily be one of pure hydration, in which the production of hydrocellulose was facilitated; for, according to Neumann, since the final yield of dextrose is dependent upon the amount of hydrocellulose originally present or upon the speed of its formation, the production of dextrose from hydrocellulose proceeds at a greater rate than the production of hydrocellulose from cellulose.

From the foregoing it is apparent that, since the publication of Simonsen's work and the obtaining of patents on his process, the production of ethyl alcohol from sawdust has received a very large degree of attention practically all over the world, and large sums have been spent on its technical development. Of the four plants which have been built in this country only two have achieved commercial success, but the failure of the others was due apparently to other things than the process itself.

# OUTLINE OF INVESTIGATIONS.

The greatest fault common to all of the work that has been done heretofore is that it was aimed chiefly at an increase in the total yield of sugars, whereas, as will be shown later, such an increase does not necessarily mean a proportionate increase in alcohol yield. Most of the fermentation work done was haphazard and not of the same scientific character as the chemical work. Without accurate fermentations, and, consequently, without complete data, the interpretation of results led to difficulty, because oftentimes total sugar yields might not vary and yet might give different alcohol yields because of variations in the proportion of fermentable and nonfermentable sugars. Since we have no good quantitative chemical means for separating these two classes of sugars and must depend on fermentation, which is a biological process, carefully standardized fermentation experiments are an absolute necessity. The importance of this point, as will be shown later, can not be too strongly emphasized. Simonsen and others after him have contented themselves with an occasional fermentation (usually under conditions that made accurate duplication impossible) to show that some of the sugar obtained was actually fermentable. A careful study of sugar and alcohol relations, especially of the effect of the different variables on that portion of the total sugar that is fermentable, has not, to the knowledge of the writer, been made public heretofore.

Simonsen, Neumann, and other investigators obtained contradictory data because they used as a variable different amounts of an acid solution of constant strength, thereby simultaneously varying both the ratio of water to wood and that of acid to wood. As will be shown in a study of these variables, these ratios are not mutually dependent, and the "acidity." of the solution used for hydrolysis is of little or no importance, except where it varies the actual ratio of acid to wood; the concentration of the catalytic agent expressed in per cent, or other terms, of the dry wood is the decisive factor.

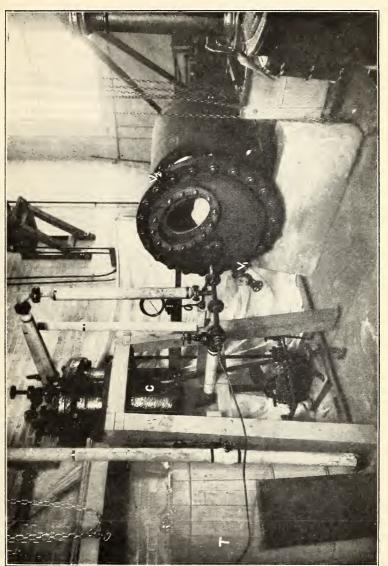
In view of these facts, it was decided to reinvestigate certain of the variables studied heretofore, in addition to such others as might be considered necessary to the technical solution of this problem. This bulletin presents the results of the first part of such a systematic study.

The variables under investigation were: (1) Influence of the temperature and pressure of digestion; (2) length of the time of digestion; (3) ratio of the water to the dry sawdust; (4) ratio of the catalyzing agent to the dry sawdust; (5) concentration of the catalyzing agent in water; (6) size of the sawdust, hogged slabs, etc.; (7) effect of adding the catalyzing agent (acid) after the preliminary heating of the wood; (8) effect of varying the amount of bark in the sawdust; or, more specifically, the tannin and other ingredients in the bark; (9) special chemical treatments other than or in addition to acid catalysis; (10) yields from different species and mixtures; (11) the fermentation variables; (12) steam consumption for each ton of sawdust digested.

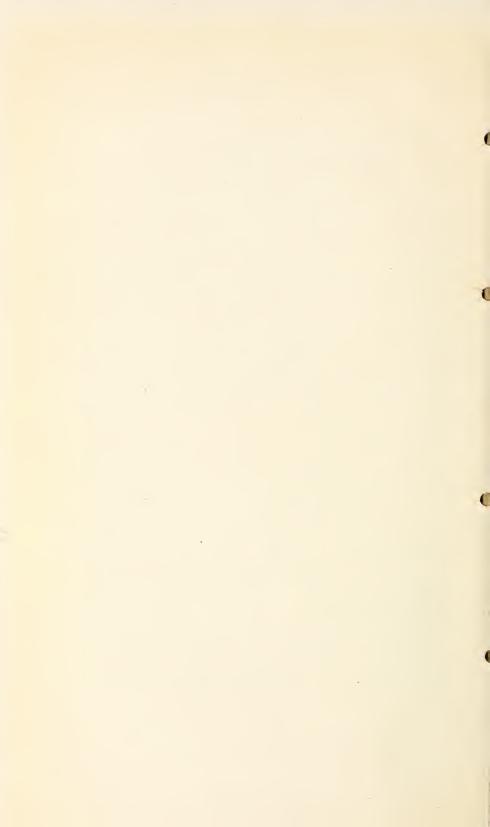
#### APPARATUS AND PROCEDURE.

The apparatus used and the method of procedure in each experiment were as follows: The hydrolysis of the wood was carried on in a rotary digester consisting of a thin cast-iron inner shell lined with acid-proof enamel and an outer shell of steel, the two being separated by several inches. The inside length of the inner shell was about 5 feet, the diameter about  $2\frac{1}{2}$  feet, and the total capacity about 22 cubic feet. Steam was admitted simultaneously to the inner shell and to the space between the inner and outer shells, the digester being similar to a steam-jacketed apparatus, except that the inner shell could be readily taken out and replaced. After a cook had been completed, the digester was blown off, the blow-off vapors being condensed in a quartz coil. A cast-iron tank, also lined with acidproof enamel, was connected with the digester in such a way that its contents might be introduced into the digester when the latter was under pressure. The steam flowed to the inner shell and to the space between the two shells through separate pipes. The one leading to the inner shell connected with the acid tank. All pipes in contact with acid liquor or acid vapor were enamel-lined, and the valves were of special bronze, so as to reduce corrosion to a minimum and avoid as much as possible those complications in fermentation that arise from the presence of iron, copper, and zinc salts. The pressure was

Bul. 983, U. S. Dept. of Agriculture.



FRONT VIEW OF DIGESTER SHOWING MANHOLE COVERS OFF AND SAWDUST IN INTERIOR.



measured by means of a gauge protected from the acid vapors, and the temperature was shown by a recording thermometer, the bulb of which projected into the sawdust. The digester was filled and emptied through a pair of concentric manholes in the inner and outer shells. The usual procedure was to make the charge of sawdust equivalent to about 100 pounds dry weight. The exact weight and moisture-content were recorded. The dilute acid was then added, the manhole covers were bolted on, steam was admitted, and rotation was begun. Before the temperature reached 100° C., the air in the inner shell and in the space between the two shells was vented to get a more accurate gauge reading. The admission of steam was continued until the desired pressure was reached and then regulated so that the heating period was always 20 minutes, or as near that length of time as possible. The steam was then throttled to maintain the desired pressure for the necessary time.

At the completion of the reaction (or in cooks of 15 minutes or more, 2 or 3 minutes before the time was up) the rotation was stopped and the vapors were blown off and condensed as rapidly as possible. The time of blow-off varied somewhat, depending on the pressure at which the cook was made and the amount of material in the digester. The condensing and cooling capacity of the coil, however, was not equal to the demands made upon it, so that blowing-off the digester took much longer than it should have done—about  $1\frac{1}{4}$  hours from 7 or 8 atmospheres to atmospheric pressure.

The condensed blow-off was weighed and analyzed for volatile acid. The condensation from the steam between the two shells was drained out and weighed. It was also tested qualitatively for dextrose to detect any leakage through stuffing boxes or flanges from the inner shell. Whenever liquor was present in the inner shell, it was drained out through the blow-off valve, after which the digester was rotated so that the manholes were at the bottom, and the sawdust was raked out. After the preliminary series of experiments, this material was centrifuged. The digester liquor, centrifugal liquor, and treated sawdust were first weighed and then analyzed for acidity, total solids, dextrose, etc.

#### METHODS OF ANALYSIS.

The following methods of analysis were used:

#### MOISTURE.

About 15 grams of air-dry sawdust or 80 grams of digested or extracted sawdust were weighed into tared crystallizing dishes of glass and dried over night in an oven at 105° C. Although the digested sawdust samples at times charred somewhat, a comparative series in

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which the acid was neutralized before drying show no difference between neutralized and unneutralized material.

#### LEACHED TREATED SAWDUST.

Duplicate 100-gram samples of digested sawdust were weighed into Jena beakers holding 600 c. c., approximately 300 c. c. of water was added, and the beakers were placed on the steam bath. After 30 minutes the solution was filtered, and the filtrate was collected in a 2-liter volumetric flask. More water to the amount of 300 c. c. was then added to the sawdust, and the heating was repeated, a longer time being allowed for each extraction. The total time of extraction was 2 days, two of the extractions extending over night. The filtrate was made up to 2,000 c. c., and the latter was used for analysis.

#### SUGARS.

Allihn's method of determining the reducing sugars, by means of Fehling's solution, as given in Bureau of Chemistry Bulletin 107 Revised, was followed. The copper oxide was filtered in an asbestos Gooch crucible, washed with hot distilled water, and dissolved with 7 c. c. of concentrated nitric acid. It was then diluted and filtered into a 300 c. c. beaker and electrolyzed after the addition of  $5 \text{ c. c. of a saturated sodium acetate solution. Hollard's stationary electrodes, consisting of a gauze cathode and a wire-frame anode, were used with a current density of 7.5 amperes at 2.4 volts. After all the copper was deposited, the electrodes were washed in water, alcohol, and ether, dried and weighed, and the dextrose was calculated from the copper numbers in the above-mentioned bulletin.$ 

#### TOTAL SOLIDS.

One hundred cubic centimeters of the extract was evaporated to dryness in a tared crystallizing dish (in a tannin oven or on the steam bath), then placed in the 105° C. oven one hour, and finally cooled in desiccator and weighed.

#### VOLATILE ACIDS.

A 100 c. c. sample of the extract was distilled to heavy frothing with 10 c. c. of 85 per cent phosphoric acid. Distilled water to the amount of 100 c. c. was then gradually added from a separatory funnel, as fast as distilled, until the volume of distillate approximated 200 c. c. The distillate was then made up to 250 c. c. A 100 c. c. sample of this distillate was treated in a covered beaker with about 2 grams of mercuric oxide for three hours on the steam bath. After removal from the steam bath, 10 or 15 c. c. of phosphoric acid was added, and the sample was redistilled. Three titrations were then made, as follows: On the original sample, which gave the total acid, fixed and volatile; on the first distillate, which gave acetic and formic acid; and on the second distillate, which gave acetic acid only.

After the proper samples had been taken, the digested sawdust was placed in the leaching tank, where the sugar was extracted from it with a number of portions of warm water. The liquor from the digester and centrifugal (whenever there was any) and the leach liquors were then combined, neutralized with calcium carbonate and allowed to settle. After settling, the clear liquor was decanted and concentrated to a heavy sirup in a single-effect vacuum evaporator. These sirups were saved until the concentrates from two or three runs were obtained, and then they were diluted to proper strength for fermentation.

## YEASTING AND FERMENTATION.

The yeast used was a pure-culture strain of Saccharomyces cerevisiæ isolated from a yeast used in a Hungarian distillery producing alcohol from beet-sugar molasses. This yeast is well adapted to the fermentation of sugar solutions obtained from the hydrolysis of wood and is considered to be the best strain available for this purpose.

The yeast was propagated in a beer wort, which was made up as follows: To 100 parts of water, 3 parts of hops were added, and the mixture was boiled vigorously for 15 minutes. The hops were filtered off while hot and from 25 to 35 parts of ground barley malt were added. The mixture was kept at 70° C. for four or five hours until the starch had all been converted, as shown by the iodine test. The malt was then pressed off and the liquid filtered. The filtrate polarized 18° to 20° in a saccharimeter. One liter was then put into a 2 or 5 liter cotton-plugged Erlenmeyer flask and sterilized in an Arnold sterilizer on three successive days. A small drop of the culture yeast kept in sterile sugar solution was added, and the fermentation was allowed to go to completion in about four or five days at 30° C., after which the resulting beer was poured off the yeast, and a 10 per cent sterile sucrose solution was added. About 10 c. c. of the yeast solution was then placed in a 50 c. c. sterile Florence flask, and these samples were used for starting the yeast. All transfers were made and similar work was done in a Hansen culture cabinet under sterile conditions.

The more recent practice for control work at the Forest Products Laboratory has been to propagate the yeast in a Pasteur flask in beer wort made as above, from which it is transferred to Freudenreich flasks which have side necks. Under these conditions it has been possible to propagate a yeast of strict purity, and all possible sources of contamination have been eliminated. The yeast will keep for long periods in the sugar solution, and, therefore, a three or four months' stock can usually be made up at one time.

When a series of fermentations was ready to be run, the small yeast sample in either the 50 c. c. Florence flask or the side-neck Freudenreich flask was added to 50 c. c. of 12° Brix sterile molasses, which was kept at 30° C.  $\pm 0.5^{\circ}$  C.<sup>35</sup> for about 24 hours. Of this solution 10 c. c. was then added to 250 c. c. of 18° Brix molasses after about 18 hours at 30° C. Of this sample 50 c. c. was transformed to 1,500 c. c. of 18° Brix molasses, which was allowed to work off to about 12° Brix in 15 to 18 hours, when it was ready for use as the starting yeast.

Meanwhile a sprout mash was made up of 8.5 grams of malt sprouts and 2.5 grams of ammonium sulphate, boiled for 15 minutes in 250 c. c. of the wood-sugar solution to be fermented. This was cooled to 30° C., and 75 c. c. of the starting yeast (in 12° Brix molasses) was added. After 6 hours the mash was washed into a 20-liter bottle (a half carboy) with 100 c. c. of the wood-sugar solution. The latter varied in concentration from about 10° to 13.5° Brix, the average being 12° to 12.5°. The exact concentrations for each fermentation are shown on the fermentation sheet. After 3 hours 100 c. c. of wood-sugar solution was added; after 3 hours, 200 c. c.; after 3 hours, 200 c. c.; after 2.5 hours, 400 c. c.; and each hour thereafter, 400 c. c., until a final volume of 2 gallons (7,570 c. c.) was made up. The general plan of starting and filling the fermenters is shown in the following table:

	-	Volume addęd.	Total volume.			Volume added.	Total volume.
Sept. 24 Sept. 25	9.30 a. m. <sup>1</sup> 3.30 p. m. 6.30 p. m. 9.30 p. m. 12.30 a. m. 3.00 a. m. 4.00 a. m. 5.00 a. m. 6.00 a. m. 7.00 a. m. 5.00 a. m.	$\begin{array}{c} C. \ c. \\ 250 \\ 100 \\ 100 \\ 200 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \end{array}$	$\begin{array}{c} C.\ c.\\ 250\\ 350\\ 450\\ 650\\ 1,250\\ 1,650\\ 2,050\\ 2,450\\ 2,850\\ 3,250 \end{array}$	Sept. 25	9.00 a. m. 10.00 a. m. 12.00 m. 2.00 p. m. 3.00 p. m. 3.00 p. m. 4.00 p. m. 5.00 p. m. 5.00 p. m. 7.00 p. m.	$\begin{array}{c} C. c. \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 400 \\ 320 \end{array}$	$\begin{array}{c} C. \ c. \\ 3, 650 \\ 4, 050 \\ 4, 450 \\ 5, 250 \\ 5, 650 \\ 6, 050 \\ 6, 850 \\ 7, 250 \\ 7, 570 \end{array}$

Fermentation No. 2.	Cooks 29, 3	33, 34, 35, 36, 37	, 38, and 39.	Starting yeast, 12° Brix.
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<sup>1</sup> Time sprout mash was set.

The above scheme was intended to duplicate as nearly as possible the times at which the different amounts of the solutions would be added from the different yeast tubs to the fermenters on a commercial scale. The first steps up to 3 a. m. are acclimating and propagating the yeast in the yeast tubs, and the final transfer into the fermenters is made at 3 a. m. From then on it takes from 12 to 18 hours in

<sup>25</sup> All fermentations were made in a fermentation room, the temperature of which was kept constant at 30° C.±0.5° C., and which could be sterilized and kept in a clean condition.

commercial practice to fill the fermenters. The length of this period depends on the size of the coolers and on the temperature of the cooling water and of the neutral juice to be cooled; for the extraction of sugar is carried on either at  $70^{\circ}$  to  $80^{\circ}$  C. or at  $40^{\circ}$  to  $45^{\circ}$  C. in the diffusion battery, and the solutions cool only slightly while being neutralized and settled.

At 9.30 a. m., September 26, the first-day Brix reading was taken; and at 9.30 a. m., September 29, the beers were distilled for analysis. This gave a fermenting period slightly in excess of 96 hours (the time allowed by the Bureau of Internal Revenue for a sour-mash fermentation), for the filling of the fermenters was actually begun at 3 a. m., September 25, instead of at 9.30 a. m., September 24. However, the error introduced is practically of no significance, for the attenuation on the fourth day is usually only 0.2° Brix at the most. The work was greatly simplified by this scheme, and most of it could be done in the usual laboratory working hours.

As will be noted on the sugar and alcohol yield sheets, the fermentation efficiencies are high; that is, higher than the 90 to 94 per cent that would ordinarily be obtained, the chief reasons for this are to be found in: (1) The alcohol added along with the starting yeast; (2) the unfermented molasses in the starting yeast, which subsequently fermented; <sup>36</sup> (3) errors in sugar determinations; (4) errors in sampling.

The fermentable sugars and fermentation efficiencies are calculated as follows, and the effect of the above errors and their magnitude will be shown. The wood-sugar solution obtained after neutralization and settling, and hereafter called the neutral juice, is analyzed before and after fermentation. The solution after fermentation is called the beer. The sugar is always expressed in grams of dextrose to the liter, although it was actually a mixture of dextrose, possibly other hexoses (as in the case of western larch), and pentoses. The following formula gives the percentage of the total sugars that are fermentable, expressed as dextrose:

Dx. in neutral juice, grams per liter	Dx. in beer, grams per liter	
Sp. gr. of juice	Sp. gr. of beer	100 = Percentage of total
Dx.in neutr Sp. gr. of		sugars fermentable.

The fermentable sugars are defined as all sugars that have disappeared during fermentation, whether the resulting product is alcohol or not. If the product is not alcohol, it will appear in the fermentation efficiency figure. If the sugar in the neutral juice before fermen-

<sup>&</sup>lt;sup>26</sup> The 18° Brix molasses worked off to about 12° Brix when it was added to the sprout mash. On the fourth day, when a sample of the yeast was distilled for analysis, the Brix readings ranged between 4° and 5°.

tation, expressed in percentage, is multiplied by the percentage of sugar fermentable, and by the constant 0.5111 (which is the amount of alcohol theoretically possible from 1 unit of dextrose), the result is the amount of alcohol theoretically obtainable from the sugar present in the neutral juice. The actual percentage by weight of alcohol in the beer divided by this theoretical alcohol figure will give the fermentation efficiency.

The first and second causes for the high fermentation efficiencies may be grouped together. As shows on the fermentation record sheets, the starting yeast will average 6 per cent of alcohol on the fourth day, which is equivalent to 4.5 c. c. of absolute alcohol. Α 12.5° Brix neutral juice will give a beer averaging 2.4 per cent of alcohol, the 2 gallons being equivalent to 181.7 c. c. of absolute The alcohol from the yeast is, therefore, 2.48 per cent of the alcohol. total alcohol and causes the fermentation efficiency to be high by approximately this amount. In beers having less than 2.4 per cent of alcohol the error will be greater, and in beers of greater alcohol content, the error will be correspondingly less. The figure obtained for fermentation efficiency is very important for comparative purposes; and, as the error is nearly a constant one, no corrections were made in calculating these values in order to eliminate the alcohol derived from the yeast. In addition, it permits of the expression of yields in the way they would be obtained commercially, for, in either case, commercial or experimental, about 1 per cent by volume of the total mash consists of the starting yeast solution.

The magnitude of the errors involved in the sugar determination is more difficult to determine. In addition to sugars, there are other reducing substances present. These are principally aldehydes and formates, for it is known that considerable quantities of formic acid are present before neutralization. Further, although the sugar is determined and calculated as dextrose (*d*-glucose), the sugar is actually a mixture of this hexose, sometimes with pentoses and sometimes with other hexoses. Moreover, the ratio of these sugars to each other varies in the different samples taken, and, although after fermentation the sugars consist entirely of pentoses and nonfermentable hexoses, they are determined and calculated as dextrose. This opens two possibilities for error: First, varying quantities of pentoses affect the accuracy of the actual dextrose determination; second, the reducing powers of sugars other than *d*-glucose are not the same as the power of *d*-glucose.

Stone  $^{37}$  and Browne  $^{38}$  differ as to the relative reducing powers of arabinose and xylose as compared with *d*-glucose, but both writers

<sup>&</sup>lt;sup>37</sup> Stone, W. E., Berichte 23, 3796.

<sup>&</sup>lt;sup>38</sup> Browne, C. A., Jour. of the Am. Chem. Soc. 28, 439.

show that the first two are not the same as d-glucose in reducing power. Browne says:

The statement has been made that in a mixture of sugars the reducing power of the individual sugars is somewhat modified by the other members present. The writer has subjected this statement to a thorough test and can discover no such influence.

However, no wide ranges of varying sugar concentrations were tried by Browne, and therefore this point was checked up at the Forest Products Laboratory with mixtures of arabinose, xylose, and *d*-glucose, and no modifying influence in mixtures was found at widely varying concentrations. Since there is no mutual influence, it is possible to correct the analyses after fermentation, if desired, for the nonfermentable sugars may in most cases be regarded as pentoses such as xylose. As the ratio of the reduction factors of xylose and dextrose, as found by Browne and corroborated by the work at the Forest Products Laboratory, was 0.983, no great error has been introduced, because the reducing power of xylose is so nearly that of dextrose.

The main error due to sampling, other than unavoidable ones arising from the sampling of large quantities of material, has been found and in a great measure overcome. On the sugar and alcohol yield sheets it will be noticed that up to cook 43 it was necessary to discard one or more of the fermentations in many of the cooks, because of poor yields or abnormal fermentation efficiencies. The reasons for these discrepancies were apparent. After the sugar was extracted from the wood and the acid solution had been neutralized, the clarified, settled neutral juice was concentrated to a heavy sirup in vacuo and stored as such. In the different cooks, varying amounts of sludge were precipitated when the neutral juice was concentrated. This sludge consists mainly of calicum sulphate, some calcium acetate, and calcium formate, with possibly some crystallized sugar. When a fermentation was to be made, the heavy sirup was diluted with water to give a solution of about 12.5° Brix. Previous to cook No. 43, samples of the 12.5° Brix juice were taken for analysis along with the sludge remaining in the carboy before sterilization. After that time the samples were made up and allowed to stand all night; the clear juice was then siphoned out into a clean 5-gallon bottle and given two intermittent sterilizations. The sample for analysis and the final Brix reading were taken at the same time that the 240 c. c. sample was taken for making up the sprout mash. This apparently obviated all of the former difficulties, and but few fermentations were discarded after this scheme was inaugurated. In addition, the sugar data in each run were much more concordant.

A constant-temperature bath, which will regulate to  $+0.05^{\circ}$  C., also materially assisted in the accuracy of the alcohol determination. The beers were cooled to 20° C. Portions of 100 c. c. each were taken for distillation, to which were added 50 c. c. of distilled water and about 5 grams of precipitated calcium carbonate, along with 3 or 4 drops of high-boiling paraffin oil to prevent foaming. A portion of 100 c. c. was distilled over and caught in a volumetric flask. If a drop or two of paraffin oil came over, it was readily removed with a small strip of absorbent paper toweling. The 100 c. c. of distillates was then placed in the 15.6° C. constant-temperature bath; after it came to temperature the volume was made up, and the specific gravity was determined by means of a Boots double-wall vacuum pycnometer. In this way the alcohol-content of the beer could be determined very accurately. Although only a small amount of calcium carbonate was necessary to neutralize the volatile acid present, as can be shown by a redistillation of the first distillate, an excess of calcium carbonateabout 5 grams-was used to prevent bumping.

Beginning with fermentation No. 10, the total solids in the neutral juice and beer were determined. This was done in an attempt to correlate the specific gravity, Brix, and sugar data in the neutral juice, and also to give a check on the fermentable sugars and alcohol determinations. It has been found, for instance, in the fermentation of waste sulphite liquors that sufficient volatile compounds, mostly sulphur compounds, distill over in the alcohol determination to make this determination from the gravity of the distillate practically worthless. The addition of alkali and the redistillation helped, but even then there was not much correlation between the alcohol as determined and the sugar data. When determinations were made of total solids, however, it was found that, if the difference in the two determinations before and after fermentation were assumed to be alcohol and carbon dioxide, and if the alcohol were calculated from that difference, the results agreed quite well with the other analytical data and especially with the yields of alcohol obtained commercially or on a large scale experimentally. The data obtained at the Forest Products Laboratory on total solids, however, have not been of such assistance, and frequently the total solids do not even follow the specific gravity or Brix readings; much less do they give a good indication of the alcohol yields, as the following table will show:

#### Соок 56.

Mash before fermentation.				Beer.									
Fer- men- tation No.	Sp. gr. at 15° C.	Brix,	Total solids.	Dex- trose per liter.	Sp. gr. at 15° C.	Brix.	Total solids.	Alco- hol by weight.	Alco- hol from total solids.	Differ- ence in total solids.	Fer- ment- able sugar, aver- age.	Alco- hol yields, aver- age.	Alco- hol per ton.
16 16 17 17 18 20 20	$\begin{array}{c} 1.\ 0514\\ 1.\ 0509\\ 1.\ 0475\\ 1.\ 0498\\ 1.\ 0487\\ 1.\ 0487\\ 1.\ 0429\\ 1.\ 0542 \end{array}$	12. 4 12. 4 12. 0 12. 4 12. 3 10. 7 13. 1	Per ct. 9.567 8.858 9.428 9.978 11.110 8.606 10.750	$\begin{array}{c} Grms.\\ 65, 20\\ 64, 60\\ 57, 34\\ 60, 10\\ 66, 52\\ 56, 90\\ 71, 29 \end{array}$	$\begin{array}{c} 1.\ 0270\\ 1.\ 0268\\ 1.\ 0268\\ 1.\ 0268\\ 1.\ 0278\\ 1.\ 0262\\ 1.\ 0227\\ 1.\ 0287\\ \end{array}$	6.8 6.8 6.8 7.2 6.6 5.8 7.1	$\begin{array}{c} P. \ ct.\\ 5.\ 842\\ 5.\ 848\\ 5.\ 873\\ 6.\ 160\\ 6.\ 070\\ 5.\ 278\\ 6.\ 493\\ \end{array}$	Per ct. 2.390 2.395 2.259 2.345 2.543 1.968 2.590	$\begin{array}{c} 1.899\\ 1.548\\ 1.817\\ 1.952\\ 2.576\\ 1.701\\ 2.176\\ \end{array}$	$\begin{array}{c} 3.\ 715\\ 3.\ 010\\ 3.\ 555\\ 3.\ 818\\ 5.\ 040\\ 3.\ 328\\ 4.\ 257\end{array}$	}77. 15	P. ct. 8.295	Gauls. 25.09

#### Соок 57.

17 18 18 19	$\begin{array}{c} 1.0509 \\ 1.0463 \\ 1.0400 \\ 1.0474 \end{array}$	12.5 11.5 10.0 11.7	$\begin{array}{c} 10.\ 614\\ 10.\ 076\\ 9.\ 962\\ 8.\ 705\\ 8.\ 539\\ 8.\ 465 \end{array}$	$\begin{array}{c} 66.\ 32 \\ 62.\ 02 \\ 52.\ 26 \\ 60.\ 98 \end{array}$	$\begin{array}{c} 1.0263 \\ 1.0247 \\ 1.0213 \\ 1.0240 \end{array}$	$     \begin{array}{r}       6.8 \\       6.3 \\       5.3 \\       6.3 \\     \end{array}   $	6.023 5.781 4.948 5.805	2.543	$\begin{array}{c} 2.072\\ 2.137\\ 1.920\\ 1.398 \end{array}$	4.053 4.181 3.757 2.734	78.90	8.222	24. 87
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#### Соок 58.

18 19 19	$\begin{array}{c} 1.0497 \\ 1.0523 \\ 1.0540 \end{array}$	$12.4 \\ 12.7 \\ 13.3$	$ \begin{array}{r} 11.006 \\ 9.679 \\ 10.598 \end{array} $	81. 48 83. 15 87. 19	1.0275 1.0285 1.0300	7.0 7.3 7.6	6. 484 7. 196 7. 453	$\begin{array}{c} 2.\ 218\\ 2.\ 117\\ 2.\ 421\\ 2.\ 500\\ 2.\ 456 \end{array}$	2.311 1.780 1.608	$\begin{array}{c} 4.522 \\ 3.483 \\ 3.145 \end{array}$	60. 48	6.768	20. 47
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In this table two extremes of fermentable sugars were chosen, namely, cooks Nos. 56 and 57, with high fermentable sugar and alcohol yields, and cook No. 58, with low fermentable sugar and alcohol yields. There were always some alcohol yields, as calculated from the total solids, that were above, as well as some that were below, those actually determined by distillation. It seems, therefore, that the figure for total solids is no criterion, especially when the ratio of sugar to total solids given on the digester record sheet is examined. Extreme cooking conditions, such as high acid, high pressure, and long-time cooks, increased the production of total solids other than sugars. In these experiments, however, the total solids were determined in the acid extracts. After the latter had been neutralized, as in the neutral juice before fermentation, the proportion of sugars to total solids was still lower because of the large amount of calcium salts of the organic acids present. Because of the latter, apparently, there was a large variation of alcohol, as calculated from the total solids and by distillation. The Brix and specific gravity sometimes varied with the total solids and sometimes with the sugars. In cook No. 56, fermentation No. 17, there was a 12° Brix juice with 57.34 grams per liter of dextrose; and in the same cook, fermentation No. 20, there was a  $10.7^{\circ}$  Brix juice, with 56.90grams per liter of dextrose. The whole question here seems to be one of the solubility of the sludge at the time the neutral juice is diluted before fermentation. Commercially no such variation would be obtained. Concentration of the neutral juice in an evaporator is unnecessary, for the reason that a juice of proper concentration for fermentation is obtainable directly from the diffusion battery.

As outlined previously, the variations in sugar data in the different fermentations seem to have been caused by the presence of sludge when the sample was taken. With the adoption of the method by which the clear neutral juice was siphoned from the sludge before sampling, these variations practically vanished. Cook No. 41, however, affords two check sets of fermentable sugar data with wide variations, as shown in the following table:

	Total sugar, per cent of dry wood.	Denerat	[	Alcohol yield.	
Fermentation No.		Per cent of total sugars fer- mentable.	Fermenta- tion efficiency.	Per cent of dry wood.	Gallons absolute. per ton.
4 	23. 09	$\begin{array}{r} 32.95 \\ 57.18 \\ 56.80 \\ 47.20 \\ 45.46 \end{array}$	102.6689.1495.46101.06101.90	$\begin{array}{c} 3.992 \\ 6.690 \\ 6.399 \\ 5.629 \\ 5.466 \end{array}$	$\begin{array}{c} 12.\ 075\\ 20.\ 235\\ 19.\ 355\\ 17.\ 026\\ 16.\ 533 \end{array}$

Соок 41.

Fermentation No. 4 is evidently of no value. This was found to betrue of most of the other cooks included in this fermentation, although no reason can at present be assigned for it, as the acidity and attenuation of the yeast seemed to be normal. Fermentations Nos. 5 and 6, however, show an average of 56.99 per cent of sugars fermentable, and fermentations Nos. 7 and 8 an average of 46.33 per cent of sugars fermentable. Both fermentations show fairly good checks and apparently normal fermentation.

The first of the above averages was chosen as the cook average, for, as will be shown later, that point is on the curve in the series in which cook No. 41 belongs and is, no doubt, the proper value. The reason for the second set of results is still unknown. It is to be regretted that lack of material prevented further fermentations on this cook, as the above is the only case in which a condition of this kind was noticed.

The acidity of the yeast, neutral juice, and beer is expressed in degrees, each degree being the number of cubic centimeters of N/10 alkali required for 20 c. c. of solution, phenolphtalein being used as an indicator. Another unit that is frequently used in this country is

based upon the number of cubic centimeters of N/10 alkali required to neutralize 10 c. c. of this solution and is, consequently, equivalent to two of the above-mentioned degrees. In general, both the yeast and the neutral-juice acidity increased about 4° during fermentation. However, as outlined previously, the yeast was propagated in molasses which had not been sulphited, and in the course of several years the acidity of the molasses increased about 15°. A microscopic examination showed the presence of both bacteria and cocci, and the increase in acidity was probably caused by both of these. Sometimes the acidity increased during the fermentation almost double the average amount without doing any apparent harm.

## RESULTS.

The first series of digestion experiments was more or less preliminary in character, as it was necessary to overcome a number of technical difficulties growing out of unusual conditions that required a combination of high pressure and high temperature in the presence of an acid. It required further time to organize and coordinate the work properly, especially in view of the fact that each successive run or pair of runs represented different experimental conditions. The fermentation equipment was not ready at the time; and, although some fermentations were necessarily made, no great confidence was placed in the value of the results. The total sugar data may be considered accurate, however, in view of the fact that confirmative runs were made later, in which the necessary fermentations also were carried out.

The complete data for all the runs, beginning with No. 21, will be found in the Appendix. These include the digester record, fermentation record, sugar and alcohol yields, and volatile-acid yields. An analysis of the various results obtained is given under the different subheadings that follow.

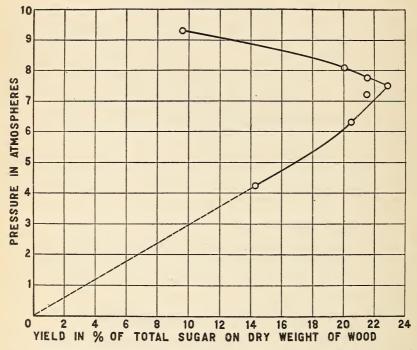
#### EFFECT OF TEMPERATURE AND PRESSURE.

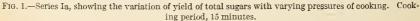
The effects of various temperatures and pressures are grouped into two classes—the first series, in which the ratio of water to wood was 400 per cent (four times as much water as dry wood), the ratio recommended by Simonsen; and the second series, in which the ratio of water to wood was 125 per cent. In the first series the ratio of acid to wood was 1.8 per cent, which was found by Simonsen to yield the best results; and in the second series the ratio of acid to wood was 2.5 per cent. In addition, the first series was run with two time variables—first, a 15-minute cooking period, and, second, an instantaneous (0-minute) cook. The results are given in the following tables and curves. SERIES Ia.

400 per cent of  $H_2O$ ; 1.80 to 1.85 per cent of  $H_2SO_4$ ; cooking time, 15 minutes.

Cook No.	Pressure (atmos- pheres).	Yield of total sugars (per cent of dry wood).
8	4, 25 6, 3 17, 1 to 7, 3 7, 5 7, 75 8, 1 9, 3	14.2920.4821.5022.8521.5420.0511.58

<sup>1</sup> The steam pressure varied between these points and it was not possible to keep the digester pressure uniform.





	ΙE	Ι	

400 per cent of H<sub>2</sub>O; 1.80 to 1.85 per cent of H<sub>2</sub>SO<sub>4</sub>; cooking time, 0 minutes.

Cook No.	Pressure (atmos- pheres).	Yield of total sugars (per cent of dry wood).
20	6.5 7.5 7.5 9.0	1%, 34 22, 59 22, 70 21, 29

#### SERIES II.

125 per cent of H<sub>2</sub>O; 2.5 per cent H<sub>2</sub>SO<sub>4</sub>; cooking time, 15 minutes.

Cook No.	Pressure (atmos- pheres).	Yield of total sugars.	Per cent of total sugars ferment- able.	
58 (2)	6.0 7.5 9.0	22, 82 23, 50 21, 08	60, 48 69, 36 78, 90	$ \begin{array}{r} 6.768 \\ 8.260 \\ -8.222 \end{array} $

<sup>2</sup> Not an actual cook. The data are interpolated from cooks Nos. 45, 46, and 47, which are similar to the above but for 0, 10, and 20 minutes. The 15-minute cook was not made, but may easily be derived from the above series.

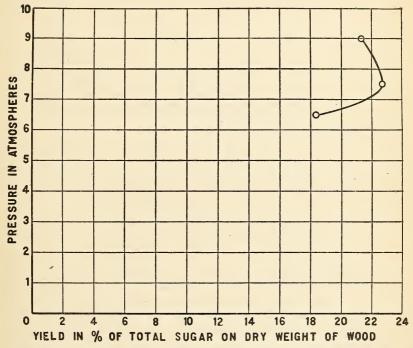


FIG. 2.—Series Ib, showing the variation of yield of total sugars with varying pressures of cooking. Cooking period, 0 minutes (instantaneous).

From the above results a maximum is observed in total sugar yields at 7.5 atmospheres gauge-pressure and the temperature corresponding thereto, which was  $174^{\circ}$  C. or  $344^{\circ}$  F. Above this point decomposition set in, and if Ia series is compared with Ib, it is seen that this decomposition was naturally much greater in the 15-minute cook than in the instantaneous (0-minute) cook. Below the above temperature, as was to be expected, the yield was not so great, because the speed of reaction and the yield of the final product is a function of the temperature. The higher the temperature the greater the speed of the reaction and the greater the yield in a given time, so

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long as decomposition does not become appreciable; but the following table <sup>39</sup> on the decomposition of dextrose in sulphuric-acid solution shows a marked decomposition above 175° C. (1 gram of dextrose in 25 c. c. of sulphuric acid of various concentrations heated for 30 minutes at the indicated temperatures.)

Per cent of sulpharic acid.	Per cent of undecomposed dextrose.				
r ei oen of sinputric acti.	At 150° C.	At 160° C.	At 175° C.	At 185° C.	
0.1 .5. 1.0. 1.5. 2.0. 2.5. 3.0. 5.0.	100.0 96.1 94.4 85.5 85.6 83.3 80.5	94. 4 92. 3 50. 5 75. 0 72. 2 71. 0 35. 8	94.2 91.6 855.5 37.2 33.8 25.0 5,5	SS. 8 50.0 33.3 31.1 5.5 5.0 2.7 .0	

Neuman calls 175° C. the "critical point" and claims that technically there is no need of investigating the production of sugars at higher temperatures. The results of the work at the Forest Products Laboratory support this statement.

Series II, however, shows that even though the total sugar yield decreased at temperatures higher than that corresponding to 7.5 atmospheres, the portion of the total sugars which was fermentable increased sufficiently to balance the decrease in total sugars, and hence the final alcohol yield was practically the same at 7.5 and 9 atmospheres. As outlined before, the necessity for complete data is at once apparent, and much of the value of series Ia and Ib would be lost if the data for series II were not at hand. The increase, or rather, the percentage of increase, in fermentable sugars may be explained in part by the fact that there is a selective decomposition; that is, the pentose or reducing substances other than the hexose present are more easily decomposed at the high temperature than is the dextrose. The following table of volatile-acid yields shows that there was greater sugar decomposition, with consequent formation of formic acid, at 9 atmospheres than at 6 atmospheres.

Cook No.	Pressure (atmos- pheres).	(percent of	Formic acid (per cent of dry wood).		Total acid (per cent of dry wood).
55. 57. 47. 46.b.	6.0 9.05 7.5	2.47 3.53 2.62 2.86	0.399 .659 .340 1.450	6.19:1 5.36:1 7.71:1 1.65:1	2.569 4.189 2.960 3.510

s 10-minute cook.

b 15-minute cook.

<sup>24</sup> Neumann, Dissertation, Dresden, 1910, p. 31.

A similar increase in formic acid is shown in the curve for series Ib on page 50.

As no data were available in the literature on the decomposition of pentoses under the conditions that obtained in these experiments, a number of autoclave cooks were made in which both pure dextrose and pure xylose in sulphuric-acid solutions were used. The solutions were made in such a way that the concentrations of sugar would be comparable to those obtained in the regular runs, and the data from these cooks are given in the two following tables:

Solution of 0.8 of a gram of xylose in 100 c. c. of 0.5 per cent sulphlric acid, heated to 112 pounds in 20 minutes. Held at 112 pounds for 15 minutes.

	· 1.	Ι	лп.
Original xylose	0. 4000	0. 4000	0. 4000
	. 2268	. 2044	. 2056
	56. 7	51. 15	51. 4
	None.	None.	None.
	. 0232	. 0280	. 0280

Solution of 4 grams of dextrose in 100 c. c. of 0.5 per cent sulphuric acid, heated to 112 pounds in 20 minutes. I, held at 118 pounds for 15 minutes; II, held at 112 to 116 pounds for 15 minutes.

	· 1.	11.	111.
Original dextrose	$1.000 \\ 1.072 \\ .0046 \\ .0926$		

In addition to the above two series, one cook was made on an aqueous solution of dextrose instead of a sulphuric-acid solution and with the following results:

Aqueous solution of 4 grams of dextrose in 100 c. c. of solution, heated to 112 pounds in 20 minutes. Held at 112 pounds for 15 minutes. Reducing sugar 0.1891 gr. cu.=0.09685×10×4=3.874 grams of dextrose per 100 c. c.

Acetic.	Formic.	Total.
0.70 c. c. .70 c. c. .00406 g.	0.25 c. c. .25 c. c. .0011 g.	0.95 c. c. .95 c. c.

The above data confirm the experimental results obtained in cooks in the series mentioned and are also extremely interesting, as it is found that approximately 50 per cent of xylose was decomposed under the conditions used, even in a solution containing only 0.8 of a gram per 100 c. c. of 0.5 per cent sulphuric acid. Furthermore, only formic acid is produced, with no acetic acid whatever. A dextrose solution having five times the concentration of dextrose showed on an average 64 per cent of the original dextrose remaining; and, although formic acid was the main constituent of the total volatile acid, some acetic acid was formed. Another series of experiments was made on autoclaving the neutralized juice before and after fermentation. The results of these experiments are given in the following tables. It is of particular interest to note here that a greater sugar decomposition occurred in the autoclave of both the neutral juice and the beer when no sulphuric acid was added than when sulphuric acid was added before autoclaving. The volatile-acid figures, although somewhat erratic, show a decided tendency toward an increase of volatile acid, although here we have a mixture of conditions in which, undoubtedly, a number of secondary reactions take place, and some of the combined acids that are present either as calcium salts or organic combinations are liberated during the autoclave process.

No.Reducing sugars per $100 c. c.$ Acetic acid per $100 c. c.$ Sulphuric acid per $100 c. c.$ Total volatile acid,Neutral juice: $74-6 (1)$ $39.72$ $14-6 (2)$ $0.248$ $15.148$ $0.380$ $-522$ $0.628$ $-522$ $74-6 (1)$ $74-6 (3)$ $39.72$ $-526$ $0.248$ $-582$ $0.380$ $-522$ $0.628$ $-522$ $74-6 (1)$ $-74-6 (4)$ $39.72$ $-74-6 (4)$ $0.628$ $-522$ $-938$ $-533$ Beer: $74-7 (1)$ $-74-7 (2)$ $-74-7 (3)$ $17.99$ $-129$ $387$ $-665$ $-665$ $-1.052$ $-74-7 (4)$ Neutral juice: $73-6 (1)$ $-73-6 (2)$ $14.24$ $-29$ $-713$ $-14.29$ $-184$ $-129$ Neutral juice: $73-6 (2)$ $-73-6 (4)$ $29.03$ $-20.33$ $-184$ $-29.03$ $-184$ $-29.03$ $73-7 (1)$ $-73-7 (1)$ $-73-7 (2)$ $-16624$ $-10.98$ $-1900$ $-12.94$ $-1430$ $-14.526$ $73-7 (2)$ $-73-7 (3)$ $-16624$ $-10.98$ $-1430$ $-12.96$						
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	No.	sugars per	acid per	acid per	acid per	volatile
	$\begin{array}{c} 74-6\ (1) \\ 74-6\ (2) \\ 74-6\ (3) \\ 74-6\ (3) \\ 74-6\ (4) \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\ \\$	$\begin{array}{c} \bar{1}8, 148\\ 31, 824\\ 36, 08\\ 17, 99\\ 7, 70\\ 12, 29\\ 13, 11\\ 41, 29\\ 14, 24\\ 22, 04\\ 29, 03\\ 41, 624\\ 12, 94\\ 20, 75\\ \end{array}$	.356 .263 .333 .387 .480 1.208 .341 .713 .883 .868 .527 1.038 1.418 1.418	$ \begin{array}{r}     582 \\     540 \\     .338 \\     .665 \\     .754 \\     .095 \\     .671 \\     .184 \\     .238 \\     .404 \\     .238 \\     .439 \\     .392 \\     .540 \\     .410 \\   \end{array} $		938 933 671 1.052 1.234 1.303 1.012 .897 1.116 .966 1.430 1.958 1.866

Autoclave cooks on fermented and unfermented wood-sugar extracts.

(1) original; (2) original autoclaved, 112 pounds for 15 minutes; (3) original plus 0.5 per cent of sulphuric acid autoclaved, 112 pounds for 15 minutes; (4) original plus 0.5 per cent of sulphuric acid autoclaved, 112 pounds for 15 minutes.

The experimental procedure covering the previous series of autoclave cooks on fermented and unfermented extracts, also sugar solutions in both water and sulphuric acid, was carried on as follows: Fifty cubic centimeters of the solution was put into a pear-shaped, porcelain-stoppered, rubber-gasketed pressure flask of glass, which was put into an autoclave, the flask being surrounded by water. The autoclave was gas heated, and steam pressure was turned into the autoclave so fast as to make the autoclave cooks comparable with the digester cooks. As the flasks were stoppered, none of the volatile acids could escape, and they were determined along with the sugars in the solutions after they were cooked in the autoclave.

Comparing these yields with those shown above for series II, it is seen that the yields were higher at 6 and 9 atmospheres, no doubt on account of different amounts of water present. For all technical purposes, therefore, there is no advantage in exceeding 7.5 atmospheres (112 to 115 pounds per square inch gauge pressure), for above this point decomposition of sugar sets in. The decomposition of the fermentable sugars was apparently not so great as that of the nonfermentable sugars, and the final alcohol yield was, therefore, not greatly affected; but the increased amounts of volatile acids formed are undesirable because of the possibility of an inhibiting action during fermentation.

# LENGTH OF TIME OF COOKING.

When the pressure of 7.5 atmospheres was used as a constant, and when the time of cooking was varied, the following results were were obtained:

#### PRELIMINARY SERIES III.

Time variable; 1.8 to 1.85 per cent of H<sub>2</sub>SO<sub>4</sub>; 400 per cent of H<sub>2</sub>O; 7.5 atmospheres.

Cook No.	Time of cook, min- utes.	Per cent of total sugars.	Per cent of total sugars fer- mentable.		Gallons ab- solute per ton.
15. 16. 21.1 18. 17.	0 0 0 15 30	22, 59 22, 70 23, 16 22, 85 22, 95	54.87	6. 096	

<sup>1</sup> Cook 21 had 300 per cent water instead of 400 per cent.

#### SERIES III.

Time variable; 2.50 per cent of  $H_2SO_4$ ; 125 per cent of  $H_2O_2$ ; 7.5 atmospheres.

	(Dime of	Per cent of	Per cent of	Alcohol yields.		
Cook No.	Time of cook, min- utes.	sugars.	total sugars fer- mentable.	Per cent of dry wood.	Gallons ab- solute per ton.	
31. 45.	0 0	21. 45 22. 77	<b>63.</b> 66 62, 65	6. 859 6. 994	20.75 21.15	
Average	0	22.11	63.16	6.927	20, 95	
32. 47.	10 10	$\begin{array}{c} 21.32\\ 23 40\end{array}$	69, 79 67, 27	7. 339 7. 984	$22.20 \\ 24.15$	
Average	10	22.36	68, 53	7.662	23.18	
46	$\begin{array}{c} 20\\ 45\\ 90 \end{array}$	$23. \ 61 \\ 21. \ 56 \\ 18. \ 06$	71. 44 77. 15 81. 40	8, 537 8, 295 7, 387	$25.82 \\ 25 09 \\ 22.34$	

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## SERIES IIIa.

	Time of	Per cent of	Per cent of	Alcohol yields.		
- Cook No.	cook, min- utes.	total sugars.	total sugars fer- mentable.	Per cent of dry wood.	Gallons ab- solute per ton.	
33 40	0 0 - 0	$23.\ 17 \\ 23.\ 74 \\ 22.\ 37$	54. 20 53. 67 53. 92	$\begin{array}{c} 6.972 \\ 6.319 \\ 6.214 \end{array}$	$21.09 \\ 19.11 \\ 18.80$	
Average	0	23.46	53.94	6. 646	20. 10	
24 41	10 10	$23.81 \\ 23.09$	53, 16 56, 99	6.362 6.550	19. 24 19. 81	
Average	10	23.45	55.08	6.456	19. 53	
42	30	22.34	63.22	6. 862	20.75	

1.4 per cent of H<sub>2</sub>SO<sub>4</sub>; 125 per cent of H<sub>2</sub>O; 7.5 atmospheres.

1 Blow-off open, not averaged.

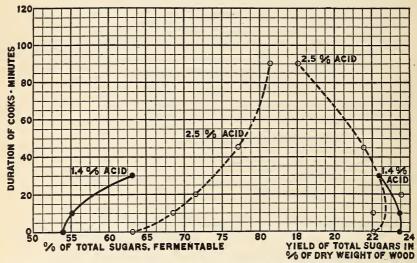


FIG. 3.—Series III and IIIa, showing the variation in yield of total sugars and percentage of total sugars that is fermentable with varying cooking periods at two different acid concentrations. Cooking pressure, 7.5 atmospheres.

These results are further illustrated graphically in figures 3, 4, and 5.

In a previous publication,<sup>40</sup> based on preliminary series III, the author stated that the time of cooking was apparently without effect. This statement was based on a total sugar data only, as practically all other alcohol data up to that time had been, and must now be, modified.

Altogether three concentrations of acid and two concentrations of water were used, except in cook 21, as noted in the table. For

<sup>40</sup> Jour. of Ind. and Eng. Chem. 1914, 625.

the instantaneous cooks no great difference in total sugars will be noted. Figure 3, series III*a*, shows a gradual decrease in the 1.4 per cent acid series, and a slight increase followed by a decrease in the 2.5 per cent acid series in total sugar yields; whereas figure 4, preliminary series III, shows practically no difference in a 0, 15, or 30 minute period. As shown in figure 3, however, decided increases were attained in the portion of total sugars which is fermentable, with increased alcohol yields (fig. 5), especially in the 2.5 per cent acid series. In this series the maximum yield was 8.54 per cent of alcohol, after which the yield dropped, although the yield of fermentable sugars kept on increasing. The total sugar yield, however,

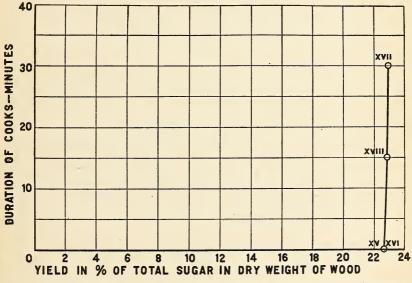


FIG. 4.—Preliminary series III, showing the variations in yield of total sugars with varying periods of cooking. Cooking pressure, 7.5 atmospheres.

decreased at a greater rate than the above increase, and gave lower alcohol yields.

Here again the data would permit of only doubtful interpretation or would lead to erroneous conclusions if only the total sugars were taken into account. The explanation of these results will be taken up later.

# RATIO OF WATER TO WOOD.

All of the preliminary cooks were made with 400 per cent of water that is, four times the dry weight of wood. This was the water ratio used by Simonsen, although that given in United States Patent No. 938308 by Ewen and Tomlinson was somewhat less than 4 to 1.

From an operating standpoint the reduction of the amount of water used is greatly to be desired. The reasons for this are: First,

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the steam consumption in cooking is greatly decreased. Second, there is difficulty in obtaining a juice sufficiently concentrated to ferment and distill economically, because, if an excess of liquor is present in the digester, a large portion of the sugar is dissolved therein. Third, there is difficulty in handling a dripping, digested sawdust from which the acid liquor must be separated. The following table shows the results obtained by decreasing the ratio of 4 parts of water to 1 of dry wood down to equal parts of each.

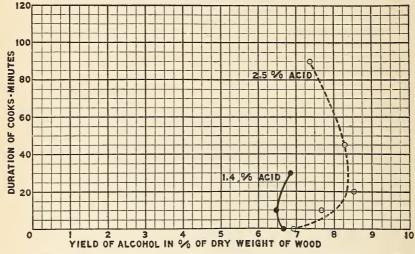


FIG. 5.—Series III and IIIa, showing variation in alcohol with varying cooking periods at two different acid concentrations. Cooking pressure, 7.5 atmospheres.

SERIES IV.

Water to wood ratio variable; 1.80 to 1.83 per cent of H<sub>2</sub>SO<sub>4</sub>; 7.5 atmospheres; 0 minute.

Cook No.		Den sent	Per cent	Alcohol yields.		
	Per cent H <sub>2</sub> O.	Per cent of total sugars.	of total sugars fer- mentable.	Per cent of dry wood.	Gallons absolute per ton.	
26. 21. 22. 34. 30.	$400 \\ 300 \\ 250 \\ 125 \\ 100$	$22. 24 \\ 23. 16 \\ 23. 75 \\ 21. 96 \\ 21. 09$	$56. 19 \\ 54. 87 \\ 55. 31 \\ 59. 29 \\ 60. 68$	$\begin{array}{c} 6.154\\ 6.096\\ 6.648\\ 6.805\\ 6.440\end{array}$	$     18.61 \\     18.44 \\     20.11 \\     20.58 \\     19.48 $	

SERIES IVa.

1.40 per cent of H<sub>2</sub>SO<sub>4</sub>; 7.5 atmospheres; 0 minute.

			Per cent	Alcohol	yields.
Cook No.	Per cent H <sub>2</sub> O.	Per cent of total sugars.	of total sugars fer- mentable.	Per cent of dry wood.	Gallons absolute per ton.
43 44	200 100	21.84 23.00	57. 94 57. 16	6.369 6.665	19.62 20.16

In the experiments of series IV, 1.80 per cent of sulphuric acid was used. The experiments were then repeated with the use of 100 and 200 per cent of water with 1.40 per cent of sulphuric acid, and the results were the same. These results are shown graphically in

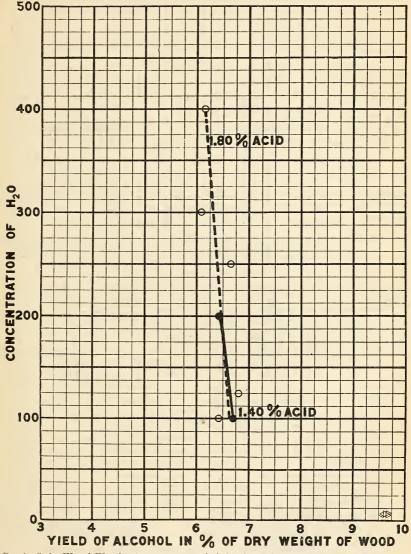
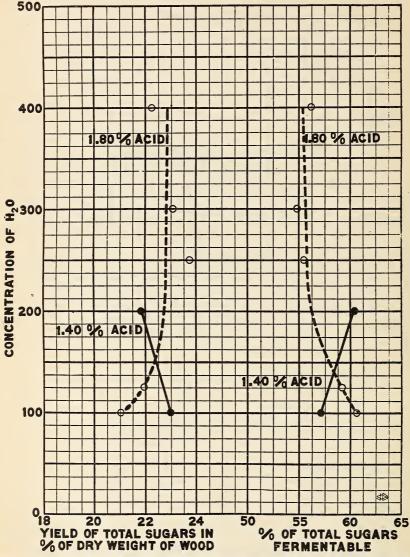
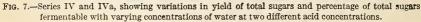


FIG. 6.—Series IV and IVa, showing variation in alcohol yields with varying concentrations of water at two different acid concentrations.

figures 6 and 7. Figure 6 shows that the average alcohol yields were very nearly constant for the different concentrations of water used, and figure 7 shows that this also held true for both the amounts of total sugars and the percentages of sugars that were fermentable. There was a little more variation in the latter figures than in the alcohol values, because in the sugar data there was apparently a combination of variables in opposite directions. The 1.80 per cent





acid curves show that there was a slight decrease in total sugars with decreasing amounts of water; but the percentage of fermentable sugars increased at the same time, with the result that the actual alcohol yield was practically constant. A larger number of cooks would probably have reduced these variations, but they were not deemed necessary to establish the principle involved.

Although 100 per cent of water gave results practically as good as did larger amounts, the operating conditions chosen for future work was 125 per cent of water. This was done for two reasons: First, to insure a better mixture of the acid in the wood, which, of course, is easier to accomplish with larger amounts of water, and, second, to use the maximum amount of water possible and still obtain a digested sawdust that has no drip. The advantages from the use of 125 per cent of water were felt to outweigh the small increase in steam consumption and to justify its adoption for future work. From calculations that have been made to determine the steam load on a commercial digester it was found that it takes 1,816,000 B. t. u. to a cook for heating the wood and acid solution, and 1,183,000 B. t. u. to heat the digester-a total of 2,999,000 B. t. u. to a cook. Decreasing the ratio of water to wood from 125 per cent to 100 per cent would decrease the total steam load to 2,709,000 B. t. u., a difference of 290,000, or about 10 per cent. The digester load, however, is only 25 to 30 per cent of the total steam load of the plant; consequently, the above would make a difference of only 2 or 2.5 per cent of the total steam load of the plant.

# RATIO OF ACID TO WOOD.

The above ratio of water to wood (125 per cent) and a 0-minute cooking period were the constants used in the next series, in which the ratio of acid to wood was the variable. Sulphuric acid was used as the catalytic agent and in amounts varying from 0.5 per cent to 4 per cent of the dry weight of the wood. The results are given in the following table:

			Per cent of	Alcohol yields.		
Cook No.		Per cent of total sugars.	total sugars ferment- able.	Per cent of dry wood.	Gallons absolute per ton.	
38	$\begin{array}{c} 0.5 \\ .75 \\ 1.00 \\ 1.40 \\ 1.40 \\ 1.40 \\ 1.40 \end{array}$	17. 42 21. 83 21. 68 23. 17 23. 74 22. 37	43. 13 56. 03 56. 43 54. 20 53. 67 53. 92	$\begin{array}{c} 4.\ 172\\ 6.\ 085\\ 6.\ 506\\ 6.\ 972\\ 6.\ 319\\ 6.\ 214 \end{array}$	12, 62 18, 41 19, 68 21, 09 19, 11 18, 80	
Average	1.40	23. 455	53, 935	6, 6455	20, 10	
34	1.80 1.80 2.50 2.50	21.9621.0921.4522.77	59, 29 60, 68 63, 66 62, 65	6, 805 6, 440 6, 859 6, 994	20, 58 19, 48 19, 75 21, 15	
Average	2.50	22.11	63. 155	6.9265	20.95	
36	4.00	21.10	66.63	7.000	21.17	

#### SERIES V.

125 per cent of H<sub>2</sub>O; 7.5 atmospheres; 0 minute.

<sup>1</sup> Blow-off open, not averaged.

 $^{2}$  30 to 100 per cent  $H_{2}O$ , not averaged.

SERIES Va.-Shortleaf pine.

			Per cent of	Alcohol	yields.
Cook No.	Per cent $H_2SO_4$ .	Per cent of total sugars	total sugars	Per cent of dry wood.	Gallons absolute per ton.
45 49	2.50 4.00	$17.15 \\ 14.02$	60. 27 66. 59	5.201 4.262	15.73 12.89

125 per cent of H<sub>2</sub>O; 7.5 atmospheres; 10 minutes.

The results of series V and Va for both spruce and shortleaf pine are shown graphically in figures 8 and 9. Figure 8 shows the actual alcohol yield based on the dry weight of the wood, and figure 9 gives

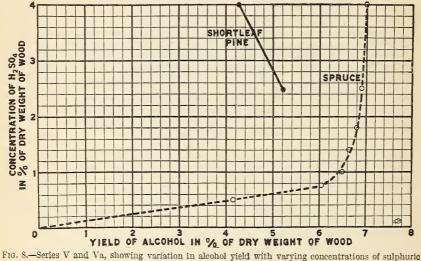


FIG. 8.—Series V and Va, showing variation in alcohol yield with varying concentrations of sulphuric acid for spruce and shortleaf pine.

the variations in total sugars and the portion of total sugars that is fermentable.

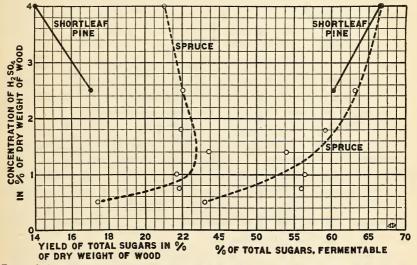
Figure 8 shows that the yield of alcohol from spruce increased rapidly with the lower concentrations of acid, but that above 1 per cent of acid the increase was comparatively small, there being little difference between 1 per cent and 3.5 per cent of acid. There was an actual decrease in the alcohol yield from shortleaf pine. This was because of the decrease of total sugars (fig. 9), although the amount of sugars fermentable increased from 60.29 per cent to 66.59 per cent of the total. The shortleaf pine used was a mixture of band sawdust and hogged slabs and edgings containing about 8 per cent of cypress and a considerable quantity of bark.

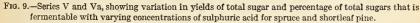
Even from spruce a decrease in total sugars is noticeable with acidities above 1.4 per cent, but here again the percentage of the total

fermentable sugars increases. As a result the total yield is not appreciably lessened and the alcohol yield remains practically constant.

In this series is seen again the importance of complete data; that is, data on total sugars, percentage of total sugars fermentable, and alcohol yields. All of these are necessary for a proper interpretation of the results, especially in an experiment like this, in which there is an apparent neutralization of two factors that vary in different ways.

A study of these two variables—namely, the ratio of water to wood and of acid to wood—shows that they are not mutually dependent,





that the acidity of the solution used for hydrolysis is of minor or no importance, but that the concentration of the catalytic agent expressed in percentage of dry wood is the decisive factor. As stated previously, Simonsen expressed all of his results in total sugar. When such long cooking periods are used (two hours in his work on variable No. 3-the influence of the amount of water present and of the acid concentration), it is not surprising that he found variable sugar yields with a constant amount of acid and varying amounts of water, or varying "acidities," as he called them. The decomposition products so obtained would be largely reducing agents, formic and lævulinic acids, which would show high sugar yields. Practically always, especially in the above work on cellulose, Simonsen used amounts of water which were technically not feasible. Neuman realized the technical importance of decreasing the ratio of water to wood, but carried it no further than 3 to 1-300 per cent of water.

He inverted 50 grams of air-dry sawdust with 150 c. c. of 0.5 per cent sulphuric acid (equivalent to 1.5 per cent of sulphuric acid based on air-dry wood) for one-half hour at 175° C., with 112 pounds of pressure to the square inch, and duplicate experiments showed yields of 20.4 and 20 per cent of total sugars. The percentages of total sugars fermentable and the alcohol yields were not given. In the discussion of his results he gives the following:

These experiments prove that one can work with small amounts of liquid without exerting a deleterious action on the yields. In fact, the yields in experiments 63 and 64 (the two referred to above) are higher than in former ones in which larger amounts of liquids were used. At the same time, the extracts contained a greater percentage of sugar which is also favorable for fermentation. On the contrary, a small amount of liquid (acid solution) is not advisable, since in another experiment with sawdust and 2 parts of liquid (0.5 per cent sulphuric acid) at 175° C. a considerable evolution of sulphur dioxide took place with partial cooking of the materials used.

It must be remembered that the above experiments were made on 50-gram samples of wood heated in an autoclave indirectly; whereas the results obtained at the Forest Products Laboratory were based on 100-pound samples cooked with steam. At the laboratory there was no coking in instantaneous or short-time cooks with 1 part of water to 1 of wood; although, with the higher acid concentrations, irrespective of the amount of water used, there was always some coking—that is, a darkening of the digested wood. When Neumann used sulphuric acid as the catalytic agent, he nearly always employed a 0.5 per cent solution and simply varied the amount. He thereby confused the effect of his ratios of water and acid to wood, since they were both varied simultaneously.

If steam was used as the heating agent, of course some further dilution occurred during cooking; the more water used to begin with, the greater was this dilution. In an experimental apparatus, like that used at the Forest Products Laboratory, the amount of steam required to heat the digester was greater in proportion to the amount necessary to heat the wood and acid solution than it would be in a large commercial digester holding two or more cords of wood. The following data from cooks Nos. 30 and 34 show in general how much this dilution was:

	Cook 30, June 30, 1914.	Cook 34, July 29, 1914.
Water.	1.80 0. 7.5. 38.48. 271.20. 103.55. 103. 5.	125. 1.80. 0. 7.5. 41.41. 288.06. 100.68. 126. 61.38. 1.86 to 1.

In cook No. 30, with an original ratio of water to wood equal to 1 to 1, the ratio increased to 1.62 to 1, an increase of 62 per cent. In cook No. 34, with an original ratio of 1.25 to 1, the ratio increased to 1.86 to 1, an increased of 61 per cent, which was practically identical with that of cook No. 30. In both cooks the yields of total sugars, percentages of total sugars fermentable, and yields of alcohol were practically identical, as shown in the table of results on series IV, page 36. It seems, therefore, that if there is sufficient water to insure a good mixture of the catalytic agent with the wood, both the water and acidity of the solution added are without effect; and that, of these two variables, the only one affecting the yields is the concentration of the catalytic agent based on the amount of dry wood present.

Increasing the concentration of the catalytic agent caused increased vields of total sugars up to about 1.5 per cent of sulphuric acid; then the yields begin to decrease, although the portion of the sugars that is fermentable increased without a break in the curve. The increase in the amount of sugars fermentable is sufficient to offset the decrease in total sugars, and consequently the resulting alcohol yield is practically constant. The explanation of this condition, as shown on pages 30 to 33, is that the nonfermentable sugars (the pentoses) are the more unstable under the conditions used, and pentose decomposition takes place with increasing amounts of the catalytic agent. This pentose decomposition accounts for a decrease of the total sugar yield and an increase of the fermentable sugars, especially if there is little or no hexose decomposition; and the result is a practically constant alcohol yield similar to that obtained. As in the inversion of cane sugar in the presence of an acid, here also the speed of the reaction is probably determined by a combination of the catalytic effects of both the hydrogen ions and the non-ionized acid; moreover, the increased amounts of sugar formed with increased acid concentration may in part be a result of the non-ionized molecule. However, this is not the full explanation of the conditions observed. If it were, the 4 per cent acid series should give yields as much greater than those of the 2.5 per cent acid as the 2.5 per cent acid yields are greater than those of the 1 per cent; but this is not the case.

The above discussion has been limited entirely to the results of the experiments on spruce. Only two cooks were made on shortleaf pine, and these were insufficient to warrant any conclusions, especially in view of the complex nature of the raw material.

### SIZE OF CHIPS.

Two cooks were made on chips of two different sizes and under the conditions outlined in the table below.

	Length of	Per cent	Per cent	Alcoho	l yields.
Cook No.	chip with the grain (inches).	of total sugars.	of total sugars fer- mentable.	Per cent of dry wood.	Gallons absolute per ton.
59	1/5 to 3/16 Mixture.	$20.21 \\ 21.005 \\ 20.14$	73.50 69.07 71.16	$7.126 \\ 7.214 \\ 7.109$	$21.55 \\ 21.82 \\ 21.50$

125 per cent of H<sub>2</sub>O; 2.5 per cent of H<sub>2</sub>SO<sub>4</sub>; 7.5 atmospheres; 15 minutes.

Even the larger chips were thoroughly penetrated and cooked, and the yield obtained from them compares favorably with that obtained from sawdust under similar conditions. The chips, however, did not extract readily, and while they were being stirred in the leaching tank they were ground to a powder. In a commercial diffusion battery there would not, of course, be a similar mechanical action, but the time of extraction and the capacity of the battery would be decreased. A lack of material prevented leaching experiments, and these results were obtained primarily to determine whether material of this size would give yields similar to those from sawdust. Further work along this line is necessary.

Cook No. 61 was a mixture of sawdust, small chips, and large chips in the following amounts:

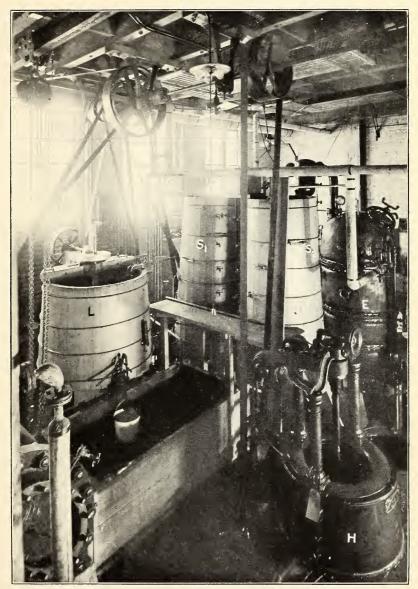
	Moisture.	Air-dry weight.	Dry weight.
Sawdust. Large chips. Small chips.	Per cent. 8.78 9.67 9.37	Pounds. 31.35 46.96 41.04	Pounds. 28.60 42.41 37.20

This mixture was cooked at 7.5 atmospheres with 125 per cent of water and 2.5 per cent of sulphuric acid for 15 minutes. After it was cooked, a sample of the mixture was taken and extracted and the extract analyzed in the usual way. Another part of the sample was screened through a screen having openings three-eighths of an inch square, and the materials passing through the screen and remaining on it were also extracted and analyzed in the usual way. The following table shows the results of these analyses:

Digested mixture.	Moisture (per cent).	Total sugars, (per cent of wet weight).	Total solids (per cent of wet weight).	Acidity of extract (C. c. N/10 NaOH).
Unscreened		6. 616	7. 196	7.5
Remaining on ‡-inch screen		6. 853	7. 28	7.6
Passing through ‡-inch screen		6. 688	7. 32	7.6

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PLATE II.



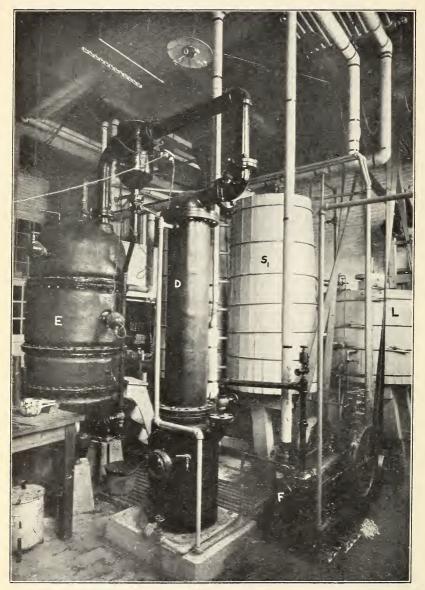
Leaching Tank, L: Settling Tanks,  $S_1$  and  $S_2$ : Single-Effect Vacuum Evaporator, E; Hydro-Extractor, II.

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PLATE III.

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EVAPORATOR E, CONNECTED TO SURFACE CONDENSER D, AND DRY VACUUM PUMP F. CORNER OF FERMENTATION ROOM BUILT IN UPPER CORNER OF LABORATORY BEYOND EVAPORATOR AND CONDENSER.

From these figures it will be seen that all the material was equally cooked and gave practically the same yields, irrespective of size within the limits used. In addition, the material leached readily without formation of fine stuff, and 88.4 per cent of the sugar present was extracted without difficulty in three leachings. A certain amount of sawdust or material of similar size, therefore, seems necessary in order to obtain a good extraction, even though chips as large as five-eighths of an inch with the grain may be cooked under the conditions outlined in as short a time as 15 minutes and with good yields.

It has been observed in commercial practice that a mixture of dust and chips in the proportion of 10 to 15 per cent of dust and 90 to 85 per cent of chips gives the best results in the diffusion battery. If all dust or too much dust is used, the cooked material packs in the cells, high pressures are required to force the extracting water through it, and the material hangs in the cells at the time of discharge, causing loss of time in operation. If dust-free chips are used, the opposite is true; the extracting water percolates too fast, and extraction is not good. With the proper combination of dust and shredded chips, a 92 per cent extraction of the total sugar may be obtained.

# LEACHING EXPERIMENTS.

The determination of the number of cells in a diffusion battery necessary to get a maximum extraction in a minimum time, with a minimum amount of water, is a very important point in this process. Furthermore, it was desirable to determine whether there was any selective solubility between the different sugars present or the soluble solids other than sugars. With this end in view, two series of cooks were made under the same conditions, viz, 125 per cent of water, 2.5 per cent of sulphuric acid, a pressure of 7.5 atmospheres, and a cooking period of 15 minutes. It is regrettable that an accident to some of the condensing apparatus prevented blowing off these cooks, and after each cook it was necessary to allow the digester to cool gradually to 212° F. or less before the contents were discharged. Undoubtedly reducing substances were formed and retained which were the cause of erratic sugar data being obtained.

Cooks Nos. 51 and 52 were preliminary and were made for the purpose of outlining a method of procedure. The digested sawdust from both cooks was put into the leaching tanks and successively leached by sprinkling small quantities of boiling water over the digested sawdust and collecting the drip as soon as it had drained through the sawdust. The different extracts were then analyzed, neutralized, and fermented in the usual manner with the following results.

	Ees	before	al juice fermen- ion.	Beer.		Per cent of		Fer-							
Leach No.	Fer- men- tation No.	Sp. gr. at 15° C.	Reduc- ing sugars, (grams per liter).	Sp. gr. at 15° C.	Reduc- ing sugars (grams per liter).	Alcohol (per cent by weight), (liter).	total retical sugars fermen- y table.	total sugars fermen-	total sugars fermen-		retical	menta- tion effi- ciency.	Neutral juice.	Beer.	In- crease.
1	10	1,0502	62.28	1.0262	17.40	1.910	71.40	2,164	88.26	6.2	12.0	5,8			
1	10	1.0502	61.70	1.0202	11.74	2.267	80.55	2.418	93.76	2.4	9.6	7.2			
2	10	1.0482	61.56	1.0277	14.88	2.233	75.34	2. 262	98.72	4.4	11.2	6.8			
-	11	1.0509	66.04	1.0270	14.74	2.313	77.16	2.478	93.34	4.4	10.4	6.0			
3	10	1.0492	59.52	1.0273	17.08	2.171	70.68	2.050	105.90	6.4	12.5	6.1			
	11	1.0480	60.24	1.0269	16.00	2.152	72.89	2.142	100.47	6.0	11.2	5.2			
4	10	1.0486	62.12	1.0286	18.52	2.129	69.62	2.108	101.00	3.6	11.4	7.8			
5	11 10	1.0521 1.0488	$68.70 \\ 57.28$	1.0296 1.0304	18.88 17.86	2.360 1.844	71.91 68.26	2.400 1.905	98.33 96.80	4.0 2.4	9.6 10.4	5.6 8.0			
9	10	1.0488	62.28	1.0304	17.80	1. 844	69.05	2.087	93.39	3.8	10.4	6.2			
	11	1.0002	02.20	1.0000	10. 02	1. 515	00.00	2.001	50.05	0.0	10.0	0.2			

Cooks Nos. 53 and 54.

Cooks Nos. 53 and 54 were combined and handled in the same way; but not until previous experiments had indicated the quantities of water necessary to leach out approximately equal quantities of sugar. In all, nine leaches were made, although only the drip and the first eight leaches contained enough sugar to make a fermentation possible. The following table shows the amounts of water added for each leach, the amounts of extract recovered, the sugars and total solids in each extract, and the percentages of total sugars fermentable, with the fermentation efficiencies: 0)

COOKS	Nos.	53	AND	54.
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Leach	Amou water		Amount of ex- tract re-	of ex-	Reduc- ing sugars	Weight of sugars in ex-	Total solids	Total solids in ex-	Ratio of sugars to total	Acidity (c. c. N/10
No.	Liters.	Pounds.	(pounds).	tract at 15° C.	(grams	tract (pounds).	(per cent).	(pounds).	solids (per cent).	NaOH per 100 c. c. ).
Drip 1. 2. 3. 4. 5. 6. 7. 8. 9.	$\begin{array}{c} 20 \\ 30 \\ 30 \\ 50 \\ 75 \\ 100 \\ 300 \\ 500 \end{array}$	$\begin{array}{c} & 44.0 \\ & 66.1 \\ & 66.1 \\ & 66.1 \\ & 110.2 \\ & 165.3 \\ & 220.5 \\ & 661.4 \\ & 1,102.3 \end{array}$	83. 29 38. 39 61. 70 63. 76 71. 32 94. 21 160. 93 194. 21 647. 14 997. 94	$\begin{array}{c} \textbf{1.0482}\\ \textbf{1.0518}\\ \textbf{1.0522}\\ \textbf{1.0501}\\ \textbf{1.0501}\\ \textbf{1.0504}\\ \textbf{1.0443}\\ \textbf{1.0360}\\ \textbf{1.0258}\\ \textbf{1.0109}\\ \textbf{1.0015} \end{array}$	$\begin{array}{c} 74.54\\ 80.12\\ 79.48\\ 78.48\\ 75.36\\ 65.75\\ 50.08\\ 34.29\\ 14.00\\ 1.66\end{array}$	$5.93 \\ 2.92 \\ 4.66 \\ 4.77 \\ 5.12 \\ 5.94 \\ 7.77 \\ 6.51 \\ 8.96 \\ 1.655$	$\begin{array}{r} 8.187\\ 8.897\\ 8.866\\ 8.195\\ 8.180\\ 7.314\\ 5.757\\ 4.188\\ 2.198\\ 0.220\\ \end{array}$	$\begin{array}{c} 6.72\\ 3.41\\ 5.47\\ 5.22\\ 5.83\\ 6.90\\ 9.27\\ 8.14\\ 14.21\\ 2.19\\ \end{array}$	88.3 85.7 85.3 91.4 87.9 86.1 83.4 81.9 63.1 75.6	73. 4 75. 0 74. 5 76. 8 74. 6 74. 0 63. 0 63. 0 45. 1 18. 9 2. 1

**46** 

	Tan	Neutra befor menta					Per cent of		Fer-		Acidity		
Leach No.	Fer- men- tation No.	Sp. gr. at 15° C.	Reduc- ing sugars (grams per liter).	Sp. gr. at 15° C.	Reduc- ing sugars (grams per liter).	Al- cohol (per cent by weight).	total sugars	rs retical - alcohol. nt-	alcohol.	men- tation effi- ciency.	Neu- tral juice.	Beer.	In- crease.
Drip 1 2 3 4 5 6	$\left\{\begin{array}{c} 12\\ 13\\ 12\\ 13\\ 12\\ 12\\ 13\\ 12\\ 13\\ 12\\ 13\\ 12\\ 13\\ 12\\ 13\\ 14\\ 12\\ 13\\ 14\\ 12\\ 13\end{array}\right.$	$\begin{array}{c} 1.0515\\ 1.0506\\ 1.0497\\ 1.0460\\ 1.0473\\ 1.0510\\ 1.0520\\ 1.0520\\ 1.0502\\ 1.0502\\ 1.0505\\ 1.0502\\ 1.0515\\ 1.0538\\ 1.0504\\ 1.0456\\ 1.0490 \end{array}$	$\begin{array}{c} 62.38\\ 61.92\\ 62.12\\ 55.12\\ 57.08\\ 63.92\\ 59.52\\ 61.96\\ 57.74\\ 57.34\\ 54.76\\ 58.34\\ 53.22\\ 52.52\\ 47.38\end{array}$	$\begin{array}{c} 1.\ 0337\\ 1.\ 0284\\ 1.\ 0422\\ 1.\ 0367\\ 1.\ 0298\\ 1.\ 0284\\ 1.\ 0275\\ 1.\ 0401\\ 1.\ 0287\\ 1.\ 0299\\ 1.\ 0321\\ 1.\ 0475\\ 1.\ 0329\\ 1.\ 0343\\ 1.\ 0308\\ \end{array}$	$\begin{array}{c} 23.52\\ 10.84\\ 44.70\\ 33.86\\ 18.72\\ 11.74\\ 10.04\\ 35.30\\ 10.68\\ 12.36\\ 13.12\\ 41.16\\ 15.24\\ 41.8,14\\ \end{array}$	$\begin{array}{c} 1.\ 625\\ 2.\ 188\\ 0.\ 648\\ 0.\ 897\\ 1.\ 690\\ 2.\ 266\\ 2.\ 087\\ 1.\ 019\\ 2.\ 090\\ 1.\ 913\\ 1.\ 836\\ 0.\ 549\\ 1.\ 646\\ 2.\ 016\\ 1.\ 790\\ \end{array}$	$\begin{array}{c} 61.65\\ 82.12\\ 27.51\\ 38.03\\ 66.64\\ 81.22\\ 82.77\\ 42.38\\ 81.11\\ 78.02\\ 75.60\\ 29.03\\ 70.89\\ 84.91\\ 82.51\\ \end{array}$	$\begin{array}{c} 1.869\\ 2.474\\ 0.832\\ 1.024\\ 1.856\\ 2.525\\ 2.399\\ 1.276\\ 2.278\\ 2.177\\ 2.012\\ 0.824\\ 1.836\\ 2.180\\ 1.905\\ \end{array}$	$\begin{array}{c} 86.94\\ 88.44\\ 77.88\\ 87.60\\ 91.06\\ 89.74\\ 86.99\\ 79.86\\ 91.75\\ 87.87\\ 91.25\\ 66.87\\ 89.65\\ 92.48\\ 93.96\end{array}$	$\begin{array}{c} 3.9\\ 8.0\\ 3.8\\ 6.8\\ 9.4\\ 3.7\\ 8.8\\ 4.3\\ 9.2\\ 3.9\\ 8.6\\ 8.0\\ 3.3\\ 4.4\end{array}$	$\begin{array}{c} 12.0\\ 13.2\\ 7.0\\ 8.0\\ 10.8\\ 12.4\\ 12.1\\ 11.8\\ 12.8\\ 12.4\\ 12.9\\ 11.4\\ 12.4\\ 12.2\\ 11.0\\ \end{array}$	8.1 5.2 4.8 1.2 7.2 8.4 3.0 8.5 9.0 2.8 4.4 8.9 6.6	
7 8 9 <sup>1</sup>	$\left\{ \begin{array}{c} 14\\ 12\\ 13\\ 14\\ 12\\ 13\\ 14\\ 12\\ 13\\ 14\\ 14\\ \end{array} \right.$	$\begin{array}{c} 1.0515\\ 1.0576\\ 1.0496\\ 1.0481\\ 1.0547\\ 1.0519\\ 1.0489\\ \end{array}$	$\begin{array}{r} 49.56\\ 53.04\\ 46.19\\ 43.76\\ 47.32\\ 44.14\\ 42.46\end{array}$	$\begin{array}{c} 1.\ 0324\\ 1.\ 0371\\ 1.\ 0326\\ 1.\ 0314\\ 1.\ 0380\\ 1.\ 0357\\ 1.\ 0338\\ \end{array}$	8.28 8.48 8.00 7.78 9.00 8.52 7.56	$1.951 \\ 1.851 \\ 1.724 \\ 1.682 \\ 1.772 \\ 1.685 \\ 1.610 $	82. 92 83. 69 82. 39 81. 94 80. 67 80. 39 81. 94	$\begin{array}{c} 1.999\\ 2.145\\ 1.853\\ 1.748\\ 1.850\\ 1.724\\ 1.695\end{array}$	97.60 86.29 93.03 96.22 95.78 97.74 94.98	$\begin{array}{c} 6.0 \\ 4.1 \\ 3.8 \\ 6.4 \\ 3.6 \\ 6.6 \\ 6.4 \end{array}$	$ \begin{array}{c} 10.0 \\ 12.4 \\ 11.2 \\ 11.0 \\ 12.0 \\ 10.8 \\ 12.0 \\ \end{array} $	$ \begin{array}{r} 4.0\\ 8.3\\ 7.4\\ 4.6\\ 8.4\\ 4.2\\ 5.6 \end{array} $	

COOKS NOS. 53 AND 54-Continued.

<sup>1</sup> Insufficient sugar for fermentation.

A considerable amount of variation will be noted in the percentages of total sugars fermentable, although all of the extracts, excepting Nos. 1 and 5, have at least one fermentation with over 80 per cent of the total sugars fermentable. Extract No. 5 shows one fermentation with 75.60 per cent of sugars fermentable, but both fermentations of extract No. 1 are very low. The latter is only 2.72 per cent of the total weight of extract obtained, but contains 5.40 per cent of the total sugar calculated as dextrose. Apparently we have here some strongly reducing substances present which are very soluble, which react as a sugar toward Fehling's solution, but which do not ferment. This material was also extracted in a greater or less quantity in the succeeding leaches up to and including No. 5, and it was, therefore, difficult for the yeast to get control and furnish a good fermentation. Not until the beginning of the sixth leach were fairly uniform and constant fermentations obtained.

From the foregoing results it is apparent that the sugars should be extracted as completely as possible, as the last extracts are apparently the purest from a fermentation standpoint. This work should be repeated, however, for additional data are necessary before final judgment is passed on a phase of the work of so great technica importance. The data given are indicative but insufficient to be conclusive in this respect. Commercially, the subject of extraction, covering the questions of size, design, and number of cells in a diffusion battery, is one that has received a great deal of attention in the beet-sugar industry. In the light of data obtained in units of commercial size in the ethylalcohol industry, it may be said that at least a 5-cell draw—that is, passing the extracting medium through 5 cells in rotation—is necessary, and that a 6 or 7 cell draw is more desirable. The amount of water to be used and the proper end point or time of drawing must be determined for each particular battery, and with proper care and supervision the efficiency of the entire operation can be maintained at a 92 per cent extraction with a 10° to 11° Brix acid juice corrected for temperature.

## VOLATILE ACID YIELDS.

In addition to the sugars obtained as hydrolytic products, acetic and formic acids were also obtained in varying amounts. The yields are given on the acid-yield sheet in the Appendix, in addition to the following tables, in which the cooks have been outlined in the different series as given before under pressure, time, water to wood and acid to wood ratios:

### SERIES Ib.

## PRESSURE VARIABLE.

1.80 per ce	ent of H	$SO_4$ ; 400 I	per cent of H	$L_2O$ (water); (	) minute.

Cook No.	Pressure.	Yield of vol	atile acid (pe veight of woo	er cent of dry od).
	-	Acetic.	Formic.	Both.
20 15 16 19	6.5 7.5 7.5 9.0	$1.25 \\ 1.22 \\ 1.62 \\ 1.60$	$\begin{array}{c} 0.105\ .185\ .220\ .443 \end{array}$	$     \begin{array}{r}       1.355 \\       1.405 \\       1.840 \\       2.043     \end{array} $

#### Series III.

### TIME VARIABLE.

2.5 per cent of H<sub>2</sub>SO<sub>4</sub>; 125 per cent of H<sub>2</sub>O; 7.5 atmospheres.

Cook No.	Time of cook	Yield of vo	latile acid (pe eight of wood	er cent of dry
	(minutes).	Acetic.	Formic.	Both.
31	$\begin{array}{c} 0\\ 0\\ 10\\ 10\\ 20\\ 45\\ 90 \end{array}$	$\begin{array}{c} 1.\ 755\\ 1.\ 670\\ 1.\ 67\\ 2.\ 62\\ 2.\ 360\\ 2.\ 915\\ 2.\ 140 \end{array}$	$\begin{array}{c} 0.\ 572\\ .\ 790\\ 1.\ 090\\ .\ 340\\ 1.\ 450\\ 1.\ 492\\ .\ 440 \end{array}$	$\begin{array}{c} 2.827\\ 2.460\\ 2.760\\ 2.96\\ 3.81\\ 4.407\\ 2.580\end{array}$

#### SERIES IIIa.

## TIME VARIABLE.

1.4 per cent of H<sub>2</sub>SO<sub>4</sub>; 125 pcr cent of H<sub>2</sub>O; 7.5 atmospheres.

Cook No.	Time of cook	Yield of vol	atile acid (pe eight of wood	er cent of dry
	(minutes).	Acetic.	Formic.	Both.
33. 40	0 0 10 10 30	$\begin{array}{c} 1.\ 457\\ 1.\ 560\\ 2.\ 39\\ 1.\ 416\\ 1.\ 55\end{array}$	0.355 .470 .059 .430 .570	$ \begin{array}{r} 1.812\\ 2.030\\ 2.449\\ 1.846\\ 2.120 \end{array} $

#### PRELIMINARY SERIES III.

# TIME VARIABLE.

## 1.80 per cent of $H_2SO_4$ ; 400 per cent of $H_2O$ ; 7.5 atmospheres.

Cook No.	Time of cook	Yield of vol	atile acid (pe	er cent of dry
	(minutes).	Acetic.	Formic.	Both.
15 16 18 17	0 0 15 30	$1.22 \\ 1.62 \\ 1.32 \\ 1.48$	$\begin{array}{c} 0.\ 185 \\ .\ 220 \\ .\ 598 \\ .\ 570 \end{array}$	$1.\ 405 \\ 1.\ 840 \\ 1.\ 918 \\ 2.\ 050$

# SERIES IV.

#### WATER TO WOOD RATIO.

#### 1.80 to 1.83 per cent of H<sub>2</sub>SO<sub>4</sub>; 7.5 atmospheres; 0 minute.

Cook No.	Per cent of water.	we	atile acid (per eight of wood	r cent of dry l).
	water.	Acetic.	Formic.	Both.
26	$400 \\ 300 \\ 250 \\ 125 \\ 100$	$1.75 \\ 1.52 \\ (^1) \\ 2.11 \\ 1.38 $	0. 431 . 239 ( <sup>1</sup> ) . 480 . 642	2. 131 1. 753 ( <sup>1</sup> ) 2. 590 2. 022

<sup>1</sup> Data not complete.

## SERVES IVa.

## WATER 19 WOOD RATIO.

1.40 per cent of H<sub>2</sub>SO<sub>4</sub>; 7.5 atmospheres; 0 minute.

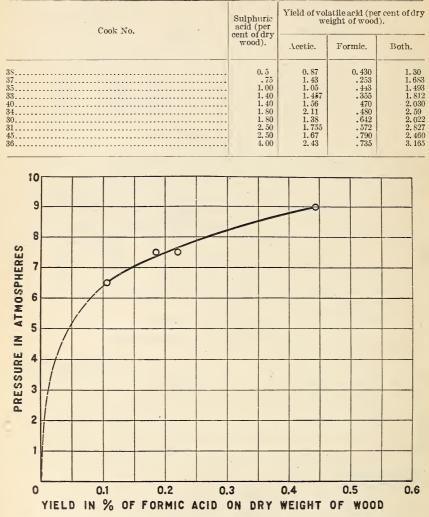
Cook No.	Per cent of water.	Yield of volatile acid (per cent of dry weight of wood).			
		Acetic.	Formic.	Both.	
43	200 100	1.51 1.69	0. 160 . 290	1.660 1.980	

54976°-22-Bull. 983----4

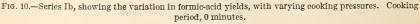
#### Series V.

## ACID TO WOOD RATIO.

125 per cent of H<sub>2</sub>O; 7.5 atmospheres; 0 minute.



0



Both the yields of acetic and formic acid varied with the cooking conditions, although the former was the more constant of the two. The variations in the water to wood ratio in general seemed to be without effect on the yields of both acids, and this was to be expected. Increasing the pressure of cooking increased the amount of formic acid, particularly as shown in the curve for series Ib in figure 10, and this was apparently due in part to sugar decomposition. Increasing the acidity based on the dry wood increased particularly the amount of formic acid produced, whereas increasing the time of cooking seemed to increase both acids, although the increase of formic acid was proportionately much greater than that of acetic acid.

The source of these acids at the temperatures used is apparently a hydrolysis of the acetyl and formyl groups present in the lignin complex, as suggested by Cross and Bevan<sup>41</sup> and by Cross,<sup>42</sup> who determined these acids as results of the acid hydrolysis of a number of materials. Bergström<sup>43</sup> also obtained these acids as a result of hydrolysis without the aid of an acid catalyst, with the use of water at 6 atmospheres of pressure. Formic acid, however, with lævulinic acid, carbon dioxide, etc., is a decomposition product of the sugars, and undoubtedly the large increases in formic acid yields with increased pressure, time of cooking, and concentration of catalyst are due to sugar decomposition. The data are of particular interest as furnishing an approximate index of the degree of this decomposition.

These acids in certain concentrations also have a toxic effect on yeast growth and no doubt inhibit fermentation to some extent, even in the neutralized juice in which the acids are present as their calcium salts. In addition, nearly the same amount of acid was obtained at the Forest Products Laboratory as is usually obtained by the destructive distillation of this species of wood, and in a few cases more. It is known that the rapid decomposition of wood does not begin till a temperature of 275° to 280° C.44 is reached, after which the decomposition is exothermic. Since the formic acid is derived from the wood and also from sugar decomposition, the amount hydrolyzed from the wood directly is difficult to determine. If this amount were known, it would give a helpful index, similar to the methoxy number now used, of the value of a species of wood for certain purposes. It has been shown beyond question 45 that the destructive distillation of cellulose, sulphite, or soda wood pulp, as well as cotton, yields acetic acid, but no methyl alcohol, and Klason and his coworkers have obtained a yield of 2.79 per cent of acetic acid from spruce sulphite cellulose. More than this amount of acetic acid was obtained in cook No. 56 at the Forest Products Laboratory by hydrolysis of the wood at a temperature 100° C. lower.

In view of the work of Cross and Bergström, it seems that the greater part of the acetic and formic acids, with the exception of the formic acid that results from sugar decomposition, comes from the hydrolysis

<sup>&</sup>lt;sup>41</sup> Cross and Bevan, Berichte, 28, 1940.

<sup>&</sup>lt;sup>42</sup> Cross, W. E., Dissertation, Göttingen, 1910, Ueber das Vorkommen der Formyl- und Acetyl Gruppen im Lignin.

<sup>&</sup>lt;sup>43</sup> Bergström, Der Papierfabrikant, 2, 305.

<sup>44</sup> Klason, P., Jour. für prakt. Chemie, 1914, 90, 413-447.

<sup>&</sup>lt;sup>46</sup> Buttner, G., and Wislicenus, H., Jour. für prakt. Chemie, 79, 177-234; Klason, von Heidenstam, and Norlin, Zeit. für ang. Chemie, 1909, 1205.

of the acetyl and formyl groups in the lignin. Bergström obtained as much as 1.53 per cent acetic acid and 0.23 per cent of formic acid from spruce boiled in water at a pressure of 6 atmospheres for two hours without any catalyst, whereas cellulose produced from spruce yielded only 0.08 per cent of combined acids under similar treatment.

This being true, the maximum amount of acid obtainable from wood is the sum of the acid produced by the destructive distillation of the cellulose and the hydrolysis of the lignin. This would be 5.7 per cent total acid, which, so far as the writer's knowledge extends, has never been obtained by the destructive decomposition of wood. Undoubtedly secondary decomposition products are formed, such as  $2CH_3COOH = (CH_3)_2CO$  plus H<sub>2</sub>O plus CO<sub>2</sub>, 2 HCHO plus H<sub>2</sub>O = CH<sub>3</sub>OH plus HCOOH, and similar reactions, which may account for the acetone and in part for the formic acid obtained from destructive distillation.

0

A series of destructive distillations has been made at the Forest Products Laboratory on spruce and spruce-digester residues cooked with sulphuric acid for sugar and alcohol production. These distillations were made, some slowly and some quickly, some very wet and some very dry, to determine whether the hydrolytic effect noted above could be reproduced in the destructive-distillation process. The details of this work are being reserved for later publication; but it may be stated here that the distillation of either wet wood or wet residue produced more total acid than was obtained from the dry wood or dry residue under similar distillation conditions, showing that there was a hydrolytic effect from the added water. The increased amount of acid, however, was not sufficient to pay for its recovery from the more dilute pyroligneous acid produced.

Aside from the scientific interest of the subject and its bearing on the chemistry of wood, the technical importance of the recovery of these acids is to be considered. As outlined previously, this has been attempted in France and in the processes covered by the patents granted to Cohoe in this country. Cohoe, however, worked with broad-leaved woods, which would give even larger amounts of these acids. The table in the Appendix, however, shows that the amount of acid which might be recovered in the condensed blow-off averages only about 10 per cent of each, and this would yield a condensate of little value. As the average concentration of acetic acid in the condensed blow-off was only about 0.20 per cent, it is questionable whether recovery in commercial amounts is possible. If some means could be devised, however, for washing out the volatile acids with steam and making a practically complete recovery of them without too great expense or delay to the digester cycle, these acids might form a valuable by-product of this industry.

## EFFECT OF TANNIN AND BARK.

Various mixtures of spruce and spruce bark were cooked under the usual conditions of 7.5 atmospheres of pressure, 125 per cent of water, and 2.5 per cent of sulphuric acid based on dry weight. The results are given in the following table:

		Total sugars		Per cent	Alcoho	l yields.
	Cook No,	(per cent of origi- nal dry weight).	gi- fermen-	Per cent of origi- nal dry weight.	Gallons absolute per dry ton.	
All spruce wood. 91.5 per cent of wood, 8.5 per cent of bark 74.5 per cent of wood, 25.5 per cent of bark All bark	84 87 85 88	$22.11 \\ 19.60 \\ 22.49 \\ 17.07$	70.3869.4760.0431.95	7.4576.7656.3642.730	22.5520.4619.258.25	

From the result it is seen that as much as 25 per cent of spruce bark may be mixed with the wood without appreciably decreasing the yield of alcohol. Other barks were not available in sufficient quantity to determine the practical limits to which they may occur in waste mixtures without appreciable effect. Commercial experience however, has shown that not more than 10 per cent of the total of yellow pine may be bark without seriously affecting the yield. Spruce bark has long been used in Europe as a tanning material and has found favor because of its high sugar content, which in the "mellowing" or fermentation and acidification of the tanning liquors produced a comparatively large amount of acid and was therefore used as a plumping agent.

The tannin present shows no inhibitory action toward yeast growth, for even sugar liquors produced from red and white oak gave normal fermentation and fermentation efficiencies. In commercial practice other factors have been found, such as long cooking periods with increased formations of acids, acetone, and aldehydes, which influence yeast growth and fermentation much more than does the tannin found in the sugar liquors usually produced from mill waste.

# EFFECT OF CATALYZERS OTHER THAN SULPHURIC ACID OR IN ADDITION THERETO.

As indicated previously, Körner, Cohoe, and others have attempted the production of sugar and alcohol from wood with the use of hydrogen peroxide, potassium dichromate, and potassium persulphate as hydrating and oxidizing agents in addition to sulphuric acid, and these investigators have also recommended the use of hydrochloric acid. The experiments at the Forest Products Laboratory have been only preliminary to an investigation of the field, but they have confirmed several known facts besides establishing several new ones.

Theoretically, hydrochloric acid, because of its maximum ionization should produce even greater sugar yields than sulphuric acid. Hydrochloric acid presents greater technical difficulties than does sulphuric acid, and its cost is greater; however, if the yield were increased sufficiently, the other difficulties could probably be overcome. Two concentrations of hydrochloric acid were therefore tried, one of 1.80 per cent and another of 2.50 per cent, based on dry-wood weight, and the respective total sugar yields were 19.69 per cent and 17.02 per cent. The complete data on cooks Nos. 89 and 90 are given in the tables in the Appendix. That yeast is intolerant of the Cl ion is well known, and the laboratory workers were unable, as others before had been, to obtain any fermentations. Unless, therefore, the Cl ion is removed by precipitation, as with silver nitrate, fermentation is impossible. No fermentation was obtained when chlorine or chlorides were used. The chief interest of the following experiments, therefore, is in the data adduced with respect to total sugars, for, as stated before, if it were possible materially to increase the total sugars obtained, other difficulties might possibly be overcome.

	s pruce.	
	7.5 atmospheres: 125 per cent of $H_2O$ .	
Cook No.	Catalyst.	Total reducing sugars (per cent of original dry wood).
93 90 89 92 94 25 96 97	<ul> <li>}1.8 per cent of hydrochloric acid.</li> <li>2.5 per cent of hydrochloric acid.</li> <li>1.8 per cent of sulphuric acid plus salt (NaCl) for equation H<sub>2</sub>SO<sub>4</sub>+NaCl= NaHSO<sub>4</sub>+HCl.</li> <li>1.8 per cent of sulphuric acid plus salt (NaCl) for equation H<sub>2</sub>SO<sub>4</sub>+2 NaCl=Na<sub>2</sub>SO<sub>4</sub>+ 2 HCl.</li> <li>2 per cent of sulphuric acid plus 10 per cent of KClO<sub>3</sub> to make 2 KClO<sub>3</sub>+H<sub>2</sub>SO<sub>4</sub>= K<sub>2</sub>SO<sub>4</sub>+2 HClO<sub>3</sub>.</li> <li>1.8 per cent of sulphuric acid plus 50 per cent of KClO<sub>3</sub> to make 2 KClO<sub>3</sub>+ H<sub>2</sub>SO<sub>4</sub>=K<sub>2</sub>SO<sub>4</sub>+2 HClO<sub>3</sub>.</li> </ul>	$\left\{\begin{array}{c} 18.25\\ 19.69\\ 17.02\\ 20.44\\ 19.44\\ 19.59\\ 20.34\\ 21.45\end{array}\right.$

The above table shows no yields of total sugars greater than it is possible to obtain with the use of sulphuric acid only. Even if all the sugars produced were fermentable and could be fermented, the yield would not justify the added expense. Simple chloride and chlorine treatments in their technical aspect may, therefore, be dismissed, although the results are of interest in their bearing on the chemistry of wood.

The next experiment was with the use of ferrous sulphate in conjunction with sulphuric acid—that is, with an increase in the concentration of sulphate ions. The result, especially when taken in connection with the experiments on niter cake described below, is of sufficient interest and value to warrant further investigation. Reference should be made to all the data of cook No. 104, which was a standard cook using 1.8 per cent of sulphuric acid plus 0.1 per cent of ferrous sulphate. The results are given in the following table, along with data from cook No. 34, showing the results from the use of 1.8 per cent of acid without the addition of ferrous sulphate. Although the differences between the two cooks are small, the slight increase in fermentable sugar when the ferrous sulphate was used is worthy of consideration.

125 per	cent of	$H_2O;$	7.5 atmos	pheres.
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	Cook No. Catalyst.	Total sugars.	Per cent of total sugars	Alcohol yield.		
Cook No.			ferment- able.	Per cent of dry wood.	Gallons per ton.	
54 <b></b>	1.8 per cent of H <sub>2</sub> SO <sub>4</sub> . {1.8 per cent of H <sub>2</sub> SO <sub>4</sub> . (0.1 per cent of FeSO <sub>4</sub> .	$\left. \begin{array}{c} 21.96\\ 21.40 \end{array} \right\}$	59.29 63.23	6.805 6.927	20. 58 20. 97	

Two cooks were made with niter cake furnished through the courtesy of the E. I. du Pont de Nemours Co. In cook No. 107, niter cake only was used, and the amount of it was equivalent to 1.8 per cent of concentrated acid. In other words, 5.55 pounds of cake are equal to 1 pound of 100 per cent acid. In cook No. 108 a mixture of 0.9 per cent of acid and niter cake equivalent to 0.9 per cent of acid, making a total of 1.8 per cent of acid, was used. The results follow:

Cook No.	Catalyst.	Total	Per cent of total sugars	Alcohol yields.		
		sugars.	ferment- able.	Per cent of dry wood.	Gallons per ton.	
107. 108	Niter cake. 0.5 niter cake, 0.5 H <sub>2</sub> SO <sub>4</sub>	17. 93 19. 25	60. 13 59. 18	5. 583 5. 077	16. 89 15. 38	

When these results are compared with those obtained from the use of acid alone, it is found that niter cake will produce, either alone or when mixed with as high as 50 per cent of the usual acid concentration, 75 or 80 per cent of the yield obtained with sulphuric acid alone. These experiments suggest interesting possibilities, provided the niter cake can be obtained at an appreciable saving as compared with the cost of sulphuric acid.

## 56 BULLETIN 983. U. S. DEPARTMENT OF AGRICULTURE.

Three cooks were made with the use of phosphoric acid  $(H_3PO_4)$  alone or in mixture with sulphiric acid, and the results were as follows:

Spruce.

7.5 atmospheres; 125 per cent H <sub>2</sub> O.								
Cook No.		Total	Per cent of	Alcohol yields.				
	Catalyst.	sugars (per cent of original dry wood.)	total sugars ierment- able.	Per cent of original dry wood.	Gallons per ton.			
100	1.5 per cent of sulphuric acid, 0.1 per cent of phosphoric acid.	21, 12	64.32	7.726	23.24			
101	0.9 per cent of H <sub>2</sub> SO <sub>4</sub> , 0.9 per cent of H <sub>3</sub> PO <sub>4</sub> .	20, 83	55, 20	5. 635	19.62			
102	1.5 per cent of H <sub>3</sub> PO <sub>4</sub>	19.38	51. 58	5,180	15.69			

The above results are even better than were to be expected, if the relative degrees of dissociation of the two acids are considered. The slight increase in yield of cook No. 100, containing 0.1 per cent of phosphoric acid in addition to 1.8 per cent of sulphuric acid, as compared with cook No. 34 (p. 55), containing 1.8 per cent of sulphuric acid only, is not sufficient when its cost is considered to warrant the commercial use of even small quantities of phosphoric acid. Phosphates, of course, are necessary for yeast growth: and if they are present in considerable quantities in the mash during fermentation they act as a yeast stimulant. It has not been found necessary, however, to add any great quantity of phosphates in order to secure satisfactory fermentation. A pound or two of ammonium phosphate or a pint of "sirupy" acid added to the starting yeast in the early stages of its propagation is sufficient to insure vigorous growth.

# STUDY OF DIFFERENT SPECIES.

All the cooks in this series were made under the following conditions: 2.5 per cent of sulphuric acid: 125 per cent of water: 7.5 atmospheres of pressure; 20 minutes cooking period. The results obtained divide the different species into their natural botanical classifications, namely, the coniferous and broad-leaved species. Although not all species in each class were tested, authentic samples of enough species were used to demonstrate the value of most of those woods that are commonly available for this process.

#### CONIFEROUS WOODS.

Per cent of total Alcohol yields. reducing sugars. Total reducing Gallons sugars Cock of 190-Species of wood. (per cent Per cent Gallons proof per No. of origi-Fermen-Unferof origiof absodiy ton, naldiy table. mentable. nal dry lute per allowing wood). dry ton. wood. 5 percent distillation loss. 63 . . . . . White pine, Idaho..... 21.00 74.49 25.517.762 23.4823.43 Red spruce. Douglas fir, Montana..... 74.161 67.42 64..... 20.4825.847.565 22.88 22.841 32.58 6.8227.4378.28220.5922.4624.9020.64 65..... 21.10 75.6773.3272.4922.4825.0566 . . . . . White pine. 20.02 24.33 23.0623.2526.68 67.... Long leaf pine..... 68..... 27.51 25.1621.75Lodgepole pine. 8.330 25.2069..... 70..... 21.93 67.37 32.63 7.205 21.79 Norway pineextra large or small 23.38 ships 25.6266.8833.127.745 23.4271.... 50<sup>1</sup>.... 72.6737.8957.88Red spruce .. 22.06 27.337.956 24.0624.01 Western larch..... 29.7230.5262.114.97715.0515.0383<sup>2</sup>.... 72<sup>3</sup>.....  $42.12 \\ 45.31$  $8.687 \\ 6.934$ 26.2620.97.....do..... 26.2126.21 21.15 54.6977.63 72.55 do. Western hemlock 20.93 86..... 22.37 7.622 23.05 23.01 99..... Sugar pine..... 18.03 27.45 6.276 18.96 18.93 103 . . . . 66.4975.16  $7.115 \\ 7.934$ .....do. . 20.23 33.51 21.51 21.47 . . . . . . . . . . . . . . Douglas fir, Washington 74 . . . . . 21.13 24.84 23.99 23.95 46..... White spruce..... 23.61 71.44 28.56 8.537 25.82 25 78

The results of the experiments on the various coniferous species are given in the following table:

<sup>1</sup> 1.8 per cent of acid; 10 minutes cook. <sup>2</sup> 2.5 per cent of acid; 20 minutes cook. <sup>3</sup> 2.5 per cent of acid; 40 minutes cook.

The experiments on western larch will be considered separately. No great differences were found among the various other species. Those highest in cellulose, like white spruce and white pine, gave the best yields. The West Coast Douglas fir gave higher yields than the Montana mountain-grown fir. Cook No. 70 was on Norway pine chips left from the Yaryan extraction process, in which the turpentine had been steamed out and the rosin had been dissolved out with gasoline. The yield was probably 3 to 5 per cent higher than it would have been on the original wood basis, because of the difference in cellulose content calculated on a basis of freedom from volatile oil and rosin, as compared with the original wood. At the same time, the results of cooks Nos. 67 and 68 on true longleaf pine showed that the turpentine, pine oil, and rosin do not interfere in the production of the sugars from the wood or in the fermentation of the sugar liquors produced. However, sugar liquors produced from woods having appreciable quantities of volatile oils do contain some of the oils mentioned, and, unless particular care is exercised in the distillation and refining of the beers and alcohol made from such liquors, the alcohol finally obtained will contain some of those oils. On the other hand, it is not impossible to remove the oils, as evidenced by the fact that the finest Cologne spirits being produced in this country to-day is manufactured from longleat pine.

Practically all the commercially available coniferous woods of this country are of equal value for alcohol production, and, as the waste from them constitutes the greater portion of the mill waste of the country, the results obtained from the experiments with them are naturally the most interesting. Cypress and the cedars were not tried; and the writer has no data to present regarding them except commercial results obtained from cypress in mixture with longleaf pine. As the exact composition of the mixture is not known, no positive data can be offered; it may, however, be said that cypress seems to give yields only from 60 to 75 per cent as good as pine.

The different results obtained in cooks Nos. 50, 83, and 72 on western larch are due to differences in material as well as in treatment. The differences will be considered in greater detail later. Cook No. 50 was made on material from the butt log, which is usually left in the woods, and with 1.8 per cent acid at 7.5 atmospheres for 10 minutes. Cooks Nos. 72 and 83 were made with 2.5 per cent of acid, the first for 20 minutes and the second for 40 minutes. Unfortunately, all three cooks were not made on material from the same sample, although cooks Nos. 72 and 83 were on the same sample and indicate a tendency toward an increase of fermentable sugars with an increase in the time of cooking. This tendency is probably due to pentose decomposition, as it was with spruce. The high figure for total sugars is due to the production of galactose, which is not fermentable under ordinary conditions, and the figures for fermentable sugars are therefore correspondingly lower than they are in the other coniferous woods.

# BROAD-LEAVED WOODS.

The broad-leaved woods neither give the yields obtained from the coniferous woods, nor do they exhibit the uniformity of yield shown by the coniferous species. The yields of total sugars are sometimes nearly as great as those obtained from the coniferous species; but, as the following table shows, the portion of the total sugars fermentable is very much less than that from the coniferous species:

		Total re- ducing sugars, per cent of origi- nal dry wood.	Per cent of total reducing sugars.		Alcohol yields.		
Cook No.	Species of wood.		Fermen- table.	Unfer- mentable.	Per cent of origi- nal dry wood.	Gallons of abso- lute per dry tons.	Gallons of 190- proof per dry ton allowing 5 per cent distilla- tion loss.
75 76 77 78 79 80	Hard maple. Silver maple Beech. White oak Red oak Svcamore Slippery elm Red gum.	$18.93 \\ 20.74 \\ 21.24 \\ 17.30 \\ 18.38 \\ 18.30$	$\begin{array}{c} 46.29\\ 34.04\\ 47.22\\ 22.22\\ 50.48\\ 30.40\\ 38.86\\ 26.79\\ 38.81\\ 32.86\end{array}$	$\begin{array}{c} 53.\ 71\\ 65.\ 96\\ 52.\ 78\\ 77.\ 78\\ 49.\ 52\\ 69.\ 59\\ 61.\ 14\\ 73.\ 21\\ 61.\ 19\\ 67.\ 14 \end{array}$	$\begin{array}{c} 4.\ 288\\ 3.\ 029\\ 4.\ 661\\ 1.\ 995\\ 4.\ 102\\ 2.\ 675\\ 3.\ 205\\ 1.\ 382\\ 3.\ 658\\ 2.\ 392 \end{array}$	$\begin{array}{c} 12.97\\ 9.16\\ 14.10\\ 6.03\\ 12.40\\ 8.09\\ 9.69\\ 5.99\\ 11.06\\ 7.23\end{array}$	$12.95 \\ 9.14 \\ 14.07 \\ 6.02 \\ 12.38 \\ 8.07 \\ 9.67 \\ 5.98 \\ 11.03 \\ 7.21 \\$

The reason for these marked differences between the evergreens and the deciduous trees must reside in their chemical composition rather than in any physical or structural differences and, no doubt, may be ascribed primarily to differences in cellulose content. As a class, the deciduous trees contain less cellulose and more lignin and wood gums than the coniferous species. The gums, such as xylan and araban, are in part converted into the respective pentose sugars. This accounts for the comparatively high total sugar yields and also for the comparatively small portion of the sugar that is fermentable. Those constituents, lignin and gums, which make the hardwoods as a class, and especially beech, birch, and maple, desirable in destructive distillation, bring about a different result in alcohol production, and it is questionable whether commercial recovery would be possible from any of those species alone that are mentioned in the above table. If mixed in small amounts with coniferous waste, the broad-leaved species will not interfere with the production of sugar and alcohol from the coniferous waste, in spite of the large amounts of acetic and formic acids produced. In fact, it is when the recovery of these acids is considered in conjunction with the alcohol process that hardwood waste utilization appears possible if the waste is available in sufficient quantity.

Ordinary destructive distillation practice recovers 180 pounds of crude acetate of lime per cord of wood. Under good conditions this may be brought up to 200 or 220 pounds per cord.

The following table shows the amounts of acetic and formic acid produced from beech, birch, and maple, the three species commonly distilled:

Cook No.	Species.	Acetic acid (per cent of original dry wood).	Formic acid (per cent of original dry wood).	Total vola- tile acid (per cent of original dry wood).
76	Beech.	4. 800	0.445	5.245
62	Birch.	4. 700	.706	5.406
73	Hard maple.	3. 770	.512	4.282

The amounts of acetic acid produced, as shown in the above table, are equal to or greater than the amounts produced by the destructivedistillation process; and, aside from the technical value of the facts disclosed, additional light is thrown on the difference in chemical composition of those woods and woods of the coniferous species.

# SOURCE OF FERMENTABLE SUGAR.

As previously outlined, the source of the fermentable sugar obtained from the hydrolysis of wood has long been a mooted question. Cellulose and materials higher in cellulose than wood, however, have yielded fermentable sugars and alcohol in proportion to their cellulose content. In order to throw additional light on this subject, and also to ascertain whether another common waste material other than mill waste could be used for the production of alcohol, cook No. 50 was made on western larch.

The logging of western larch shows a woods loss of about 8 per cent, caused by butting off the lower portion of the tree.<sup>46</sup> The presence of shakes in the butt is chiefly responsible for this practice. In addition, the base of the tree is usually swollen. This portion is denser than the rest of the trunk, and usually sinks, thus preventing rafting. The length of the butts left in the woods varies from 4 to 8 feet, although a 16-foot piece is sometimes rejected.

Hitherto the utilization of this waste material has not met with success, and it was hoped that it might profitably be employed as a raw material in the production of alcohol. A sample of sawdust from a butt log was cooked with 1.8 per cent of sulphuric acid, 125 per cent of water, 7.5 atmospheres of pressure, for 10 minutes. A yield of sugars equal to 29.72 per cent and of total solids equivalent to 35.18 per cent of the dry weight of the wood was obtained. Under the same conditions white spruce would yield from 22 to 23 per cent of total sugars of which 60 to 65 per cent would be fermentable, making an alcohol yield of 6.8 to 7 per cent of the dry weight of the wood. The extracts obtained from the hydrolysis of the larch were fermented under standard conditions, the fermentation records and the alcohol yields being shown in the tables in the Appendix.

The larch yielded about 35 per cent more of total sugars than did the spruce, and yet only 37.9 per cent of that sugar fermented as compared with 60 or 65 per cent of the total sugar from spruce. A. W. Schorger, of the Forest Products Laboratory, has analyzed both of these woods with the following results:

;	Western	White spruce (4 samples).			
	larch (base) (per cent).	Range (per cent).	Mean (per cent).		
Soluble in ether	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	0.90 to 1.95 .82 to 1.45 1.88 to 2.52 6.72 to 8.84 11.18 to 13.87 10.04 to 10.78 3.08 to 3.95 51.95 to 58.47 .285 to .326	1. 36 1. 12 2. 14 7. 70 12. 21 10. 39 3. 55 56. 17 . 307		

It will be noticed that the larch contained a large amount of material soluble in water and a proportionately small amount of cel-

<sup>&</sup>lt;sup>45</sup> U. S. Department of Agriculture, Forest Service, Bulletin 122, "The Mechanical Properties of Western Larch," by O. P. M. Goss.

lulose. The chief constituent of this material soluble in water was a galactan that yielded approximately 10 to 12 per cent of the dry weight of the wood of galactose, and this in turn accounts for the high sugar yields from the larch. If the sugar yield of the larch were recalculated, so that it would be proportionate to the cellulose content (22 per cent being assumed as the yield of sugar from spruce), there would then be 16.7 per cent of sugar instead of 29.7 per cent, as actually obtained. However, as noted above, about 10 or 12 per cent of galactose was obtained. If this were subtracted from the total sugar yield of 29.7 per cent, there would remain 18 or 19 per cent of sugar comparable to the yield obtained from spruce. Under normal conditions, with a good fermentation and on the assumption that 60 to 65 per cent of the total sugar would be fermentable, there would be an alcohol yield of 4.8 to 5.2 per cent; whereas the actual alcohol yield obtained from the larch is 4.997 per cent. This corresponds to about 62 per cent of the total sugars fermentable, which is the average of the above figures chosen for spruce. It appears, therefore, that the yield of fermentable sugars and of alcohol is proportionate to the cellulose content of the wood, as suggested by Körner,<sup>47</sup> but disputed by Gallagher and Pearl,<sup>48</sup> irrespective of other materials that may be present in the wood.

Western larch butts will be a good raw material for the production of ethyl alcohol if a yeast is found that will ferment the galactose as well as the dextrose within the time limit and under the other conditions as prescribed by the Bureau of Internal Revenue.

In addition to the evidence regarding the source of the fermentable sugars that was presented in the description of the results obtained from the broad-leaved woods, cook No. 98 was made on the leached residue from cook No. 95, which had been given a chlorine and sulphuric acid treatment. Cooking this residue with 2.5 per cent of sulphuric acid gave only 6.56 per cent of total sugars as compared with the 22 per cent normally obtained from spruce. Through an accident the sugar liquor was lost and no fermentations were made, but the data obtained shows the small yield of sugars obtainable on reinversion, and also indicates that the source of the sugars is the cellulose; for, if the lignin as well as the cellulose were the source, as much sugar would be obtained from the residue as from the original wood, equal weights of material being compared.

# **BY-PRODUCTS.**

From longleaf pine and the other pines of the South, about 1 gallon of crude turpentine per cord, consisting of the higher-boiling fractions of turpentine and pine oil, can be recovered from the blow-

<sup>47</sup> Zeit. für ang. Chemie., 1908, 2353.

<sup>&</sup>lt;sup>46</sup> Proc. Eighth International Congress of Appl. Chemistry, vol. 13, p. 147.

off. The commercial values of the beer-still slops that contain the unfermented pentoses, and of the solid residue from the diffusion battery are being investigated. Recent investigations point to the use of the latter as a stock food. The potential value of both of these by-products is very great, and their investigation is exceedingly interesting both as a chemical and as a commercial question.

# ANALYSIS OF RESULTS.

In a review and summary of the results obtained from the experiments described in this paper, the following points seem to be established for coniferous woods:

1. The temperature and pressure of cooking should not exceed 7.5 atmospheres (112 to 115 pounds per square inch).

2. This temperature and pressure should be reached as soon as technically possible, and the boiler capacity of the plant should be such that this may be accomplished in 15 or 20 minutes.

3. The digester contents should be cooked at the above pressure and temperature for 15 or 20 minutes. This requires only enough steam for radiation.

4. The ratio of water to dry wood should be about 125 parts of water to 100 parts of dry wood.

5. The ratio of sulphuric acid (100 per cent) to dry wood should be from 1.8 to 2.5 parts of acid to 100 parts of dry wood.

6. Under the above conditions a yield of 25 gallons of 190-proof alcohol per dry ton has been obtained, allowance being made for a distillation loss of 2.5 per cent, but no allowance being made for any manufacturing losses.

7. After the cooking, the digester should be blown off as quickly as possible. If the plant is operating on longleaf pine or a similar resinous wood, about 1 gallon of crude turpentine can be recovered per cord. If the plant is operating on other coniferous species, the amount of turpentine in the blow-off will not pay for recovery; and the same thing is true of the volatile acid in all coniferous species. Usually the digester may be blown off directly into the air.

8. Under the above conditions a complete cycle for each digester would be about as follows:

	Minu	ites	s.
Loading	5 to	0	5
Heating	15 to	0 2	20
Cooking			
Blowing-off.	5 t	Э	8
Discharging	5 to	Э	7
=			
Total	45 to	5 6	0

9. Broad-leaved woods produce only about one-half as much fermentable sugars and alcohol as do coniferous woods, although the volatile-acid production from some of the broad-leaved woods is as great as that from the same species by the destructive-distillation process.

10. Sulphuric acid and possibly some sulphates are the best catalysts if cost, technical conditions, and yields are all considered.

11. Chlorides prohibit fermentation, but tannin in the concentrations as ordinarily obtained does not.

# PLANT EQUIPMENT AND OPERATION.

The essential parts of a plant equipped to produce ethyl alcohol from wood, considered in the order of their use, are as follows:

- 1. Adequate sawdust storage.
- 2. Disintegrating equipment-hogs, screens, and shredders.
- 3. Sawdust storage above digesters, and acid storage.
- 4. Digesters.
- 5. Diffusion battery.
- 6. Neutralizing and settling tanks.
- 7. Coolers.
- 8. Fermenters and yeast equipment.<sup>49</sup>
- 9. Beer still.49
- 10. Rectifying still.49
- 11. Bonded warehouse.49
- 12. Boilers and engines.
- 13. Laboratory and office.
- 14. Charcoal rectifiers (desirable, but not absolutely necessary).

### SAWDUST STORAGE.

What constitutes adequate sawdust storage will depend upon the location and the continuity of operation of the sawmill and upon the character of the logging operation. The operation of the alcohol plant and distillery must be continuous. The storage, therefore, must be adequate to make it possible for the plant to comply with the regulations of the Bureau of Internal Revenue governing the operation of distilleries. These alcohol plants are surveyed as to their output and must produce daily the amount required in the survey; if they do not produce that amount, they are penalized with the tax on such a quantity of alcohol as is necessary to make up the amount required by the survey. In general, therefore, the alcohol plant should have at least 15 days' supply of wood on hand; and, if the logging operations require frequent shutdowns, the alcohol plant should always have sufficient material in storage to last twice as long as the average shutdown. The waste may be best stored and handled in the condition in which it is ready for use, that is, hogged and shredded. Protection from the rain is all that is needed, and any type of open-walled, covered building would be suitable. Belt conveyors may be used to handle the material, and

<sup>&</sup>lt;sup>49</sup> These items must be approved and supervised by the U. S. Bureau of Internal Revenue.

a long, open, covered shed with an inclined bottom sloping into a trough, similar to those used for the storage of sugar beets, would answer the purpose; or the material may be windrowed in piles and permitted to hopper itself, the danger of its rotting in the hoppers being obviated by this plan.

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All hoppers, both in the storage building and over the digesters, as well as all other parts of these buildings coming in contact with the hogged and shredded waste, should be either of steel or of heavily creosoted timber construction in order that decay may be avoided. The green shredded wood makes an ideal medium for the cultivation of wood-destroying fungi, and even in exposed places too dry, it would seem, for decay to take place, the writer has found it progressing rapidly wherever there were accumulations of fine stuff.

# DISINTEGRATING EQUIPMENT.

The disintegrating equipment should consist of hogs or chippers, shredders, and screens. A chip one-half an inch long in the direction of the grain will be penetrated thoroughly with acid, but the ease with which the sugar can be leached out is a problem that requires attention. However, as the residual digested sawdust or waste after extraction is ample for power production, and as all exhaust steam from the engine has value for heating and distillation purposes, the extra power required to chip down to a three-sixteenths or onequarter inch chip would not be prohibitive, and the greater efficiency of extraction would probably make the chipping down very desirable. After being screened—for the screenings should be reshredded—the fine stuff should go by belt to the loading bin over the digester.

## SAWDUST AND ACID STORAGE.

The loading bins should be of sufficient size to serve as intermediate storage for the material as it comes from the screen on its way to the digester. Each of the bins should hold several digesterfuls and should be placed over the digester, being tapered down so that the material may flow directly into the digester, according to the arrangement in chemical-pulp plants.

The acid intended for the plant should be in concentrated form, to permit of shipment in tank cars and storage in steel tanks. The concentrated acid should be pumped into a lead-lined tank above the digester and be diluted in order that the dilute acid may flow into the digester along with the sawdust. If rotating digesters are used, no special mixing apparatus will be necessary; at least, no appreciable quantities of uncooked material have ever been found at the Forest Products Laboratory when such digesters were used.

### DIGESTERS.

The digesters should be of the rotating kind, and may be spherical or in the form of a short cylindrical section with dished ends. If they are of the latter type, the diameter should be double the length of the cylindrical section in order that the digester may be filled as nearly full as possible. A number of satisfactory acid-proof linings are now obtainable. During the cooking the mass shrinks in volume and settles, the final volume being only about two-thirds the original volume. There is, therefore, ample room for thorough mixing during the cooking.

The size of the digesters will be governed by the daily capacity of the plant, the heating period, and the time of the complete cycle for each digester. If the heating period is 15 minutes out of a total of 1 hour for each cook, four digesters or multiples of four should be used; whereas if the heating period is 20 minutes out of a total of 1 hour, only three or multiples of three should be used. In this way the steam load on the boilers will be made as uniform as possible. The boiler capacity will be largely determined by this load, since that for power and distillation purposes will usually be constant. In addition, the hogging, shredding, and digester capacity of the plant should be sufficient to give enough digested sawdust in 18 or 20 hours to run the rest of the plant 24 hours, thereby allowing time for breakdowns and repairs.

By the rotation of the digester the cooked sawdust is discharged and falls into a large bin, which receives this material from all the digesters. From this bin it goes by mechanical conveyor to the different cells of the diffusion battery.

## DIFFUSION BATTERY.

Closed cells similar to those used for the extraction of sugar beets or dyewood chips may be used. These should be lined and made acid-resistant like the digesters, and are usually fitted with bronze and copper. The top and bottom should be so arranged that charging and discharging may be readily accomplished. Cells of this type may be obtained from which the extracted material will empty itself when the bottom of the cell is released.

The temperature of the extracting water will rise  $30^{\circ}$  to  $50^{\circ}$  F. during extraction because of the hot dust. In cold weather it would be advisable to warm the water before it is used. If the acid juice comes from the battery with a temperature between  $125^{\circ}$  and  $150^{\circ}$ F., a good extraction will be obtained, and the large volumes of water that would be required to cool hotter juice will not be necessary.

The size and number of cells in the battery and the amount of water in each cell will be governed by the size of the plant and the 54976°-22-Bull 983-5

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size of the material that is to be cooked, as, for instance, sawdust will extract more readily than larger material. As the sugars are readily soluble, only a short extraction period is necessary—that is, from 5 to 10 minutes on each cell—making a total extraction period of 50 to 75 minutes. This period, however, will be governed in part by the length of time that it takes the water to drain through each cell, and this in turn depends on the size of the cell.

The cells should not be too large, else the extracting water will not pass through the material easily, and there will be a tendency to channel. The amount of water used should be such as to make the resulting acid extract 11° to 12° Brix, the proper concentration for fermentation. The Brix will rise another degree on neutralization.

As in laboratory work, so in regular practice, a large number of extractions or washings with small amounts of liquid will give a better extraction or a more thorough washing with a more concentrated extract than will fewer extractions with larger amounts of water for each extraction.

### NEUTRALIZATION AND SETTLING.

After extraction the acid extract is nearly neutralized with lime or a high-grade limestone. For this purpose a magnesia stone is undesirable. For a number of reasons it has been found preferable to cool the acid juice to 100° F. or less before it is neutralized. During neutralization the temperature will rise a few degrees. The extract is then allowed to stand and settle out the sludge of calcium sulphate. As this usually requires from 15 to 18 hours, adequate tank capacity is required.

## COOLERS.

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The clear juice is then drawn off and passed through coolers to reduce its temperature to from 80° to 90° F., after which it goes into the fermenting tanks. The coolers should be of copper, and their size will depend upon the temperature of the water supply available. As the calcium sulphate in the neutral juice will partially crystallize out during the cooling, the coolers should be so designed as to be easily taken apart and cleaned.

# FERMENTATION, DISTILLATION, ETC.

A 96-hour fermentation period is permitted; hence a 4-day fermenter capacity is required. The size of the individual fermenter will be determined largely by the outdoor mean temperature and other local conditions. The other apparatus is the standard distillery equipment in use in grain and molasses distilleries.

#### POWER REQUIREMENTS.

The steam load of the plant will be distributed about as follows:

	Per cent.
Pumps-boiler, fire, general water supply, beer, alcohol	. 20
Digesters	. 30
Hogs and shredders	
General power for driving conveyors, digesters, etc	. 15
Distillation and rectification, (including all exhaust steam not used for heating	ıg
boiler-feed and extraction water)	. 15
	100
	100

A large supply of pure, cool water is also necessary. It should be pure for boiler and extraction purposes, and cool for use in cooling and condensing. The disposal of the beer-still slop requires attention, because it contains a large amount of pentose carbohydrate and dead yeast; the latter of which is highly nitrogenous. However, the slop does not putrefy on standing and will maintain a nearly sterile condition for a long time.

### COSTS.

As outlined before, the commercial production of alcohol by this process, with two exceptions, has not been a success. If such a yield is assumed, however, as that obtained at the Forest Products Laboratory, and if the necessary manufacturing losses are allowed, as for instance, the extraction loss in the sludge of the settled juice, and the distillation and rectification losses (which, combined, should not be over 20 per cent of the total product), a yield of about 20 gallons a dry ton is obtained. Assuming this yield, and a location in which the supply of waste will be uniform and constant for a period of 20 years, and in which plenty of good water may be had, with a supply of sulphuric acid and lime reasonably nearby, the cost of a gallon of 190-proof alcohol from wood in a properly designed and constructed plant having a capacity of 2,500 or 3,000 gallons a day, is estimated to be as follows:

Yeast nutrients	\$0.015 to	\$0.020
Repairs and materials (exclusive of fuel and wood)	.030 to	. 040
Labor	.015 to	. 030
Wood and fuel	. 020 to	. 020
Interest at 7 per cent	. 019 to	. 020
Depreciation at 10 per cent	. 023 to	. 035
Overhead, taxes, etc	.015 to	. 030
Total	. 137 to	. 195

In the above table wood has been valued at 40 cents a cord, 1,800 pounds of dry wood being considered to be a cord. This wood should consist of sawdust and hogged refuse, but should not contain over 10 per cent of bark, as the yield of sugars and alcohol from bark is very low. A large quantity of bark would mean running a large volume of inert material through the alcohol plant at considerable expense and without return. Moreover, the use of most barks would add large quantities of tannin to the solutions to be fermented, and this also is undesirable.

If all waste is disposed of for this purpose, a sawmill could not only net the price of 40 cents a cord mentioned above, but it could also avoid the cost of burning the waste, which, as given before, ranges from 30 to 66 cents a cord. To the sawmill this would mean a net gain practically double the figure at which the waste is sold.

The successful production of ethyl alcohol from sawdust seems to depend upon the proper design, equipment, and management of the plant, rather than upon the improvement of the chemical or fermentological features of the process. The problem involves the quick and efficient handling of large volumes of low-grade material under unusual technical conditions, the perfecting of the necessary acidresisting pieces of apparatus, a study of the experience of the plants that have been built and operated, and the efficient utilization of material whose mere removal is now an expense. This industry unquestionably is worthy the serious study of experimental and practical investigators of the utilization of forest products.

			Total	Per cent of total reducing sugars,	Per cent of total educing sugars,		Alcohol	Alcohol in beer.		IA	Alcohel yields.	.s.	
Spectes of wood.	Fer- menta- tion No.	Cook No.	reducing sugars (per cent of origi- nal dry wood).	Ferment- able.	Nonfer- mentable.	Fermen- tation efficiency.	Actual alcohol yield.	Theo- retical alcohol yield.	Per cent of total sugars obtained as alco- hol.	Per cent of origi- nal dry wood.	Gallons of abso- lute per dry ton.	Gallons of 190- proof per dry ton, allowing 5 per cent distilla- tion loss.	Remarks.
W hite spruce.		33	23, 16 23, 16	54. 57 55. 17	45.43 44.83	83. 20 104. 38	1. 818 1. 452	2, 185 1. 391	45, 40 57, 59	5.374 6.817	16, 255 20, 619	16. 224 20. 581	
				54.87	45.13					6. 096	18, 44	18, 40	Average.
		2222	23. 75 23. 75 23. 75	55, 01 55, 61 54, 44	44, 99 44, 39 35, 56	96.68 101.34 107.70	2.443 2.271 .783	2. 527 2. 241 . 727	53, 18 56, 36 69, 40	6, 455 6, 841 8, 424	$\begin{array}{c} 19.524 \\ 20.692 \\ 25.480 \end{array}$	$\begin{array}{c} 19.\ 488\\ 20.\ 653\\ 25.\ 432\end{array}$	Discard.
-				55.31	44,69					6.648	20, 11	20.07	Average.
		88	23, 55 23, 55	54. 22 55. 09	45.78 44.91	101. 19 94. 37	2.128 2.045	2.103 2.167	54.87 51.99	6.607 6.260	$19,984 \\ 18,935$	19. 946 18. 899	
				54, 66	45.34					6.434	19.46	19, 42	D0.
-		24 24 24	23, 81 23, 81 23, 81	53. 76 52. 45 53. 26	46, 24 47. 55 46, 74	95. 21 93. 73 106. 08	1. 849 1. 778 2. 364	1. 942 1. 897 2. 228	51.18 49.16 56.50	6, 228 5, 982 6, 876	18. 838 18. 094 20. 798	18.802 18.060 20.759	
				53, 16	46.84					6, 362	19. 24	19.21	D0.
-		25 25	25.01 25.01 25.01	54, 93 58, 20 54, 75	45.07 41.80 45.25	97.69 96.97 98.37	2, 115 2, 340 2, 180	2. 165 2. 413 2. 216	53. 66 56. 44 53. 86	6.859 7 210 6.885	20.746 21.808 20.825	20, 707 21, 767 20, 786	
				54.84	45, 16					6.985	21, 13	21.09	D0.
;		26 26 26	22, 24 22, 24 22, 24	58, 18 53, 79 56, 61	. 41.82 46.21 43.39	92, 56 97, 93 98, 77	2. 141 2. 314 2. 334	2.313 2.363 2.363	53, 85 52 67 55, 91	6, 121 5, 987 6, 355	18 514 18 109 19. 222	18.479 18.075 19.186	
			-,	56.19	43.81					6.154	18.61	18, 58	D0.
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TABLE 1.—Sugar and alcohol yields.

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TABLE

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		-	1 - Frank	Per cent of total reducing sugars.	of total sugars.		Alcohol in beer.	n beer.		IV	Alcohol yields.	ls.	
Species of work.	Fer- menta- tion No.	Cook No.	Total reducing sugars (per cent of origi- nal dry wood).	Ferment-	- Nonfer- mentable.	Fermen- tation efficiency.	Actual alcohol yield.	Theo- retical alcohol yield.	Per cent of total sugars obtained as alco- hol.	Per cent of origi- nal dry wood.	Gallons of abso- lute per dry ton.	Gallons of 190- proof per dry ton, allowing 5 per cent distilla- tion loss.	Remarks.
Shortleaf pine		27 27	18, 57 18, 57	56, 21 54, 25	43. 79	91. 74 103. 85	1.811 2.40	1.974 2.311	51.57 56.34	4, 893 5, 858	14. 800 17. 719	14.772	
				55.23	44. 77		-			5, 376	16, 255	16, 228	A verage.
		28	5.34	32.29	67.71	101.11	. 453	. 448	32, 65	168.	2,710	2.690	
		888	16 37 16.37 16.37	56, 52 53, 15 53, 85	43. 48 46. 85 46. 15	84.69 106.30 87.84	1. 494 1. 265 2. 008	1. 764 1. 190 2. 286	47.87 56.50 47.30	4.00 4.727 3.957	12.039 14 208 11.959	12.076 14.271 11.946	
				54.51	45.49					4.226	12.78	12, 76	D0.
White spruce		88	21 09 21 09	60,09 61 26	39.91 38.74	91, 89 104, 98	1.619 2.322	1. 762 2. 213	55, 22 64, 27	5, 952 6, 928	18.003 20.955	17, 969 20, 916	
				60.68	39.32					6.440	19.48	19.44	D0.
		2222	21.45 21.45 21.45	63. 56 63. 76 63. 76 42. 14	36. 44 36. 24 57. 86	101, 58 95, 00 90, 74	2, 189 2, 299 1, 586	$\begin{array}{c} 2.155\\ 2.420\\ 1.590\end{array}$	64. 56 60. 57 42. 03	7.078 6.640 4.096	21.400 20.084 12.389	21.368 20.046 12.366	Discard.
				63, 66	36.34					6, 850	20.75	20.71	Average.
		32222	21.32 21.32 21.32 21.32	69.45 58.97 38.47 70.12	30, 55 41, 03 61, 53 20, 88 20, 88	99. 18 146 77 95. 68 93. 85	2, 169 2, 956 1, 153 2, 016	2, 187 2, 014 1, 205 2, 148	68, 88 86, 55 36, 81 65, 82	7.506 9.431 4.011 7.171	22, 703 28, 526 12, 132 21, 600	22.661 28.472 12.100 21.649	Discard. Do.
				69, 79	30, 21					7 339	22, 20	22.16	Average.
		88 FR	23. 17 23. 17	54, 46 53, 93	45, 54 46, 07	113. 64 103. 57	3 066 2.409	2, 698 2, 326	61. 89 55. 86	7.320 5.615	22 168 20, 008	22. 126 19. 971	
				54.20	45, 80					6.972	21.09	21.05	D0.
	-	-				-	-	-	-				

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Discard.	Average.		D0.		D0.		D0.		D0.		D0.	Discard.	Average.	Discard. Do. Do.	Average.
20. 173 20. 913 17. 414	20.54	20, 119 19, 162	19.64	20, 457 21, 794 21, 151	21.13	18. 522 18. 509 18. 984	18.37	$\begin{array}{c} 13.\ 142\\ 13.\ 380\\ 11.\ 777\end{array}$	12.60	$\frac{18,232}{19,418}$ 18, 633	18.76	$\begin{array}{c} 21.492\\ 13.546\\ 19.077\end{array}$	19.08	$\begin{array}{c} 20.197\\ 19.319\\ 12.052\\ 16.994\\ 16.502 \end{array}$	19.77
$\begin{array}{c} 20.\ 211\\ 20.\ 952\\ 17.\ 446\end{array}$	20.58	20, 157 19, 198	19.68	20, 495 21, 835 21, 191	21.17	18. 557 18. 544 18. 118	18.41	$\begin{array}{c} 13.\ 167\\ 13.\ 405\\ 11.\ 799\end{array}$	12.62	$\frac{16.266}{19.455}$ 18.668	18.80	$\begin{array}{c} 21.533\\ 13.572\\ 19.113\end{array}$	19.11	20. 235 19. 355 12. 075 17. 026 16. 533	19.81
6, 682 6, 927 5, 768	6.805	$6.664 \\ 6.347$	6, 506	6. 776 7. 219 7. 006	7.000	6. 135 6. 131 5. 990	6.085	4. 363 4. 432 3. 901	4.172	6. 039 6. 432 6. 172	6.214	7.1194.4876.319	6.319	6. 690 6. 399 3. 992 5. 466 5. 466	6. 550
59.53 61.72 51.39		60. 14 57. 28		62, 83 66, 94 64, 97		54, 98 54, 95 53, 70		48. 89 49. 78 43. 82		52.82 56.26 53.98		58.67 36.98 52.08		56.69 54.22 33.83 47.70 46.32	·
2, 869 2, 374 2, 338		3. 121 2. 714		2.422 2.180 1.829		3, 094 2, 428 2, 493		$1.502 \\ 1.500 \\ 1.336 \\ 1.336$		2. 550 2. 005 2. 059		$\begin{array}{c} 2.637\\ 1.377\\ 2.559\end{array}$		$\begin{array}{c} 2.\ 436\\ 2.\ 511\\ 1.\ 354\\ 1.\ 987\\ 2.\ 158\end{array}$	
2 887 2. 463 2. 243		3. 348 2. 737		<ul> <li>2.316</li> <li>2.114</li> <li>1.824</li> </ul>		$\begin{array}{c} 3.\ 079\\ 2.\ 403\\ 2.\ 336\end{array}$		$\begin{array}{c} 1.732 \\ 1.673 \\ 1.382 \end{array}$		$2.564 \\ 2.121 \\ 1.983$		$\begin{array}{c} 2.424 \\ 1.501 \\ 2.483 \end{array}$		$\begin{array}{c} 2.\ 415\\ 2.\ 397\\ 1.\ 390\\ 2.\ 008\\ 2.\ 199\end{array}$	
100. 63 103. 75 95. 94		107.27 100.85		95. 62 96. 97 99. 72		99. 52 98. 97 93. 70		$115.31 \\111.53 \\103.44$		$\frac{100.55}{105.79}$		$\begin{array}{c} 91.92\\ 109.00\\ 97.03\end{array}$		$\begin{array}{c} 99.\ 14\\ 95.\ 46\\ 102.\ 66\\ 101.\ 90\\ 101.\ 90\end{array}$	
40, 92 40, 51 46, 44	40.71	$\frac{43}{43}, \frac{94}{20}$	43.57	34, 29 30, 97 34, 85	33. 37	44, 75 44, 48 42, 69	43.97	57.60 55.37 57.64	56.87	47. 47 46. 82 43. 95	46.08	36. 17 66. 07 46. 33	46.33	$\begin{array}{c} 42.82\\ 43.20\\ 67.05\\ 52.80\\ 54.54\end{array}$	43.01
59.08 59.49 53.56	59. 29	56. 06 56. 80	56. 43	65. 71 69. 03 65. 15	66. 63	55, 25 55, 52 57, 31	56.03	42. 40 44. 63 42. 36	43.13	52. 53 53. 18 56. 05	53.92	63. 83 33. 93 53. 67	53.67	57.18 56.80 32.95 47.20 45.46	56.99
21.96 21.96 21.96 21.96		21.68 21.68		21, 10 21, 10 21, 10		21, 83 21, 83 21, 83 21, 83		17. 42 17. 42 17. 42		22.37 22.37 22.37		23. 74 23. 74 23. 74		888888 888888 88888888	<u> </u>
34 34 34 34		35		36 88 36		37 37 37		30 30 30 30 30 30		0, 0, 0, 0, 0, 0,		40 40 40		4444	

TABLE 1.-Sugar and alcohol yields-Continued.

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		Romarks.	Discard. Do. Do.	Average.		Do.		Do.	Diseard.	A verage.
	s.	Gallons of 190- proof per allowing 5 per cent distitla- tion loss.	13, 785 21, 441 19, 841 10, 962 20, 230 21, 541 21, 257 21, 257	20.72	19. 944 19. 865 19. 134 19. 134 19. 134 18. 588 18. 588	19. 23	18, 929 19, 856 20, 970 20, 725	20, 12	20, 354 21, 299 20, 668 11, 044 22, 138	21.11
	Alcohol yields.	Gallons of abso- hite per dry tou.	13. 811 21. 480 19. 878 10. 983 20. 269 21. 581 21. 297	20, 760	19. 981 19. 903 19. 171 19. 171 19. 455 18. 623 18. 448	19.26	18, 965 19, 893 21, 010 20, 765	20, 16	20, 393 21, 339 20, 707 14, 071 22, 180	21.15
	ſV	Per cent of origi- nal dry wood.	4, 566 7, 102 6, 572 8, 531 6, 701 7, 135 7, 135 7, 041	6,862	6, 606 6, 580 6, 580 6, 338 6, 432 6, 157 6, 099	6.369	6. 270 6. 577 6. 946 6. 865	6, 665	6. 742 7. 055 6. 846 4. 652 7. 333	6.994
		Per cent of total sugars obtained as alco- hol.	39, 99 57, 56 58, 56 58, 56 58, 59 61, 67 61, 67		59, 18 57, 18 57, 16 54, 16 54, 16 54, 16		53, 34 55, 95 58, 40 58, 40		57. 93 60. 62 58. 83 39. 97 63. 01	
	Alcohol in beer.	Theo- retical alcohol yreld.	L. 542 2. 650 2. 650 2. 571 2. 571 2. 498		2, 105 2, 278 2, 2195 2, 2195 2, 261 2, 261		2. 764 2. 667 2. 551 2. 543		2, 513 3, 063 2, 866 1, 258 2, 539	
	Alcohol	Actual alcohol yield.	L 533 L 533 L 138 L 138 L 138 2,445 2,445 2,445 2,439		2, 258 2, 278 2, 720 2, 815 2, 207 2, 116		2. 589 2. 752 2. 258 2. 536		2, 319 2, 990 2, 632 1, 217 2, 595 2, 595	
		Fermen- lation efficiency.	99.42 94.38 94.38 94.38 91.50 91.50 97.64		107.27 100.00 97.32 99.86 93.34 93.59		93, 67 103, 19 100, 31 99, 73		92. 28 97. 62 91. 84 96. 74 102. 20	
	of total sugars.	Perment- Nonfer- able, mentable.	59, 78 39, 01 35, 86 35, 86 35, 86 86, 84 86, 84	36.78	44. 83 41. 65 42. 30 40. 90 41. 62	42.06	43. 06 45. 78 41. 09 41. 44	42.84	37. 22 37. 90 35. 94 58. 68 38. 35	37.35
	Per cent of total reducing sugars.	Ferment- able.	40, 22 53, 44 53, 44 60, 99 64, 14 64, 15 64, 58 63, 16	63.22	55, 17 58, 95 58, 34 57, 70 58, 34 58, 34	57.94	56, 94 54, 22 58, 91 58, 56	57, 16	62, 78 62, 10 64, 06 61, 65 61, 65	62.65
	·	Total reducing sugars (per cent of crigi- nal dry wood).	322222222 *******		21.84 21.84 21.84 21.84 21.84 21.84 21.84 21.84 21.84		8888 88888		22.77 22.77 22.77 22.77 22.77 22.77	•
-		Cook No.	24444 244444 244444 244444 244444 244444 244444 244444 2444444		****		44 44 44		45 45 45 45	
		Fer- menta- tion No.								
		Species of wood.	White spruce							

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Discard.	A verage.		D0.		Do.		D0.		D0.		Do.			D0.
$\begin{array}{c c} 26,317\\ 25,191\\ 14,684\\ 25,815\\ \end{array}$	25.77	23, 935 24, 692 24, 946 22, 845	24.10	$\begin{array}{c} 15.967\\ 14.195\\ 16.946\end{array}$	15.70	$13.030 \\ 12.701$	12.87	$15,004 \\ 15,240 \\ 14,829 \\ 1$	15.03	22, 703 22, 884 22, 597 22, 597 22, 021 21, 308	22, 30	25.079 25.345 25.172	25.224 26.087 23.475 24.925	25.04
26.366 25.238 14.712 25.864	25.82	23. 980 24. 739 24. 993 22. 888	24.15	$\begin{array}{c} 15,998\\ 14,222\\ 16,978\end{array}$	15.73	13. 055 12. 725	12.89	$\begin{array}{c} 15,033\\ 15,269\\ 14,857\end{array}$	15.05	22. 746 22. 927 22. 640 22. 062 21. 348	22.34	25.126 25.392 25.220	25, 271 26, 136 23, 520 24, 972	25.09
8. 717 8. 344 4. 864 8. 551	8. 537	7, 928 8, 179 8, 263 7, 567	7.984	5.289 4.702 5.613	5.201	4. 316 4. 207	4, 262	4. 970 5. 048 4. 912	4.977	7.520 7.580 7.485 7.294 7.058	7.387	8. 307 8. 395 8. 338	8.355 8.641 7.776 8.256	8. 295
72, 24 69, 15 40, 31 70, 86		66. 29 68. 39 69. 09 63. 27		60.34 53.65 64.04		60. 24 58. 71		32.34 31.80 33.24	<u> </u>	81, 48 82, 11 81, 09 79, 02 76, 46		75. 41 76. 20 75. 67	75.82 78.42 70.57 74.92	
$2.502 \\ 2.235 \\ .925 \\ 2.225 \\ $		2. 890 2. 716 2. 446 2. 380		$2.620 \\ 2.248 \\ 2.094$		1.844 1.756		$1.539 \\ 1.841 \\ 1.71$		2, 239 2, 239 2, 289 2, 328 2, 328 3,		2.446 2.440 2.412	2, 367 2, 515 2, 143 2, 650	
2. 548 2. 130 1. 089 2. 226		2, 830 2, 713 2, 485 2, 321		2.588 2.070 2.186		1.707 1.514		$1.320 \\ 1.470 \\ 1.578 \\ 1.57$		2, 311 2, 259 2, 259 2, 259 2, 224 2, 224		2. 390 2. 395 2. 382	2.315 2.543 1.968 2.590	
$\begin{array}{c} 101.84\\ 95.30\\ 117.73\\ 100.04 \end{array}$		97.92 99.89 101.59 97.52		98. 78 92. 08 104. 39		$\begin{array}{c} 92.57\\ 86.22 \end{array}$		85. 77 79. 85 92. 23	*	98.95 99.11 99.11 98.34 95.53		97. 73 98. 09 98. 75	97.80 101.11 91.83 97.73	-
29. 07 27. 44 65. 76 29. 17	28.56	32. 30 31. 53 31. 99 35. 12	32.73	$38.91 \\ 41.74 \\ 38.65$	39.73	$\frac{34.92}{31.91}$	33, 41	62.30 60.17 63.96	62.11	17.65 18.59 18.18 18.18 19.96	18.60	22. 84 22. 32 23. 37	22, 47 22, 44 23, 15 23, 34	22.85
70. 93 72. 56 34. 24 70. 83	71.44	67.70 68.47 68.01 64.88	67 27	61.09 58.26 61.35	60.27	65.08 68.09	66. 59	37. 70 39. 83 36. 04	37.89	82.35 81.41 81.82 81.37 80.04	81.40	77. 16 77. 68 76. 63	77. 53 77. 56 76. 85	77.15
23.61 23.61 23.61 23.61		23, 40 23, 40 23, 40 23, 40 40 23, 40 23, 40 23, 40 23, 40 23, 40 23, 40 23, 40 23, 40 23, 40 23, 40 24 24 24 24 24 24 24 24 24 24 24 24 24	<u> </u>	17. 15 17. 15 17. 15		14.02 14.02		29, 72 29, 72 29, 72		18, 06 18, 06 18, 06 18, 06 18, 06 18, 06			21.56 21.56 21.56	
46 46 46 46		47 47 47		48 48 48		49		50 50		55 55 55 55 55 55 55 55 55		56 56 56	20 20 20 20 20 20	
				Shortleaf pine				Western larch		White sprace				

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Remarks. Average. Do. D0. Do. Gallens of 190-proof per dry ton, allowing 5 per cent distilla-tion loss.  $\begin{array}{c} 24.400\\ 25.625\\ 24.904\\ 24.170\\ 24.768\\ 24.768\\ 25.060\end{array}$  $\begin{array}{c} 21.\ 100\\ 18.\ 793\\ 21.\ 109\\ 20.\ 816\\ 20.\ 339\end{array}$  $\begin{array}{c} 20.381\\ 21.538\\ 22.591\\ 21.544\\ 21.544\end{array}$ 21.903 21.190 21.317 22.830 21.661 24.82 21.5121.7843 20. Alcohol yields. Gallons of abso-lute per dry ton.  $\begin{array}{c} 24.446\\ 25.674\\ 25.674\\ 24.951\\ 24.216\\ 24.815\\ 25.108\\ 25.108\\ \end{array}$  $\begin{array}{c} 21.\,140\\ 18.\,829\\ 21.\,149\\ 20.\,855\\ 20.\,377\end{array}$  $\begin{array}{c} 20.420\\ 21.578\\ 22.634\\ 21.584\\ \end{array}$  $\begin{array}{c} 21.944\\ 21.230\\ 21.357\\ 22.872\\ 21.703\end{array}$ 21.8228 47 55 24. 20. 21. Per cent of origi-nal dry wood. 7.214 8. 082 8. 488 8. 249 8. 206 8. 204 8. 301 6.989 6.992 6.992 6.895 6.737 6.768  $\begin{array}{c} 6.751 \\ 7.134 \\ 7.483 \\ 7.136 \\ 7.136 \end{array}$ 2227.126 7.255 7.019 7.061 7.562 7.175 ø Per cent of total sugars obtained as alco-hol. 75.01 78.78 76.52 76.15 76.15 77.05 65.36 69.07 72.44 69.08 67.58 65.38 65.77 70.44 66.83 92 95 12 76 539.  $\begin{array}{c} 2.541 \\ 2.541 \\ 2.393 \\ 2.334 \\ 2.334 \\ 2.329 \\ 2.329 \\ 2.329 \\ \end{array}$ 2. 244 2. 419 2. 435 2. 535 2. 535 2. 575  $\begin{array}{c} 2.071\\ 2.306\\ 2.432\\ 2.824\\ 2.824\end{array}$  $\begin{array}{c} 2.341\\ 2.240\\ 2.191\\ 2.367\\ 2.552\\ \end{array}$ Theo-retucal alcohol yield. Alcohol in beer  $\begin{array}{c} 2.558\\ 2.558\\ 2.543\\ 1.909\\ 2.266\\ 2.274\\ \end{array}$  $\frac{1.843}{2.147}$ 2.382
2.694  $\begin{array}{c} 2.246\\ 2.072\\ 2.469\\ 2.474\\ 2.517\end{array}$ 218 117 421 500 456 Actual alcohol yield. ก่ก่ก่ก่ Fermen-tation efficiency. 00 940 40 98. 99. 95. 95. 92. 95. 95. 93. 95. 97. 93. 93.  $\begin{array}{c} 19.74\\ 21.27\\ 21.00\\ 21.91\\ 21.57\\ 21.09\\ \end{array}$ 21.10 26.5625.8126.0427.5929.5629.32 141.63 32.61 32.24 32.24 30.93Ferment- Nonfer-able. mentable.  $^{38}_{20}$ 5250Per cent of total reducing sugars. 39.939. 39. 26. 73. 44 74. 19 72. 41 69.07 5498238 6 56 95 98 98 98 98 98 98 98 98 98 48 50 72.8.23.80. 78. 6.20.000 60. 73. 520°. Total reducing sugars (per cent of origi-nal dry wood).  $\begin{array}{c} 21.08\\ 21.08\\ 21.08\\ 21.08\\ 21.08\\ 21.08\\ 21.08\\ \end{array}$ \*\*\* 21.005 21.005 21.005 21.005 21.005 2222 222222 88888 57 557 557 557 539 539 999999 Cook No. Fer-menta-tion No. Species of wood. White spruce.

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TABLE 1.-Sugar and alcohol yields-Continued.

	Discard.	D0.	Average.			D0.			D0.			D0.				Do		D0.	
6	21.538 16.390	21.38/	21.46		13.05 12.84	12, 95		24. 51 22. 81 23. 43	23.43	22. 89 22. 93	22.11 23.41	22.84	21.70 20.46	20.24	21.11	20.59	23.03 22.54 21.79	22.46	24.67 24.96 24.00 25.44 24.77
T	21. 578 16. 421	17. 583	21.50		13.08 12.86	12.97		22.25 23.48 23.88 23.48	23.48	22. 93 22. 98	22. 16 23. 46	22.88	21.74	20.28 19.47 20.08	21.15	20.64	23.07 22.58 21.81	22.48	$\begin{array}{c} 24.71\\ 25.00\\ 24.04\\ 25.49\\ 24.81\\ 24.81\end{array}$
	7. 134 5. 429	5.813	7.109		4.323 4.253	4.288	7. 799 7. 761 7. 577	8, 118 7, 557 7, 762	7.702	7.582 7.597	7.325	7.565	7.189 6.776	6, 436 6, 436 6 936	6. 991 6. 720	6. 822	7.627 7.465 7.219	7.437	8. 171 8. 267 7. 949 8. 426 8. 204
	69. 31 52. 74				$\begin{array}{c} 21.06\\ 20.62\end{array}$		37. 14 36, 96 36, 08	- 38.66 35.98 17.87		37.02 37.09	35.77		34.07 32.11	30.50 30.50 32.87	33. 14 31. 85		38. 10 37. 29 26. 06		35.43 35.43 34.47 36.54 29.88
)	2. 431	2. 394 3. 525			$     \begin{array}{c}       1.306 \\       1.067     \end{array}   $		2. 216 2. 522 2. 560	2, 195 2, 306 866		2.884	2.450 3.182		2, 250	2, 630 2, 630 2, 845	2. 555		2. 668 2. 636 2. 822		2.842 2.741 2.855 2.646 3.562
	2. 381	2. 596			1.095		2, 184 2, 450 2, 172	2.244 2.105		2.852 2.908 2.908	2. 284 3. 222		2, 197	2, 338	2.361		2. 769 2. 482 2. 563		2, 599 2, 560 2, 589 2, 599 2, 599 2
	97. 94 69. 28 66. 18	73.64			83. 83 92. 91		98.54 97.14 95.72	102.24 91.26 80.18		98, 90 96, 46	101.27		97.63 95.57	94, 33 90, 58 98, 96	97.16 92.42		$103.77 \\ 94.16 \\ 90.82$		99. 79 94. 81 89. 68 97. 83 74. 12
	29.23 23.88 23.88	23. 32	28.84		50.85 56.57	53.71		25. 51 25. 51	25.51	26. 76 24. 76	24. 95 26. 87	25.84	31.72 34.26	34. 11 34. 11 35. 01	33. 27 32. 58	32.58	28. 17 22. 52 22. 32	24.34	30.53 26.02 24.80 26.92 27.07
	70. 77 76. 12	76.68	71.16		49.15 43.43	46.29		73. 98 77. 14 74. 49	74.49	73. 24	73. 13	74.16	68.28 65.74			67.42	71. 83 77. 48 77. 68	75.67	69.47 73.98 75.20 73.08 72.94
)	20.14 20.14	20.14			20.53 20.53		21.00 21.00 21.00	21.00 21.00		20.48 20.48	20.48 20.48		21.60 21.60	21.60 21.60 21.60	21.60		20.02 20.02 20.02		33.23.08 33.23.08 33.23.08 33.23.08 33.23.23 33.23.28 33.23.28 33.23.28 33.23.28 33.23.28 33.23.28 34.24 34.26 34.24 344
	19 19	01 61		62	222		888 88	888		64 64	64 64		65 65	6.5 5.5 5	65		99 99 99		67 67 67 67
				22	2522		23 23 23 23	888		535	25		523	3 2 2	26		22 24 23		8 52 57 53 58 57 57 53 58 57 57 53 58 57 57 57 57 57 57 57 57 57 57 57 57 57
				Birch			White pine, Idaho.			Red spruce			Douglas fir, Montana				White pine	•	Longleaf pine

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<sup>1</sup> Large chips of white spruce were used in this cook.

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r and alco	
Sugar	
TABLE 1	
R	

	iks.		ge.							
	Rcmarks.		A verage.		D0.		D0.		D0.	
š	Gallons of 190- proof per dry ton, allowing 5 per cent distilla- tion loss.	26, 73 24, 22	24.97	$\begin{array}{c} 24.43\\ 25.50\\ 23.20\\ 25.13\\ 25.13\\ 25.13\\ 24.97\\ 24.97\\ \end{array}$	24.98	22.30 21.79 20.99 21.69 21.99	21.75	20. 75 23. 63 25. 77	23.38	26, 17 24, 35 22, 63 22, 63 23, 67
Alcohol yıelds.	Gallons of abso- lute per dry ton.	26, 78 24, 26	25.05	25,48 25,55 25,55 25,66 25,18 25,19 25,19	25.196	22. 34 21. 84 21. 03 21. 73 22. 03	21.79	20. 79 23. 67 25. 82	23.42	26, 22 24, 40 22, 68 23, 72
ĮĄ	Per cent of origi- nal dry wood.	8, 855 8, 021	8.282	8.424 8.424 8.448 7.683 8.325 8.325 8.325 8.330 8.330	83.30	7.219 7.219 6.953 7.183 7.285	7.205	6.873 7.827 8 536	7.745	8.668 8.067 7.497 7.841
	Per cent of total sugars obtained as alco- hol.	38.40 34.78		36.23 36.34 33.05 35.49 35.49 37.07 31.91		33.68 32.92 31.71 32.42 33.22		26.83 30.55 33.32	30. 23	39.29 36.57 33.98 35.54
Alcohol in bcer.	Theo- retical alcohol yield.	2. 666 2. 567		2.142 2.492 2.816 2.577 2.586 1.357		2, 527 2, 725 2, 397 2, 504		2, 591 2, 638 2, 024		2, 968 2, 671 2, 592 1, 258
Alcohol	Actual alcohol yield.	2.681 2.379		2,126 2,539 2,552 2,552 2,552 2,552 1,283		2, 507 2, 509 2, 219 1, 943 2, 373		2.014 2.421 1.940		<b>2.</b> 949 2. 624 2. 366 2. 073
	Fermon- tation efficiency.	100.57 92.66		99.26 96.13 90.97 98.11 95.60 100.20 94.55		99. 19 95. 75 92. 59 93. 78 94. 77		$\begin{array}{c} 77.\ 74\\ 91.\ 78\\ 95.\ 83\end{array}$		99.35 98.22 91.29 91.80
Per cent of total reducing sugars.	Ferment- Nonfer- able.	25.30 26.50	26.67	28,58 28,92 28,92 27,22 27,35 27,35 27,35	27.49	33. 58 32. 74 33. 01 32. 36 31. 42 31. 42	33, 66	32.48 34.88 31.98	33.10	28.77 27.16 27.16 24.24
Per cent reducing	Ferment- able.	74. 70 73. 50	73.32	71.42 71.08 71.08 72.28 72.39 72.49	72.49	66.42 67.26 67.64 67.64 68.58	67.37	67. 52 65. 12 68. 02	66.88	71. 23 72. 84 72. 84 75. 76
E	Total reducing sugars (per cent of origi- nal dry wood).	23. 06 23. 06		នននននននន នំនំនំនំនំនំនំនំ		21.93 21.93 21.93 21.93 21.93		25,62 25,62 25,62		22,06 22,06 22,06 22,06
	Cook No.	67 67		68 68 68 68 68 68 68 68 68 68 68 68 68 6		55555 5555		70 70 70		EEEE
	Fer- menta- tion No.	27 28		82282828		282288		25 26 27		26 28 29
	Species of wood.	Longleaf pine.				Lodgepole pine		Norway pine, extracted chips		Red spruce

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		D0.		Dc.		D0.		D0.			D0.		D0.
1	21.31 23.95 23.02	24.01	$\begin{array}{c} 20.66\\ 21.27\\ 29.51\\ 22.16\\ 21.56\\ 20.43\end{array}$	20.93	8.85 9.76 9.35 9.35 9.34 9.34	9.14	23, 49 23, 23, 49 23, 23, 23, 23, 23, 23, 23, 23, 23, 23,	23, 95	13. 16 13. 37 14. 41 14. 26	15.17	14.07	6.18 6.65 5.23 5.23	6.02
7	24. 39 24. 00 23. 07	24.06	20.70 21.31 19.55 21.60 21.60 20.47	20.97	9.8.458 9.9.9.8.72 9.8.858 9.866 9.866 9.866 9.878 9.8778 9.878 9.877 9.7777 9.7777 9.7777 9.7777 9.7777 9.7777 9.7777 9.7777 9.77777 9.7777 9.7777 9.7777 9.77777 9.77777 9.77777 9.777777 9.77777 9.77777777	9.16	23, 53 24, 33 24, 33 24, 01 25, 25 25, 25, 25, 25, 25, 25, 25, 25, 25, 25,	23.99	13, 18 13, 40 14, 44 14, 28	15.20	14.10	6.19 6.67 5.25	6.03
	8, 063 7, 934 7, 626	7.956	6.841 7.046 6.342 6.341 7.341 7.143 6.768	6.934	2, 852 2, 933 3, 234 3, 234 3, 095 3, 095	3.029	7, 780 7, 904 8, 904 7, 939 7, 939 7, 888 8, 310 7, 888 7, 888 7, 888 7, 888 7, 888 7, 888 7, 888 7, 888 7, 1988 7, 1986 7, 1988 7, 1988 7, 1988 7, 1988 7, 1988 7, 1988 7, 1988 7, 1988 7, 1988 7, 1986 7, 19867 7, 19867 7, 19867 7, 19867 7, 19867 7, 19867 7, 1986	7.934	4. 358 4. 429 4. 774 4. 722	5.025	4.661	2.045 2.204 1.997 1.734	1.995
	36. 55 35. 97 31. 57		26.11 26.11 24.65 23.01 25.25 25.25 25.82		15.06 15.49 17.09 16.35 16.35		36, 82 37, 41 37, 41 37, 57 36, 38 36, 38 39, 33 37, 27		21.01 21.36 23.02 22.77 22.77	24.23		9.63 10.38 9.40 8.16	
~	2. 816 2. 816 2. 884		2. 153 1. 766 1. 766 1. 764 1. 764 1. 931 1. 931		1.222 1.136 1.214 1.214 1.096 1.096		2,533 2,533 2,510 2,510 2,533 2,734 2,734 2,734 2,734		1.720 1.531 1.723 1.503	1. 136		.713 .912 .610 .668	
	2. 752 2. 786 2. 709	•	2.007 1.724 1.583 1.755 1.911 1.690		$\begin{array}{c} 1.086\\ .988.\\ 1.118\\ 1.103\\ 1.008\\ 1.008\\ .952 \end{array}$		2,521 2,521 2,568 2,568 2,568 2,568 2,568 2,568 2,568 2,568 2,568 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,591 2,592 2,593		1.474 1.450 1.534 1.448 1.448	1.140		. 552 . 709 . 576 . 528	
	98.05 98.94 93.95		93.20 97.06 87.53 99.46 98.80 91.92		88.88 86.94 92.07 91.95 91.94 97.29		97, 59 97, 59 96, 18 96, 18 95, 79 99, 80 94, 77		85.69 94.72 89.06 96.34			81.61 77.78 94.46 78.99	
	27.06 28.87 28.00	27.32	45.19 45.81 45.81 44.89 44.90 44.03 45.04	45.31	66.84 65.13 65.13 65.88 65.88 67.06 67.12	65.95	8,558 8,558 8,566 8,578	24.83	52.02 55.89 49.43 53.76	52.76	52.77	76.91 73.90 80.52 79.78	77.77
	72. 94 71. 12 72. 00	72.67	54.81 55.11 55.11 55.10 53.97 54.96	54.69	33. 16 34. 87 34. 87 34. 12 32. 94 32. 94 32. 88	34.04	73.82 74.72 77.44 71.84 71.84 71.84 77.10 76.95	75.16	47.98 44.11 50.57 46.24	47.24	47.12	23.09 26.10 19.48 20.22	22.22
>	22.06 22.06 22.06		26.21 27.21 26.21 27.21 26.21 27.210	26.21	18,93 18,93 18,93 18,93 18,93 18,93 18,93 18,93		22222222222222222222222222222222222222		20.74 20.74 20.74 20.74			21.24 21.24 21.24 21.24	
	12		222222	72	3333333		47 74 47 74 47 74 47 74 47 74 47 74 74 7		75 75 75 75	75		76 76 76	
	30 31 32		828828	36	2233 <b>2</b> 3222		3233333 33333 33333 33333 33333 33333 3333		3333628	38		32 32 32 32 32 32 32 32 32 32 32 32 32 3	
			Western larch		Hard maple.		Douglas fir, Washington		Silver maple.			Beech.	

TABLE 1.--Sugar and alcohol yields -Continued.

	Remarks.		A verage.		Do		Do <sup>*</sup>		Do.	
ds.	Gallons of 190- proof per dry ton, allowing 5 per cent dishifa- tion less.	555555 555555 555555 55555 55555 55555 5555	12.38	7.81 7.49 8.90	8.07	8, 78 10, 00 9, 47 10, 42 10, 42	9, 67	6.05 6.40 7.06 5.97 4.35	5.08	10.34
Alcohol yields,	Callons of abso- fule per dry fon.	22222222222222222222222222222222222222	12.40	7.85 7.51 8.92	8, 09	8. % 9. 49 10. 9. 49 10. 43 10. 43 10	9.69	6. 06 6. 51 7. 07 7. 30 4. 36	6.99	10.36
IV	Per cont of origi- nal dry wood.	4, 051 4, 017 4, 017 3, 978 4, 038 4, 107	4,102	2, 595 2, 483 2, 948	2.675	2,908 3,311 3,218 3,218 3,452	3.205	2, 161 2, 151 2, 151 1, 978 1, 442	1.382	3. 426 3. 853
	Per cent of total sugars obtained as aleo- hot,	222222222 222222222 22222222		11.12 13.51 16.01		15, 89 18, 09 17, 15 17, 58 18, 86		7994 <u>7</u> × 288828		10.78
A leahol in beer,	Theo- relicut ulcohol yield.	1.666 1.722 1.933 1.788 1.619 1.613		1,082 1,087 1,058		1. 169 1. 131 1. 173 1. 378 1. 378		1.408 1.058 1.058 .912 .912 .709		1.303
Alcohol	A chual alcohol yield.	L. 575 L. 623 L. 713 L. 407		. 971 . 988 1. 059		0.973 1.070 1.039 1.148 1.148		1, 203 872 919 665 635		1. 190 L. 336
	Permen- Lation efficiency.	91, 56 91, 56 82, 55 88, 56 88, 56 88, 56 88, 56 88, 56 88, 56 88, 56 88, 56 88, 56 89, 56 88, 56 89, 56 80, 19 80, 10 80, 10, 10 80, 10, 10, 10 80, 10, 10, 10, 10, 10, 10, 10, 10, 10, 1		89, 73 90, 91 100, 10		83, 26 91, 59 83, 56 91, 69 91, 69		28.23 28.23 28.23 28.23 29.23 29.23 20.23		85.45
Per cent of Lotal reducing sugars.	Nonfer- mentable.	51, 51 50, 53 50, 53 48, 61 48, 61 48, 50	19.61	69, 21 70, 93 68, 65	69, 59	62, 65 62, 58 62, 12 59, 57 59, 57 59, 57 59, 57	61.14	71. 35 73. 79 70. 76 73. 98 76. 14	73.20	61. 59 60. 83
Por cent reducing	Perment. able,	18, 16 19, 17 50, 36 51, 39 51, 11 51, 11	50, 18	30. 79 29. 07 31. 35	30.40	37.35 37.42 37.88 41.30 40.35	38, 86	25, 25 26, 25 26, 25 26, 25 26, 25 26, 25 26, 25 26, 25 27 26, 25 27 26, 25 27 26, 25 27 26 26 27 26 26 26 27 26 26 26 26 26 26 26 26 26 26 26 26 26	20.79	38, 41 39, 17
	Total reducing sugurs (pargent of origi- mal dry wood.	17.30 17.300		18, 38 18, 38 18, 38		888888 888888 8888888 8888888 88888888		555555 888888 888888		20.42
	Cook No:	77 77 77 77		78 78 78		22222		*****		<u>8</u> 8
	For- monta- lion No.	2887382 2887382		222		88288		828328		33
	Species of wood.	While oak.		Red Out.		Syeamore		Slippery elm		ked gum

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	D0.		D0.		D0.		$D_0$		D0.		D0.
11. 86 10. 31	11.03	8.35 6.92 6.66 7.09 7.07	7.21	25, 25 25, 25, 25 25, 25, 25, 25, 25, 25, 25, 25, 25, 25,		22, 16 21, 83 21, 61 22, 52 24, 45	22.51	19.71 18.12 19.10 19.92	19.21	21.98 23.47 23.58	23.01
$11.88 \\ 10.33$	11.06	8.37 6.93 6.68 7.11 7.09	7.23	26, 12 24, 56 25, 57 25, 57 57, 57, 57, 57, 57, 57, 57, 57, 57, 57,	26.26	$\begin{array}{c} 22.20\\ 21.87\\ 21.65\\ 22.57\\ 24.49\\ 24.49\end{array}$	22.55	$\begin{array}{c} 19.74 \\ 18.16 \\ 19.14 \\ 19.96 \end{array}$	19.25	22. 03 23. 52 23. 62	23.05
3. 928 3. 415	3.658	2, 766 2, 292 2, 350 2, 343 2, 343	2, 392	8, 635 8, 121 8, 902 8, 950 9, 859 9, 859 9, 859 8, 950 8, 950 9,	8.687	$\begin{array}{c} 7.\ 338\\ 7.\ 158\\ 7.\ 158\\ 7.\ 460\\ 8.\ 098 \end{array}$	7.457	6. 528 6. 003 6. 328 6. 598	6.364	7.282 7.776 7.810	7.622
19.24 16.72		15.21 12.60 12.14 12.92 12.88		25, 29 26, 61 27, 70 27, 70 70, 70, 70 70, 70, 70, 70, 70, 70, 70, 70, 70, 70,	70.07	33. 19 32. 70 32. 38 33. 74 36. 62		$\begin{array}{c} 29.02\\ 26.69\\ 28.14\\ 38.63\\ 38.63\\ 38.63\\ \end{array}$		34. 43 36. 76 36. 93 28. 02	
1. 439 1. 131		$\begin{array}{c} . 900\\ 1.110\\ 1.660\\ . 970\\ . 994\end{array}$		L 788 L 710 L 710 L 7148 L 714	L, 444	2. 005 2. 948 2. 535 1. 687 1. 687		$\begin{array}{c} 1.903\\ 2.584\\ 2.546\\ 2.688\\ 1.793\\ 1.793\end{array}$		2. 502 2. 935 4. 206	
1. 390 . 956		. 999 . 955 . 888 . 784 . 829		1. 664 1. 714 1. 514 1. 514 1. 743 1. 743 2. 415 2. 415	L, ±10	1. 886 2. 597 2. 533 2. 553 1. 642		$\begin{array}{c} 1.704\\ 2.310\\ 2.385\\ 2.591\\ 2.842\\ 2.842 \end{array}$		2. 175 2. 716 2. 785 2. 785	
96. 56 84. 55		99. 89 86. 00 83. 50 83. 44 83. 44 84 83. 44		93, 05 87, 57 97, 32 90, 74 130, 38 85, 06 85, 06	33. 40	94.08 88.09 97.34 97.35		$\begin{array}{c} 89.54\\ 89.54\\ 93.67\\ 96.39\\ 96.39\\ 158.50\end{array}$		86.95 92.54 92.99 66.22	
61.03 61.30	61.18	70.22 71.33 55.61 68.74 69.79	67.13	40.50 28.59 29.59 29.59 20.59		30.97 27.36 31.17 32.18 26.39	29.63	$ \begin{array}{c} 36.58\\ 41.58\\ 41.22\\ 40.45\end{array} $	39.98	22. 52 22. 27 22. 30	22, 36
38.97 38.70	. 38.81	29.78 28.67 44.39 31.26 30.21	32.86	59, 50 59, 50 58, 64 58, 64 58, 64 57, 52 71, 48 71, 48	57.88	69. 03 72. 64 68. 83 67. 82 67. 82 73. 61	70.38	63, 42 58, 42 58, 78 59, 55	60.04	77. 48 77. 73 77. 70	77.63
20, 42 20, 42	-	18, 19 18, 19 18, 19 18, 19 18, 19 18, 19	)	2222222223 232222223 2322222223 23222222		22, 11 22, 11 22, 11 22, 11 22, 11 22, 11		$\begin{array}{c} 22.49\\ 22.49\\ 22.49\\ 22.49\\ 22.49\\ 22.49\\ \end{array}$		21.15 21.15 21.15 21.15 21.15	
. 18 18		****		• 88888888888	8	20 20 20 20 20 4 4 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2		********		***	
35 36		40 33 37 88 89 38 7		858886444 858886444	5 <del>1</del>	37 37 38 39 41		37 33 40 41		33 39 41 41	
	-	Cottonwood		Western larch		White spruce.		White spruce and bark (25.53 per cent bark).		Western hemlock	

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		-	E 10401	Per cent of total reducing sugars.	of total sugars.		Alcohol in beer.	in beer.		IA	Alcohol yields.	s.	
Species of wood.	Fer- menta- No.	Cook No.	reducing reducing regreation of origi- nal dry wood).	Ferment- able.	Nonfer- mentable.	Fermen- tation efficiency.	Actual alcohol yield.	Theo- retical alcohol yield.	Per cent of total sugars obtained as alco- hol.	Per cent of origi- nal dry wood.	Gallons of abso- lute per dry ton.	Gallons of 190- proof per dry ton, allowing 5 per cent distilla- tion loss.	Remarks.
White spruce and bark. Bark, 8.49 per cent of total.	%8944	877 87 87 87	19.60 19.60 19.60 19.60	$\begin{array}{c} 72.22\\71.47\\66.77\\66.78\\66.78\end{array}$	27. 78 28. 53 33. 23 33. 22	$\begin{array}{c} 95.08\\ 96.90\\ 90.72\\ 90.72\\ 83.59\\ 83.59\end{array}$	$\begin{array}{c} 2.532\\ 2.615\\ 2.407\\ 2.493\\ 1.651 \end{array}$	$\begin{array}{c} 2.663\\ 2.699\\ 1.994\\ 2.762\\ 1.975\end{array}$	35.10 35.40 41.20 28.53 28.53	6.879 6.938 8.075 6.343 5.592	20.81 20.98 24.42 19.19 16.91	20.77 20.94 24.38 19.15 16.88	
				69.47	30.52					6.765	20.46	20.42	Average.
White spruce bark	39 40	88 88 88 88	17.07 17.07	36.00 27.91	64.00 72.09	82.10 118.31	1.231	1.499	15.11 16.88	2. 579 2. 881	7.80 8.71	7.79 8.70	
				31.95	68.04					27:30	8.25	8.24	D0.
White spruce (2.5 per cent HCl)	40 41 42	68 68 68 68	$17.02 \\ 17.02 \\ 17.02 \\ 17.02$	$ \begin{array}{c} 5.10 \\ .95 \\ (2) \\ (2) \end{array} $	$ \begin{array}{c} 94.90 \\ 99.05 \\ (^2) \end{array} $	173. 71 412. 58	.171 .128 .056	.038	4. 53 2. 00	.341	2.33 1.03	2.33 1.03	
		-	_	3.02	96.97					5.56	1.68	1.68	D0.
White spruce (1.8 per cent HCl)	41 42 43	866	$19.69 \\ 19.69 \\ 19.69 \\ 19.69 \\ 19.69 \\ 10.61 \\ 10.6$	$\begin{array}{c} 1.79 \\ (2) \\ (2) \end{array}$	$98.21 \\ (2) \\ (2) \\ (2) $		.147	.080					
Spruce (1.8 per cent 11 <sub>2</sub> SO <sub>4</sub> + Narl= NaHSO <sub>4</sub> )	44 44 45 44 45 44 45 46	855555 85555 85555 8555 8555 8555 8555	20.44 20.44 20.44 20.44 20.44	$\begin{array}{c} 4.06\\ 5.22\\ 9.55\\ 11.22\\ 32.43\\ 32.43\end{array}$	95.94 94.78 94.78 90.45 88.78 67.57	$\begin{array}{c} 504.46\\ 158.40\\ 65.09\\ 70.80\\ 197.10\end{array}$	$\begin{array}{c} 790\\ .223\\ .223\\ .282\\ .282\\ 2.24\\ 2.24\end{array}$	$ \begin{array}{c}     157 \\                                    $	$\binom{3}{4.222}{4.06}{32.67}$	. 863 . 650 . 830 6. 677	$\begin{array}{c} 2.61\\ 1.97\\ 2.51\\ 20.20 \end{array}$	$\begin{array}{c} 2.60\\ 1.96\\ 2.50\\ 20.16\end{array}$	
				12.49	87.50				-	2.255	6.32	6.58	Do.
Spruce (1.8 per cent HCl neut, with CaCO <sub>3</sub> ).	42 44 45	3333	18.25 18.25 18.25	7.78 .105 .50	92. 22 99. 895 99. 50	598.00 257.15	0571	.0096	(3) •66	.120	.36	36	

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			D0.						D0.				D0.			D0.	
	4.63	$3.06 \\ 4.26$	3.66	3.06 4.27 2.91	2.64			$16.04 \\ 19.59$	17.81	18.00 16.80 18.01			17.60		19.67 17.59 19.53	18.93	<sup>6</sup> This fermentation was valueless on account of being chilled.
	4.64	3.06 4.26	3.66	3.07 4.28 2.91	.34 2.65		••••••	16.07 19.62	17.84	18.04 16.84 18.04			17.30		19. 71 17. 63 19. 56	18.96	count of be
	1.532	1.013 1.409	1.211	1.014 1.416 .963	.113 .511 .511 8.032			5.314 6.488	5.901	$5.963 \\ 5.966 \\ 5.966 \\ 6.40 $	$6.34 \\ 7.075$	1.182	3.855		6.516 5.835 6.470	6.276	eless on ac
	7.882	$5.21 \\ 7.25$		5.176 7.227 4.92				26.12 31.90	-	27.80 25.95 27.81 29.80	29.54 32.90	55.00			36.62 36.14 32.35 35.90		n was value
	. 843	.481 331		.130 .177 .143	.044 .1858 2.262			2. 748 2. 624		2.432 1.939 2.292 2.470	2.590 2.221	1.881			$\begin{array}{c} 2.132\\ 2.549\\ 2.317\\ 2.300\end{array}$		rmentation
	. 522	.331		247 334 228	.0284 .123 .067			2.145 2.356		$\begin{array}{c} 2.182\\ 2.029\\ 1.890\\ 2.199\\ \end{array}$	2. 185 2. 203	1.646			2. 472 2. 482 1. 989 2. 268		<sup>5</sup> This fer
	61.92			$\frac{190.56}{188.33}$ 159.03	64.39 66.24 2.962			1, 849. 40 78. 05 89. 78		89.70 104.62 82.46 88.88	84.35 99.75 965 00	87.75			115, 94 97, 36 86, 00 98, 75		Ŀ.
	75.09	95.18 89.55	87.86	94.69 92.49 63.95	98.25 92.18 55.68	82.87		99.69 34.51 30.48	54.89	39.37 51.47 34.01 34.29	91. 00 31. 18 35. 48 60 66	32.75	46.58		27.38 26.25 28.70	27.45	4 More sugar in beer.
	24.91	$14.82 \\ 10.45$	12.63	5.31 7.51 6.05	$   \begin{array}{c}     1.75 \\     7.82 \\     44.30   \end{array} $	12.12 (†)	(1)	.31 65.49 69.52	45.10	60.63 65.99 65.71 65.71	68.82 64.52 24.52	69.25	62.64		72.62 73.75 71.30	72.55	4 More St
18.25 18.25	19.44	19.44	<u> </u>	19.59 19.59 19.59	19.59 19.59 19.59			$120.34 \\ 20.34 \\ 120.34$		21.45 21.45 21.45 21.45 21.45	21.55	21.45		6.56	18, 03 18, 03 18, 03 18, 03 18, 03		0.
83 83	94 04	94 94		95 95 95	95 95 95	992 922	CA	96 96 96	-	66666	6666	26		86	66666		<sup>3</sup> Over 100.
46 55	42	44		42 44 43	45 53 53 53	54	66	43 44 46		44 46 46 46 46 46 46 46 46 46 46 46 46 4	812 82	242			5 45 49 50		
	Spruce (1.8 per cent H <sub>2</sub> SO <sub>4</sub> +NaCl=Na <sub>2</sub> SO cn nont with CoCl.)	4976		$\sum_{i=1}^{2} Spruce(2 \text{ per cent } Cl_2KClO_3 + H_2SO_4 + 10HCl = K_2SO_4 + 6Cl_2 + 6H_2O).$	Sull.	983-				Spruce (1.8 per cent $H_2SO_4+\frac{1}{2}$ KCIO <sub>3</sub> to make 2 KCIO <sub>3</sub> +H <sub>2</sub> SO <sub>4</sub> =HCIO <sub>3</sub> ).				Residue from (cook No. $95+2.5$ per cent H <sub>2</sub> SO <sub>4</sub> ).	Sugar pine (Green 53.06 and $H_2O$ 2.5 per cent $H_2SO_4$ 150 per cent $H_2O$ ).		<sup>3</sup> Final sugar higher than original.

ed.
-Continu
ol yields-
<i>id</i> alcoho
1.—Sugar and
TABLE

													Statement Statement Statements
				Per cent of total reducing sugars.	Per cent of total reducing sugars.		Alcohol in beer	in beer.		W	Alcohol yields.	ß.	
Species of wood.	Fer- menta- tion No.	Cook No.	Total reducing sugars (per cent of origi- nal dry wood).	Ferment-	Nonfer- mentable.	Fermen- tation efficioncy.	Actual alcohol yiold.	Theo- retical alcohol yield.	Per cent of total sugars obtained as alco- hol.	Per cent of origi- inal dry wood.	Gallons of abso- lute per dry tou.	Gallons of 190- proof per dry ton, allowing distilla- tion loss.	Remarks.
Spruce (1.8 per cent $H_2O_4$ , 0.1 per cent $H_4$ , $PO_{4_1}$ 125 per cent $H_2O_3$ .	645 51 52 52 52	00011000 10001000100010001000100001000	21.12 21.12 21.12 21.12 21.12 21.12	$\begin{array}{c} 57.93\\ 57.93\\ 68.10\\ 69.45\\ 69.10\\ 51.40\\ 66.15\end{array}$	42.07 31.90 38.00 33.85 33.85	$\begin{array}{c} 118.55\\92.34\\106.08\\177.4\\98.00\end{array}$	2.785 2.44 2.869 2.537 2.482 2.482	$\begin{array}{c} 2.349\\ 2.642\\ 2.642\\ 2.590\\ 1.430\\ 2.508\end{array}$	35.10 32.14 32.65 37.31. 37.31. 33.38	7. 413 6. 788 6. 788 7. 835 9. 850 7. 085	22, 42 20, 53 22, 14 22, 14 22, 14 22, 14 21, 43	22, 38 20, 49 22, 09 22, 09 21, 38 21, 38 21, 38	
Spruce (0.9 per cent HaSO, 0.9 per cent	55 54 50 50 50 50 50 50 50 50 50 50 50 50 50	100 101 101	21.12 21.12 20.83 20.83	63. 50 69. 00 64. 32 61. 11	36.50 31.00 44.10 38.89	112. 7 102. 80 96. 50 78. 98	2, 366 2, 383 2, 551 2, 551 2, 551 2, 551	2, 990 2, 320 2, 643 1, 870	37.58 36.18 30.10 19.14	7.900 7.620 7.726 6.280	22.89 23.04 23.24 18.99	31. 20 22. 97 24. 23 18. 95	Average.
13F ( 0).	2222222	101 101 101		57.80 58.70 55.35 56.10 42.25	42. 20 41. 30 44. 75 57. 75 57. 75	90.35 100.5 111.95 164.8	2.221 2.241 3.3.197 3.478 3.478	2.144 2.144 2.730 2.860 2.109	32.00 32.000 32.000 32.000 32.000 32.000 32.0000 32.0000 32.0000 32.0000 32.00000 32.00000 32.0000000000	5.53 6.256 6.650 6.660 7.413 7.413	$\begin{array}{c} 16.72\\ 18.92\\ 20.01\\ 22.42\\ 22.42\\ 19.65\end{array}$	16.69 18.88 20.07 20.10 22.38 19.62	Do.
Spruee (1.8 per cont H <sub>4</sub> PO <sub>4</sub> )	49 51 53 53 54	$102 \\ 102 $	19.38 19.38 19.38 19.38 19.38	53, 20 51, 70 56, 65 56, 65 50, 05 50, 05 50, 05 50, 05	41. 13 46. 25 48. 30 48. 30 53. 48. 30 49. 95 49. 95 49. 15	$\begin{array}{c} 9.175\\ 9.175\\ 104.0\\ 86.7\\ 122.3\\ 104.0\\ 104.0\\ 104.9\end{array}$	2. 286 2. 355 2. 355 2. 545 2. 676	<b>2</b> , 498 <b>2</b> , 280 <b>2</b> , 710 <b>2</b> , 548 <b>2</b> , 548	$\begin{array}{c} 25.10\\ 27.40\\ 28.14\\ 28.65\\ 27.28\\ 27.28\\ 27.28\\ \end{array}$	5. 29 5. 15 5. 15 5. 29	14.73 16.09 14.70 17.05 15.57 16.00	14. 70 16. 06 14. 67 114. 67 114. 67 115. 54 15. 97	5
Sugar pino (18 per cont H <sub>2</sub> SO <sub>4</sub> )	49 51 52	103 103 103 103	20.23 (e)	51.58 72.60 70.85 51.35	44.74 27.40 79.15 48.65	$\begin{array}{c} 96.7\\ 84.0\\ 96.3\\ 136.8\end{array}$	2, 255 2, 521 2, 306 2, 306	2, 333 3, 001 2, 082 1, 688	35, 81 27, 45 34, 78 35, 80	5.18 7.244 7.036 7.242	15.69 21.91 21.28 21.90	15.66 21.86 21.24 21.86	D0.

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	D0.				D0.	ĸ	D0.		
21.55 20.94 21.40	21.47	20.86 20.68	23. 89	$\begin{array}{c} 21.17\\ 20.54 \end{array}$	21.42	$\begin{array}{c} 17.70\\ 16.00 \end{array}$	16.85	$\begin{array}{c} 14.79\\17.40\\12.74\\16.67\\15.34\end{array}$	
21.59 20.98 21.44	21. 51	20.92 20.82		$\frac{21.21}{20.58}$	20.97	$17.73 \\ 16.03$	16.89	14.51 17.43 12.86 16.70 15.38	
7. 139 6. 939 7. 091	7.115	6.910	7.052	7.015 6.805	6.927	5.865 5.300	5.583	4. 800 5. 765 5. 522 5. 077	
35. 24 34. 30 35. 05		32. 25 32. 08	33.00 31.95	25.66 32.79 31.81		33.00 29.50		25.00 30.00 21.92 28.70 1.64	
2. 413 2. 558 2. 945		2.778 1.665	2.586 3.272	2.400 2.672		1.615 1.500		2. 500 2. 290 2. 842 2. 482	
2. 416 2. 402 3. 195		2.802 3.107	2. 630 2. 641	2.179 2.916 2.674		1.699 1.677		2. 166 2. 365 2. 115 2. 330 4. 585	
$ \begin{array}{c} 100.1 \\ 94.1 \\ 108.3 \\ 108.3 \end{array} $		101.00 186.7	101.50 80.75	90.85 100.0		$\begin{array}{c} 105.20\\ 111.8 \end{array}$		86.80 103.4 70.95 94.00	
30, 80 36, 55 36, 50	33.51	37.32	36.28	35.85 37.65	- 36.77	38.50 41.24	39.87	40. 20 40. 82	
69.20 71.45 63.50	66.49	62.68	63.72	64. 15 62. 35	63.23	61.50 58.76	60.13	59.50 56.84 60.50 59.80 59.18	
<b>()</b>		21.40 21.40	21.40	21.40 21.40 21.40		17.93 (7)			
103 103		104	104	104		107 107		$108 \\ 108 $	
53		49 50	353	2.57.52		51		55 53 53 57 55 57 57 57 55 57 57 57 57 57 57 57 57 57 57 57 57 57 57 57 57 5	-
		Spruce (1.8) per cent H <sub>2</sub> SO <sub>4</sub> , 0.10 per cent	T 60 04).			Spruce		Spruce (0.9 per cent II <sub>3</sub> SO <sub>4</sub> , 0.9 per cent niter cake).	

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 $^6$  Recalculate alcohol yields.  $^7$  Nither cake with  $\rm H_2SO_4$  equivalent of 18 per cent used to 19.25 make 1.8  $\rm H_2SO_4$ 

Cook.	Acetic acid (per cent of	Formic acid (per cent of	Ratio of acetic acid to formic	Per cent of tile acid in	tota. vola- n blow-off.	Remarks. •
	origin <b>a</b> l dry wood)	original dry wood).	acid.	Acetic.	Formic.	
$\frac{21}{22}$	1. 52	0.239	6.36 to 1	9.24	9.00	Data not complete.
23	2.27	. 540	4.20 to 1	7.54	8.70	Blow-off open.
24.	2.39	. 059	40. 40 to 1			Do.
25	1.77	. 337	5.25 to 1			200
26	1.77 1.75	. 431	4.07 to 1	5. 22	9.95	Do.
27	1.29	. 171	7.55 to 1			Data not complete.
28	1.00		A 16 to 1	5.78 11.70		
29. 30.	1,29 1,38	.310 .642	4.16 to 1 2.15 to 1	0.78 11.70	$\begin{array}{r} 4.94 \\ 12.27 \end{array}$	
31	1.755	. 572	3.05 to 1	8.53	14.23	-
32	1.67	1.090	1.53 to 1	11.48	10.64	
33	1.457	. 355	4.10 to 1	12.65	6.01	
34	2.11	. 480	4.40 to 1	$12.65 \\ 7.91$	24.55	
35	1.05	. 443	2.37 to 1	16.30	4.49	
36	2.43	.735	3.31 to 1	6.80	16.28	
37	1.43	. 253	5.65 to 1	10.75	3.82	
38 39	. 87 1. 33	.430	2.02 to 1 23.75 to 1	7.48	. 47	Blow-off open.
40	1, 56	. 470	3.32 to 1	11.68	8.14	влом-он орен.
41	1, 416	. 430	3.30 to 1	8.24	13. 50	
42	1.55	. 570	2.72 to 1	12.25	12.80	
43	1.51	. 160	9.44 to 1	11.80	23.40	
44	1.69	. 290	5.82 to 1	8, 80	9.40	
45 46	1.67 2.36	. 790 1. 450	2.11 to 1 1.63 to 1	9.48 9.45	8.26	-
47	2.62	. 340	7.71 to 1	9.45	9.75 12.32	
48	2.97	460	6.33 to 1	5. 19	3. 55	
49	1.97	. 230	8.57 to 1	7.59	18,48	
50	1.715	. 254	6.75 to 1	5.26	1.74	
51						Data not complete.
52 53						Do. Do.
54						Do.
55	2.14	. 440	4.86 to 1	13.01	23.18	
56	2.915	1.492	1.95 to 1	7.32	3.88	
57	3, 53	. 659	5.36 to 1	7.69	14.79	
58 59	2.47 2.16	. 399 . 869	6. 19 to 1 2. 49 to 1	7.84 10.23	6.28 8.91	
60		. 809	2.49101	10.25	8. 91	Do.
61		. 933	3.58 to 1	7.39	5.45	201
62	4.70	. 706	6.66 to 1	15.53	4,09	Birch.
63	2.96	. 846	3. 50 to 1	8.09	11.82	White pine, Idaho.
64	2.70 2.24	1.08	2.50 to 1	9.06	5.93	Red spruce. Douglas fir, Montana.
65 66	2.24 2.68	1.42 .626	1.58 to 1 4.28 to 1	6.45 14.87	7.75 29.75	White pine.
67		1. 44	1. 80 to 1	8. 53	6.95	Longleaf pine.
68		1, 18	2.09 to 1	18, 11	7.38	Do.
69	2.84	. 447	7.34 to 1	6.76	3.25	Lodgepole pine.
70		1.19	1.48 to 1	7.53	2.32	Norway pine.
71	2.75	1.18	2.33 to 1	8.92	6. 59	Red spruce.
72 73	2. 84 3. 77	1.46 .512	1.945 to 1 7.36 to 1	7.72 12.52	9.31 4.26	Western larch. Hard maple.
74		. 962	2. 28 to 1	7.92	4.20	Douglas fir, Washington.
75		. 079	13. 80 to 1	49.7	20. 3	Silver maple.
76	4.80	. 445	10.75 to 1	15.65	2. 51	Beech.
77	2.57	. 789	3.26 to 1	2.84	1.77	White oak.
78	3.98	. 180	22.1 to 1	11.55	10.9	Red oak.
79	5.65	.634	8.91 to 1	10.48	4.09 9.42	Sycamore.
80 81	4.04 4.18	.738	5. 48 to 1 5. 97 to 1	$11.02 \\ 7.66$	9.42 6.30	Slippery elm. Red gum.
82	2, 82	.406	6.94 to 1	7.79	26,90	Cottonwood.
83	1.90	. 667	2,83 to 1	14.24	6.62	White spruce.
86	2.14	1.59	1.35 to 1	9.63	7.14	Western hemlock.
	1					

TABLE 2.—Acid yields.

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I	r	.(Jdgi9w	818 818 818 818 819 814 811 811 811 811 811 811 811 811 811
	t pà	Alcohol (per cen	
	ent by	o tego (tego construction). (transferring).	
Ī		Acidity.	0 0 0 0 0 0 0 0 0 0 0 0 0 0
		Sp. gr. at 15° C.	$\begin{array}{c} 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 \\ 1 $
Beer.	(grams)	Reducing sugars per liter).	222 222 222 222 222 222 222 222 222 22
	m.	Hour.	$\begin{array}{c} a. & a. \\ a. & a. \\ b. & a. \\ b. & a. \\ b. & b. & b. \\ b. & b. & b. \\ b. & b. &$
	Distillation.	.93£G.	Sept. 18, 1914 10. 7 Oct. 19, 1914 10. 7 Oct. 19, 1914 10. 7 Mar. 23, 1915 1914 10. 7 Mar. 23, 1915 1914 10. 7 Sept. 18, 1914 10. 7 Sept. 1914
		I COD HA DOL VILG	000000100000104010000000000000000
	:(022	Brix fourth day.	00000000000000000000000000000000000000
-	(500	Attenuation (degree	66146444444444446441984984486666886
ase it		Fifth day.	
lecre:		Fourth day.	
brix	First day. Second day. Fourth day. Futh day. Futh day.		° +
r ree	Second day.		
Bris	First day.		สุญ่ ,4สุมุมุญสุมุญสุมุญสุมุญสุมุญญ.104 สุญี ,4มุมุญสุมุญสุมุญสุมุญสุมุญญ.104 พลัญพลั–0%ลิ–ยั%พยุ–ยั%ญ.255%
ion.		Brix.	1.1.0.1.1.1.0.1.0.2.0.2.0.1.0.1.0.1.0.1.
nentat	.(Jne	Total solids (per ce	
fern		Acidity.	ଷ୍ଣ୍ୟୁୟୁଷ୍ଣ ଷୁଷ୍ଟୁ କୁକ୍କୁକ୍କୁ କୁକ୍କୁ କୁକ୍କୁ କୁକ୍କୁକ୍କୁ କୁକ୍କୁକ୍
Mash before fermentation	emerz)	Reducing sugars. per liter).	88888428488888888888888888888888888888
Mash		Sp. gr. at 15° C.	1, 1, 0, 1, 1, 0, 1, 1, 1, 0, 1, 1, 1, 0, 1, 1, 1, 0, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1,
Suibi	ing. s, inclu	nitnsmtsi stuoH Uft to smit	Comparing the state of the
		Hours filling ferme	aaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaaa
et.		. •JuoH	
Fermenter set			$\begin{array}{c} 1914 \\ 1915 \\ 1916 \\ 19$
Ferm		Date.	Sept. 13, Sept. 14, Sept.
	ŝt.	.VibibA	
	r yea	Alcohol (per cent by weight).	884 884 884 884 884 884 884 884 884 884
Yeast.	96-hour yea:	Sp. gr. at 15° C.	1         0208         5.6           1         0208         5.7           1         0208         5.7           1         0208         5.7           1         0208         5.7           1         0208         5.7           1         0208         5.7           1         0208         5.7           1         0235         5.7           1         0235         5.7           1         0236         5.7           1         0236         5.7           1         0236         5.7           1         0236         5.7           1         0223         5.7           1         0223         5.7           1         0223         5.7           1         0223         5.7           1         0223         5.7           1         0223         5.7           1         0223         5.7           1         0220         5.7           1         0220         5.7           1         0220         5.7           1         0220         5.7 <t< td=""></t<>
		Acidity.	
	Seed yeast.	Brix.	14.7
		Cook No.	22222222222222222222222222222222222222
		Fermentation No.	

TABLE 3.—Fermentation record.

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		Yd Ju	Alcohol (per ce weight).	2003 2014 2015 2015 2015 2015 2015 2015 2015 2015
		the test	Total solids (per 0. ()tdgi9W	
		·	Acidity.	00000000000000000000000000000000000000
			Sp. gr. at 15° C.	(037) (037) (1022) (102
	Beer.	emary)	Reducing sugars per liter).	\$
-		'n.	.100H	
		Distillation.	.93£G.	8. 9 Sept. 29, 1914 8. 4 Sept. 29, 1914 8. 5 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 7 Feb. 10, 1915 8. 6 Sept. 29, 1914 7. 7 Sept. 29, 1914 9. 9 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 8 Sept. 29, 1914 9. 9 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 8 Sept. 29, 1914 8. 9 Sept. 29, 1914 9. 1000 8. 1004 9. 1004
			Brix fourth day.	
		+(000		001-04000001-010000000000400000 001-0100
	in	(500	Fifth day. Attenuation (degr	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
ned	rease		Fourth day.	000000000000000000000000000000000000000
ntin	ix).		Third day.	0 000400004040000000000000000000000000
	Brix record (decrease in Brix).		Зесонд дау.	11111111111111111111111111111111111111
ecord	Brix	Brix. First day.		ぷぷぷぷ すぷぷぷ すきえ すぷええ すぷぷぷ れぷぷぷぷぷぷぷぷぷぷぷぷ すらしょうしょう しょうちょう ちゅうしょう ちゅうしょう ちゅうしょう ちゅうしょう ちゅうしょう ちゅうしょう しょうしょう しょう
i no	n.			484888811988989191411811818818886891989 881-98888888891914118118118118118118118118188 881-98888888888
Fermentation record—Continued	entatio	•(juə	Total solids (per e	
erm	ermo		Acidity.	ほびょよほぼよいようののの、10000000000000000000000000000000000
3.—	Mash before fermentation.	emers)	Reducing sugars.	5888.28288.28288.2848.2888.28288.28288.28288.28288.28288.28288.28288.28288.28288.282888.282888.282888.2828888.2828888.282888888
TABLE	Mash		Sp. gr. at 15° C.	0.0598 1 0.0531 1 0.0531 1 0.0532 0 0.0532 0 0.0
$\mathbf{T}_{I}$	Suibi	illing.	tidnsmisl sinoH i do smid	
			Hours faillft anoH	
- 1		1	.TuoH	\$m0m01m0mm0m0m0100100000000000000000000
	er set			
	Fermenter set.		Date.	Sept. 24, 1914 Sept. 24, 1914 Sept. 27, 1914 Occt. 7, 1914 Sept. 24, 1914 Sept. 24, 1914 Sept. 24, 1914 Sept. 24, 1914 Sept. 24, 1915 Sept. 24, 1915 Sept. 24, 1915 Sept. 24, 1915 Sept. 24, 1915 Nov. 11, 1914 Nov. 11, 1914
			Acidity.	00000000000000000000000000000000000000
		yeas	by weight).	6.5.331 6.6.5.337 6.7.37 6.
	Yeast.	96-hour yeast.	Sp. gr. at 15° C. Alcohol (per cent	1.1.0206         5.807           1.1.0206         5.807           1.1.0206         5.807           1.0205         5.807           1.0205         5.807           1.0205         5.807           1.0205         5.807           1.0205         5.807           1.0205         5.807           1.0205         5.807           1.0206         5.807
	Ye			
		Seed ycast.	Acidity.	7 7 7 7 7 7
		- A	Brix.	88888888888888888888888888888888888888
			Cook No.	<u>୰୴୰୳୴ୠ୰୴୷୶୷୶୷୶୷୶୷୶୷୶୷ଡ଼ଡ଼</u> ୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷୷
			Fermentation No	

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5.5         6.5           5.5         5.5           5.5
$ \begin{array}{c} 888 \\ 5.6 \\ 0.1 \\ 0.3 $
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $
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$ \begin{array}{c} 888 \\ 5.6 \\ 0.1 \\ 0.3 $
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		Td Just	Total solids (per e. .(inglew	6. 5 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
	Beor.		Acidity.	22222222222222222222222222222222222222
-			Sp. gr. at 15° C.	(1, 10, 10, 10, 10, 10, 10, 10, 10, 10, 1
		emary)	Reducing sugars per liter).	22, 23, 25, 25, 25, 25, 25, 25, 25, 25, 25, 25
		÷	.moH	6, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1,
		Distillation.	Date.	May 11, 1915 May 18, 1915 May 18, 1915 May 18, 1915 May 11, 1915 May 1915 May 2, 1915 Nov. 21, 1915 Nov. 21, 1915 Nov. 21, 1915 Nov. 42, 1915 Nov. 43, 1915 Nov. 44, 1915 Nov. 45, 1915 Nov. 46, 1915 Nov. 46
			Briz fourth day.	0         0 <th0< th=""> <th0< th=""> <th0< th=""> <th0< th=""></th0<></th0<></th0<></th0<>
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	i.		Fifth day.	
nued	crense		Fourth day.	* + + + * * * *
Permentation record—Continued	Brix record (docrease in Brix).		Third day.	0 
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reco.	Bri		First day.	いちょう あんえん なんかん かんしょう ひょうしょう ひょうしょう ひょうしょう ひょうしょう ひょうしょう ひょうしょう しょうしょう しょうしょう しょうしょう しょうしょう しょうしょう しょうしょう
tion			Brix.	12222200000000000000000000000000000000
venta	ontali	.(tne	Total solids (per co	3.3         10,996           3.3         10,996           3.4         10,538           3.5         10,538           3.5         10,538           3.5         10,538           3.5         10,538           3.6         11,138           3.7         8.838           4.4         10,1538           5.5         9,467           5.5         9,467           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         9,470           5.5         10,475           5.5         10,48           5.5         10,48           5.5         10,48           5.5         10,48           5.5         10,48           5.5         10,48           5.5         10,48
ern	orm		Acidity.	さごろうなるないれたかかかんごしろうにいれたいいますのないか。
	Mash before fermentation.	emers)	Reducing sugars per liter).	821822838888888888888888888888888888888
Тавие З	Mash		Sp. gr. at 15° C.	0.0108 0.017 0.017 0.017 0.017 0.017 0.017 0.0170 0.01190 0.01190 0.01190 0.01190 0.010000000000
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			Acidity.	
		r year	Alcohol (per cent by weight).	000 000 000 000 000 000 000 000 000 00
	Yoast.	96-hour yeast.	Sp.gr.at 15°C.	L 0210 6, 000 22, 3 May 1, 10 2210 6, 000 22, 3 May 1, 10 22215, 5411 6, 55 May 1, 10 2225, 55 H 16, 5 May 1, 10 2235, 55 H 16, 5 May 1, 10 2245, 53 May 1, 10 10 2245, 50 May 1, 10 10 10 10 10 10 10 10 10 10 10 10 10
		st.	Acidity:	20002222222 22222222222 22222222222222
		Seed yeast.	Brix.	558114 558129 558129 558129 558129 558129 558129 558129 558129 558129 558129 558129 568129 56112 561120 521100 52100 52100 52100 52100 52100 52100 52100 52100 52100 52100 52100 52100 52000 52000 52000 52000 52000 52000 52000 50000 50000 50000 50000 500000000
			Cook No.	**************************************
			Fermentation No.	

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		Alcohol (per cent by weight).	$\begin{array}{c} 1.1 \\ 1.2 \\$
		Total solids (per cent by weight).	%F\$
		Acidity.	000000000000000000000000000000000000000
		Sp. gr. at 15° C.	33         10         0338         10           33         1         0321         0323         10           75         1         0231         10         0338         10           75         1         0231         10         0338         10           75         1         0231         10         0338         10           75         1         0231         10         0338         10           75         1         0231         13         10         0338         10           76         1         0231         33         1         0231         33         1         0231         33           7         1         1         0231         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1         0331         33         1 <t< td=""></t<>
	Beer.	Reducing sugars (grams per liter).	22222222222222222222222222222222222222
		inoH	
		Distillation	Feb. 7, 1916 Jan. 12, 1916 Jan. 12, 1916 Jan. 12, 1916 Feb. 7, 1916 Mar. 32, 1916 Jan. 13, 1916 Jan. 13, 1916 Jan. 13, 1916 Feb. 27, 1916 Feb. 27, 1916 Feb. 27, 1916 Feb. 27, 1916 Mar. 33, 1916 Mar. 31, 1916 Mar. 31, 1916 Mar. 31, 1916
		Briz fourth day.	\$
		Attenuation (degrees).	<i>₩₩₽₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽₩₽</i>
<del>.</del> .	e in	Fifth day.	- 0. 0.
ont	reas	Fourth day.	0 
Jontin	Brix record (decrease in Brix).	.7ab bridT	+++++++++++++++++++++++++++++++++++++++
)—p	reco.	Second day.	0,0000446666666666666666666666666666666
recor	Brix	First day.	
tion	on.	Brix.	10111101111111111111111111111111111111
rentat	ontatio	Total solids (per cent).	60000000000000000000000000000000000000
rern	form	Acidity.	44%4%4%4444%86444%86%444%86%4444% 8%2%2%2%6%2%2%2%2%2%2%2%2%2%2%2%2%2%2%2%
3.— <i>Permentation record</i> —Continued	Mash before fermentation.	Reducing sugars (grams per liter).	8855585737735557888272338858883538 888855885828282828282828888888888
Тавьв	Mash	Sp. gr. at 15° C.	5 5 1, 0190 5 5 1, 0190 5 1, 0103 5 1, 0103 5 1, 0203 5 1, 0200 5 1, 0210 5 1, 0210 5 1, 0210 5 1, 0210 5 1, 0210 5 1, 0220 5 1, 0321 5 1, 0321 5 1, 0320 5
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		Hours filling fermenter.	
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		Acidity.	
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	Yeast.	Sp. gr. at 15° C. Micohol (per cent by weight).	81, 0215, 5, 2012           81, 0215, 5, 3772           10, 0226, 5, 3772           10, 0226, 5, 3772           10, 0226, 5, 3772           10, 0226, 5, 3772           10, 0226, 5, 3772           10, 0226, 5, 3772           10, 0226, 5, 3772           10, 0226, 5, 3771           10, 0226, 5, 3771           10, 0226, 5, 3771           10, 0226, 5, 3771           10, 0226, 5, 3771           10, 0226, 5, 3771           10, 0236, 5, 1651
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			Total solids (per c weight).	6 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2
			Acidity.	6 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2
			Sp. gr. at 15° C.	$\begin{array}{c} 0.11 \ 0.576 \ 10 \ 8 \ 9 \ 9 \ 10 \ 10 \ 10 \ 10 \ 10 \ 10 \$
	Beer.	(grams)	Reducing sugars per liter).	776 041 041 041 041 041 041 041 041 041 041
	-		.1uoH	**************************************
		Distilation	Date.	$\begin{array}{c} 0.3 & 2.7 & J.my & 27 &$
			Brix fourth day.	
		•/(\$22		
		(500	Attenuation (degree	
	seim		Fifth day.	0 20
	erea		Fourth day.	° <del>1</del> + + + + -
	Brix record (decrease in Brix).		Third day.	10000121
	X rect		Second day.	0.0001080000000000000000000000000000000
	ßri		First day.	0-0-0-0-12-10-0-2010-00-201-0-10-10-0-10-10-0-0-10-0-0-10-0-10-0-0-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-0-10-
	on.		Brix.	41122222222222222222222222222222222222
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	arm		Acidity.	12.810 10.111 10.110 10.111 10.110 10.1111 10.11111 10.11111 10.11111 10.11111 10.11111 10.11111 10.11111 10.111111 10.111111 10.1111111 10.11111111
	Mash before formentation .	smarg)	Reducing sugars. per liter).	211 0 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2
	Mash		Sp. gr. at 15° C.	51.0510 51.0510 51.0175 51.017
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	_			10, 25 uty 20, 20, 20, 10, 25 uty 20, 20, 20, 20, 20, 20, 20, 20, 20, 20,
		ast.	Acidity.	63323995239566553566556655665566566656665666566665666656666
		ur ye	Alcohol (per cent.).	228629262666666666666666666666666666666
	Yeast	95-hour yeast	Sp. gr. at 15° C.	2 2 3 6 1 19, 8 1, 0157 6, 170 19 2 2 3 7 1, 6 22, 0 1, 0305 6, 013 22 1 9 2 7, 15 6 22, 0 1, 0307 6, 013 22 1 9 2 7, 15 6 22, 0 1, 0307 6, 013 22 2 9 3 7, 11, 0 1, 0186 5, 772 11 2 9 3 7, 710 10, 036 5, 772 11 2 9 3 7, 72 11, 0 1, 0386 5, 772 11 2 9 3 7, 72 11, 0 1, 0386 5, 772 11 2 9 3 7, 72 11, 0 1, 0386 5, 772 11 2 9 4 7, 15 22, 0 1, 0207 6, 141 17 2 9 4 7, 15 22, 0 1, 0207 6, 141 17 2 9 4 7, 15 22, 0 1, 0207 6, 141 17 2 9 5 7, 12 1, 0203 6, 011 22 2 9 5 7, 12 1, 0203 6, 012 20 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7, 52 1, 011 0203 5, 061 2 2 9 7 7,
		od st.	Acidity.	8222225266022222222222222222222222222222
		Seed yeast.	Brix.	1111         1
			Cook No.	644445888888888888888888888888888888888
			Fermentation Xo.	\$

TABLE 3.—Fermentation record—Continued.

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1	10.17	weight).	22,2310 22,2310 22,2310 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,23100 23,231000 23,231000 23,231000 23,231000 23,231000 23,231000 23,231000 23,231000 23,231000 23,231000 23,231000 23,231000 23,231000 23,2310000 23,231000 23,2310000 23,2310000 23,2310000 23,2310000 23,23100000 23,2310000000000000000000000000000000000
	nt by		1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
	rd ins	Total solids (per o weight).	44994954949495956496664966666666
		Acidity.	5641546608364263844138413841681148 0084488466068968441884684148416841148
		Sp. gr. at 15° C.	1,0202 1,02202 1,02202 1,02275 1,02275 1,02275 1,02205 1,0205 1,0
Boer.	emarg)	Reducing sugars per liter).	2000 2000 2000 2000 2000 2000 2000 200
•	÷	.woH	
	Distillation.	.91£C	% 5% 5% 5% 5% 5% 5% 5% 5% 5% 5% 5% 5% 5%
	Dié		00 7 100 00 7 100 00 7 100 00 7 100 00 100
_		Briz fourth day.	KROCKCKKKXKCCKKKCKKX-XXKXCXCX
	.(299	Tg9b) noitennettA	+ 10 10 10 1- 11 11 11 11 11 1- 14 + 4 + 4 + 4 + 4 + 4 + 4 + 4 + 4 + 4
so in		Fifth day.	0. 
Brix record (docrease in Brix).		Fourth day.	
Brix).		.vab budT	
x rec		Second day.	
Bri		First day.	00000-000000000-00-0-0-000000000000000
ion.		Brix.	600 600 600 600 600 600 600 600
ental	.(109	Total solids (per c	\$2x - 666 - 6 - 2 667 62 62 666 62 x 2 66 - 6 x 2 x
form		Acidity.	
Mash before formentation	emarg)	Reducing sugars. Per liter).	44444588888888888888888888888888888888
Mash		Sp. gr. at 15° C.	2010 1000 1000 1000 1000 1000 1000 1000
Sui	, includ.	gainemiei enue 1 lo emit	
1	.төзпе	mrsi gnillît erwoH	
set.		.moH	
Formonterset		Date.	21212121212121212121212121212121212121
Pea			Production of the second secon
	ast.	Acidity.	70 IS 2012 12
	96-hour yeast	Alcohol (per cent by weight).	6 (2014) (201
Yeast.	96-he	Sp. gr. at 15° C.	22.0         1.0233         6.170         18.           22.0         1.0233         6.170         18.           22.0         1.0233         6.170         18.           22.0         1.0233         6.170         18.           22.0         1.0233         6.170         18.           22.0         1.0233         6.170         18.           22.0         1.0233         6.170         18.           22.0         1.0235         5.388         20.           22.1         1.0235         5.388         20.           22.2         1.0235         5.388         20.           22.2         1.0235         5.388         20.           22.2         1.0235         5.738         20.           10.8         1.0236         5.737         27.           10.8         1.0205         5.71         27.           10.8         1.0270         5.71         27.           10.8         1.0270         5.71         27.           10.8         1.0270         5.71         27.           10.8         1.0270         5.71         27.           10.8         1.0270         5.71 <t< td=""></t<>
	Scool yoast.	Acidity.	$\begin{array}{c} 222.0\\ 222.0\\ 101.8\\ 10$
-	Y S	Brix.	
		Cook Xo.	

	Britth HD	0011		1 1 100			02								00
	Total reducing sugars (per cent of total soluble solids).	82.08	84.13	$\begin{array}{c} 91.10\\ 85.04\\ 95.17\end{array}$			38.03 70.50	81.84 01 35	100.00	94.98	83. 42 78. 18	90.96 75.08	88. 18 86. 64	87.20 93.43 81.95	07 20
	Total soluble solids (per cent original dry wood).	28.21	28.23	$\begin{array}{c} 25.85\\ 28.00\\ 28.00\\ 28.28\end{array}$		19.63	14.04 23.22							26.65 28.91 26.65	
d.,	Reducing sugars dry di- gested sawdust compared with original dry wood.	125.13	123.75	106.45 109.45		111.15	103.3 114.78	106.64	112.29	107.10	114.45	$105.36 \\ 102.98$	102.64 102.95	106.84 108.68 106.36	100. 40
Sugar yield	Reducing sugars (per cent of original dry wood).	23.16	23. 75	23.55 23.81 23.01			5.34 16.37	21.09	21.32	21.96	21.08	21.83 17.42	22.37 23.74	857.33 27.33	8.0
Sug	Reducing sugars (per cent of dry digested sawdust).	28.98	29.39	25.07 26.06 26.96	17	64	$\left. \left. \right\} \begin{array}{c} 5.52 \\ 18.79 \end{array} \right. \right\}$	22.49	23.94	23.52	24.15	22.98 17.94	22.96 24.44	24.67 24.28 23.23 23.23	No 107
	Reducing sugars in di- gested liquor (grams per liter).	[61. 16] [60. 84]	${75.28}$				$\{11.02\47.76$	Ē							
	Reducing sugars in di- gested sawdust (per cent).	4.152	5.104	8. 544 8. 161 9. 496	3. 556	5.816	0. 696 2. 864	8.049 7.594	7.312	7.675	7.408	7.616	8. 033 8. 216	8.009 7.524 8.604	000 00
	Original wood obtained as digested sawdust (per cent).	79.91	80.82	93.92 91.36 92.76			96.66							93. 59 91. 98 94. 00	
large.	Dry weight digested saw- dust (pounds).	78.84	83.45	92.77 100.70 95.60			96. 21 85. 06							96.34 106.59 111.43	
Digester discharge	Moisture in digested saw- dust (per cent).	61.44	59.83	65.78 68.81 64.78			55.32							67.52 69.00 62.93 73.48	
Digest	Digested liquor (pounds).	$\left\{ \begin{array}{c} 98.03\\ 144.55 \end{array} \right\}$	89.86				93.95 [130.51]								
	Digested as wdust (pounds).	204.45	207.74	271. 11 322. 85 271. 45		264.90	207.71	271.20 200.93	316.64	288. 06	284.85	288.93	285.00 293.47	296.61 343.85 300.59	110.000
ě	Moisture in wood as Charged.	17.89	14.78	18.63     10.92     10.96			32.22	9.94 9.105	8.74	9,15	8.54 8.54	× ×	8.85 10.69	9.52 10.59 10.95	Pounds.
Digester charge.	Dry weight of wood charged (pounds).	98.65	103.25	$\begin{array}{c} 98.78 \\ 110.22 \\ 103.07 \end{array}$			99. 53 97. 65							102.94 115.89 118.54	1 P0
Digeste	Acid (100 per cent) to wood ratio (per cent).	1.85	1.82	1.40 1.84 1.83	-		1.80 1.80	1. 2	d'r	-ii -	-i -tri	• •		1.1.1 40 40 40 40 40 40 40 40 40	-
	Water to wood ratio (per cent).	300	250	$112 \\ 127 \\ 100 $	400	127	400	125	125	125	125	125	125 125	125 125 100	07
	Time required for blow- off (minutes).	73	80	66	75	∞ ¢	110	75	93	66	103	9 <del>4</del> 73	65.8	8888	3
	Blow-off treatment.	Cond.	op	Open. do.	Cond.	Open.	cona.	do.	-op	do.	op	do	Open. Cond.	do	
itions	Total time steam on di- gester (minutes).	28.5	21.0	23.5 22.5 17.0	20.0	19.0	19. U 29. 5	20.2	30.2	20.5	19.50	20. / 19. 5	$19.2 \\ 18.5$	30.5 23.0 23.0	07
Cooking conditions.	Maximum temperature dur- ing cook (° F.).						340 346	346 345	346	340	341	341 341	341 340	340 340 340	
ookir	Time cooked (minutes).	0	0	1010	0	10	0	00	10	000	00	00	00	0 30 0 30	>
Ŭ	Cooking pressure (atmos- pheres).	7.5	7.5	1110	7.5		7.5		1-1	- r- r				1.1.1.1 1.1.1.1	:
	Minutes to reach desired pressure.	28.5	21.0	13.5 22.5 17.0		19.0	19. 19.	20.	20	388	961 S	0761	19.19	20.05 23.00 23.00	3
	Соок Ио.	21	22	23 24 25	26	27	8 8	85	32	3 75 5	381	38	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	4444	
	Species of wood.	White spruce				Shortleaf pine		White spruce							

TABLE 4.—Digester record.

P.

<del>7</del> 0		BULLETIN 983, U.	S. DEPARTMENT OF AGRICULTURE.
-	ield.	Reducing sugars dry dl- gested sawdaus; compared with original dry wood. Total reducing sugars (per cent original dry wood). Total reducing sugars (per cent of total soluble solids).	$\begin{array}{ c c c c c c c c c c c c c c c c c c c$
	Sugar yield.	Reducing sugars (per cent of dry digested sawdust). Reducing sugars (per cent of original dry wood).	25,25,22,25,22,25,22,25,22,25,22,25,22,25,22,25,22,25,25
		Reducing sugars in di- gested liquor (grams per liter).	888 701 916 771 916 772 976 976 976 113 846 846 77, 456 677, 456 677, 456 677, 456 677, 456 677, 456 677, 456 70, 70 70, 76 70, 76 67, 456 67, 456 70, 70 70, 76 70, 76 88 80, 70 70, 76 70, 76 80, 70 70, 76 70, 76 80, 70 70, 76 80, 70 70, 76 80, 70 70, 76 80, 70 70, 76 70, 76 80, 70 70, 76 80, 70 70, 76 70, 76 70, 76 70, 76 70, 76 70, 76 70, 76 70, 76 70, 70 70, 70, 70 70, 70, 70, 70, 70, 70, 70, 70, 70, 70,
-	•	cent). Reducing sugars in di- gested sawdust (per cent).	マンプランサング (1) (1) (1) (1) (1) (1) (1) (1) (1) (1)
	se.	se banistdo boow langinO digested sawdust (per	88         97, 56           64         98, 52           83         86, 97, 56           84         89, 52           85         85, 52           86         97, 56           87         99, 56           88         87, 19           88         89, 57           88         87, 19           88         87, 19           88         87, 19           88         87, 19           88         87, 19           88         87, 19           88         88, 37, 19           88         88, 37, 19           88         88, 37, 19           88         88, 38, 77           88         88, 38, 77           88         88, 38, 77           88         88, 38, 77           88         88, 38, 77           88         88, 38, 70           776         90, 89           88         88           88         88           88         88           88         88           88         88           89         88           89         86
	lischarg	Dry weight digested saw- dust (pounds).	50         50<
-na	Digester discharge	Moisture in digested saw- dust (per cent).	800887 312888 3 1 1 8 8853589888888888888888888888888888888
חחזחחו	Di	Digested liquot (pounds).	65. 25. 35. 35.
		Digested sawdust (pounds).	238.83 236.672 235.172 235.172 235.172 235.173 4577.30 4577.30 4577.30 4577.30 561.40 561.40 561.40 51.40 315.55 333.47 333.47 333.47 333.47 333.47 333.47 333.47 333.47 335.56 315.55 335.56 35.56 35.
L recoi	rge.	Moisture in wood as Charged.	$\begin{array}{c} 10.59\\ 10.04\\ 11.004\\ 11.004\\ 11.090\\ 11.090\\ 12.04\\ 21.375\\ 2.88$
TABLE 4.—Dugester record—Collinued	Digester charge	Dry weight of wood charged (pounds).	103, 338, 103, 338, 103, 338, 103, 339, 559, 596, 595, 596, 595, 596, 596, 576, 1038, 536, 1038, 535, 1038, 535, 1038, 538, 1038, 1
4	Dige	Acid (100 per cent) to wood 13tio (per cent).	୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶୶
ABLE		Water to wood ratio (per	<u> </u>
Ĩ		Time required for blow- off (minutes).	73 73 73 75 75 75 75 75 75 75 75 75 75 75 75 75
	ons.	Blow-off treatment.	Cond. 
	Cooking conditions.	Total time steam on di- gester (minutes).	0         0         1         0
	king	Maximum temperature dur- ing cook (° F.).	3371 3386 3386 3386 3386 3386 3386 3386 338
	Coo	Time cooked (minutes).	2222992 22222 15 15 15 15 15 15 15 15 15 15 15 15 15
		Cooking pressure (atmos- pheres).	なななな なんなん な なっちゃなななななななななななな
		Minutes to reach desired pressure.	3888538         5 </td
ł.		Соок Ио.	
		Species of wood.	White s p r u ce- (Con.) Shortleaf pine Western larch Western larch White spruce.(fine chips). White spruce (large chips). White spruce (large chips). White spruce (large chips). White pine Dougtas fin, Mon- White pine Longe pole pine

TABLE 4.-Digester record-Continued.

BULLETIN 983, U. S. DEPARTMENT OF AGRICULTURE.

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<b>94. 3</b> 99. 3 99. 3 99. 3 99. 5 99. 5 99. 4 94. 0 94. 0	87.9 97.6	69.9 67.8 87.8 72.8	86.4 75.2	75.3 83.3	99.9	87.2	90.0	95.8 78.2 78.8		87.8 81.3 82.4		
$\begin{array}{c} 775 \\ 95 \\ 95 \\ 95 \\ 95 \\ 95 \\ 95 \\ 95 $	05		61 . 32 . 61	6. 80 6. 58	0.40	65	43		3.21	24.52 25.78 21.89	5.92	SO4.
52177010102232323 5221770101022323232 5222355222323232323232323232323	6 24. 6 20.		5 23. 1 24.	9 25. 8 23.	9 20.	0 24.	<u>,</u> 4₁	0 21. 3 24. 0 26.		0.010	. 0	.8 H <sub>2</sub>
$\begin{array}{c} 1108\\ 1128\\ 1119\\$	110. 114.	that had been been	104. 122.	107.	110.	107. 96.	4 9-4	103. 106. 103.		103.		ke=1 ke.
<b>22. 06</b> <b>22. 06</b> <b>22. 06</b> <b>22. 06</b> <b>22. 06</b> <b>22. 06</b> <b>23. 06</b> <b>23. 06</b> <b>25. 13</b> <b>25. 13</b> <b>26. 13</b> <b>27. 14</b> <b>27. 15</b> <b>27. 15</b> <b>27</b>	$21.15 \\ 19.60$		20.44 18.25	19.44 19.59	20.34	21.45 6.56 18.03	21.12	20.86 19.37 20.84	21.42	21.52 20.92	19.47	Niter cake=1.8 H <sub>2</sub> SO <sub>4</sub> . Niter cake.
0000040000000000	23.4	$   \begin{array}{c}     19.6 \\     20.1 \\     23.2 \\     15.0 \\   \end{array} $	21.4 22.3	21.0 22.1	22.6	6.3	00	21.5 20.6 21.5	0	25.1 21.6 18.9	6	IN NI
11: 92223351:	10	10							:			-
156         156           818         818           818         818           818         85.           552         552.           552         552.           552         552.           552         552.           552         552.           66.         82.           67.         50.           68.         82.           522.         522.           532.         522.           63.         67.           64.         82.           64.         82.           64.         82.           64.         82.           64.         82.           64.         87.           74.         74.	594	304 62. 967 882 380	848 730	42 24	31	308 668 	540	40 08 32	560	6'/2 322 336	939	
<b>7.15</b> <b>7.15</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.18</b> <b>8.215</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.15</b> <b>1.151.15</b> <b>1.15</b> <b>1.15</b> <b>1.1511.1511.1511.1511.1511.15111.151111111111111</b>	6. 3 9. 3	సి. అద్దార్ సి. అద్దార్	6.8	7.142 6.624	7.031	7.308 1.668 5.810		7.340 6.808 7.632	7.5	9.5.9	6.9	<sup>8</sup> H <sub>3</sub> PO <sub>4</sub> . <sup>9</sup> FeSO <sub>4</sub> .
92.0 10.3	$90.1 \\ 87.6$	87.1 84.8 85.0 96.4	95.7 81.8	92.5 88.3	90.25	93.25 104.0 82.29	85.30	97.10 94.20 97.2	89.25	89.3 96.8 84.8	98.0	18
90.20 1127,95 86.0 78,5 78,5 78,5 112,0 78,5 128,4 884,0 884,0 884,0 107,15 53,10 53,100 53,100,100 53,100,100 53,100,100,100,100,100,100,100,100,100,10	94.5 94.10	$ \begin{array}{c} 49.9\\ 102.8\\ 102.4\\ 156.8\\ \end{array} $	117.3	115.5 113.6	114.0	125.03 69.40		127.29 126.51 104.0	120.2	134.8 130.1 137.3	145.2	04.
40 440 552 113 113 113 1555 1 1 555 1 1 555 1 1 555 1 1 555 1 1 555 1 1 555 1 1 555 1 1 552 1 552 1 5 552 1 5 552 1 5 552 1 5 5 5 5	88 49	237 237 24	99 38	39	66	59 08 14	222	22 22 22 22 22 22 22 22 22 22 22 22 22	46	288	69	6 KClO <sub>3</sub> +H <sub>2</sub> SO <sub>4</sub> .
	20.02	62. 65.	67.	67.	68.	67. 73.	69	65. 60.	.68.			103+
$\begin{array}{c} 123.38\\ 115.0\\ 66.87\\ 735.79\\ 135.79\\ 135.79\\ 254.54\\ 226.40\\ 26.40\\ 26.40\\ 176.57\\ 176.57\\ \end{array}$		72.85										6 KC
	323.40 319.09	$\begin{array}{c} 132.80\\ 346.19\\ 344.69\\ 451.06\end{array}$	366.67 342.09	350.42 374.64	363. 76	386.81 257.91	381.82	372.29 381.58 265.40	381.41	424. 21 385. 40 411 85		
	43.04 3. 26.12 3	96 334 43 43	32 3 72 3	37	52 3	29 08 29		10 00		11.94 4 15.35 3 13.60 4		4 H <sub>2</sub> SO4. t NaCl.
0.010.00000000000000000000000000000000		$^{9.27}_{27.14}$	23.	21.	22.	19. 40.	19.	16. $53.$	3,	-i i i i i	13.	32
												- <b>H</b> Z
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$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	328         40        do         60-120         125         2.50         104.21           40        do         125         2.50         107.34	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	20 327 40do. $125 \left\{ \begin{array}{c} 41.60 \\35 \end{array} \right\} 126.51$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	20 331 40 $125 \begin{cases} 41.8 \\ 8.1 \end{cases} \ 136.30$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	5 20 328 40 $\dots$ do. 125 $\begin{pmatrix} 4 1.8 \\ 9 & 1 \end{pmatrix}$ $134.55$	330         59        do         115         125         4 2.0         151, 11           328         38        do        do        do         175         4 25         134, 55	20 324 40do. $125 \left( \frac{4.9}{11.9} \right) 146.3$	formly cooked; cook No. 68 was made as a check.
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## LIST OF PATENTS.

Patent.	No.	Date.	Name.	Title.
United States.		Apr. 12,1870	Sten Sternberg	sugar and alcohol from lichens.
Do Do		July 26,1881 May 29,1883	S. H. Johnson. A. C. Landry, Chas. Lauga.	Production of saccharine substances. Method of and apparatus for convert- ing amylaceous and ligneous sub-
Do	607091	July 12,1898	E. Simonsen	stances into grape sugar. Treatment of materials containing cel- lulose.
Do		Apr. 17,1900	A. Classen	Process of converting wood into fer- mentable sugars.
Do		July 24,1900	do	sugar.
Do Do	696800	Mar. 18, 1902 Apr. 1, 1902	do	Do.
Do		May 20, 1902	do	Process of converting cellulose into fer- mentable sugar.
Đo	1 707903 1 12069	May 5,1903 Aug. 26,1902 Dec. 30,1902	}do	
Do Do	707903	July 24, 1900 Aug. 26, 1902	}do	Process of converting cellulose into sugar. Process of converting wood into sugar. Process of making glucose. Manufacture of dextrine.
Do	745675 755390	Dec. 1,1903 Mar. 22,1904		
Do Do	763472	May 31, 1904 June 22, 1904	M. F. Ewen, G. H. Tomhnson.	Process of making achro-o-dextrose. Process of converting wood cellulose.
Do Do		Dec. 12,1905 July 10,1906	C. F. Gross. A. Classen.	Manufacture of sugar from cellulose. Process of converting cellulose of wood into fermentable sugars.
Do	930274	Aug. 3,1909	H. W. Doughty, F. E. Wetees.	Method of treating wood during distil- lation.
Do		Oct. 26, 1909	Wetees. M. F. Ewen, G. H. Tomlinson.	Process of producing fermentable sugar from ligno-cellulose.
Do Do	970029 985725	Sept. 13, 1910 Feb. 28, 1911	Gista Ekström W. P. Cohoe	Process of making grape sugar. Method of making a fermentable prod- uct from cellulosic and ligneous mate- rials.
Do	985726	do	do	Method of making a glucoselike product from cellulosic and ligneous materials.
Do Do		July 2,1902 July 16,1912	A. F. Richter. M. F. Ewen, G. H.	rocess of producing fermentable sugar
Do	1032440	do	Tomlinson. G. H. Tomlinson do	from ligno-cellulose. Do.
Do Do Do	$\begin{array}{c} 1032441 \\ 1032442 \\ 1032443 \end{array}$	do	do	Process of feeding materials to digesters, Do. Process of treating ligno-cellulose for
Do			do	recovering turpentine and sugar. Apparatus for treating ligno-cellulose
Do			do	for recovering turpentine and sugar. Apparatus for feeding materials to
Do			do	digester. Apparatus for treating comminated
Do	1032447	do	do	
Do	1032448	do	do	cellulose. Process of producing fermentable sugars from cellulosic materials.
Do Do	$\frac{1032449}{1032450}$	do	do do	Apparatus for treating ligno-cellulose. Process of producing fermentable sugars.
Do	1033064		F. E. Gallagher, H. S. Mork.	Process of producing fermentable
Do	1037185	Aug. 27,1912	Mork. do	Process of producing fermentable sugars.
Do	1042332		G. Ekström	Method of manufacturing alcohol from sulphite liquor.
Do	1046160			Manufacture of ethyl alcohol by fer- menting sulphite liquor.
Do				Manufacturing alcohol from sulphite liquor.
Do Do			F. E. Gallagher F. E. Gallagher, H. S.	Process of producing fermentable sugars. Process of producing sugars from cellu-
Do	1056163	do	Mork. F. E. Gallagher	Process of producing fermentable
Do				sugars. Method of removing organic constiu- ents from residues obtained in pro-
Do	1087743	do	do	ducing alcohol from waste sulphite cellulose lyes of similar liquid. Method of producing cellulose. Converting of cellulose into ferment- able sugar
	1001111		1.D	able sugar.

<sup>1</sup> Reissue.

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List of patents-Continued.

Detunt	N	Dete		- (1):41-
Patent.	No.	Date.	Name.	Title.
United States.	1091327	Mar. 24, 1914		Process of producing fermentable sugars.
Do	1096030	May 12,1914	F. E. Gallagher, H. S. Mork.	Do.
Do	1101061	June 23,1914	A. Classen	material into glucose and other
German	29025	Jan. 22, 1884	P. Fliessbach	soluble and insoluble carbohydrates. Verfahren der Behandlung von Pülpe aus der Kartoffelstärkefabrikation zur Erzeugung von Dextrin, Trau- benzucker, Syrup u. s. w. Von der Unwandlung von Stärke und Cellulose in Rohrzucker (Saccharose) unter Anwendlung von Elektrizität. Ver. zur Herstellung von zuckerhalti- gen Füssigkeiten und Alcohol aus Torf.
Do	32388	Dec. 3, 1884	L. Aubert, V. Giraud	benzucker, Syrup u. s. w. Von der Unwandlung von Stärke und Cellulose in Rohrzucker (Saccharose)
Do	66158	Nov. 15, 1891	C. Kappesser	ver. zur Herstellung von zuckerhalti- gen Flüssigkeiten und Alcohol aus
Do	77158	Aug. 22, 1891	C. Pieper	u. Zuckersyrup unmittelbar aus Kartoffelreibse Kartoffelpülpe,
Do	92079	Dec. 2, 1894	E. Simonsen	Schlammstärke u. s. w. V. zur schnellen Umwandlung von Holz, Sagespähnen u. dgl. in Gahr- fähige Products mit Hülfe von Säuren.
Do	111868	July 15, 1899	A. Classen	
Do	118540	Sept. 24, 1899	do	V. zur Ueberführung von Holz u. and. cellulosehaltigem Material in Zucker
Do	118541		do	Stärke haltigen u. s. ähnlichen Mate-
Do	118542		do	Zucker unter Aufschliessen mit Chlor.
Do	118543	May 1,1900	do	Neuerung in Verf. zur Ueberf. von Holz u. s. w. in Zucker.
. Do	118544	May 12,1900	do	Holz u. s. w. in Zucker. Neuerung in V. zur Ueberf. von Holz u. s. w. in Zucker.
Do	121869	Oct. 17,1900	do	Verf. zur Ueberführung von Holzfaser in Dextrose.
Do	130980	June 9, 1901	do	Verf. zum Verzuckern von Holz durch schweflige Säure in Druckgefassen.
Do	123911	Oct. 17, 1900	do	Addition to Patent No. 121,869. Title same as 121,869. Specifies heating to 125°-135° C.
Do	147844a	May 26, 1901	R. Gentzen, L. Roth	Verf. zur Gewung von für die Spiritus- fabrikation verwendbaren Maiselen aus Pflanzen und pflanzlichen Abfall- stoffen.
Do	193112	Jan. 17,1906	G. Ekström	Verf. zur Herstellung von Trauben- zucker oder Ethyl Alcohol aus Zellu- lose haltigen Stoffen.
Do Do	$207354 \\ 318203$	Aug. 8, 1907 Oct. 9, 1902	do Reynaud	Addition to Patent No. 193,112. Pour nouveau procédé de transforma- tion de la cellulose des végétaux en
English	358696	Màr. 3, 1906		glucose et ses derives. Compagnie Industrielle des Alcools de L'Ardèche, France. Rotary apparatus for saccharifying
Do	380358	Dec. 6,1907	Ekström, G	wood. Procédé pour la fabrication du sucre de raisin ou éventuellement de l'alcool ethylenigne, au moyen de
Do	358696	do		matières contenant de la cellulose. Compagnie Industrielle des Alcools de l'Ardèche. Rotary apparatus for saccharifying
Do	393336	do	Bouchand Praceign	wood. See Zeit. für Angw. Chemie, 1910,
French	405187	Dec. 22, 1909	J. J. D'Orlowski	23, page 916. Procédé pour la fabrication de l'alcool avec de la sciure de bois de la cellu- lose, de l'amidon et dcs matières
Do Do	12872 34	1849 1854	C. Montgomery M. Poole	amylacées. Brewing, distilling, and rectifying. Processes for the manufacture of dex- trine and glucose for distillation and application of the product thereof
Do Do	$1246 \\ 2281$	$     1854 \\     1854 $	H. Bordier R. H. Brooman	application of the products thereof. Manufacture of ligneous alcohol. Obtaining alcohol from organic sub- stances.
Do Do	2433 1283	1856 June 7,1858	T. F. Henley J. B. A. Lombard, X. T. Esquiron.	Producing alcohol and food for cattle. Obtaining saccharine substances from cereal and vegetable matters, etc.

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Patent.	No.	Date.	Name.	Title.
English	268	Jan. 21,1880	A. M. Clark	Obtaining glucose and alcohol from ligneous materials.
Do Do	$     4334 \\     4514 $	Oct. 23,1880 Nov. 4,1880	S. H. Johnson Wm. F. Nast	Production of saccharine substances. Manufacture of sugar, etc., from cellu-
Do	11407	Aug. 18,1884	H. H. Lake	lose or ligneous materials. Improvements relating to apparatus for treating ligneous substances for the production of glucose.
Do	11557	Aug. 22,1884	A. M. Clark	the production of glucose. Improved apparatus for use in treat- ing wood and other ligneous matters by means of hydrochloric acid gas, in order to obtain glucose therefrom.
Do	1767	Feb. 9,1885	Adolph Behr	Improved apparatus and process for obtaining cellulose and glucose from wood and other vegetable matter.
Do	10164	Aug. 9,1886	H. J. Hadden	solution and other vegetable inter- improvement in the treatment of Jeru- salem artichoke to prepare it for use in distilling in the manufacture of glucose and similar industries.
Do	13653	Aug. 29, 1889	Paul Marix	A new or improved process for the purification of mineral oils and for
Do	21059	Dec. 2, 1891	C. Kleyer, C. Kappesser	for the production of cellulose sugar
Do	13492	July 12,1894	R. Zdarek	and alcohol. Improvements in the manufacture of alcohol.
Do	10762	May 30, 1895	E. Simonsen	An improved process for the treatment of materials containing cellulose for the production of spirits.
Do	21314	Nov. 11, 1895	A. L. Tedesco	Process for manufacturing pure dex-
Do	21878	Sept. 24, 1897	R. Zdarek	Improvements in the manufacture of ethyl alcohol.
Do Do	$24013 \\ 1035$	Nov. 15, 1898 June 16, 1899	N. Basset. C. F. Cross, J. S. Rem-	Method of treating cellulose. Improvement in the production of
Do	12 241	June 12,1899	ington. Paul Magnier, P. A. Brangier.	starch and saccharine matters. Improved process for convering wood, wood shavings, woody fiber, saw- dust, and other substances into dex- trine glucose, and alcohar
Do	258	Jan. 4,1900	Alex Classen	trine, glucose, and alcohol. Process for converting cellulose and starch into fermentable sugar.
Do	259	do	do	Process of converting wood into ler-
Do	4199	Feb. 27, 1901	do	Improved process for converting cellu- lose into sugar (dextrose).
Do	12588	June 20, 1901	do	Process for converting wood and other cellulose materials into sugar.
Do Do	8545 11113	Apr. 13, 1904 May 13, 1904	C. F. Cross W. H. Wheatley	Treatment of cottonseed hulls to ob- tain useful products therefrom. Method of preparing must for the man-
D0	11113	May 13, 1904	W. H. Wheathey	ufacture of spirits of wine, the feeding of cattle, and the manufacture of dextrose.
Do	16262	July 22,1904	Arno Börner	A new or improved process for the manufacture of starchlike or Amy-
Do	22709	Nov. 6, 1905	Alex Classen	loidlike substances and sugar. Process for facilitating the fermenta- tion of sugar solutions obtained from wood that contains tannin.
Do	16510	July 21, 1906	B. E. E. Newlands	
Do	5128	Mar. 2, 1907	C.S. Lake	Improvements in and relating to the treatment of peat for the production
Do	24503	Nov. 5, 1907	Boren Hafner, Frank Krist.	of alcoholic and other products. Improved manufacture of fermentable sugar from materials containing starch or cellulose.
Do	18341	Aug. 13, 1907	G. Ekström	An improved process for making grape sugar (glucose) and ethyl alcohol from materials containing cellulose.
Do	5128	Mar. 2, 1907	H. H. Lake	Improvements in and relating to the treatment of peat for the production of alcoholic and other products.
Do	26619	Dec. 8, 1908		of alcoholic and other products. Compagnie Industrielle des Alcools de L'Ardèche. Improvements in or relating to apparatus for converting wood into fermentable sugars and other products.

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