

# INDUSTRIAL ALCOHOL

THE PRODUCTION AND USE OF ALCOHOL  
FOR INDUSTRIAL PURPOSES, FOR USE  
AS AN ILLUMINANT AND AS A  
SOURCE OF MOTIVE POWER

BY  
JOHN GEDDES M'INTOSH

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BY  
H. B. STOCKS, F.I.C.

WITH NINETY-ONE ILLUSTRATIONS AND THIRTY-SEVEN TABLES



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## REVISER'S PREFACE.

THE formation of alcohol is a natural biological function adapted to industrial requirements, depending, as it does, upon the fermentation of saccharine solutions by the agency of the minute organism *Saccharomyces cerevisiæ* and other varieties of yeast which abound everywhere. Thus, when the ripening of fruit has attained its zenith, the juices containing the sugars formed are spontaneously fermented to alcohol, which quickly passes to acetic acid, and eventually to carbonic acid and water.

It is therefore not a matter of surprise that in very early days, alcoholic beverages, such as mead, beer, ale, wine, etc., were made by the fermentation of saccharine juices, either spontaneously or after the addition of yeast. These products contain however only a moderate quantity of alcohol, which is limited by the percentage of sugar in the original liquor and also by the vitality of the yeast employed, strong solutions of sugar not being fermentable.

It was however found that when such liquids were distilled, the alcohol, being more volatile, passed over in greater abundance at first leaving water and extractive matters behind, so that by distillation a much stronger spirit could be produced. By this method "strong waters" or "spirits" were obtained which were named whisky, brandy, rum, gin, etc.

In these spirits the flavour and aroma are still of great importance, but the introduction of the Coffey's still and the modern rectifying stills allowed of the removal of the odoriferous substances and impurities so that practically pure alcohol of high strength could be continuously produced on a very large scale.

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The studies in fermentation, and of the diseases of beer by Pasteur, and the adaptation and elaboration by later workers of the methods introduced by him to the cultivation of pure ferments have had a profound influence on the brewing and distilling industries, which are now carried out on a scientific basis and under expert control. Previous to this the losses due to defective fermentation were a serious problem, and the operation of brewing an uncertain one. With increase of knowledge the fermentation and distillation operations become more easily directed and controlled.

Alcohol for industrial purposes does not differ essentially from alcohol for potable use, except that for some applications—*v.g.*, for burning and motor purposes—it need not be so carefully refined. For many industries it should however be approximately pure and of high strength, and alcohol of this description can be manufactured in modern distilleries on an enormous scale.

For industrial purposes the question of cost is of paramount importance, and it is this factor alone which prevents it being used on a much larger scale. In its manufacture the choice of raw material must be given due consideration. Such material should be as rich in carbohydrates as possible consistent with a low cost; sugar being preferable to starch since it requires no preliminary conversion. A very poor material may, however, be suitable if the price is sufficiently low, as instanced by sulphite-cellulose waste liquors which are now employed.

The manufacture of alcohol synthetically from ethylene, or from acetylene made from calcium carbide, has been proved to be possible, but, although such methods are of great interest and importance, they cannot at present compete with the ordinary fermentation process.

In the present work the methods of manufacturing alcohol from beets, grain, potatoes, low wines, sugar, and molasses are described, also its production from wood waste and sulphite-cellulose liquors and synthetically from ethylene. Other raw materials are considered. The preparation, fermentation and distillation of the washes are fully explained,

under this head being included an account of the propagation of pure yeast and the many forms of distillation and rectification plant.

Chapters are devoted to the properties of alcohol, the preparation and properties of the derivatives of ethyl alcohol and also of methyl alcohol and amyl alcohol and their derivatives, and of chloroform, acetone, and similar products. For industrial purposes alcohol is usually methylated or denatured, the denaturants authorised in various countries being dealt with. Statistics and particulars are given relating, among other things, to the use of alcohol in the arts and manufactures, and considerable space has been devoted to the employment of alcohol for heating and lighting purposes and as a motor fuel, and the economical manufacture of alcohol for such purposes.

In the preparation of this edition the reviser is indebted to articles and abstracts in the "Journal of the Society of Chemical Industry," the "Board of Trade Journal," and other sources.

H. B. STOCKS, F.I.C.

BIRKENHEAD, *August*, 1923.



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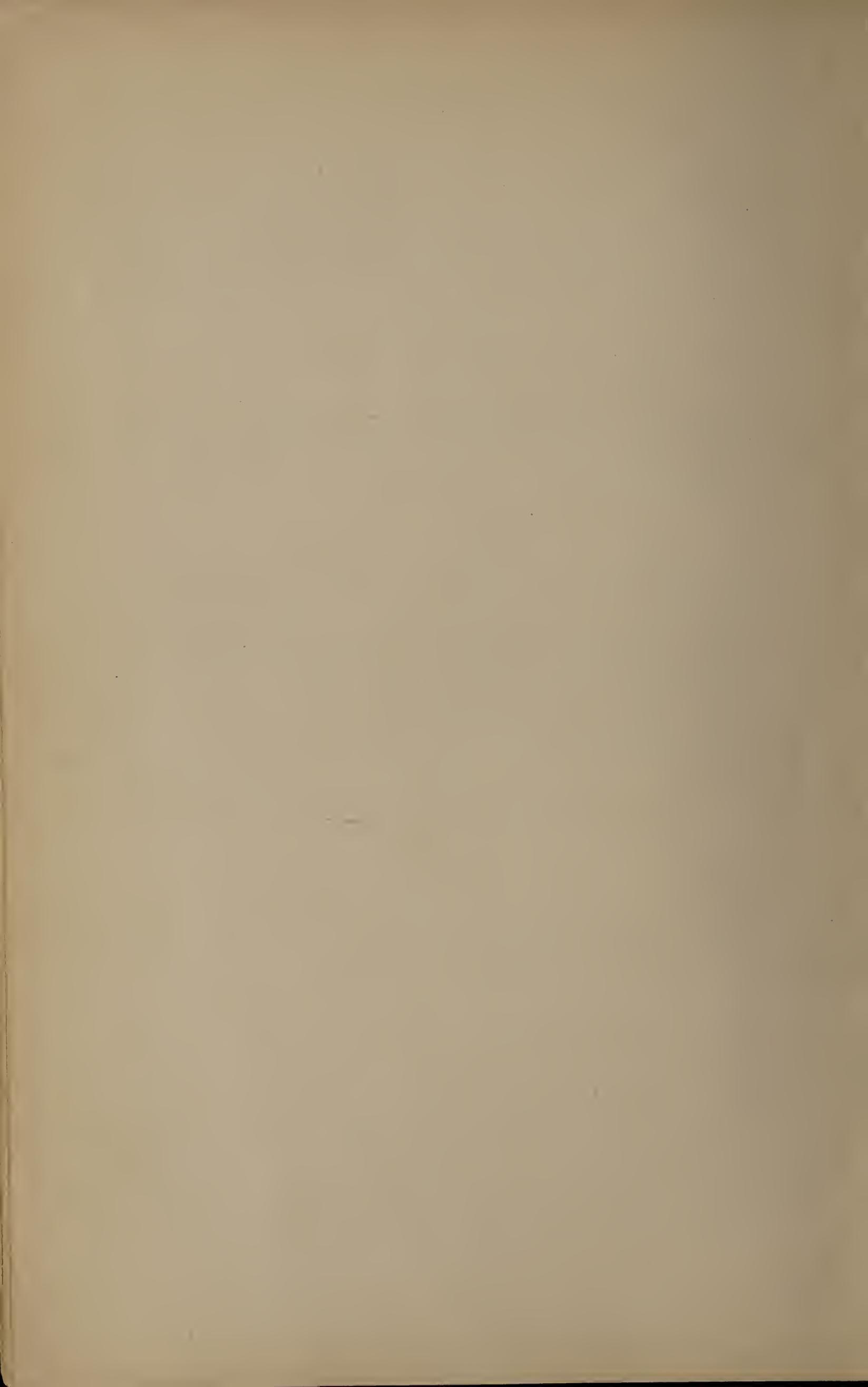
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## CHAPTER I.

### ALCOHOL AND ITS PROPERTIES.

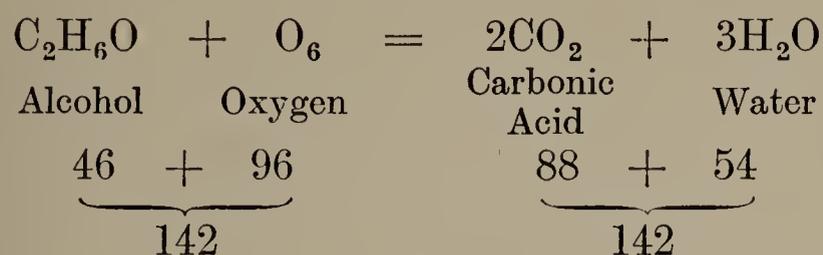
DEFINITION.—*Ethyllic alcohol—common alcohol*— $C_2H_5HO$ .—This is the principal spirit obtained by distillation from fermented liquors, e.g. wine, beet juice, malt worts, etc., technically termed wash. For a good many years the distilling plant employed has been so perfected that by a continuous operation an alcohol of 95 per cent. strength may be obtained free from ethers and aldehydes on the one hand, and fusel oil (amylic alcohol, etc.) on the other.<sup>1</sup> Alcohol being more volatile than water, on distilling a mixture of the two the portion which passes over first is much richer in alcohol than the original liquid, and after a certain amount has been distilled the residue is practically free from alcohol. When these vapours are condensed a spirituous liquor is collected, the strength of which depends upon the extent to which the distillation has proceeded. The longer the duration of distillation the weaker the spirit. But if, instead of condensing the vapours *per decensum*, we cause them to ascend into a series of receivers arranged one above the other, or into a column containing receptacles at different heights to receive the condensed liquid, it is clear that the vapour of water will condense first and nearest to the still in which the worts, etc., are being heated, and to which the condensed liquid is constantly being returned, whilst the alcoholic vapour, on the other hand, will rise and ascend further and further away from the source of heat towards the cooler portions, where the more condensable vapour of water cannot follow it. Finally, the alcoholic vapours are themselves condensed in the cooler parts of the condenser most distant from the still, but those portions which condense first are more dilute

<sup>1</sup> See footnote 3, p. 18.

than the portions which condense further away. Only this latter portion is collected for sale. The more watery portions flow back into the heated parts of the column, where they are again deprived of their more volatile constituents. The alcohol in the "foreshot" of the "pot-still" is rich in aldehydes and ethers, that of the last runnings contains much fusel oil. In the continuous rectification distillation columns there is a continuous feed of raw spirit and simultaneous separate elimination therefrom of (a) ethers and aldehydes, (b) pure alcohol, (c) fusel oil, and (d) water.

*Properties of alcohol.*—Alcohol is a liquid with a spirituous odour and ardent aromatic taste, more mobile and far more volatile than water and of much lighter weight. Alcohol is miscible in all proportions with most liquid substances, with the exception of the fatty oils, which dissolve in it in very limited proportion—castor oil and croton oil excepted, both of which it freely dissolves. In fact, next to water, it is the most useful and important solvent. Its solvent action on resins, balsams, camphor, etc., is well known. As met with in commerce it invariably contains some water. The term *absolute* alcohol is really relative, not absolute. Absolute alcohol, that is, alcohol free from water, even if it were capable of being prepared, the moment it was exposed to air would absorb water from it. A definite volume of alcohol weighs much less than an equal volume of water. An imperial gallon of water weighs 10 lbs. An imperial gallon of absolute alcohol weighs 7.938 lbs. at 60° F. As alcohol is miscible in all proportions with water, it is obvious that the weight of a gallon of more or less dilute alcohol will be a measure of its strength provided the liquid contains only pure alcohol and water (Tables II.-V.). Alcohol is easily inflammable. It burns with a pale bluish flame hardly perceptible in broad daylight, but the heat of its flame is very intense, as may easily be demonstrated by suspending in it a coil of fine platinum wire, which becomes white hot. It deposits no soot on the surface of cold bodies held over it. The pale blue colour of the flame is accentuated when *the alcohol* is diluted with a little water, and not even a trace of sooty matter is then deposited. The products formed by the combustion of alcohol under ordinary conditions

are carbon dioxide and water. The weight of the water considerably exceeding that of the alcohol burnt, thus :—



Saussure, Junr., found by actual experiment that 100 parts of alcohol, when burned, yielded 136 parts of water.

*Products formed by the destructive distillation of alcohol.*—When alcohol is passed through a red-hot tube, it is decomposed more or less perfectly according to the temperature and to the rapidity of its passage. The most accurate results were obtained by T. de Saussure, who passed alcohol through a red-hot porcelain tube; on the interior walls of the tube were deposited a little charcoal, a volatile crystalline substance (naphthaline), and a brown empyreumatic oil; a gas was also evolved, the specific gravity of which was 0.856. This gas consisted of a mixture of hydrocarbons (methane, ethane, acetylene, benzene), carbonic oxide and hydrogen, and a great number of derivatives. When alcohol vapour and oxygen are mixed in certain proportions and fired by an electric spark, a violent explosion occurs, carbonic acid and water being formed. One volume of alcohol vapour requires three volumes of oxygen for its perfect combustion, the result being two volumes of carbonic acid and three volumes of aqueous vapour. But substances very rich in oxygen, like chromic acid, may actually inflame alcohol by mere contact. A few drops of alcohol, spotted on dry chromic acid, from a separating funnel, immediately become inflamed, the chromic acid ( $\text{CrO}_3$ ) losing half its oxygen, and being reduced to sesquioxide of chromium (chrome green,  $\text{Cr}_2\text{O}_3$ ).

When alcohol is oxidised at a lower temperature than that required for its inflammation, as by the action of spongy or finely divided platinum, the products of its combustion are very different; the proportion of carbonic acid is lower, while aldehydic and acetic compounds are formed. There are many substances which communicate *colour* to the flame of alcohol; boric

acid and the soluble salts of baryta impart a green tint ; soda salts cause it to burn yellow ; the soluble salts of strontia give it a beautiful rose-colour, and chloride of calcium also reddens its flame ; copper salts impart a fine green tinge. Graham has shown that alcohol may, in many instances, be combined with salts in the same way as water of crystallisation ; such combinations have been called *alcoholates*. They are obtained by dissolving the substances by heat in absolute alcohol, and are deposited as the solution cools, more or less regularly crystallised. They appear to be definite compounds, and in some cases the alcohol is retained by an attraction so powerful as not to be evolved at a temperature of 400° or 500° F. Graham examined the alcoholic combinations of chloride of calcium, nitrate of magnesia, nitrate of lime, chloride of zinc, and chloride of manganese. Chloroform, chloral, bromoform, iodoform are the result of the action of chlorine and other halogens on alcohol. Under certain conditions it reacts with nearly all the acids, giving rise to an important and varied class of compounds, resulting from their mutual action. A series of curious phenomena, arising out of the mixture of alcohol and acids, observed by Chevreul, were examined by Pelouze. When a little sulphuric acid, for instance, is mixed with alcohol, the mixture has no action upon any *neutral* carbonate, and yet it decomposes acetate of potash, evolving acetic acid. A mixture of alcohol and hydrochloric acid does not act upon carbonate of potash, but it decomposes the carbonates of soda, lime, strontium, and magnesium. A mixture of alcohol and nitric acid is without action upon carbonate of potash, but it acts powerfully on carbonate of lime, and of strontium, and slowly on the carbonates of soda, barium, and magnesium. Alcoholic solutions of acetic and of tartaric acid decompose none of the carbonates : a similar solution of citric acid decomposes the carbonates of potash and magnesium, but not carbonate of barium, strontium, or calcium ; and the alcoholic solution of oxalic acid decomposes carbonate of strontium, of calcium, and of magnesium, but not carbonate of potash. The addition of a small quantity of water does not affect these mixtures, for when a saturated solution of carbonate of potash is mixed with the

alcoholic solution of acetic acid, the carbonate is precipitated without effervescence: an alcoholic solution, therefore, may appear neutral to certain tests, whilst, in reality, it is strongly acid. It is difficult to suggest an explanation of these statements.

Alcohol dissolves a small quantity of sulphur, especially at its boiling temperature, but the greater portion is deposited, on cooling, in small brilliant crystals: the solution has a peculiar odour. When a flask of alcohol is suspended in the head of a still, containing sulphur, and the latter melted, so that as its vapour rises it may be condensed with that of the alcohol, a reddish-yellow liquid passes over, containing sulphuretted hydrogen: this solution becomes milky upon the addition of water, and appears to contain about a hundredth part of sulphur. A very similar solution may be obtained by passing sulphuretted hydrogen into alcohol, under slight pressure. Alcohol also dissolves phosphorus, taking up about  $1/240$ th part at its boiling-point, and retaining  $1/320$ th part when cold. This solution is luminous in the dark on exposure to air, and produces a beautiful pale but ineffectual flame when poured upon hot water. Alcohol dissolves carbon-disulphide, and the solution is decomposed by alkalis. Potassium and sodium slowly decompose alcohol at common temperatures; heated with it, they evolve hydrocarbons. Caustic potash and soda are soluble in alcohol, and it is sometimes resorted to as a means for the purification of those alkalis; if the alcohol is not pure the solutions darken considerably on keeping. Ammonia and its carbonates are soluble at common temperatures in alcohol: it also absorbs a large quantity of ammonia gas, and of several other gases. The oxides of lithium, barium, strontium, and calcium are almost insoluble in alcohol, even in their hydrated states; so also are the fixed alkaline carbonates: the alkaline sulphides are, however, soluble. The greater number of the chlorides, iodides, and bromides, which are soluble in water, are soluble also in alcohol, and with many of them the definite compounds above mentioned are produced. Thus there are well-defined compounds of chlorides of calcium, zinc, manganese, etc., with alcohol (*alcoholates*); the same is the case with some of the nitrates; but the sulphates are almost all

insoluble ; use is often made of this fact in the analysis of mixtures of salts. Zeise studied the mutual reaction of chloride of platinum and alcohol. He showed the existence of a peculiar class of salts, of which a hydrocarbon and the chlorides are the elements ; he terms them *etherised salts*. Hellot a long time ago obtained a crystallisable compound of chloride of antimony and alcohol ; and later Lewy described an analogous compound of perchloride of tin and alcohol. The uses of alcohol in the arts, and its applications to various economical purposes, are extremely numerous ; to the chemist it is a most valuable form of fuel, but it is not freely used owing to its high price ; and for the same reason many manufactures, in which alcohol is an essential agent, cannot be economically carried on in this country. Its solvent power, in relation to resins, oils, and other organic products, has been elsewhere noted ; its employment for medicinal and pharmaceutical purposes are well known. It is the chief raw material used in the manufacture of ether, chloral, and chloroform. It is one of the essential raw materials in the manufacture of numerous intermediate products in coal-tar colour manufacture, and in the form of its radical ethyl enters into the constitution of both these and the finished coal-tar colours, and hundreds of other substances. (See Chaps. XII. and XIII.)

*Proof spirit* is defined by law (58 Geo. III. c. 28) to be such spirit “ as shall at the temperature of 51° by Fahrenheit’s thermometer weigh exactly  $\frac{1}{3}$ ths of an equal measure of distilled water.” The temperature of the distilled water is not specified, but there can be no doubt that its weight at 51° is intended to be taken as the standard. Taking water at 51° F. as unity, the specific gravity of “ proof spirit ” at 51° F. is .92308. When such spirit is raised to the more usual temperature of 60° F., its specific gravity, compared with water at 60° F., is .91984. To calculate the quantity of spirits at proof in a given quantity of spirit over or under proof strength : Multiply the quantity of spirit by the number of degrees of strength of the spirit, and divide the product by 100. The number of degrees of strength of any spirit is 100 *plus* the number of degrees overproof, or *minus* the number of degrees underproof.

*Example.*—19·8 gallons of spirit at 64·5 overproof.

$$100 + 64\cdot5 = 164\cdot5 \text{ proof strength.}$$

$$164\cdot5 \times 19\cdot8 \div 100 = 32\cdot571$$

taken as 32·5 gallons at proof.

The facility with which the hydrometer can be used is such as to render it the best instrument for determining the strength of alcohol for Excise purposes. A Committee of the Royal Society many years ago recommended to the Government a form of the instrument which they considered best adapted to the purpose, accompanied by proper tables. The following extract from their report needs no explanation: “With regard to the substance alcohol upon which the Excise duty is to be levied, there appears to be no reason either philosophical or practical why it should be considered as absolute; a definite mixture of alcohol and water is as invariable in its value as absolute alcohol can be. It is also invariable in its nature, and can be more readily, and with equal accuracy, identified by that only quality or condition to which recourse can be had in practice, namely, specific gravity. A diluted alcohol is therefore that which is recommended by us as the only excisable substance, and as, on the one hand, it will make no difference in the identification, and, on the other, will be a great commercial advantage, it is further recommended that the standard be very nearly that of the present *proof spirit*. The proposition of your Committee is that standard spirit be that which, consisting of water and alcohol alone, shall have a specific gravity of 0·92 at the temperature of 62° F., water being unity at that temperature, or, in other words, that it shall at 62° F. weigh  $\frac{92}{100}$ ths or  $\frac{23}{25}$ ths of an equal bulk of water at the same temperature. The temperature of 62° F. is recommended as the standard, because it was that at which water was taken in the late National survey and adjustment of weights and measures. The specific gravity of 0·92 is taken rather than 0·918633 (the specific gravity of present proof spirit at 62°), because the fraction expressing its relation to water is much more simple, and will facilitate the construction of the tables and the verification of the instruments proposed to be used. This definition of *standard spirit* appears to your Committee to be very simple, and yet as

exact as it can be, or as any *other* standard spirit can be. This standard is rather weaker than the old *proof spirit*, in the proportion of nearly 1·1 gallon of the present *proof spirit per cent*. But this disadvantage your Committee consider as trifling compared with the great convenience which will result if the specific gravity of 0·92 be taken rather than 0·918633. It may be interesting hereafter to ascertain what proportion of *absolute alcohol* enters into the composition of the recommended standard spirit, should the latter be adopted by the Government ; but the point possesses not the slightest practical importance in relation to the present question. The proposed standard is, in fact, more definite, more sure, and more ascertainable than that of the alcohol which it must contain. Philosophers are not yet agreed upon the density of *absolute alcohol*, and the differences of specific gravity assigned to it vary from ·7910 to ·7980. But assuming the truth to be somewhere within these extremes, the proposed standard would contain nearly one-half by weight of absolute alcohol. (·7947 at 59° F., Berzelius ; ·7960 at 60° F., Turner, from *Saussure?* ; ·7910 at 68° F., Brande ; ·7980, Chaussier ; ·79235 at 64° F., Gay-Lussac.) In any mixture of alcohol and water, the *specific gravity* appears to be the only quality or condition to which recourse can be had for the practical purposes of the Excise, in order to indicate the proportion of standard spirit present. Your Committee are of opinion that the *hydrometer* is the instrument best fitted in the hands of the Excise officer to indicate that specific gravity, and they think it ought to be so graduated as to give the indication of strength, not upon an arbitrary scale, but in terms of specific gravity at a fixed temperature, which in the present case should be 62° F., for that of the standard spirit. The graduation in terms of specific gravity will not only supply a very minute yet sensible scale for the purpose of ascertaining smaller differences in the density than is done by the present scale, but will also afford an easy means of verifying the instruments when required.”<sup>1</sup>

*Heat developed by and contraction ensuing on mixing alcohol*

<sup>1</sup> This recommendation of the Royal Society, made several generations ago, has never been adopted by the Excise. By the use of a hydrometer, with direct indications 0·800-0·850 according to the suggestions of the Royal Society, the strength over proof of any industrial alcohol is easily ascertained from Table II., pp. 20-22.

*and water.*—Equal mixtures of alcohol ( $D = 0.825$ ) and water each at  $10^{\circ} \text{C.}$  ( $= 50^{\circ} \text{F.}$ ) register, when suddenly mixed, a temperature of  $21.1^{\circ} \text{C.}$  ( $= 70^{\circ} \text{F.}$ ), and a mixture of equal measures of proof spirit and water each at  $10^{\circ} \text{C.}$  ( $= 50^{\circ} \text{F.}$ ) register, under like conditions,  $15.6^{\circ} \text{C.}$  ( $= 60^{\circ} \text{F.}$ ). On thus mixing alcohol and water the contraction increases till the mixture consists of 100 parts of alcohol and 116.23 of water. One hundred volumes of this mixture at  $59^{\circ}$  contains 53.739 of anhydrous alcohol and 49.836 of water; the condensation therefore amounts to 3.575. The specific gravity is 0.927 at  $15^{\circ} \text{C.}$  ( $59^{\circ} \text{F.}$ , Rudberg). From this point the contractions produced by fresh addition of water become more and more feeble, and terminate in apparent expansion. When equal volumes of dilute alcohol ( $D = 0.954$ ) and water are mixed the density becomes 0.9768, whereas if there were no expansion the density would be 0.9770.

TABLE I.—CONTRACTION OF MIXTURE PER CENT. ON DILUTING ALCOHOL WITH WATER.

A	B	A	B	A	B	A	B
100	0.0	75	3.19	50	3.745	25	2.24
95	1.18	70	3.44	45	3.64	20	1.72
90	1.94	65	3.615	40	3.44	15	1.20
85	2.47	60	3.73	35	3.14	10	0.72
80	2.87	55	3.77	30	2.72	5	0.31

A, volume of alcohol per cent. B, contractions in hundredths of the volume of the mixture when 100 per cent. alcohol is reduced by water to extent indicated in A.

The maximum of contraction, according to Dumas, indicates 55 per cent. of alcohol, but Rudberg's experiments place it at 54 per cent., which is equivalent to 23 parts by weight of alcohol and 27 of water = 1 molecule of alcohol  $\text{C}_2\text{H}_6\text{O}$  and 3 molecules of water. The absolute amount of the contraction varies with the temperature; according to Tralles, at  $4^{\circ} \text{C.}$  ( $39^{\circ} \text{F.}$ ) it amounts to 3.97, at  $11^{\circ} \text{C.}$  ( $52^{\circ} \text{F.}$ ) to 3.77, at  $18^{\circ} \text{C.}$  ( $64^{\circ} \text{F.}$ ) to 3.60, and at  $37^{\circ} \text{C.}$  ( $100^{\circ} \text{F.}$ ) to 3.31.

*Absolute alcohol.*—Absolute alcohol is prepared by rectifying

the alcohol of commerce by substances which take up water. Quick-lime is the substance most generally used. The alcohol is digested with a large quantity of quick-lime in a flask for two days. The latter is then connected with a Liebig's condenser, and the alcohol is distilled off. The quicklime does not appear to slake much. The first and last portions are rejected, because even when working with almost absolute alcohol the former contains a large proportion of water; whilst, on the other hand, owing to the high temperature the last portion is apt to contain water extracted from the calcium hydrate by the absolute alcohol. These two portions being collected apart, the remainder is absolute alcohol, potassium permanganate does not redden it, but imparts a faint brown tint. Instead of directly distilling the alcohol through a Liebig's condenser, the flask may be attached to a vertical reflux condenser, and boiled on the water-bath for an hour, when the condenser is changed to the ordinary position and the alcohol distilled off as before; in this case the lime, some lumps of which should originally have projected above the surface of the liquid, is completely disintegrated. Care must be taken not to use too much alcohol, as the heat generated by the slaking of the lime may cause such sudden and violent ebullition as to project a mixture of alcohol and lime through the condenser. When the alcohol originally contained more than 5 per cent. of water, a single rectification is not enough, and less quicklime must be used, otherwise the flask may be broken by the heat developed in slaking. Better results are obtained by digestion with, and distillation over, anhydrous barium oxide, made by decomposing the nitrate. A. Kailan found that on boiling alcohol of 92-93 per cent. strength with quicklime on the water bath for three and a half hours the strength was increased to 99.5 per cent., and after six hours to 99.9 per cent.

A very pure absolute alcohol is obtained on a larger scale by simple filtration through quicklime. Any convenient apparatus may be used, such as an inverted two-gallon tin can, the bottom being removed and a lid fitted in its place, the cylindrical part of the can being lined inside with a cylindrical perforated vessel, in the centre of which a tube is fixed. The can acts as a jacket.

The inner vessel is filled with quicklime and as much alcohol as it will hold. The central tube communicates with the interior of the jacket. The inverted neck of the can is fitted with a stop-cock for running off the dehydrated alcohol after being left in contact for fourteen days.

In the manufacture of absolute alcohol by very slow, cold percolations through large successive portions of quicklime, it not unfrequently comes from the rectifying still of a specific gravity below that given in the tables of the best and most recent authorities; and the entire product of the process for years has been of such strength that all the hydrometers tried have sunk below the reading scale. A U.S.A. Government Inspector pronounced the alcohol to be in one case 102 per cent. strong! Another inspector made it 99.8 per cent., but he could not possibly have done this with his official instruments, because his hydrometer would sink below the reading scale, even when the alcohol had been exposed to the air in several trials. From these observations upon alcohol that could not be completely anhydrous, since very strong alcohol takes moisture from the air very rapidly indeed, and changes proportionately in specific gravity, Squibb concluded that the figures in the tables were all too high for the present time, and reinvestigated the subject, Table III. giving the results. In this case metallic sodium was used to remove the last traces of alcohol. A small piece is dissolved in the alcohol, and the whole distilled at a steam heat. The sodium forms ethylate of sodium with disengagement of hydrogen, whilst the traces of water which the alcohol contains decompose the ethylate with the formation of caustic soda and alcohol.



Sodic Ethylate and Water = Caustic Soda and Alcohol.

But according to Mendelejeff, when either sodium or sodium amalgam are used to dehydrate alcohol, traces of sodium or sodium and mercury are found in the distillate. Potassium carbonate has been used, but it is too weak a dehydrating agent (see p. 10).

According to a German patent,<sup>1</sup> alcohol of 90 per cent. strength can be rapidly and completely dehydrated by the action of anhydrous sodium sulphide. The sulphide is not affected in any way by the alcohol, and it can be recovered by heating the residue till all the water is driven off.

The phlegms furnished by the best distilling columns are far from being a mixture of water and pure alcohol only. Amongst the substances which deteriorate them the following may be mentioned: Propylic, butylic, isoamylic, and hexylic alcohols, the aldehydes of ethylic and homologous alcohols, acetone, glycols, acrolein, furfural, the acids produced by the oxidation of these different alcohols, or from the reduction of pre-existing acids, pelargonic acid, etc., the ethers (esters or ethereal salts) produced by all the possible combinations of all these alcohols with the various acids. If some of these bodies by their presence impart to the liquors a flavour and aroma which constitute their value, the majority, on the contrary, are noxious in a high degree and very unpleasant to taste and smell. The industrial alcohol distiller is therefore in duty bound to eliminate them to satisfy both hygiene and his customers' requirements. It cannot be done by chemical processes. Continuous rectification and "pasteurisation" is alone effectual.

*Assay of alcohol.*—(1) *Water* may be detected by adding anhydrous sulphate of copper, which changes from greyish white to blue in dilute alcohol, but this reaction fails to detect minute proportions of water. Slight traces of water may be detected by adding a small amount of the alcohol to be tested to a saturated alcoholic solution of liquid paraffin; in presence of traces of water the liquid becomes turbid immediately (Crismer). Also by permanganate of potash, which under like circumstances turns red. (2) *Amylic alcohol* (fusel oil) may be detected by agitating 5 c.c. of the alcohol to be tested with 6 c.c. of water and 15 to 20 drops of chloroform. The chloroformic solution is decanted and evaporated, leaving the fusel oil; about 0.05 per cent. may be detected in this manner. (3) Impurities in the first runnings may be recognised and roughly estimated by aid of the reaction of

<sup>1</sup> Chem. Fabr. Griesheim—Elektron Ger. Pat. 236591, Dec. 22, 1909.

rosaniline bisulphite on the *aldehydes* which it always contains. (4) *Furfurol* may be recognised and approximately estimated by the red reaction which it gives with aniline acetate, and by its reaction with a solution of phloroglucinol, with which it gives a blackish-green precipitate. Savalle used an acid reagent which he described as very satisfactory as regards rapidity and exactitude in detecting and estimating *en bloc* the impurities in an alcohol; 10 c.c. of the alcohol are run into a small flask with 10 c.c. of the reagent, and the whole is heated over a spirit lamp with constant shaking. As soon as the liquid boils, the heat is withdrawn, and the whole is run into one of the empty bottles in the case. The tint of the liquid is then compared with that of a typical plate to get the percentage of impurities.

MOHLER'S METHOD FOR THE ANALYSIS OF COMMERCIAL ALCOHOL.—There are well-known methods for determining extract, alcohol, acids, and furfurol. The method now described renders it possible in half-litre samples to determine also the ethers, the aldehydes, the higher alcohols, and the nitrogenous products. These determinations have to be made on the distilled liquid brought to the standard of 50° G.L., except as regards the nitrogenous products, which are determined in the sample itself.

(a) *Acidity*.—The total acidity is determined by means of a standard solution of calcium succrate (100 c.c. = 1 gram of acetic acid), the alcohol being diluted with boiled distilled water to a strength of about 50 per cent. Litmus solution is used as indicator, and the comparison is made with boiled distilled water to which an equal volume of the litmus solution has been added (Barbet).

(b) *Determination of ethers*.—Boil 100 c.c. of the distilled alcohol for an hour along with 20 c.c. of decinormal potash (Barbet uses a standard solution of calcium succrate which does not resinify aldehydes), the flask being fitted with an ascending condenser. The alkaline liquid is titrated back by decinormal acid, and the results are calculated as ethyl acetate. A correction should be applied for any free acid present which is determined in the cold.

(c) *Aldehydes*.—The intensity of the violet colour developed

by the action of rosaniline bisulphite upon alcohols containing aldehydes is proportional to the quantity in solution. To apply this reagent to the determination of the aldehydes it must be caused to act upon a solution of known strength, and to bring the alcohol to be analysed by dilution to contain a quantity of aldehyde equal to that of the standard. To 10 c.c. of a solution of ethyl aldehyde at a strength of  $\frac{1}{10000}$ , and to 10 c.c. of the alcohol under analysis (both at 50° of alcoholometric strength), add at the same time 4 c.c. of rosaniline bisulphite. The tints are allowed to develop for twenty minutes, and their intensity is then compared by means of the Duboscq colorimeter. The operation is recommenced by diluting the alcohol in question until the colours have the same intensity. If  $m$  represents this dilution, the weight of ethylic aldehyde per litre will be  $m \times 0.050$ . Furfurol is estimated colorimetrically by the depth of colour produced by aniline acetate. The colour reactions with *m*-phenylene diamine and quinol-sulphuric acid are also employed.

(*d*) *Higher alcohols*.—Sulphuric acid in the conditions in which it is employed acts only on the aldehydes and the higher alcohols. The aldehydes are kept back by means of aniline phosphate or meta-phenylene diamine hydrochloride. To 100 c.c. of the distilled sample add 1 c.c. of aniline and 1 c.c. of phosphoric acid at 45° B. The liquid is boiled for an hour with an ascending condenser, and is then distilled to dryness in a salt bath. The distillate is treated with 10 c.c. of sulphuric acid of specific gravity 1.84 (168° Tw.), and the mixture heated at 120° C. in a chloride of calcium bath for one hour, and the tint observed is examined comparatively in the colorimeter with that given by an alcoholic solution containing 0.250 isobutylic alcohol per litre, operating, as in the case of the alcohols, by diluting the alcohol until the tints are equalised. If the results are to be calculated to amyl alcohol, multiply by 1.6.

*Separation of the aldehydes : Girard and Rocques' method*.—The authors dissolve in 200 c.c. of alcohol, at 50°, 3 grms. of meta-phenylene-diamine hydrochloride, and boil for half an hour with an ascending condenser. The liquid assumes a pale yellow colour. It is allowed to cool for half an hour and slightly stirred

towards the end of this time. The colour of the liquid darkens, and if aldehyde is present it assumes a fine green fluorescence. It is then distilled quickly, and 125 c.c. of distilled alcohol are collected, marking 75°. This is then submitted to Savalle's test, and the tints obtained are compared with those given by alcohol at 75°, to which known quantities of pure amyl alcohol have been added.

(e) *Nitrogenous products*.—The weight of ammonia corresponding, on the one hand, to the amides and to saline ammonia, and on the other to the pyridine bases and the alkaloids, is determined by submitting the alcohol in question first to the action of sodium carbonate, and then to that of alkaline permanganate, titrating the small quantities of ammonia produced in each operation with Nessler's reagent. To 100 c.c. of the sample not distilled add 2 c.c. of phosphoric acid at 45° B., and expel all the alcohol by boiling. The phosphoric solution of the bases is diluted with about 1 litre distilled water; 10 grms. of pure sodium carbonate are added, and the mixture is distilled until no more ammonia passes over. The permanganate and the potash are then introduced, and the distillation is continued, the ammoniacal water being collected in another receiver. The ammonia obtained from each operation is determined with Nessler's reagent comparatively with a solution containing 0.00001 gm. ammonium chloride per c.c. By the method just described 500 c.c. samples of alcohol (containing not more than 1-200,000th of acids, 1-200,000th of aldehydes, 1-1,000,000th of ethers, 1-1,000,000th of furfural, 1-20,000th of higher alcohols, and 1-100,000th on operating with alcohol at 90°, 1-1,000,000th of ammonia corresponding to saline ammonia and amides, and 1-10,000,000th of ammonia corresponding to the alkaloids and to pyridine bases) may be analysed.

*Methyl Alcohol: Detection*.—Many tests have been devised for the detection of methyl alcohol in presence of ethyl alcohol, one of the best being that of Denigès.<sup>1</sup> This author found that on oxidation with potassium permanganate ethyl alcohol is converted into acetaldehyde, while methyl alcohol is oxidised to formaldehyde, which can readily be detected by the colouration

<sup>1</sup> *Comptes. rend.*, 1910, 529-531, 832-834.

produced with Schiff's reagent. This reagent, as ordinarily prepared, is a solution of magenta decolourised by sulphurous acid,

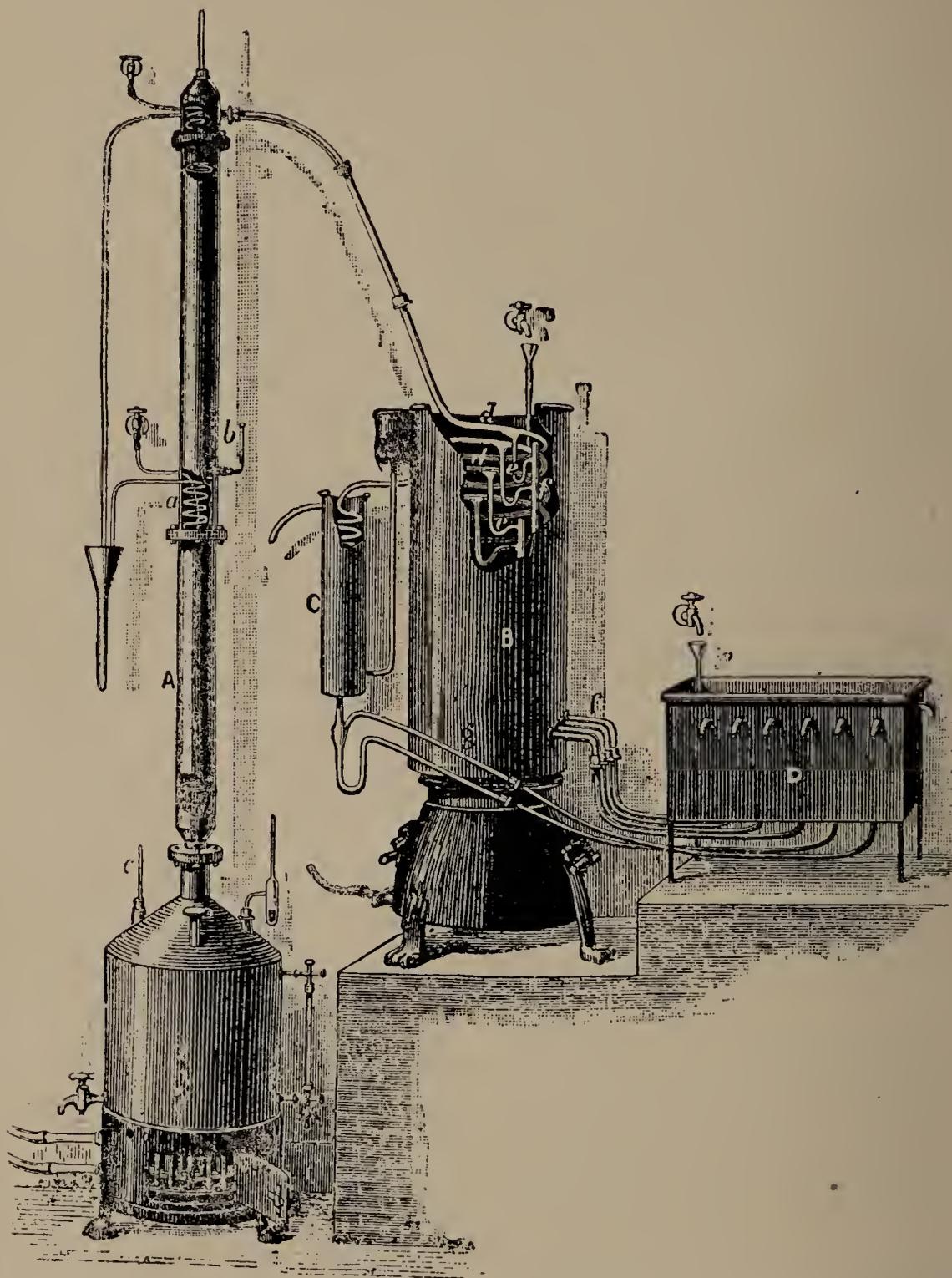


FIG. 1.—Laboratory Rectifier (SOREL). A, rectifying column; *a*, auxiliary refrigerator; *b*, thermometer; B, condenser No. 1 regulated to  $77.5^{\circ}$  C.; *dd*, coil of 10 spirals, each with S tube (*e*) for products condensed therein; *ff*, separate collectors of four different lots from *e* (viz. 1 and 2; 3 and 4; 5, 6, and 7; and 8, 9, and 10), so that bulk from each pipe (*f*) is appreciably equal; C, hot condenser No. 2 fed by water from B; *g*, pipe for vapour from C (liquefied in refrigerator D).

and with aldehydes generally it gives an intense rose-red colour. Denigès discovered that if the solution be made strongly acid with sulphuric acid, the colour produced by acetaldehyde fades away in a few hours, while that of formaldehyde is permanent.

The delicacy of the test is such that 1 part of formaldehyde in 1000 parts of acetaldehyde can be detected.

The reagent for testing is prepared by dissolving 1 gram of magenta and 20 c.c. of a saturated solution of sodium bisulphite in distilled water and making up to 1 litre ; after standing about ten minutes 20 c.c. of hydrochloric acid (specific gravity 1.18) is added, and in an hour or so the solution will be decolourised and may then be used.

The method of carrying out the test is as follows : 0.1 c.c. of the alcohol to be tested and 5 c.c. of a 1 per cent. solution of potassium permanganate are mixed in a test tube, and about 0.2 c.c. of pure sulphuric acid added ; the mixture is well shaken for a few minutes, and then 1 c.c. of an 8 per cent. solution of oxalic acid is added, and the mixture again well shaken. The liquid is acidified with 1 c.c. of pure sulphuric acid, and 5 c.c. of the bisulphite-magenta solution immediately added. A rose-red colour will appear, which, if permanent, shows the presence of formaldehyde and indicates methyl alcohol. A rough estimation of the amount of methyl alcohol can be arrived at by estimating the depth of colour.

Aweng's method<sup>1</sup> is a modification of Denigès', the procedure being the same in all respects, except that in place of the bisulphite magenta solution, phenylhydrazine hydrochloride and ferric chloride are employed for the detection of the formaldehyde. To the decolourised liquid 2 c.c. of a 4 per cent. solution of phenylhydrazine hydrochloride, 6 drops of 10 per cent. solution of ferric chloride, and 5 c.c. of strong hydrochloric acid are added. With acetaldehyde only a pale yellow colouration is produced, but formaldehyde gives a bright rose-red colour. Other methods of confirming the presence of methyl alcohol are also given by Aweng in the reference quoted, but these need not be referred to further here, as the above tests are sufficiently characteristic to serve for the purpose.<sup>2</sup>

*Estimation.*—The amount of methyl alcohol in spirits can be

<sup>1</sup> *Apoth.-Zeit*, 1912, 27, 159.

<sup>2</sup> For further information on this subject, see *Jour. Soc. Chem. Ind.*, 1905, 128 ; 1909, 23 ; 1910, 451, 585 ; 1912, 296 ; 1915, 812 ; 1919, 874A.

accurately estimated by the method of T. E. Thorpe and J. Holmes,<sup>1</sup> which is carried out as follows: The liquid to be tested is first diluted till it contains no more than 1 gram of methyl alcohol or 4 grams of the mixed alcohols in 50 c.c. 50 c.c. of the solution is then treated with 20 grams of potassium bichromate and 80 c.c. of sulphuric acid (diluted 1 to 4) in a stoppered flask, which is fitted with a stoppered funnel and delivery tube. After standing for about 18 hours, 10 grams more of potassium bichromate and 50 c.c. of sulphuric acid (1 : 1) are added and the mixture boiled for ten minutes, while a current of air is drawn through the liquid. The vapours from the flask pass through drying tubes and then through weighed soda lime tubes in which the carbonic acid evolved is absorbed. The increase in weight due to the carbonic acid is calculated to methyl alcohol. The authors found that pure ethyl alcohol is oxidised to acetic acid with the exception of 0.5 per cent., which is converted into carbonic acid, so that a correction of 0.01 gram must be subtracted from the weight of carbonic acid for each gram of ethyl alcohol present. Methyl alcohol is entirely converted into carbonic acid. The total percentage of alcohols can be determined in the usual manner by taking the density, the specific gravities of ethyl alcohol and methyl alcohol being practically identical.<sup>2</sup>

Commercial "absolute" alcohol always contains water, sometimes as much as 1 to 2 per cent. ; in addition to this, aldehyde is, according to J. B. Tingle, frequently present in varying quantity. The methods of purification in general use in laboratories, such as distillation over lime, barium oxide, sodium, etc., although adequate to remove the water, fail to affect the aldehyde, and it cannot be eliminated by fractionation.<sup>3</sup> The problem of its

<sup>1</sup> *Trans. Chem. Soc.*, 1904, 1-6.

<sup>2</sup> For a full account of the methods used in the analyses of spirits see E. A. Mann and C. E. Stracey, *Jour. Soc. Chem. Ind.*, 1907, 287, 450.

<sup>3</sup> There is no necessity for every chemist to be his own absolute alcohol purifier, nor for commercial absolute alcohol to contain even a trace of aldehyde, as the following analysis by Delbruck of the industrial alcohol from one of Barbet's continuous distillation rectification stills of 176 gallons capacity per hour in this case shows:

Acids . . . .	Nil.	Colour .	Transparent like water.
Aldehydes . . .	Nil.	Odour .	Fine.
Savalle's test .	Colourless.	Taste .	Pure.

Percentage of alcohol, 94.4 per cent. by weight at 15° = 96.39 by volume.

Such commercial alcohols when rendered absolute should give a chemically pure alcohol without the necessity of any such chemical treatment as that suggested by Winkler.

removal has been attacked recently by L. W. Winkler<sup>1</sup> in the following manner: Silver oxide, prepared from the nitrate, is well washed and dried at the ordinary temperature. It is then triturated with a little of the alcohol and the thin paste added to the remainder. The quantity of oxide used depends, of course, on the particular sample of alcohol; it need not exceed a few grammes to the litre, and may be less. To neutralise the acetic acid which is produced, potassium hydroxide, 1 to 2 grams per litre, is added; the mixture is frequently shaken and allowed to remain for several days at the ordinary temperature, until a portion of the alcohol fails to give the test for aldehyde, viz., a silver mirror, with ammoniacal nitrate of silver solution.

Winkler recommends metallic calcium, in the form of filings, for the removal of water from alcohol; 20 grams are usually sufficient to dehydrate 1 litre of commercial "absolute" alcohol. The substances are mixed, and boiled in a distillation flask with a reversed condenser, and then distilled; cork connections must not be used. The product has a purity of 99.9 per cent. A second treatment with 0.5 per cent. of its weight of calcium appears to remove the last trace of water, because a third treatment, also with 0.5 per cent. of calcium, was found to produce no further change. Certain physical properties of alcohol, purified in this manner and fractionally distilled, were determined with the following results: Specific gravity at 0° = 0.80629, at 10° = 0.79787, at 15° = 0.79363, at 20° = 0.78937; these figures are reduced to a vacuum, and referred to water at 4°. The corresponding values given by Mendeleeff are 0.80625, 0.79788, 0.79367, and 0.78945 respectively. The boiling points are 77.81° (743.5 mm.), 78.20° (754.9 mm.), 78.29° (757.8 mm.), and 78.37° (760 mm.); therefore a difference of 1 mm. pressure causes a change of 0.034° in the boiling-point. Winkler mentions two rather curious facts which he observed in the course of his work.

As to its being impossible to free alcohol from aldehyde by ordinary fractionation in a laboratory, that is possibly impracticable, even in Sorel's or in Claudon Merin's laboratory rectifiers (Fig. 1). But there is no reason why it should not be freed from aldehyde, etc., in Barbet's laboratory rectifier constructed on the same principle as his continuous rectifiers. The results obtained by Sorel's apparatus prove that the condenser BC is not an analyser, as generally believed. The analysis is effected on the plates (Figs. 54 and 59).

<sup>1</sup> *Ber.*, 1905, xxxviii., 3612.

The reaction between calcium and alcohol is the more vigorous the less water is present below 5 per cent., but ordinary alcohol, containing 5 to 10 per cent. of water, also attacks calcium with considerable energy. A. Kailon<sup>1</sup> has also determined the specific gravity of alcohol dehydrated by metallic calcium and found it to be 0.78513 at  $\frac{25^\circ}{4^\circ}$  C. Corresponding figures for Winckler and Mendeleeff are 0.78509 and 0.78522. Alcohol absolutely free from water is not nearly so hygroscopic as is usually supposed. For example, 200 c.c. of it were allowed to remain in an uncovered beaker, exposed to the air of the laboratory, for fifteen minutes; it was then found that the amount of water which had been absorbed was less than 0.1 per cent. (Tingle).

TABLE II.—SHOWING THE RELATIONSHIP BETWEEN THE SPECIFIC GRAVITY AND PER CENT. OF ALCOHOL OVER AND UNDER PROOF AT 60° F. (URE).

Specific Gravity	PerCent. over Proof.	Specific Gravity.	PerCent. over Proof.	Specific Gravity.	PerCent. over Proof	Specific Gravity.	PerCent. over Proof.
0.8156	67.0	.8252	62.3	.8347	57.5	.8441	52.5
.8160	66.8	.8256	62.2	.8351	57.3	.8445	52.3
.8163	66.6	.8259	62.0	.8354	57.1	.8448	52.1
.8167	66.5	.8263	61.8	.8358	56.9	.8452	51.9
.8170	66.3	.8266	61.6	.8362	56.8	.8455	51.7
.8174	66.1	.8270	61.4	.8365	56.6	.8459	51.5
.8178	65.6	.8273	61.3	.8369	56.4	.8462	51.3
.8181	65.8	.8277	61.1	.8372	56.2	.8465	51.1
.8185	65.6	.8280	60.9	.8376	56.0	.8469	50.9
.8188	65.5	.8284	60.7	.8379	55.9	.8472	50.7
.8192	65.3	.8287	60.5	.8383	55.7	.8476	50.5
.8196	65.1	.8291	60.4	.8386	55.5	.8480	50.3
.8199	65.0	.8294	60.2	.8390	55.3	.8482	50.1
.8203	64.8	.8298	60.0	.8393	55.1	.8486	49.9
.8206	64.7	.8301	59.8	.8396	55.0	.8490	49.7
.8210	64.5	.8305	59.6	.8400	54.8	.8493	49.5
.8214	64.3	.8308	59.5	.8403	54.6	.8496	49.3
.8218	64.1	.8312	59.3	.8407	54.4	.8499	49.1
.8221	64.0	.8315	59.1	.8410	54.2	.8503	48.9
.8224	63.8	.8319	58.9	.8413	54.1	.8506	48.7
.8227	63.6	.8322	58.7	.8417	53.9	.8510	48.5
.8231	63.4	.8326	58.6	.8420	53.7	.8513	48.3
.8234	63.2	.8329	58.4	.8424	53.5	.8516	48.0
.8238	63.1	.8333	58.2	.8427	53.3	.8520	47.8
.8242	62.9	.8336	58.0	.8431	53.1	.8523	47.6
.8245	62.7	.8340	57.8	.8434	52.9	.8527	47.4
.8249	62.5	.8344	57.7	.8438	52.7	.8530	47.2

<sup>1</sup> Ber., 1911, 44 2881-2884.

TABLE II.—Continued.

Specific Gravity.	PerCent. over Proof.						
.8533	47.0	.8702	36.4	.8876	24.5	.9056	11.4
.8537	46.8	.8706	36.2	.8879	24.3	.9060	11.1
.8540	46.6	.8709	35.9	.8883	24.0	.9064	10.8
.8543	46.4	.8713	35.7	.8886	23.8	.9067	10.6
.8547	46.2	.8716	35.5	.8890	23.5	.9071	10.3
.8550	46.0	.8720	35.2	.8894	23.2	.9075	10.0
.8553	45.8	.8723	35.0	.8897	23.0	.9079	9.7
.8556	45.6	.8727	34.7	.8901	22.7	.9082	9.4
.8560	45.4	.8730	34.5	.8904	22.5	.9085	9.2
.8563	45.2	.8734	34.3	.8908	22.2	.9089	8.9
.8566	45.0	.8737	34.1	.8912	21.9	.9093	8.6
.8570	44.8	.8741	33.8	.8915	21.7	.9097	8.3
.8573	44.6	.8744	33.6	.8919	21.4	.9100	8.0
.8577	44.4	.8748	33.4	.8922	21.2	.9104	7.7
.8581	44.2	.8751	33.2	.8926	20.9	.9107	7.4
.8583	43.9	.8755	32.9	.8930	20.6	.9111	7.1
.8587	43.7	.8758	32.7	.8933	20.4	.9115	6.8
.8590	43.5	.8762	32.4	.8937	20.1	.9118	6.5
.8594	43.3	.8765	32.2	.8940	19.9	.9122	6.2
.8597	43.1	.8769	32.0	.8944	19.6	.9126	5.9
.8601	42.8	.8772	31.7	.8948	19.3	.9130	5.6
.8604	42.6	.8776	31.5	.8951	19.1	.9134	5.3
.8608	42.4	.8779	31.2	.8955	18.8	.9137	5.0
.8611	42.2	.8783	31.0	.8959	18.6	.9141	4.8
.8615	42.0	.8786	30.8	.8962	18.3	.9145	4.5
.8618	41.7	.8790	30.5	.8966	18.0	.9148	4.2
.8622	41.5	.8793	30.3	.8970	17.7	.9152	3.9
.8625	41.3	.8797	30.0	.8974	17.5	.9156	3.6
.8629	41.1	.8800	29.8	.8977	17.2	.9159	3.3
.8632	40.9	.8804	29.5	.8981	16.9	.9163	3.0
.8636	40.6	.8807	29.3	.8985	16.6	.9167	2.7
.8639	40.4	.8811	29.0	.8989	16.4	.9170	2.4
.8643	40.2	.8814	28.8	.8992	16.1	.9174	2.1
.8646	40.0	.8818	28.5	.8996	15.9	.9178	1.9
.8650	39.8	.8822	28.3	.9000	15.6	.9182	1.6
.8653	39.5	.8825	28.0	.9004	15.3	.9185	1.3
.8657	39.3	.8829	27.8	.9008	15.0	.9189	1.0
.8660	39.1	.8832	27.5	.9011	14.8	.9192	0.7
.8664	38.9	.8836	27.3	.9015	14.5	.9196	0.3
.8667	38.7	.8840	27.0	.9019	14.2	.9200	Proof.
.8671	38.4	.8843	26.8	.9023	13.9	Under Proof.	
.8674	38.2	.8847	26.5	.9026	13.6	.9204	0.3
.8678	38.0	.8850	26.3	.9030	13.4	.9207	0.6
.8681	37.8	.8854	26.0	.9034	13.1	.9210	0.9
.8685	37.6	.8858	25.8	.9038	12.8	.9214	1.3
.8688	37.3	.8861	25.5	.9041	12.5	.9218	1.6
.8692	37.1	.8865	25.3	.9045	12.2	.9222	1.9
.8695	36.9	.8869	25.0	.9049	12.0	.9226	2.2
.8699	36.7	.8872	24.8	.9052	11.7	.9229	2.5

[TABLE CONTINUED.]

TABLE II.—*Concluded.*

Specific Gravity.	PerCent. under Proof.	Specific Gravity.	PerCent. under Proof	Specific Gravity.	PerCent. under Proof.	Specific Gravity.	PerCent. under Proof.
.9233	2.8	.9426	20.0	.9615	41.7	.9810	73.5
.9237	3.1	.9430	20.4	.9619	42.2	.9814	74.1
.9241	3.4	.9434	20.8	.9623	42.8	.9816	74.8
.9244	3.7	.9437	21.2	.9627	43.3	.9822	75.4
.9248	4.0	.9441	21.6	.9631	43.9	.9826	76.1
.9252	4.4	.9445	21.9	.9635	44.4	.9830	76.7
.9255	4.7	.9448	22.2	.9638	45.0	.9834	77.3
.9259	5.0	.9452	22.7	.9642	45.5	.9838	78.0
.9263	5.3	.9456	23.1	.9646	46.1	.9842	78.6
.9267	5.7	.9460	23.5	.9650	46.7	.9846	79.2
.9270	6.0	.9464	23.9	.9654	47.3	.9850	79.8
.9274	6.4	.9468	24.3	.9657	47.9	.9854	80.4
.9278	6.7	.9472	24.7	.9661	48.5	.9858	81.1
.9282	7.0	.9476	25.1	.9665	49.1	.9862	81.7
.9286	7.3	.9480	25.5	.9669	49.7	.9866	82.3
.9291	7.7	.9484	25.9	.9674	50.3	.9870	82.9
.9295	8.0	.9488	26.3	.9677	51.0	.9874	83.5
.9299	8.3	.9492	26.7	.9681	51.6	.9878	84.0
.9302	8.6	.9496	27.1	.9685	52.2	.9882	84.6
.9306	9.0	.9499	27.5	.9689	52.9	.9886	85.2
.9310	9.3	.9503	28.0	.9693	53.3	.9890	85.8
.9314	9.7	.9507	28.4	.9697	54.2	.9894	86.3
.9318	10.0	.9511	28.8	.9701	54.8	.9898	86.9
.9322	10.3	.9515	29.2	.9705	55.5	.9902	87.4
.9326	10.7	.9519	29.7	.9709	56.2	.9906	88.0
.9329	11.0	.9522	30.1	.9713	56.9	.9910	88.5
.9332	11.4	.9526	30.6	.9718	57.6	.9914	89.1
.9337	11.7	.9530	31.0	.9722	58.3	.9918	89.6
.9341	12.1	.9534	31.4	.9726	59.0	.9922	90.2
.9345	12.4	.9539	31.8	.9730	59.7	.9926	90.7
.9349	12.8	.9542	32.3	.9734	60.4	.9930	91.2
.9353	13.1	.9546	32.8	.9738	61.1	.9934	91.7
.9357	13.5	.9550	33.2	.9742	61.8	.9938	92.3
.9360	13.9	.9553	33.7	.9746	62.5	.9942	92.8
.9364	14.2	.9557	34.2	.9750	63.2	.9946	93.3
.9368	14.6	.9561	34.6	.9754	63.9	.9950	93.8
.9372	14.9	.9565	35.1	.9758	64.6	.9954	94.3
.9376	15.3	.9569	35.6	.9762	65.3	.9958	94.9
.9380	15.7	.9573	36.1	.9766	66.0	.9962	95.4
.9384	16.0	.9577	36.6	.9770	66.7	.9966	95.9
.9388	16.4	.9580	37.1	.9774	67.4	.9970	96.4
.9392	16.7	.9584	37.6	.9778	68.0	.9974	96.8
.9396	17.1	.9588	38.1	.9782	68.7	.9978	97.3
.9399	17.5	.9592	38.6	.9786	69.4	.9982	97.7
.9403	17.8	.9596	39.1	.9790	70.1	.9986	98.2
.9407	18.2	.9599	39.6	.9794	70.8	.9990	98.7
.9411	18.5	.9603	40.1	.9798	71.4	.9993	99.1
.9415	18.9	.9607	40.6	.9802	72.1	.9997	99.6
.9419	19.3	.9611	41.1	.9806	72.8	1.0000	100.0
.9422	19.7						

TABLE III.

Showing the specific gravities of mixtures of alcohol and water, ascertained by actual observation at intervals of 4 per cent., on Squibb's basis for absolute alcohol of sp. gr. 0.79350, at 15.6° C. = 60° F. compared with water at 15.6° C. = 60° F. as unity; and showing both the apparent and corrected specific gravities at different temperatures by the two common standards for unity, namely, water at its maximum density of 4° C. = 39°.2 F. and at 15.6° C. = 60° F. The Table is sufficiently correct to be practically useful to the fourth decimal place (Squibb).

Per Cent. by Weight.	COMPARED WITH WATER AT 4° C. = 39.2° F. AS UNITY. Weighed at—						COMPARED WITH WATER AT 15.6° C. = 60° F. AS UNITY. Weighed at—		
	15° C. = 59° F.		15.6° C. = 60° F.		25° C. = 77° F.		15.6° C. = 60° F.		Corrected for Expansion of Glass.
	True.	Apparent.	Corrected for Expansion of Glass.	Apparent.	Corrected for Expansion of Glass.	Apparent.	Corrected for Expansion of Glass.	True.	
100	0.80257	0.79348	0.79326	0.79301	0.79279	0.78537	0.78496	0.79350	0.78573
99	0.80579	0.79667	0.79645	0.79618	0.79595	0.78847	0.78806	0.79669	0.78882
98	0.80875	0.79965	0.79943	0.79916	0.79893	0.79131	0.79089	0.79967	0.79186
96	0.81467	0.80555	0.80533	0.80509	0.80486	0.79744	0.79702	0.80558	0.79782
92	0.82593	0.81680	0.81658	0.81631	0.81607	0.80865	0.80823	0.81684	0.80896
88	0.83649	0.82751	0.82728	0.82699	0.82675	0.81929	0.81886	0.82755	0.81963
84	0.84681	0.83770	0.83747	0.83719	0.83695	0.82953	0.82909	0.83775	0.82987
80	0.85683	0.84773	0.84750	0.84718	0.84694	0.83959	0.83915	0.84779	0.84001
76	0.86655	0.85742	0.85718	0.85699	0.85674	0.84937	0.84892	0.85749	0.84971
72	0.87600	0.86702	0.86678	0.86649	0.86624	0.85895	0.85850	0.86711	0.85933
68	0.88516	0.87655	0.87631	0.87607	0.87582	0.86847	0.86801	0.87665	0.86885
64	0.89479	0.88625	0.88601	0.88578	0.88552	0.87832	0.87786	0.88636	0.87863
60	0.90401	0.89549	0.89524	0.89506	0.89480	0.88766	0.88719	0.89561	0.88800
56	0.91297	0.90452	0.90427	0.90405	0.90379	0.89687	0.89640	0.90465	0.89724
52	0.92177	0.91349	0.91324	0.91309	0.91283	0.90597	0.90549	0.91365	0.90632
48	0.93045	0.92231	0.92206	0.92187	0.92160	0.91489	0.91441	0.92247	0.91525
44	0.93875	0.93082	0.93056	0.93045	0.93018	0.92361	0.92313	0.93101	0.92405
40	0.94655	0.93901	0.93875	0.93865	0.93838	0.93217	0.93168	0.93923	0.93253

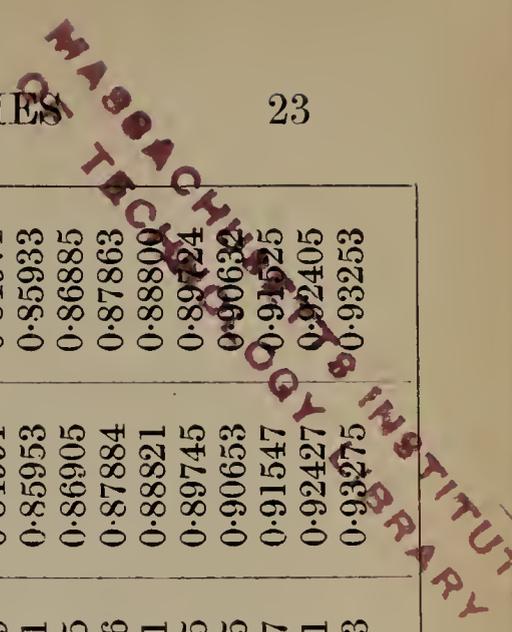


TABLE IV.

DENSITY AND PERCENTAGE OF ALCOHOL BY VOLUME AND PERCENTAGE BY WEIGHT AT 15.56° C. (TRALLES). WATER = 0.9991.

Percentage of Alcohol by Volume.	Percentage of Alcohol by Weight.	Density at 15.56° C.	Percentage of Alcohol by Volume.	Percentage of Alcohol by Weight.	Density at 15.56° C.	Percentage of Alcohol by Volume.	Percentage of Alcohol by Weight.	Density at 15.56° C.
0	0	0.9991	34	28.13	0.9596	68	60.38	0.8941
1	0.80	0.9976	35	28.99	0.9583	69	61.42	0.8917
2	1.60	0.9961	36	29.86	0.9570	70	62.50	0.8892
3	2.40	0.9947	37	30.74	0.9556	71	63.58	0.8867
4	3.20	0.9933	38	31.62	0.9541	72	64.66	0.8842
5	4.00	0.9919	39	32.50	0.9526	73	65.74	0.8817
6	4.81	0.9906	40	33.39	0.9510	74	66.83	0.8791
7	5.62	0.9893	41	34.28	0.9494	75	67.93	0.8765
8	6.43	0.9881	42	35.18	0.9478	76	69.05	0.8739
9	7.24	0.9869	43	36.08	0.9461	77	70.18	0.8712
10	8.05	0.9857	44	36.99	0.9444	78	71.31	0.8685
11	8.87	0.9845	45	37.90	0.9427	79	72.45	0.8658
12	9.69	0.9834	46	38.82	0.9409	80	73.59	0.8631
13	10.51	0.9823	47	39.74	0.9391	81	74.74	0.8603
14	11.33	0.9812	48	40.66	0.9373	82	75.91	0.8575
15	12.15	0.9802	49	41.59	0.9354	83	77.09	0.8547
16	12.98	0.9791	50	42.52	0.9335	84	78.29	0.8518
17	13.80	0.9781	51	43.47	0.9315	85	79.50	0.8488
18	14.63	0.9771	52	44.42	0.9295	86	80.71	0.8458
19	15.46	0.9761	53	45.36	0.9275	87	81.94	0.8428
20	16.28	0.9751	54	46.32	0.9254	88	83.19	0.8397
21	17.11	0.9741	55	47.29	0.9234	89	84.46	0.8365
22	17.95	0.9731	56	48.26	0.9213	90	85.75	0.8332
23	18.78	0.9720	57	49.26	0.9192	91	87.09	0.8299
24	19.62	0.9710	58	50.21	0.9170	92	88.37	0.8265
25	20.46	0.9700	59	51.20	0.9148	93	89.71	0.8230
26	21.30	0.9689	60	52.20	0.9126	94	91.07	0.8194
27	22.14	0.9679	61	53.20	0.9104	95	92.46	0.8157
28	22.99	0.9668	62	54.21	0.9082	96	93.89	0.8118
29	23.84	0.9657	63	55.21	0.9059	97	95.34	0.8077
30	24.69	0.9646	64	56.22	0.9036	98	96.84	0.8034
31	25.55	0.9634	65	57.24	0.9013	99	98.39	0.7988
32	26.41	0.9622	66	58.27	0.8989	100	100.00	0.7939
33	27.27	0.9609	67	59.32	0.8965			

N. S. Osborne, E. C. McKelvey, and H. W. Bearce<sup>1</sup> have determined the specific gravities of several samples of pure alcohol prepared by different methods, selecting those which appeared to be homogeneous on fractionation. They state that alcohol dehydrated by metallic calcium has a slightly higher specific gravity than that rectified with lime or aluminium amalgam. The mean specific gravity of fifteen samples of the purest alcohol which could be obtained was 0.78506 at 25° C. The specific gravities of mixtures of pure alcohol and water were found to be as follows :—

TABLE V.

Per cent. of Alcohol by Weight.	Sp. gr. at 25° C.	Per cent. of Alcohol by Weight.	Sp. gr. at 25° C.
	Grams/c.c.		Grams/c.c.
0	0.997077	55	0.898502
2	0.993359	60	0.886990
5	0.988166	65	0.875269
6	0.986563	70	0.863399
10	0.980434	75	0.851336
15	0.973345	80	0.839114
20	0.966392	85	0.826596
25	0.958946	90	0.813622
30	0.950672	95	0.799120
35	0.941459	98	0.791170
40	0.931483	99	0.788135
45	0.920850	100	0.785058
50	0.909852	—	—

They also determined the specific gravities of twelve mixtures of alcohol and water at temperatures of 10° to 40° C., and give the method of calculating the specific gravities of mixtures at any temperature between those limits. In these determinations the weighings were made in specially constructed pycnometers, the mixtures being saturated with air.

<sup>1</sup> *Bull. Bureau of Standards*, Vol. IX., Scientific Paper, No. 197.

TABLE VI.—CONVERSION OF PER CENT. BY VOLUME INTO PER CENT. BY WEIGHT CORRECTED FOR ALCOHOL.

Volume.	Weight.	Volume.	Weight.	Volume.	Weight.	Volume.	Weight.
Per Cent.							
1	0·80	12	9·68	60	52·20	89	84·46
2	1·60	13	10·51	70	62·50	90	85·75
3	2·40	14	11·33	80	73·59	91	87·09
4	3·20	15	12·15	81	74·74	92	88·37
5	4·0	16	12·98	82	75·91	93	89·71
6	4·81	17	13·80	83	77·09	94	91·07
7	5·62	20	17·28	84	78·29	95	92·46
8	6·43	25	20·46	85	79·50	96	93·89
9	7·24	30	25·69	86	80·71		
10	8·05	40	33·39	87	81·94		
11	8·87	50	42·52	88	83·19		

TABLE VII.—ALCOHOLIC CONTENT OF BOILING LIQUID AND VAPOUR OF AQUEOUS ALCOHOL AT DIFFERENT BOILING-POINTS (GRONING).

Temperature of Vapour.	Alcohol by Volume in Boiling Liquid.	Alcohol by Volume in Distillate.	Temperature of Vapour.	Alcohol by Volume in Boiling Liquid.	Alcohol by Volume in Distillate.
Degrees C.	Per Cent.	Per Cent.	Degrees C.	Per Cent.	Per Cent.
77·2	92	93	87·5	20	71
77·5	90	92	88·7	18	68
77·8	85	91·5	90·0	15	66
78·2	80	90·5	91·2	12	61
78·7	75	90	92·5	10	55
79·4	70	89	93·7	7	50
80·0	65	87	95·0	5	42
81·2	50	85	96·2	3	36
82·5	40	82	97·5	2	28
83·7	35	80	98·7	1	13
85·0	30	78	100·0	0	—
86·2	25	76			

Later results by P. N. Evans <sup>1</sup> are as follows :—

TABLE VIII.

Temperature of Vapour.	Alcohol by Weight in Liquid.	Alcohol by Weight in Vapour.	Temperature of Vapour.	Alcohol by Weight in Liquid.	Alcohol by Weight in Vapour.
Degrees C.	Per Cent.	Per Cent.	Degrees C.	Per Cent.	Per Cent.
78.2	91	92	87.0	17	70
78.4	85	90	87.5	16	69
78.6	82	88	88.0	15	68
78.8	80	87	88.5	13	67
79.0	78	86	89.0	12	65
79.2	76	85	89.5	11	63
79.4	74	85	90.0	10	61
79.6	72	84	90.5	10	59
79.8	69	84	91.0	9	57
80.0	67	83	91.5	8	55
80.2	64	83	92.0	8	53
80.4	62	82	92.5	7	51
80.6	59	82	93.0	6	49
80.8	56	81	93.5	6	46
81.0	53	81	94.0	5	44
81.2	50	80	94.5	5	42
81.4	47	80	95.0	4	39
81.6	45	80	95.5	4	36
81.8	43	79	96.0	3	33
82.0	41	79	96.5	3	30
82.5	36	78	97.0	2	27
83.0	33	78	97.5	2	23
83.5	30	77	98.0	1	20
84.0	27	76	98.5	1	15
84.5	25	75	99.0	—	10
85.0	23	74	99.5	—	5
85.5	21	73	100	0	0
86.0	20	72			
86.5	18	71			

*Boiling points of mixtures of ethyl alcohol and water at different pressures (A. Doroszewski and E. Polianski).—<sup>2</sup>*

<sup>1</sup> *Jour. Ind. Eng. Chem.*, 1916, 8, 260-262.

<sup>2</sup> *J. Russ. Phys. Chem. Ges.*, 1910, 42, 109-134; *Jour. Soc. Chem. Ind.*, 1910, 586.

TABLE IX.—BOILING-POINTS OF MIXTURES OF ETHYL ALCOHOL AND WATER.

Percentage of Alcohol by Weight.	Boiling Points of Mixtures of Ethyl Alcohol and Water.		
	700 m.m. °C.	760 m.m. °C.	800 m.m. °C.
0	97.72	100	101.44
10	89.28	91.47	92.86
20	84.89	87.05	88.43
30	82.42	84.58	85.94
40	81.00	83.13	84.49
50	79.78	81.91	83.26
60	78.92	81.04	82.38
70	78.03	80.14	81.47
80	77.22	79.32	80.64
90	76.40	78.54	79.86
100	76.26	78.35	79.66
95.57	76.16	78.23	79.54

TABLE X.—SHOWING THE CONTRACTION OF ALCOHOL ON COOLING FROM ITS BOILING-POINT CALCULATED BY DUMAS FROM GAY-LUSSAC'S EXPERIMENTS.

Temperature.		Volume.	Temperature.		Volume.
° C.	° F.		° C.	° F.	
78.14	173	1000.0	38.4	101	954.4
73.4	164	994.4	33.4	92	948.9
68.4	155	988.6	28.4	83	943.6
63.4	146	982.5	23.4	73	938.6
58.4	136	975.7	18.4	65	934.0
53.4	128	970.9	13.4	56	929.3
48.4	119	965.3	8.4	47	924.5
43.4	110	960.0	3.4	39	919.9

TABLE XI.—AMOUNT OF WATER TO ADD TO ALCOHOL OF GIVEN STRENGTH TO REDUCE IT.

	90 Per Cent. Alcohol.	85 Per Cent. Alcohol.	80 Per Cent. Alcohol.	75 Per Cent. Alcohol.	70 Per Cent. Alcohol.	65 Per Cent. Alcohol.	60 Per Cent. Alcohol.	55 Per Cent. Alcohol.	50 Per Cent. Alcohol.
85	6.56								
80	13.79	6.83							
75	21.89	14.48	7.20						
70	31.10	23.14	15.35	7.64					
65	41.53	33.03	24.66	16.37	8.15				
60	53.65	44.48	35.44	26.47	17.58	8.76			
55	67.87	57.90	48.07	38.32	28.63	19.02	9.47		
50	84.71	73.90	63.04	52.43	41.73	31.25	20.47	10.35	
45	105.34	93.30	81.38	69.54	57.78	46.09	34.46	22.90	11.41
40	130.80	117.34	104.01	90.76	77.58	64.48	51.43	38.46	25.55
35	163.28	148.01	132.88	117.82	102.84	87.93	70.80	58.31	43.59
30	206.22	188.57	171.05	153.53	136.34	118.94	101.71	84.54	67.45
25	266.12	245.15	224.30	203.61	182.83	162.21	141.65	121.16	100.73
20	355.80	329.84	304.01	278.26	252.58	226.98	201.43	175.96	150.55
15	505.27	471.00	436.85	402.81	368.83	334.91	301.07	267.29	233.64
10	804.50	753.65	702.89	652.21	601.60	551.06	500.50	450.19	399.85

EXAMPLE.—It is required to bring alcohol containing 80 litres of alcohol per 100 litres to 40 per cent. by volume. Running the finger down the 80 per cent. column until it comes opposite 40, we find that 104.01 litres of water have to be added to 100 litres of 80 per cent. alcohol by volume to bring it to 40 per cent. by volume.

TABLE XII.—BOILING-POINT IN DEGREES C. OF ALCOHOL OF DIFFERENT STRENGTHS (NOYES AND WARFRE).

Per Cent.	Boiling Point.								
100.0	78.300	94.0	78.195	84.0	78.723	69.0	80.042	18.0	87.92
99.5	78.270	93.5	78.211	83.0	78.806	67.0	80.237	13.0	90.02
99.0	78.243	93.0	78.227	82.0	78.879	65.0	80.438	10.0	91.80
98.5	78.222	92.5	78.241	81.0	78.968	63.0	80.642	8.0	93.10
98.0	78.205	92.0	78.259	80.0	79.050	55.0	81.77	7.0	93.73
97.5	78.191	91.0	78.270	79.0	79.133	48.0	82.43	5.5	94.84
97.0	78.181	90.0	78.323	78.0	79.214	37.0	83.76	4.5	95.63
96.5	78.179	89.0	78.385	77.0	79.354	35.0	83.87	3.0	97.11
96.0	78.174	88.0	78.445	76.0	79.404	29.0	84.86	2.0	98.05
95.5	78.176	87.0	78.530	75.0	79.505	26.0	85.41	1.5	98.55
95.0	78.177	86.0	78.575	73.0	79.683	22.0	86.11	1.0	98.95
94.5	78.186	85.0	78.645	71.0	79.862	20.0	87.32	0.5	99.65

*N.B.*—The alcoholic content shown by hydrometer and boiling-point is only true of mixtures of *pure* alcohol and *pure* water. The weight of ethylene generated by sulphuric acid, and absorbed by bromine, is useful if methyl-alcohol co-exists. Iodoform test is undecisive. The Swiss monopoly require that 90 per cent. "extra fin" and "surfin" do not react with metadiamidobenzol hydrochloride, nor must "extra fin" decolorise permanganate in less than five minutes, nor "surfin" in less than fifteen minutes. Otherwise the goods are only accepted as inferior; 90 per cent. "fin" showing more than 0.3 per 1000 of aldehyde, or which decolorises permanganate in less than a minute, is rejected.

*Phenol test for aldehyde.*—Run 2 c.c. of sample into test-tube, add 0.02 grammes of either carbolic acid, naphthol  $\alpha$ , naphthol  $\beta$ , resorcin, hydroquinone, phloroglucin, pryogallol, guaiacol, thymol, gallic acid. The reagent dissolved, pour 1 c.c. pure colourless  $H_2SO_4$  down side of tube; if aldehyde be present, a coloured ring forms at the junction of the two layers. After shaking, the colour varies with the nature of the aldehyde and reagent, susceptible to  $\frac{1}{200000}$ th of aldehyde.

*Indices of Refraction of Mixtures of Alcohol and Water.*—A. Doroshewski and S. Dworzanczyk<sup>1</sup> have determined the indices of refraction for sodium rays of mixture of ethyl alcohol and water at different temperatures, their results being as follows:

TABLE XIII.

Percentage of Alcohol by Weight.	$15^\circ$ $n \frac{D}{D}$	$30^\circ$ $n \frac{D}{D}$	$40^\circ$ $n \frac{D}{D}$	$50^\circ$ $n \frac{D}{D}$	$55^\circ$ $n \frac{D}{D}$
0	1.33345	1.3318	1.3306	1.3290	1.3281
10	1.34020	1.3384	1.3368	1.3349	1.3339
20	1.34778	1.3450	1.3429	1.3406	1.3393
30	1.35470	1.3510	1.3481	1.3452	1.3435
40	1.35948	1.3550	1.3518	1.3484	1.3468
50	1.36290	1.3578	1.3543	1.3506	1.3488
60	1.36505	1.3597	1.3560	1.3522	1.3501
70	1.36645	1.3608	1.3570	1.3528	1.3505
80	1.36690	1.3611	1.3569	1.3525	1.3502
90	1.36626	1.3603	1.3561	1.3515	1.3491
100	1.36332	1.3573	1.3521	1.3487	1.3465

<sup>1</sup> *Jour. Russ. Phys. Chem. Ges.*, 1908, 40, 908-931; *Jour. Soc. Chem. Ind.*, 1909, 853.

## CHAPTER II.

### CONTINUOUS FERMENTATION AND STERILIZATION IN THE MANUFACTURE OF INDUSTRIAL ALCOHOL.

THE manufacture of industrial alcohol comprises : Saccharification, Fermentation, Distillation and Rectification.

*Saccharification.*—Sucrose, glucose, and maltose <sup>1</sup> are directly capable of undergoing fermentation. These do not, therefore, require any special preliminary preparation. Other carbohydrates, inuline, for instance, before they can undergo fermentation, must be previously hydrated, either (*a*) by superheated steam, or (*b*) by the action of dilute acids, at a temperature of 100° C. Starch and dextrine, as such, do not undergo fermentation. They must previously be saccharified, *i.e.*, they must be changed into dextrose or maltose (*a*) by the action of dilute acids (5 lb. of acid per 100 lb. of grain) acting under a pressure of 5 kilogrammes per sq. cm. ; or (*b*) by the diastase of malt at 12° C. (53.6° F.) ; about 25 lb. of malt being used for 100 lb. of starch to be saccharified. (See Chaps. IV. and V.)

*Fermentation.*—The transformation of all the above saccharine matters into alcohol is effected by an *organised* ferment belonging to the numerous class of saccharomyces or yeasts. The great difficulty of industrial fermentation is to produce a vigorous growth of the proper species of saccharomyces. This minute organism does not propagate itself and develop unless it can find, besides the mineral and nitrogenous matter required for its production, a suitable amount of carbohydrates capable of undergoing fermentation. If the quantity be too small, reproduction is slow, and the fermentative power soon disappears. If too

<sup>1</sup> For a detailed description of these sugars, densities of their solutions, etc., see the author's *Technology of Sugar*, 2nd ed., published by Scott, Greenwood & Son, London.

large, it reproduces itself very abundantly, to the loss of the distiller, whose object is not to obtain unlimited numbers of saccharomyces, but the greatest amount of alcohol which the latter can produce. As 1 oz. of saccharomyces require for their production at least 1 oz. of sugar, to work in that way would spell ruin. It is thus necessary to continually regenerate a sufficient quantity of suitable ferment to complete the fermentation, but no more, and to use up completely the ferment produced. The presence of other ferments must be avoided. They not only use up, to waste, some of the sugar treated, but produce substances



FIG. 2.—Yeast Cells (*Saccharomyces cerevisiae*).

either injurious to the quality of the alcohol, or poisonous and capable of paralysing or killing the ferment. Fortunately the most frequently occurring of these dangerous organisms (disease ferments) are much weaker than the alcoholic ferment. Some, without being decidedly anærobic, are not active in the presence of oxygen. They may thus be dealt with by aerating the wort, prior to fermentation. Some succumb to an acid, others to an antiseptic, incapable from the small dose of seriously hindering the development and action of the alcoholic ferment. But when the distillery residuals are not to be used as cattle food, the

development of disease ferments may be prevented by oxidising the worts, or by treating them with sulphuric or hydrochloric acids or by organic acids derived from the action of the former on the organic salts present in the substance treated, which secondary products are capable of paralysing the disease ferment without interfering with the alcoholic ferment. Thus, in beet-juice fermentation, the acidity of the liquor is regulated so that in 1 litre the acidity equals about 2.5 grammes of sulphuric acid. Other substances are injurious to the alcoholic ferment, viz., the higher acids of the fatty series, such as butyric and capric acids, etc., and particularly nitrous acid and the compounds of these present, for instance, in beet molasses. They may, owing to the volatility of the acids, be eliminated by boiling the diluted molasses with a slight excess of sulphuric acid, after which the excess of acid is neutralised. Moreover, certain extractive matters are eliminated by the action of animal charcoal. When the fermentation residuals have to be directly consumed by animals (as in the case of tubers and cereals saccharified by malt), the above process is not applicable, since mineral acids engender troubles in the digestive system. Until lately, recourse was had to lactic acid produced at the expense of sugar by a special form of fermentation, effected on a portion of the products undergoing fermentation. This lactic acid is, in fact, poisonous to the highly dangerous *clostridium butyricum*, the existence of which even attenuates the lactic ferment itself. But the production of this lactic acid necessitates minute precautions and involves a great loss of sugar. Small doses of hydrofluoric acid are used in its stead, more especially in France. At all times, when working by intermittent fermentation, it is necessary to produce the amount necessary for each fermenting tun, but afterwards precautions must be taken to avoid producing at the expense of the sugar a useless amount of ferment, and thus causing the ferment in a regression stage to excrete products injurious to the quality of the alcohol.

Care must be taken to allow the ferment to live, without its field of action being invaded by foreign ferments. This is done by carefully watching the temperature at each phase of the operation. The greater number of disease ferments only develop

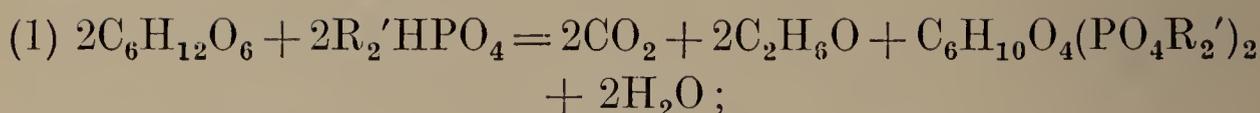
rapidly at a temperature above 30° C. (86° F.). Some of them even, like the distiller's lactic ferment, thrive at a temperature of 50° C. (122° F.), a temperature which paralyses many others. The alcoholic ferment, on the other hand, may live and thrive at decidedly lower temperatures, 15°-18° C. (59°-63.4° F.). The lower temperature brewery ferments act slowly, about 0° C. (32° F.). On the other hand, two phases may be differentiated in the life-history of the higher temperature distillery ferment: (a) The phase of abundant development most active at the temperature of 25°-26° C. (77°-78.8° F.); (b) the phase of predominant fermenting power at higher temperatures. When it is desired to develop the ferment so as to form what is known as leaven or yeast, or the *piéd de la cuve* (vat bottoms) of the French, the operation is carried on at a comparatively low temperature, never exceeding 25° C. (77° F.), working with a medium rich in mineral and albuminoid food, and in sugars capable of undergoing fermentation. By successive additions of saccharine wort, a high percentage of sugar is maintained in the mass. Much young ferment is thus produced capable of further propagating itself prolifically. Thus, by fermenting the concentrated worts from potatoes, and grain saccharified by malt, yeasts are produced on liquids containing as much as 18, and even 22, per cent. of maltose, and the percentage of sugar is never allowed to fall below 9. When that figure is reached, freshly saccharified wort is run into the yeast, until the ferment is judged to be sufficiently developed. The yeast once applied, or set to work, the temperature of the whole mass rises to 28°-29° C. (82.4°-84.2° F.), so that the yeast exerts to the fullest possible extent its fermentative power without propagating itself beyond measure. Moreover, it must not be allowed to reach the retrogressive phase, when it appears to secrete products deleterious to the quality of the alcohol. The former temperatures of 32°-34° C. (89.6°-93.2° F.) have been completely forsaken. This operation, especially in the case of rich worts, is a very delicate one. It is, in fact, admitted in actual practice that 1 per cent. of sugar transformed into alcohol raises the temperature 0.9° C. (1.62° F.). The temperature of the worts to be treated with yeast must therefore be carefully watched,

or resource had to the use of refrigerants, and this latter process is being most generally adopted, in the case of very rich wash.

Yeast is also used in the fermentation of beet juice, but with a different point in view. Where substances capable of undergoing fermentation like beet juice may be rendered unsusceptible to disease ferments by the addition of a suitable amount of strong acids, and contain in themselves not only the sugar, but a suitable amount of nutritive mineral matter and assimilable albuminoids, the operation is remarkably simplified by treating one vat with the requisite amount of the contents of the previous one. Yeast is only made at the beginning of the season, or when it is necessary to renew it owing to its contamination with foreign ferments. Working normally with a vat treated with yeast, fresh juice of density 1.035-1.04 is run in, the flow being so regulated that the fermentation constantly absorbs the sugar introduced, and that the density in the vat be only 1.0015 at the maximum, and the acidity at 2.5 grammes of normal sulphuric acid per litre. Working in that way there is an abundant production of ferment. When the vat is full it is "mixed," that is, one-third of the contents is run into an empty vat: fresh vat bottoms are thus obtained for the next fermentation, then juice is run in to fill the two vats, but in the mother vat the density is maintained not at 1.0015 as formerly, but at 1.00075, so as to prevent the useless production of yeast and utilise as much as possible its fermentative power. The mother vat once full is exhausted of its sugar in four hours. In the case of beet juice treated in this way the fermentation process lasts twenty-four hours, with molasses forty-eight hours, with grain saccharified under pressure, or for thick grain or potato worts saccharified by malt, seventy-two hours.

A. Harden and W. J. Young<sup>1</sup> assume that during fermentation, if phosphates are added, the phosphate combines with the sugar to form a hexosephosphate, the rate of fermentation being considerably increased for a time; after which it falls rapidly to the normal. They give the following equations to show the course of the reactions:

<sup>1</sup> *Proc. Chem. Soc.*, 1908, 115-116; 1910, 321-330. See also A. v. Lebedoff, *Compt. rend.*, 1911, 136-139; *Biochem. Zeits.*, 1910, 213-229; W. J. Young, *Proc. Roy. Soc.*, 1909, 181 B, 528-545; *Biochem. Zeits.*, 1911, 177-188.



As Pasteur's principles of rational fermentation have become more extensively adopted in fermentation industries, it has been acknowledged that the first condition to fulfil to obtain the best results as regards purity and the largest yields in alcohol consists in the daily production in sufficient quantity of pure ferments from a strain of yeast appropriate to the industry and acclimatised to the nature of the saccharine wort. Having secured such yeast, the fermentation proper may be conducted in the open air, because when the wort is copiously treated with yeast in full activity, it is able to defend itself against bacteria during the whole course of the alcoholic fermentation. The main object is to simplify the apparatus and impart very great vitality to the yeast. There should only be a single yeast-producing vessel, from which three or four batches of yeast may be drawn off in the twenty-four hours. The apparatus should be sufficiently large for each batch of yeast to serve directly as "vat bottoms" (*piéd de la cuve*) without any intermediate proliferation in the open air. When once inoculated, the apparatus ought to yield yeast for more than a month without any other precaution being taken against contamination. All the taps are therefore cleaned in basins of water containing a little formalin, and all the valves possess a small vessel in which the packing is protected by anti-septic water. The prolific propagation of the yeast is intensified and its fermentation strength increased by carrying out on a large scale what Pasteur did in the laboratory: To revivify a languid yeast it must be cultivated in a thin film, that is, in a very thin layer of bouillon spread out in contact with air. Pasteur used large flasks, the flat bottoms of which were covered with only a thin layer of liquid. How the sterilised wort is introduced into the apparatus will be described directly; suffice it to say that the wort is sterilised outside the apparatus. The yeast apparatus (Fig. 3), a cylindrical copper or wrought-iron vessel, resting on a cast-iron foundation, consists of two distinct parts: the bottom

forms a reservoir of juice in pure fermentation, whilst the top comprises four to six aeration plates, on which the liquid forms a very thin layer about 2 cm., say,  $\frac{3}{4}$ -inch thick. The liquid of the

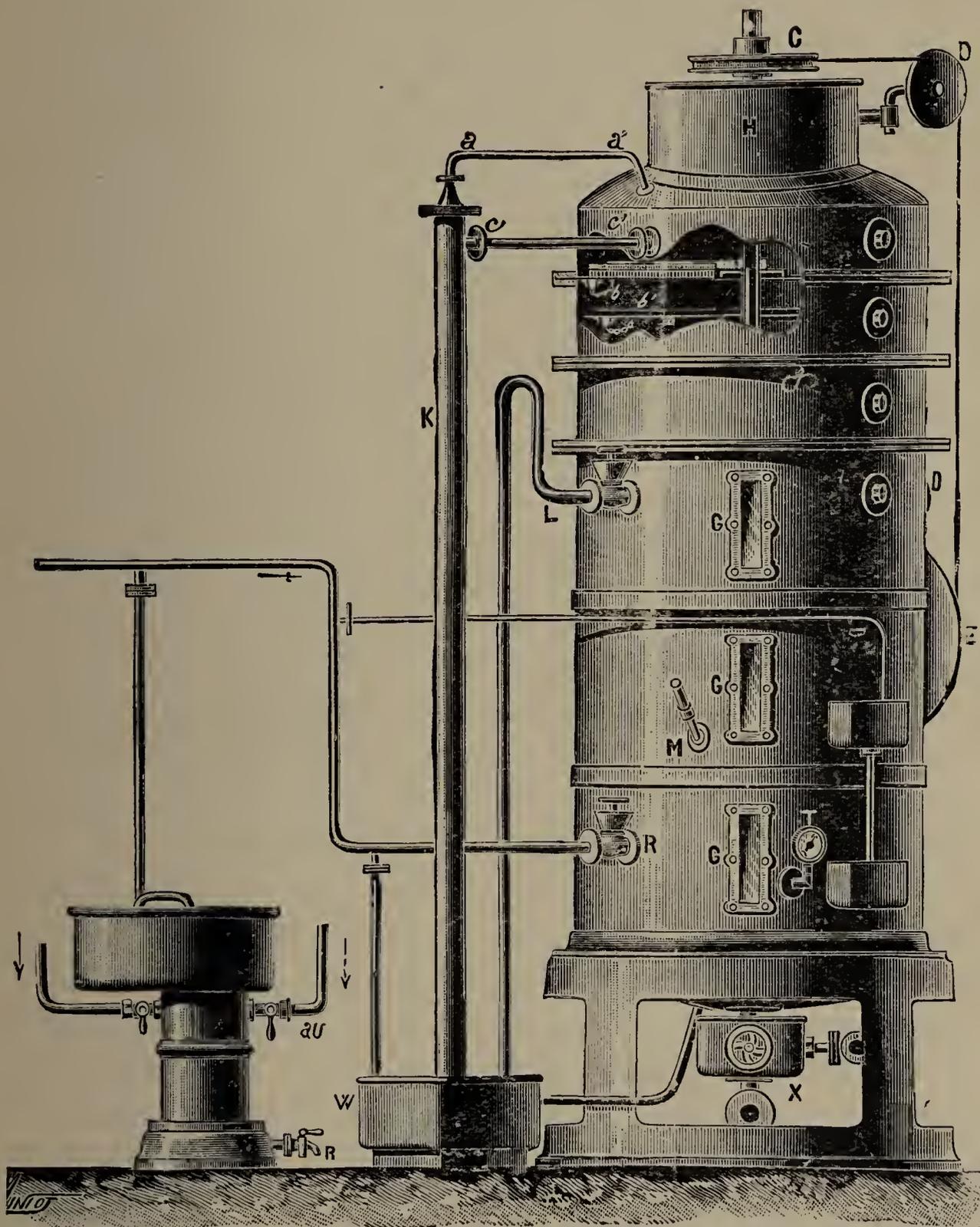


FIG. 3.—Plant for cultivation of pure ferments (aerobic cultivation) (E. BARBET, Paris).

lower reservoir is continuously raised on to the upper plate by means of an emulsifier K. The principle of the emulsifier is known. Zambeaux has used it in a very ingenious way to raise sulphuric acid on to the upper purifying towers. The emulsifier

K is a small elongated tubular vessel which contains some 6-10 thin copper pipes. In the lower orifice of each pipe is a small vertical nozzle through which a jet of sterilised air issues. The air is sub-divided into a series of air-bubbles which occupy the whole width of the pipe, and which are separated one from another by rings of liquid (*liquid pistons*). If the entering air be sufficient, the sum total of the liquid pistons in any one of the tubes forms a column of liquid of less height than the height of the liquid in the bottom of the yeast apparatus. Equilibrium is destroyed, and, in virtue of the law of communicating vessels, the liquid assumes a continuous ascending movement in the tube to spread itself through *c c'* on to the upper aerobic plate *b'*. This plate bears in its centre a small rim which forms a funnel. The excess of liquid falls on to the second plate, which, however, has no funnel. The liquid flows alternately from the circumference to the centre, and reciprocally. Throughout the whole of this course the saccharine wort in fermentation is spread out in contact with the air brought by the emulsifier. The carbonic acid is disengaged, the wort being totally freed from it, and in its place oxygen is dissolved in an analogous manner to respiration by the lungs. Without doubt the air in this way acts much better on the leaven than air injected in large globules in the bottom of the receiver. Suppose, instead of yeast, we place in the wort a decidedly amphibious organism, *e.g.*, one of the mucedinæ. The culture on the plates will forthwith produce aerial mycelia; whilst in the lower reservoir it would be in vain to inject air, only the anaerobic or submerged form of the organism would be produced; the mycelia would divide and assume the form of oval globules, like yeast, and yield alcohol.

A vertical axis traverses the yeast apparatus, and carries metallic brushes to keep in suspension the yeast deposited on the plates. It is turned from time to time by hand mechanism CDE. H is a small vessel containing a dilute solution of formalin to clean the packing and the safety valves. L is the exit of the mixture of air and carbonic acid; it bubbles into the vessel W of the emulsifier. M, pipe for inoculation with pure yeast. R, entrance of sterilised air for direct bubbling. G, gauge glasses.

T, thermometer. X, discharge pipe (cleansed) carrying a lateral pipe to receive steam or sterilised air. The air steriliser placed to the left of the figure consists of a cotton waste filter permanently enclosed in a steam autoclave. The air commences to circulate in a cleansed coil in the steam jacket, where it can be heated to a high temperature, then it traverses from the bottom to the top of the cotton heated by the filter jacket. In this manner every portion of the waste is brought to the *sterilising* temperature. Steam may then be turned off, the filtration of the air sufficing to free it from germs, provided the cotton be purified from time to time by steam. A small jet of steam may be left on permanently to lukewarm the air, because aeration appreciably cools the worts. Fig. 4 shows Egrot's combined steriliser and fore-warmer.

Before the time of Pasteur every operation was empirical (rule of thumb); whilst at the present day, Pasteur's methods, which he himself had already applied to brewery and vineyard practice, have been adopted in industrial distilleries. By their means the distiller can regulate his fermentation processes, whereas formerly he had no effective control. The work of Pasteur has been considerably extended in many directions. In particular, Barbet's process of spirit manufacture now about to be described is only an extension of his methods. There is one fact, says Barbet, that the Pasteur school left a little in the dark. As a ferment easily accustoms itself to culture media of different composition, and as it reproduces itself in all saccharine solutions provided they contain nitrogenous matter, phosphates, and salts, it was customary not to attach any great importance either to the nature of the sugar or of the accessory substances. If, instead of cultivating the wine ferment in the juice of the grape, we cultivate it twenty consecutive times in malt wort, after the twentieth culture it still remains grape ferment, and not beer-yeast or barm—the medium has not modified the original strain of the ferment in any appreciable way; but Pasteur found that beer wort, treated with pure Chablis ferment, imparted to that beer a vinous character recalling the flavour of Chablis wine. In breweries the strain of ferment used has a manifest influence on

the aroma of the beer, and if the strain of ferment (whilst still keeping to yeast) be changed, the taste of the beer is immediately altered, even if the wort has not varied. These facts have led to

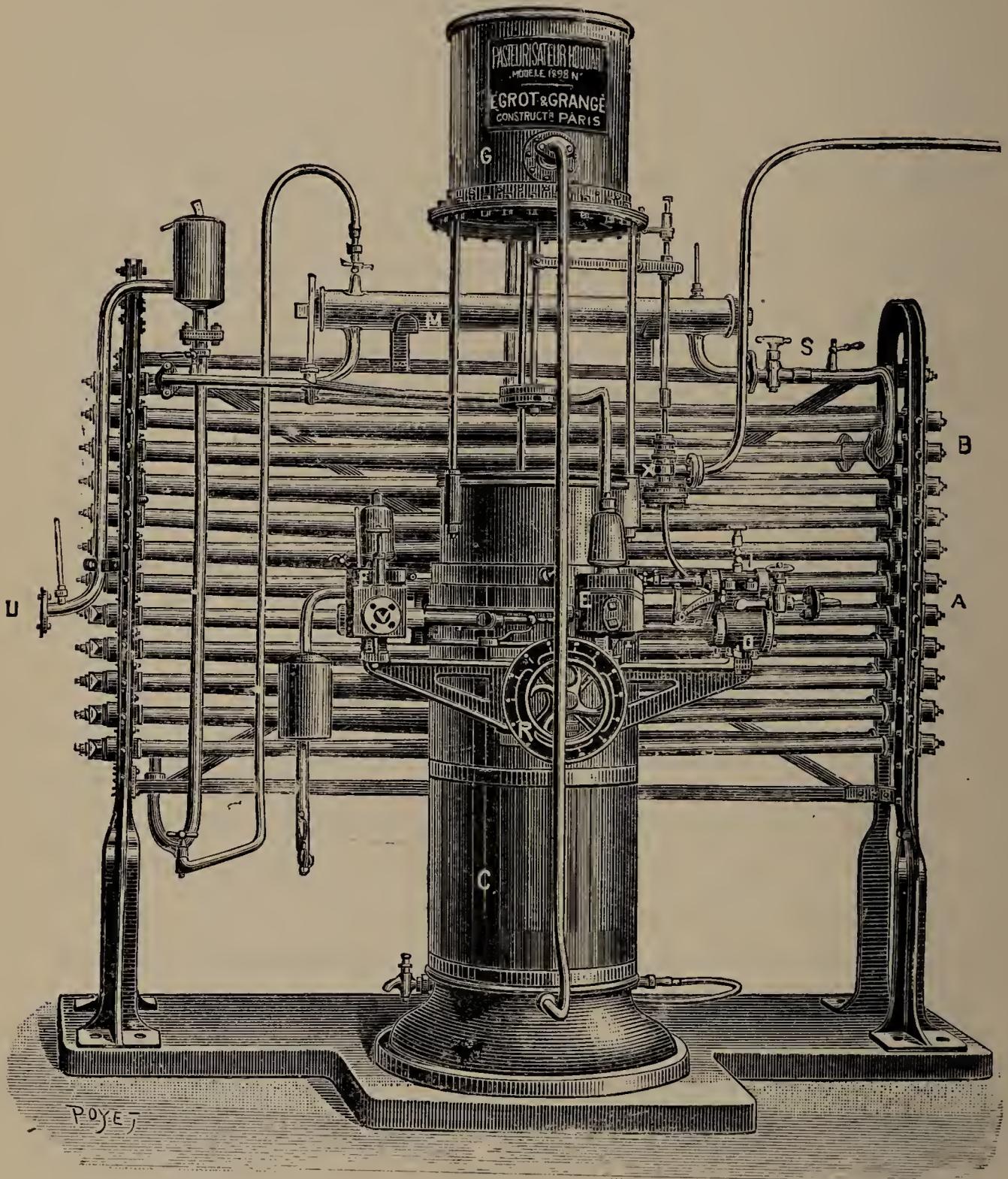


FIG. 4.—Forewarmer steriliser (EGROT and GRANGÉ). A, B, jacketed tubular pipes, B, heating A, cooling.

an altogether preponderating influence being attributed to the strain of ferment, while the composition of the saccharine wort has not been sufficiently taken into account. Now, if the strain of ferment persists for a very long time, even though in a medium

differing from its natural and favourite food, it is none the less true that there is a change in its mode of existence, and this change resolves itself into a modification in the nature and *bouquet* of its excretions.

It must be borne in mind that the splitting up or resolution of sugar, under the influence of the ferment, is a vital phenomenon ; it is a complex digestion, which, in addition to alcohol and carbonic acid, gives birth to numerous by-products. Amongst these accessory substances, Pasteur found per 100 grammes of sucrose, 3.16 of glycerine, 0.67 grammes of succinic acid, and 1 gramme of building materials, ceded to new ferment cells, but he did not weigh, and no one has yet weighed, the accompanying aromatic excretions, the simultaneous generation of which is incontestable. Now, in brandy, it is not the alcohol which has such a high commercial value—the chief value of brandy rests most decidedly in the perfumed excretions peculiar to this form of fermentation. The alcohol is simply the vehicle for the perfumed excretion. If we cultivate a fine champagne ferment in malt wort, the fermented liquid will possess a certain vinous illusion—it will be *cervoise*, neither wine nor beer ; but if we distil it, it will be found that the perfumes are fugitive and in no sense equal to the characteristic aroma of wine, because the culture medium has altered the digestive excretions of the ferment. If to the residual lees from the distillation of white wine we add a sugar, exempt from any intrinsic odour whatever, and if the wort be kept under the same conditions of acidity, density, and temperature as grape juice, the vine ferment is once more in its favourite medium : the same acidity, the same albuminous, saline, and organic matter as originally. Its vital evolution produces a second edition of all the phenomena of the first fermentation, it secretes the same fatty, œnanthic, and other acids, the same perfumed aldehydes, the same ethers, and essences, so that on distillation it would be impossible to differentiate the second brandy from the first. Every one knows the radical difference that is generally made between the industrial distillery and the old malt whisky distillery, on the one hand, and those which produce the so-called natural brandies on the other hand. One hundred years ago, as far as

natural brandies were concerned, only wine brandy, cherry brandy, rum, etc., produced by altogether crude rudimentary processes by so-called "spontaneous" fermentation, and distillation in an alembic, were known.

On the introduction of industrial distillation raw material was sought for amongst cheap agricultural products, such as raw grain, potatoes, beets, Jerusalem artichokes, and sugar residuals, treacle or molasses, and recourse had to be made to much more complicated processes. Without mentioning the preliminary saccharification of starchy substances, attempts were made to induce very rapid industrial fermentation by means of yeast, to carry continuous distillation into practical effect, and to subsequently purify the product (because it in no way resembled natural brandy) by rectification, filtration, chemical or electrical treatment, addition of perfumes, etc. All these improvements, however, only served to widen still further the gulf separating the two kinds of distilleries: the old distillery still retaining its supremacy, whilst the industrial distillery was devoted to the production of a cheap product—in one word, alcohol. It strove to eliminate all smell which would betray its origin, and to approach neutrality, and chemical purity, as near as possible. But this neutrality itself only shows that qualities and grades have been renounced, and that the least possible amount of impurities is aimed at in the production of plain spirits (Chap. IV.).

In order to produce spirits resembling natural brandy, Barbet commenced by treating potatoes or beets so as to extract their starch or sugar by the usual methods. He saccharified the starch by the following method. Normal sulphuric acid is used in the proportion of 1 to  $1\frac{1}{2}$  per cent. on the weight of starch, first boiling in the open air to dissolve the starch, then charging into a copper autoclave and heating at a pressure not exceeding 1 to  $1\frac{1}{2}$  kilogrammes per square centimetre at the most for a period of about one hour (page 31). Then followed saturation by chalk (the syrup being extremely pale), filtration through a filter press and through animal charcoal, and also, if necessary, through wood charcoal. The liquid must be rendered colourless and inodorous. In preparing the saccharine liquor, it is necessary to approach as

near as possible to the composition of the must of fruits, especially as regards saline matters and acids. Mineral acids must on no account be present. The most convenient manner of imitating fruit must consists in using the vinasse from fruit distillation. It may seem difficult at first sight, for those engaged in the industry on the large scale, to procure such musts in sufficient quantity for daily use; but this is not impossible since the original must can serve at least five times, and even more, provided special heating precautions are observed during distillation. The principal danger to be avoided is caramelisation, in order to obviate which Barbet only heats his distilling plant with exhaust steam. The industry formerly demanded acidity as indispensable to fermentation, either by mineral acids or by the manufacture of lactic ferments, or by the re-utilisation of a good portion of vinasse (manufacture of pressed yeast). Barbet claims that his process has nothing subversive in it. He employs other vegetable acids and other vinasses. Certain precautions render this process far from laborious, and it is one to which the manufacturer should devote all his care and attention.

Barbet reserves to himself the use of his patented process of fermentation by pure ferments and continuous sterilisation. He claims that he has improved the fermentation process, especially the production of ferments by aerobic culture, so as to yield the maximum activity and vitality to the ferment in very acid musts. The purity and the high strength of the three-six<sup>1</sup> are in direct proportion to the organic acidity of the musts. Moreover, he claims that his system of pure ferments and aeration is pushed by him as far as possible with the view of producing a prolific reproduction of ferment cells. The distillation of the concentrated lees yields highly perfumed products of great body sufficient to perfume large quantities of industrial alcohol. Ebullition is necessary to destroy the cell and liberate the perfume. The greater number of cells propagated, the greater the amount of perfume produced. Operations must therefore be conducted as in pressed yeast factories. A small percentage of alcohol is sacri-

<sup>1</sup> *Trois-six* is a term applied in France to 90 per cent. alcohol, because three volumes of it mixed with three volumes of water yield a mixture marking 19° Cartier.

ficed, in order to produce enough ferment, as it is the source of the desired aroma. Therefore, at the end of the first pure ferment vessel, two or three large vessels are placed, which serve to leaven the whole of the must, and to start the fermentation process therein. Barbet also uses emulsion with sterilised air, and aeration, so as to make reproduction as prolific as possible. Hence the musts, copiously treated with ferment, and maintained up to then in absolute purity, are run into tuns in the open air, there to complete the fermentation process, because there is now no danger of contamination, the alcohol already formed, and the great acidity, preventing infection from all kinds of bacteria. It is very difficult to induce forced ageing of spirits after or during distillation. These are very delicate culinary questions, and success is not often attained. Barbet overcomes this difficulty in a great measure by ageing the fermented liquor before distillation by prolonged heating of the fermented wash in a series of vessels, P P<sup>1</sup> P<sup>2</sup> P<sup>3</sup> (Fig. 5). By pumping the fermentation gases into P<sup>3</sup>, the aroma is retained, and the excess of gas condensed in S falls as a perfumed liquid into the still D, or into a distilling column.

*Fermentation in closed vats.*—E. Lühder<sup>1</sup> gives an interesting account of fermentation in closed fermentation vats, using a mash made from potatoes saccharified by means of oat-malt. The vats employed were upright and cylindrical, each having a capacity of 3478 litres (765 galls.), and whenever required, which, however, was not often, they were cooled by a flow of water over the exterior. The carbonic acid was taken off by a pipe and was washed with water to recover the alcohol, which added about 0.3 to 0.4 per cent. to the yield, the total being 68 to 69 litres of 100 per cent. alcohol for 100 kilos of starch (6.8 to 6.9 galls. per 100 lb. of starch).

*Catalysers or Energisers in Alcoholic Fermentation.*—It is well known that yeast grows most vigorously in a slightly acid medium, and although ordinary acids, such as sulphuric, hydrochloric, or hydrofluoric are employed, it is probable that they act by liberating organic acids from the material to be fermented, and that

<sup>1</sup> *Z. Spiritusind.*, 1913, 36, 213-214.

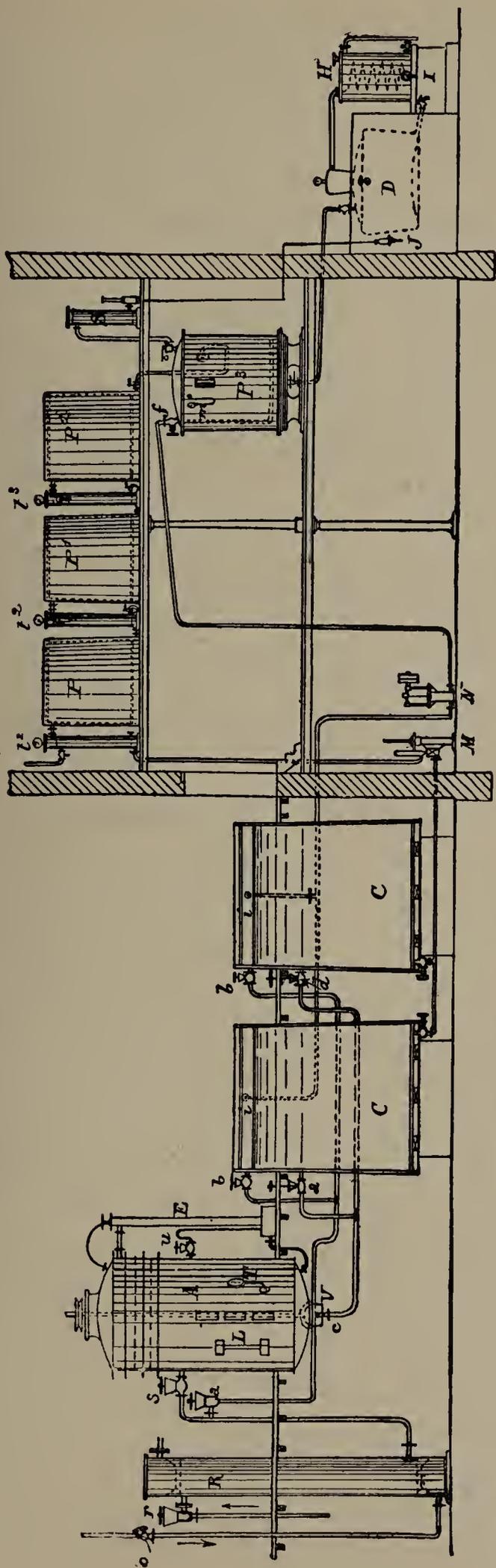


FIG. 5.—Plant for aseptic fermentation, collection of fermentation gases, ageing by prolonged heating and distillation of spirit (E. BARBET). A, ferment vessel; CC, fermentation tuns; K, steriliser; M, fermented wash pump; N, fermentation gas pump; *ii*, fermentation gas pipes; *t<sup>1</sup>* *t<sup>2</sup>* *t<sup>3</sup>* calorisers; P P<sup>1</sup> P<sup>2</sup> P<sup>3</sup> closed vessels; P<sup>3</sup>, autoclave to stand pressure of CO<sub>2</sub> from CC; E, condenser for unabsorbed fermentation gas; D, still; H, condenser; I, test safe; J, test safe for perfumed liquid run into still or distilling column E. BARBET).

these organic acids are beneficial to the growth of the plant. H. Euler and H. Cassel<sup>1</sup> state that the growth of yeast cells is considerably stimulated by the addition of salts of aliphatic and hydroxyacids, *e.g.*, ammonium formate and acetate, sodium lactate and racemate. In certain experiments which they carried out 2 grammes of sucrose was dissolved in 110 c.c. of water and 0.25 grammes of yeast added, the volume of carbonic acid evolved without any addition was 30 c.c. in ninety minutes, 51 c.c. in 180 minutes, 80.5 c.c. in 330 minutes, and 102 c.c. in 490 minutes. In a similar experiment in which 0.04 gramme of ammonium formate was added, the carbonic acid amounted to 33.5 c.c. in 90 minutes, 65 c.c. in 180 minutes, 120.5 c.c. in 330 minutes, and 180.5 c.c. in 490 minutes.

Sodium pyruvate appears to act as a catalyst when added to fermenting liquids,<sup>2</sup> and formic acid is also regarded as beneficial by H. Lange.<sup>3</sup> The last-named author states that small amounts of formic acid stimulate yeast growth, and that fermentation in presence of this acid is much more regular throughout, less violent and more uniform, but with a vigorous after fermentation. Mash tuns may therefore be filled much higher than usual. The amount of formic acid most suitable is 50 to 60 c.c. of pure formic acid to a mash of 3000 litres capacity. The strong formic acid may be diluted in the proportion of one part to eight parts of water, and 100 c.c. of this solution is added for each 100 litres of mash after it has been acidified and cooled to 37° or 38° C. In successive fermentations the proportion of formic acid may be increased, first to 200 c.c. and then to 300 c.c. per 100 litres, and may remain at that proportion in subsequent operations, the temperature of pitching being raised to advantage 1½° to 2° C.

*Influence of rosin in fermentation.*—The effect of abietic acid or rosin in the fermenting tun is beneficial, according to J. Effront,<sup>4</sup> favouring yeast growth and inhibiting bacterial action. Effront took out a patent for this discovery,<sup>5</sup> which included the use of

<sup>1</sup> *Z. physiol. Chem.*, 1913, 86, 122-129.

<sup>2</sup> M. Oppenheimer, *Z. physiol. Chem.*, 1914, 93, 233-261.

<sup>3</sup> *Z. spiritusind.*, 1905, 28, 341-342.

<sup>4</sup> *Compt. rend.*, 1903, 136, 1556-1557; *Monit. Scient.*, 1905, 19, 721-722.

<sup>5</sup> *Eng. Pat.* 19,354, Sept. 3, 1902.

fatty acids, resin acids, fatty soaps or resins, which are stated to act beneficially by altering the physical condition of the fermenting liquid. The proportions given in the patent specification are one ton of molasses diluted to 650 to 700 gallons, to which is added a 10 per cent. solution of colophony containing 2.5 per cent. caustic potash, in the proportion of  $1\frac{1}{8}$  galls. to  $3\frac{3}{8}$  galls. Effront states that when a pitching yeast is employed containing, say, 5 per cent. of bacterial impurity, after addition to the wort sedimentation takes place, a layer of nearly pure yeast being at the bottom of the vessel, but that the proportion of bacteria in the higher layers of the liquid increase, so that at the surface the ratio of bacteria to yeast cells may be as high as 4 to 1. This being the case, the bacteria, not being held in check by the activity of the yeast in those regions, will multiply rapidly, and may do infinite harm. The addition of 0.3 to 0.5 gramme of rosin soap per litre to the mash at the time of pitching causes the whole of the fluid to become turbid, which subsequently breaks to form a flocculent precipitate, owing to the separation of rosin in a colloidal form, and this adheres to the bacteria so as to weight them relatively more than the yeast, with the result that they become more uniformly distributed among the relatively more numerous yeast cells. According to the author, this process is employed in the manufacture of 91 per cent. of the total molasses spirit produced in France. No sterilisation is necessary, and the amount of sulphuric acid employed need not exceed that required to neutralise the molasses.

## CHAPTER III

### THE MANUFACTURE OF INDUSTRIAL ALCOHOL FROM BEETS

*Alcohol from beets.*—The sugar beets used for distilling purposes are not of so fine a strain as those used for sugar manufacture. Their juice is of less density and is more impure. When a new variety of sugar beet, richer in sugar than those formerly in vogue, has been discovered by pedigree selection, those formerly in favour are degraded from sugar-house rank and consigned to the distiller. When a beet is pulped and the juice pressed, the juice contains, besides sugar and water, soluble mineral salts, soluble albumen, and other organic substances, such as certain acids, asparagin, etc., as shown in accompanying table.

TABLE XIV.—COMPOSITION OF SUGAR BEET AND SUGAR-BEET JUICE.

	Per cent.
Water.	79 to 84·5
Solid matter:—	
(1) Soluble in water, 11·5 to 17·0 per cent.	
(2) Insoluble in water, 4 to 5 per cent.	15·5 to 21·0
Ingredients in juice:—	
(1) Water.	80
(2) Solid matter.	
(A) Sugar, 15 per cent.	
(B) Non-saccharine matter, 5 per cent.	20
(a) Ash:—	0·8
Sulphates, phosphates, silicates, and nitrates of potassium, sodium, rubidium, vanadium, calcium, magnesium, iron and manganese.	
(b) Oxalates, citrates, malates, and succinates of the same metals transformed by combustion into carbonates.	
(c) Nitrogenous constituents: proteins (albumen, etc.), asparagin ( $C_4H_8N_2O_3$ ), and various amides; betaine ( $C_5H_{11}N_2O_2 + H_2O$ ); glutamine ( $C_3H_5(HN_2)(CONH_2)CO_2H$ ); leucine ( $NH_2C_5H_{10}CO_2H$ ); tyrosine ( $C_6H_4(OH)(C_2H_5)(NH_2)CO_2H$ ),	1·6

(d) Non-nitrogenous substances : raffinose ( $C_{18}H_{32}O_{16} + 5H_2O$ ); arabinose ( $C_5H_{10}O_5$ ) <sup>n</sup> ; dextrine ( $C_6H_{10}O_5$ ) <sup>n</sup> ; Soluble pectic substances; chlorophyll; chromogene; fat; coniferin ( $C_{16}H_{22}O_8 + 2H_2O$ ); vanillin ( $C_8H_8O_3$ ).	1·0
(e) Cellulosic substances,	1·0
Pectic bodies and colouring substances,	0·6
(f) Cholesterin ( $C_{26}H_{43}OH + Aq$ ).	1·6

Distillery beets contain, as a rule, rather less sugar and conversely more saline matter than that shown in the above table.

*Valuation of beets for distillery purposes.*—Beets are valued for distillery purposes by the density of the expressed juice. The hydrometer is graduated into Excise degrees; thus 7·0 Excise degrees means that the juice has a density of 1·070. But even the most rule of thumb distillers are ready to admit that there is nothing more deceptive than the *hydrometer* in determining the value of beets, and that the poorer the beet, the greater the liability to mistakes due to its exclusive use. In buying beets the percentage of sugar should alone be the sole basis of contract with the distiller, as with the sugar manufacturer, since the alcohol comes solely from the sugar. The salts, let it not be forgotten, though they contribute to the density, are eliminated with the spent wash. But, whilst in a sugar works each lot of beets is tested by the saccharimeter,<sup>1</sup> there are few agricultural distilleries where the instrument is in current use. It is used, occasionally, in the course of a “campaign,” for example, when very high or very low beets are under treatment, just “to get a rough idea of what they do contain,” but it is not yet established on the working bench, along with the microscope and the acidimeter. Whether distillery beets continue to be bought by density, or by contract, matters little; it is desirable, however, to establish that the distiller has an immediate interest in keeping an exact account of the percentage of sugar in his beets, if only to ascertain whether the alcohol extracted corresponds with their sugar content. Besides, the rational use of this apparatus tells the distiller that the beets of equal density of such and such a district, of such and such a farmer, and from

<sup>1</sup> *i.e.*, the polariscope, see the author's *Technology of Sugar*, published by Scott, Greenwood & Son, London.

such and such a field, are more saline or more saccharine, than others, that certain strongly manured fields produce beets of sufficient density, but poor in sugar, and hence poor in yield ; on the other hand, that in such a parish, in such a district, in consequence of the nature of the soil, or the kind of manure employed, the beets grown there are purer and their density not factitious but a real alcohol producer. He will observe, amongst his own growers, those who, with equal weight, deliver the most sugar, and, fortified by all these observations, he will buy so that, with an equal outlay, the amount of alcohol obtained, due to this foresight, will be considerably more than that of his neighbour, who allows himself to be guided solely by the misleading indications of the hydrometer. It must not be lost sight of that for densities bordering on  $7^{\circ}$  the sugar content corresponds closely with that generally accepted, viz., twice the density. This coefficient decreases in proportion as the density becomes lower, so that a beet at  $4^{\circ}$ , for example, rarely yields more than 6.5 per cent. of sugar. Densities of 4.2 have been known not to reach 6 per cent. A typical case, submitted to expert distillers, is the following : “ How is it that the Excise finds that this year I required 150 kilos (3 cwt.) of beets more than last year to produce a hectolitre (22 gallons) of alcohol, whilst the average density of my beets is, at least, as high as that of other years, and the distillery is in good working order ? ” The report on examination is : That the system of fermentation is good ; pulp, wash, exhaustion, normal ; no loss during extraction. Examination of plant shows no leakage, neither in condensers, nor anywhere else. Everything is in good order. We can only come to the conclusion that since nothing is being lost we ought to find again as alcohol all the sugar introduced, and reciprocally, if the alcohol be deficient, it is the sugar itself that is present in smaller quantity. The distiller, if questioned, will declare that the season before, beets being scarce, he had bought almost anything that was offered to him, and that numerous lots of beets which in ordinary seasons would have been used for cattle feeding had been consequently mixed with his usual beetroots. The cause of the deficiency is found. The

density of the beet being that season in general rather above the average, the average density of the deliveries reached almost those of previous years ; but the salts, introduced by these beets, the purity of which was deplorable, could not take the place of the sugar in the fermentation, hence the deficiency in the yield of alcohol. Had the distiller, from time to time, made some saccharimetrical tests, as a guide to the real value of certain lots of beets, he would have refused them ; and thus avoided working at a loss, and have been spared several weeks' worry in trying to find the reason for the deficiency in his stock. K. Windisch<sup>1</sup> gives the yield of alcohol per 100 kilos of roots, containing 16 per cent. of sugar, as 9.5 to 10 litres.

The first stage in the process of manufacturing alcohol from beets is the washing of the beets and the elimination of stones, etc. This is done in the apparatus shown in Fig. 6. The beets are brought by truck to the foot of the archimedean screw elevator, which lifts them and carries them to the washing machine and stone eliminator, from which they pass by the chain elevator fitted with cups, which carry them up and drop them into the beet-slicing machines (Fig. 10, H), from whence they descend into the conveyor which feeds the diffusers (Fig. 7 ; Fig. 10, I).

*Feeding the washing machines.*—The cheapest way of feeding the beet washers is by *hydraulic carriers*, a narrow channel or sluice of about 20 inches wide, rounded at the bottom, running right through the factory yard, in which a stream of water flows rapidly. When the beets are thrown into this stream, as their density is only slightly greater than that of water, they are carried along with the current to the end of the channel, which dips into the washer. There is no fear of a barrow load of beets, thrown in at one point, stopping the flow of water, because at that point the level of the liquid rises, passes over the heap of beets, carrying away the top ones in its train, disintegrates the heap, and, under the ever-increasing pressure from behind, forces the individual beets to float rapidly to the washer. The yard may be intersected by similar channels, and the labour in handling the beets is much diminished. If the different channels be

<sup>1</sup> *Z. Spiritusind.*, 1914, 37, 580-581.

completely covered with boards, the heaps may be piled above the channel itself, and by lifting the boards in rotation a single labourer can dismantle them from the one end of the heap to the

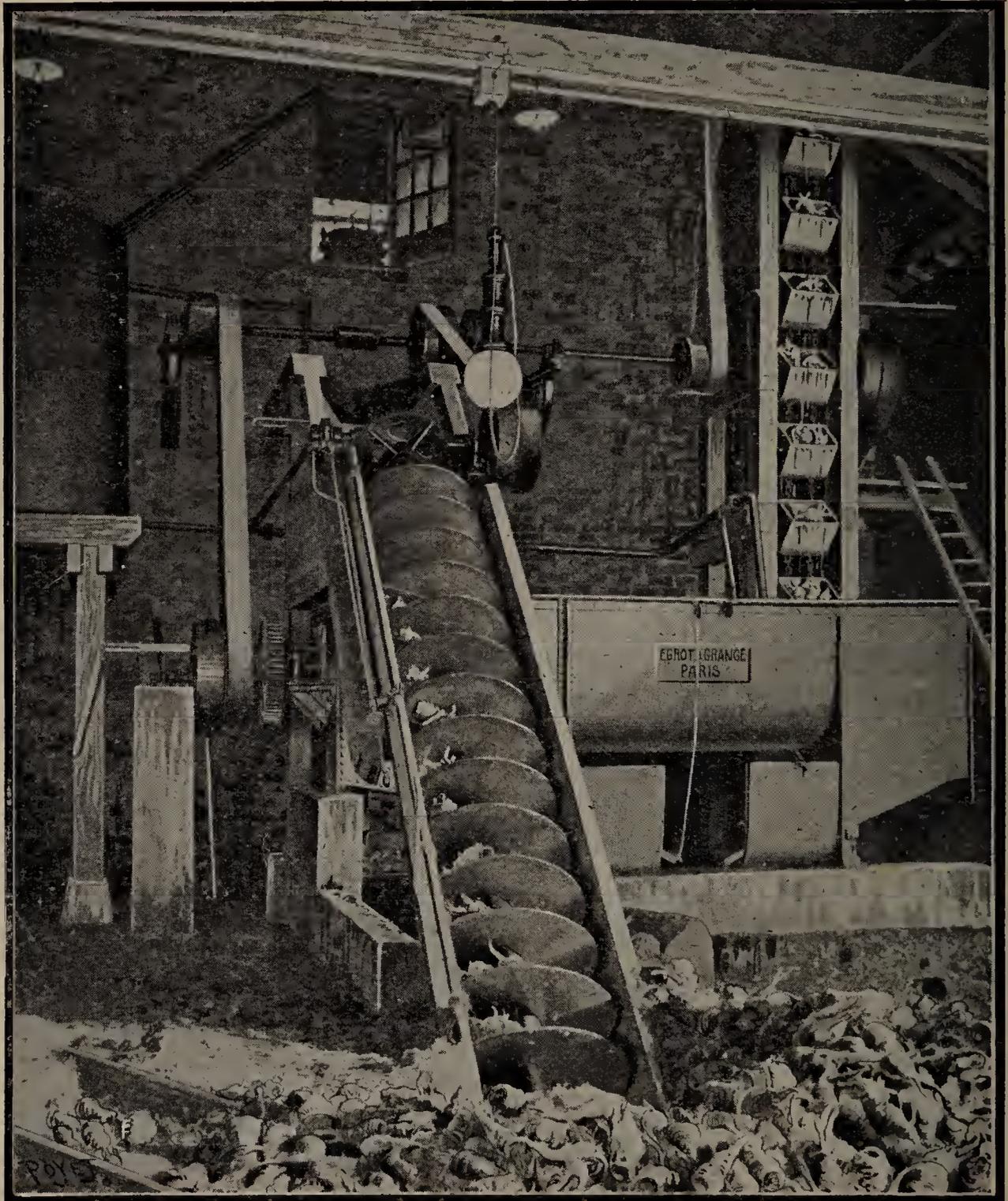


FIG. 6.—Screw elevator for beets—Beet washer—Stone eliminator—Cup elevator (EGROT and GRANGÉ).

other, and thus feed the washing machine at the same time. The *hydraulic carrier* is thus the most convenient and cheapest method of transport when there is a fall towards the factory. Its feed water is used for the washing machinery, for which there

has to be a very abundant supply. Whatever method of transport be adopted, the beets have first to pass through one or two washing machines, to free them from the mud with which they are encumbered, a very difficult operation when they have been lifted in wet weather on heavy soils.

*Beet-slicing machinery.*—The essential part of a beet-slicing machine is a circular horizontal plate revolving rapidly round a vertical shaft. This plate is pierced with apertures in which knives, arranged like the cutting edge of a plane, are inserted. If the beet, therefore, comes in contact with this revolving plate the knives will plane the beet, and the slices thus cut off fall beneath. Moreover, if the knives have an appropriate shape, the shavings or slices cut off will have the desired roof-ridge-tile shape. The revolving plate is enclosed in a frame surmounted by a hopper, and shaped underneath like a large funnel. The beets are fed into the hopper, and, as the latter is sufficiently high, *i.e.*, 20 to 40 inches, the weight of the beets above, pressing on those below, which rest immediately above the plate, act as an automatic pusher, causing the latter to be caught by the revolving knives and thus to be cut into slices of the desired size and shape. The beet-slicer is thus nothing more than a special adaptation of the turnip-slicer so very extensively used in Britain for cattle-feeding purposes. There are eight to ten apertures in the plate, into which the blades are fixed. The knives are not fixed directly into the plate. They are mounted in movable, easily changeable frames, called knife-holders, and it is these knife-holders which rest in the apertures of the plate. As the knives soon become blunted, there is always a complete complementary set, adjusted beforehand, in reserve, so that when the knives at work do not cut properly, the knife-holders with the blunt blades are removed bodily, and immediately replaced by fresh sets (page 55). The change is thus effected very rapidly, for the construction of the apertures is such that the change involves no difficult operation, the knife-blades simply fitting into the grooves which hold them fast during rotation. They are, in fact, kept in place by centrifugal force. The plate is driven by conical gearing acting on its shaft, the gearing being driven by pulleys and belts. The

shaft rests in a socket, and is kept in the vertical position by strong plummer-blocks surrounding it on the top so that it cannot become displaced. The diameter of the plates is variable, the average being 5 feet—some are 3 feet and others over 6 feet. Plates of great diameter are much in vogue in Austria. But as the speed of the knives ought always to be the same, plates of large diameter must be made to revolve more slowly than smaller ones. Plates of 5 feet in diameter make 100 to 120 revolutions, so that  $6\frac{1}{2}$ -feet plates need only make 60 to 90 revolutions for their circumference to have travelled the same distance as the 5-feet plates in the same time.

Knives may be divided into three classes: (1) *Naprawil knives*.—The first, the oldest, called Naprawil knives, make rectangular slices. They consist of a straight cutting blade surmounted at intervals by cutting ridges, which divide the slices cut off by the knife into smaller sections. (2) *Goller knives*.—The second class of knives, known as Goller knives, make triangular slices. They are steel blades  $\frac{1}{8}$  to  $\frac{1}{6}$  inch in thickness, cut in the body in a zig-zag form at an angle of  $60^\circ$ . They are also made of wrought-iron bent into an undulating form having the same profile. The beet can thus be cut into *triangular* slices. But the form of the slice is quite irregular, because, when the knife passes through the beet, it leaves its triangular mark upon it. When the next knife comes into play, it therefore cuts the ridges of the triangle, forming an irregular-shaped slice. This is bad, because the new slice has not the thickness requisite for good diffusion working. The thin slices are exhausted sooner than the thick ones, and the exhaustion of the latter may be imperfect and incomplete. (3) *The roof-ridge-tile-shaped knives*.—The third class of knives, the most extensively used, combining the principles of the Goller and Naprawil knives, are the roof-ridge-tile shape. They have the same profile as Goller's knives, only on the upper part the summit of the angle carries cutting ridges like the Naprawil knives. When a knife has passed, leaving its mark on the beet, when the next arrives it passes into the same furrow, and therefore lifts a roof-ridge-tile-shaped slice of perfect shape, and sharply cut on all its faces. Care must be taken to mount

the knives so that their blades are correctly placed one behind another. The knives are mounted in a knife-holder like the block of a plane. The slope of the knife and the length of steel which overlaps the plane of the knife-holder is varied, so that the knife may project more or less according to whether the slice is to be larger or smaller. The knives are sharpened on very hard steel discs, which revolve rapidly on their axes, and the circumference of which is dressed like a file. The profile of the bottom of the blade is given to the circumference, and it is enough to place the knife in front, firmly held in a clip which guides it, and to press lightly with the hand, to sharpen its cutting edge. There are also similar discs for sharpening the cutting edges of the ridges. A knife is thus sharpened very quickly. The sharpening is finished by files, also of the desired profile or shape. Some knives are made of hardened non-tempered steel, and are sharpened immediately they are taken out of the beet-slicer. These are the class of knives most generally used. Some, made of tempered steel, require softening before sharpening, and tempering afterwards. The operation of sharpening the knives requires great care to do it well, and consequently necessitates a special smithy and a skilful, experienced smith; it is therefore practised in but few factories. The knives in current use only have been mentioned. There are others derived from the three described, only differing in unimportant details.<sup>1</sup>

*Extraction of beet juice by maceration.*—About 1830, Mathieu de Dombasle (1777-1843), a celebrated French agriculturist, who not only in his day made improvements in many agricultural implements, but who was also one of the original creators of the beet-sugar industry, introduced a maceration process for extracting the juice from the sugar beet without pulping, rasping, or grating, and without hydraulic presses. The beets were cut into thin slices by a rotary machine. The slices were then transferred to the first of a series of casks arranged in the form of a battery, the juice from No. 1 being run on to No. 2. The beet slices were macerated in No. 1 for about an hour with an equal bulk of water, at a temperature of about 212° F. After this treatment the

<sup>1</sup> See author's *Technology of Sugar*.

liquid—having now acquired a density of 2° Baumé, sp. gr. 1.014—was run into No. 2, containing fresh beet slices. From No. 2 it was run into No. 3, and so on until it had passed through No. 5, charged in the same way, when, its density having reached 5½° Baumé, sp. gr. 1.040, it was suitable for defecation. No. 1 was thus charged with hot water, and No. 5 yielded a juice suitable for further treatment. To obviate cooling, it was re-heated in its passage through the casks, the maximum amount of sugar possible thus being extracted. The exhausted slices scarcely contained any sugar. Juice obtained thus, although transparent and requiring little lime for purification, was liable to ferment, or, owing to the dilution water, it was difficult to granulate. Hence the process was generally abandoned as far as sugar works were concerned, since the juice obtained in this way could not be successfully treated by the method then in vogue for pressed juice, and because the exhausted slices were too wet for cattle-feeding. The wet nature of distillery beet diffusion and maceration pulp is one reason why extraction of beets by pressing still prevails in districts where the farmers are prejudiced against maceration or diffusion pulp. When carbonation (alternate treatment of beet juice with lime and carbonic acid) led to the adoption of diffusion in sugar works, its employment by distilleries followed as a natural sequence. It was first used in a distillery by E. Barbet at Telques in 1880.

*A diffusion battery* consists of a series of eight to fourteen cylindrical vessels called *diffusers* arranged consecutively. These communicate with each other by piping, so that the juice issuing *from the bottom* of one diffuser flows into the next *from above*. The current may be reversed by taps if need be, so as to pass from top to bottom of the diffusers, instead of from bottom to top, or the juice may be heated during its passage from the one battery to the other. A steam reheater, or *calorisor*, keeps the liquid always hot. Again, taps are so arranged that water may be run into each diffuser, instead of juice. There are also taps for running off the liquid after the beets have been exhausted. The diffusers have a top door for charging them with fresh beet slices, and a bottom door through which the exhausted beet slices

are discharged. Supposing all the diffusers are charged with fresh beet slices, there will still be a certain amount of vacant space between the slices. When enough water is run into the diffuser to occupy this vacant space, the diffuser will then contain about equal weights of water and beet slices. Not only so, but the space occupied by each is also almost identical, the density of the beet being only slightly above that of the water. If the first diffuser of the series be now charged with water, *osmosis* at once comes into play in the cells of the beet so as to cause a certain proportion of the sugar which they contain to pass into the water. The batteryman, however, does not wait until equilibrium in density is established between the saccharine fluid in the beet cells and the exterior saccharine liquid, since that would take too long. After a few minutes' contact, however, there is no very great difference in density between the two. The liquid from the first diffuser is then run into the adjoining diffuser, being reheated in its passage. The saccharine liquid is now in contact with fresh beet slices, the juice of which is of greater density; *osmosis* is again energetically started. It thus soon becomes still more highly charged with sugar, until, in fact, the densities are nearly equal. The saccharine liquid is once more run into a fresh diffuser, and the same interaction of fluids takes place, and the same operations are continued until the density of the liquid is only slightly inferior to that of the primitive juice. The *osmotic* action is then almost *nil*. If the crude juice be allowed to stand for some time, the pectic matters ferment and are transformed into two gelatinous acids—the pectic and the pectosic. The juice then segregates into a jelly, or, if diluted with water, it strings, or becomes ropy like certain white wines after they have gone wrong.

*Simplified diffusion.*—The system of simplified diffusion installed in the distillery of M. Postel (Fig. 10), consists of six diffusers in line; they have a capacity of 12 hectolitres (264 imperial gallons) each, and are 13 to 14 feet (4 metres) in height. The body of the diffuser is cylindrical or very slightly conical, being wider at the bottom. The bottom (Fig. 7) of each diffuser is covered with a discharge door, which on being opened the

whole contents of the diffuser are discharged directly into a truck underneath. The opening (Fig. 7) and the closing of this door is manipulated from above by the man in charge of the diffusers (Fig. 10, I, J); for each diffuser a single tap enables

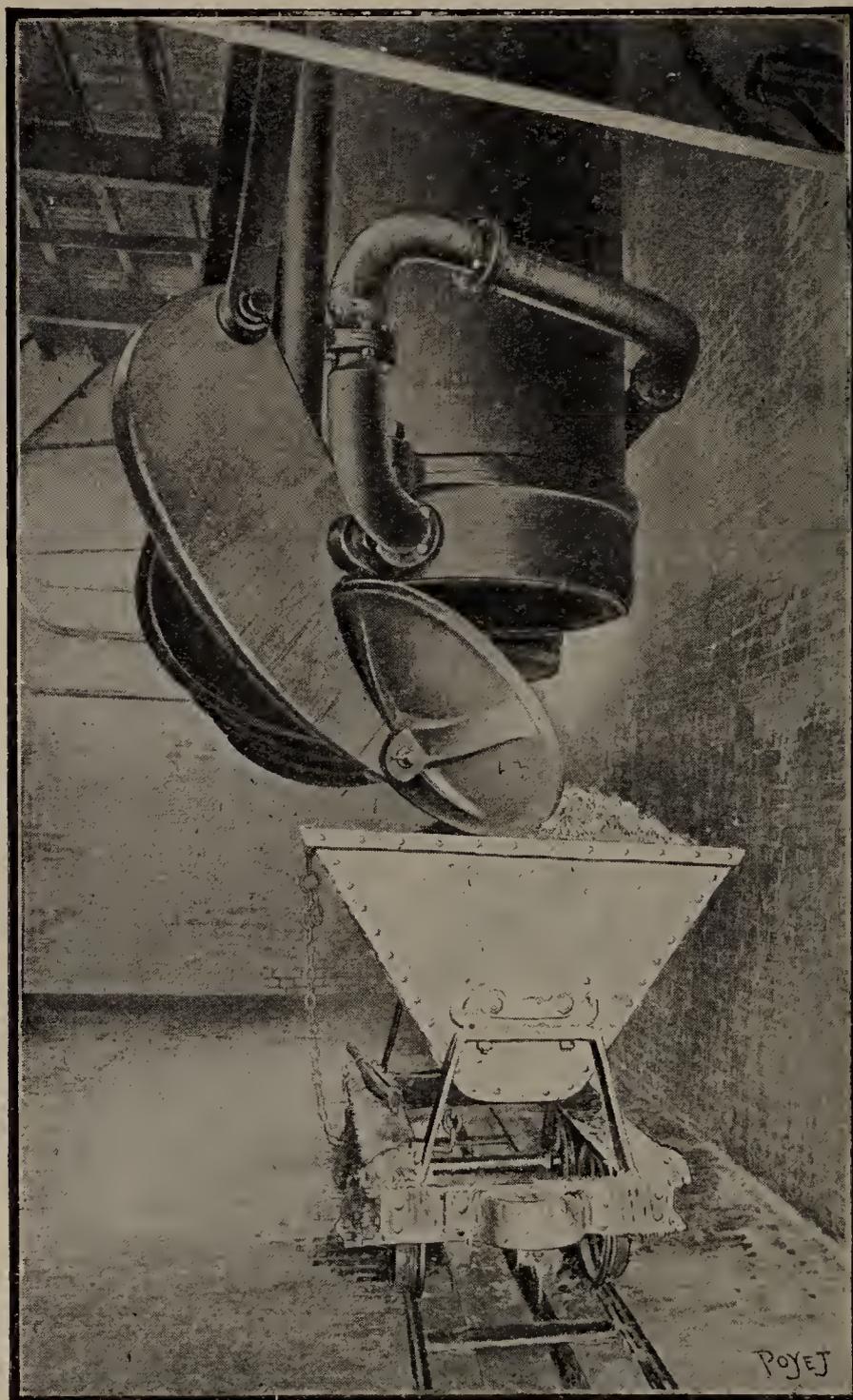


FIG. 7.—Beet diffusers (bottom part) showing doors and method of discharge of spent slices into lateral tilting trucks on rails *en route* for spent pulp silo (Fig. 10) (EGROT and GRANGÉ).

him, at will, to run in vinasse, establish circulation with the adjacent diffuser, or to run the juice to the measuring tank. Moreover, the weak juice does not issue from the battery, but is directly passed on to the next diffuser by driving it from the

end diffuser by compressed air. Further, a special but very simple arrangement enables either vinasse or compressed air to be run into the tail-end diffuser, and the latter under such constant pressure as may be desired, so as to have a very active circulation in the battery in spite of the extremely reduced section of the diffusers. The working and management of this system of diffusion is thus rendered very simple, and requires only a very small covered-in space as compared with either ordinary diffusion or maceration. The juice runs from the measuring tanks into a tubular Egrot & Grangé cooler, with great circulation and easily removable tubes, where it is cooled to 23° C. (77° F.) prior to proceeding directly to the fermentation vats (similar principle to Fig. 4).

A common fault, in distillery working, whichever method of extraction be adopted, consists in exaggerating the proportion of juice withdrawn per kilogramme of beets. The over-extraction is made for fear of not sufficiently exhausting the slices ; but, very often, the volume of juice withdrawn may be very naturally reduced without the sugar increasing in the pulp, on that account. The three factors which determine exhaustion must be better supervised : (1) the state of division of the pulp or slices and the uniformity of the same ; (2) the temperature in the macerators or diffusers ; (3) the length of contact. Very often an attempt is made to remedy a defect in one of the above three factors by increasing the proportion of juice withdrawn. It would be more rational to see that these three points received due attention and were in harmony with each other. The juice withdrawn could thus be reduced to a minimum, exactly as in a beet-sugar works. The advantages to be gained from this reduction in volume of extracted juice are an increase in the output, and reduction of the general expense. The further treatment of the juice, whether it be evaporated or distilled, being on a smaller bulk, results in economy. The distiller, when producing a smaller volume of juice, need not fear that it will be too concentrated, juices of 3.5° to 4° ferment quite as well as those at 2.5°. The expense of acid will be less, the only thing requiring to be supervised being the exhaustion of the pulp. Over-extraction is therefore

perfectly useless, if extraction has been well done ; it is only a costly palliative for imperfect extraction.

*Continuous fermentation.*—The system of fermentation practised at Dammard Distillery is peculiar. Formerly there were three modes of fermentation. (1) Fermentation in successive vats, the fermentation of the juice or the wort being induced by a new ferment, pure or not of greater or less value. (2) Fermentation *par coupages* in successive vats, the *coupages* being furnished by a unique mother vat, or vat nurse, the life of which might vary from one to eight days or more. The function of the mother vat or nurse is to furnish all the necessary *coupages* to induce the fermentation in the fermentation proper ; the fresh juice flows, therefore, concurrently into the mother vat to sustain it, and into the fermentation vats, which have received the *coupages* from the mother vat, to fill them. (3) Fermentation by *coupages* in successive vats as before, only that each fermentation vat becomes successively and in rotation the mother vat to furnish the *coupage* necessary for one of the succeeding vats. This method of fermentation is only differentiated from the preceding by the fact that the mother vats are integrally renewed and rejuvenated by this system. It is this last method that is the most generally adopted in beet distilleries, the second method described relative to periodic or permanent mother vats being only an exception, leaving out of account those factories producing strong leavens from pure ferments. Now the method adopted at Dammard differs essentially from these three methods. It constitutes real continuous fermentation effected constantly in the same vat, which is never emptied nor distilled except, of course, at the end of the campaign. It *alone* receives the fresh juice, and absolutely the *whole* of the fresh juice produced by the diffusion. This vat is not, therefore, a mother vat, nor a nurse vat, as it is still improperly termed, to use an already known designation. It is this vat which supports the whole fermentation ; no other vat receives juice to ferment, and none can, in fact, receive it, as the piping does not admit of it. To send the fermented wort to the distillery, a continuous draw off is made from the fermentation vat, and the rate of flow is regulated so as to maintain always in

the latter the correct volume of juice for proper fermentation to the desired extent of all the fresh juice which it receives. This fermented juice is run into a *cuve de chute et de liquidation* (falling vat), in which the fermentation subsides, and from which it passes to the direct distillation and rectification plant. At Dammard Distillery, fermentation is performed in this way, in two twin vats AA instead of a single one of double capacity, so as to facilitate installation, and the liquidation of the fermented juice issuing from these vats is accomplished by means of four *cuves de chute* BBBB of much smaller capacity. It may be added that three of these suffice after working for fifteen days and at the full capacity of the factory. Fermentation is very fine, very active, and very complete, being maintained at 29° C. in the fermentation vats without ever going beyond 30°, and falling towards 26°-27° in the *cuves de chute*. The vinasse or residue from beet maceration is used for cattle feeding, but is of lower value than that from potatoes.

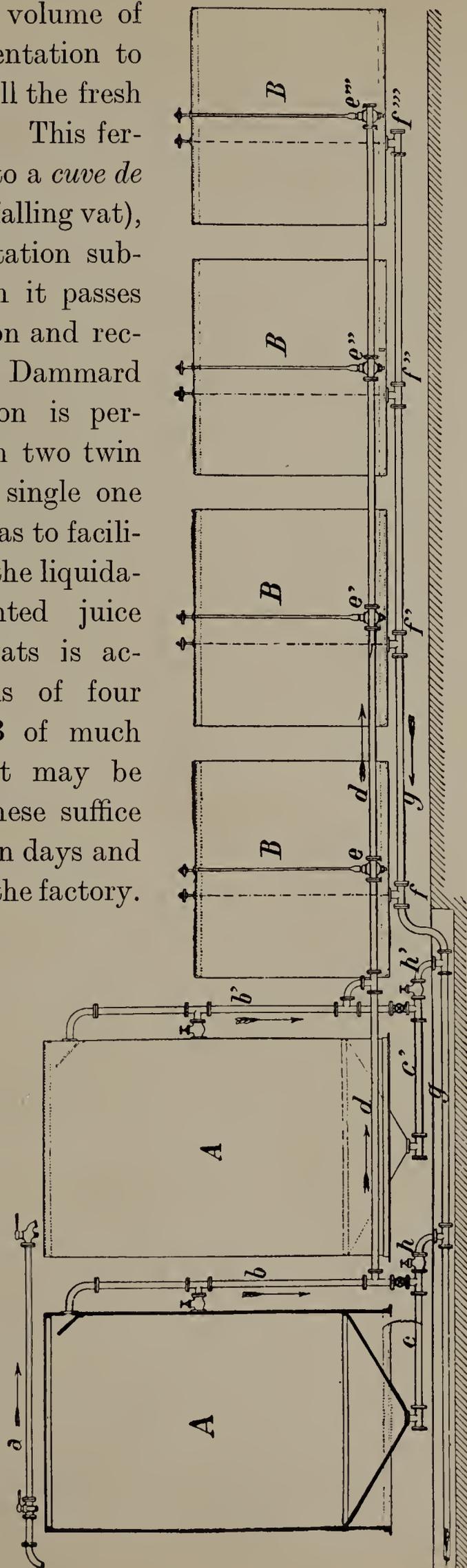


FIG. 8.—Diagrammatic illustration of Fermentation Department of Dammard Beet Distillery (Fig. 10, M, N, O) (EGROT and GRANGÉ). AA, chief fermentation vats; B, “Cuves de chute;” a, juice pipe; b b' c c', draw-off pipes; d, feed pipe to BBBB; e e' e'' e''', feed taps; f f' f'' f''', exit taps; g, pipe to distillery; h, pipe to discharge A (either vessel into g (if need be)).

*Antiseptic fermentation.*—Fermentation is often initiated by stirring a quantity of beer yeast into a portion of the beet juice, and when fermentation has become vigorous, letting the juice flow in ; when in full swing several fermenting tuns are kept at work simultaneously by starting a fresh one when the end one is full. That is working by *coupage*, or mixing of the tuns, almost exclusively used in distilleries working with beer yeast. In some distilleries the use of beer yeast has been abandoned on account of its doubtful purity. Starting from the principle that to obtain a really pure ferment it is necessary that the yeast itself be perfectly pure, ferments are used which are carefully protected during cultivation from all contamination. The disadvantage of the use of these pure ferments is that they require expensive plant, *cuves de reveil* (rousing vats) (Fig. 5) and ferment tuns of different capacities. Besides, the precautions required to prevent contamination are somewhat too elaborate and difficult to realise with not too intelligent workmen. Another disadvantage of this system is that the aseptic fermentation cannot be maintained to the end. The juice of the beet contains at certain periods a crowd of bacteria and foreign ferments, the spores, at least, of which resist the imperfect pasteurisation produced in the apparatus which exhausts the pulp. Consequently, whilst admitting that contamination by extraneous germs is avoided, contamination from those contained in the juice itself cannot be prevented. If the portion of the beet juice required for the making of the leaven be sterilised—which uses up an appreciable amount of heat—and these remain pure, contamination will perforce occur as soon as it reaches the large tun into which the juice coming directly from the presses, the macerators, and the diffusers flows after passing through condensers, gutters, and pumps, which too often are highly infected with bacteria and moulds. This contamination will make itself manifest in a more apparent manner if juices are decomposed. Some distillery specialists, therefore, urge that the system of true aseptic fermentation can never be applied integrally in the working of beets.

There is therefore, they argue, some justice in the remark,

“ Why ask us by the use of costly plant and elaborate precautions to refrain from introducing foreign ferment into our leaven, if it only be to mix the same leaven in a state of purity with a considerable quantity of unsterilised juice ? ” The partisans of this system reply, insisting too, and rightly so, that by commencing each tun with pure leaven, if the fermentation does not maintain throughout its initial purity, it will always be less imperfect than in the system of mixing the tuns, where the contamination in one tun may affect all which succeed it until the vats are cleaned and the leaven is renewed. The system of fermentation which includes a pure leaven for each tun, or the system of “ mother ” tun which is analogous thereto, is therefore preferable when the initial stage is carefully maintained pure. But if fermentation in an aseptic medium is not wholly applicable in beet distilleries, the difficulty can be surmounted by fermentation in an antiseptic medium ; that is, by rendering the wash incapable of developing bacteria by the addition to the beet juice of suitable antiseptics. Sulphuric acid has itself a very decided antiseptic action, but other agents, and amongst them hydrofluoric acid and fluorides, possess this power in a high degree. Generally, when fermentation is effected in antiseptic washes, a commencement is made with leavens acclimatised to a strong dose of hydrofluoric acid. According to the purity of the juice, a greater or less proportion of hydrofluoric acid is used. The following experiment was with an ordinary yeast fermentation fed by butyric juices. The fermentation was active but impure, in spite of the large amount of sulphuric acid employed. Fluoride was tried in small doses, and antiseptic effects were at once apparent, so that the dose of sulphuric acid could be diminished by 1 gramme per litre. Now the quantity of fluoride was only 0·010 to 0·015 gramme per litre. Attempts were made to increase this proportion, but the fermentation slowed and became weak, and the use of fluoride had to be suspended for several hours. The same result occurred by a repetition of the experiment, and the dose had to be restricted to the above ; otherwise the leaven itself was affected by the presence of the antiseptic. In dealing with very impure juices from heated or frosted beets, the dose of 3 to 6 grammes per

hectolitre is sometimes necessary ; it follows that beer yeast could barely live in such a medium ; it is necessary to start from pure yeast acclimatised for the purpose. Those leavens which Effront has acclimatised to vegetate in worts containing 36 grammes and more of acid per hectolitre are unaffected by washes containing 3, 6, and 12 grammes of hydrofluoric acid per hectolitre. By these comparatively large doses the most active bacteria are rendered powerless.

The solution of the problem of pure fermentation in the agricultural distillery lies, they say, in the use of pure leaven acclimatised to antiseptics and in the judicious use of these latter in fermentation. This system of fermentation in an antiseptic medium has the great advantage of not requiring for its application any special installation. One can work by leavening or by mixing the tuns. On the other hand, there is no necessity to take any more precautions, or any more care, in regard to cleanliness than in ordinary working. Besides the advantages secured by pure fermentation, increased yield, and a larger quantity of better *bon gout* alcohol, the use of antiseptics allows the use of sulphuric acid to be materially reduced. Finally, by destroying the cause of fermentation mishaps, which it suppresses, it gives absolute security in working.

*Aseptic fermentation in beet distilleries.*—Barbét's plant (Fig. 9) meets all the objections already raised. AA are the measuring tanks of the diffusion or maceration juice. The whole of the juice is sterilised at a temperature approaching boiling, not only to destroy bacteria, but also the saccharogenic diastase present in beet juice, and which, according to Barbet, is the enemy of the invertase of yeast. Sterilisation is effected in the wrought-iron tank C, and to reduce steam and water to a minimum the juice before entering therein traverses a tubular apparatus B, where it receives by methodical exchange the heat of the sterilised juice issuing from C, and entering by the valve R into the tubular vessel ; the very effective refrigerator V completes the refrigeration of the juice. The tubular vessels, as well as all the connection pipes, before use are carefully sterilised by steam valve J on the recuperator, the effect of the steam passed through being com-

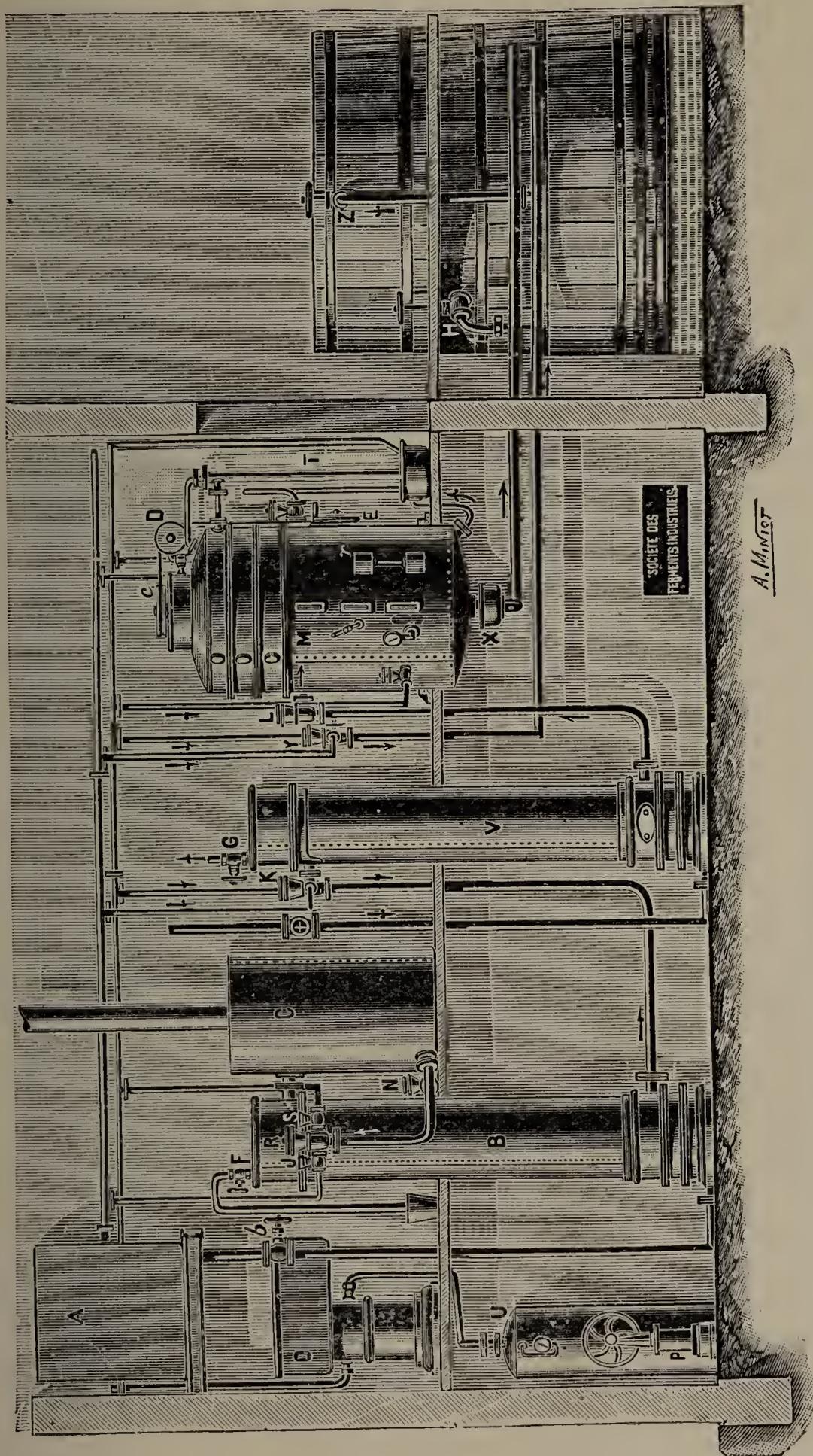


FIG. 9.—Plant for preparing pure aerobic yeast for aseptic fermentation of beet juice (E. BARBET).

pleted by the injection of a little formaldehyde, which is instantaneously diffused by the steam through all parts of the apparatus. The cooled sterilised juice is directed into the yeast apparatus M

through the valve L, or to the tuns Z by the valve Y. The juice should be distributed by pipes, and not by open troughs, so as to reach the tuns without contamination. The advantage of total sterilisation is that the destruction of the saccharogenic diastase enables the diffusion of the juice to be made at as high a density as may be desired, whilst it is otherwise rather difficult to ferment at 4°. Beet juice thus becomes as amenable as molasses, which ferments at much higher densities. Now, since the expense of distillation is proportional to the volume of the wash, there is great advantage in reducing it one or two tuns per day, which effects economy which more than covers the cost of sterilisation. Besides, diffusion costs less in steam when only 120 per cent. of juice is drawn, instead of 160, 180, and even 200 per cent., as is sometimes done in distilleries. The beet sugar works exhaust well with 115, and even 110 per cent., and it is quite as easy to do the same in distillery working (Fig. 10). Half the acid is economised owing to sterility of the juice and its smaller volume; the juice does not require such efficient protection against bacteria. Another advantage consists in not putting any acid in the diffusers and diffusing with water alone, since the butyric and other germs are killed by the steriliser; therefore no more use of perforated wrought-iron, and no more making of antiseptic salt of iron, is necessary. Finally, at the beginning of the season, the first *piéd de levain* (leaven) can no longer refuse to work. It is well to make the first yeast with molasses, so as not to start the diffusion until good yeast is available. Once fermentation is well started in the yeast apparatus, only four-six hours are required to form each batch of yeast, consequently four-six batches of yeast are produced per twenty-four hours. If there are many tuns, one batch of yeast will be distributed between two, or even three tuns, which does not seem inconvenient; the tun treated with yeast will be mixed with its neighbour. If a batch of yeast be desired for each tun, two sets of yeast apparatus will be necessary. This plant has been in operation at the distillery of Marquette lez Bouchain, where it has worked regularly, yielding the advantages just summarised. They have been able, particularly with pure yeast, to bring the juice quickly into active fer-

mentation, and feed the tun rapidly without killing the ferment, which would infallibly be the case with juice not freed from its saccharogenic diastase.

Fig. 10 shows a general view of the interior structural arrangements, and the correlation of the individual machines, stills, and other accessories of the plant and apparatus of a modern beet distillery. It will be observed that with the exception of the conveyance of the beets to the elevator, and the removal of the spent pulp by trucks, everything is done practically automatically.

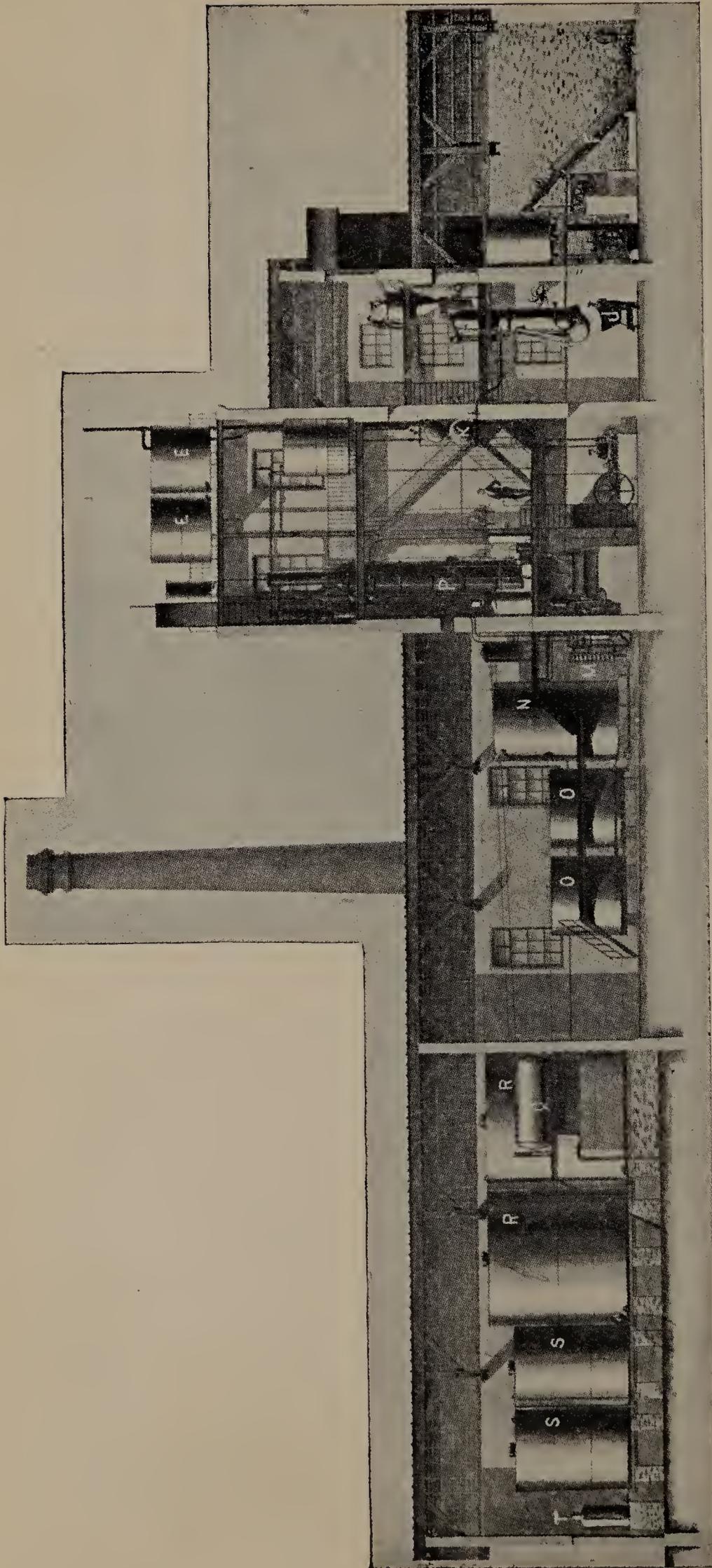
I. MOTIVE POWER.—A, Boiler chimney. B, steam boiler, very capacious, to furnish all the steam required by the whole factory, with two feed pumps, one of which is in reserve. C, Steam engine.

II. WATER SUPPLY.—D, Pump to elevate the water from a well into the two cisterns, E, communicating with each other.

III. WASHING AND REMOVING STONES FROM THE BEETS : BEET-SLICING MACHINES.—F, Elevator which lifts the dirty beets fed into it at the ground-level, where they are shot out of the trucks, and carries them into the washing machine. G, Powerful washer specially arranged to effect the perfect separation of stones before automatically transferring the washed beets into the elevator, which conveys them to the beet-slicing machines. H, Horizontal plate beet-slicing machine producing ridge-tile-shaped slices as in beet sugar works.

IV. DIFFUSION.—I, Diffusers (Line), six in number, forming Guillaume, Egrot & Grangé's simplified diffusion battery; a horizontal conveyor distributes the fresh slices into the diffusion battery. J, Waggon on rails to receive the pulp from the diffusers and convey it to the silos; the waggon is made to hold the contents of a diffuser. K, Receiver for vinasse and compressed air for diffusion; the double feeding of this common reservoir is effected under a pressure capable of being regulated at will by a special pump for the vinasse and by another for the compressed air. L, Measuring-tanks for diffusion juice.

V. FERMENTATION.—These tanks are placed above a small settling tank, which serves to clarify the juice and to aerate it



Rectified alcohol warehouse.

Continuous fermentation.

Continuous direct distillation rectification.

Beet-slicing simplified diffusion.

Beet-washing elevator.

Fig. 10.—Agricultural distillery (beets) installed on the farm of Dammard, belonging to M. Postel, farmer, near La Ferté Milon (Aisne), France, on Guillaume, Egrot, and Grangé's system. Capable of treating 50 tons of beets in twenty-four hours. Interior view showing structural arrangements, with ground plan of same. (Details are given above.)

prior to fermentation. M, Combined cooler and heater, to cool the vinasse to  $75^{\circ}$ - $80^{\circ}$  C. and utilise the heat to the profit of the fermented wash going to the direct distillation rectification plant, and to cool to about  $25^{\circ}$  C. the fresh juice prior to fermentation. This combined cooler and heater (p. 59) is of the multiple and removable tubular bundle type, the same type as that used by Egrot & Grangé (Fig. 3) in the pasteurisation of wine. N, Principal fermentation vats into which all the fresh diffusion juice is run to be continuously fermented. O, Vats into which the fermented wash is run from the principal fermentation vats to allow the fermentation of the wash to completely subside, after which it is sent to the direct distillation rectification apparatus.

VI. DIRECT DISTILLATION : RECTIFICATION PLANT (Guillaume's system specially simplified for agricultural distilleries).—This plant P consumes no more coal than an ordinary distilling column, and is easier to manage ; it produces, on the one hand, and as principal product, a perfectly rectified alcohol, marketable forthwith as such

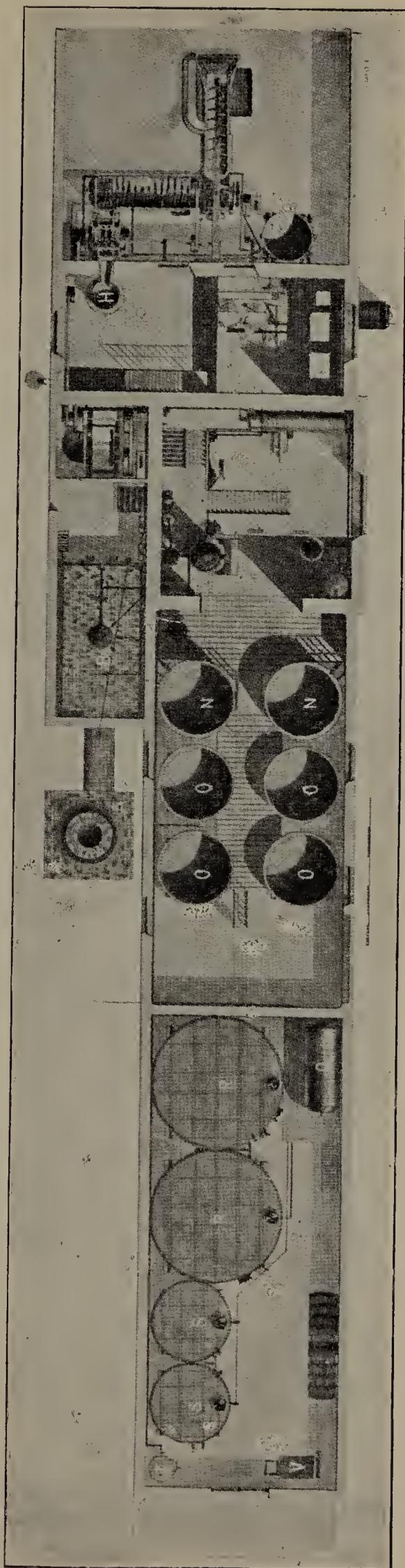


FIG. 10A.—Ground plan of Dan mard Distillery (EGROT and GRANGÉ).

on the Exchange or to retail customers; and, on the other hand, as a by-product, alcohol of the right strength to be sold for methylation. Q, Testing reservoir for testing the rectified alcohol coming from the direct distillation rectification apparatus; this receiver is divided longitudinally into two distinct compartments, into one of which the alcohol produced by the day-shift gang is run, and into the other that produced by the night-shift gang. In this way the alcohol produced by both the day and night shift gangs of workmen can be tested. These compartments are arranged to receive compressed air, by which



FIG. 11.—View of rear part of beet distillery, showing silos, mud settling ponds from washers (EGROT and GRANGÉ). (See Fig. 6; Fig. 10, G.)

the rectified alcohol is pumped after being tested to the vessels allocated to receive it. R, Large reservoirs to receive and mix the rectified alcohol, the quality of which has been verified in Q. These large reservoirs enable samples for sale to be taken which can leave no doubt as to the identity of the alcohol to deliver with such samples. S, Reservoirs for alcohol intended for methylation. T, Cask-filling machine. V, Weighing machine.

The plant was supplied to treat 50 tons of beets per day of an average density of 6°. The buildings were specially built to the plans of the constructors of the plant. The natural slope of

the ground was utilised so as to facilitate the rolling of the trucks bringing the beets from the silos to the washer, and of the truck running the spent pulp from the diffusers to the pulp pit. Moreover, the general situation favoured the evacuation of the muddy liquors in the mud ponds, the overflow from which flowed freely on to the land at a lower level, thus fertilising it with the plant food contained in the portion of the spent wash which was run out, and in the drainage water. The pulp silos are excavated out of the ground. The truck bringing the pulp is tilted directly into the pits, the foundation of which is on a slope, so as to ensure the evacuation of the drainage water. A single labourer suffices at any one time to attend to the bottom discharge of the diffusers and the trucking of the pulp to the silos. The pulp silos abut on the road, and the masonry foundation being on a level with the road, the carts can go on to the very centre to get loaded, and carry it as required to the rather far-distant farm-steadings. It will be seen from Figs. 14 and 15 that the selection of a site for a beet distillery is a matter for very serious consideration, and that a slight eminence or rising ground with advantageous slopes presents many useful features from the point of view of economical working.

STAFF.—For a turnover of 50 tons of beets daily, the inside staff, each shift (day and night) of the Dammard Distillery comprises : (1) A man for the diffusion ; (2) a man to attend to the bottom of the diffusers and remove the spent pulp from the silos ; (3) an engine-driver, who also acts as stoker ; (4) an overseer for inside and outside work. The latter supervises, at the same time as all the other departments, the fermentations and the continuous direct distillation rectification, no other workmen being specially told off for either department. The combined stokers and engine-drivers attend to all the lubrication and do all the interior cleaning, and face, temper, and case-harden the knives of the root cutters. The guaranteed daily production of 30 hectolitres (660 gallons) at 100° (*i.e.*, absolute alcohol) was slightly exceeded, and for the month of December, 1905, including all stoppages, was 35 hectolitres (770 gallons). At this speed, in spite of raising water 90 metres (295·2 feet) and the expense of steam for the

electric light, the monthly coal consumption was only 78 tons, say less than 85 kilos per hectolitre of alcohol at 100° (8½ lbs. of coal per gallon of absolute alcohol).

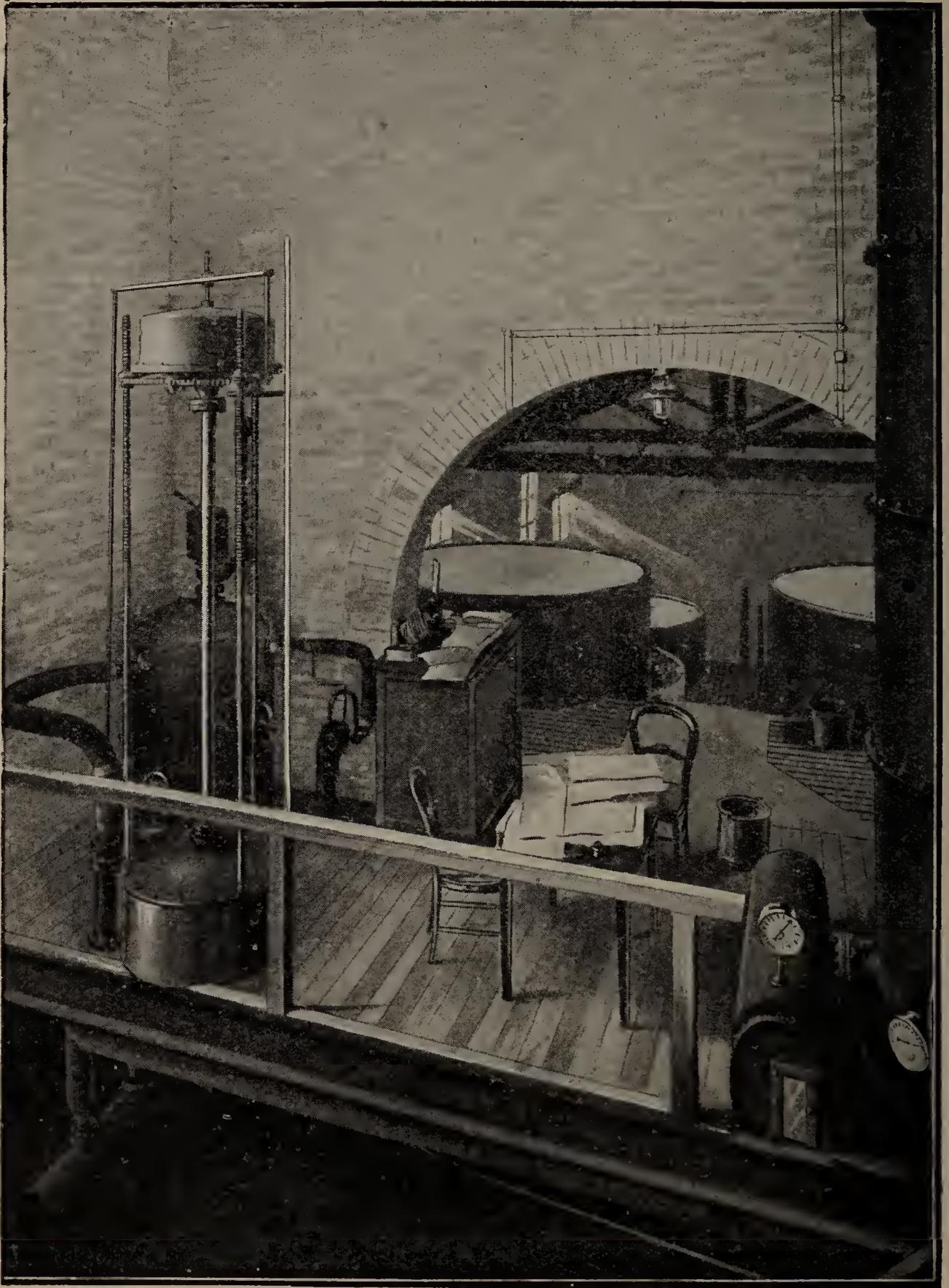


FIG. 12.—Point from which foreman distiller controls plant (shown in Fig. 10), showing Guillaume's steam regulator and dial thermometers of (a) inclined distilling column ; (b) bottom of rectifier (EGROT and GRANGÉ).

*Application of Barbet's pure fermentation process to the distillation of beet molasses.*—The first idea would be to work in an analogous manner to that for beets (Figs. 9, 18), replacing the

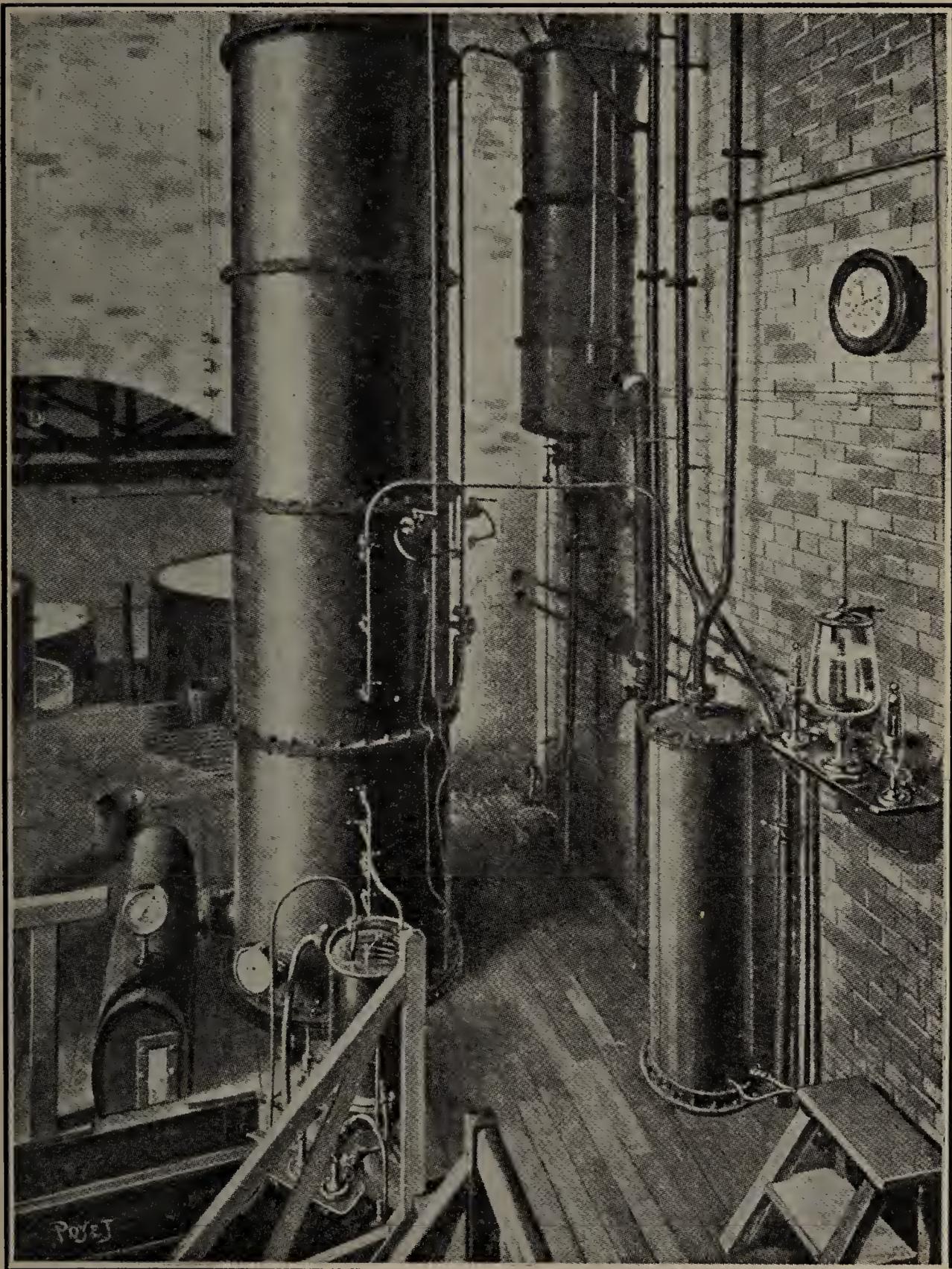


FIG. 13.—Guillaume's distillation, rectification plant (shown in Fig 10)  
(EGROT and GRANGÉ).

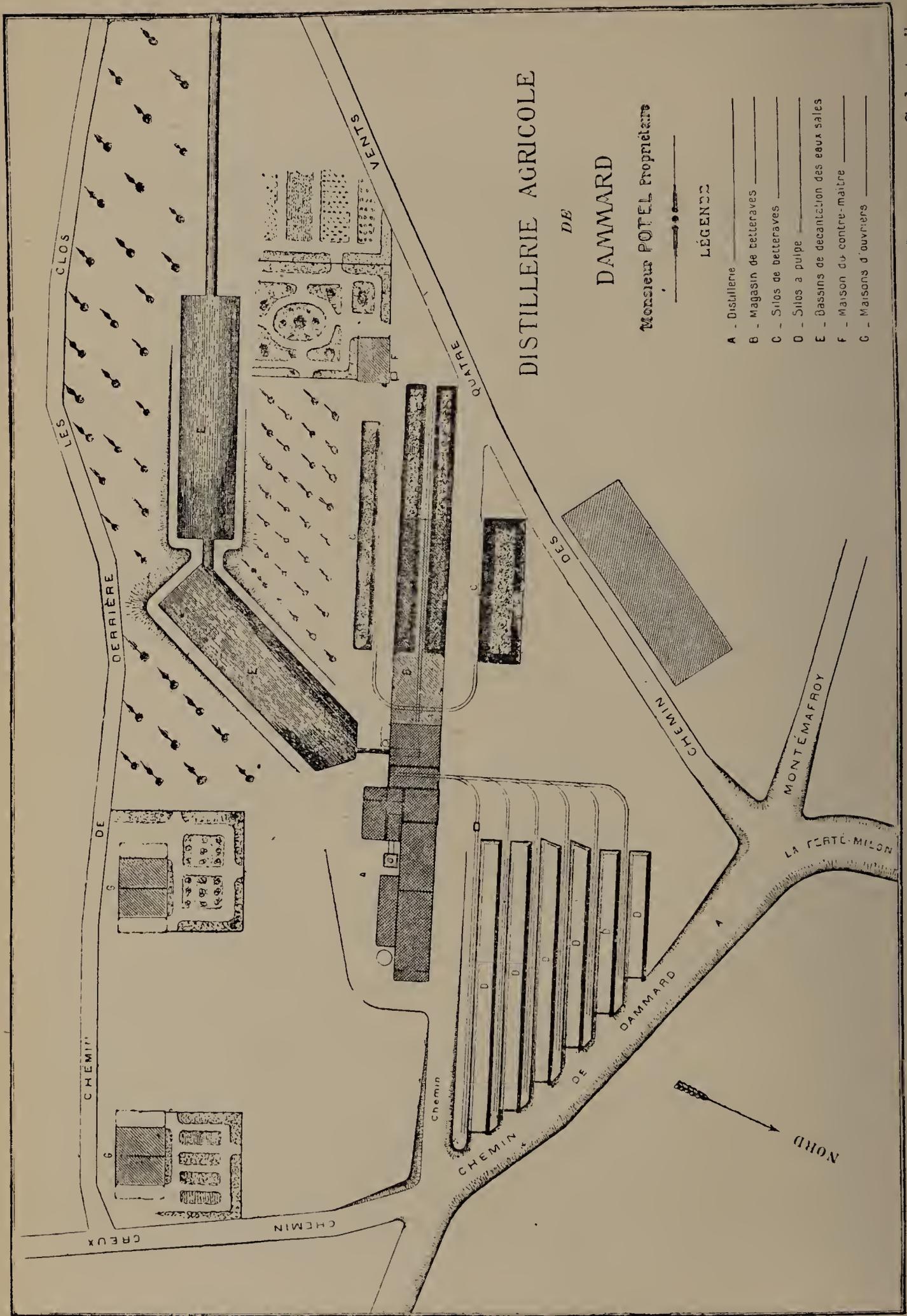


FIG. 14.—General plan of an agricultural distillery and its surroundings (Egror and Grangé). A, distillery; B, beet store; C, beet silos; D, pulp silos; E, settling ponds for muddy water from beet washing; F, foreman's house; G, workmen's houses.

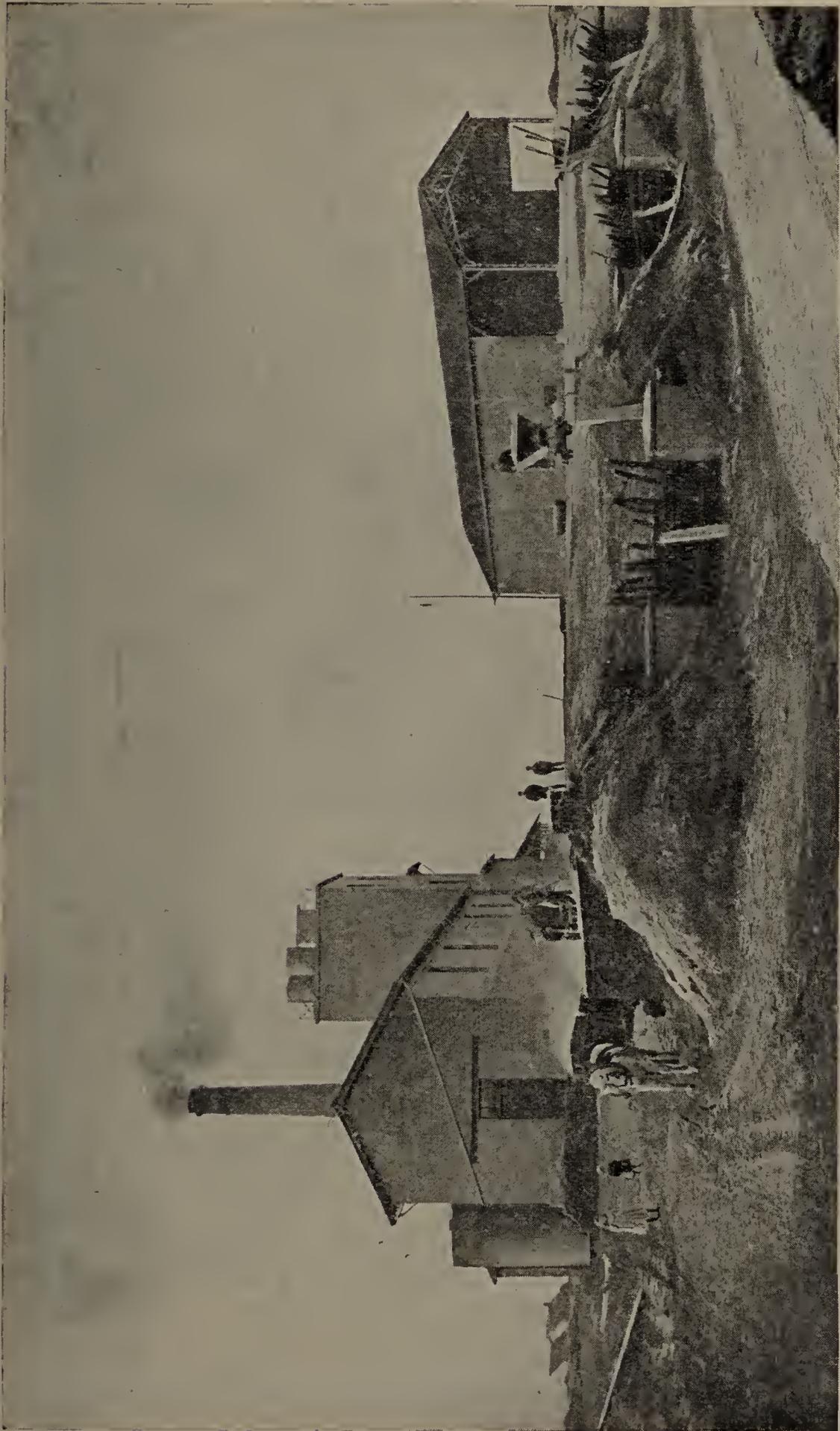


FIG. 15.—Exterior view of beet distillery (Figs. 10, 14), showing beet silo, and silos for spent beet slices, with tilting truck full of spent beet slices ready for discharge, and cart loading pulp for neighbouring farms for cattle feeding EGRROT and GRANGÉ).

measuring tanks by two large tuns to dilute the molasses to 1.080 or more, and the operations would follow in the same order, except that the steriliser would actually be brought to the boil so as to

denitrate the molasses. But denitration is not fully accomplished in dilute washes, because the acidity is not strong enough. The process is therefore modified as follows. At A are two dilution vats for diluting to  $28^{\circ}$ - $30^{\circ}$  Bé, by addition of a little water and the whole of the sulphuric acid required for fermentation; *b* is

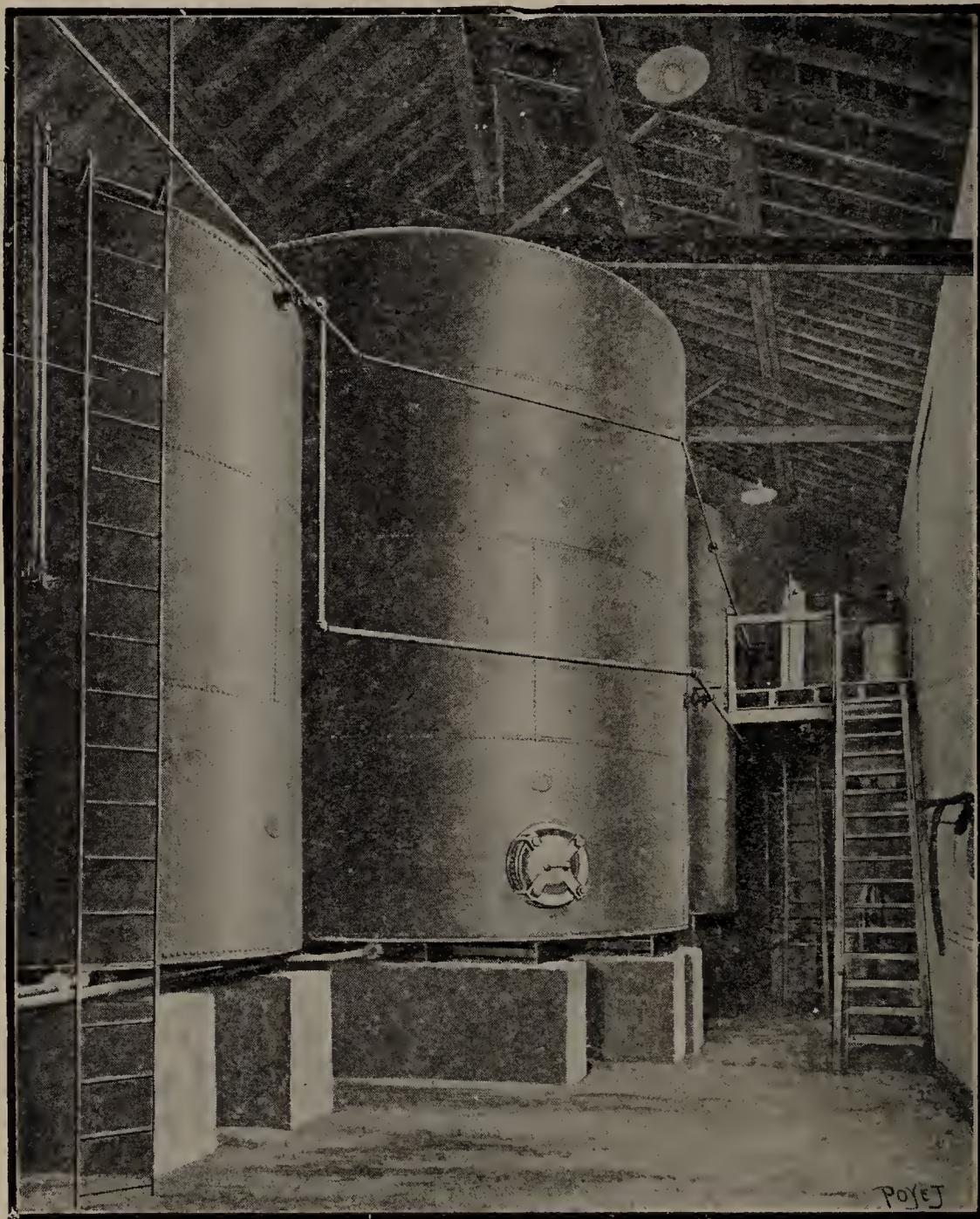


FIG. 16.—Tanks for storing alcohol (EGROT and GRANGÉ). (Sec R, S, Fig. 10, sec. 13.)

a regulating feed tank. B is the recuperator (fore-warmer). The molasses heated to about  $80^{\circ}$  C. by the fore-warmer enters the continuous denitrator C, where it is boiled fifteen to twenty minutes before issuing continuously through the bottom of the apparatus. From there instead of going to the recuperator, it

returns to a copper mixer D, closed by a cast-iron lid and fitted with a mechanical agitator. To dilute the boiling molasses, the hot water from the condensers, and even a certain proportion of boiling lees, is used, these liquids being regulated respectively by the taps *n* and *m*, and are mixed with the molasses in a kind of mixer before entering the dilution vessel. The dilution temperature is about 80° C. It may be increased a little by injecting steam. But, in fact, owing to the presence of acid, the tempera-

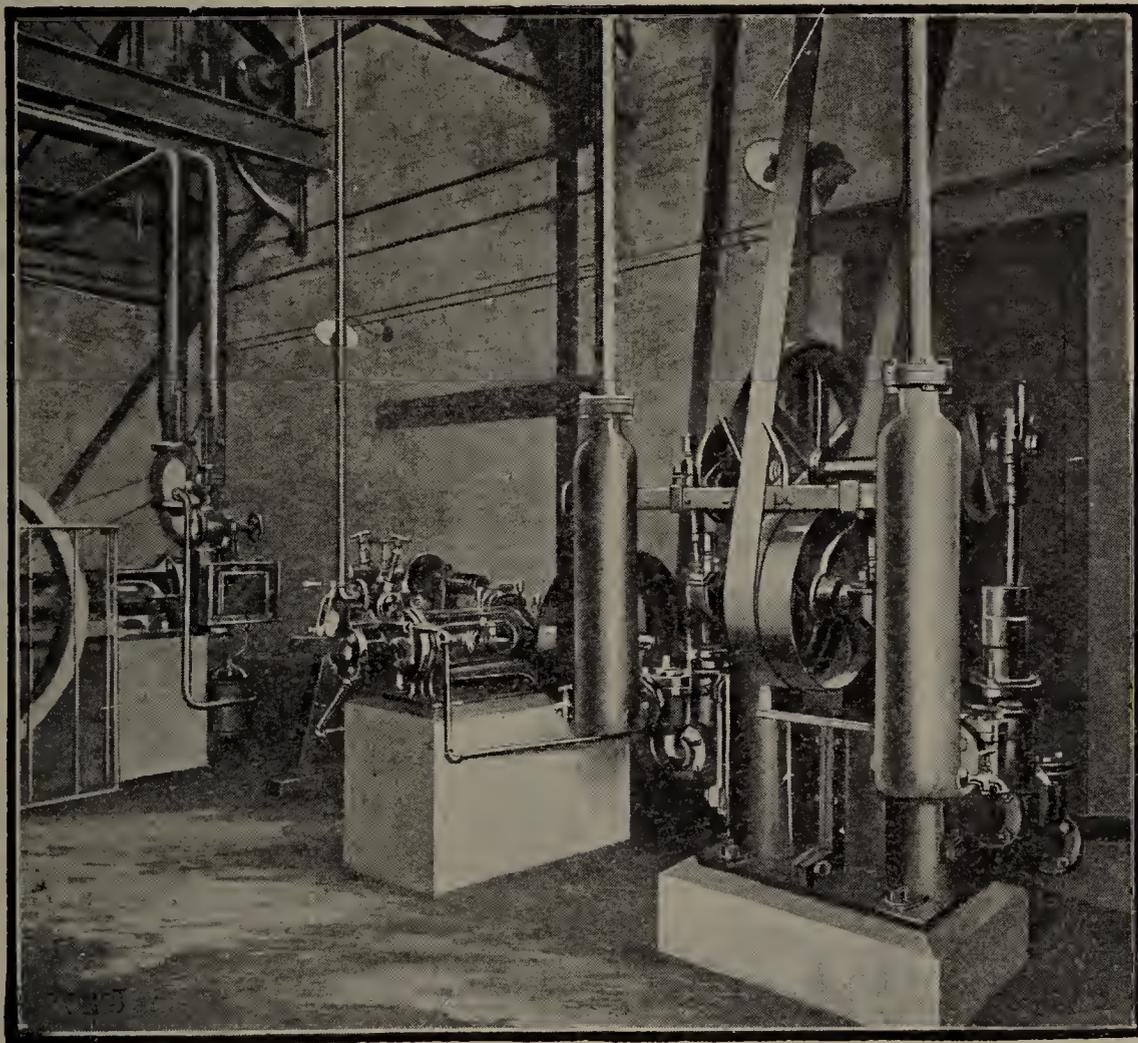


FIG. 17.—Pumps and motor power arrangements (EGROT and GRANGÉ). (See C, D, Fig. 10.)

ture of effective sterilisation is not very high, and 80° effects a practically efficient purification for the short duration of industrial fermentation. At the exit is a test glass E, where the temperature and density is permanently indicated, 1.060 at 80° = 1.082 at 21° C., the temperature at which it is sent to the fermenting tuns. At the exit from this test-glass the diluted wash returns to the recuperator B to heat the molasses to be denitrated. From thence it passes to the refrigerator V and to the fermenting

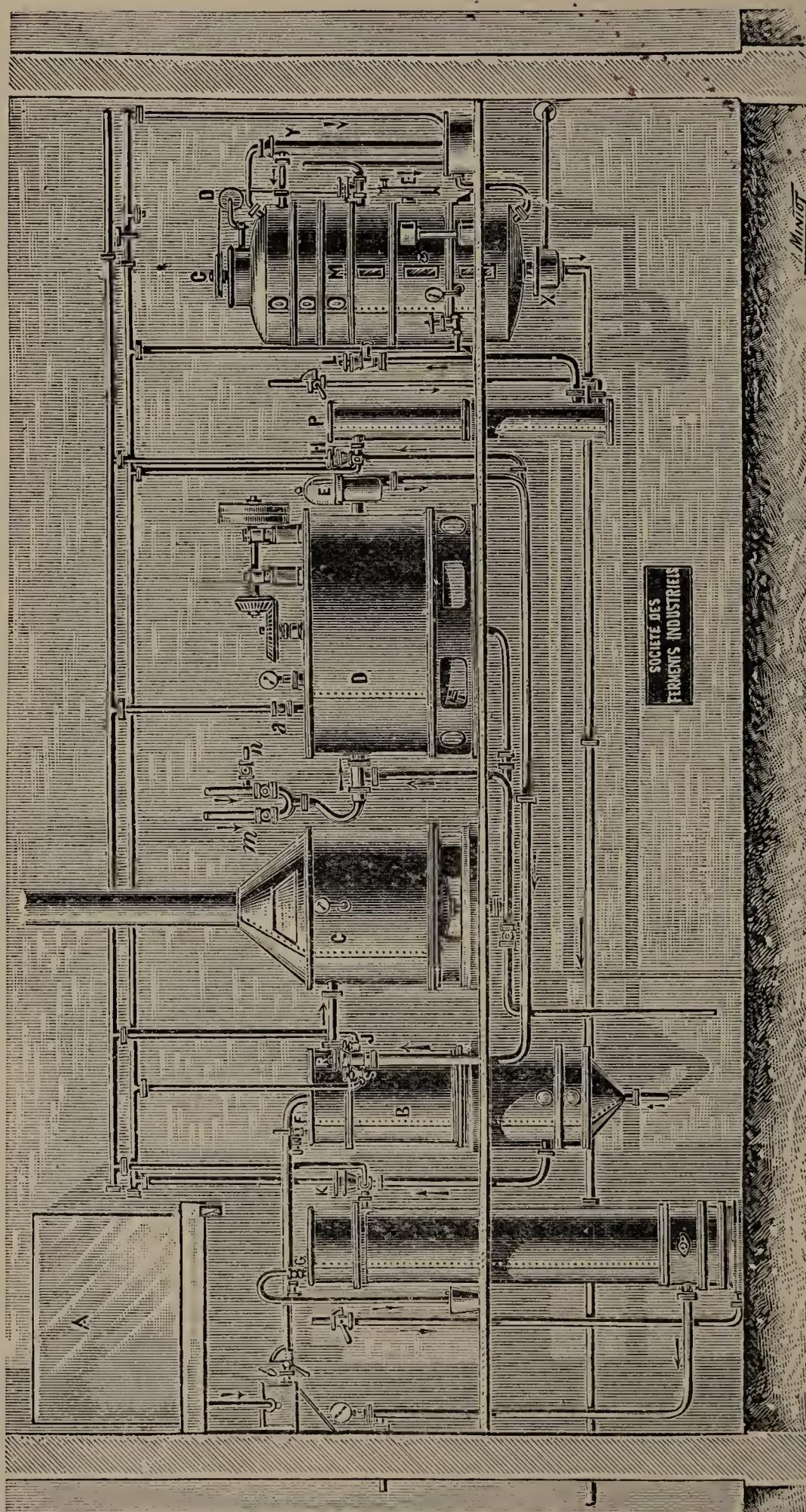


FIG. 18.—Plant for aseptic fermentation of beet-sugar molasses (E. Barbet). A, tanks to dilute to  $28^{\circ}$ - $30^{\circ}$ B.; B, fore-warmer; C, continuous denitrator and steriliser; D, mixer to dilute with hot water or hot spent wash; m, spent wash tap; n, hot water tap; E, test glass; H, regulating valve; M, pure yeast producer (Fig. 2); P, cooler for yeast apparatus; V, refrigerator to left of B.

tuns. For the yeasts M, direct draw-offs of wash are made on the mixer D, which for the occasion are re-heated to  $97^{\circ}$ - $98^{\circ}$ , so as to have a more certain sterilisation. Regulated by the valve H, this wash is cooled in the special refrigerator P, and enters through L into the yeast apparatus, which works as already described. A little syrup of maize saccharified by acid and filtered may be run into D, so as to furnish elements more favourable than molasses alone; or one may rest content by adding maltopeptone. The use of very active yeasts enables the tuns to be charged at a very high density up to 1.100, which economises coal in the potash department. This great activity of fermentation enables the same end to be attained by the re-use of a certain proportion of lees for dilution. If, for example, one-fourth of the lees be made to re-enter, there are only three-fourths of the volume of lees to be evaporated, and once this routine is established these three-fourths contain the whole of the salts and organic matter which should be discharged each working day; the lees are also more concentrated and require less coal. Finally, pure yeasts diminish the expenditure in acid, enrich the potash salts in carbonate of potash, and yield purer spirits.

Instead of heating the distilling column by direct high pressure steam, a small triple, or even double, effect system working under pressure may be installed. The live steam boils the vinasse in No. 1 under a pressure of 3 kilos, the steam from No. 1 (E) heats No. 2 (D), which boils at 1 kilo, and, finally, this steam at 1 kilo heats the base of the column A either by a pipe, coil, or steam jacket. In the case of molasses the vinasse thus concentrated is auto-evaporable on the furnace, *i.e.*, the combustion of the organic matters on incineration suffice to complete the evaporation of the water without expense of fuel, except in the beginning, to light up the potash furnaces. Neither in the triple effect nor in the furnace is there any consumption of fuel. The salts are thus obtained gratuitously. With beets, Barbet's beet diffusion method, in which the spent wash is terminated by aqueous diffusion, enables the pulp to be pressed, and thus produces a cattle-food identical with that obtained in sugar factories. With grain and potato wash, *i.e.*,

with turbid washes, multiple effect evaporation deserves attention. Barbet has rendered tubular heating practicable even with

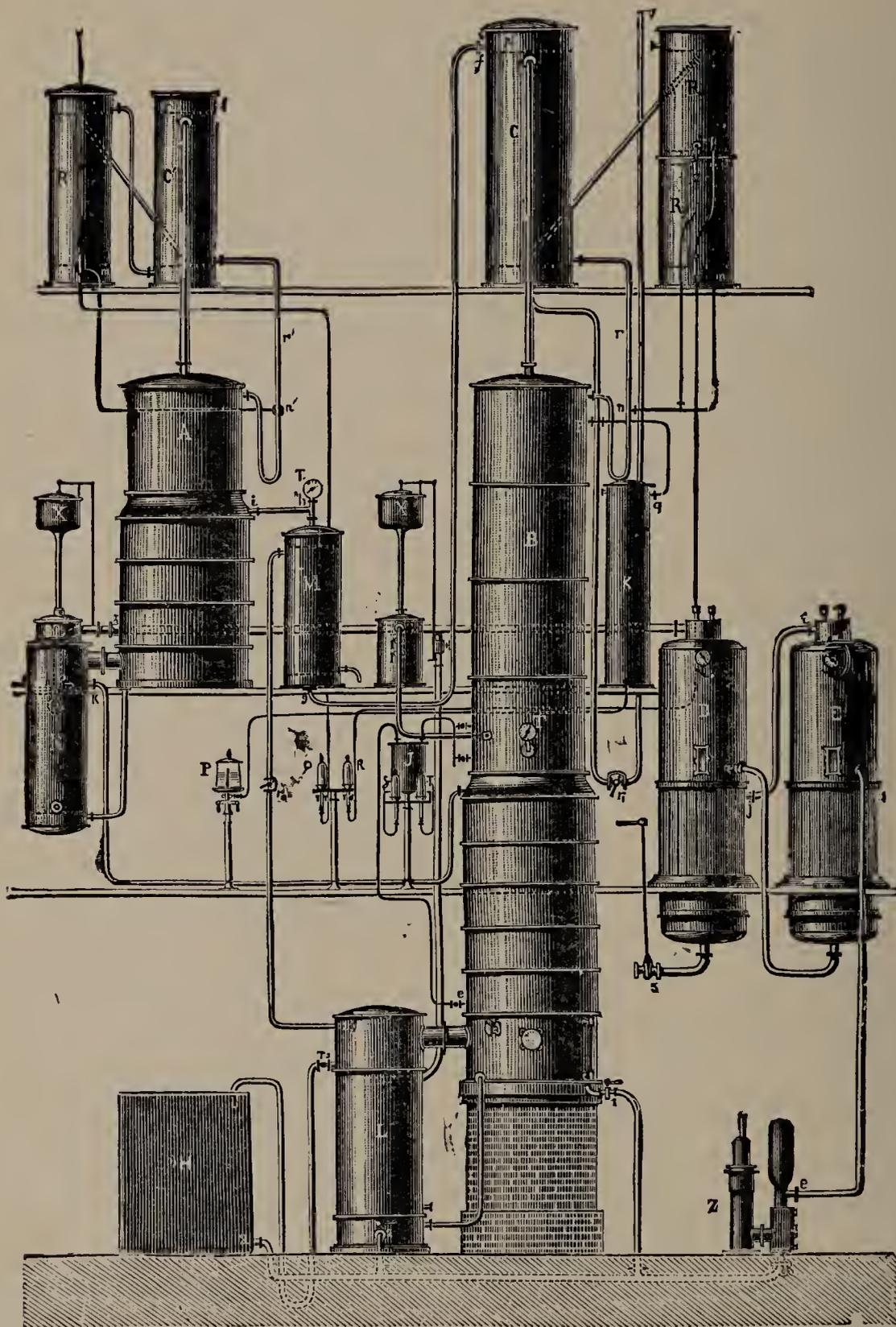


FIG. 19.—Direct distillation rectification plant (heated by steam from triple effect used to evaporate vinasse). A, preliminary purifier; B, rectifier; C C', condensers; D E E, multiple effect vinasse evaporators; K, pasteurised alcohol condenser; R R', refrigerators (E. Barbet).

thick washes. Consequently 100 litres of fermented wash, instead of yielding 105 to 110 litres of dregs, only yield about

85. They are afterwards concentrated by triple effect apparatus of special construction, in which the heating surfaces are constantly brushed. A residue is thus obtained of a consistency thick enough to be siloed under perfect conditions of preservation. This fodder, being less aqueous, is better for cattle, especially in the case of potatoes. Freight is reduced to one-third, and a stock of food is available for many months after the season is over.

*Beet distillation in Britain.*—Prior to 1870, there were some thirteen distilleries in Britain working experimentally on beet, and every one of them came to grief. The beets, no doubt, rotted before they could be distilled, owing to our arbitrary and despotic Excise restriction against brewing and distilling simultaneously, and against continuous fermentation. The excellent plant shown in Fig. 10 contravenes our Excise laws in every direction.

K. Windisch<sup>1</sup> states that the excessive “head” arising during the fermentation of beet infusion, and the “fobbing” which occurs during the distillation, can be obviated by an addition of green malt after the mash has been transferred to the cooling vat. This he ascribes to the malt enzymes dissolving the hemicelluloses contained in the cell walls of the beet, thus forming a more fluid mash. The fermentation can be carried on with a “lactic acid” or “sulphuric acid” yeast, and will be completed in about three days, the “head” being kept down by stirring. In vats of 1000 litres capacity the charge should be 600 to 700 kilos of beets, and the mash will attenuate to about 1.2°-1.3° Balling.

Raw beet sugar can be used in distilleries provided certain modifications in the methods of procedure are followed. K. Windisch,<sup>2</sup> in discussing this problem, points out that in order to obtain successful results the beet sugar should be dissolved in water at a temperature of about 75° C., to destroy bacteria and undesirable ferments. The strength of the mash should be about 16 per cent., but not higher than 17 per cent. of

<sup>1</sup> *Z. Spiritusind.*, 1914, 37, 580-581.

<sup>2</sup> *Z. Spiritusind.*, 1915, 38, 121, 129; *Jour. Soc. Chem. Ind.*, 1915, 1160.

extract at time of pitching. Acidification with 1 to 1.5 c.c. of concentrated sulphuric acid (free from arsenic) is recommended, and in order to give nitrogenous food for the yeast an addition of 20 to 60 grams of ammonium chloride should be added per 100 litres. The author, however, expresses some doubt as to whether yeast produced under these conditions can be used indefinitely. Fermentation should be commenced immediately the mash has been brought to a temperature of 30° C., and should not be allowed to fall below that temperature till the fermentation is practically completed.

## CHAPTER IV.

### THE MANUFACTURE OF INDUSTRIAL ALCOHOL FROM GRAIN.

THE cereals (corn, grain) are the fruits or seeds of certain plants, all of which almost exclusively belong to the graminaceous family. The stem or stalk, termed the straw, long and slender, bearing large sheathing leaves which fall on maturity, ends in an inflated part called the ear, consisting of the seeds and their envelopes. When the ears are ripe the stalks are cut down nearly level with the ground, an operation now almost invariably performed mechanically by reapers and self-binders, except in outlying districts. The grain is then separated from the straw by threshing and from the outer envelopes by winnowing.

*Storage of grain and its liability to damage during storage.*—Grain is capable of being damaged in many ways during storage, either from the attacks of insects or the development of fungi, or even from the germination of the seed itself. It is

TABLE XV.—SHOWING AVERAGE COMPOSITION OF THE GRAIN OF CEREALS (LAWES AND GILBERT).

	Old Wheat.	Barley.	Oats.	Rye.	Maize.	Rice.
Water . . . . .	11.1	12.0	14.2	14.3	11.5	10.8
Starch . . . . .	62.3	52.7	56.1	54.9	54.8	78.8
Fat . . . . .	1.2	2.6	4.6	2.0	4.7	0.1
Cellulose . . . . .	8.3	11.5	1.0	6.4	14.9	0.2
Dextrin and sugar	3.8	4.2	5.7	11.3	2.9	1.6
Albuminoids . . . . .	10.9	13.2	16.0	8.8	8.9	7.2
Ash . . . . .	1.6	2.8	2.2	1.8	1.6	0.9
Loss . . . . .	0.8	1.0	0.2	0.5	0.7	0.4
<b>Total . . . . .</b>	<b>100.0</b>	<b>100.0</b>	<b>100.0</b>	<b>100.0</b>	<b>100.0</b>	<b>100.0</b>

preserved by covering it with substances capable of killing all living germs, by depriving it of moisture by means of air or heat. The chief cereals are wheat, barley, oats, rye, maize, and rice.

*The Manufacture of alcohol from grain by saccharification within torrefied or kiln-dried malt.*—Malt is barley which has been caused to germinate to a certain extent, after which the process is inhibited by heat. During the germination of grain a small quantity of a white, insipid, nitrogenous substance termed diastase is developed. Diastase is a soluble ferment which possesses the property of causing starch, which is naturally insoluble, to become soluble, *i.e.*, by changing it into dextrin and maltose. The barley is steeped in cold water for about fifty hours at a constant temperature of 14°-15° C., and is then made into a heap, or couch, upon the malting floor—a floor of slate or cement—until it germinates. Here it absorbs oxygen, and evolves carbonic acid; the temperature of the heap rises, and then it is occasionally turned to prevent it becoming too warm. In this process the radicle lengthens, and the plumule, called by the malsters acrospire, elongates. When it has nearly reached the opposite extremity of the seed, its further growth is arrested by drying at a temperature slowly rising to 150° F. or more. This slight torrefaction is effected in a kiln—a large stove traversed by a current of hot air, the construction of which need not be entered into here. If dark-coloured malt be desired, the heat may be raised to 164° F. But Payen and Persoz state that at 75° C. (167° F.) diastase loses its property of rendering starch soluble. Therefore, when malt is intended for the brewer or distiller, care should be taken that the heat is not allowed to rise too high, otherwise its saccharifying power will be diminished, as shown on page 85.

After appropriate drying in the kiln the malt is then cleansed of the rootlets by screening it through wire sieves, so that the sprouted radicles called combs or chives are broken off and separated. According to Dr. Thomson, barley loses about 8 per cent. by converting it into malt, which is distributed as follows :—

Carried off by the steep-water.	1·5
Dissipated on the floor.	3·0
Roots separated by cleansing.	3·0
Waste.	0·5
	—
	8·0

TABLE XVI.—SHOWING INFLUENCE OF HIGH KILN HEAT ON INFUSION PRODUCTS OF MALT.

	80° C.	100° C.	120° C.
Maltose . . . . .	37·01	52·44	51·32
Dextrin . . . . .	14·92	18·49	19·35
Lactic acid . . . . .	6·56	0·49	0·31
Soluble albuminoids . . . . .	2·09	1·60	1·50
Colouring matters, ash, etc. . . . .	1·49	1·38	1·32
Total dry solids . . . . .	76·07	74·40	73·80

The system of pneumatic malting more especially in vogue on the Continent is for the moment beyond the limits of this treatise; it would be dealt with more appropriately in a treatise on potable alcohol. According to Cameron, from 3 to 6 per cent. of the weight of the grain is lost in the process of malting, exclusive of the amount eliminated in the form of dust, or combings, which is about 3 to 4 per cent. According to Proust, barley also contains a peculiar substance, insoluble in hot water, which he called hordein, and which, during malting, is diminished in quantity, being converted into sugar or starch. Hordein appears to have been confounded with starch. The starch of malt also differs in some of its properties from that of barley. Germination thus partly converts the starch of the grains into a kind of sugar, which is capable of vinous fermentation, by which process alcohol is formed. In brewing, the malt is steeped until the sugar is dissolved out, forming the "sweet wort."

The chief change which takes place during malting is the conversion of about one-eighth part of the starch of the grain into maltose; probably nearly as much starch is converted into

soluble compounds—dextrin, etc. But the real rationale of the process of malting is to develop the diastase necessary for the saccharification not only of the malt itself, but of any raw grain, potatoes, etc., which may be used in conjunction therewith under the somewhat anomalous term of malt adjuncts. The malt is in reality the saccharification adjunct, the so-called adjunct being the main product to be saccharified. A large amount of diastase formed during germination remains until the last stage of the process. It is this excess of diastase which enables the distiller to ferment starch, or unmalted corn, by mixing them with from 10 to 50 per cent. of malted grain.

Diastase (from *διίστημι*, "I separate") was first obtained from barley malt by Payen and Persoz. It may be procured from brewers' malt, but in greater quantity from germinated barley carefully prepared for the purpose, in which the germ has been allowed to attain about the length of the seed. The malt is pulverised and macerated in, or triturated for a few minutes with water, at the temperature of 70° or 80° F.; the pasty mixture is then strongly pressed, and the turbid liquor which runs from it filtered. The filtrate is then heated in a water bath to about 170° F., at which temperature the greater part of the foreign matter coagulates and may be separated by filtration. The clear filtered liquor retains the diastase and may be used for many purposes as a solution of that substance; it, however, also retains other principles, from which it may be to a great extent separated by the addition of anhydrous alcohol, which forms a flocculent precipitate of diastase insoluble in that liquid; it may be collected and carefully dried at a low temperature, for when heated in a moist state above 190° F. its properties are materially affected. It may be further purified by a second solution in water and precipitation by alcohol, and if the solutions are brown, animal charcoal may be resorted to as a means of decolorising them. Diastase may also be obtained without the aid of heat, but the process requires caution: it consists in triturating the finely-ground malt as before with a little water, pressing out the liquor, and carefully adding a little alcohol to it so as to coagulate its albuminous contents without precipitating the diastase; it

is then filtered, and the diastase is separated by the further addition of strong alcohol: it may be purified by a second aqueous solution and alcoholic precipitation, and should be dried at a temperature not exceeding 100° F., or *in vacuo*. It is white, soluble in water and in dilute alcohol, but insoluble in strong alcohol; its aqueous solution is tasteless, and soon becomes sour and decomposes; its effect upon starch is entirely destroyed by boiling; it contains nitrogen, but its ultimate composition has not been accurately determined.

The manufacture of alcohol from grain, etc., may be naturally divided into two stages, brewing and distilling. The raw materials are wheat, rye, barley, oats, maize, rice, etc. The yield in spirits which these grains afford depends on their starch content. Mr. Young of the Inland Revenue gave the following average figures for different raw materials:—

	Gallons Proof Spirits.
1 quarter of barley malt yields . . . . .	18
1 „ „ malt grain „ . . . . .	20
1 cwt. „ sugar . . . . .	10
1 „ „ molasses „ . . . . .	7½
1 ton „ beetroot „ . . . . .	15

Ure gives the following yields:—

	Alcohol of Specific Gravity 0·9427 9/11 of British Proof Spirits in lb.
100 lb. wheat . . . . .	40-45
100 „ rye . . . . .	36-42
100 „ barley . . . . .	40
100 „ oats . . . . .	36
100 „ buckwheat . . . . .	40
100 „ maize . . . . .	40

the mean of the whole being about 40 lb., *i.e.*, 4¼ gallons of density 0·9427, or 3·47 gallons at Excise proof. Pooley states that by actual practice he found the average produce from undried foreign corn used in the following proportion—malt, 12; oats, 16; barley, 112—to be 1 gallon of spirit from 20¼ lb. of the mixed

grist, or very nearly 5 proof gallons per 100 lb. of grist. Again, he quotes the case of an Irish distillery where only home-grown barley and oats highly kiln-dried and one-fifth malt are used, 1 gallon of proof spirit is produced from 18 lb. of the mixed grist ; and he asserts that sometimes in favourable seasons, working with high-class corn, even less than  $17\frac{1}{4}$  lb. mixed grist will produce 1 gallon of proof spirit. Ure gives the following proportions as those used by some experienced Scotch distillers, 250 bolls, containing 6 bushels each, being used for a mash consisting of :

25 bolls oats weighing	284 lb. per boll, or	$47\frac{1}{3}$ lb. per bushel.
42 „ malt	„ 240 „ „	40 „ „
25 „ rye	„ 320 „ „	$53\frac{1}{3}$ „ „
158 „ barley	„ 320 „ „	$53\frac{1}{3}$ „ „
—		—
250		$48\frac{1}{2}$
—		—

From each boil weighing 291 lb., 14 imperial gallons of proof spirit are obtained on an average, equivalent to 11.2 gallons at 25 overproof, which is about 4.8 proof gallons per cent. of mixed grist grain, which thus agrees fairly well in the main with Pooley's figures. Ure says 100 lb. of starch from Hermstedt's experiments should yield 7.8 gallons of proof spirit. The Scotch and Irish distillers use the following mixtures :

	Scotch.	Irish.
Malt . . .	2	2
Oats . . .	1	1
Rye . . .	1	0
Barley . . .	7	7

Moreover, in a London distillery using about 1000 quarters of mixed grain per week, the production from that quantity is between 19,000 and 20,000 gallons of proof spirit per week. At 50 lb. per bushel, a yield of 20,000 gallons per 1000 qrs. of grain exactly corresponds to 5 proof gallons per 100 lb. of grain.

Barley, therefore, is the predominant grain used in distilleries for making potable spirit in Britain. But maize and molasses

are the chief raw material employed for alcohol for methylation. In Ireland, large quantities of home-grown barley and oats highly kiln-dried are mashed. Barley is generally used either wholly or partially in the malted state, whilst other grain is not malted, but is merely mixed with a certain amount of barley malt to induce the conversion of the starch in it into maltose and dextrine. The main reason for using the mixture of different kinds of grain as indicated above is because it is preferable to use a mixture of several kinds of grain instead of using all of one kind, because, for example, with wheat, with barley and oats, or barley with rye and wheat, the husks of the oats diffused through the wheat flour and rye meal keep it open and porous when mashed, and thus favour the extraction of the wort. But when the whole of the grain used is malted grain, a much more limpid wort is obtained than that produced from a mixture of malt with raw grain; hence pure malt is preferable for the ale and porter brewer, whilst the mixture affords a larger yield of product at the same cost of materials to the distiller. When barley is the only other grain used besides malt, from one-third to one-sixth of malt is usually mixed with it; but when wheat and rye are also taken, the addition of from one-eighth to one-sixteenth of barley malt is sufficient. Oats are very useful for mixing with wheat to keep the meal open in the mashing.

*Mashing.*—Raw grain and unmalted barley are ground to meal by millstones, but malt is simply crushed between rollers (Fig. 20). To facilitate drainage of the mass some oat husks are added, *i.e.*, when as much as 87.5-90 per cent. of barley is taken for 10-12.5 per cent. of malt. But when mashing is done in the proportion of 40 bushels of barley to 20 of malt, from 600 to 700 gallons of water heated to 150° F. are mixed with each 60 bushels in the mash tun, and carefully incorporated by the agitation produced by a mechanical agitator with blades (Figs. 21 and 22). The mixer is kept at work for two or three hours, with the gradual admission of about 400 additional gallons of water at a temperature of 190° F. to counteract the cooling of the materials, unless the operation be performed in a steam-jacketed vessel, by which the temperature can be regulated at about 160° F. If the wort be

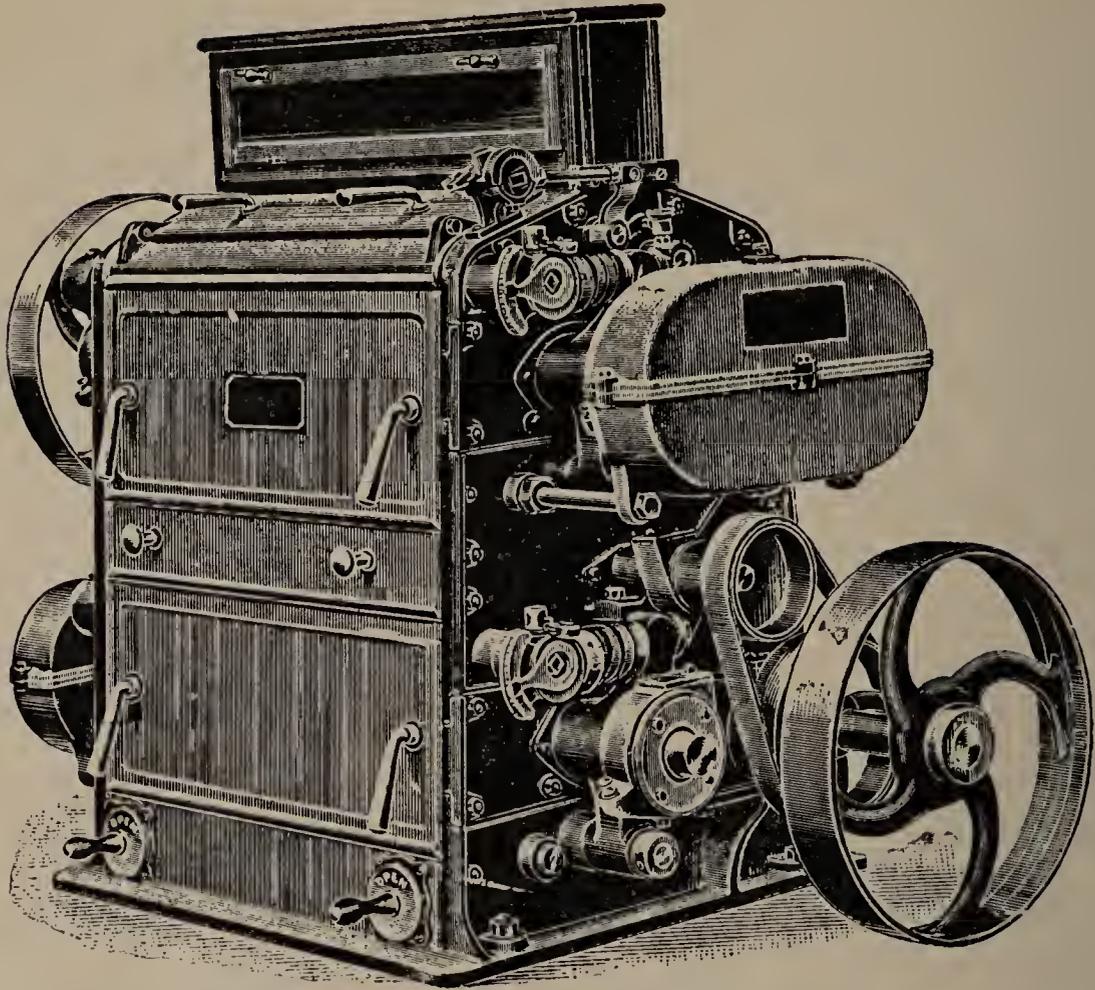


FIG 20.—Patent two-high malt mills (BLAIR, CAMPBELL & McLEAN, Ltd).

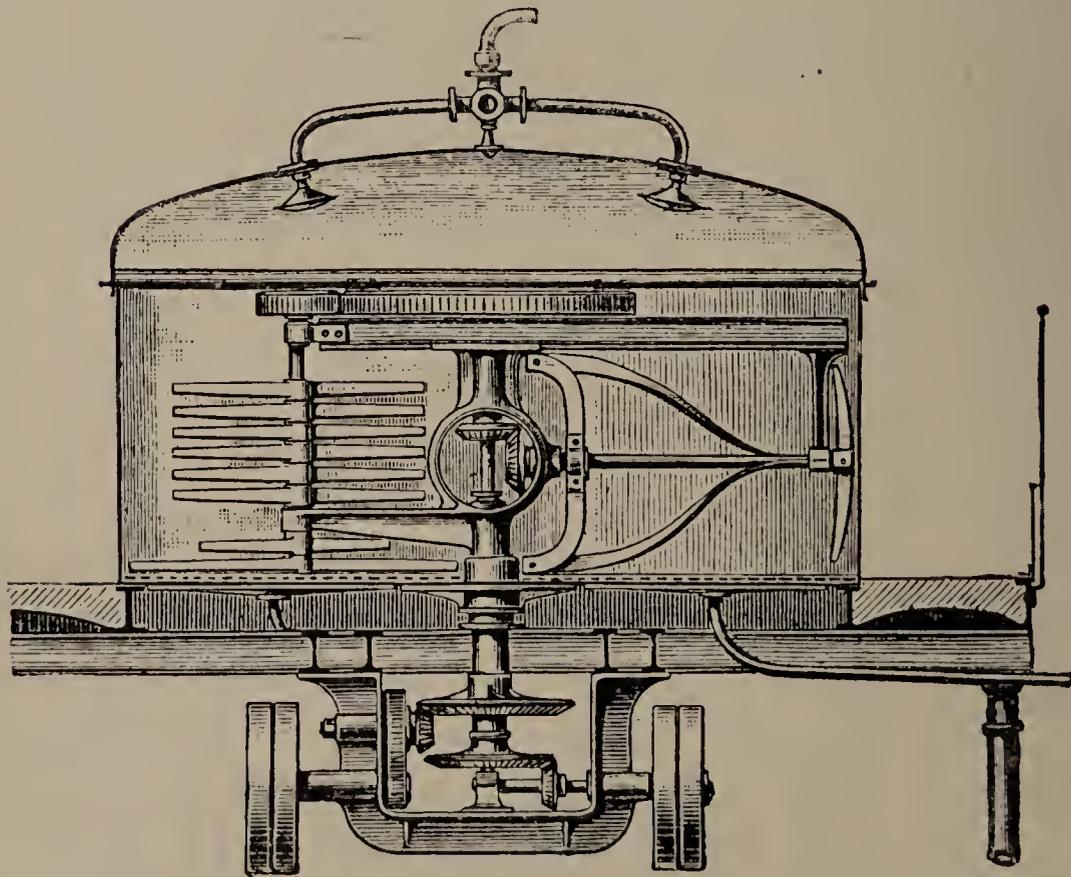


FIG. 21.—Mash tun.

tested every half-hour during mashing, it will be found to become gradually sweeter and more fluid, but of greater density. The wort is drawn off from the grain whenever it has reached its maximum density, which seldom exceeds 150 lb. per barrel, *i.e.*,

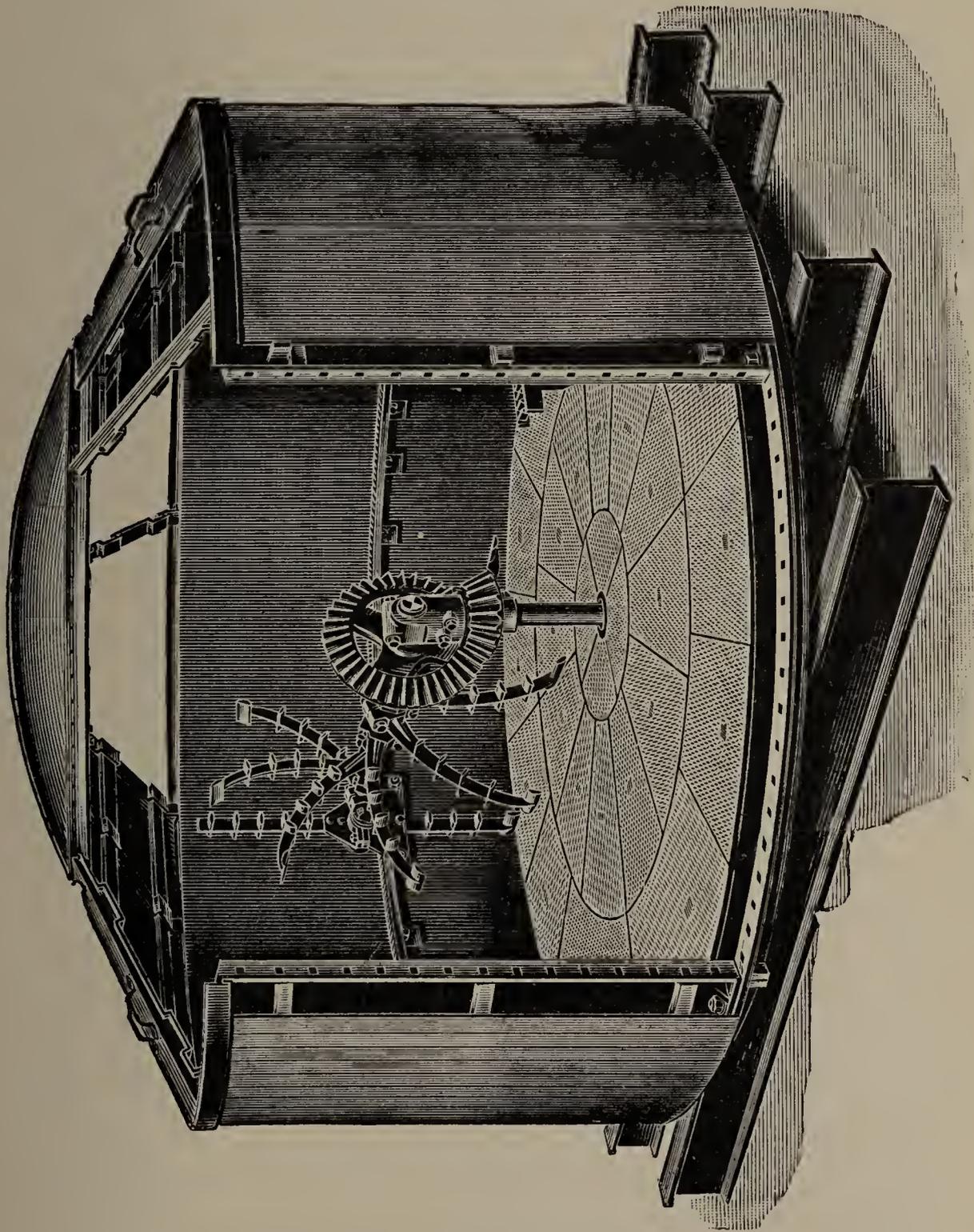


FIG. 22.—Mash tun (BLAIR, CAMPBELL & McLEAN, Ltd).

$\frac{360 + 150}{360} = 1.42$ , or 42 per cent. The distiller's corn not being so porous as the brewer's, the wort cannot be drawn off from the bottom of the tun, but through a series of holes at the level of the liquor bored in a pipe fixed in the corner of the vessel. About

one-third only of the infusion water can thus be drawn off from the pasty mass. Fresh water is then run in at the temperature of 190° F., well mixed by agitation for half an hour, then quietly infused for an hour and a half, and then drawn off as before. Fully 400 gallons of water are used upon this occasion, and nearly as much liquor may be drawn off. Lastly, to extract from the grains the whole of the soluble ingredients, about 700 gallons of boiling water are run in, mixed, left to infuse, and drawn off as before. This weak wort is commonly reserved for the first liquor of the next mashing operation along with a fresh quantity of meal and malt. With the above proportions of malt, raw grain, and water, the first infusion may have a strength of 20 per cent. = sp. gr. 1.082, or 73 lb. per barrel, the second of 50 lb. per barrel or 14 per cent., the two together having a strength of 61.2 lb. per barrel = 17 per cent., sp. gr. 1.070. But direct experiments on a large scale show that no more than four-fifths of the soluble starchy matter of the worts is brought into solution in the best regulated fermentation of the distiller from raw grain. For every 2 lb. so decomposed, 1 lb. of alcohol, sp. gr. 0.825, is generated; and as every gallon of spirits of sp. gr. 0.909 contains 4.6 lb. of such alcohol, it will take twice 4.6 lb. of saccharine matter to produce the said gallon. To these 9.2 lb. actually converted into alcohol, one-fifth, *i.e.*, 1.84 lb., must be added, which will raise it to 11.04 lb. the amount of solid matter required for producing a gallon of above spirits.

*English infusion process—Factors of hydration*<sup>1</sup> (GRAHAM).—The malt was mixed with cold water, and the temperature raised in 30 minutes to the various heats given, and the infusion process then conducted for two hours at the respective heats.

The malts and barleys in Series I., II., III., and IV. were different, though the same for any given series. It will be seen that no advantage is obtained by boiling barley previous to mashing, owing to the activity of the albuminoids of barley; with rice boiling is essential.

*Manufacture of alcohol from grain by saccharification with green malt.*—Formerly the distillation of fermented grain wash

<sup>1</sup> For influence of kiln-drying temperature, see Table XVI., p. 85.

TABLE XVII.—INFLUENCE OF TIME.

(Malt, 100. Water, 1000. Temperature, 145° F.)

No. of Malt.	Dura- tion of Mash. Hours.	Maltose.	Dextrine.	Total Sugars.	No. of Malt.	Dura- tion of Mash. Hours.	Maltose.	Dextrine.	Total Sugars.
1	$\frac{1}{2}$	48.60	14.61	63.21	2	3	59.52	8.71	68.23
„	1	52.35	12.26	64.61	„	5	61.47	7.91	69.38
„	2	53.56	11.39	64.95	3	$\frac{1}{2}$	47.46	13.89	61.35
„	3	54.60	11.05	64.65	„	1	48.69	14.27	62.96
„	7	61.47	3.50	65.00	„	2	52.81	12.08	64.89
2	$\frac{1}{2}$	49.99	14.98	64.97	„	4	54.34	10.67	65.01
„	1	53.56	13.43	66.99	„	6	57.24	8.67	65.91
„	2	57.69	10.76	68.45					

TABLE XVIII.—INFLUENCE OF HEAT.

(Ratio of Malt to Water 1 to 10.)

No. of Malt	°F.	Maltose.	Dextrine.	Total Sugars.	No. of Malt.	°F.	Maltose.	Dextrine.	Total Sugars.
1	150	47.46	10.70	58.16	2	160	41.65	17.42	59.07
„	160	43.50	13.42	56.92	„	170	30.24	25.09	55.33
„	170	32.17	17.61	49.78	3	140	51.36	10.60	61.96
2	140	52.81	12.08	64.89	„	150	45.30	14.35	57.65
„	150	48.61	13.83	62.44	„	160	39.90	18.06	59.96

TABLE XIX.—INFLUENCE OF QUANTITY OF WATER.

(Malt, 100. Temperature, 140° F. Time, 2 hours.)

No. of Malt.	Water.	Maltose.	Dextrine.	Total Sugars.
1	1000	53.56	11.39	64.95
„	500	49.99	12.92	62.91
„	200	49.00	13.88	62.88
„	100	46.80	15.08	61.88
2	1000	52.81	12.08	64.89
	500	53.56	9.82	63.58
	100 <sup>1</sup>	35.70	16.18	57.88

<sup>1</sup> In this experiment a portion of the water was allowed to evaporate, and therefore the real quantity of water was less than 100, hence the great fall in the amount of maltose.

TABLE XX.—INFLUENCE OF NATURE AND RATIO SUBSTITUTE OF MALT.  
 TOTAL GRAIN, 100. WATER, 1000. TIME, 2 HOURS. TEMPERATURE,  
 145° F.

	Maltose.	Dextrine.	Total Sugars.		Maltose.	Dextrine.	Total Sugars.
I.	A 33.33	11.42	44.75	III.	N 57.69	5.65	63.34
	B 37.87	15.96	53.83		O 53.56	10.87	64.43
	C 41.65	16.96	58.61		P 49.99	15.27	65.26
	D 44.64	15.90	60.54		Q 53.56	11.89	65.45
	E 58.72	7.12	65.84		R 49.99	15.97	65.96
	F 60.00	10.97	70.97		S 46.87	19.99	66.86
	G 62.49	11.26	73.75		T 50.87	14.43	65.30
	H 57.69	1.73	59.42		U 48.10	17.05	65.15
	I 47.62	17.38	65.00		V 45.11	19.88	64.99
II.	J 46.15	18.48	64.63	IV.	W 49.99	18.94	68.93
	K 42.50	22.11	64.61		X 46.87	19.98	66.85
	L 41.34	22.48	63.82		Y 44.11	22.07	66.18
	M 54.15	22.46	76.61				

A, barley, 100. B, barley, 90; malt, 10. C, barley, 80; malt, 20. D, barley, 50; malt, 50. E, rice, 25; malt, 75. F, rice, 50; malt, 50. G, rice, 75; malt, 25. H, malt, 100 (rice = 72 per cent. starch). I, raw barley, 50; malt, 50. J, boiled barley, 50; malt, 50. K, high dried barley, 50; malt, 50. L, high dried barley boiled, 50; malt, 50. M, rice boiled, 50; malt, 50. N, raw barley, 25; malt, 75. O, raw barley, 50; malt, 50. P, raw barley, 75; malt, 25. Q, boiled barley, 25; malt, 75. R, boiled barley, 50; malt, 50. S, boiled barley, 75; malt, 25. T, high dried barley, 75; malt, 25. U, high dried barley, 50; malt, 50. V, high dried barley, 75; malt, 25. W, high dried barley boiled, 25; malt, 75. X, high dried barley boiled, 50; malt, 50. Y, high dried barley boiled.

entailed the grinding of the grain employed, the cooking of the resultant meal, and the saccharification thereof in macerators by means of kiln-dried malt also ground to meal. In this style of working a whole series of mills and kilns and a much larger proportion of malt than is now used were employed.

It has been found that the saccharification capacity of green malt is exactly the same as kiln-dried malt, thus showing an advantage of 40 per cent. in favour of green malt, and advantage has been taken of this fact. It was in 1885 that Warein and Defrance of Lille introduced into France the new process which up to then had been confined to Germany, and by the improve-

ments they brought to bear upon it caused it to be adopted by a large number of distillers. The plant consists of (1) a wort refrigerator ; (2) a mash tun ; (3) a fermenting tun. A complete working distillery of grain and potatoes operated by steam was exhibited by the above firm on the occasion of Parmentier's centenary at Montdidier. By new improvements brought to bear in the arrangement of the plant, in the method of working and in the use of sulphurous acid, the constructors claim that the yield obtained may be increased to 36 litres of alcohol per 100 kilos of maize,<sup>1</sup> and 10 to 12 litres of alcohol at 100 per cent. per 100 kilos of potatoes,<sup>2</sup> a result which leaves previous ones far behind. The residues resulting from this method of working form a cattle food highly conducive to fattening, and are sold in farming districts under the form of liquid or drained distillery dregs. Filtration tanks which evacuate naturally the liquid portion of the dregs dispenses with mechanical filtration. A hectolitre of dregs run into the filtration tanks yields 18 to 20 kilos of solid dregs ; 18 kilos of these dregs contain :—

Nitrogenous matter	. . . . .	1.536
Fat	. . . . .	0.630
Glucose	. . . . .	1.105
Non-nitrogenous matter	. . . . .	2.340
Mineral matter	. . . . .	0.308
Water	. . . . .	0.860

One kilo corresponds to 1 kilo of meadow hay. To produce 5 tons of dry dregs in twenty-four hours, four filtering tanks are required. These dregs may also be converted by hydraulic presses into dry edible cakes in the proportion of 25 per cent. of its weight, and will yield maize oil in the proportion of 2 per cent.

*The manufacture of alcohol by the acid saccharification of grain.*— This method of working has been adopted by many continental distillers, some distillers working up beets in winter, by making certain additions to their existing plant for producing alcohol

<sup>1</sup> 3.6 bulk gallons of 100 per cent. alcohol per 100 lb., or 6.3 proof gallons.

<sup>2</sup> 1.2 bulk gallons of 100 per cent. alcohol, or 2.1 proof gallons per 100 lb .

from beets, have been enabled to produce grain alcohol in the summer. It is claimed that by this method the yield of alcohol is increased from 2.7 gallons to 3.2 gallons per 100 lb., or close upon 20 per cent. more.

A *cuisseur saccharificateur*, or boiler saccharifier, is shown in

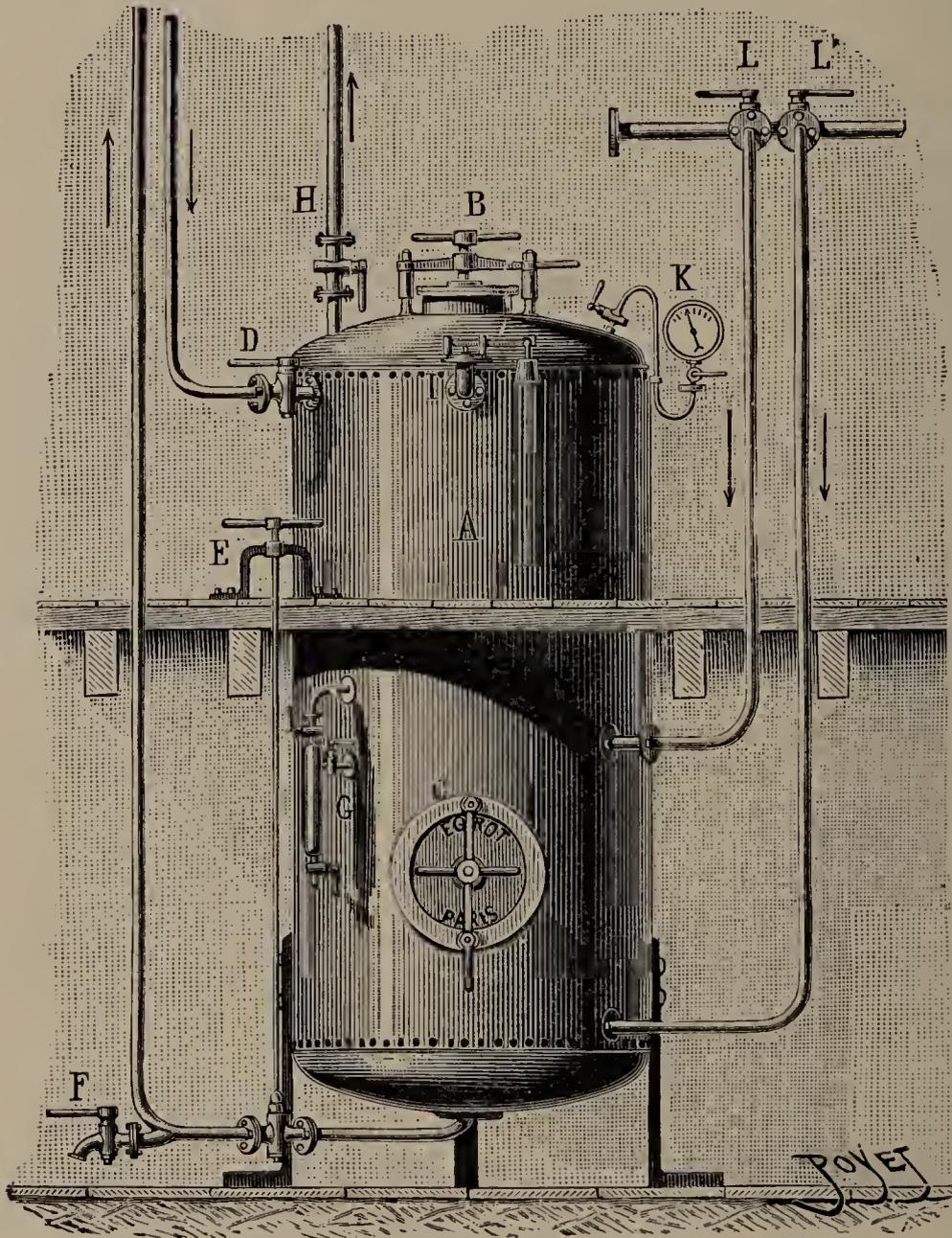


FIG. 23.—Digester for saccharification of grain, etc., with acid under pressure (EGROT and GRANGÉ). A, Digester; B, C, manholes; D, feed pipe; E, rod for manipulating bottom discharge valve; F, sampling pipe; H, discharge pipe; K, pressure gauge; LL, steam pipes.

Fig. 24, in which by appropriate treatment the starch of grain or corn may be transformed into glucose. The entire grain is submitted in presence of water to heat and a pressure of three atmospheres; then, when the grains are completely transformed into paste, acid is forced into the boiler from the globular vessel

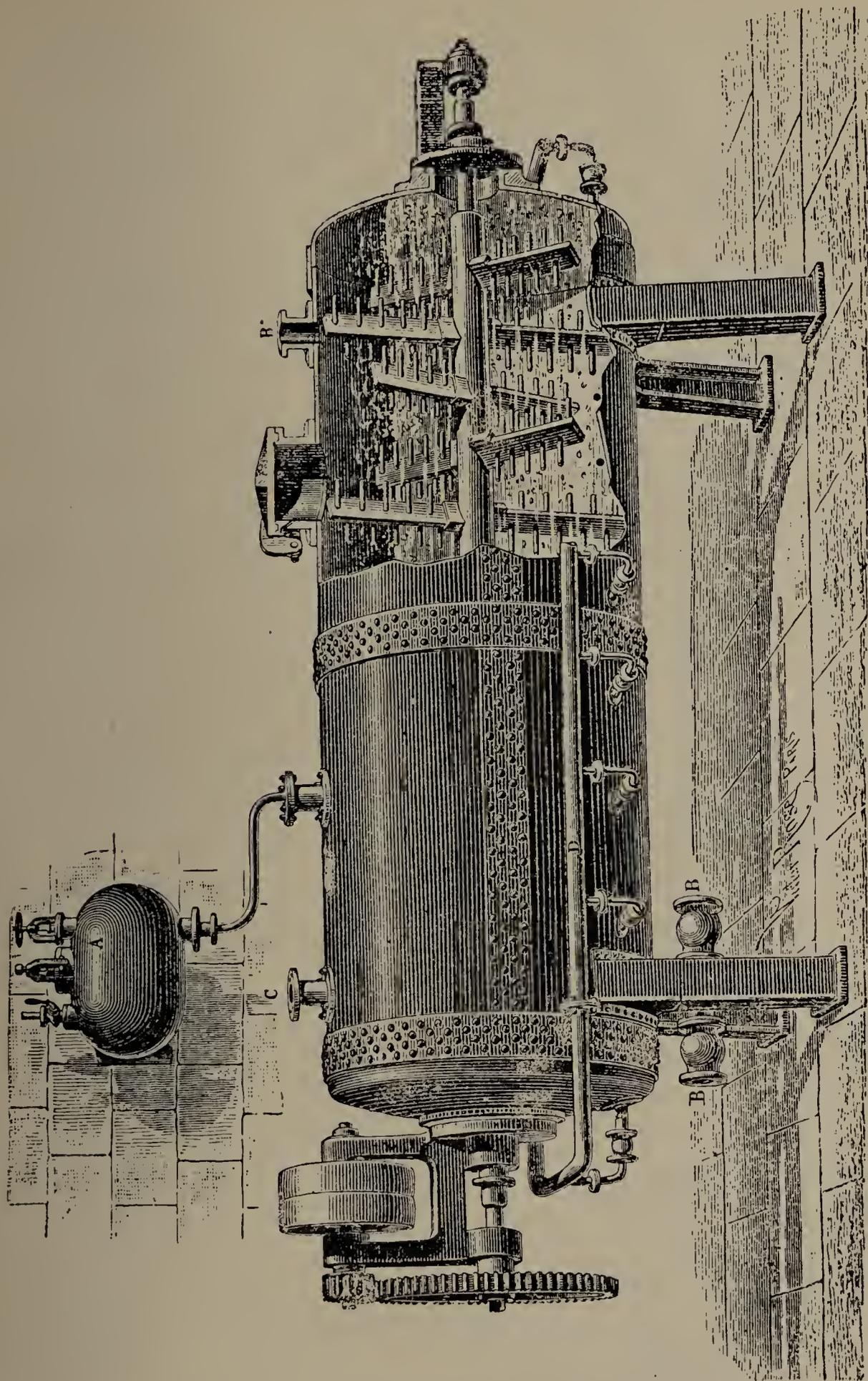
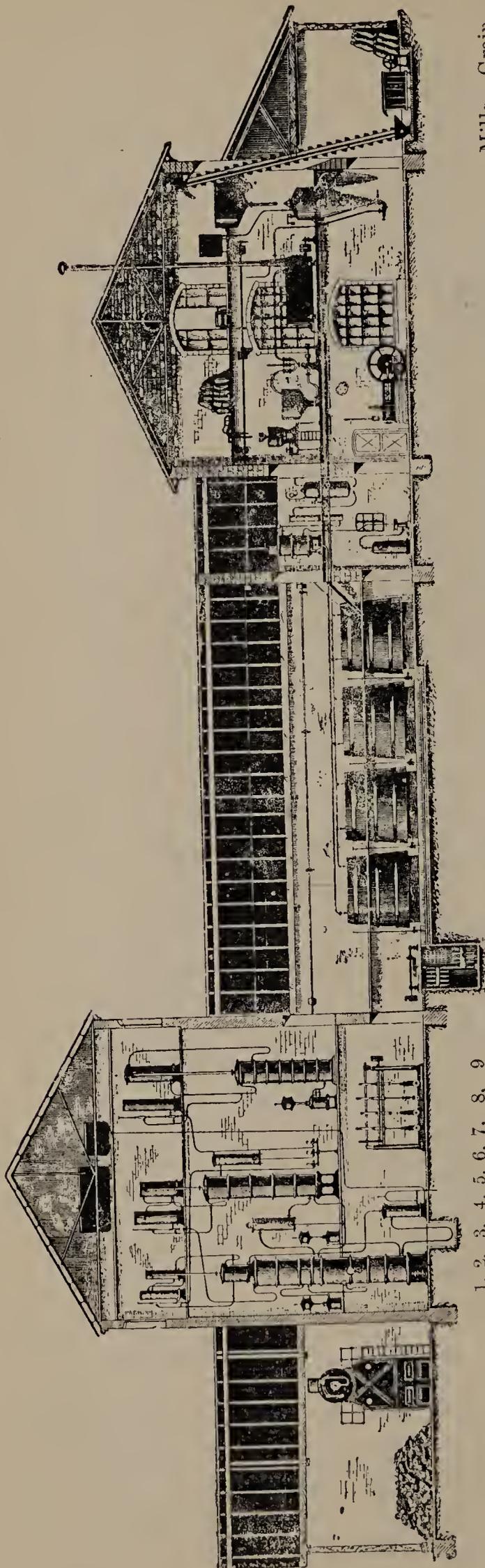


FIG. 24.—Horizontal mixing saccharifier for treating grain with acids (WAREIN and DEFRANCE).

A by steam pressure from the boiler. By this treatment saccharification is complete in fifteen to twenty minutes. This plant is made in several sizes, according to the scale on which the

INDUSTRIAL ALCOHOL



- Coal. Boiler.
- 1, 2, 3, 4, 5, 6, 7, 8, 9
- Continuous rectification distillation department.
  - 1 Refrigerator, steam regulator.
  - 2 Condenser; purifying column, first runnings (aldehydes and ethers) eliminator.
  - 3 Steam regulator.
  - 4 Cooler, special condenser recuperator or forewarmer.
  - 5 Water tank, condenser, and rectification column.
  - 6 Special condenser, test vessels in glass cases, with floating hydrometer.
  - 7 Reservoir for raw spirit from column.
  - 8 Refrigerator steam regulator.
  - 9 Condenser, distilling column, pumps.
- Continuous fermentation department.
  - Yeast mixer.
  - Fermentation tun.
  - Fermentation tun.
  - Fermentation tun.
  - Fermentation tun.
- Department for continuous preparation of pure aerobiose yeast.
  - Pure aerobiose ferment vessel —Air vessel.
  - Air steriliser.
  - Cooler.
  - Boiler for yeast.
- Continuous saccharification department.
  - Malt in sacks, malt mills, malt mixer, centrifugal mixer, air pump.
  - Horizontal saccharifier and extractor (mash tun).
  - Water tank.
  - Cylindrical measurer containing charge for one boiling —Boiler.
- Mills.—Grain crushing and sifting Department.
  - Elevator.
  - Mill and stone remover.
  - Sacks of grain.

FIG. 25.—Grain distillery on Barbet's principles. Plan of buildings and plant for the combined continuous manufacture of alcohol from raw grain by malt, and simultaneous continuous rectification of the raw spirit, all in one building.

N.B.—The various items are as near as may be virtually above title, and in descending order.

operations are conducted. A conical form is given to small plants, which are installed vertically. The operation is conducted thus : 25 gallons of hot water are run into the saccharifier for every 100 lb. of maize to be treated, then, after having opened the steam taps and set the agitator at work, it is charged with grain. After half an hour the air tap is closed and the pressure increased to three atmospheres. This pressure is kept up for two and a half hours, and the state of the paste is then ascertained. When the paste is fully formed, concentrated hydrochloric acid is run in, the proportion being  $2\frac{1}{2}$  per cent. on the weight of the maize being treated. Saccharification requires twenty-five minutes, and the worts obtained in this way are very pale. The yields of glucose and dextrin are respectively 68 and 1.75 per cent., and  $3\frac{1}{2}$  British imperial gallons of rectified alcohol of  $90^\circ$  are obtained per 100 lb. of maize. The distillation residuals (dregs) passed through the filter press are dried and pressed in a hydraulic press in order to extract the oil which they contain.

The weight of oil obtained per 100 lb. of maize is as follows : Oil, 2.3 lb., or about 1 quart, leaving 10 lb. of maize cake.

The oil is disposed of to soap makers, and the cakes are sold *as manure*.

Maize and rice oil may also be extracted from distillery residuals by volatile solvents. The plant is illustrated and described in De la Coux' "Industrial Uses of Water."

The spirit produced in Britain for methylation is substantially the same as that produced for consumption. Distillers who produce only for methylation generally use molasses, or a very large proportion of that material. Approximately three-quarters of the spirit which goes for methylation is produced from molasses, which at the present time is the cheapest material, but the price of molasses is affected by the price of sugar. It has risen rapidly, so that it may at any time become prohibitive for this purpose. Less plant is required in the manufacture of spirit from molasses, but all the plant used in the manufacture of molasses is used in the manufacture of grain spirit. With regard to the question whether the spirit produced for methylation could be used for consumption by being blended without rectification, it would be

potable, although it would not be desirable to use it for consumption. As to whether the spirit produced for methylation is a less pure spirit than that produced for consumption, it may be said that it is not such a stable spirit, and it would be liable to oxidation.

## CHAPTER V.

### THE MANUFACTURE OF INDUSTRIAL ALCOHOL FROM POTATOES.

THE potato plant is known botanically as the *Solanum tuberosum*. Several varieties of potato were long ago examined by Einhof and Lampadius, their composition being shown in the following table :—

TABLE XXI.—SHOWING COMPOSITION OF UNIMPROVED VARIETIES OF POTATOES.<sup>1</sup>

Variety of Potato.	Water	Starch.	Gum.	Albumen.	Cellulose.
Red . . .	75·0	15·0	4·1	1·4	7·0
Sprouted . .	73·0	15·2	3·7	1·3	6·8
Large red Surinam	78·0	12·9	—	0·7	6·0
Kidney . . .	81·3	9·1	—	0·8	8·8
Sweet . . .	74·3	15·1	—	0·8	8·2
Peruvian . .	76·0	15·0	1·9	1·9	5·2
English . . .	77·5	12·9	1·7	1·1	6·8

The quantity of solid matter in the potato varies with its state of ripeness : the ripest lose from 68 to 70 per cent. in drying ; the least ripe from 70 to 80 per cent. The proportion of starch varies considerably. Davy obtained from 18 to 20 per cent. Körte obtained, as the mean result of the examination of fifty-five varieties of potato, 24·9 per cent. solid matter ; the average amount of starch being 11·85 per cent. Those potatoes keep best in which starch is most abundant ; but the starch diminishes by keeping, probably passing into dextrin and sugar ; thus, from the same variety of potato, Payen obtained 17·2 per cent. of starch in

<sup>1</sup> This table is given to show by contrast the increase in the amount of starch effected by pedigree selection in the present-day distillery potatoes. See p. 109.

October, and only 14·5 per cent. in April. A portion of the albumen also at the same time disappears. Thus in new potatoes Boussingault found 2·25 per cent. of albumen (gluten), but in old potatoes only 1·5 per cent. The analyses of diseased potatoes threw no light upon the cause of the malady, or the means of cure. But pedigree selection and *bouillie bordelaise* have performed wonders. Vauquelin examined forty-seven kinds of potatoes and found the amount of starch to vary in 100 parts from 12 to 24 parts; the average result was found to be from 17 to 19 per cent. Pedigree selection has increased the percentage of starch, the alcohol-yielding constituent of potatoes.

Alcohol may be produced from potatoes either directly or indirectly. By the first or direct method the starch of the potato is fermented without any previous separation from the cellular tissue, and subsequently converted into sugar by sulphuric acid as in the indirect manner. The points in favour of converting potatoes into alcohol are that they are cheap, yield a good spirit, the residuals form a good cattle food, and a minimum of yeast is required. In order to obtain spirit directly from potatoes in the older processes, they were first steamed for an hour, and then crushed between wooden or stone rolls. Ground malt was then made into a pulp with warm water, the potato paste added, and the whole stirred until uniform, renewing the stirring until cold. Yeast was then added, but as potatoes ferment readily, this was employed in less amount than with grain. Beets or carrots were said to improve the flavour and increase the quality of the spirit. After the fermentation was pushed to its fullest extent the wash was distilled in the ordinary way. In Siemens' process (said to produce one and a half times as much spirit as the ordinary plant used in Germany), which was at one time used in Denmark, 3 to 4 tons of potatoes were steamed at a temperature slightly above 212° F., then mashed in the steaming vessel by a revolving iron cross, and sufficient warm water, rendered slightly alkaline by 1¼ lb. of caustic potash, to form a thin paste added. The resulting starch paste was passed through a sieve, leaving the skin of the potato behind. The starch liquor was rapidly cooled, yeast added, and the process finished as usual. By this method the yield of spirit

from a given quantity of potatoes was greatly increased. Fifty hectolitres 30 litres (137 imperial bushels) of potatoes, along with 8 hectolitres of ground malt, yielded 9 hectolitres or 198 imperial gallons of spirit. Cadet stated that 800 lb. of potatoes yielded 30 lb. of spirit, costing the distiller 36 francs, and selling for 48. From 50 kilogrammes (110 lb.) of potato starch converted into glucose by sulphuric acid, 20 to 25 litres (= 4.4 to 5.5 imperial gallons of alcohol) at 0.935 were obtained. Oersted, at Copenhagen, from a ton of potatoes obtained the poor yield of  $16\frac{1}{2}$  to 17 quarts of spirit at  $50^{\circ}$  of Tralles' alcoholometer.

About the year 1832, says Muspratt, a gentleman visited the distillery of Messrs. Calder at Eyemouth in Berwickshire, and found that they had worked for a short time on potatoes. He considered the spirit, which had the flavour of Hollands, to be pure and good, and although it was affirmed that no grain or malt had been used, he strongly suspected the contrary. The fermentation was described as beautiful, the head rising 7 or 8 feet like clouds of cotton, and when beaten down to the surface of the worts it rose again in the same manner. The gravity worked at was  $40^{\circ}$ , and the attenuation was good. The potatoes were ground in a mill like a common pepper-mill in shape, but made of sheet-iron perforated like a grater. The pulp thus produced was mashed in the keeve with boiling water, and the extract run off quite pure and freely. A sperge or small wort of about  $20^{\circ}$  gravity was obliged to be used, otherwise the worts at the gravity of  $40^{\circ}$  could not be got off; the produce was good, as there was no deficiency. The spirit sent to the London market, when called "grain spirit" in the permits, was highly prized; when the error was corrected, and the product was denominated "spirit distilled from potatoes," the price fell and it was not so much in vogue. About the same time, Mr. Jameson of Fairfield, near Enniscorthy, commenced distilling from potatoes. They were sliced, dried on a corn kiln, ground to flour mixed in certain proportion with grain, and mashed in the ordinary manner. But the manufacture was abandoned in consequence of the opposition of the peasantry through fear of a scarcity in the article of food. That was shortly

prior to the appearance of the potato disease and the sad famine which ensued.

At the present time alcohol is distilled from potatoes on a large scale in Germany. The following account of a German potato distillery is from the report of the Excise Committee :—

*Marienfelde agricultural distillery.*—There is in the neighbourhood of Berlin an example of an agricultural distillery. It is situated at Marienfelde, some ten miles to the south of Berlin, on a large and apparently very flourishing farm. The potatoes (which must be produced on the land of the proprietor) are first washed by machinery. They are then steamed and pulped and driven through a strainer into the mash-tun (Figs. 21 and 22, Chap. IV.), where they are mixed with a small percentage of malt. The wort is then passed into the fermenting vats. Each vat is gauged, and its content marked on the outside, together with the number of the vat. The wash is left to ferment for thirty hours, and is then conveyed to the still, *which is of the patent-still type*. On issuing from the condenser the spirit passes first through a domed glass case in which is a cup (Figs. 47 and 48). In this cup, into which the spirit flows and from which it overflows, there float a thermometer and a hydrometer, to indicate the strength of the spirit passing. From this apparatus the spirit flows into a (Siemens) meter, fitted with an indicator which records the quantity, reduced to the standard of pure alcohol, of spirit transmitted, and from the meter the spirit passes on to the receiver. The system of control does not require the continuous attendance of Excise officers, but is compounded of—(1) mechanical contrivances ; (2) book entries ; (3) liability to visitation at any time.

(1) *Mechanical contrivances.*—Up to the point at which the wash passes into the still, these are limited to the gauging of the vats and to the plumbing under Revenue seal of all joints of the pipes leading from the vats to the still. From that point onwards to the receiver every vessel is locked and sealed, so that no access to the spirit can be obtained by the distiller. Up to this point the manager is treated as an honest man. Afterwards he is no longer trusted. In the smaller distilleries, the meter, an expensive

apparatus, is dispensed with, the quantity of spirit distilled being ascertained by the Excise officer from the receiver. Whether there be a meter or not, the receiver is, of course, under lock, and is not accessible to the distiller.

(2) *Book entries.*—The regulations require entry of the quantity of materials used. These are regarded as of small practical value, and little attention is paid to such records. It is manifest that they cannot be susceptible of any real check. The important entries are those of the times of charging and discharging the several fermenting vats, and of the quantities of wash in each. These entries can, of course, be checked against the spirit found in the receiver, and from them is computed the vat-tax and the distillery tax, which have to be paid by the distiller.

(3) *Liability to visitation.*—It will be seen that the control under (1) and (2) provides no security against abstraction of wash from the fermenting vats. Visitation at frequent and uncertain intervals would seem to be an essential feature of the system, and at Marienfelde the visits of Excise officers are regarded as even unpleasantly frequent. Whether they are so in more remote distilleries may be open to doubt. In any case, the system of control rests so heavily upon confidence that, if satisfactory with a low duty on spirits and with a system of rebates of duty that makes the Excise a source of profit to the smaller distiller, it could not safely be adopted where the duty is as high as it is in the United Kingdom, and invariable in its incidence. The distillery at Marienfelde is one of the best and largest type of agricultural distillery. Its "contingent" is 600 hectolitres per annum, or about 23,000 proof gallons of spirit. Of the total number of agricultural distilleries in the German Empire there are not more than 2000 or 3000 of similar size and character. The vast majority of the agricultural distilleries are to be found in the eastern provinces of Prussia and Saxony, where the soil is poor, and the cost of conveying agricultural produce to a remunerative market is high: it is not quite clear how it can be commercially profitable on a fertile farm close to Berlin to convert potatoes into spirit. In 1905, even with the abnormally high price of spirit, no more than from £2 to £2 5s. per ton would be

realised on potatoes used for distillation, whereas if sold for consumption as food they would have realised £4 per ton. The distiller is, however, compelled to use them in the distillery, in order to maintain his "contingent," which might be reduced if he should

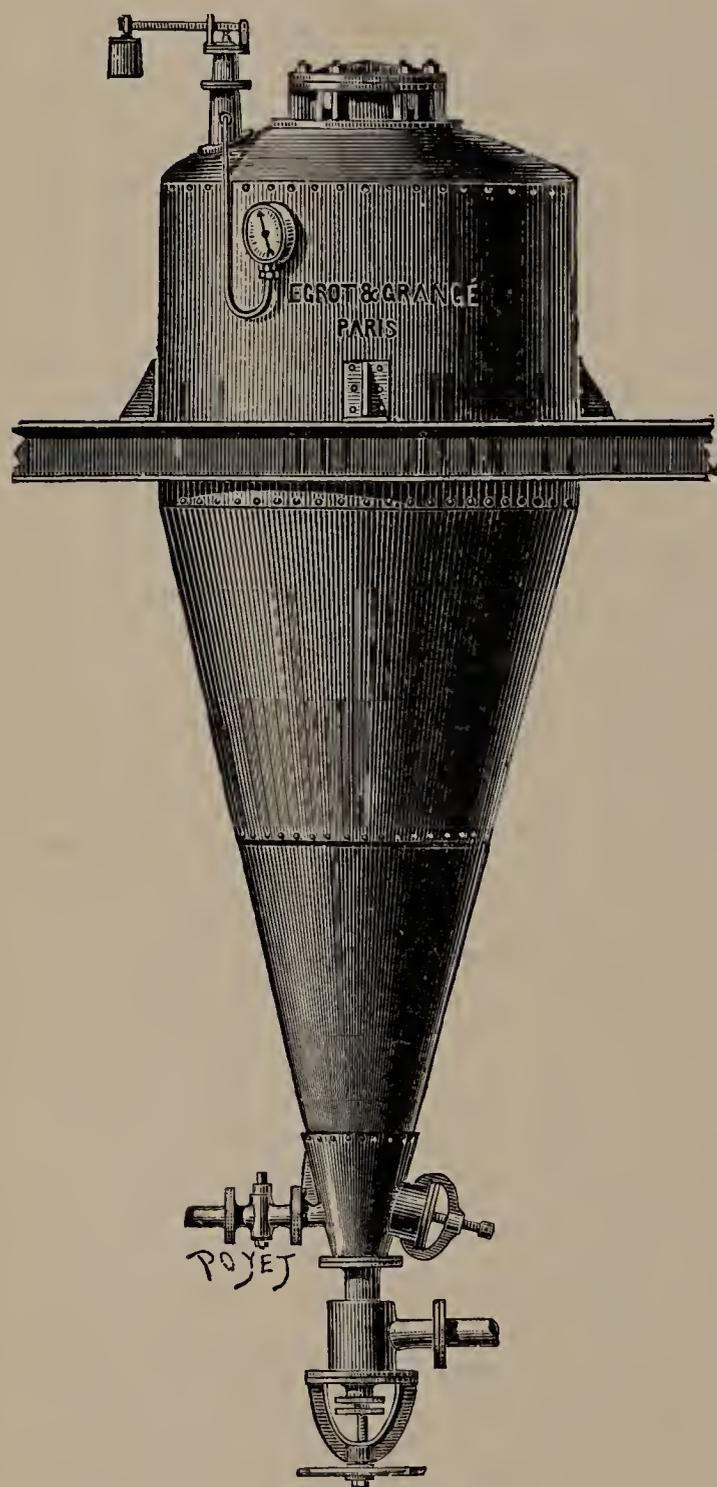


FIG. 26.—Autoclave for saccharification of potatoes by malt, under pressure (EGROT and GRANGÉ).

fail in any season to reach his prescribed production of 600 hectolitres. Moreover, the above figure of return on the potatoes very possibly does not include the bonus of 20 marks (£1) per hectolitre on the amount of the contingent. This would be equal to more than another £1 per ton for the potatoes used. The explanation of the maintenance of this distillery may be that it is kept up to some extent as a convenient object-lesson in the neighbourhood of Berlin, for the instruction of Excise officers, and to illustrate the teaching at the Institut für Gärungsgewerbe und Starke fabrication in Berlin, an institution established by the trades, with assistance from the Government, for the purpose of giving instruction in

brewing, distilling, and other processes in which fermentation is employed. In normal years the return from potatoes used in the agricultural distilleries does not exceed 25s. per ton (exclusive presumably of bonuses), and in many cases is less, the

average being about 20s. per ton. The yield of alcohol may be taken from a ton of potatoes at about 25 gallons of pure alcohol, or about 44 proof gallons.

The distilleries in Bavaria are often co-operative, and if anything came of this industry in Ireland the distilleries there would probably be co-operative. Dr. Hinchcliff, on behalf of the Department of Agriculture and Technical Instruction for Ireland, inquired into the system adopted in Germany for the manufacture of alcohol from potatoes. The industry is a fairly large one in Bavaria. In some districts within a few miles of Munich, where the soil in many parts is fairly fertile, there is a distillery in every village. The industry is, however, confined to the less fertile parts, though it is fairly well established in Saxony, where there is good land. It is, of course, more extensive still on the sandy lands of East Prussia and to the east of Berlin. The unsorted potatoes, large and small, sound and bad, are first run through a washing apparatus and afterwards conveyed to an automatic weight recorder before being delivered into the steamer. The potatoes are weighed mainly in order that the payment to the producer may be calculated. The machine used is a weight recorder very much like the weighbridges used for automatically weighing corn. It automatically cuts off the supply and tips up when charged with a certain amount, be it 1 ton or 3 tons. The spirit as it leaves these agricultural distilleries is not nearly pure, and for potable purposes has to be rectified. One cwt. of potatoes produces potato spirit = 1.43 gallon of absolute alcohol, or 2.5 proof gallons, *i.e.* about 28.6 gallons of absolute alcohol per ton, or almost 57.2 proof gallons. According to Bucheler, the *actual* amount of starch obtained from potatoes is 20 per cent.; the actual yield from a cwt. of potatoes being  $2\frac{1}{2}$  proof gallons; this is, roughly, two-thirds of the theoretical yield. Several of the distilleries near Munich reach just over 60 up to 64 per cent. of the theoretical yield. When redistilled and further purified, the alcohol from potatoes is used as potable spirit and sold under various names. Of all the spirit which is produced in Germany, something like four-fifths is produced from potatoes. Therefore, clearly, a good deal of the potato spirit must be drunk. In the

seasons 1897-98 to 1901-02 the average production per season was slightly over 381 million litres of alcohol, whilst during the period 1898 to 1903 the average yearly consumption of spirits was 238 million litres of alcohol.

The co-operative distilleries purchase potatoes from the adjoining farmers. The price varies to a great extent, somewhat according to season, as in Britain, but also according to the district. They are cheaper on the sandy soils to the east of Berlin, where there is not such a big demand. But in 1904, for instance, they were paying about 27 marks per metric ton, which was roughly 27s. per imperial ton—a high price, but potatoes were then somewhat scarce owing to a lighter crop than usual. The normal price is 20s. in an ordinary year, according to authorities, but 23s. per ton is said to be usual. Potatoes were at times in Berlin even cheaper than 20s. per ton for alcohol production purposes. The price of potatoes for domestic consumption was usually somewhat higher.

Potatoes used for distillation purposes in Germany are not all manipulated in so-called co-operative distilleries. In Bavaria there are in certain districts several proprietary distilleries where each farmer has a distillery. In Saxony also the owner of a large farm may have his own distillery, a single farm in Germany being in some cases able to keep a distillery supplied; also some of the large estates in Bohemia and Austria possess distilleries. But, as a general rule, in Bavaria more than one farm, or even estate, is needed to keep a distillery going. In East Prussia the farms are large, and owing to the sandy nature of the soil, that is where potatoes are mainly grown; there a single farmer would be able to grow sufficient potatoes to run a distillery. The distilleries are co-operative in the strict sense of the word,—they divide their profits amongst those who run them. The potatoes are paid for on the basis of percentage of starch, which is estimated by a not very exact method in some cases, but by means of the specific gravity, and by using Maercker's formula, so that for potatoes containing, say, 20 per cent. of starch the price would be regulated accordingly. A certain standard is first taken as the basis, and for every unit per cent. of starch over and

above this, a certain additional amount is paid per metric ton or centner. In one distillery visited by Dr. Hinchcliff, in addition to barley, a mixture of imported maize and potatoes was used, but these were the only products met with. In Bavaria the varieties of potato specially suited for the production of spirit give a somewhat larger yield than the kinds grown for household consumption; 7 tons per acre, however, is considered a satisfactory yield. About 6 tons per statute acre is an average crop in Britain, and about 4 tons per statute acre in Ireland, but it is not considered a good crop if under 7 tons.

The quality of the potato for distillery purposes has improved of late years, those having a high starch content having been chosen for planting. The duty is not imposed primarily with the object of encouraging the production of a high class quality, but rather with the idea of encouraging potato culture; in fact, it is not imposed in relation to the weight of the raw material that is dealt with in an agricultural distillery, but only with regard to the output of alcohol; but the capacity of the mash-tun being also taxed, it is advantageous to put in as concentrated a mash as possible, and hence to use potatoes that have as high a starch content as possible. At the Alcohol Exhibition at Vienna, several varieties of potato were exhibited, these having been tested at some of the experimental stations and found to contain over 22 per cent. of starch; and not only should the potatoes contain a high percentage of starch, but they ought to be of a character not to yield very much dextrin. The relative quantities of maltose and dextrin in the mash should be as far apart as possible, and in that respect not only the starch content, but also the character of the potatoes as regards the dextrin formed, has much improved. There is a prevalent misunderstanding regarding the possibility of converting diseased potatoes directly into alcohol. This can only be done by first separating the starch from the unsound portion of the potato. The attempt to work up diseased potatoes *directly* into alcohol would be a very doubtful economy.

M. Delbrück<sup>1</sup> is of opinion that it is more economical to dry

<sup>1</sup> *Z. Spiritusind.*, 1904, 27, 301.

potatoes than to keep them in storage over a long period, the cost of drying being more than counterbalanced by the loss occurring during storage. He also states that the cost of producing alcohol has been very materially lowered by the introduction of the "amylo" process from Japan, the saccharification of the mash, after liquefaction has been brought about by a minimum quantity of malt, being performed by inoculation with the fungus *Amylomyces Rouxii*. By this process almost the theoretical percentage of alcohol is produced.

Dried potatoes are obtained either in the form of thin slices, which have been dried on frames by means of a current of hot air, or in the form of flakes produced by crushing boiled potatoes between heated rollers, and, according to G. Heinzelmann,<sup>1</sup> it is necessary to submit these to the same treatment as maize, having an efficient stirring arrangement attached to the steamer, so that they may be acted upon throughout, otherwise they are liable to agglomerate together into solid masses into which the steam cannot penetrate. In thick-mash distilleries the yield amounts to 33 to 35 litres absolute alcohol per 100 kilos of dried potatoes, varying with the proportion of starch present.

<sup>1</sup> *Z. Spiritusind.*, 1904, 27, 358.

## CHAPTER VI.

### THE MANUFACTURE OF INDUSTRIAL ALCOHOL.

#### FROM SURPLUS STOCKS OF WINE, SPOILT WINE, WINE MARCS, AND FROM FRUIT IN GENERAL.

INDUSTRIAL ALCOHOL FROM WINE AND WINE MARCS.—*The grape vine and its varieties.*—The raw material is the grape or fruit of the vine, a perennial climbing plant which flourishes in moderately warm climates, and of which there are several varieties, termed *cepages*. The quality of the wine depends not only on the variety, but also on the soil, position in relation to the sun, the manure, the nature of the ferment, and the care exercised in the fermentation. Moreover, the different varieties do not prosper in all soils and climates: thus *le Gamay* flourishes in Beaujolais; *le Vionnier*, *la petite syrah*, and *la Roussane* in l'Hermitage; and so on. Generally, a high temperature yields red wines, rich in alcohol, but poor both in acid and in bouquet. Red grapes may be treated so as to yield red, rose, or even white wine; white grapes are only used to produce white wines. The grape ripens in September or October, ripening being hastened, in cold climates, or in wet weather, by removing the leaves. The degree of ripeness to attain varies with the wine to be made, and maturity is estimated by instruments termed *mussimètres*, or, judged by taste, the juice becoming more or less sweet.

*Collection and pressing.*—The grapes are detached from the plant by a knife, placed in baskets, etc., and carried into large brick, cement, or wooden vats, where the juice is pressed out by the feet, the hand, or mechanically. The saccharine juice, or must, separates from the envelope, and, under the influence of the *mycoderma vini* present on the grape, ferments. This

TABLE XXII.—COMPOSITION OF WINES FROM DIFFERENT WINE-GROWING DISTRICTS, ALSO OF “PIQUETTE” OR SOUR WINE FROM THE MARC.

SOURCE OF ORIGIN.	Alcohol Per Cent. vol.	EXTRACTS		Ash.	Tartar.	Reducing Substances as Glucose.	Sulphate of Potash.	Acidity as H <sub>2</sub> SO <sub>4</sub> .
		at 100°.	In Vacuo.					
Aude, Corbière, 1882 . . . . .	10·3	24·6	29·6	4·35	1·30	3·80	3·67	3·76
„ Narbonne, 1881 . . . . .	9·6	22·4	26·3	4·10	2·25	1·70	2·80	5·00
„ „ 1883 . . . . .	10·5	22·6	26·3	4·30	2·63	1·08	3·48	5·39
Charente, 1883 . . . . .	8·2	18·5	23·1	2·40	2·73	1·21	0·31	5·09
Cher <i>blanc</i> , 1883 . . . . .	7·2	16·0	19·6	1·68	3·30	0·86	0·25	6·61
„ rouge, 1883 . . . . .	6·6	20·6	25·6	2·60	4·22	1·63	0·34	6·81
Côte-d'Or, Beaune . . . . .	9·3	21·7	25·8	2·10	3·76	2·40	0·40	3·19
„ Pommard vieux . . . . .	11·9	21·6	24·3	2·03	1·51	0·40	0·65	3·25
„ Puligny, 1879 . . . . .	6·8	23·3	27·5	1·87	2·83	1·00	0·23	5·31
„ rouge, 1883 . . . . .	7·1	17·7	22·9	1·63	3·49	0·91	0·25	7·05
„ <i>blanc</i> , 1883 . . . . .	7·9	14·1	20·0	2·04	3·02	1·04	0·15	5·00
Gard, Nîmes, 1882 . . . . .	9·4	22·7	25·9	3·81	3·49	0·90	1·82	3·43
Gironde, Saint-Estèphe, 1878 . . . . .	11·1	22·4	28·3	2·20	1·31	1·50	0·49	2·96
„ Saint-Emilion vieux . . . . .	10·9	22·1	27·9	2·60	2·00	0·80	0·72	4·60
„ Château-Larose, 1864 . . . . .	10·9	21·5	26·2	3·00	1·90	0·50	0·53	3·90
„ Mouton-Rothschild, } 1878 . . . . .	11·7	22·3	27·2	3·00	1·60	0·50	0·58	3·50
Hérault, Capestang, 1880 . . . . .	8·0	18·4	22·4	3·56	2·44	3·80	2·33	3·82
„ Ramejan, 1881 . . . . .	8·9	20·9	25·2	2·85	3·00	1·30	1·32	2·86
„ „ 1883 . . . . .	10·0	18·2	22·4	2·12	3·67	0·52	0·69	4·85
„ rouge, 1883 . . . . .	6·6	23·7	25·9	4·60	3·01	0·91	3·50	6·51
„ Minervois, 1883 . . . . .	7·9	23·4	24·2	4·08	3·03	0·89	2·81	6·02
Indre-et-Loire, Bléré, 1881 . . . . .	8·2	23·7	26·3	2·11	2·03	1·40	0·18	3·00
Loir-et-Cher, Blois, 1881 . . . . .	7·6	18·3	21·9	2·15	3·20	1·80	0·25	4·83
Loire-Inf. Nantes <i>blanc</i> , 1883 . . . . .	6·1	15·1	19·1	1·48	1·89	1·02	traces	6·81
Lot, Cahors, 1881 . . . . .	10·0	21·8	25·0	1·97	3·92	1·70	0·16	3·40
Pyr.-Orient. Roussillon, 1881 . . . . .	12·3	24·7	28·9	3·87	1·04	2·50	3·02	2·92
Saône-et-Loire, Mâcon, 1881 . . . . .	10·5	18·7	24·1	1·85	2·10	0·70	0·53	5·07
„ Thorins, 1878 . . . . .	12·2	24·0	31·6	2·14	2·43	1·80	0·30	4·96
Yonne, Augy, 1881 . . . . .	7·0	19·3	23·7	2·30	2·78	2·10	0·41	5·00
„ Joigny, 1884 . . . . .	8·0	20·3	24·8	2·24	3·29	1·74	0·15	5·72
„ <i>blanc</i> , 1883 . . . . .	7·7	19·2	21·9	1·64	3·58	0·81	0·02	7·25
Algeria, Bône, 1881 . . . . .	10·3	19·1	24·6	2·89	0·82	0·60	1·65	6·37
„ Staouéli, 1880 . . . . .	10·4	22·3	28·8	4·62	0·80	0·70	4·07	4·80
Spain, red, 1881 . . . . .	14·8	25·6	30·0	4·03	1·90	3·50	3·00	2·70
„ „ 1883 . . . . .	10·7	19·2	24·2	4·32	1·98	1·09	3·32	4·50
Italy, Riposto, 1880 . . . . .	13·2	24·1	28·4	3·88	0·86	3·90	1·51	2·90
„ red . . . . .	13·0	32·0	37·6	4·52	2·92	4·38	3·74	6·17
„ Sicily, 1883 . . . . .	13·8	27·2	33·0	3·08	1·51	3·62	0·37	4·80
Portugal, red, 1882 . . . . .	13·5	20·8	26·0	2·92	3·15	2·90	0·27	3·72
Turkey, Adrianople, 1878 . . . . .	11·4	22·9	29·6	2·47	2·06	5·00	0·71	3·10
Piquette, lixiviation of, 1883, } mars, Midi . . . . .	5·9	17·9	20·8	4·68	3·59	traces	2·75	4·07

fermentation is favoured by aeration, keeping it at a temperature between 15° C. and 30° C., and using, if need be, selected ferments. Choice ferments transform all the sugar into alcohol, increase the strength of the wine, and prevent the formation of secondary products which impart a bad taste. Formerly the skins, brought to the surface by the carbonic acid, remained on the surface of the liquid. Now, it is preferred to keep them under the liquid, so that they do not decompose in the air and impart a bad taste to the wine. When fermentation has nearly ceased, the liquid is drawn off whilst it is still mild, and the transformation of the sugar into alcohol is finished in casks. The skins left behind retain a certain amount of saccharine juice, and constitute the *marc*, which, if pressed and fermented, yields an inferior quality of wine. If water be added to the grape skins, and they are then pressed a second time, *piquette* (sour wine) is produced. The casks of wine must always be kept full, but open at the bung hole, so that the surface exposed to evaporation and air is not too great, in order that there may be little loss of alcohol, and that the wine does not turn sour. (1) *Vinage* is the adding of alcohol to a poor wine; (2) *mouillage* is reducing the proportion of alcohol by adding water; (3) *avivage*, brightening the colour by adding sulphate of lime or tartaric acid; (4) *coupage* is the blending of several qualities to produce an average one. White wines may be made from white grapes, or red grapes, but in the latter case they are termed *vins faits en blanc*. If white grapes are used, the process is practically the same as for red wine. But if red grapes be used, the grapes must be pressed gently so as to cause the juice to exude without the envelopes falling into the liquid; the skins containing the colouring matter should not remain in contact with the liquid after fermentation sets in.

*Barbet's process of cultivating pure wine ferment.*—The application of Pasteur's principles to wine presents special difficulties. Up to recently all that has been done is to despatch to wine-growers when required a few bottles of ferment, leaving the culture thereof to the wine-growers themselves when a second crop of ferment enables them to set their fermentation tuns. But the

delicate preparatory operations required are beyond the capabilities of wine-growers, who have not the equipment indispensable for maintaining the ferment in the proper state of purity. Successive fermentations are made in non-sterilised tuns with a must which has not itself been suitably freed from extraneous micro-organisms, so that very often when the ferment reaches the wine-grower's fermenting tuns it is not at all pure, and the wine-grower does not recoup himself in the resultant wine for the expense and trouble to which he has put himself. To work satisfactorily with pure and active ferments, special plant is required—a steam boiler for sterilisation and plant for pumping sterilised compressed air; in a word, it requires a mechanical and engineering plant on the large scale, which on account of its price cannot be erected except in very large viticultural exploitations. But what costs too much for an individual, becomes realisable and practicable for a sufficiently large group of wine-growers. What is required for each district is not a laboratory distributing small quantities of pure ferment, but a small factory supplying each vineyard, and, for each vat, an ample supply of pure ferment in full activity.

Fig. 27 shows a plan of such a factory, which can be annexed to an existing wine-cellar. The necessary grape juice is supplied by the wine-press to the pure ferment. The juice is pumped into the upper reservoir A, from whence it passes to the tubular heater B, which it traverses from below upwards—an apparatus in which the juice advantageously benefits by absorbing the greater part of the heat of the hot sterilised juice, so that when it reaches the steriliser C it requires but little steam to be brought to the boil, or to a temperature bordering on ebullition. The sterilised juice issues by the tap *a*, descends to the heater B, then passes to the cooler D, from which it should issue at the right temperature for the fermentation for which it is to be used. Another part of the sterilised juice enters the cooler F, and from thence passes into the pure ferment apparatus G, in which the ferment is cultivated on very shallow plates in its upper part, and on to which the must is continually elevated by a compressed sterilised air emulsifier H. The “*aerobiose*” treatment recom-

mended by Pasteur is thus realised. The air steriliser L is on a new plan. It consists of a cotton-wool filter enclosed in a steam autoclave, so that the cotton can be from time to time sterilised with the minimum amount of trouble. J is the steam pump to

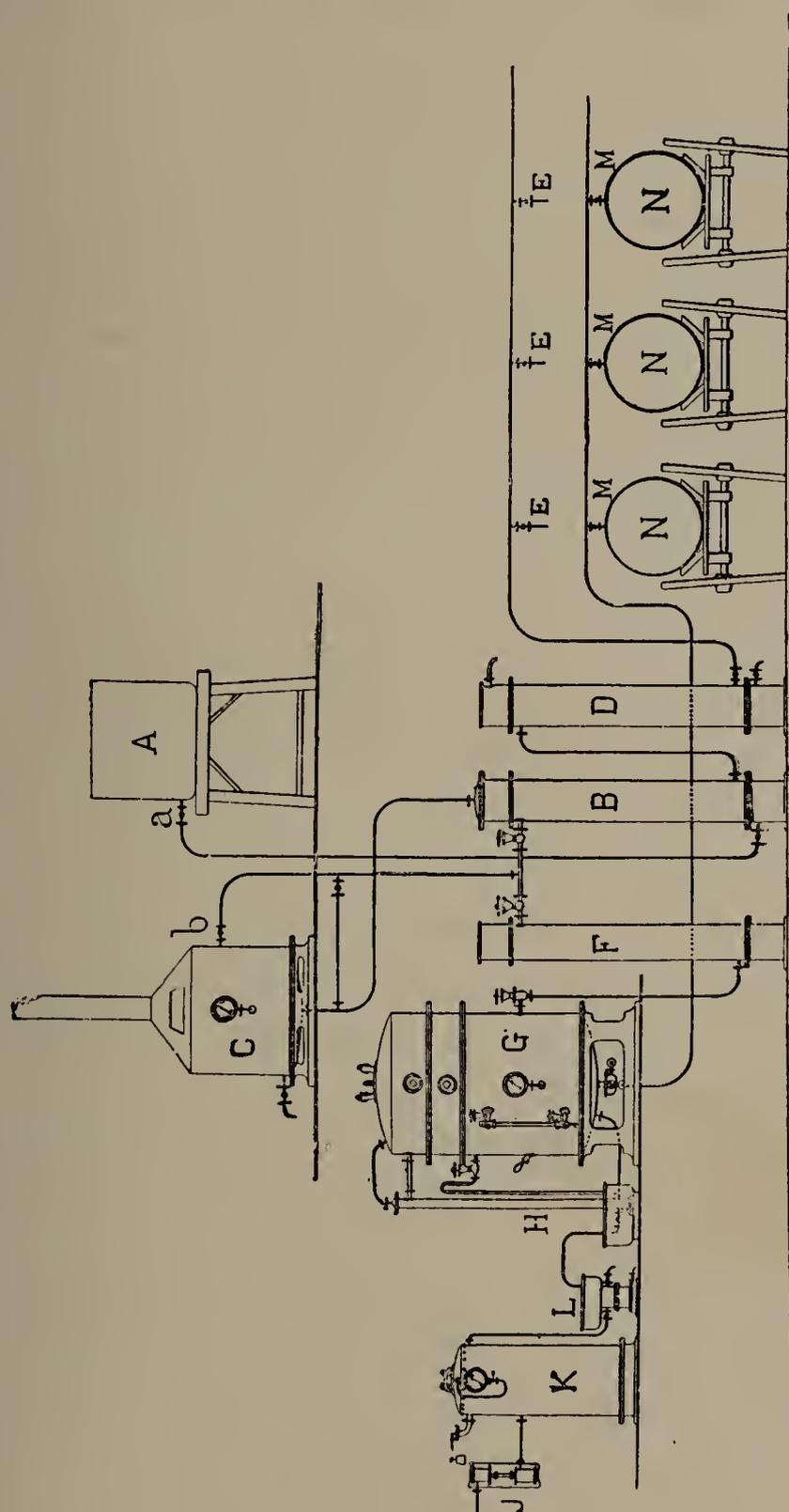


FIG. 27.—Plant for production of pure aseptic ferments for fermentation of wine.

compress the air. K is the compressed air receiver. The tubular vessels BDF are fitted with sterilisable tubular cases. NNN are galvanised iron casks tinned inside. They are intended for transporting the pure ferments to the wine-cellars in the

neighbourhood ; for this purpose they are mounted on convenient trucks. After washing and sterilisation, the leaven casks N are filled with sterilised juice cooled in B and D and treated with a little of the pure ferment drawn from apparatus G. Fermentation in the casks is allowed to go on, and the carbonic acid gas is not allowed to escape, the casks possessing safety valves gauged to 1 kilogramme per square centimetre. When the casks reach the wine-cellar for which they are intended, the contents are in active fermentation and under a pressure, which will be sufficient to elevate the ferment into the fermenting vats without requiring a pump. This simple device avoids contaminating the ferment. Fermentation develops instantaneously in the vats, thus ensuring that no mishap occurs in fermentation—mishaps which occurred too frequently by the old methods. If in the region in which this system is carried on two or three very distinct kinds of wine are produced which require different strains of ferment to be used, it suffices to have in the works two or three apparatus G, each with its own strain of ferment, the plant otherwise remaining as before. The continuous sterilisation plant is a perfect instrument for use afterwards in pasteurising the juice drawn from it. A double advantage is thus derived from the same plant.

*The Distillation of Alcohol from Wine.*—The agricultural depression in the South of France and Algeria and similar wine-growing districts is due to the fact that the consumption of wine has not increased in the same proportion as its annual production. The annual production of these two districts have exceeded the annual consumption in France by 440 million gallons. This unsaleable excess, deprived by fiscal enactments of all chance of exportation, as such, must needs find a new outlet, the only outlet possible being distillation. French opinion encourages alcohol from fruit being used widely for consumption, preferably as a beverage ; whilst, on the other hand, the French Minister of Agriculture strives to open new and illimitable outlets for the industrial alcohol of the North of France as an illuminant, a heating agent, and a source of motive power. But, on the other hand, the wine is not exportable, as such ; brandy is not subjected to the same disability. In fact,

France, twenty-five years ago, exported 13,200,000 gallons of brandy, calculated at 100° G.L. at high prices. This wide outlet has gradually narrowed in proportion as unscrupulous merchants blended the Charentais product, decimated by the phylloxera, with du Nord alcohol; and now, when abundance has once more returned, the French regard it as not impossible to reconquer the foreign market by new brandies equal to those of their predecessors. The wine-growers of Southern France (Armagnac excepted) and Algeria found that their brandies were not generally esteemed, either by direct customers or by wholesale merchants, and that the latter prefer red du Nord alcohol for making their cheap blends. If the industrial raw material is afflicted with an original unpleasant odour, it cannot be denied that its fermentation is accomplished with the greatest of care, and that it engenders few fermentation impurities. If we examine wine alcohol of bad quality, we find in such a product special impurities which do not generally exist, in appreciable quantity, in good industrial alcohols. For instance, there is often noticeable in wine alcohols a piquant odour, recalling burnt paper or burnt caoutchouc. This is due to acrolein, formed by fermentation at far too high a temperature. This defect is almost general in Algerian spirits. Often, again, the dominant odour is of sulphurous acid, which may be very pronounced. If a solution of baryta be poured into this alcohol, an abundant precipitate, not only of barium sulphite, but even of oxide of iron is produced, because this alcohol has strongly corroded the iron tuns of the warehouse. Sulphurous acid exists not only in the free state, but it has formed very unpleasant sulphited ethers in contact with the boiling alcohol. If the ethers in the above alcohol be destroyed by potash, traces of sulphuretted hydrogen may be detected, which was previously combined with the alcohol, and formed a sulphuretted ether of very unpleasant odour. Finally, chemical analysis reveals the presence of another category of impurities, amines or ammoniacal compounds analogous to those given off by over-ripe cheese or fish. None of these defects should exist in wine alcohol, if made from sound wine. But until the southern district of France protects itself against the effect of a hot climate, until they produce

wines from a sufficiently pure fermentation, it will be necessary to rectify the brandy carefully by distillation. The apparatus must be constructed in such a way that the plant can produce a sound and proper brandy even from the worst wines. The origin of the acetic, sulphuretted, and sulphurous ethers, the obnoxious presence of which has just been indicated, must be sought for. By experiment we find that ethers are formed when boiling alcohol of great strength comes in contact with acids, mineral or organic, liquid or gaseous. Acetic ether arises from the acetic acid in the piquées wines. Sulphurous ether comes from the sulphurous acid added to the wine, on the one hand by repeated *michages* of the casks and on the other hand as bisulphite, to make *mutage* or to preserve the wine against cryptogamic maladies, or, finally, to bleach the wine when it is of a rose colour.

Sulphuretted ether, or mercaptan, arises from sulphuretted hydrogen, which one would scarcely expect to see figuring amongst the ingredients of wine. But it must be borne in mind that the vines have been drenched several times with *bouillies bordelaises* containing sulphate of copper, and that flowers of sulphur are also used; the sulphides may also be due to reduction. All these chemical products are met with to a great extent in the fermentation tun, and certain wine-growers assert that they have sometimes found natural sulphur agglomerated in the lees of a tun. It is not astonishing under such conditions that the formation of sulphides has resulted, which, under the influence of the acidity of the wine, disengage sulphuretted hydrogen. Acetic acid, sulphurous acid, and sulphuretted hydrogen are three volatile acids, which on distillation become disengaged from the wine, and rise into the upper parts of the distilling column at the same time as the alcohol becomes stronger and stronger, till the strength of the alcohol is such as to enable it to react with the acids which accompany it, and combine therewith, in the form of ethers. To allow this combination to be accomplished is an almost irremediable mistake, because the ethers, so formed, can henceforth only be destroyed by energetic chemical reagents, such as potash or soda, which act in their turn on the alcohol,

and on the vinous perfumes, in a very objectionable manner. Prevention is better than cure.

The danger of chemical combination does not exist until the alcohol becomes concentrated. It is advisable, therefore, to free the vapours from their dangerous associates as soon as they are liberated from the wine, and recourse may be made for that purpose to the methods adopted by chemists in laboratories for purifying vapours or gases. The vapours are sifted, or scrubbed over fragments of marble, to retain acid gases, and the action is completed by bubbling through a liquid which contains fixed salts, without odour, capable of retaining the last traces of sulphuretted hydrogen. One of the liquid washers or scrubbers is intended for another purpose, that of absorbing ammoniacal compounds, products arising principally from fermentation made in unsuitable localities or tuns. Once the alcoholic vapours are thoroughly scrubbed by appropriate solutions, they may be allowed to continue their upward course, and it will soon be seen that if the wine being treated has not now the defects already mentioned, we have only to stop introducing the reagents, and every defect will reappear (Fig. 30).

The scrubbers are almost always effective when rectified alcohol is desired, because it is necessary to obtain absolute freedom from odour. When it is desired to make brandy, the cleansing is less needed by reason of the fact that the product has a sufficient aroma in general to mask completely small defects.

*By-Products.*—The vine contains cream of tartar, the price of which in the crude state is greater than that of alcohol; it is economically extracted by the same multiple effect evaporators as are used in sugar works (Fig. 30). Wine also contains a notable proportion of glycerine, 5-9 lb. per 100 gallons, at £40 per ton. In a day's turnover of 2200 gallons this is worth 55s. to 90s. A well-equipped distillery should not neglect such an important source of profit, which would pay all its general expenses. The treatment of the marcs, from the pressing, ought also to be undertaken, so as not only to extract all the spirit, but also to obtain cream of tartar and tartaric acid; the wine-distiller ought

also to separate the seeds, which serve as a food for poultry, or 12 to 15 per cent. of good quality oil may be extracted therefrom. A small wine distillery concentrating the working of 1000 to 1250 acres of vines ought to fulfil the following programme: Make pure ferments at the moment of pressing. Centralise all the marcs for distillation. Profit by use of apparatus for sterilisation of juice for pure ferments so as to effect the pasteurisation of the wines from the first pressing. Distil the wines. Profit by the plant for the exhaustion of marcs to work other alcohol-producing plants, beets, sorghum, asphodel, or Barbary figs in Algeria, etc., with the view of producing denatured alcohol for lighting. Utilise all the by-products, tartar, salts, glycerine, seeds. Such a factory (Figs. 29, 30) would have work for nine months out of twelve, and consequently would be in excellent condition to maintain a capable technical staff and have but slight depreciation expenses. In France agricultural distilleries of this nature may be easily formed on co-operative principles, making, if need be, appeal to the financial aid of the district agricultural banks constituted by the law of 1897. The privileges of the Bank of France and that of the Bank of Algeria were only renewed on condition of placing a substantial amount of capital at the disposal of agricultural enterprises of this nature.

*Continuous steam still.*—More particularly intended for “Vins de bon cru.” 1. *Prolonged boiling.* It is of great importance that the wine undergoes prolonged boiling. That is the chief reason of the superiority of intermittent stills over *ordinary* columns. The wine to be distilled is pumped, in a continuous manner, into the tank P (Fig. 28). The feed is regulated by the dial tap (15), and the wine is first warmed in the tubular vessel M, absorbing heat from the boiling vinasse issuing from the tap (17) of the still D. It is heated automatically without any fuel consumption whatever. The hot wine enters (by 16) the bottom of a very large reservoir G, which contains sufficient to feed the plant for three or four hours. The heating of the wine is continued up to 92°-93° C., *i.e.*, to a point bordering on ebullition. The wine thus undergoes intense and prolonged pasteurisation. The heating to 92°-93° C. is effected by a steam coil, receiving

steam from the dome of the still D by the tap 19. The boiled wine enters the column of plates, and is exhausted in descending

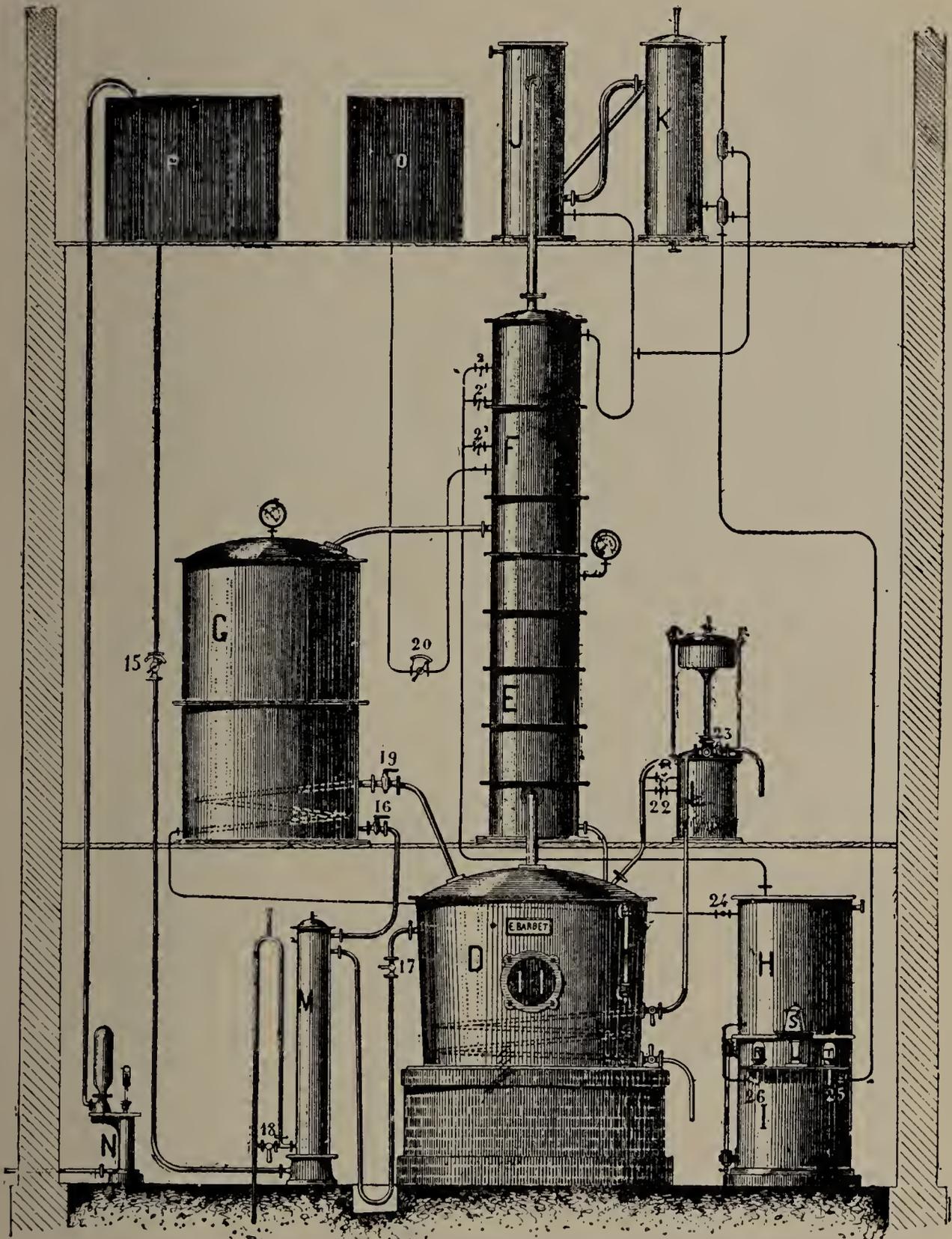


FIG. 28.—Plant for distillation of wine. D, still; E, F, rectifier; G, wine boiler; J, condenser; K, refrigerator; M, wine forewarmer; N, pump; O, fatty acid liquor; P, wine tank (E. BARBET).

through the plates E to the still D. There it enters a still, so capacious that the boiling of the vinasse is prolonged for three hours at least, allowing the volatile acids in the wine, or in the

cells of the ferment, to be disengaged. 2. *Etherification*. Part of the acid vapours ascend through the plates E and F, to come

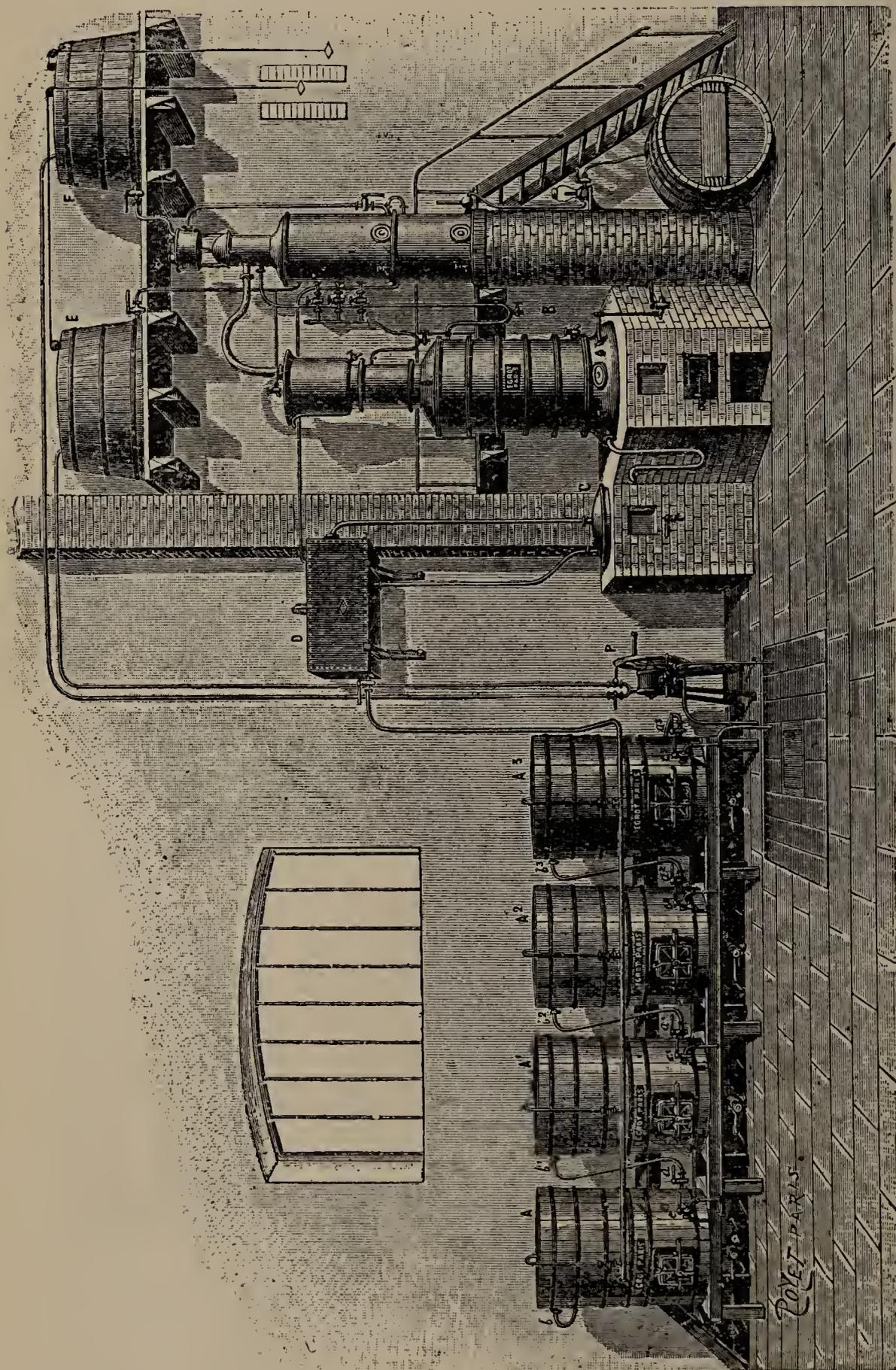


FIG. 29.—Plant for manufacture of alcohol marc brandy, from marc of wine (EGROT and GRANGE). A A<sup>1</sup> A<sup>2</sup> A<sup>3</sup>, maceration vats; a a<sup>1</sup> a<sup>2</sup> a<sup>3</sup> b b<sup>1</sup> b<sup>2</sup> b<sup>3</sup>, connecting pipes conveying juice obtained by maceration from the bottom of the one macerating tun to the top of the next; C C<sup>1</sup> C<sup>2</sup> C<sup>3</sup>, taps for drawing off macerated juice; P, pump; D E F, maceration juice tanks; B, continuous still; G, rectifying column.

in intimate contact with the vapours of the wine, and to etherify them. But as these acids are not very volatile, possibly they

would be arrested in the lower stages of the column, and it would be impossible to reach the level where the brandy is of greatest alcoholic strength and easily etherifiable. The steam which completes the heating of the wine in G is the *vinasse* steam. The condensed liquid which results is therefore charged with volatile acids and affords an exit for them. This perfumed water is collected, after cooling, in the double refrigerator H. It issues through the tap R, where a delicate alcoholometer permanently controls the exhaustion of the *vinasse*. When the plant is well regulated, no more alcohol remains in the still D, and the liquid from the coil of G ought to mark zero on the alcoholometer. This acid liquid is pumped into the tank O, and through the dial-gauged tap 20 it is introduced into the column F, above the wine, a stage at which it finds brandy, at 40° or 50°. The perfumed acid water may alternatively be pumped to the wine tank P. Etherification is effected, and once accomplished cannot be destroyed. 3. *Purification*. The brandy becomes more and more concentrated in the plates F, owing to total retrogradation from the tubular condensers J and K. At the exit of the refrigerator K there is allowed a permanent flow of first runnings of 2 to 5 per cent., which is collected at the tap T. It contains some aldehydes and unpleasant gases. This product reaches 90°, and even 93°. The remainder of the condensed liquid re-enters the apparatus, and, as may be imagined, at this great strength the etherification of the œnanthic and other acids is rapidly effected. The brandy redescends on the plates F, where it is actually boiled as in the *Charentaise* “*repasse*.” In proportion as it descends, it loses in strength, and the distiller continuously extracts it through taps 2, 2<sup>1</sup>, or 2<sup>2</sup>, as he thinks fit, according as he finds more *finesse* at one or other of these stages. Generally, with “*bon cru*” products, the best quality is at 60°-70° (alcoholic). Brandy, very mellow, and matured by automatic reboiling and well charged with œnanthic perfumes, flows through the safe S. A tap fixed at the entrance to the worm safe enables the flow to be regulated in a very precise manner. When the brandies have a taste of *marc*, which it is desired to eliminate, it is drawn from the upper tap 2, whilst by the lower tap 2<sup>2</sup> a slight extraction is made of

1 to 2 per cent. containing volatile oils. It is there they concentrate. The plant is provided with a steam regulator.

In large establishments where the supply of marc to be dis-

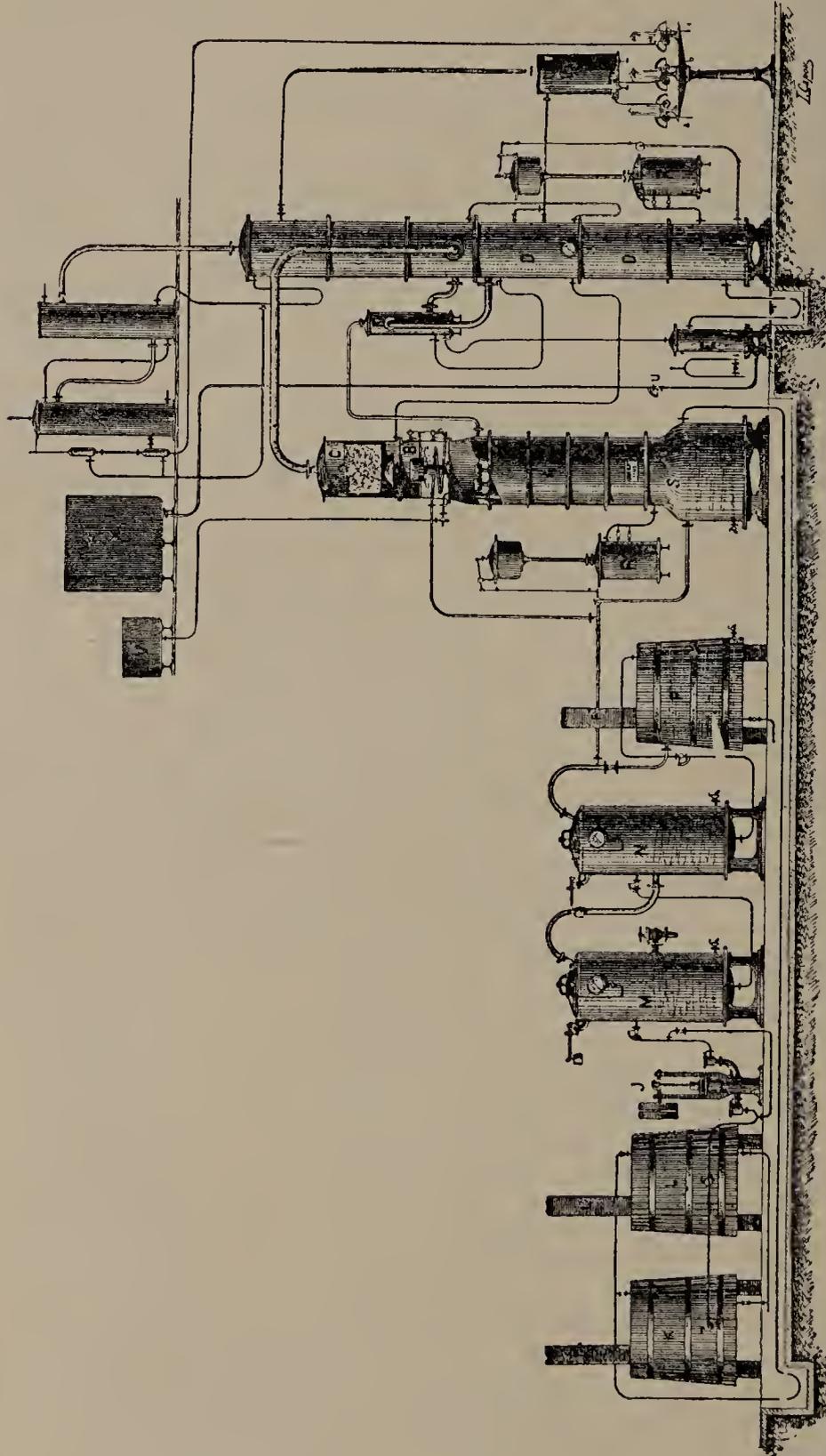


FIG 30.—Plan for producing 96 per cent. rectified wine alcohol and recovering cream of tartar by multiple effect evaporation, the steam used for distillation coming from the multiple effect apparatus (E. BARBET).

tilled is abundant, four or five maceration vats are used (according to the richness of the marcs), so as to form a battery. Each has a false bottom 4 inches above the true one, and it is upon this false bottom that the marc is run. A wide pipe leads from the

top of one vat underneath the false bottom of the following one. This pipe is, moreover, open on the top, so as to allow of water being run in direct. The maceration liquid passes consecutively through each of the vats, in which it remains some hours, and issues from the last as "piquette" (sour wine). The displacement of the liquid from one vat into another is made by running water into the false bottom of the first vat. The maceration liquid driven from below upwards flows from the top part of the vat, and enters under the false bottom of the following one, which discharges into No. 3, and so on to the last. After a number of macerations corresponding to the number of vats, the exhausted marc of the first is withdrawn and replaced by fresh marc. This vat now becomes the last of the series, that from which the sour wine is withdrawn, No. 2 becoming the first. Afterwards No. 2 will be the last, and No. 3 the first, and so on. This methodical maceration yields good results, furnishing high-strength piquette and completely exhausting the marcs. The piquette is pumped to D E F, and continuously distilled.

Some wines, owing to defective fermentation, as already mentioned, are inferior in quality, which ordinary distillery plant is powerless to remedy, special measures being necessary to cope with the difficulties they present. It is more especially the acid gases that cause trouble in the distillation; sulphurous acid and sulphuretted hydrogen ascend the column and combine with the spirit to form nauseous ethers, which it is impossible afterwards to destroy or eliminate. The remedy, therefore, consists in preventing these gases from rising into the upper stages of the column. It is necessary, in fact, to act upon the alcoholic vapours at the point where they are disengaged from the wine, and to free them from their impure gases by appropriate cleansing, so that they do not ascend upwards to the high-strength plates. This washing of the vapours is effected in special scrubbers termed the reagent vessels. In the first B, they pass over fragments of marble to free them from sulphurous acid and acetic acid; in the succeeding vessel C the vapours bubble through a solution having no action on the spirit itself, which absorbs sulphuretted hydrogen and ammoniacal compounds. In fact, this vapour

cleansing is done in apparatus similar to the Woolf's bottles used in chemical laboratories. If the reagent to the latter vessels are outside the column, the cleansed vapours return. A supplementary condenser is then used in connection with these reagent columns to force down the amylic vapours so abundant in such wines into the lower part of the column. The right-hand portion of Fig. 30 shows an arrangement intended to make from wine in a single operation rectified 96 per cent. alcohol. It will at once be observed that this apparatus may be placed in a low building, so that it meets the wants of the greater number of distillers in the South of France. When it is a question of making odourless alcohol, and not a vinous brandy, the perfume of which masks certain impurities, it is indispensably necessary to eliminate all unpleasant elements. No precaution must be neglected, because the wines used for alcohol making are generally of low quality. A is the wine exhaustion column, surmounted by reagent vessels. B contains salts in solution. C contains marble chips. From there the purified vapours pass to the rectifier proper K. Underneath the column K, D D<sup>1</sup> intended to specially purify the last runnings. The wine is first heated in the recuperator E by absorbing the heat from the residual liquor issuing from the column D D<sup>1</sup>. It then passes into the tubular vessel F, where the heating is completed. Owing to the chemical purification of the alcoholic vapours, pasteurisation expels the first runnings products. At the same time, the elimination of the odours of the marc by the special column D D<sup>1</sup> is perfect, a 96 per cent. "bon gout" alcohol, quite fresh and pure, being obtained. The vinasse is run alternately into K and L, and the tartaric acid saturated by potassium carbonate. The clarified liquor is pumped into M N, heated by live steam from the boiler. No. 1 steam heats No. 2 (N), the liquid in which comes from No. 1; the steam disengaged from the second vessel N (No. 2) is used to heat both the distilling column A by the coil S and a final wooden vat P fitted with a steam coil, into which the vinasse from N (No. 2) is allowed to run continuously. After this final concentration, the vinasse is sent to the crystallisers. The concentration is such that almost the whole of the bitartrate of potash is obtained. In

a distillery on a large scale, the utilisation of by-products may be carried a stage further, the concentrated liquor thus obtained being utilised to extract the glycerine. The mother liquors from the cream of tartar contain a sufficient quantity of glycerine to render the rational extraction easy and economical. Such work entails the addition of special supplementary plant; in particular, plant for the production of superheated steam and for distillation *in vacuo*; but the product obtained being of great value, the capital sunk yields a very profitable return, which distillers should not overlook (see also Chap. III., p. 79).

*Fruit distillation on a small scale.*—In Great Britain we are accustomed to regard the distillation of fermented liquors as only profitable when conducted on a large scale, and therefore lose sight of the fact that abroad, more especially in France, distillation is carried on by small farmers and proprietors on what is often a miniature scale. This they are enabled to do by less restrictive Excise laws, which permit a farmer or market gardener to distil his own produce with a minimum amount of trouble. An idea of the simplicity of these restrictions may be gathered from the fact that portable stills are moved about the country propelled sometimes by hand, sometimes by horses, sometimes by automobile, all catering for work from the country growers of horticultural produce. The distillation is carried on in the open air. No fruit is wasted in France, it is distilled. In Great Britain, in seasons when there is a glut of fruit, many tons of it are wasted or sold at a loss in districts like the Vale of Evesham, which under a wise and economical administration of our Excise policy would be distilled. Again, it would obviously be a wise measure if jam manufacturers were allowed to distil for the production of industrial alcohol fruit which reaches them in a state unfit for human food. This would be a special advantage to jam manufacturers who grow their own fruit, and who deserve every privilege that can be granted them. The French fruit-grower has no interest in despatching bad fruit to market, he can distil it and utilise it to better profit than by accepting the poor price it would fetch as damaged fruit.

Figs. 31, 32 show a small portable fire-heated still, one of many

such kinds used in France. The liquid to be distilled is introduced into the body of the still A placed in the wrought-iron furnace B. The still head G is fixed in its place by means of bolts Z. The extremity *m* of the flexible tube F is screwed on to the still head, then the condenser R being filled with water, the furnace fire is made up, and the noise of the liquid boiling in the still is soon

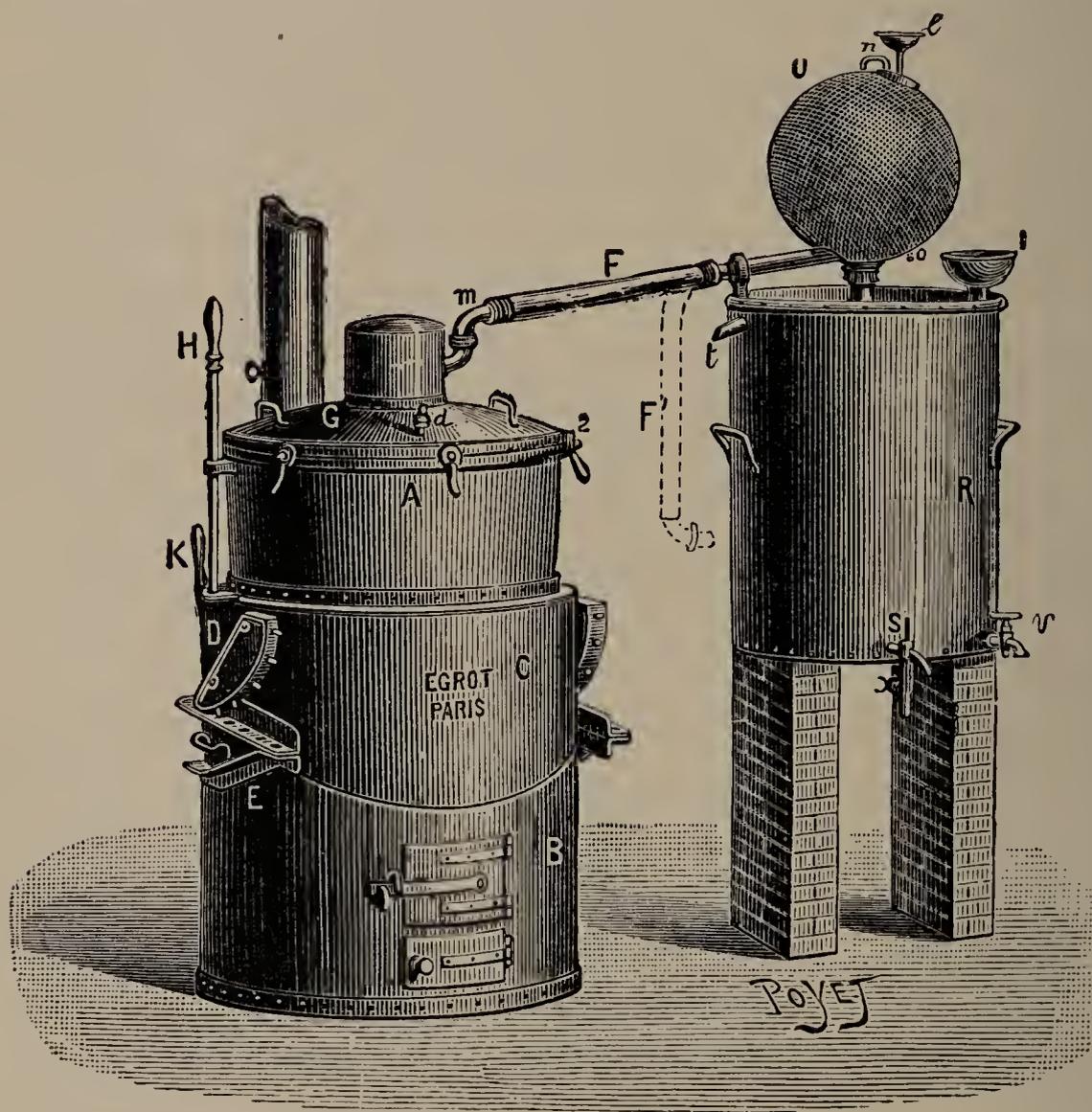


FIG. 31.—Fire heated portable tilting still for small scale distillation of wine, fruit, etc. A, body of still; B, wrought-iron furnace; C, part of furnace fixed to body of still; D, cam; E, rail on which D rolls; G, still head; *d*, screw box; Z, bolt; *m*, goose neck joint; F, goose neck during tilting; H, handle; K, lever bolt; U, spherical rectifier; *l*, water funnel; R, condenser; I, funnel; V, discharge tap; S, worm safe; *x*, test-glass (EGROT and GRANGÉ).

heard. When the distillation commences is easily ascertained by following the course of the hot vapours by running the hand along the goose neck. A small stream of water is run into the funnel of the rectifier U. Then, when the spirit reaches the mouth of the worm at S, water is run into the funnel I to cool the worm contained in the tank R. The flow of water into the rectifier is

regulated according to the alcoholic strength of the product to be distilled and the strength desired in the distillate. Towards the end of the distillation, when the distillate marks less than 40° G.L., the latter is collected apart and the heat is increased. The feed of water into the rectifier is therefore stopped. The distillate collected apart is repassed through the still with the next lot. If rectified spirits are not required, or aromatic waters are distilled, water is not run into the rectifier, the still then working like an ordinary still. On the completion of the operation the still head is taken off, the body of the still emptied and cleaned with the right hand, the handle H is drawn forward by removing the bolt K with the left hand, the cam D attached to the body of the still rolls on the projecting rail E, and the still advances, tilting itself towards the horizontal in so doing. To raise it, the handle H is lifted without touching the bolt K, which shuts automatically.

This form of still is suitable for distillers, spirit dealers, dispensers, chemists, perfumers, amateurs, etc. For distilling semi-solid matters, metallic baskets may be used to prevent burning owing to contact with the heated surface.

The still head, when taken off, forms a copper pan very useful in agricultural work for the heating of milk, the manufacture of cheese, the melting of sugar, the heating of water, the manufacture of cream of tartar, the cooking of food for cattle.

Morin (*Comptes Rendus*, 105, 1019) distilled 92 litres of genuine cognac made in 1883 from Charente Inferieure wine in Claudin and Morin's apparatus. The first runnings contained the more volatile substances, the second fairly pure ethyl alcohol, the third the higher alcohols. The residue, mainly water, was tested for free acids—*isobutyl alcohol* and glycerin. The first three portions were then fractionated, 5 litres of light alcohol, 55 litres of pure ethylic alcohol, and 3.5 litres of higher boiling compounds were obtained. The latter portion had a strong odour of fusel oil and a burning taste. The residual water was added to that previously obtained. The fractions were then redistilled from Le Bel and Henninger's apparatus. The fusel

oil fraction, which after dehydration by potassium carbonate weighed 352 grammes, yielded :

	Grammes.
Water . . . . .	7
Ethyl alcohol . . . . .	130
Normal propyl alcohol . . . . .	25
Isobutyl alcohol . . . . .	6
Amyl alcohol . . . . .	175
Furfurol . . . . .	2
Wine oils . . . . .	7
	} 352

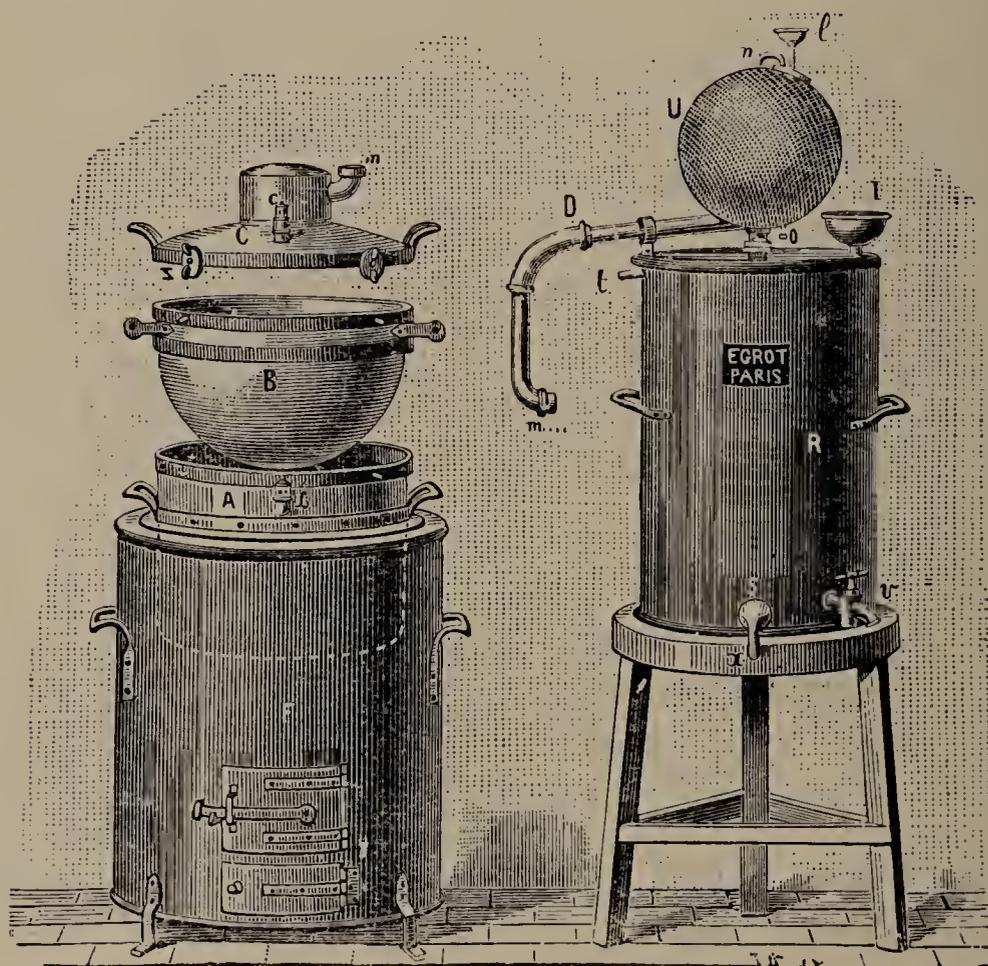


FIG. 32.—Egrot's still, with water bath for various purposes (EGROT and GRANGÉ). A, flat-bottomed pan capable of being used for various purposes ; B, still body, may be used as round bottom pan ; C, still head. This plant may serve as :—

- |  |  |
|--|--|
| 1. Ordinary still . . . . .              | } For the distillation of aromatic liquors and spirits of 60°-70° G.L. at the first operation. |
| 2. Still, with water bath . . . . .      |  |
| 3. Round bottom pan . . . . .            | } For the manufacture of syrups, pastes, pomades ; and for the concentration of all products.  |
| 4. Flat-bottomed pan . . . . .           |  |
| 5. Double pan, with water bath . . . . . |  |

The water contained a little acetic and butyric acids and a small quantity of a viscous liquid, which distilled undecomposed under diminished pressure, and appeared to consist of isobutyl alcohol and glycerin. The residue contained tannin and other

principles extracted from the wood. Column I. of the following table shows the compounds contained in 100 litres of the cognac ; column II., those contained in the distillate obtained in the same way by the fermentation of 100 kilos of sugar.

	I.	II.
	Grammes.	Grammes.
Aldehyde . . . . .	traces.	traces.
Ethyl alcohol . . . . .	50,837.0	50,615.0
Normal propyl alcohol . . . . .	27.17	2.0
Isobutyl alcohol . . . . .	6.52	1.5
Amyl alcohol . . . . .	190.21	51.0
Furfurol aldehydes . . . . .	2.19	0.0
Wine oil . . . . .	7.61	2.0
Acetic acid . . . . .	trace.	
Isobutyl glycol . . . . .	2.19	
Glycerin . . . . .	4.38	

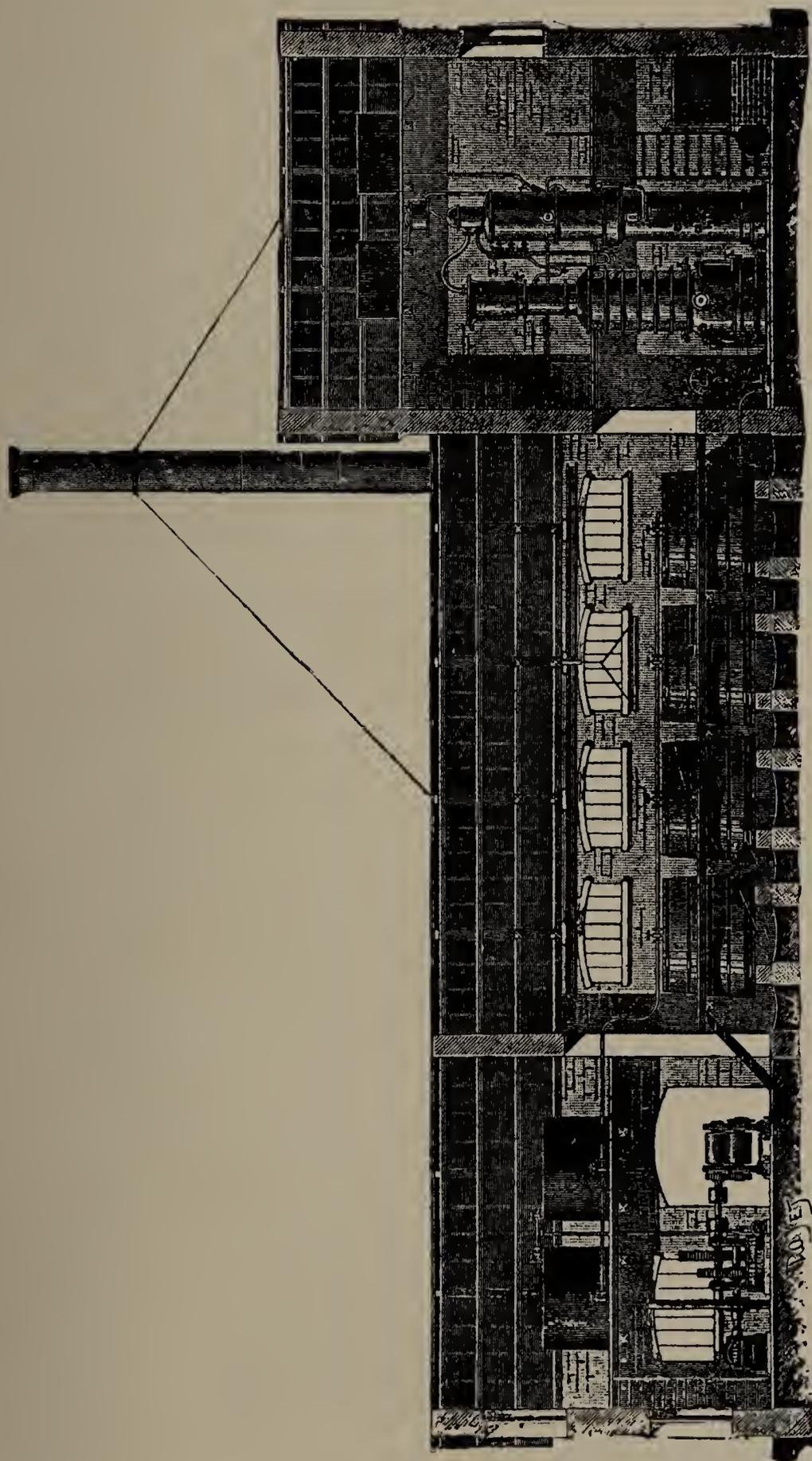
Butyl alcohol was absent. Furfurol was detected directly by the addition of aniline to the cognac, a red coloration being produced in the presence of acetic acid.

## CHAPTER VII.

### THE MANUFACTURE OF ALCOHOL FROM THE SUGAR CANE AND SUGAR-CANE MOLASSES.

THE sugar cane is the main source of alcohol in tropical countries, whether it be manufactured by direct elaboration from the cane juice (vesou) or by fermentation of molasses. Like the must of the grape, cane juice (containing as much as 12-16 per cent. of sugar) enters into fermentation spontaneously, but often in a much more energetic manner. An inferior quality of spirit is made from molasses mixed with the skimmings and washings of sugar pans. When molasses is diluted with twenty times its weight of water, and the mixture has cooled to 78° F., if one-twelfth its weight of yeast be added, fermentation will speedily ensue, and an ardent spirit will be generated, which, when distilled, has none of the aroma of rum produced from cane juice, the odorous products having been dissipated at the high temperature employed in the production of molasses. This product is called *Tafia* in the West Indies, *Caña* in the Argentine Republic, *Cachaca* in Brazil. When cane juice is to be used for making rum, it is extracted by single or double pressure in mills, in the same way as for sugar manufacture (Figs. 33, 34). It is generally strained to remove bagasse débris, and then fermented. The natural ferment is found on the exterior waxy surface of the cane. The cane-sugar ferment differs from European ferments, the alcoholic ferments of Venezuela being much smaller than European species. In form they approach an octagon, especially the protoplasm. They require for their development and propagation in good condition a temperature of 30° C. (86° F.), otherwise fermentation is very slow and tedious. Cooling is never done at zero ; injurious secondary fermentation seizes the

top very easily, whilst at 35° C. (95° F.), the temperature which suits this alcoholic ferment best, fermentation occurs very



Distillery.

Fermentation tuns.

Molasses mixers.  
Sugar mills.

FIG. 33 — Rum Distillery (EGROT and GRANGÉ). Front view of interior.

rapidly and in good condition with “chutes” at zero. The alcoholic ferment can withstand strongly acid liquors. Media

in which European ferments would be paralysed, the Venezuelan ferment can stand very well. The reproduction of the alco-

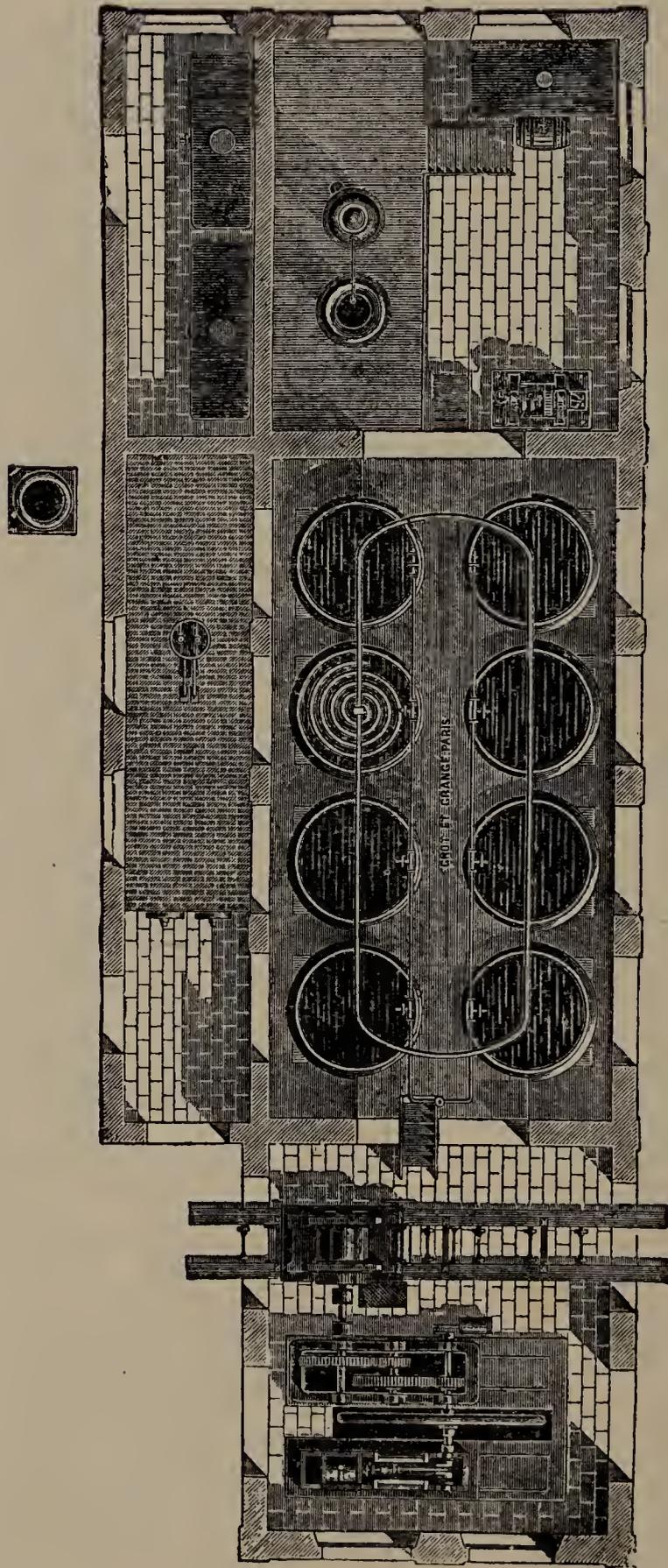


FIG. 34.—Rum distillery (EGROT and GRANGÉ). Ground plan of Fig. 33.

holic ferment is effected at a temperature between  $30^{\circ}$  and  $36^{\circ}$  C. ( $86^{\circ}$ - $96.8^{\circ}$  F.). Barbet points out that some slight exception must be taken in regard to Delafond's remarks on acidity.

European ferments, he says, do not grow well in presence of mineral acids, but they tolerate certain organic acids very well. Bakers' yeast ferments with great activity in media containing lactic acid, equivalent to 6-7 grammes of sulphuric acid per litre (*i.e.*, 6-7 lb. of sulphuric acid per 100 gallons), and wine ferments in certain years contain tartaric acid exceeding the equivalent of 7-9 grammes of sulphuric acid per litre (7-9 lb. per 100 gallons).<sup>1</sup> The hotter the wash up to a certain point, the more need it has of some acidity (preferably organic) to protect it against injurious ferments. This law holds good to a greater extent with cane juice than with European washes and musts, because non-acidified cane juice is a nidus for bacterial and mould growth.

Sometimes it is a film or web of mycoderma which forms on the surface of the vat, so matted and consistent that it can be lifted in enormous strings without breaking. Sometimes viscous fermentation sets in, against which disease the only remedy is high acidity, falling little short of that which would be injurious to the proper ferment itself. In cane juice stringing like thickened oil, says Delafond, I have found the viscous ferment under the microscope forming a compact mucilaginous mass, which, thinned down with distilled water, formed a froth or "head," and it was found in pairs when detached. The viscous ferment does not develop in strongly acid media. The addition of sulphuric acid stops viscous fermentation, even when in active condition, and as the organism acts on levulose, this ferment is not propagated until the alcoholic ferment has commenced the work of splitting up the sugar. The *acetic* ferment may be combated by avoiding the heating of the juice, and by realising (as near as possible) an *anaerobic* fermentation. In regard to other bacteria, the acidity of the juice acts best. The best way of securing cheap acidity in the mashes is to utilise the spent wash from previous fermentations. Between the first and second pressure of the cane the bagasse is moistened *with water*. It would be more rational, says Barbet, to cause the bagasse to *reimbibe the spent wash*; this, however, is a question of trial. Another way of checking too energetic fermentation is the rational use of antiseptics,

<sup>1</sup> Chapter VI., p. 112.

refrigeration of the wort, previous sterilisation, etc. The refrigeration of the wort is not often practicable owing to scarcity of fresh water, if carelessness, bad air, polluted water, and general laxity are the order of the day, no care being taken in the manufacture and preservation of the molasses in the boiling-house, and it is not thought worth the trouble to exercise any microscopical or chemical supervision—for the alleged reason that the whole management of a distillery is so simple that it may be left in the hands of any overseer or driver—then the very finest distillery plant might be set up and would prove a failure financially, even though all the exhaust steam was utilised and the very best rectifying apparatus employed. Under such circumstances it cannot be a matter for wonder that the percentage yield—which is, and will always remain, the only figure for comparison—instead of being 80, or more, goes down to 60, or less, even though the percentage of wash attains a very high (but fallacious) figure, and the yield from the molasses appears to be satisfactory.

*Fermentation.*—The sediment from molasses wash, after fermentation, is seen, under the microscope, to consist of innumerable cells, smaller in size than those of the common beer-ferment (yeast). They are round and shining, mixed with small granules and separate from one another, not arranged in masses or in the form of long bands. Rum ferment remains unchanged so long as the nutritive matter in which it grows remains the same. If, however, it is put into a liquor containing more sugar than the original wash, or into solutions of starch and dextrine, there appears in about forty-eight hours a dirty kind of mycelium, the threads of which occupy the whole of the liquor. From this mycelium the ferment is easily reproduced when it is put back into ordinary wash. The mycelium is always present along with the rum ferment in all fermented wash in the distillery, particularly when fermentation has proceeded slowly or the wash has been much exposed to the air. The rum ferment not only differs in structure from beer ferment, but its products and action are also of a special nature. (1) The rum ferment has the strongest effect at a temperature of 30° to 35° C. (2) It is very sensitive

in regard to cold. (3) At 18° to 20° C. the fermentation slows down, the acidity increases, and the yield of alcohol diminishes. (4) The degree of concentration of the wash has a distinct influence on the vegetation of the ferment. (5) A solution of saccharose of 18 to 19 per cent. appears to give the best yield. Pure sugar is here referred to ; the statement does not apply to liquids such as molasses. (6) The rum ferment, both in its fermentative form and that of mycelium, separates an enzyme which converts saccharose into invert sugar (Stade).

In Jamaica and some of our colonies, says Ure, fifty gallons of spent wash or lees are mixed with six gallons of molasses, thirty-six gallons of sugar-pan skimmings, a substance rich in aroma, and eight gallons of water, in which mixture there is about one-eighth of its weight of solid saccharose. The fermentation is seldom complete in less than nine days, and most commonly it requires from twelve to fifteen, the period being dependent on the capacity of the fermenting tun and the quality of its contents. The liquid now becomes clear the froth having subsided, and few bubbles of gas are liberated from it, whilst its specific gravity falls from 1.050 to 0.992. The sooner it is subjected to distillation following this period the better, to prevent loss of alcohol by the acetic fermentation, an accident very liable to supervene in the sugar colonies. The crude spirit obtained from the large still at the first operation is rectified in a smaller still. About 114 gallons of rum, proof-strength, specific gravity 0.920, are obtained from 1200 gallons of wash. Now these 1200 gallons weigh 12,600 lb., and contain nearly one-eighth of their weight of sugar = 1575 lb., which should yield nearly its own weight of proof spirit, or in bulk  $\frac{1575}{9.2} = 171.2$  gallons,

whereas only 114 are obtained, proving (according to Ure) the process to be conducted in a manner far from economical, even with every reasonable allowance. Ure, by his own experiments on the quantity of proof spirits obtainable from molasses, found that one gallon of sweets should yield one gallon of spirits, and hence the above 1200 gallons should have afforded the same bulk of rum.

Dr. Ure also quotes an experiment he made as follows:— 150 lb. of West Indian molasses were dissolved in water and mixed with 2 gallons of yeast weighing exactly 20 lb. The wash measured 70 gallons and had a specific gravity of 1.0647 at 60° F. In two days the gravity had fallen to 1.0055, in three days to 1.0022, and in five days to 1.001. The temperature was kept at from 80° to 90° F. during the last two days by means of a steam-pipe, to favour the fermentation. The yield of spirits was 11.35 gallons. Now, 150 lb. of the above molasses were found to contain of solid matter, chiefly uncrystallisable sugar, 112 lb., so the yield was in accordance with the above statement.

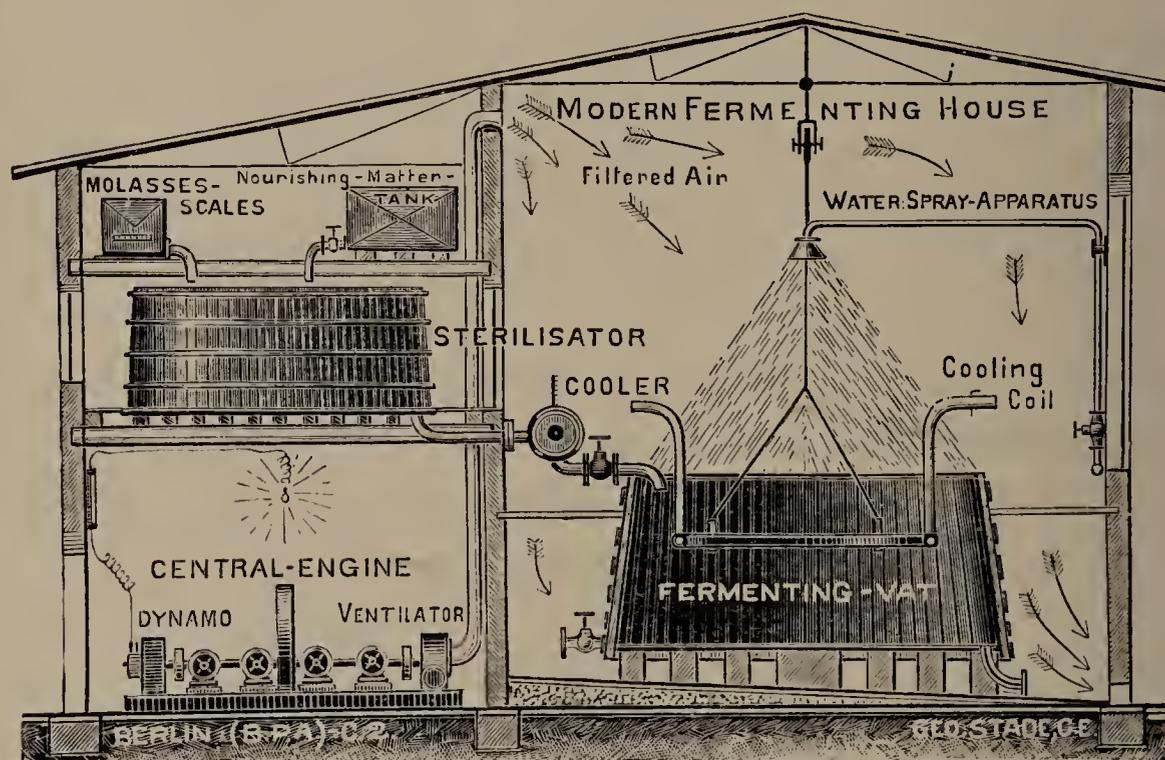


FIG. 35.—Modern fermenting room (STADE).

More than one million tons of cane-sugar molasses are produced annually, sufficient to produce 132,000,000 gallons of spirit of 60 per cent. strength. Not a sixth part of it is used for this purpose, because, especially in the French colonies, they have discontinued to a large extent the manufacture of rum, or Tafia as they call it, and even, it is alleged, run their molasses into the sea or into streams.

*Composition of molasses.*—The analysis of molasses is complicated by the co-existence of several sugars and other similar substances, saccharose, dextrose, levulose, mannose, glucose,

raffinose, caramel, etc., the choice and ratio of the defecation agent greatly influencing the product.

TABLE XXIII.—ANALYSES OF CANE-SUGAR MOLASSES.

	Java (Prinsen Geerligs).		Average.	Thomson Average Composition of Molasses from Different Sources.
	Extreme Limits.			
	Min.	Max.		
Crystallisable sugar Calculated by inversion	5.7	44.3	35.3	26.34
Reducing substances cal- culated as glucose .	18.8	39.4	27.6	28.13
Dextrose . . . . .	8.2	22.9	14.2	—
Levulose . . . . .	5.6	16.5	13.4	—
Total ash . . . . .	4.38	9.04	8.08	8.26
Water . . . . .	17.1	42.3	19.1	26.60
Organic impurity . . . . .	—	—	11.23	10.67
Density . . . . .	1.315	1.481	1.481	—
Acidity as acetic acid . . . . .	—	0.50	0.19	—
Real purity in ratio to saccharose . . . . .	9.8	53.7	43.6	—
Nitrogenous matter . . . . .	—	—	—	2.21

The analytical determination of the different sugars in molasses is no safe guide as a basis for estimating the amount of alcohol which it will yield. There is no satisfactory method available for this purpose, except that of Dr. Ure, viz., a comparative fermentation test in the laboratory.

ANALYSES BY BARBET OF TWO MOLASSES FROM EGYPT.

	No. 1. Per Cent.	No. 2. Per Cent.
Crystallisable sugar (Clerget's method)	39.10	39.60
Reducing sugars . . . . .	19.33	16.90
Total sugar calculated as uncrystallisable . . . . .	60.48	58.53
Total by direct inversion of the molasses . . . . .	59.96	58.56

Judging from these analyses, No. 1 ought to yield more alcohol than No. 2, but direct experiment led to rather unlooked-for results. No. 1 yielded 27.59 c.c. of absolute alcohol per 100 grammes of molasses, whilst No. 2 gave 31.03, or 3½ per cent.

more. The direct fermentation method is therefore indispensable. The acidity of the molasses is first determined so as to calculate the amount of sulphuric acid to be added to get a wash with 2 grammes of acidity per litre before fermentation. Then a sterilised 2-litre flat-bottomed flask is taken, tared, and 200 grammes of molasses weighed into it. Water up to nearly 1 litre total volume is added to make the fermentation test on the basis of 200 grammes of molasses per litre. The sulphuric acid, calculated to amount to 2 grammes at least, is added, and the liquid boiled for ten to fifteen minutes. "Denitration" sterilisation and slight complementary inversion are thus simultaneously effected. The mouth of the flask is plugged with cotton-wool, and cooled as rapidly as possible to 30° C. (86° F.). Whilst this has been going on, 10 grammes of good yeast fresh from the bakery is dissolved in a little sterilised water, and run into the flask with a little rinsing water, about 1050 grammes of liquid, which makes exactly a litre, since the density is about 1.050. The flask is weighed exactly, including cotton-wool plug, and the whole brought to 28°-29° C. (82.4°-84.2° F.). The progress of the fermentation is watched by weighing at intervals of six hours. When fermentation is finished the weight remains constant. The volume of the liquid is then determined, to ascertain to what exact weight of molasses the volumes drawn off for distillation, polarisation, and determination of acidity will correspond. Suppose that the CO<sub>2</sub> is removed by insufflation, 1020 grammes of liquid remain. Cool the liquid to 15° C. = 59° F., and take the density with an exact hydrometer, say 1018. The real weight in vacuo of a litre of this fermented liquor is 1018 × 0.99916, this latter coefficient being the weight of water at 15° C. In vacuo 1020 grammes weigh 1021.1 grammes, air displaced by the liquid, less air displaced by brass weights. The real volume of the wort is—

$$v = \frac{p}{d} = \frac{1021.1}{1018 \times 0.99916} = \frac{1021.10}{1017.1} = 1004 \text{ c.c.}$$

The 500 c.c. taken for distillation correspond to 200 grammes ×  $\frac{5.00}{10.04} = 99.601$  grammes of molasses. To get the exact alcoholic strength, neutralise exactly the 500 c.c. of wort, for without that

the volatile fatty acids distilled would cause a slight loss of alcohol, continue the distillation to 200 c.c. The two molasses from Egypt gave the following results after fermentation in the laboratory :—

Yield in alcohol per 100 grammes . . . . .	27.59	31.03
Density on cooling wash to 15° C. . . . .	1.034	1.020
Polarisation . . . . .	0.0	0.0
Acidity per litre . . . . .	1.93	2.10
Unfermented sugar per litre . . . . .	9.55	4.64
Sugar brought to 100 grammes of molasses	3.72	1.82

With pure ferments it is not impossible to realise on the large scale the results obtained in the laboratory, but the molasses have to be treated to eliminate injurious ferments as well as the bacteria with which they are infected. In most colonies, molasses fermentation is often completely unattended. Fermentation is spontaneous, *i.e.*, the germs in the air fall into and multiply in the liquid. At the end of the season, since germ spores are rare in the air, rule-of-thumb processes, apparently justified by strange theories, are resorted to by the foremen. Sulphuric acid, lime, or sal-ammoniac are capriciously added. These may be useful in modifying the composition of the wash, but they do not supply ferment. Again, bakers' yeast thinned down is added, which yields more lactic acid and foreign germs than real ferment. The best plan is to take the ferments from the cane. Fresh green bagasse is placed in a previously sterilised vat, which is filled with dilute sterilised molasses. Fermentation soon becomes active, and a leaven with an indigenous ferment adapted both to the high temperature and to the composition of the culture medium is thus obtained. When the fermentation of the first vat is started, a little of its contents is drawn off to leaven the next vat, and so on. Although better than spontaneous fermentation, this method still leaves much to be desired, because infection of the wash gradually becomes more and more accentuated. It is usual to make very large dilution vats, so as to produce the wash of the same average strength, and to make sufficient wash for the night-work. It is the fermentation ante-chamber, and there the wash left to itself is contaminated,

especially since the vat is rarely emptied, and still more rarely washed and sterilised. The wash ought to be sent to the fermentation tuns as soon as it is thinned down. It is not difficult to design very simple and compact arrangements to mix instantaneously and continuously water and molasses and to regulate the two feeding taps so as to obtain a liquid of the right density. Such plant is in use in Austria to dilute the crude phlegm before filtration or rectification.

In spite of its concentration, cane-sugar molasses often decomposes to such an extent in the casks as to blow out the bungs and even the bottoms of the casks, with disengagement of disagreeable smelling gases. Such molasses are often so acid that on dilution they yield a wash with an acidity of 3 grammes per litre. All volatile fatty acids are antiseptic and retard fermentation; they may even stop alcoholic fermentation and favour injurious secondary fermentations, acetic, butyric, etc. The molasses should then be heated or treated with sulphuric acid, and air injected into the boiling mass to expel the volatile acids. It is not a "denitration" as in beet distilleries, but a sterilisation with an equipment identical to that used in the "denitration" of beet molasses. The most simple process is Barbet's continuous sterilisation process. In this the copper boiling and aeration pan is of such a capacity that the molasses, previously diluted to 25°-30° Baumé, remains there for over a quarter or an hour. The molasses before entering is methodically heated by the issuing boiling molasses, so that the plant requires little or no steam. Next to the heat recuperator a tubular refrigerator brings the sterilised wash back to a proper heat for fermentation. It may thus be diluted to 9° or 10° Baumé for the fermenting tuns. This process gives good results, but it is better to adopt in its entirety the processes of the *Société anonyme des ferments industriels* (Brevets Barbet), particulars of which follow. The molasses are diluted, as described above, to about 30° Baumé, and, if need be, a small dose of sulphuric acid is added so as to liberate any fatty acids. The dilution to 30° is done in two alternate wooden or copper vats A. At the exit a beck with a ball valve *b*, and a tap with graduated dial, enables

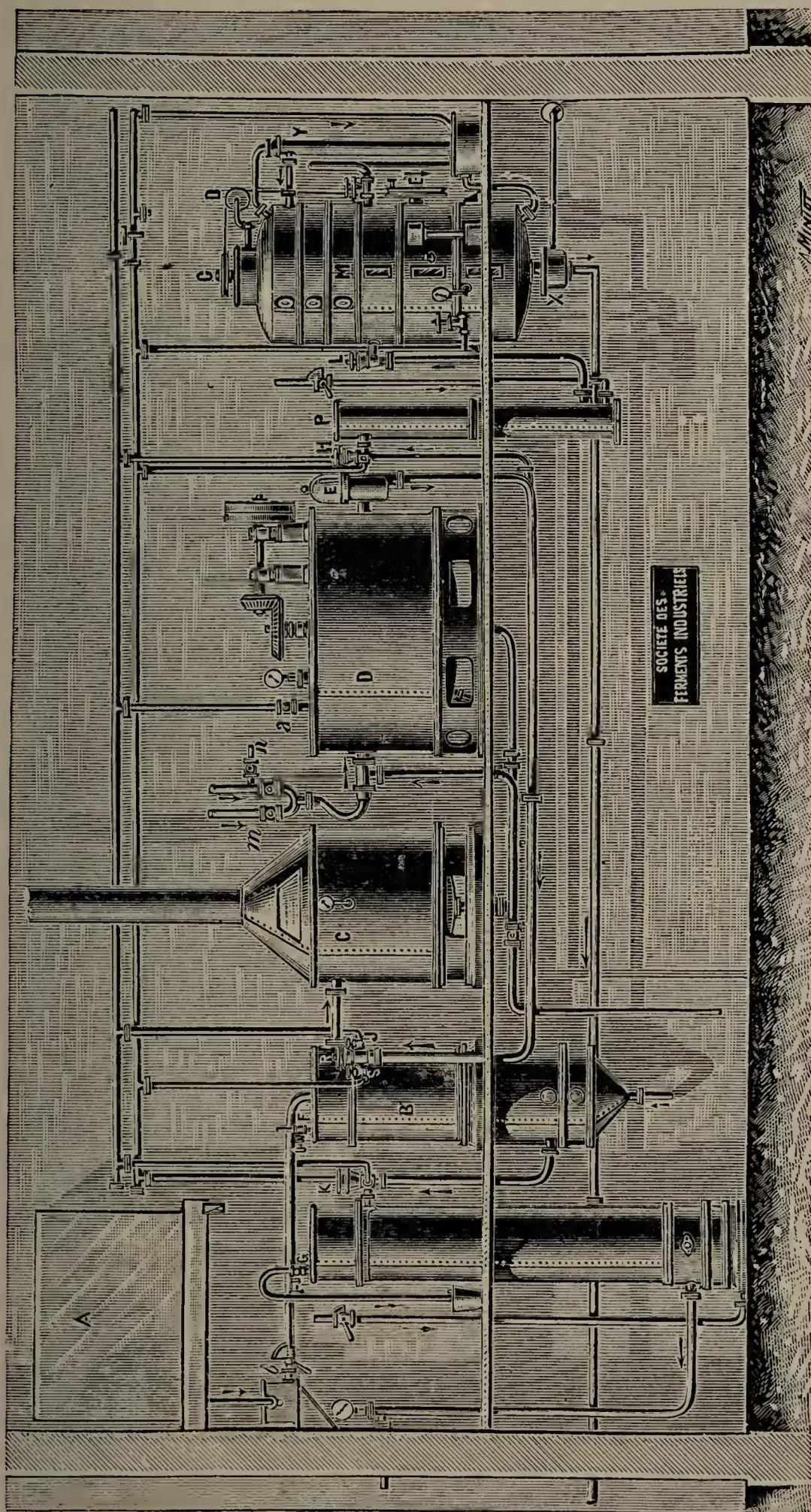


FIG. 36.—Plant for aseptic fermentation of cane-sugar molasses (E. BARBET). A, tank to dilute to 28-30° B.; b, regulating feed tank; B, forewarmer; C, continuous steriliser; D, mixer to dilute with hot water or hot spent wash; m, spent wash tap; n, hot water tap; E, test-glass; H, regulating valve; M, pure yeast producer (Fig. 2); P, cooler for yeast apparatus; V, refrigerator to left of B.

the molasses to be fed into the heat recuperator in a very regular manner. They pass into the inside tubes of the heat recuperator B, and enter almost boiling into the vessel C, made of copper. An open steam-pipe brings them to the boil, and air injected through a perforated pipe stimulates the elimination of the liberated fatty acids. The disengaged vapours are led into a tubular refrigerator, where they are condensed, yielding an acid liquid with a more or less pleasant odour. When the molasses is of good quality, the perfume of the acid water is not unpleasant; it is then utilised either to mix it with the fermented wort or with the crude rum of the first distillation. If the smell be bad, there is no need to condense the vapours, and the utility of continuous sterilisation is proved by the elimination of the bad smelling odours. Without this expulsion all these products left in the wash would be disengaged on distillation, thus depreciating the good quality of the rum. As the molasses come from the steriliser C, the boiling liquid, which is at 30° B, runs into a supplementary dilution pan D before being sent to the recuperator B, and this supplementary dilution is done not with cold but with very hot water from the condensers, and even with a little spent wort as it comes from the distilling column. The three liquids, sterilised molasses, hot water, and spent wort, enter into the apparatus through a mixer *m* and *n*. A special arrangement shows whether the three ingredients are regulated in the right proportions for fermentation.

According to circumstances, the diluted wash is at a temperature of 80°-85° C. (176°-185° F.). A perforated steam-pipe enables the temperature to be raised to 95°-97° C. (203°-206°·6 F.) if desired, but it is well known that the sterilisation temperature required is lower the greater the amount of free acid in the wash. Thus natural wines are pasteurised at as low a temperature as 67° C. (152·6° F.), because their tartaric acidity is such that suitable sterilisation is arrived at at this temperature. With molasses washes which have more than 2 grammes of acidity per litre a temperature of 80° C. (176° F.) is amply sufficient—at least, from an industrial point of view. At the exit tap of the dilution vat D, the capacity of which ensures for the wash a stay

of twenty minutes at a high temperature, there is a special test-glass E which indicates the density. A table is prepared of the relation between the true density of the liquid at 15° C. (59° F.) and that which it shows at the test-glass: thus, 1060 at 80° C. (176° F.) = 1.084 at 15° C. If it be desired to produce wash at 1.084 at 15° C., and if the temperature of the exit is 80° C. (176° F.), the operator ought to have a flow of density 1.060 at the exit test-glass E. From there the wash goes to the recuperator B, then to the refrigerator, which should bring it to the temperature suitable for fermentation. The refrigerator is constructed according to new rules, which reduce the consumption of water to a minimum and prevent all contamination of the wash during cooling. The wash obtained as a final result at a constant density and temperature has been twice sterilised—first at 30° Baumé, then after complete dilution. It is therefore aseptic, and is thus as well prepared as possible to yield a pure fermentation, provided it be mixed with a pure ferment. The means to be adopted to run a certain amount of spent wort into the wash have already been described. In the case of small rum distilleries the plant described above is simplified, the dilution vessel A being dispensed with, and there is then only one recipient for the steriliser C and the dilution vessel D. In it the molasses is first diluted, boiled, the boiling spent wort then added to bring the dilution to the right density for fermentation. Dilution is done by hand.

*Pure ferments for cane spirit.*—Several chemists have attempted for some years to extend the use of pure ferment; in the first place, researches were made to select strains of acclimatised ferments. But it is not enough to have a good strain of ferment, it must be in full vigour, and at the same time furnish a sufficient number of active cells to the fermenting tuns for fermentation to operate with as little delay as possible, *i.e.*, about thirty-six to forty hours for molasses wash. The difficulty in the way of almost all these attempts was the preparation of pure leaven from the ferments furnished by laboratories. Commencing with 2 or 3 litres of ferment, a first leaven of 50 to 70 litres was prepared, which was run into a second tank of 300 to 400 litres, then into a vat of 20 or 30 hectolitres, and finally it was this leaven which

served as "yeast" for the large fermenting tuns. Throughout the whole of this long series of operations it was impossible to avoid contamination, so that on a final test of the leaven it was no more pure nor more active than the pressed yeast formerly employed. Moreover, these preparations were very complicated. Barbet's equipment is much more practical, and realises in a single operation the preparation of numerous abundant ferments charged with a mass of vigorous cells. Only one pure leaven apparatus is required; this is closed and tested at a kilogramme of pressure. At the outset it is sterilised by passing in steam at 115° C. (239° F.) for several minutes, then the excess of pressure is relaxed, sterilised air is introduced, and also 8 to 10 hectolitres of wash sterilised in a vessel on one side and cooled in the refrigerator. Cold or tepid water is made to circulate in a coil in the apparatus as required, to correct the temperature of the wash and bring it to the desired point. Then the ferment "sowing" tubulure is "singed" or purified by fire, and about 1 litre of pure ferment from the laboratory is introduced and aerated briskly to actively force the propagation of the leaven. When fermentation is well started, fresh wash is run into the apparatus to fill its lower capacity. At this point Barbet's system may be brought into action. This consists in cultivating the ferment on a large aerated surface, that is to say, *en aerobiose*. Pasteur showed that highly aerated surface culture imparted to leaven quite a characteristic vigour, and even went so far as to modify the form of the cells. At the same time, it accentuates in the organism the faculty of developing spores. In a word, when a leaven is fatigued it is revived by surface cultivation in a wash in as shallow a layer as possible. Pasteur realised this in a very simple manner by putting a very small quantity of liquid in a large flat-bottomed flask. Barbet followed Pasteur's precepts, recalled to him by Dr. Calmette, by installing in the upper part of the apparatus a series of shallow copper plates on to which he forces the wash to continually ascend and spread over them. He utilises in the compressed sterilised air the force required to produce the continuous circulation of the wash, and that by emulsifying tubes, the number of which depends on the capacity of the leaven

apparatus. For instance, a reservoir A (Fig. 37) containing a liquid which it is desired to cause to ascend into B. A reverse siphon is fixed, and at the lowest point of the longest limb com-

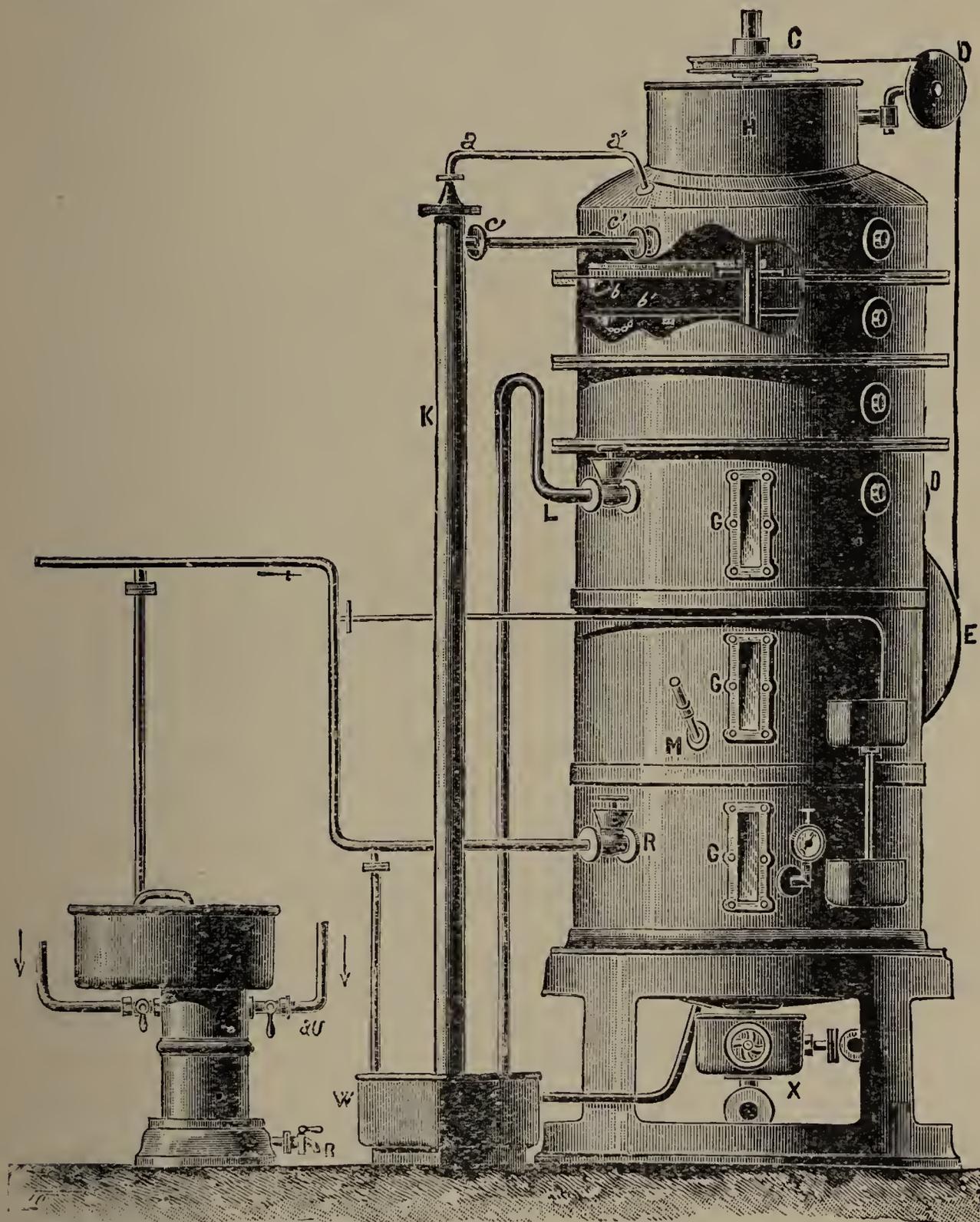


FIG. 37. — Plant for producing pure ferment for fermentation of cane-sugar molasses (E. BARBET).

pressed air is admitted through a tap. By conveniently regulating the size of the air-bells inside the pipe, a series of air-bells are formed therein—"liquid pistons," as they have been termed. If the number of these air-bells be so increased that the total

number of liquid pistons represent a column of liquid less than that of the descending limb, the liquid rises in the second limb, which causes the liquid A to ascend regularly into B. This system, called "emulsion," is used for lifting acids without the aid of pumps. Barbet has applied it to his pure leaven process so much the more profitably because the use of compressed air was already obligatory in any case. Instead of forcing the air to the bottom of the wash, a method which inefficiently utilises the oxygen of the air, Barbet applies it to produce a continuous ascending current of wash on to the upper plates, where it spreads itself out over a wide surface of liquid, dissolving therein, and revivifying the yeast cells. The pure leaven apparatus is fitted with a dial thermometer, a water coil, a gauge glass, and a safety valve. It is likewise fitted with brass brushes on each plate, so that when desirable the leaven adhering to the plates may be detached and forced to descend into the mass of leaven underneath, and from thence to the fermenting tuns. When the leaven is ripe, only some two-thirds of it are taken for the fermenting tuns, the remainder being used to perpetuate the fermentation in the apparatus, which is again filled with fresh wash, sterilised, and cooled. Fresh leaven for the fermenting tuns may thus be produced every five or six hours. All the accessories are so designed as to entirely prevent interior contamination, and in this way the leaven may be preserved pure for months. It is only at long intervals that the apparatus is emptied, cleaned, and again sterilised, to commence again with a fresh culture. Molasses alone would not be an excellent culture medium for ferment. It may be improved by the addition of different substances, ammoniacal salts, phosphates, etc., or better by adding a small proportion of maize saccharified by acid, neutralised and passed to the filter presses, or by a little maltopeptone.

*Continuous fermentation.*—The pure leaven plant just described yields a batch of leaven every six, or even every five, hours, which corresponds to four or five tuns per day. If the tuns be small and numerous, one batch of leaven will do for two or three tuns in virtue of the usual method of mixing. But it is better to work as follows. Next to the autoclave which makes the pure

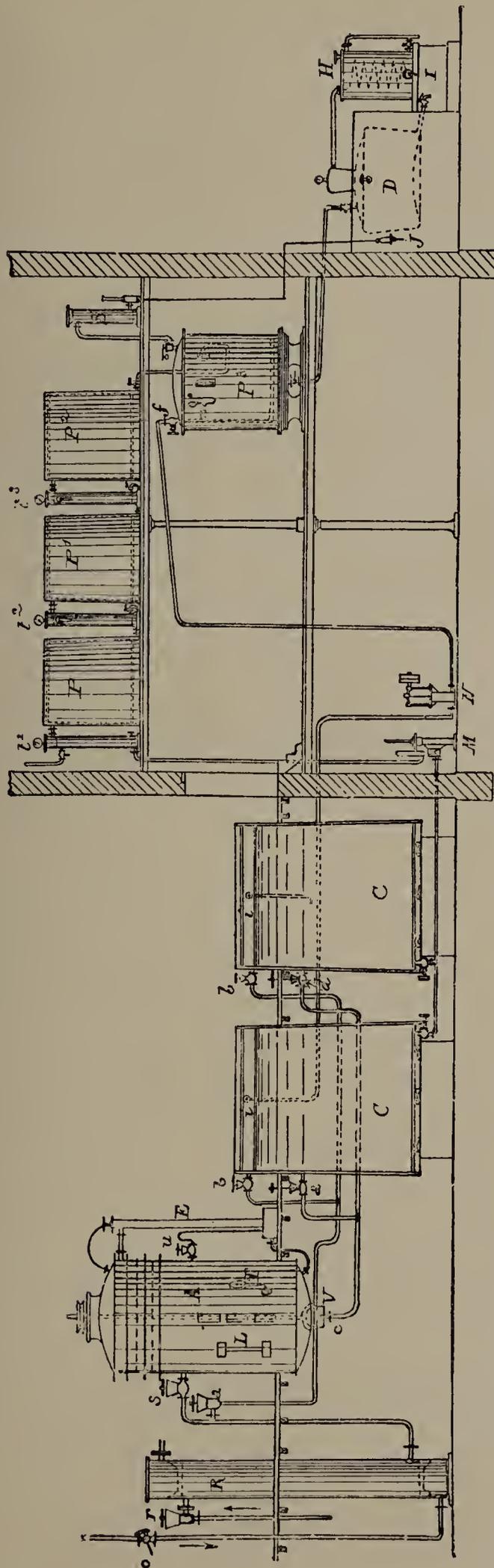


FIG. 38.—Plant for aseptic fermentation, collection of fermentation gases, ageing by prolonged heating and distillation of rum (E. BARBET). *A*, ferment vessel; *CC*, fermentation tuns; *R*, steriliser; *M*, fermented wash pump; *N*, fermentation gas pump; *i i*, fermentation gas pipes; *t t* *t*<sup>3</sup>, calorisers; *P P*<sup>1</sup> *P*<sup>2</sup> *P*<sup>3</sup>, closed vessels; *P*<sup>3</sup>, autoclave to stand pressure of CO<sub>2</sub> from *C C*; *E*, condenser for unabsorbed fermentation gas; *D*, still; *H*, condenser; *I*, test safe; *J*, test safe for perfumed liquid rum into still or distilling column (E. BARBET).

leaven, two or three large copper autoclaves are placed fitted with one or two aerobic plates and an emulsifier. These autoclaves are arranged almost similarly to the pure leaven apparatus. The second autoclave B receives at regular intervals a certain amount of pure leaven extracted from A. Moreover, it is fed continuously by sterilised molasses in the proportion of 10 to 20 per cent. of the hourly production. The wash is completely inoculated by the addition of the pure leaven, the emulsifier, and the "aerobic" treatment stimulate fermentation, and the liquid from B constitutes in its turn a more bulky source of leaven than could be extracted from A. There can thus be extracted from B continuously or at predetermined intervals as much liquid as the amount of wash fed into it. This liquid, charged with vigorous leaven cells in full fermentation, is used in its turn to inoculate the autoclave B<sup>1</sup> in proportion as the remainder of the sterilised juice is fed into it. In B<sup>1</sup> the same phenomena occur as in B. The "aerobic" action is pushed to such an extent that as the wash issues from A it possesses a sufficient number of active cells to be able to conduct in the open air the total fermentation of the sugar. The whole of the wash is thus inoculated without access of ordinary air, and strictly following the method of Pasteur. When it issues from the last autoclave it is pure, and fermentation has got a good start, owing to the number of cells which it possesses and their activity. The wash may henceforth finish its fermentation in the open air. Bacteria cannot easily develop in the liquid in a medium so thoroughly inoculated; that is a well-known fact in regard to micro-organisms. Once the fermentation is well started, contamination can no longer get any appreciable hold on the wash. The capacity of the closed vats has been reduced to a minimum, but existing vats cannot be altered. All the vats are filled one after the other without any precaution in regard to speed, mixing, or supervision. It is enough if the vats be well washed before use. When one vat is full, the next is charged, and fermentation is finished very quickly, and in twenty-four to thirty-six hours the wash can be distilled. The fermentation is so rapid that it is completed in advance of the distillation. This, however, is no

disadvantage, as it gives a certain elasticity to the manufacture, because a certain amount of interval before distillation seems to improve the perfume of the rum.

*The saline matter of cane-sugar molasses.*—*Aerobic* leavens being very vigorous, a large proportion of spent wort may be allowed to enter the wash in the tuns without fear of the accumulation of salts and organic matters arresting the fermentation. The spent wort thus becoming richer in salts, the potash salts may subsequently be profitably extracted by evaporation and incineration. Now potash is a product of some value, because all tropical countries have to import their potash from Europe and the U.S.A. Suppose 25,000 kilogrammes of molasses to be the daily turnover, yielding 1000 hectolitres of wash. All the potash of the 25,000 kilogrammes of molasses is dissolved in the residual 1000 hectolitres. But if 500 hectolitres of wash are taken daily to help to dilute the 25,000 kilogrammes of fresh molasses, the daily routine being established in that way, it follows that every day there will be sent to the potash evaporators only 500 hectolitres, containing the potash from 25,000 kilogrammes of molasses. That is obligatory, so that the output of potash may be equal to that entering the factory. Thus the wash to be burnt daily is reduced to half the preceding volume, which results in a saving of half the fuel. It remains to be ascertained whether this method of working the fermentation with a high density and a high percentage of impurities does not act prejudicially on the fermentation and the yield. Take the two Egyptian molasses, 1 and 2 previously mentioned. In the distilled vinasses dilute respectively a fresh quantity of No. 1 and No. 2 in such proportions that we get once more 250 grammes of fresh molasses per litre, *i.e.*, the highest dose used on the large scale. That gives the high initial densities of 1.109 and 1.102, say 14° Baumé, owing to the re-use of the spent wort. Using the same dose of leaven as before, 5 grammes per 100 grammes of molasses, fermentation is over 42, and good yields in alcohol are obtained.

	No. 1.	No. 2.
Initial density at 15° C. (59° F.) . . . . .	1·109	1·102
Fresh molasses per 100 c.c. . . . .	25 gr.	25 gr.
Acidity per litre at the outset . . . . .	1·9	1·8
Density at the " chute " . . . . .	1·063	1·043
Polarisation of the fermented wort . . . . .	0·000	0·000
Non-fermented sugar per 100 grammes of molasses . . . . .	5·33	4·48
Yield in alcohol per 100 grammes of molasses	26·7	31·3
Instead of first distillation . . . . .	27·59	31·03
Ash per 100 c.c. of wash . . . . .	5·44	4·42

Too much importance must not be attached to the percentage of unfermented sugar, because it shows the accumulation of two distinct fermentations ; what is important is the yield in alcohol. With the first molasses, which was of bad quality, the yield is lowered 0·89 per cent. ; but from the molasses No. 2, which was a good sample, the alcoholic yield was very slightly better, say 0·27 per cent. at the most. One may therefore claim on an average the same industrial yield. Beet molasses as sent to be incinerated contain only about 3 per cent. of ash, but here is a liquid much richer in salts, which ought to be profitably evaporated and incinerated. Pellet gives numerous analyses of cane molasses ashes. Generally, there is a large proportion insoluble in water, sometimes even as high as 50 per cent., consisting principally of carbonate of lime, silica, and carbon, due to the excessive use of lime in the fermentation process. The carbonate of potash varies in inverse ratio to the insoluble from 10 to 35 and even 40 per cent. Considering that with the least concentration in the triple effect, bringing the spent wort to 11° Baumé, it may be incinerated with no expense for coal, the extraction of potash salts will yield a substantial profit, whatever price fuel may be in the colonies. The most simple triple effect system for this purpose is Barbet's, in which, instead of heating the distilling column with high pressure steam, a triple or even a double effect working under pressure is installed. The live steam boils the vinasse at a pressure of 3 kilogrammes, the steam from No. 1 heats No. 2, which boils at about 1 kilogramme, and finally this steam at 1 kilogramme heats the base of the column either by a

pipe, a coil, or a jacket. The spent wort thus concentrated is *evaporable* in the furnace without fuel, the combustion of the organic matters on incinerating sufficing to complete the evaporation of the water without expense of fuel, except when the potash furnaces are first lighted. Neither in the triple effect nor in the furnace are there any expenses for coal. The potash salts are thus obtained at little cost.

*Distillation with purification and ageing.*—With good quality molasses and fermentation as described, little requires to be done but to distil, either in the classical alembic of Père Labat, or in still shown in Fig. 46. The ageing of the wort (Fig. 38) prior to distillation dispenses with ageing in the distillery plant. But adherence to routine and objection to new fermentation methods has to be taken into account, as well as the use of molasses from bad cane, in which cases the still should act as a corrective. Barbet's rum still may act as a Charentais still by utilising only the pot-still proper and the condenser. A heater for the wash is added only when this has not been pasteurised, in which case the wine is very hot when charged into the still. The wash heater consists of a copper vessel of the same capacity as the still, in which the coil for the alcoholic vapours occupies the bottom, afterwards going to the condenser. A dial thermometer shows the temperature of the wort in the wort heater, and when it is high enough the regulation of taps directs the vapours to the condenser. The plant comprises a column with plates of the so-called slit-cap pattern, and above a condenser and a refrigerator with a variable steam regulator. By closing one tap and opening another, the vapours from the still, instead of going direct to the condenser, are forced to ascend the rectifying column, in which, owing to the condensation, a methodical separation of the volatile compounds is effected according to their respective degree of volatility. The plate column forms a real winnowing machine adapted to volatile substances, separating the useless impurities from the valuable products, just as the fan separates flour from groats and bran. The column separates in a rational way aldehydes and methylic alcohol and the higher alcohols from the good spirits. But, in addition, it plays the important *role*

of ageing by inducing the fatty acids to combine with the strong alcohol to form fruity ethers and perfumes.

*Rectification of rum.*—Rum is as easily rectified as other crude alcohols from beet or molasses, but it is evident that this operation is simplified in proportion as the rum is less impure and strong smelling. Pure fermentation is therefore more necessary in this instance than for common rum distilling, only care must be taken not to use spent wash in excess. In making neutral alcohol, it will be well to resort to the continuous rectification of the low

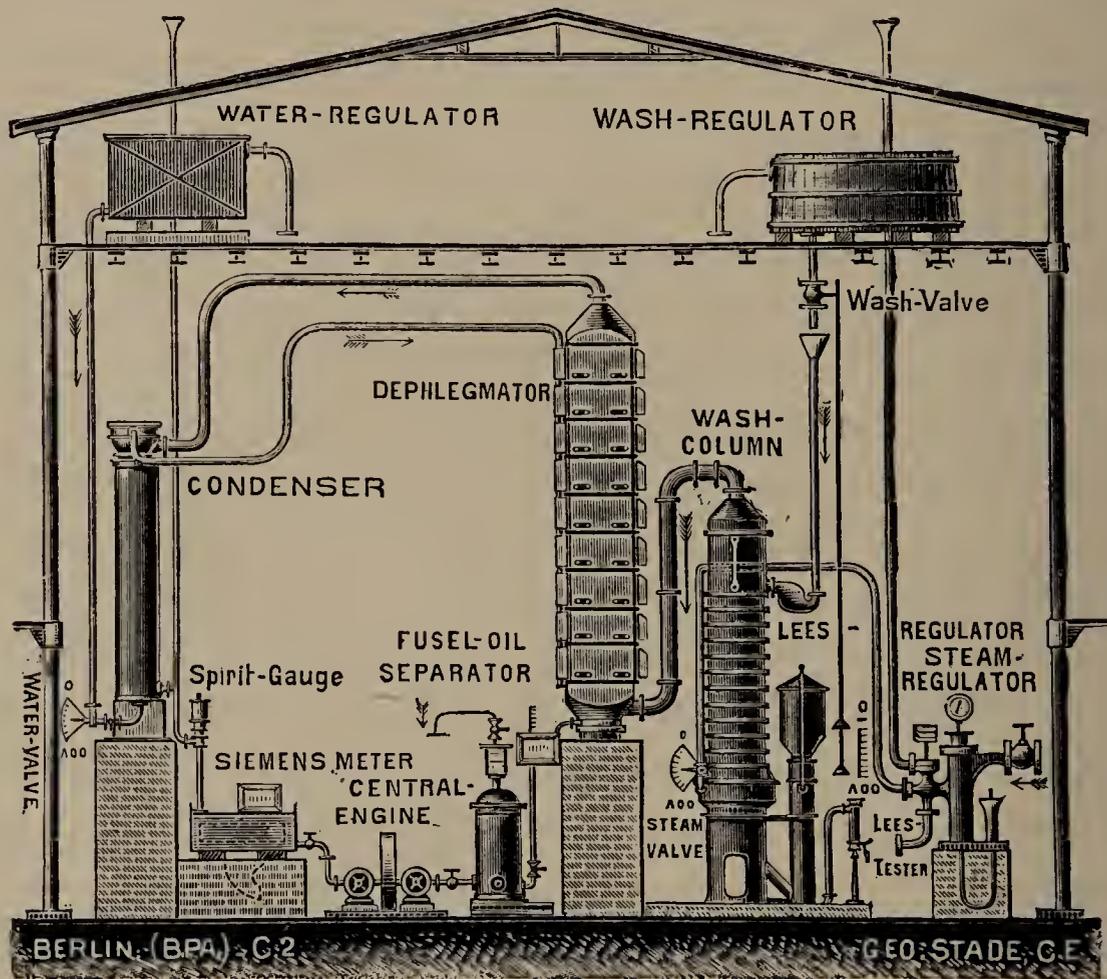


FIG. 39.—Rum distillation and rectification plant (STADE, Berlin).

wines, which has all the advantage of simplicity of installation and of working, as well as a saving in time and fuel. By this operation the first runnings, the last runnings, and the ultra last runnings are fractionated. In addition to the plant illustrated in Fig. 39, Stade makes a simple distilling column, a description of which will be given first. This distilling column, type A, is identical with Fig. 39, except that it has no dephlegmation column. The wash regulating tank is open at the top, and a pipe from its lower part connects with the still, so that the wash

is kept throughout at a uniform height. The regulating valve must be adjusted so as to allow a stream of wash of unvarying uniformity to flow into the apparatus. By this simple contrivance a perfectly uniform influx of wash into the apparatus is secured so long as the reservoir is kept supplied by the wash pump. The wash enters the wash column through the funnel pipe, and effects, in the upper part of the column, which contains several narrow and contracted passages, what the maker describes as "an advantageous dephlegmation of the through-going distillation vapours," the lower part being appropriated to the distillation proper. The spent lees run out through a valve in the lees regulator, and spontaneously emit some vapour into the lees tester, which becomes condensed therein, and this condensed vapour affords the surest indication of the de-alcoholisation of the lees. In Stade's type B, Distillation Rectification apparatus, the vapours generated in the wash column of dephlegmation pass through the latter and thence into the condenser. Each section of the dephlegmator consists of a square cast-iron box containing a number of cooling pipes arranged in horizontal rows and secured between two partitions. The cooling water flows inside the pipes, and the spaces between the latter are filled up with porcelain balls, thus producing alternately powerful dephlegmation and rectification of the vapours. The cooling water flows down from one row of pipes to the other with the assistance of the water boxes screwed to the sides, and as the latter contain several outlets, the cooling pipes can be cooled without difficulty whilst the machinery is working. The same applies to the vertical cooling pipes in the condenser, which, as the cooler is open at the top, can at any time be cleaned from the top with a brush.

*Preservation and transport of molasses.*—At the outset it was mentioned that the cane molasses now produced would yield 132,000,000 gallons of spirit of 60 per cent. strength yearly. Much less than this is, however, produced, due chiefly to the fact that in Cuba, Java, Louisiana, the molasses are not fully utilized; some factories even try to burn them. Sugar factories have in many cases given up distilling them on the spot, owing to the poor yields in quality and quantity; moreover,

there being no outlet or market near, the rum casks must be transported to them, and rum casks are very dear. Tropical climates are not suited to the transport and preservation in good condition of empty casks, the sun dries them up and they fall to pieces, and in spite of re-coopering and steeping them in water the damage is considerable, and the returned empties are a great expense. Very often the value of the cask is at least double that of the molasses it contains. Molasses distilleries should be installed at the seaports, cheap transit of the molasses thereto should be arranged, and warehousing and preservation must be provided for. Barbet solves the problem by boiling the molasses *in vacuo* to the broken proof "cassé," and then runs it into loaf-sugar moulds or into square or oblong moulds with a lining. As all factories possess plant for boiling molasses, only a few moulds are required. To minimise the stock of these, the loaf moulds may be dipped in wrought-iron tanks through which cold water circulates. The loaf sets quicker, and the mould is available again sooner. Before running the molasses into the mould, the latter is lined with a sheet of packing paper. The molasses is then run in, and, when set, turned out. The molasses is thus packed in the paper, which hinders the blocks from sticking together in transit, and protects the somewhat hygroscopic molasses from humidity. Transit of such molasses loaves becomes an extremely simple matter: manipulation being identical with sugar loaves there is no dead weight to transport, the weight of the paper, being negligible compared with that of the casks, which often exceeds 2 per cent. of the whole. The concentration of the molasses ensures a very appreciable economy in freight, since it removes 10 to 12 per cent. of water; there is only 88 to 90 per cent. of the original weight. This saves carriage, which is a point of great importance, especially in countries with indifferent roads in which conveyance is made on mules or horses. The semi-solid molasses may be stored in warehouses and not in huge tanks, which are required for the liquid product. Besides this the molasses is not liable to ferment, and the trouble of burst casks is done away with. The consistency of the product, and the fact that it has been heated to a high temperature, ensure that it is sterilised and not liable to deteriorate.

The Prussian Government allowed the use of raw sugar for distillery purposes provided that it was denatured by the addition of 2 per cent. of lamp black. The use of raw beet sugar must, however, as pointed out by O. Reinké,<sup>1</sup> be counterbalanced by the addition of cereals, yeast extract, etc., to yield the quota of nitrogenous material and phosphates. The raw sugar must be sterilised by heat, and any alkalinity corrected by the addition of lactic or sulphuric acid, or by increased acidity in the malt and rye mash. If, however, the sugar is slightly acid, the addition of about 0.01 per cent. of sulphuric acid will be sufficient, the correct acidity of the wash being 0.2°. Pure sugar gives a yield of 49.8 per cent. of alcohol, which is equivalent to 0.6274 litre per kilo. Raw beet sugar contains 90 to 96 per cent. of sugar and 0.9 to 2.8 per cent. of ash, and a raw sugar containing 92 per cent. of sucrose will yield 57.7 litres of alcohol per 100 kilos.

<sup>1</sup> *Chem. Zeit.*, 1915, 39, 149.

## CHAPTER VIII

### ALCOHOL FROM MISCELLANEOUS SOURCES.

ALCOHOL can be produced from any material containing sugar or starch by the methods already described, but the value of such materials for the purpose depends upon several factors, including relative abundance of the material, cost at which it can be delivered, percentage of sugar or starch contained therein, and value of the bagasse for feeding or other purposes. Materials which are rich in sugar are to be preferred, since the only method of treatment required is infusion, no conversion by means of malt or acids being necessary, and therefore cost of preparation low. But very few materials of this class are to be found in any quantity, the only ones used, in addition to molasses, up to the present being mahwa flowers and the sap of certain trees, to which reference will subsequently be made. Several materials less rich in sugar have been proposed or used for the manufacture of alcohol. If these are obtainable in sufficient quantity and at a low enough price they will no doubt serve; waste materials, *e.g.*, grape marcs, waste currants, and such like are, in fact, used for this purpose in countries where they are produced, but the value of many other materials which have been recommended is somewhat problematical.

With regard to starchy materials, those containing the highest percentage of starch are naturally the best, less offals being produced and less residue left after fermentation to be dealt with. When the offals and residues have a definite feeding value they may be disposed of to farmers, as, for instance, the germ, etc., of Indian corn, or brewer's grains, but wet residues, in many cases, do not possess any such value, and must be got rid of by burning under the boilers. For this reason products, even waste products,

which contain but little starch and a high percentage of cellular tissue, although obtainable at low cost, cannot successfully compete with those that are at present in use.

Many attempts have been made to manufacture alcohol from waste wood and sawdust. Large quantities of this accumulate in timber-growing areas, and are either burnt or turned into the nearest stream, to be a nuisance for many years. Wood contains about 50 per cent. of cellulose, and in addition soluble carbohydrates, which can be converted into fermentable sugars by treatment with acids. The conversion, however, is far from complete, and the yield of alcohol falls very much short of the theoretical amount.

Processes have also been devised for the production of alcohol from the waste liquors of sulphite-cellulose factories, and although failure attended the first efforts, success was at last attained, and now large quantities of alcohol are produced in this way.

#### SUGAR-BEARING MATERIALS

*Mahwa flowers.* — The flowers known as mahwa flowers, produced by *Bassia longifolia* and *B. latifolia*, trees indigenous to India, contain a very high percentage of sugars, there being an increase up to the period just preceding their fall, when they contain from 60 to 70 per cent. Suggestions have, therefore, been made to employ the dried flowers for the manufacture of alcohol, and if sufficient quantities are available, they will no doubt prove to be a very valuable raw material. G. J. Fowler, J. D. E. Behram, S. N. Bhate, K. H. Hassan, S. Mahdihassan, and N. N. Inuganti<sup>1</sup> have made an exhaustive examination into the biochemistry of these flowers, and found them to contain dextrose, laevulose, maltose, sucrose, pentoses, and cellulose, and, in addition, several enzymes, of which maltase, catalase, and oxidases were present in all stages of growth. On infusion with water the natural yeasts occurring in the flowers immediately brought about fermentation of the mash, which was stimulated by the addition of small amounts of sulphuric acid and ammonium

<sup>1</sup> *Jour. Indian Inst. Sci.*, 1920, 81-118.

phosphate, yields of 90 per cent. of the theoretical amount of alcohol being thus obtained.

*Nipa palm*.—The Nipa palm (*Nipa fructicans*) grows extensively in the Philippine Islands, its habitat being the mouths of tidal rivers, where it appears to flourish by reason of its contact with the brackish water. According to a United States Consular report (1915), there are at least 100,000 acres of Nipa palm swamp available in the Philippines, 90 per cent. of which is still untouched, and while certain portions are privately owned, much of it is still under Government control, and may be leased for 10 per cent. of the production per season. At the present time very nearly all the alcohol produced in the Philippines is derived from one Nipa palm swamp of about 45,000 acres in extent, but only about one-third of this has been worked. The production of alcohol is approximately 2,500,000 gallons per season, nine-tenths being derived from Nipa sap. The alcohol is made by native labour, and is consumed locally for drinking, only 2 per cent. of the total being denatured for commercial use. A considerable quantity of the sap is also fermented and consumed as a beverage by the natives; this will contain 4 to 16 per cent. of alcohol. Besides the Nipa palm, fermented drinks are prepared from the coconut palm (*Cocos nucifera*), the buri (*Corypha elata*), and the sugar palm (*Arenga saccharifera*), but in smaller quantities.

According to H. D. Gibbs,<sup>1</sup> 93 per cent. of the alcohol and fermented beverages produced in the Philippines was derived from Nipa sap. There were then in existence sixty-eight distilleries, the combined production of which amounted to upwards of 2,000,000 proof gallons (U.S. proof, density 0.7939 at 60° F.), from 20,000,000 gallons of sap treated in the season.

The Nipa palm grows in a wild state, but some portions are under semi-cultivation. No sap is taken before the fifth year, but subsequently, after the tree has fruited, the fruit stalk is cut, and each day a new slice removed in order to expose a fresh surface, from which the sap or *tuba* exudes. The yield of sap varies considerably, but amounts per tree daily to about 0.58 litre (rather over a pint), or 43 litres (nearly 10 gallons) during the

<sup>1</sup> *Philippine Jour. Sci.*, 1911, 99-143, 147-206.

season. The produce per acre being 820 gallons. This yield, the author believes, could be considerably increased by cultivation and improved methods of tapping. The average composition of the fresh sap is : Density at 15° C., 1.07 ; total solids, 17.5 per cent. ; sucrose, 16.5 per cent. ; traces of acidity, reducing sugars, and nitrogenous matters, 0.54 per cent., and ash, 0.46 per cent. The methods of collection and transport are very primitive, and cleanliness is unknown, so that inversion and fermentation rapidly ensue with consequent loss of sugar ; also, the fermentation and distillation losses are considerable, the yield on the average being only 5.6 per cent. by volume of alcohol, whereas, assuming 14 per cent. of sugar to be present in the juice as fermented, this should yield 7.6 per cent. of alcohol. With improved methods of manufacture the production of alcohol from Nipa palm sap should be very profitable, the author stating that it is the cheapest source of alcohol known, the raw material costing only 1.35d. per litre of 90 per cent. alcohol. Although the sap of the cocoanut palm is used to a limited extent for distillation, the product being known as *vino de coco*, and very much esteemed, the difficulty of collecting it, owing to the inflorescence being at considerable height, is considerable. The *buri* palm only flowers during one season, and is therefore only available for that limited period.

According to a Report of the Bureau of Science, Manila, in 1913, there were seventy-five modern distilleries in the Philippines producing 2,500,000 gallons of distilled spirits, over 98 per cent. of which was used in the form of beverages and the remainder as fuel. The cost of collecting the sap was about 4s. per 100 gallons, but it is estimated that in a well-managed area this might be reduced to 3s. or even 2s. ; 100 gallons of sap yield 6 to 7 gallons of alcohol, so that the cost of raw material would amount to only about 4d. or 6d. per gallon. At that time a plant to produce 500 gallons of alcohol per day could be obtained for £3000, and allowing for wages, fuel, and interest on investment, it was computed that the alcohol could be produced at 10d. per gallon, experts even averring that, under favourable conditions, it would cost no more than 7d. It is estimated that

the Nipa sap in the Philippines would yield 50,000,000 gallons of alcohol per season.

The Nipa palm also grows extensively in British North Borneo, and although the area has not been accurately surveyed, it is believed to be greater in extent than in the Philippines. It is especially abundant in Labuk Bay and in Sandakan Bay, there being well over 50,000 acres in these districts readily accessible from Sandakan; 200 acres of Nipa palm under efficient management would yield 500 gallons of alcohol per day for the six or seven months during which the sap flows.<sup>1</sup> The Manila Bureau of Science gives the yield of sap as 4000 gallons per acre annually, equal to at least 4000 lb. of sugar. The British North Borneo Government estimate that in their territory there are at least 300,000 acres of Nipa palm, easily accessible, and discuss the possibility of starting this industry in their Department of Forestry, Bulletin No. 3.<sup>2</sup>

*Arbutos unedo*.—The ripe fruits of the strawberry tree (*Arbutus unedo*) contain, on an average, 18.8 per cent. of sugar, and by fermentation yield a wine containing 9.15 to 10.5 per cent. of alcohol.<sup>3</sup>

*Curra:nts*.—In Greece damaged currants, unsuitable for food purposes, are utilised for the manufacture of spirit. Currants are also used in Italy for the same purpose, and A. Havry<sup>4</sup> details the method employed at one distillery in which the diffusion process was employed. The diffusers, of which there are 20, are in the form of wooden tuns, tapering from top to bottom, and fitted with false bottoms consisting of perforated copper sheets. The freshly charged tun is filled with hot water, which is allowed to remain on the currants for two hours, the juice, having a density of 34° to 35° Balling, being drawn off into a reservoir. A second charge of water is then run in, and after half-an-hour it is also withdrawn, having a density of 20 to 22° Balling. For the fermentation the juice is diluted to 14° Balling,

<sup>1</sup> *U.S. Con. Rep.*, No. 281, Dec., 1915.

<sup>2</sup> *Board of Trade Journal*, Aug. 7, 1919.

<sup>3</sup> G. Sani. *Atti. R. Acad. dei Lincei. Roma*, 1913, 884, *Jour. Soc. Chem. Indt.*, 1913, 803,

<sup>4</sup> *Z. Spiritusind.*, 1910, 33, 525-526.

and fermentation commenced at 20° to 22° C., finishing at 36° C. in thirty-two to thirty-five hours. The distillation is carried on in a continuous rectifying plant, the spirit being of excellent quality. The wash contains tartrates, which are also recovered by addition of milk of lime to the spent wash, the calcium tartrate, thus precipitated, being converted into tartaric acid or cream of tartar. The currants contain 55 per cent. of sugar, and yield 28·5 to 33 litres of absolute alcohol per 100 kilos of material. The calcium tartrate (containing 50 to 51 per cent. of tartaric acid) recovered amounts to 2·5 to 2·8 per cent. on the weight of the raw material.

*Pears.*—According to K. Windisch,<sup>1</sup> small unripe pears may be used for the manufacture of alcohol, although it is more advantageous to employ ripe fruit. The yield from unripe pears, deficient of sugar, amounted to only 1 to 2 litres of absolute alcohol per 100 kilos, whereas fully ripe fruit gave 3 to 3·5 litres; 100 litres of pear pulp yield 6 litres of alcohol. The pulp is very liable to ferment and acetify, and must therefore be distilled as quickly as possible.

#### STARCH AND INULIN BEARING MATERIALS

*Jerusalem artichokes.*—Experiments conducted by M. Rudiger<sup>2</sup> have shown that Jerusalem artichokes are available as a source of alcohol. Tubers which had been left in the ground over the winter, and therefore, presumably, were deteriorated, gave a yield of 6·4 litres of alcohol per 100 kilos by direct fermentation of the mash without acid hydrolysis. Heating of the mash to 55° C. before fermentation resulted in an increase of 0·4 litre, but higher temperatures led to reduction in the yield. Treatment with acid increased the amount of alcohol, but this method of treatment is not so economical in small distilleries. The spirit is of good quality, the once-distilled material having a much better flavour than that from beets, it is stated.

*Manihot* (Tapioca).—The fresh roots of the plant, investigated by H. Lange,<sup>3</sup> treated with malt, has twice the spirit value of

<sup>1</sup> *Z. Spiritusind.*, 1905, 28, 87-88.

<sup>2</sup> *Ibid.*, 1920, 43, 203.

<sup>3</sup> *Ibid.*, 1909, 32, 200-201.

potatoes, but, as pointed out by the author, these could only be used where they are grown. If they were to be used in Europe they would have to be dried for the purpose. In this condition they contain water, 14.00 per cent.; starch, 66.89; protein, 2.06; sugar (calc. as invert.), 1.90 per cent. They therefore constitute a richer material than maize or rye, and have about the same value as dried potatoes.

The dried roots require to be broken up, since they cannot be saccharified by simply steeping or boiling. They are therefore first passed through a disintegrator or ball mill, and further reduced to grist or meal in a Gruson mill.

The ground material is heated with water, preferably in a Henze steamer, the proportions being 100 kilos of grist to 250 litres of water. Steaming is gradual, the pressure being allowed to rise slowly to 2.25 atmospheres, at which it is kept for ten minutes. The saccharification of the mash is brought about by the addition of 10 per cent. of green malt, the mash having a density of 19.65° Balling, and an acidity of 0.1 per cent. With a specially cultivated distillery yeast, known as Race XII., attenuation follows the normal course, the pitching temperature being 20° C.; after sixty hours the mash registers — 0.8° Balling. The yield of alcohol was found to be equivalent to 59.8 per cent., calculated on the starch originally present.

*Asphodel* (*Asphodelus ramosus*) is a very prevalent weed in Italy, where it is known under the name of *porazzo*, and is such a nuisance that it has been referred to as “the plague of the Mediterranean.” The roots or tubers of the plant contain a considerable quantity of starch, and at one time it was cultivated for the manufacture of spirit. It seems, however, that very great difficulty was experienced in the rectification of the alcohol, and for this reason the cultivation of the plant was given up. Further investigation would no doubt solve the problem of efficient purification, in which case Italy would possess in it a remunerative source of alcohol, which would add materially to the country's supplies. The plant is extremely prolific, the yield of roots per acre amounting to 17,850 lb., capable of producing at least 107 gallons of alcohol. When *porazzo* was fermented

in Algiers, the residue was found to be quite suitable for the manufacture of paper, but it is stated that it can be used as it is produced for cattle feeding.<sup>1</sup> A patent was taken out in France by A. Badoil<sup>2</sup> for the manufacture of alcohol from the tubers of asphodel, the method including macerating in water for removal of resins, wax, etc., heating under pressure with steam at about 3 kilos pressure for one to two hours, then crushing and pressing the material. The juice expelled is acidified, boiled for one to two hours, purified by passing through animal charcoal, and is then fermented and distilled in the usual way.

*Helianthus*.—The tubers of *Helianthus* have been proposed as suitable material for the manufacture of alcohol by J. Kocks.<sup>3</sup> He found on analysis that the tubers contained 27.38 to 27.98 per cent. of dry material, consisting of protein, 11.69 to 14.06 per cent. ; sugar (lævulose), 8.11 to 13.60 per cent. ; inulin, etc. (producing sugars on hydrolysis), 58.33 to 60.52 per cent. ; together with fat, ash, and cellulose in smaller amounts. The author gives the following yields of alcohol in litres per 100 kilos of tubers :—

	1	2	3	4
Juice fermented without treatment	5.16	5.16	5.33	4.07
Juice treated with sulphuric acid	8.21	8.10	9.40	6.60
Tubers fermented directly . . . . .	5.92	7.90	6.38	—
Tubers treated with sulphuric acid	8.83	7.90	7.97	—

*Bananas*.—Banana meal is prepared from the unripe fruit by peeling them and cutting into slices, which are dried and then ground. Banana meal contains a large quantity of starch, and according to C. Nagel<sup>4</sup> it has also weak diastatic powers, so that the starch becomes slowly saccharified without addition, but the yield of alcohol in this case is but small ; with the addition of malt extract previous to fermentation much higher yields of alcohol result, amounting to 42 to 47.8 litres per 100 kilos. With the addition of 30 per cent. of kilned malt to the meal a very

<sup>1</sup> *Annal. d'Ingen. e. d'Archit.*, April, 1918.

<sup>2</sup> *French Pat.*, 423, 043, Nov., 23, 1910.

<sup>3</sup> *Z. Spiritusind.*, 1909, 32, 161.

<sup>4</sup> *Ibid.*, 1912, 35, 185.

fine yeast crop is obtained, equal to 20 per cent. on the weight of the materials employed. At that time banana meal was obtainable at the price of 30s. per cwt. According to J. von Sury<sup>1</sup> dried unripe bananas contain 80 per cent. of starch.

*Horse chestnuts.*—Horse chestnuts have been suggested as material for the manufacture of alcohol, and would no doubt serve for the purpose if sufficiently plentiful. Prof. S. J. M. Auld<sup>2</sup> found the whole nut (dried) to contain 73·97 per cent. and the dried kernels 74·45 per cent. of carbohydrates. J. L. Baker and H. F. E. Hulton<sup>3</sup> state that the amount of sugars and starch in the peeled nuts varies from 42·1 to 59·5 per cent., while Rousset<sup>4</sup> gives the proportion of sugars, gums, and starch in the kernels as 17·2 per cent.

When the peeled nuts are heated with dilute sulphuric acid for three hours and then fermented, a yield of 27 per cent. of alcohol is obtained, calculated on the dried, peeled kernels, or 12 per cent. on the original material (Baker and Hulton); 100 kilos of dry horse chestnuts contain 20 to 30 per cent. of starch, and will produce 27 to 28 litres of alcohol (J. Goris).<sup>5</sup>

*Acorns.*—According to the same authority,<sup>6</sup> acorns removed from the cups and hydrolysed by heating with a mixture of dilute sulphuric and hydrochloric acids, gave 8·58 to 20·16 litres per 100 kilos of dried, undecorticated material, or 28 to 31 litres from the decorticated material, the variations being due to variety of plant, state of ripeness, and also particularly to the strength of the extract. Baker and Hulton found the dried peeled kernels to contain 63·9 per cent. of sugars and starch, the yield of alcohol being 27 per cent., equivalent to 12 per cent. on the moist material, the same as with chestnuts.

*Xanthorrhœa preisii.*—Several of the species of *Xanthorrhœa*, including *X. preisii*, are known in Australia under the name of "Grass tree." This particular tree is confined to *Western Australia*, and it contains in the interior of the stem a soft pulp

<sup>1</sup> *Chem. Zeit.*, 1910, 34, 463.

<sup>2</sup> *Jour. Soc. Chem. Indt.*, 1913, 173.

<sup>3</sup> *Analyst*, 1917, 42, 351-355.

<sup>4</sup> *Les Matières Grasses*, 1913, 6, 2980-2981.

<sup>5</sup> *Compt. Rend.*, 1917, 165, 345-348. *Feuille d'Inform. du Minist. de l'Agric.* (France), 1917, 20, 10. See also *Jour. Soc. Chem. Indt.*, 1918, 277a.

<sup>6</sup> *Kayser (Feuille. d'Inform. du Minist. de l'Agric.*, 1917, 22, 9.

which, after drying, can be ground to a meal. Such a meal has been prepared by Mr. Chas. Harper, of Guildford, Western Australia, and E. A. Mann<sup>1</sup> suggested this product as a raw material for the manufacture of alcohol. The author found this meal to contain: Moisture, 9.19 per cent.; fat, 0.78; albuminoids ( $N \times 6.33$ ), 2.83; fibre, 35.93; ash, 0.40; and carbohydrates (by difference), 50.87 per cent.

The carbohydrates were composed of: Reducing sugars (calculated as glucose), 10.25; non-reducing sugars (calculated as cane sugar), 15.86; and other carbohydrates (by difference), 24.76 per cent.

The meal was washed with water and fermented with yeast, the fermentation was quickly completed, the yield of alcohol in one experiment being equal to 1.24 gallons per bushel (60 lb. of meal). Experiments on a larger scale, however, gave much less alcohol: in one case, 0.8, and in another 0.5, proof gallons of spirit per bushel. The author gives the following comparative yields of alcohol from various materials:—

	Proof gallons per bushel (60 lb.)
Pure starch . . . . .	4.68
Wheat, rye, oats, buckwheat, maize (average) .	2.08
Malt . . . . .	2.00
Potatoes (containing 21 per cent. starch) . .	1.26
Sugar cane (containing 12 per cent. sugar) .	0.72
Beet . . . . .	0.60
Grass tree core . . . . .	0.5 to 1.24

#### HEMICELLULOSES

*Swedish white moss.*—According to a United States Consular Report of December 21st, 1918, the County Syndicate Aktiebolag petitioned the Swedish Government for their permission to manufacture 5,000,000 litres of alcoholic spirit from white moss. The petitioners propose to manufacture alcohol under official supervision and with the co-operation of the Government. It is stated that almost unlimited supplies of the moss are available,

<sup>1</sup> *Jour. Soc. Chem. Indt.*, 1906, 1076.

and that a very good quality of alcohol can be produced from it at a lower cost than from grain or potatoes.

*Cladonia rangiferina* (reindeer moss).—This is a lichen which occurs in large quantities in Arctic climates, and is probably the same as that referred to above. Elrodt and Kunz<sup>1</sup> report that this moss is largely composed of hemicelluloses, which, on heating with acids, are converted into fermentable sugars. The composition of this moss is given as follows: Water, 11.7; nitrogen, 0.66; ash, 4.87; ether extract, 1.6 per cent. The most suitable method of treating the plant consisted in first steaming it for an hour under a pressure of three atmospheres, then adding 2.5 per cent. of hydrochloric acid (25 per cent. strength), further steaming for one hour at three atmospheres pressure, and neutralising the juice. By such method 71.7 per cent. of extract, containing 54.5 per cent. of sugars, was obtained. Fermentation proceeded normally, the growth of yeast being good, but the process was found to be stimulated by addition of phosphoric acid.

*Marine Algæ*.—E. Kayser<sup>2</sup> suggests the use of the seaweeds *Laminaria flexicaulis* and *L. saccharina* for the manufacture of alcohol. These, when dried and comminuted, on heating yield a liquid which is capable of being fermented. Experiments have shown that the yield of alcohol amounts to 3.58 to 3.76 litres per 100 kilos. of dried material. Larger yields of alcohol are, however, obtainable by heating the dried algæ with 7 per cent. sulphuric acid and bringing the mash to an acidity of 0.1 per cent. previous to fermentation; in this case a yield of 12 litres per 100 kilos was obtained.

*Laminaria digitata* is another species of algæ which has also been examined by the same author.<sup>3</sup> On heating the dried product (containing 10 per cent. of water) with water containing 3 to 6 per cent. of sulphuric acid at 122° C. for half to one hour, then cooling and bringing the liquor to an acidity of 0.1 per cent., it fermented readily, yielding 6 litres of alcohol per 100 kilos of dry material. Fermentation was accelerated by the addition of

<sup>1</sup> *Brennerei-Zeit.*, 1918, 6171; *Chem. Zeit.*, 1919, 43, Rep. 40.

<sup>2</sup> *Ann. Chim. Analyt.*, 1919, 1, 79-80.

<sup>3</sup> *Compt. rend. Acad. d'Agric.*, 1918, 4, 450-451.

nitrogenous materials. The author is of opinion that higher yields could probably be obtained on a large scale under proper conditions, and also that the mineral matter in the vinasse might with advantage be recovered.

Marine algæ contain both potash and iodine, which can be recovered from the residual liquors, although not so economically as from seaweed ash or kelp.

H. Landmark<sup>1</sup> suggested the use of seaweeds as food for the yeast in the fermentation of sulphite-cellulose liquors. They contain nitrogenous matter and phosphoric acid, and have the additional advantage that on heating with acids fermentable sugars are formed.

*Corozo nut* or *vegetable ivory* (*Phytelephas macrocarpa*), and other plants such as *Palma dun*, indigenous to South America, have been examined by G. Mezzadrolì<sup>2</sup> with a view to their employment for the manufacture of alcohol. These products differ, however, very materially from all other products used for this purpose, in that they are largely composed of the carbohydrate mannan ( $C_6H_{10}O_5$ ), isomeric with starch, convertible on hydrolysis with acid into mannose ( $C_6H_{12}O_6$ ), isomeric likewise with glucose. Mannose, however, is not fermentable with ordinary yeast, but, according to the author, it can be fermented with the yeast of saké—the Japanese drink—although but slowly. Wild yeasts found occurring on fruits from Perugia were found more suitable, and these fermented mannose completely, 4 to 6 per cent. of alcohol on the weight of the original materials being obtained. Corozo nuts and the waste from button factories yielded 10 to 15 litres of alcohol per 100 kilos of material employed.

*Sotol*.—This plant, which grows very profusely in Northern Mexico and Western Texas, appears to be quite suitable for the manufacture of alcohol, and, according to United States Consular Reports of May 2nd, 1915, and August 7th, 1918, preparations were being made at Ciudad Juarez, Mexico, to use it for that purpose. The cost of collecting the plant was estimated at \$2 to \$5

<sup>1</sup> *Pap. tekn. Forening*, Apl., 9, 1919; *Jour. Soc. Chem. Indt.*, 1919, 1052-1055.

<sup>2</sup> *Boll. Chim. Farm.*, 1918, 57, 361-362.

per ton, and the yield of alcohol is stated to be 18 to 25 gallons (180 proof) per ton of material.

*Sisal* or *Heneguín* (*Aloe sisalina*).—The leaves of the aloe, which is indigenous to Mexico and has been transplanted in other parts of America, the Pacific Islands, India, and Africa, yield a fibre known as sisal hemp. The waste or bagasse from this material contains 12 per cent., and sometimes up to 14 per cent., of sugar. In Yucatan, from which town this fibre was first exported, alcohol is made from sisal waste, and experiments carried out in British East Africa have shown that alcohol can be prepared from it provided that the yeast is acclimatised to the liquid.<sup>1</sup> The use of special yeasts, or addition of suitable nutrients, might also be tried with advantage.

The bagasse from 1000 kilos of leaves on pressing yields 436 litres of liquid having a specific gravity of 1.04, and about 96 kilos of solid material. The liquor is heated in a copper boiler under pressure at 120° C. with a little sulphuric acid and ammonium sulphate, and then kept for one hour at 15 lb. pressure. It is run out, cooled to 38° C., and fermented with a specially cultivated yeast in a closed vessel, through which a current of sterilised air is passed. At the end of five days the density of the wort is reduced to 1.011, and on distillation a yield equal to 15.96 litres of absolute alcohol is obtained, or 16.76 litres of 95 per cent. alcohol. The yield of alcohol is very nearly the theoretical, *i.e.*, 16.17 litres.

A patent has been taken out by F. Thatcher (Assignor to L. M. Stiles)<sup>2</sup> for the use of comminuted cacti for the production of alcohol.

*Rice straw*.—A. B. C. Rogers and C. H. Bedford have patented a process for the manufacture of alcohol from rice straw and rice husk. This process consists in first steaming the material (preferably with superheated steam), then heating the pulp with dilute acid, neutralising, and fermenting. In order to assist in the reaction, hydrochloric acid vapour, chlorine, or atomised bleaching powder may be admitted during the steaming.

<sup>1</sup> *Tropical Life*, 1917, 13, 155. *Bull. Agric. Intel.*, 1918, 9, 988.

<sup>2</sup> *U.S. Patent*, 1, 096, 965, May, 19, 1914.

*Reeds.*—The Verwertung Inlandischer Produkte have obtained a patent<sup>1</sup> for the manufacture of alcohol from the roots of the common reed (*Arundo phragmites*). They claim also that the residue remaining after the distillation of the alcohol is suitable for use as a feeding material. The yield of alcohol is stated to be 10·7 to 11·5 litres per 100 kilos of roots.

*Peat.*—Several attempts have been made to manufacture alcohol from peat. According to R. Pique,<sup>2</sup> the proportion of water in peat decreases from 80 per cent. in that taken from the surface to about 44 per cent. in peat at a depth of 1 to 2 metres. In experiments carried out by the author quantities of 225 kilos of peat were heated with 400 to 460 litres of water and 3 to 4 litres of sulphuric acid, at a pressure of 3 kilos, for forty-five minutes. The juice was then removed by pressure, partly neutralised, and fermented with an active cultivation of *S. ellipsoideus*. The yeast was previously cultivated in beetroot molasses containing 3 per cent. of sulphuric acid, in order to acclimatise it to the conditions of the peat juice. By this method of working the yield of alcohol amounted to 10·02 litres of alcohol per 100 kilos calculated on the dry material. The residue left after expressing the juice, when dried, is stated to constitute a valuable fuel.

*Garbage.*—J. J. Morgan has taken out a patent for the manufacture of alcohol from garbage,<sup>3</sup> the garbage being heated with a dilute solution of an acid, by which means the carbohydrates are converted to dextrose; the acid is then neutralised and the liquid fermented and distilled.

*Alcohol from bakery ovens.*—In the manufacture of bread the dough is leavened by means of yeast. The flour contains a small quantity of sugars, which is fermented by the yeast giving rise to carbonic acid, resulting in the aeration of the dough. At the same time alcohol is formed, the greater part of which is driven off in the baking of the bread. No attempts are at present made to recover this alcohol, but there is very little doubt that it could be done. An interesting note in "The Western Brewer" (July,

<sup>1</sup> *Ger. Pat.*, 311, 217, Dec., 13, 1916.

<sup>2</sup> *Bull. Assoc. Chim. Sucr. Dist.*, 1917, 1730-1741.

<sup>3</sup> *U.S. Pat.*, 1, 114, 017, Oct. 20, 1914.

1918) in this connection is worth noting. It seems that at a bakery in New Mexico a quantity of alcohol, and also a rudely constructed plant for recovering alcohol from the bake-oven fumes, was discovered. The fumes from the oven were caused to travel through a worm cooled by immersion in a barrel of water. It appears from this journal that the discovery gave rise to much discussion as to the practicability of recovering the alcohol given off during the baking of bread. In 1919 a plant for this purpose was devised and patented by J. Kizek (assignor to P. Kitchen and J. I. Piscek).<sup>1</sup> This plant consists of a vertical drum, provided with a water jacket in the upper part, and other suitable condensing arrangements. The vapours from the baking-oven are caused to pass through the drum, and the liquid condensed, containing the alcohol, is drawn off at the bottom.

<sup>1</sup> *U.S. Pat.*, 1, 314, 082, Aug. 26, 1919.

## CHAPTER IX.

### MANUFACTURE OF ALCOHOL FROM WASTE WOOD AND SULPHITE CELLULOSE LIQUORS.

*Alcohol from wood waste.*—The problem of the conversion of cellulose into glucose has occupied the attention of chemists for a considerable period. Cellulose has the same percentage composition as starch, *i.e.*, corresponding with the formula  $C_6H_{10}O_5$ , and at first sight it would appear to be an easy matter to convert it into sugars which could be fermented. This, however, is by no means the case, since cellulose, unlike starch, is very resistant to hydrolysis, and even after long-continued boiling with acids it is very little changed, and the yield of soluble products small in amount.

In the year 1819 Braconnot first made the discovery that on heating wood cellulose with sulphuric acid a proportion of it was converted into glucose. Many attempts were made in subsequent years to utilise this reaction for the manufacture of sugar, and numerous patents were taken out for processes which were devised for this purpose, but it was not till many years later that anything approaching a workable method was invented.

In 1898 a patent was obtained by C. Pope for a method of converting starch into glucose by heating with sulphuric acid, and this has developed into an enormous industry in the United States. In the same year Einar Simonsen took out a patent for the manufacture of sugar or glucose from wood by the action of sulphuric acid, and in 1900 A. Classen was granted a patent for the same purpose, in which sulphuric acid under pressure was employed. These processes will be described later.

The composition of various kinds of wood is shown in the following table :—<sup>1</sup>

TABLE XXIV.

Wood.	Water.	Cellulose.	Aqueous Extracts.	Resin.	Other Non-Cellulose
Birch . . .	12.48	55.52	2.65	1.14	28.21
Beech . . .	12.57	45.47	2.41	0.41	39.14
Box . . . .	12.90	48.14	2.63	0.63	35.70
Ebony . . .	9.40	29.99	9.99	2.54	48.08
Oak . . . .	13.12	39.47	12.20	0.91	34.30
Alder . . .	10.70	54.62	2.48	0.87	31.33
Lignum vitæ . .	10.88	32.22	6.06	15.63	35.21
Lime . . . .	10.10	53.09	3.56	3.93	29.33
Chestnut . . .	12.03	52.64	5.41	1.10	28.82
Fir . . . .	12.87	53.27	4.05	1.63	28.18
Mahogany . . .	12.39	49.07	9.91	1.02	27.61
Poplar . . .	12.10	62.77	2.88	1.37	20.88
Pine . . . .	13.87	56.99	1.26	0.97	26.91
Teak . . . .	11.05	43.12	3.93	3.74	38.16
Willow . . .	11.66	55.72	2.65	1.23	28.74

It will be seen from these analyses that, with one or two exceptions, wood contains about 50 per cent. of cellulose, which is practically unaffected by acids or alkalis, except after prolonged heating, or when the solutions are extremely concentrated. It contains also wood gums, hemicelluloses, pentosans, lignin, resin, etc., which are known as encrusting materials, and which are removed in solution when it is subjected to the action of alkalis, bisulphites, or acids when the product is converted into paper pulp. These products are therefore contained in the waste liquors produced in very large quantities in paper manufacturing.

An enormous amount of waste wood, in the form of twigs, chippings and sawdust, is produced in all large lumbering districts, notably in the United States, Canada, and Sweden, and although many methods of utilising this waste wood have been proposed, and for a few purposes it is employed, the greater part of it is still a waste product very difficult to get rid of. In some

<sup>1</sup> Hugo Müller, *Pflanzenfaser*, 150.

places it is burnt under the boilers, but at best it is an indifferent fuel, requiring special grates for its complete and efficient combustion. In a few districts it is disposed of to charcoal burners, but even for this purpose it is not very suitable. The cost of carriage precludes its conveyance to any considerable distance to places where it might be used. Hence it is left to rot on the ground, is gathered into heaps and burnt, or is shot into the nearest stream, where it undergoes slow decomposition, fouling the banks and the water for miles, and remaining as a nuisance and a danger to health for years.

The early processes employed in the hydrolysis of the wood failed commercially principally for three reasons—first, the large amount of sulphuric acid required in the conversion; second, the length of time required; and third, in consequence of these two factors, the low yield of sugar obtained.

E. Simonsen<sup>1</sup> therefore made a critical study of the reactions, in order to determine the most favourable conditions of working. Numerous experiments were carried out on a laboratory scale, in which the conditions, quantity of acid, and water, pressure, and time were varied.

Shortly, the conclusions arrived at were: When cellulose was heated with 0.3 per cent. sulphuric acid, the yield of sugars increased from 2 per cent. (calculated on the dry material) at 1.3 atmospheric pressure to 43.1 per cent. at a pressure of 9 atmospheres. With acid of 0.45 strength the highest yield of sugar, *i.e.*, 45 per cent., resulted with a pressure of 8 atmospheres. With the most favourable temperature and pressure the maximum yield of sugar was attained in two hours. The most suitable proportions were 1050 c.c. of water to 40 grammes of cellulose.

In these experiments a considerable amount of solid residue remained, amounting to about 44 per cent. on the weight of the dry cellulose employed. On again heating this material with dilute sulphuric acid, a further 26.8 per cent. of reducing sugars was obtained.

Subsequently similar experiments were conducted with pine

<sup>1</sup> *Zeits. fur angew. Chem.*, 1898, 195-196, 219-228.

sawdust in place of cellulose ; 100 grammes of sawdust (containing 15 per cent. of moisture) being heated with 500 c.c. of dilute sulphuric acid. The highest yields of sugar (19.2 to 19.6 per cent.) in this case were obtained with acid of 0.5 to 1.0 strength. The best pressure was 9 to 10 atmospheres when the time was one hour, but in one experiment the highest yield of sugar was obtained in fifteen minutes.

In the experiments on the fermentation of the liquors, it was found that in no case was more than 64 per cent. of the total sugars fermented.

Further experiments on a commercial basis were carried out in Christiania,<sup>1</sup> using a large steam pressure boiler and a cylindrical rotating autoclave lined with lead, having a capacity of 7.5 cb. m., and also in a smaller converter, which gave more successful results. The liquor obtained in these conversions was run into large wooden tanks, in which it was nearly neutralised, and a nutrient for the yeast added, composed of 0.5 kilo sodium nitrate, 0.5 kilo of sodium hydrogen sulphate, 0.25 kilo of calcium phosphate, and 0.25 kilo of magnesium sulphate. The fermentation was started at 25° C., and required three to five days for completion.

In this case the proportions found most suitable were : Ratio of wood to liquid, 4 : 1 ; acidity, 0.5 per cent. The sugar solution obtained amounted to 10 to 25 per cent. in excess of the initial liquid, and contained usually about 5 per cent. of sugar, occasionally more when less water was used.

For the fermentation of the liquors bottom yeast was found preferable to a top yeast, and 25° C. the most suitable temperature. The fermentation was allowed to run till 75 per cent. of the sugar was fermented, determined by Fehling's solution. The alcoholic strength of the fermented liquor reached 1.0 to 1.7 per cent., and the amount of alcohol produced from 100 kilos of air-dried sawdust, containing 20 per cent. of water, amounted to 7.2 litres as a maximum.

The alcohol is stated to have been of excellent quality. Submitted to Schmelk of Christiania for examination, it gave the

<sup>1</sup> *Zeits. für angew. Chem.*, 1898, 1007-1012.

following figures on analysis : Specific gravity at 15° C., 0.8336 ; alcohol, 90.21 per cent. by volume ; solid matter, none ; methyl alcohol, trace, fusel oil, none ; aldehyde, trace ; furfural, trace ; ester, trace ; free acid, trace ; volatile oil, none.

The pressed residue from the autoclave could be burnt under the boiler.

Prof. R. F. Rutten, in his paper on this subject,<sup>1</sup> gives a considerable amount of information on this subject, which has been embodied in this chapter. He points out that if alcohol can be made from wood waste or sawdust, or from sulphite cellulose liquors, a very abundant and cheap source of supply is immediately available. The cost of alcohol manufactured from the usual raw materials precludes its use for many purposes. The most economical sources of alcohol in the United States were maize and molasses. Maize at that time cost \$21.6 per ton, and one ton of maize will yield 90 gallons of alcohol of 94 per cent. strength, so that the cost of raw material in this case was 24 cents per gallon. Molasses is a much richer material than maize. Two gallons of molasses will yield 1 gallon of alcohol of 94 per cent. strength, the cost of raw material being 21 to 22 cents per gallon. The cost of the raw material used in the German distilleries works out about the same, that is, 21 to 22 cents per gallon of the same strength.

Waste wood and sawdust could be obtained for the carting away, but in any case it would cost no more than 30 to 50 cents per ton, and as, as will subsequently be shown, 1 ton of sawdust can be made to yield 20 gallons of alcohol of 94 per cent. strength, the cost of raw material would not amount to more than 2 to 2½ cents per gallon.

Dr. Alexander Classen, of the Polytechnic School at Aachen, published in 1900 a method of treating wood with sulphurous acid under pressure for its conversion into glucose, which was subsequently fermented to alcohol. This process was patented in several countries. In the English patent <sup>2</sup> the claim is made for the conversion of cellulose or substances containing cellulose,

<sup>1</sup> *Jour. Soc. Chem. Ind.*, 1909, 1290.

<sup>2</sup> *Eng. Pat.*, 258, Jan. 4. 1900.

*e.g.*, wood, into fermentable sugar by boiling in a closed vessel at a temperature of 120° to 145° C., according to the nature of the material, with a solution of sulphurous acid and sulphuric acid, or sulphurous acid and hydrochloric acid; for instance, with a solution containing 2 per cent. of sulphurous acid and 0.2 per cent. of sulphuric acid. As an alternative sulphurous acid alone may be used, the sulphuric acid being conveniently formed by the admission of air or of an oxidising agent to the contents of the cylinder. The liquors resulting from the hydrolysis may be saturated with sulphurous acid and again used in treating a fresh charge of sawdust, and in this way liquors containing 10 per cent. of sugars may be produced, 80 or 90 per cent. of which is fermentable.

In a subsequent patent <sup>1</sup> for an improvement on this process. the moist disintegrated wood is subjected to the vapours of sulphur trioxide (SO<sub>3</sub>), or to a mixture of sulphur trioxide and sulphurous acid at 100° C., less acid in this case being required. The process can also be modified by heating the moist wood to 125° C. after treating with the acid vapours.

In a third patent <sup>2</sup> sulphurous acid is again employed, 100 parts of the wood, containing 25 to 30 per cent. of water, being heated in a closed vessel at 120° to 145° with sulphurous acid for fifty to sixty minutes. It is claimed that the sulphurous acid is largely converted into sulphuric acid, and that consequently no addition of the latter acid is needed. The excess of sulphurous acid is blown off and the mass boiled with water to extract the sugar.

In a second claim, however, both sulphurous and sulphuric acid are again included.

Experiments were carried out by Classen at Aachen in a small plant which appeared to be quite successful. With a view to enlisting the financial help of United States capitalists, a similar but larger plant was erected at Highland Park, near Chicago, and this yielded such promising results that a much larger commercial plant was installed at Hattisburg, Miss. This comprised an acid generating plant for the production of sulphurous acid solution,

<sup>1</sup> *Eng. Pat.*, 4199, Feb. 27, 1901.

<sup>2</sup> *Ibid.*, 12,588, June 20, 1901.

and the absorption of the acid blown off from the converter; a converter or digester, consisting of a revolving cylinder, 30 feet long and 36 inches diameter, in which the wood was heated with the sulphurous acid solution (this was constructed of iron, lined with sheet lead to resist the acid, and was surrounded by a steam jacket); a battery of tanks for extracting the sugars from the acid-treated wood by means of water; neutralising vats in which the excess of acid was neutralised by addition of chalk; fermentation vats and distillation plant of the ordinary type for the fermentation and distillation.

This plant, it is estimated, cost \$250,000 for building and operating for one year.

The method of working this process adopted was as follows. The digester, which had a capacity of about two tons, was nearly filled with the wood waste, and a saturated solution of sulphurous acid, equal to about one-third of the weight of the charge, was run in. The cylinder was then closed airtight and caused to revolve slowly while steam was admitted to the jacket. The temperature in the interior of the cylinder gradually rose to 290° to 300° F., the pressure being about 100 lb. Heat and pressure were kept up for four to six hours, after which the vapour was blown off into absorption tanks, and the unaltered sulphurous acid thus recovered. The cover of the cylinder was then removed and the contents turned out. The wood in this state appeared very much like finely ground coffee. It was transferred to the extraction battery, where all soluble matter was removed by repeated treatment with water. The liquor containing the sugars, amounting to 340 to 400 lb. per ton of wood treated, was pumped into the neutralising vats, in which it was neutralised by addition of chalk, and, after allowing the liquor to clarify by subsidence, it was pumped into the fermenting vats, fermented with yeast, and subsequently distilled in the usual manner.

The yield of alcohol by this process was claimed to be 18 to 20 gallons per ton of waste wood operated on.

Although great hopes were entertained at the outset that this process would prove a commercial success, many difficulties were met with which appeared insurmountable, and after operating

for a time the plant closed down. The chief difficulties were the length of time required for conversion, the large proportion of acid required, adding to the cost, and resulting in the production of too much sulphuric acid. These two factors also led to a considerable destruction of the sugars with the consequent formation of caramel and other products, non-fermentable, and rendering the extraction of the sugars much more difficult and expensive. Buckling and cracking of the lead lining of the converter also frequently occurred, resulting in considerable delay, and requiring frequent repair and renewal, adding very materially to the cost of the process.

In 1907 a plant of this type was also working in England, as described by Laskowsky.<sup>1</sup> In this case the charge of sawdust was one ton. The average amount of reducing sugar formed corresponded to 22 per cent. on the dry substance of the wood, and the most favourable results gave 60 per cent. of the sugars fermented. The yield of alcohol amounted to 5 to 6 litres per 100 kilos of dry material treated, which is only about half the above, and the wood employed contained 20 per cent. of moisture and cost £1 per ton.

Two chemical engineers who were associated with the Classen process, both in Germany and in the United States—Messrs. Malcolm F. Ewen and G. H. Tomlinson—with the financial assistance of Mr. J. M. Ewen, a Chicago engineer, commenced investigations in 1904 with a view to perfecting the methods and removing the disabilities which had been the cause of the abandonment of the old process. Knowing beforehand all the difficulties which were to be surmounted, they were after many experiments able to overcome them and evolve a process which gave the maximum yield of sugars in the minimum of time and at little cost. Their improvements consisted in shortening the time of steaming from six hours to forty-five minutes, reducing the quantity of acid required, producing a converted wood waste in a condition easily and completely extractable by water, minimising the destruction of the sugars, and lining the converter with a material almost unaffected by acids, thus prolonging its life very considerably.

<sup>1</sup> *Chem. Zeit.*, 1919, 43, 51.

Patents were taken out to cover these improvements, and an experimental plant was erected at Chicago Heights, about 20 miles from the city of Chicago, by the Wood Waste Products Company, now known as the Standard Alcohol Company, who acquired the rights in the Ewen-Tomlinson patents. This works was visited in June, 1909, by Dr. Rutten, who describes the plant and the methods of working in the paper already referred to. The plant was sufficiently large to test the process on a commercial scale, and consisted of a converter 12 feet by 8 feet constructed of steel, lined with firebrick set in a cement composed of lead oxide and glycerine. This had a capacity of two tons waste wood or three tons of sawdust. An acid generating and recovery plant, battery of extraction cells, neutralising vats, storage tanks, fermentation vats, and distillery were also included.

Wood waste, sawdust, or shavings were charged into the converter until it was completely filled, the converter was then closed airtight, and a measured quantity of sulphurous acid equivalent to one-third the weight of the wood was pumped in through a perforated pipe which passed through the trunnions of the apparatus. The sulphurous acid was then shut off and live steam blown in until the pressure rose to 100 lb., the time occupied being ten to fifteen minutes. The steam was then shut off and the cylinder slowly revolved for forty to forty-five minutes, the temperature and pressure being kept up during this period. The steam, containing the excess of sulphurous acid and acetic acid and terpenes from the wood, was then blown off into an absorption tank. As soon as the pressure was reduced to the normal, the cylinder was opened and the treated wood turned out into a conical bin, from which it was delivered on to a conveyer and thus to the diffusion tanks. In these tanks it was treated with successive quantities of hot water until it was entirely exhausted. The liquor was neutralised by the addition of lime and chalk, allowed to settle in large tanks, and then pumped into the fermentation vats, and, after fermentation, it was distilled.

Details are given of the working of two charges. The first

consisted of 3623 lb. of air-dry shavings, almost entirely pine, from Chicago mills. This contained 18.61 per cent. of moisture, and therefore the weight of dry material was 2948.8 lb. The second charge consisted of 2992 lb. of shavings, and 2105 lb. of wet hardwood sawdust, chiefly oak, but containing some pine. The total weight of wood in this case was 5097 lb., equivalent to 3058 lb. of dry material, the wet sawdust containing 70.4 per cent. of moisture.

After action of sulphurous acid, the products were analysed with the following results :

	1.	2.
Moisture . . . . .	32.23	34.63
Total reducing sugar . . . . .	13.70	14.28
Calculated to dry basis . . . . .	20.20	24.18
Total acidity as $\text{SO}_3$ . . . . .	1.23	1.12
Sulphuric acid ( $\text{SO}_3$ ) . . . . .	0.418	0.353
<hr/>		
Reducing sugars fermented . . . . .	10.70	10.97
Unfermented sugars (Xylose, etc.) . . . . .	3.00	3.21

The total weight of the waste wood treated in the two charges amounted to 8750 lb., or calculated to dry material, 6007.1 lb.

The treated wood was exhausted with hot water and the two extractions collected together in one storage tank, the liquor amounting to 2984 gallons, or 26,150 lb. in weight, and containing 5.6 per cent. of reducing sugars, calculated as glucose. The total acidity was equivalent to 0.64 per cent. of sulphuric, the real sulphuric amounting to 0.21 per cent., the balance being made up of acetic acid and organic compounds of the type of pyrogallol, whose presence was indicated by the blackening which rapidly occurred on neutralising with lime and chalk. The paucity of nitrogenous matters in the extract was corrected for by the addition of about half a bushel of malt sprouts previous to fermentation. The yeast employed in this case was a brewery yeast obtained from a neighbouring lager-beer brewery. The fermentation occupied about three days, and the subsequent distillation, which was carried out by a United States Government official, yielded 115.92 gallons of proof spirit, equivalent to 61.66 gallons of 94 per cent. alcohol. This yield corresponds to 14.14

gallons of alcohol per ton of wood waste, including the wet sawdust, or 20·55 gallons per ton of dry material.

The alcohol after rectification yielded a water-white potable spirit, having no odour or flavour of wood, and containing neither methyl alcohol nor higher alcohol, but showing traces of furfural or aldehyde.

At that time the residue from the converter was not utilised. This amounted to about 65 per cent. on the weight of the original wood, and consisted of a kind of friable cellulose. The intention, however, was to press it, and after partial drying by waste heat, burn it under the boiler. The recovery of turpentine and acetic acid from the water in the absorption tanks, after blowing off steam from the converter into them, is also a possibility.

Dr. Rutten also mentions a French process, about which little has been published, which in one experiment is said to have yielded from 3200 lb. of refuse wood, 21½ gallons of 94 per cent. alcohol, 76 lb. of acetic acid, and 1800 lb. of stock feed.<sup>1</sup> The stock feed referred to is the disintegrated cellulose. Estimates of the cost of production and selling prices in this case show a substantial profit.

The author estimates that in the Ewen-Tomlinson process, after allowing for cost of maintenance, interest, depreciation, labour, and material, with sawdust at 50 cents per ton, the nett cost of 90 per cent. alcohol would be 10·8 cents per gallon, and the Standard Alcohol Company claimed that with a larger plant it would be possible to produce it at 7 cents per gallon.

In considering the action of sulphurous acid upon wood, it has already been remarked that a solid residue amounting to about 65 per cent. of the original material is left. This material is altered in some respects, but it no doubt represents in effect the cellulose of the original wood. Dr. Rutten regards this as a significant fact, and although claim is made in the Classen patents, and in one of the Ewen-Tomlinson patents, for the conversion of cellulose into fermentable sugars, it is very doubtful if this really occurs, at any rate to an appreciable extent. The residue is practically equivalent to the cellulose obtained in paper pulp manufacture,

<sup>1</sup> G. U. Borde, *General Electric Review*, Sept., 1909

the non-cellulosic material really yielding the sugars on which the process depends.

A pamphlet was issued in 1920 from the United States Bureau of Agriculture, Forest Products Laboratory, Madison, Wisconsin, dealing with the production of alcohol from wood waste, describing the plant required and giving estimates of cost, based upon the most recent experiments. These estimates show that at a plant treating 180 tons of wood waste daily, 3600 gallons of alcohol can be produced at a cost of about 25 cents per gallon. It has been found by experience that soft woods give the highest yield, white spruce, for instance, giving 25.8 gallons of 95 per cent. alcohol per ton, and longleaf pine 25.1 gallons. Hard maple, red oak, cottonwood, and slippery elm were amongst those which gave the smallest yields, the first named giving 9.1 gallons and the last 6.0 gallons per ton.

Other agents besides sulphuric and sulphurous acid have been suggested for the conversion of wood or sawdust into sugar. For this purpose J. J. d'Orlowski<sup>1</sup> patented the use of hydrofluoric acid of 2 to 6 per cent. strength. Calcium bisulphite,  $\text{CaH}_2(\text{SO}_3)_2$ , is claimed by L. Spassky<sup>2</sup> for treating peat, sawdust, straw, moss, or cellulosic tissue, a 5 per cent. solution being used for three hours under five to six atmospheres pressure, the liquid in this case being inverted by sulphuric, hydrochloric, or hydrofluoric acid.

In a subsequent patent<sup>3</sup> A. Ponansky and L. Spassky claim, first, the removal of resins from the wood by boiling with water containing soda or lime at 140° C.; second, the removal of lignin by heating with a dilute solution of calcium bisulphite; and third, the saccharification of the residue by heating with dilute hydrochloric, sulphurous, or hydrofluoric acid for about three hours. The subsequent method of treatment comprises distillation of the liquid under diminished pressure to remove volatile acids until it has attained a density of 20° to 30° B. (sp. gr. 1.162-1.263), when a current of steam or carbonic acid is passed through at 60° to 70° C. This is followed by neutralisation with chalk, addition of tartaric acid, and fermentation.

<sup>1</sup> *Fr. Pat.*, 405,187, July, 17, 1909.

<sup>2</sup> *Ibid.*, 451,268, Feb. 9, 1912.

<sup>3</sup> *Ibid.*, 459,593, Sept. 9, 1912.

Another patent is that of G. Boulais and P. E. Lefebvre.<sup>1</sup> The patentees in this case make use of the power of wood charcoal for absorbing large quantities of sulphurous acid as a basis for their process. The charcoal thus saturated with sulphurous acid is placed in alternate layers with the wood in the autoclave, and after treating under pressure, the fibre is saccharified by heating with dilute acid and the liquid fermented and distilled. A further claim includes the dry distillation of the unaltered residue with the formation of methane, ethylene, methyl alcohol, and acetone, which serve for the purpose of carburetting the alcohol.

F. W. Cressmann<sup>2</sup> describes experiments in which the conversion was brought about by sulphuric acid. The apparatus employed consisted of a rotary digester of cast iron 5 feet long and  $2\frac{1}{2}$  feet diameter, lined with an acid-proof enamel, and covered with an outer vessel of steel. This was connected to an enamel-lined cast-iron tank containing sulphuric acid, which latter could be delivered into the digester while under pressure. Steam was admitted both to the jacket and to the interior of the cylinder, and after the operation was finished the vapours were blown off through a pipe into a quartz coil condenser. The charge consisted of sawdust equivalent to about 100 lb. of material, and the amount of sulphuric acid was 9 lb. diluted with water, the total liquid at the conclusion of an experiment being about four times the weight of the sawdust. The best results were obtained with a pressure of 7.5 atmospheres, the amount of sugar produced being equivalent to 22 or 23 per cent. on the weight of dry spruce sawdust employed; about 70 per cent. of the sugars were capable of being fermented, the yield of alcohol amounting to upwards of 91 per cent. of the theoretical. In addition, 1.4 per cent. of acetic acid and 0.1 to 0.6 per cent. of formic acid were found in the products.

Further experiments by the same author<sup>3</sup> led to the conclusion that the best results were obtained with a pressure of  $7\frac{1}{2}$  atmospheres and duration of digestion twenty minutes after this pressure had been attained. The ratio of water, dry wood,

<sup>1</sup> *Fr. Pat.*, 465,534, Feb. 7, 1913.      <sup>2</sup> *Jour. Ind. Eng. Chem.*, 1914, 6, 625-630.

<sup>3</sup> *Ibid.*, 1915, 7, 920-923.

and acid were 125 : 100 : 2½. In one experiment under these conditions white spruce yielded 23.61 per cent. of total sugars, 71.4 per cent. of which was fermentable. In another experiment with the same wood 22 per cent. of sugars was formed, 60 to 65 per cent. being fermentable.

When western larch (*Larix occidentalis*) was digested with 12.5 per cent. of water and 1.8 per cent. of sulphuric acid at 7.5 atmospheres pressure for ten minutes, the yield of sugar was 29.7 per cent., 37 per cent. of this being fermentable, the alcohol produced amounting to 4.98 per cent. on the weight of the dry wood.

The fermentation was allowed to proceed for ninety-six hours at 30° C., the yeast employed being a pure culture of *Saccharomyces cerevisiae* isolated from a yeast obtained from a Hungarian distillery working on beet molasses. The yeast was first inoculated into a molasses solution, and when in an active state of fermentation, this was added to the wood extract in the proportion of about 1 per cent. of the volume of the latter. Ammonium sulphate and malt combs were also used as a nitrogenous food for the yeast. The alcohol produced from the molasses, amounting to about 2.5 per cent. of the total, is included in the above estimates.

A comparison of the relative values of wood extract and molasses for the manufacture of alcohol is given by G. H. Tomlinson.<sup>1</sup> He states that the fermentation need not necessarily be carried out at the same time as the extraction, but that the solution can be concentrated to a syrupy consistency, and in this form conveyed to any locality where the fermentation can be more conveniently performed. The wood waste left on extraction (amounting to about 70 per cent.), after drying, is quite suitable to be used as a fuel. The amount of material rendered soluble is about 24 per cent., but only 8.5 per cent. consists of fermentable sugar. The cost of the plant employed by the Standard Alcohol Company at Fullerton, La., amounted to £456,920; the plant and equipment for converting the wood into sugars and extracting it in the form of solution costing about £200,000; the fer-

<sup>1</sup> *Chem. and Met. Eng.*, 1918, 19, 552-568.

mentation and distillation plant accounting for the remainder. This plant produced in 1913, in twenty-two working days, when working at three-quarters its full capacity, 1,688,600 gallons of sugar solution, containing 10·3 per cent. of sugars. The author estimates that, allowing for depreciation of plant, the total cost amounted to 31·8 cents per 100 gallons, or 0·318 cent. per gallon. One gallon of this wood extract yields about 0·39 gallon of proof spirit. On the other hand, molasses formed by concentrating 100 gallons of sugar solution to 12½ gallons costs approximately 3 cents per gallon, but it will yield 1 gallon of proof spirit. In the former case 1 gallon of proof spirit costs for raw materials just under 1 cent, and in the latter case 3 cents.

With regard to the utilisation as a fuel of the residue left after extraction, and also the recovery of sulphurous acid and calcium sulphate, *see* R. W. Strehlenert, "Papierfabr," 1913, 11, 645-649.

*Alcohol from sulphite-cellulose waste lyes.*—Sulphite-cellulose waste lyes are produced in enormous quantities in paper-pulp manufactories. They are essentially similar in composition to the liquors obtained from wood by the methods already described, except that they are much more dilute. According to a report by T. H. Norton,<sup>1</sup> 10 tons of lye result from the production of 1 ton of cellulose. This contains dextrose and other sugars, xylose, acetic acid, tannic acid, nitrogen compounds, resins, and also as the chief constituent, calcium lignin-sulphonic acid.

The sugars, amounting to about 1 per cent. on the weight of the lyes, are for the most part fermentable. Previous to fermentation the lyes are neutralised with carbonate of lime. The yeast is usually propagated in molasses or malt solution, and then grown in liquors containing increasing quantities of wood extract till it becomes acclimatised. The fermentation is carried on as usual, and no special difficulties are, as a rule, encountered.

For the neutralisation of the acid liquors, P. G. Ekstrom, at Skutskär, employed lime,<sup>2</sup> while at Fores, Wallin used the waste

<sup>1</sup> *U.S. Consular Report*, Nov., 1911.

<sup>2</sup> *Eng. Pat.*, 6741, March 17, 1910.

causticisation sludge.<sup>1</sup> Ekström also patented the employment of a mixture of calcium carbonate and ammonium sulphate.<sup>2</sup>

Since the crude acid liquors require a considerable amount of lime for neutralisation, it is the practice in Norway to evaporate to a certain extent before neutralisation, whereby the excess of sulphurous acid is driven off. Thus H.B. Landmark<sup>3</sup> states that 1 cb. metre of the original waste lyes requires 11 to 13 kilos of lime for complete neutralisation, whereas after evaporation only 2 to 3 kilos are needed. Ordinary brewery yeast acclimatised for 5 or 6 days to sulphite liquors is used for pitching the lye. This author also states that the addition of a little milk or whey to the liquors favours fermentation. This produces a precipitate which, on drying, forms a yellow powder, known as "lignocasein," containing 65 per cent. of casein, which can be employed as a size. In some cases yeast nutrients, *e.g.*, malt extract, dead yeast, ammonium sulphate, are also added to the cooled and aerated liquors. The liquors are very dilute, containing only 1.0 to 1.5 per cent. of fermentable sugars, with 0.5 per cent. unfermentable (W. Kiby), so that the fermented liquors are correspondingly weak in alcohol, *i.e.*, 0.5 to 0.75; therefore the consumption of steam is considerable in comparison with a molasses distillery. According to C. G. Schwalbe,<sup>4</sup> the best apparatus for distillation is the column apparatus of Kubierschky.

It is, however, preferable to evaporate the liquors previous to fermentation at 50° C. to a syrupy consistency, the pitching temperature being 26° to 28° C., and the time required four to five days. This is more in accordance with ordinary practice, and after distilling off the alcohol the residue can be evaporated to dryness, forming "cell pitch," which can be used as a fuel or for binding purposes in the manufacture of briquettes. The "cell pitch" from one ton of waste lyes is equivalent to 372 kilos of carbon in calorific value.<sup>5</sup>

The alcohol produced from sulphite cellulose liquors contains methyl alcohol, existing in the original lyes, also aldehydes, furfural, and also probably acetone. It is therefore, in a sense,

<sup>1</sup> C. G. Schwalbe, *Z. angew. Chem.*, 1910, 23, 1537-1540.

<sup>2</sup> U.S. Pat., 1,042,332, Oct. 22, 1912.

<sup>3</sup> *Chem. Zeit.*, 1915, 39, 98-99.

<sup>4</sup> *Z. angew. Chem.*, 1910, 23, 1537-1540.

<sup>5</sup> H. B. Landmark, *ibid.*

already denatured, and is in a fit condition to be used as methylated spirit for heating, lighting, and power purposes.

The yield of alcohol is, according to Schwalbe, on the average 60 litres (15.3 gallons) per ton of cellulose; Norton estimates it at 14 gallons per ton, or 6 gallons 100 per cent. alcohol per 1000 gallons of lye; Kiby 80 litres (18 gallons), and certainly not higher than 88 litres (19 gallons); Landmark gives the yield as 88 litres, and states that the cost is 1.1d. per litre (5d. per gallon) of absolute alcohol.

Considerable quantities of alcohol are now made in the United States, and also in Sweden and Norway, from sulphite cellulose waste lyes.

In 1910 Ekström<sup>1</sup> described the working of an experimental plant erected at Skutskär, Sweden, which commenced operations on May 24th, 1909. This dealt with the waste lye from a cellulose works of 5000 tons capacity. In March, 1910, the production of spirit of "normal strength" per twenty-four hours amounted to 2130 litres, the total for the month being 49,027 litres; in April the production was 2120 litres per twenty-four hours, and the total for the month 50,980 litres. The working of this experimental plant proved so satisfactory that it was decided to extend it to deal with the whole production of the lyes from 20,000 tons per annum.

W. Kiby<sup>2</sup> stated that the total production of alcohol in Sweden for the financial year 1908-9 amounted to about 22 million litres absolute alcohol, and the imports to 1.17 million litres. The sulphite pulp mills at Lärkudden were at that time producing 600,000 litres of alcohol per annum, and were extending the plant to enable them to produce 1,200,000 litres per annum. The possible production in Sweden from paper pulp mills being about 25 million litres of absolute alcohol.

At a works producing 60 tons of cellulose per day the possible production would be 1,300,000 litres of absolute alcohol per annum, at a cost of about 1.2d. per litre, after allowing 10 per cent. for depreciation. The cost of this alcohol in Sweden, after

<sup>1</sup> *Papier fabrikant*, 1910, 8, 582.

<sup>2</sup> *Chem. Zeit.*, 1910, 34, 1077-1078, 1091-1093.

paying the present tax, would therefore be about 17s. to 18s. 6d. per 100 litres (22 gallons). He estimates that in Germany 33 million litres of alcohol could be made in the paper pulp mills there, but owing to the higher taxes in that country, in addition to 10s. or 11s., the cost of the spirit, there would be an addition of 19s. per 100 litres, and he is therefore of opinion that it could not be made except at a loss.<sup>1</sup>

In reports in the "Board of Trade Journal," April 25th and July 18th, 1918, it is stated that the Swedish sulphite spirit factories had formed a company for the organisation and sale of their entire output, more particularly as a motor spirit in competition with imported petrol.

The four factories then operating in Sweden were producing about 3 million litres of spirit per annum. Four more factories were just commencing operations, their combined output being estimated at  $1\frac{1}{2}$  million litres, and ten new works were in course of construction, so that the output of spirit for that year would amount to about 12 million litres. In addition, six or seven mills were contemplating putting up plant, therefore it was estimated that in the near future the production of sulphite cellulose alcohol in Sweden would approximate to 36 million litres of 100 per cent. spirit. The average yearly import of benzine is about 25 million litres, which it is believed could be replaced by Swedish alcohol.

In Norway the annual production of sulphite cellulose is 210,000 tons, the waste lye from which contains 160,000 tons of organic matter, containing about 32,000 tons of sugars, from which 17 million litres of alcohol could be produced, a quantity considerably in excess of the requirements in Norway for motor spirit.

In Switzerland a society has recently been formed with the object of creating a cellulose alcohol industry in that country, the promoters of this enterprise being of the opinion that alcohol can be obtained more cheaply from the waste lyes than from beets or by synthetic processes.

V. K. Krieble,<sup>2</sup> in discussing the Canadian industry, states

<sup>1</sup> See also W. Kiby, *Chem. Zeit.*, 1915, 39, 212-214, 261-265, 284-285, 307-308, 350-352.

<sup>2</sup> *Paper. Ann. Conv.*, 1919, 23, 153-156.

that the sulphite liquors produced in that country are comparatively rich in sugars, *i.e.*, 2.0 to 2.9 per cent., 61.2 to 75 per cent. of the total being fermentable. The yield of alcohol amounts to 0.82 to 1.23 per cent. by volume on the original liquor, but in practice this might be increased to fully 1.5 per cent.

An improvement on Ekström's process, described by H. V. Tartar,<sup>1</sup> is of interest, since no special yeast is required, and therefore the preliminary acclimatisation of the ferment can be dispensed with.

In this modification the liquor is treated with sulphuric acid, diluted with three times its volume of water, in proportion to the sulphurous acid present. It is then concentrated *in vacuo* at a temperature not exceeding 85° C., and the sulphurous acid expelled can be recovered for use again. To the concentrated solution is added a small quantity of potassium permanganate, in the proportion of about  $\frac{1}{2}$  oz. for 500 gallons of the original liquor, with the object of destroying the last traces of sulphurous acid. The liquor is neutralised with lime, allowed to settle, the clear liquid decanted, acidified with 0.1 per cent. of sulphuric acid, and then fermented with brewer's yeast at 27° C. for forty to sixty hours, agitation being resorted to in order to aerate the mass. The concentrated liquor contains about 6 per cent. of fermentable sugar, and the alcohol recovered amounts to 0.76 to 0.86 per cent. by volume on the original lye. This author is also of opinion that in a carefully controlled plant, with suitable means for preventing loss of alcohol by volatilisation, a yield of 1.0 per cent. of alcohol from the original liquors is capable of being attained.

<sup>1</sup> *Jour. Ind. Eng. Chem.*, 1916, 226-228.

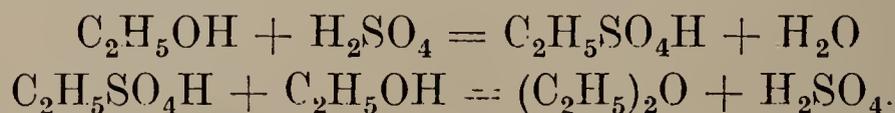
## CHAPTER X.

### PRODUCTION OF ALCOHOL BY SYNTHETIC PROCESSES.

ON heating alcohol with strong sulphuric acid ether is formed, as shown in the following equation :—



It was, however, found that the reaction takes place in two stages, ethylsulphuric acid ( $\text{C}_2\text{H}_5\text{SO}_4\text{H}$ ) being first formed, and this reacted with more alcohol to form ether, thus :—



By heating alcohol with excess of sulphuric acid, ethylene ( $\text{C}_2\text{H}_4$ ) is produced :—



In this case also ethylsulphuric or “sulphovinic” acid is formed as an intermediate product.

This reaction is, however, a reversible one, since ethylene is absorbed by sulphuric acid, forming ethylsulphuric acid, and on diluting this with water and distilling, alcohol passes over.

Faraday<sup>1</sup> in 1825 carried out an investigation of the illuminating gas produced by heating oil to a high temperature, and known as “oil gas.” This product he found to contain the gas named “olefiant gas,” previously discovered by Becher, and in 1795 investigated by four Dutch chemists, who determined its properties. Faraday also found that “olefiant gas” was soluble in sulphuric acid, one volume of the acid dissolving about 80 volumes of the gas.

Faraday gave some of the acid containing olefiant gas to Henry Hennell, who investigated it and found it to form salts which differed from the ordinary sulphates, the acid and its salts being the same as those of “sulphovinic acid,”

<sup>1</sup> *Phil. Trans. Roy. Soc.*, 1825, 440.

which is formed by mixing alcohol and sulphuric acid together. Later Hennel<sup>1</sup> stated that on heating sulphovinic acid ether is formed, and also that when distilled with dilute sulphuric acid, it is decomposed into alcohol and sulphuric acid.

The formation of ethyl sulphuric acid by the absorption of olefiant gas in sulphuric was for a long time doubted, but Berthelot<sup>2</sup> subsequently studied this reaction and confirmed Hennel's conclusions.

Berthelot also discovered that ethylene could be produced from acetylene.<sup>3</sup> Acetylene being formed from calcium carbide by the action of water, and calcium carbide being produced by heating carbon and lime in the electric furnace, a truly synthetic production of alcohol is therefore possible.

C. F. Tidman<sup>4</sup> mentions these particulars and also refers to the investigations of Fritsche, who in 1897 commenced experiments with a view to utilising the reaction for the manufacture of alcohol on a commercial scale. Fritsche made more extended trials in 1912, but eventually came to the conclusion that the very large scrubbing surface required for efficient absorption in his plant rendered the process uneconomical, and it was therefore abandoned. During the war experiments were carried out at Skinningrove Iron Works on the production of alcohol from debenzolised coke-oven gas, and a successful method was devised for this purpose by Messrs. Bury and Ollander.<sup>5</sup>

The gas produced by the coking of Durham coal contains 2.0 to 2.5 per cent. of olefines, principally ethylene, and in addition, tar, ammonia, naphthalene, benzol, sulphuretted hydrogen, higher olefines, and water vapours, which have all to be removed previous to the absorption of the ethylene. The methods of removing these constituents are the same as those employed in gas works, and which are well known. Benzol is removed from the

<sup>1</sup> *Phil. Trans. Roy. Soc.*, 1828, 365.

<sup>2</sup> *Compt. rend.*, xxxvi., 1098.

<sup>3</sup> *De la Synthèse en Chimie Organique*, 1860, 183.

<sup>4</sup> *Jour. Soc. Chem. Ind.*, 1921, 86 T.

<sup>5</sup> Bury, *Cleveland Institution of Engineers*, Dec., 1919; Prof. Bone, Third Report of the British Association Committee for the Investigation of Fuel Economy; C. F. Tidman, *ibid.*

gas by absorption in a heavy oil, from which it is recovered by distillation, and higher olefines are absorbed in a " bubbler " by means of 80 per cent. sulphuric acid, which also removes most of the water vapour. Ethylene is extracted from the gas by means of strong sulphuric acid, containing 95.4 per cent.  $H_2SO_4$ , a most important point in this connection being an intimate contact of gas and acid. The absorption is carried out on the counter current system by passing the gas up lead-lined towers, which are steam-jacketted and packed with broken silica, down which sulphuric acid is allowed to flow from a cistern at the top. The best results were obtained when the acid was caused to flow intermittently and not in a continuous stream. In this way absorptions of as high as 90 per cent. have been attained, and it was found by experiment that a 70 per cent. absorption could readily be got by two and a half minutes contact of gas and acid.

The temperature most favourable for the absorption is between  $60^\circ$  and  $80^\circ$  C.; below  $60^\circ$  the absorption is slow, and at higher temperatures more or less decomposition occurs. In the absorption tower the acid could be saturated to the extent of 18 per cent., with ethylene, but with a mechanical agitator as high as 30 per cent.

In order to obtain alcohol from the ethyl sulphuric acid thus formed it is diluted with water to 64 per cent. sulphuric acid and distilled in a vacuum of  $26\frac{1}{2}$  inches, the distillation commencing at  $94^\circ$  C. and gradually rising to a maximum of  $146^\circ$  C. The alcohol passes over quietly, no bumping or frothing occurring. It is recovered in the form of a 15 per cent. spirit, which is subsequently concentrated to 95 per cent. in a column still, the weaker alcohol passing over last being employed for diluting the next batch of acid. Instead of diluting with water, the acid may be passed directly to a distillation plant, in which it is met by a current of steam, which dilutes it and hydrolyses the ethyl sulphuric acid, the alcohol being volatilised by the heat of the reaction.

The sulphuric acid, freed from alcohol, is concentrated in a Gaillard tower and transferred again to the absorption towers.

The alcohol has a persistent and disagreeable odour which renders it unsuitable for the manufacture of organic products

This has been found to be due to the presence of diethylene disulphide, which is not removed by the concentration. The presence of this impurity does not, however, interfere with its use as a motor spirit, and it can be taken out by suitable rectification, yielding a potable spirit.

Several experiments were tried with an 87 per cent. crude spirit for motor purposes, which were quite successful. Thus comparative trials with a 4 h.p. Triumph machine (total weight fully 700 lb.) gave, with Shell petrol, 64.6 miles per gallon; 50 per cent. of benzol and 50 per cent. of alcohol, 78.5 miles per gallon; and alcohol alone, 58 miles per gallon. Similar experiments with a Buick car were also satisfactory. No trouble appeared to arise from the presence of the diethyl disulphide in the crude spirit.

Mr. Bury estimates that at the Skinningrove coking plant, with its 120 Otto regenerative ovens, calculating on a basis of 70 per cent. extraction and 70 per cent. recovery, the weekly production would amount to 9280 gallons of absolute alcohol.

A. A. L. J. Damiens, M. C. J. E. de Loisy, and O. J. G. Piette<sup>1</sup> have introduced into this process a modification by the employment of one or more catalysts for aiding the absorption of the ethylene and formation of sulphovinic acid; for instance, metallic sulphates, sulphur trioxide, phosphoric anhydride, phosphorous anhydride, or arsenious oxide. Good results are obtained by the addition of 1 to 2 per cent. of sulphur trioxide and phosphoric anhydride, or 1 to 5 per cent. of ferrous sulphate, copper sulphate, etc., to the sulphuric acid. The acid employed has a strength of 66° B. (sp. gr. 1.84), and may be used cold, but preferably temperature should be kept at 100° to 120° C. The gas, previous to the absorption of ethylene, is first dehydrated and freed from higher hydrocarbons of the ethylene and acetylene series by passing it through acid previously saturated with ethylene. Ether or alcohol, or a mixture of the two, may alternatively be produced by regulating the amount of water used in diluting the acid. The acid from which the alcohol and ether have been removed by distillation can be concentrated by gas dehydrated in the apparatus, or by waste furnace gases.

<sup>1</sup> *Eng. Pat.*, 152,495, Nov. 16, 1919.

J. Behrens<sup>1</sup> claimed the manufacture of acetic acid from coal, lignite etc., the distillation gases from these products being mixed with carbonic acid and heated to about 400° C. The aldehyde thus formed can be either oxidised or reduced with the production of acetic acid or alcohol respectively. The yields are equivalent to 20 grammes of alcohol or 26 grammes of acetic acid from the ethylenic substances contained in 1000 litres of illuminating gas, about 75 per cent. of the ethylenes being thus converted.

In 1919 the "Statist" reported the result of researches, which had been carried on by the leading Swiss hydro-electric companies in co-operation with the alcohol company, the Lonza Company, Limited, into the commercial possibilities of the manufacture of ethyl alcohol from calcium carbide. The high price of coal is one of the chief difficulties, coal from Germany costing 600 francs for one truck load, but even taking this expense into consideration, the syndicate are of opinion that it will be possible to sell spirit of second-grade quality at £21 2s., and high-grade industrial alcohol at £22 4s. per ton, which is very much lower than the price paid by the Federation for imported spirit. It is estimated that the hydro-electric installations, with a minimum capacity of 20,000 h.p. in the winter and 30,000 h.p. in the summer, would be able to produce 7500 to 10,000 tons of alcohol annually.

The Lonza factory contracted with the Swiss Federal Government to supply alcohol at about 1s. per gallon, but after working for a time and finding it did not pay, they ceased operations.<sup>2</sup>

W. K. Freeman<sup>3</sup> claims to produce ethylene direct from hydrogen by passing the latter through a chamber in which are arranged a number of carbon arcs. The ethylene thus produced is cooled and passed up an absorption tower fitted with superimposed trays filled with acid-resisting material, down which a current of warm sulphuric acid is allowed to flow. Alcohol is obtained from the acid by distillation.

<sup>1</sup> *Ger. Pat.*, 275,049, Feb. 21, 1913.

<sup>2</sup> A. P. H. Desborough, *Jour. Soc. Chem. Ind.*, 1920, 202 R.

<sup>3</sup> *Eng. Pat.*, 28,928, Dec. 15, 1913.

## CHAPTER XI.

### PLANT, ETC., FOR THE DISTILLATION AND RECTIFICATION OF INDUSTRIAL ALCOHOL.

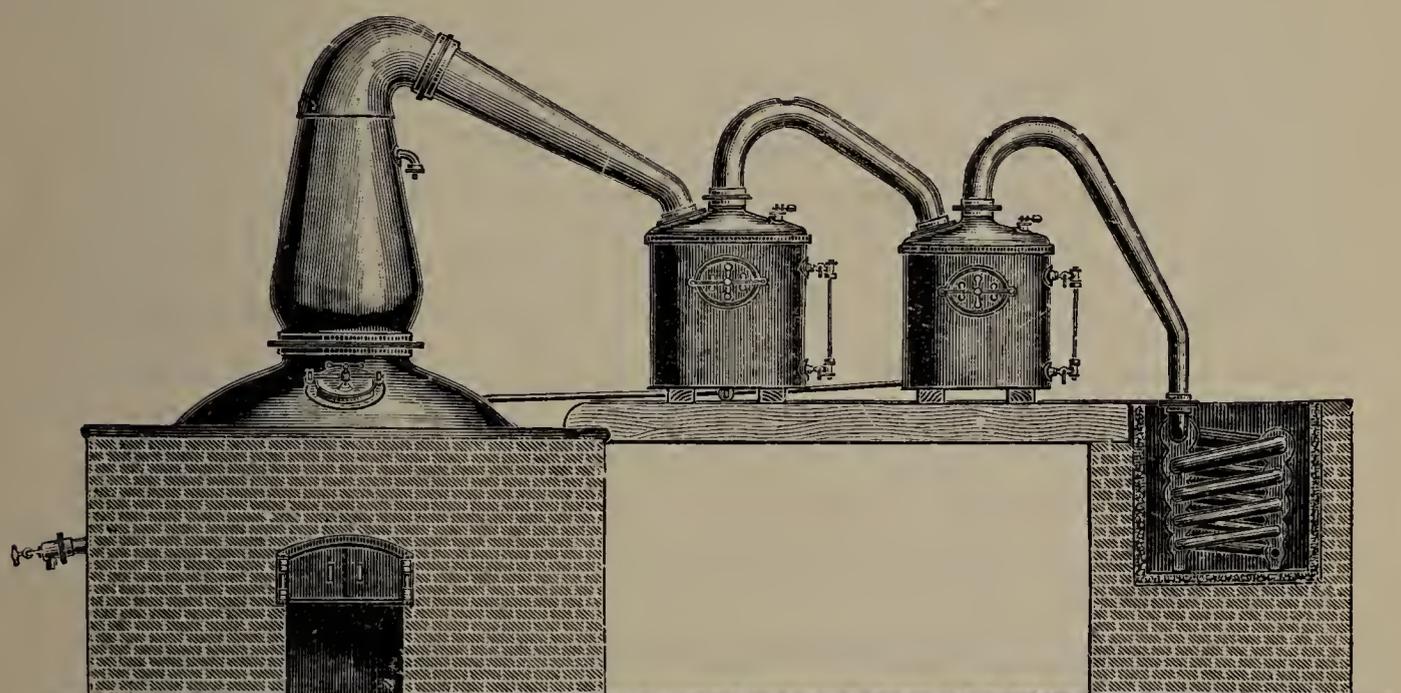
*Water and fuel.*—The purer the water for distillery purposes the better. The distiller could therefore not do better than consult the English edition of De la Coux' "Industrial Uses of Water." Each kind of water must be treated suitably in order to purify it, and to discuss such processes is outside the scope of this book. Practical distillers are accustomed at the end of each season to take into account the quantities of materials and the working expenses to produce a hectolitre of alcohol. This total is, moreover, subdivided under the headings "wages," "acid," "coal," etc; even "lubricants" have a heading. This classification is extremely useful, since each year the distiller can judge by comparison with previous seasons the cause of the increase or decrease in general expenses, and take the necessary measures to remedy any increased expenditure thus divulged. If we compare the cost of manufacture detailed in this way for different distilleries, nothing is more surprising than the enormous differences in the quantity and cost of coal. Some distilleries use per hectolitre of alcohol produced considerably more than others. What is the cause of this anomaly? The cause, or rather the causes, are complex: (a) the quality of the fuel; (b) more or less complete retention and utilisation of the heat generated; (c) equipment; (d) greater or less economy in using the heat. (a) The quality of the fuel. Though in every sugar works analyses of the coal, coke, limestone, and all purchases generally are made, yet few distillers test their coal. It would, however, be rational when trying to reduce the coal bill to take quality into account. It may be objected that if the quality be inferior the price is low,

and that the price per heat unit ought to be the same in all cases. That should be so, but it is not always so, and distillers may pay dear for indifferent coal with much ash. In any case, coal with a high ash content gives very little heat, causes dirty fire-bars, which require constant raking to keep clean, and much labour in removing and tipping the clinker. (b) More or less complete retention and utilisation of the heat generated. Here, again, there are great variations between one distillery and another. Sometimes it is a bad stoker who rakes his furnaces too much about at one time, and loses in the form of black smoke much of the heat that would otherwise be generated; again, it is the fire-bars that are defective, producing ashes containing a considerable proportion of imperfectly burnt cinders; later on, it may be the boilers themselves—perhaps of an obsolete pattern—which do not allow the economical use of the heat generated. Or it may be hard water, which deposits a hard layer of encrustation in the interior of the boilers, and this, gradually increasing, prevents the complete utilisation of the heat generated. (c) Equipment. It is a fact that some machines require and use up more heat to produce the same useful effect than more improved plant. (d) Greater or less economy in using the heat. In beet distilling the volume of juice withdrawn may be reduced; also, much of the hot vinasse is used over again, representing heat gained or saved.

*Plant.*—This point merits careful consideration. When plant becomes obsolete or worn-out, the question of what plant to put in place of it is an important one. The first stills used for the distillation of fermented liquors were *intermittent pot-stills*. The pot-still consisted of a kettle or pot built in brickwork over a furnace and partly filled with the fermented liquid to be distilled. The still head of this pot communicated directly with a condenser consisting of a copper worm suitably cooled. Subsequently (about 1800) attempts were made to *fractionate* the vapours by the intervention of vessels on the principle of the Woulfe's bottle, as shown in Fig. 40. This type of still is commonly used in Jamaica and in the West Indies generally. The old intermittent pot-still and the type just described are now used only for the production of certain potable spirits of superior quality—rum,

whisky, etc., in which the aroma of the alcoholic vapours and the spirit condensed from it, more or less modified by the heat, are regarded with special favour, and the spirit is therefore valued very highly by connoisseurs on that account.

When a mixture of pure alcohol and water is heated in a pot-still to its boiling-point, vapour is given off, and since alcohol is more volatile than water, the vapours given off in the commencement are rich in alcohol (Table VII.). Then, as the contents of the pot-still become exhausted of alcohol, they become more



**Furnace.**  
Fire-heated still showing manhole, discharge valve, dome still head fitted with pipe leading to retort (in older forms direct to condenser).

**Retorts or high and low wine vessels, with gauge glass and vacuum valves.**

**Condensing worm.** (In the ordinary pot-still the condenser connects directly with the still head without intervention of the two intermediate vessels.)

FIG. 40.—Fire-heated still with fractionating apparatus on the principle of the Woulfe's bottle (BLAIR, CAMPBELL, & MCLEAN, Glasgow).

and more aqueous, until, finally, the whole of the alcohol is eliminated, which occurs when about three-fifths of the liquid is distilled. The first portion of the distillate contains a large proportion of the more volatile alcohol, and the second a large proportion of the less volatile water. But when, for instance, the wash or liquid formed by the fermentation of malt is distilled, the first portion consists of a very volatile product, the "fore shot;" then alcohol mixed with water distils; and, lastly, a substance less volatile than alcohol—the fusel oil—passes over.

But these can only be completely separated from each other by appropriately constructed fractionating stills, the introduction of the two intervening Woulfe's bottles being a preliminary attempt at such fractionation. This apparatus aided in concentrating the alcohol and in the separation of the roughest impurities from the distillate without materially injuring the natural aroma. But let us examine what occurs in the simple pot-still a little more closely. When one charge of the still has been distilled, if a stronger alcohol be desired the still is emptied, and the same operation repeated on the distillate, until, after a series of successive distillations, alcohol of the desired strength is obtained. The fermented wash, in the first instance, consists of (1) non-volatile or very slightly volatile substances, including mineral salts, proteins, yeast, glycerin, succinic acid, lactic acid; (2) volatile substances—alcohol, water, odoriferous oils (fusel oil), and a little acetic acid. When this liquid is boiled, the vapours formed consist essentially of water and alcohol, and the condensed distillate is therefore a mixture of alcohol and water. Since pure alcohol boils at  $78.3^{\circ}$  C., and water boils at  $100^{\circ}$  C. under a barometric pressure of 760 millimetres, it will at once be seen that the richer the wash in alcohol the lower is the temperature at which it will boil. Again, just as the more volatile the substance the less heat is required to convert it into vapour, so also the temperature at which that vapour is liquefied is lower than that required to condense the vapour of a substance which boils at a higher temperature. Practice has, moreover, shown that the vapour of alcohol which is disengaged from boiling wash behaves towards the wash itself as if a current of gas were injected through the liquid at the same temperature. Steam is therefore entrained, and the more abundantly as the temperature approaches  $100^{\circ}$  C., the boiling-point of water. Pure alcohol cannot, therefore, be extracted from fermented liquors by boiling in a pot-still. But by passing the mixed vapour simultaneously through a condensing arrangement, the temperature of which is lower than the boiling-point of the less volatile, but not as low as the boiling-point of the more volatile, the vapour of the less volatile liquid will be condensed, the more volatile retaining its gaseous form.

Again, as the temperature at which the fermented wash boils increases its alcoholic strength decreases, the proportion of steam in the vapour becomes greater until the boiling-point of water ( $100^{\circ}$  C.) is reached, the vapour then containing no alcohol. When the mixed vapours are led through a pipe laid in water at a temperature below  $100^{\circ}$  C., but not as low as the boiling-point of the mixed liquid, the aqueous vapour is liquefied without affecting the alcoholic vapour until the temperature of the condenser sinks below  $78.3^{\circ}$  C. ( $176^{\circ}$  F.), when alcohol will condense. This occurs with the pot-still, the aqueous vapour being partially condensed in the first two or three spirals of the worm, whilst the alcohol retains the gaseous condition until it reaches a spiral of the worm where the temperature is below the boiling-point of alcohol, when it also is liquefied. (The cold water enters by the bottom and leaves by the top of the tub containing the worm, hence the lowest spiral is exposed to the coldest water and the top spiral to the hottest. The whole of the water and much of the other impurities which pass over with the alcohol are thus found in the condensation products of the pot-still.)

In intermittent distillation by the pot-still, therefore, the vapours contain at the outset *much alcohol* and little water, then more water and less alcohol, finally, they consist entirely of water. Therefore, since the temperature at which water boils is  $100^{\circ}$  C. ( $212^{\circ}$  F.), and that at which alcohol boils is  $78^{\circ}$  C. ( $176^{\circ}$  F.), if a mixture of these two liquids be distilled, the mixture will boil at an intermediate temperature proportional to the amount of each in the mixture, and the boiling-point of the liquid being distilled is an indication at any given moment of the percentage of alcohol present therein. Hence the necessity of repeated redistillations to obtain an alcoholic liquid of the desired strength, resulting in loss of time and entailing great expense in fuel, and therefore, as previously mentioned, intermittent pot-still distillation is now used only in the preparation of whisky, rum, brandy, and other potable spirits.<sup>1</sup>

<sup>1</sup> There are several varieties of pot-stills of very large capacity used in the manufacture of whisky and rum, the makers of the still shown in Fig. 40 and other distillery constructors listing a variety of these, but as this is not a treatise on the manufacture of potable spirits further description does not come within our province.

*The use of soap in the distillation of alcohol.*<sup>1</sup>—As the use of soap, although empirical, was brought prominently to notice in the famous “What is Whisky?” case, it may be as well to consider its use and abuse. “To arrest the formation of acetic acid,” says Muspratt, “as soon as the attenuation of the wort has reached its lowest point it is run into the still with as little delay as possible.” According to what Muspratt called the old methods, the wash was distilled in two large iron retorts or stills, each of about 600-1200 gallons capacity, according to the size of the factory. The retorts, he says, were provided with a rotary chain to prevent the lees adhering to the bottom of the still, which otherwise would deposit and become charred from the heat, thus communicating a disagreeable taste to the spirit. Previous to distillation, about 1 lb. of soap was added to every 100 gallons of the wash. When the charge of wash was 8000 gallons the distillation was carried on as speedily as possible without risk of it running foul, till about 2400 gallons were drawn off. These constituted the low wines or singlings, which were very weak, averaging not more than 63° below proof on Sykes’ hydrometer. The remainder of the spirituous product of the 8000 gallons was received in another vessel for a further distillation. The singlings were redistilled and doubled in the second still, the spirit being drawn off until it began to acquire a disagreeable taste and smell; these were what constituted the feints, and owed their peculiarity to an essential oil which was held in solution (fusel oil). The feints were collected in the feints back and were mixed with the turbid portion of the first distillate, water being added, and the whole redistilled. Very weak singlings were thus obtained, which upon a second distillation afforded finished spirit. Some distillers continued the first distillation as long as any alcohol came over, and then subjected the low wines to a second distillation in the spirit still. The first portions were more or less blue or turbid, and consequently were run into the feints back. As soon as the spirit became clear and free from disagreeable odour it was run into the spirits back. The last runnings feints

<sup>1</sup> Soap as a froth preventative is now obsolete, so also are mechanical froth preventatives like B, Fig. 42.

were mixed with the first portions. These feints were mixed, as before stated, with a considerable quantity of water, and distilled in order to free them from the disagreeable oil derived from the husks of the grain. In some distilleries a self-regulating bath was put in the capital of the still. The common Scotch stills at that time had the capital 15 to 20 feet high to prevent the wash from boiling over into the worm; it was customary to tap the capital from time to time with a rod, and from the sound emitted it was inferred whether it was empty, partially filled, or in danger of an overflow, in the latter case the fire being withdrawn or damped by means of water drawn from a pipe near the furnace door supplied from a cistern in the upper part of the building. When a very pure spirit was required, it was customary to dilute the distillate with water and submit it to a third distillation, in order that the distillate may not have the harsh taste of strong alcoholic liquids. In the improved stills a spirit of 60 per cent. O.P. was obtained even in the first distillation, and with a considerable saving of fuel, time, and labour, while the use of soap was unnecessary. But in modern stills spirit of 67 O.P. is now easily produced.

As a general rule, the lower the temperature at which the distillation is carried on, the purer will be the spirit. When an excess of soap has been used and the distillation urged too rapidly, the distillate often possesses a soapy or fatty flavour, which is occasioned by particles of the liquid being carried over mechanically in the vapour and dissolved in the alcoholic liquid. Muspratt proceeds to explain the manner in which the soap acts to prevent the charge running foul, as follows. During fermentation and the subsequent transference of the wort into the still, small proportions of acetic acid are generated which decompose part of the soap, setting free the oily compound (fatty acids), which then rises to the surface of the liquor and break the bubbles of vapour as they ascend through it from the bottom of the retort, hence the liquid cannot pass over unless the boiling be violently urged. But Muspratt's explanation of the rational and judicious use of soap for such a purpose is far from complete. If used, it should be in such proportion as to exactly neutralise the free acetic acid,

and the still hardly seems the right place in which to do this, although, it may be the only feasible one. It is the fatty acids which act as the froth preventer, not the soap. Similar inconvenience occurs with the boiling of beet juice, but in this case oil is used, not soap. Unless the alcoholic liquor and the soap solution be titrated and used in exact proportions, an excess of soap solution is bound to occur as often as not, and will certainly lead to priming. If the soap solution be intended to prevent frothing, slight excess will accentuate the evil, frothing and priming will then occur to a greater extent. Excessive heating

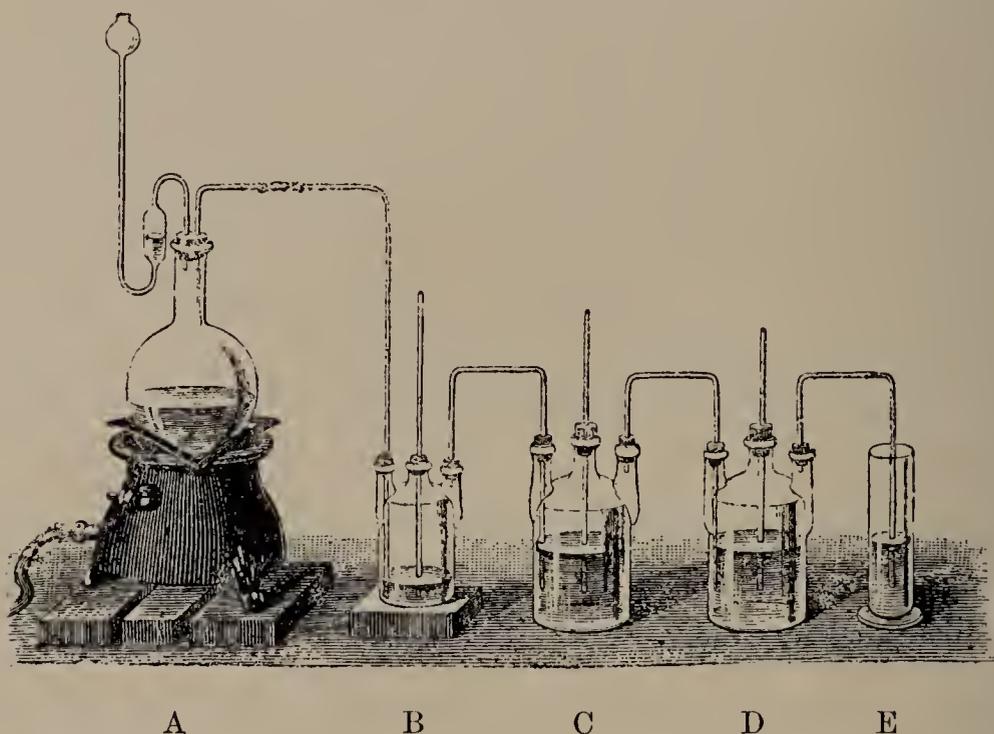


FIG. 41.—Diagrammatic representation of principle of Woulfe's bottle. A, flask fitted with safety funnel and delivery tube; B C D, Woulfe's bottles fitted with inlet and outlet bent tubes and intermediate safety tube; E, glass jar.

is not always the cause of priming with soapy liquors or liquors having a tendency to froth. Very often the still acts imperfectly, or boiling ceases for a time and then suddenly it commences to prime, and even if the heat be instantaneously withdrawn, the distiller can do nothing whilst the still empties itself through the worm. Soap is therefore a dangerous material to put into a still. Happily, with continuous rectifiers, it is unnecessary, and enormous bulks of liquid are not now subjected to heat. Moreover, the passing of the alcoholic vapour through Barbet's marble or limestone scrubbers eliminates all acidity.

A considerable improvement was made in the methods of

distillation when it first became practicable to obtain strong alcohol from fermented wash in an intermittent fire-heated still in one operation. The method of saturating water with gaseous vapours in a series of Woulfe's bottles had been known for a time long prior to the adaptation of this principle to the vapours evolved on distillation. Edouard Adam, a workman from Rouen, settled at Nîmes, near Montpellier, attended a course of chemical lectures at Montpellier, and listened to a discussion on the utility of Woulfe's apparatus. The idea then occurred to him that by applying the principles of the Woulfe's apparatus to the condensation of the vapour from a spirit still strong alcohol could be obtained in one operation. He caused the boiling-hot vapours to drive the spirits successively out of one bottle into the other, so as to obtain in the successive vessels alcohol of any desired strength and purity "at one and the same heat." Adam obtained a patent for his invention on May 29th, 1801, and soon afterwards, as a result of his success on the small scale, was able to erect a complete distillery which excited the admiration of all the practical chemists of that day. In November, 1805, he obtained a certificate for certain improvements for extracting from wine in one process the whole of its alcohol. Twenty distilleries (*bruleries*) were erected in the South of France for operating his system, with a capital of over 1,000,000 francs. Like Count Rumford, Adam thus discovered the principle of heating liquids by the condensation of their vapours. He applied it to the distillation of wine, and caused a quantity of it to boil by the transmission through it of the vapour from another portion of the same liquid. His efforts were crowned with success; he obtained at the commencement spirit at 33° Cartier instead of brandy. In six hours he distilled in one of his stills 400 velts of wine, or about 679 imperial gallons, from which he got by a single distillation 58-60 velts—100 gallons, practically, of 90 per cent. spirit. This apparatus was arranged so that the vapours rising from the still passed into a series of egg-shaped vessels partly full of wine, and there condensed until the wine reached the boiling-point owing to the latent heat liberated by the condensing vapours. The wine thus heated and rendered more alcoholic in each succeeding

vessel, gave off spirit which passed into a series of smaller empty vessels, where it deposited in transit the more aqueous portion, the phlegm of the distilleries, the amount of which diminished in each succeeding vessel. The most volatile portions were at last condensed, first in a condenser cooled with wine, then in another cooled with water. But the Nemesis which seems to shadow the fortune of all inventors began to pursue Adam. It appears that he was so overjoyed after making his first experiments, that he ran about the streets of Montpellier telling everybody of the surprising results of his invention. This created several rivals, the most formidable being Isaac Solimani, Professor of Chemistry in Montpellier, who obtained a patent involving much the same principle, but his patent was dated somewhat later than Adam's, viz., in July, 1801, therefore Solimani's claim of priority fell through. Several other adaptations of the still and condenser on Woulfe's principle were introduced, Berard's, patented on August 16th, 1805, being the most important. The Adam's still, however, was most used; not so much, it is said, on account of its merits, for it was regarded as considerably inferior to both Solimani's and Berard's, but because of the alleged quarrelsome disposition of the patentee, whose cupidity, it is further alleged, led him to suppose, as his brevet specified, that the whole of the alcohol could be obtained from wines when distilled in his apparatus, all other inventions being regarded by him as infringements on his rights, and the lawsuits to which he exposed those introducing any new invention prevented the use of any other improved form of still. After realising a handsome fortune by his own distillery and the proceeds of his patent, he became so involved in lawsuits, which he ultimately lost, that expenses and costs reduced him to complete penury, in which state he died. Some at least of Adam's compatriots, however, revere his memory. Girardin especially credits him as endowing the South of France with an industry which has brought her many millions. But however that may be, and notwithstanding the many good points of the stills of Adam, and also of those of his rivals, they all suffered from one grave defect. They were *intermittent*. Labour, fuel, and above all, time was lost by the

cooling of the stills for the discharging of the spent liquor, recharging the stills, and again getting up sufficient heat to carry on the distillation. If Berard's still was less complex and therefore more easily managed, yet it consumed more fuel than the others, owing to the fact that it was necessary to frequently discharge and recharge the still.

*History of continuous distillation, continuous rectification, and continuous distillation with simultaneous rectification.*—The first to attempt the construction of a continuous still was Baglioni. His efforts were, however, not attended with any great success; the subject was further studied by Cellier, Blumenthal, and Derosné. Blumenthal constructed a continuous still, which afterwards became the property of Derosné, who still further improved upon it. Armand Savalle also took the matter up, first in collaboration with Blumenthal and Derosné, but soon surpassed them in the perfection of his plant. Derosné immediately erected important workshops for their construction, and associated himself with Cail, who became a celebrated engineering contractor. As to Armand Savalle, he continued to improve the plant all his life, first in collaboration with his young son Desiré Savalle, and at the end of his career, abandoning industrial practice, he became a consulting engineer. His son Desiré Savalle succeeded him in the firm, and successfully created what are claimed to be the most improved types. Desiré Savalle was succeeded by Albert Savalle, the grandson, who became the head of this old firm. Other eminent French designers of distillery plant are Egrot and Grangé, a very old-established firm, and Emile Barbet. The latter was the first to bring continuous rectification to a successful issue, and not only continuous rectification, but simultaneous continuous distillation and rectification. Within recent years, Guillaume, a former pupil or assistant of Barbet, claims to have improved upon the work of his former patron; the latter has, however, most adversely criticised the work and claims of his former subordinate. The latter has *inter alia* invented an inclined distilling column, which, at any rate, has the merit of novelty, and is easily dismantled for cleaning purposes. Messrs. Blair, Campbell, & McLean, of



into the bottom of the alembic A ; *e*, a tube to lead the alcoholic vapours, generated in A, into the bottom of B, which vapours, in passing through the liquor in B, heat it, and are partially condensed ; *f*, glass tube to mark the level of the liquor in B ; *g* and *g*, level indicators ; *h*, pipe conducting the wash from the lower part of the wine-heater E upon the uppermost of the series of horizontal discs, mounted within the distillation column ; *i*, a stop-cock for emptying the wine-heater at the end of an operation ; *ll*, two tubes fitted to the wine-heater E, of which the first descends into the last compartment of the rectifier, whence it rises to the fifth ; and the second tube descends to the third compartment, whence it rises above the second. At the curvature of these two tubes stop-cocks *l* and *k* are placed on them, for drawing at pleasure a sample of the liquor returned to the rectifier ; *m*, *n*, and *o* are tubes communicating on one side with the sloping tube *p*, and on the other with the tube *l*. These three communications serve to furnish a spirit of greater or less strength. Thus, if a very strong spirit is required, the alcoholic vapours which condense in the worm enclosed in E are all to be led back into the rectifier D, to effect which purpose it is requisite merely to open the stop-cocks *n* and *o* ; again, weaker spirits may be obtained by closing the stop-cock *o*, and still weaker by closing the stop-cock *n* ; for in this case the alcoholic vapours condensed in the worm within E will flow off into the worm within the upright cooler F, and will get mixed with the richer vapours condensed in this refrigerator. The interior of the column C contains a series of movable concave scale pans (like those of a balance), with spaces between, each alternate pan having the convex side turned the reverse way to the preceding one, for the purpose of prolonging the cascade descent of the *vinasse* through C, and exposing it more effectively to the heating action of the ascending vapours ; the edges of these pans are, moreover, furnished with projecting points of copper wires, to lead off the liquor from their surfaces in a fine shower. The interior of the rectifier column D is fitted with a series of shelves, or floors, the passage from one compartment to that above it being through a short tube, bent at right angles, and open at both ends ; *p p p* is a series of tubes

for receiving the vapours condensed in each of the turns of the large serpentine within E. The axis of this worm is horizontal ; *q q q*, sight-holes in the top of the wine-heater ; *r*, a tube to conduct the alcoholic vapours not condensed in the worm of E, and also, if desired, those which have been condensed there, into the worm of the refrigerator F ; *s*, a tube to bring the *vinasse* from the reservoir G into the lower part of the refrigerator F ; *t* is a tube which conducts the wine from the top of the refrigerator F to the upper part of the wine-warmer E ; *u* is the funnel opening of the pipe leading the wine from *g* to the refrigerator ; *v*, a stop-cock regulating the flow into the tube *t* ; *x*, a tube conducting the finished spirit from the refrigerator. It contains a hydrometer to indicate the strength. The above explanation of Fig. 42 will sufficiently explain the general principle of the working of this still. The internal arrangements of the still, especially of the condenser, and the general working of the still, were somewhat intricate and over-elaborate, but as it is now obsolete we need not dwell upon it further here.

*Coffey's still*.—This ingenious, original, and powerful apparatus for distilling spirits from fermented worts or wash of all kinds was, after many struggles with the illiberal prejudices of the Excise, at last universally recognised as the best, most economical, and surest in a revenue point of view, of all the contrivances of obtaining alcohol, in the purest state, and of any desired strength, at one operation. Its outer form and internal structure differ essentially from those of all the older stills, though it possesses some of the good principles of Derosné, in continuity of action, and in causing a current of spirituous vapour to ascend, and a current of wash deprived of its alcohol to descend, in one system of continuous cells. The main structure consists of a series of wooden planks, 5 or 6 inches thick, fixed one above another, the joints being covered, or the whole being lined with sheet copper, so that the apparatus resembles a large chest, to which is attached the induction pipe of a steam boiler. as the active principle of the whole. The essential apparatus consists of three main parts : the wash collector A A A and the two rectangular columns or uprights. The front column D D D, or the analyser, is for recti-

fying the wash, the other column is intended for warming the wash; the under part F F F of the forewarmer serves as a dephlegmator and for the rectification of the feints; the upper part E E E serves to condense the strong spirituous vapour. The wash collector A is divided into two compartments B and C, by means of the copper plate *c c*; this plate *c c* is pieced like a drainer with a number of small holes, and is provided also with T-shaped valves *o o o*. The wash rectifier D is divided by the plates *r r*, of a like drainer construction, into twelve chambers, and the feint rectifier F F into ten chambers by similar plates *s s s*. These

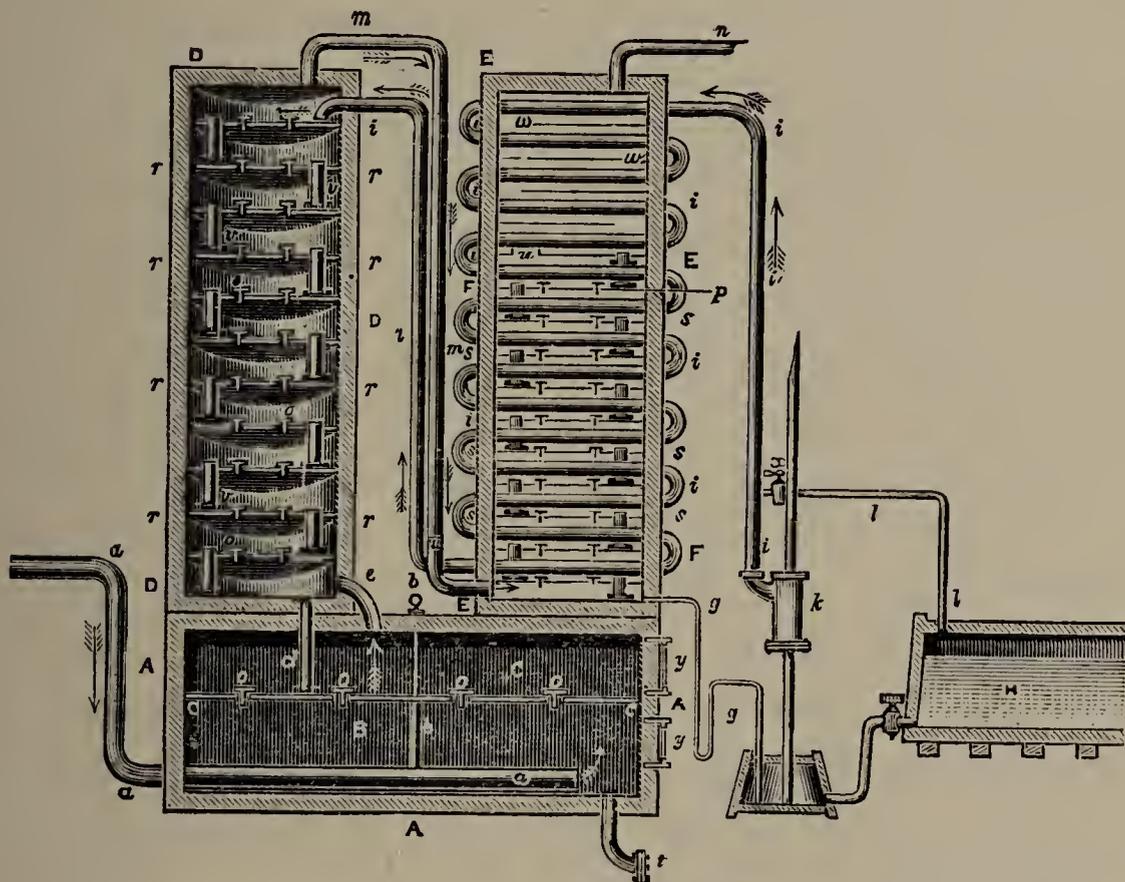


FIG. 43.—Coffey's still, section showing working.

orifices are so narrow as to allow only the passage of the rising vapour, but prevent the downward flow of the liquid resting on the plates, which passes downwards through the adjunct tubes *e* into the wash collector B, *v* into the rectifier D, and likewise into the dephlegmator F, passing successively from one chamber to another. When the steam pressure is too strong, the valves *o o* allow it to escape.

When the apparatus is in action, a continuous stream of wash is raised out of G, by means of the pump *k*, into the tube *i*, which feeds the still. This current must be regulated very carefully,

so as just to feed the tube *i*, allowing the excess to return through the stop-cock *x*, and the tube *l* back into the wash cistern H. The tube *i* enters into the uppermost partition of E, forming seven zigzag bendings in this space, and through F, and then mounts upwards from that chamber into the top chamber of D. Thence the wash flows down from chamber to chamber, and arrives through *d* into C, and finally in a similar way into B, where it is fully deprived of spirit, and is from time to time run off through *t*. It is necessary throughout that the wash in this passage into D and B should stand about an inch high upon each plate *r r*, for which purpose the adjunct tubes *v* should stand an inch above the plates, and thus give the vapour no indirect passage, as the under end of each tube *v* dips into a shallow cup and is thus shut in by the wash remaining in it. The tube *d*, which leads the wash from the plate *c c* into C, serves a like purpose. As soon as the wash has risen in it to the upper orifice of the glass tube *y*, the valve *b* is opened to allow it to flow off into B through the tube *b*. In B the very hot and nearly spent wash comes into contact with the steam, issuing from the steam boiler through the steam tube *a a*. The steam rushes through the liquid and carries off the spirit from it through the small orifices in the plate *c*, expands thus into the whole breadth of this chamber through the wash standing in it, and deprives this at once of every trace of spirit, then collects over the fluid and enters through the connection tube *e* into the undermost chamber of D, and thence into the following in succession always through the orifices of the plates *r r*. Whilst the steam meets the wash in each chamber and becomes more spirituous the higher it mounts, at the same time it becomes cooler and deposits the watery part, absorbing more alcohol, so that after this complicated rectification it passes on through the tube M M into the lowest chamber of the forewarmer J. Here it travels upwards through the plates S S, where the feints are at the same time rectified by the dephlegmation of the vapour. The steam flows through the different junction tubes into F and its subdivisions, whereby, as the wash in D forms on each plate, a layer an inch thick is to be penetrated by the steam. The remainder passes

out through the undermost plate through the tube *g g* into G, where it is carried by the pump with fresh water into circulation in the apparatus. The alcoholic vapour now reaches E. The plate which separates E and F is not perforated, it lets the vapour merely pass through the short and wide junction tube *u* into the condenser E, where in like manner the non-perforated plates *w w* compel it to follow the zigzag bendings of *i i*, so as to complete its condensation and the heating of the wash in *r*. The completely condensed vapour is collected on the bottom of E, and is conducted out of the cup of the junction tube there (which is larger) through the annexed tube sideways at *p* into the refrigerator not shown in the figure.

*Continuous working steam stills on Coffey's patent principle.*<sup>1</sup>

This apparatus is constructed to produce alcohol continuously at a strength of 66 to 67 O.P., or 42° to 43° Cartier. The still consists of an analyser and a rectifier column, both built of copper frames in flanged sections, and jointed together with wrought-iron flanges, screwbolts, and nuts. Both columns are provided with the necessary copper diaphragm plates, with their connections and fittings; and the rectifier column has, in addition, seamless copper or brass washpipes, with inside and outside bends, as shown. A refrigerator is provided for the spirits, and a condenser for the overhead and feints vapours. A copper hot feints vessel, with connections, is also supplied; a sampling apparatus of improved construction, a spirit proof jar or test case, and the necessary mercurial or steam gauges, with copper pipes, cocks, valves, and connections to make the still complete. This type of still is very economical, both as regards fuel and water, and will produce the strongest spirit continuously, at the same time entirely exhausting all the spirit from the wash. Two steam-pumps of the horizontal type are usually supplied, one for pumping wash, and one for water, but an additional pump for feints is sometimes supplied for the larger sizes. A, analyser; B, rectifier; C, overhead vapour and feints condenser; D, spirits refrigerator; E, hot feints receiver; F, wash-pump; G, water-pump; H, spirit test case; K, reducing valve for steam; L, steam stop valve; M, cold feints receiver (constructed of wood). Fig. 45 represents

<sup>1</sup> Figs. 44, 45.

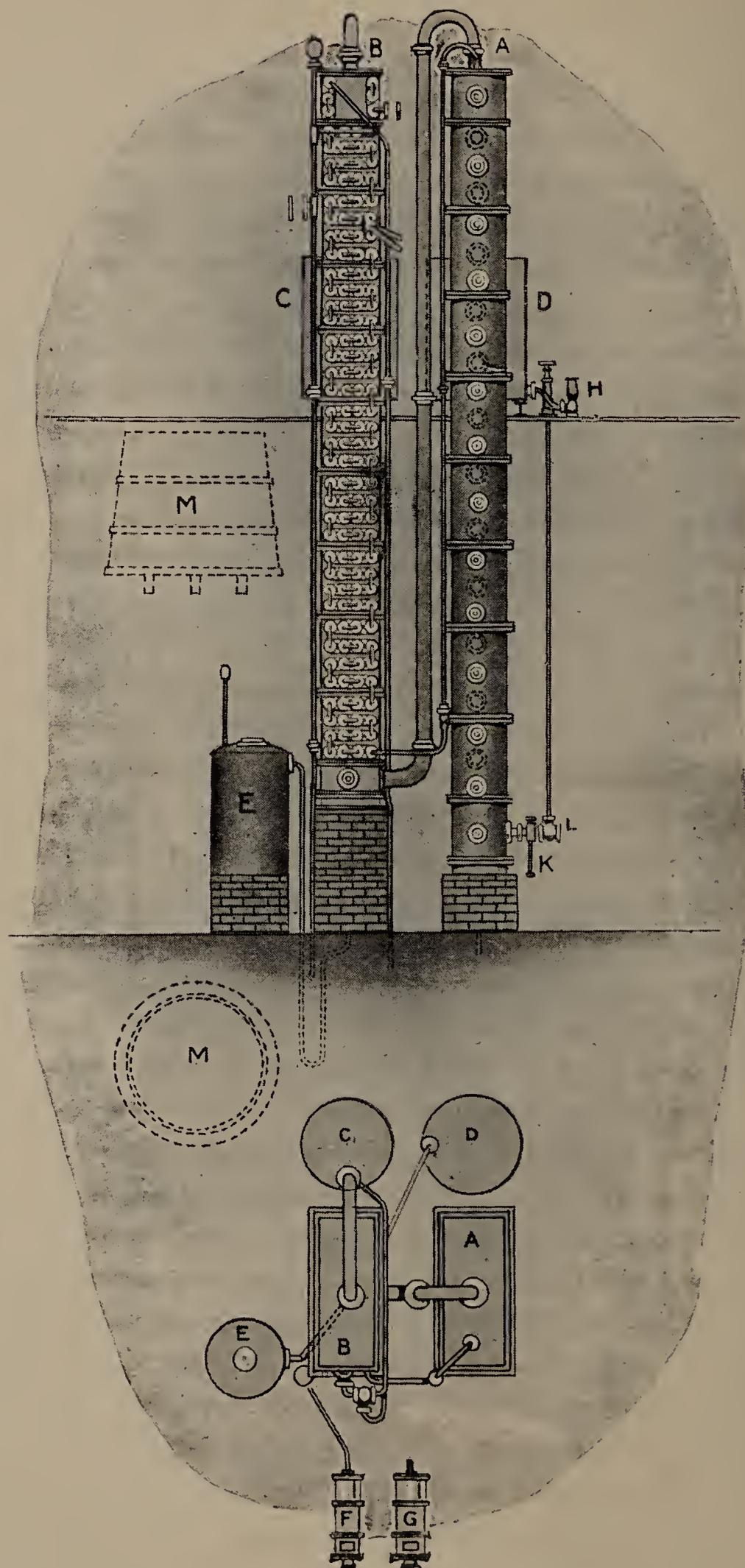


FIG. 44.—Continuous working steam-still of the Coffey type with copper frames  
(Messrs. BLAIR, CAMPBELL & MCLEAN, Ltd., Glasgow).

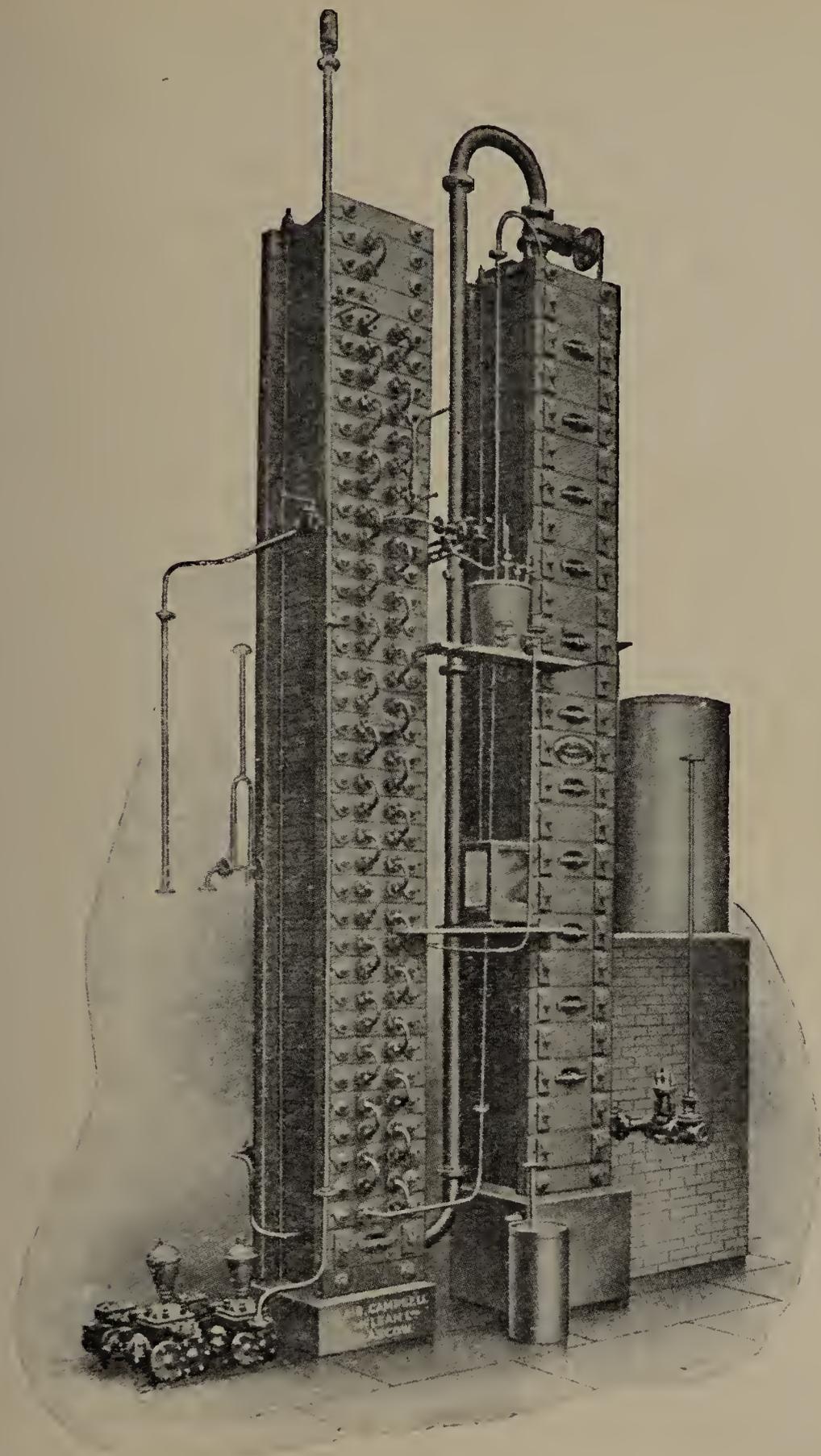


FIG. 45.—Continuous working steam-still of the Coffey type with wood frames  
(Messrs. BLAIR, CAMPBELL & McLEAN, Ltd., Glasgow).

Messrs. Blair, Campbell & McLean's continuous working steam still, on "Coffey's" principle, with all the latest improvements, for the production of pure spirit of best quality at  $43^{\circ}$

Cartier in one operation continuously. This still consists of an analyser and a rectifier column, each built of wood frames in sections, and bound together vertically and horizontally by means of wrought-iron tie rods. The columns in this case are also provided with the necessary copper diaphragm plates with their connections and fittings, and the rectifier column has, in addition, seamless copper wash pipes, with inside and outside bends as shown. The copper spirits overhead and feints worms are all tinned inside and outside, the tank for the worm being of cast-iron: the hot feints vessel and connections, sampling apparatus, spirit test safe and lockings, and the necessary mercurial and steam gauges, copper pipes, cocks, valves, and connections are also provided. The spirit measuring vessel (if required) is made in two compartments, tinned inside, and provided with inlet and outlet connections with lockings and graduated scales.

This apparatus is also most economical in the consumption of fuel and water, producing automatically and continuously the strongest alcohol, with entire removal of spirit from the wash.

The wood frames (even in the warmest climates) last many years, and can be easily replaced at small cost, when worn out. These stills are easily worked and require but little attention.

In the first distilling columns made by Savalle about 1870, the plates were perforated. These were cheap and simple, and, while new, worked perfectly; but the perforations of the plates through which the vapours passed became enlarged, and the proper ratio between the passage of vapour and the work accomplished ceased. This entailed loss of alcohol in the *vinasse*, a loss which continued to increase in proportion to the wear and tear of the plates. To cope with this evil the plant shown in Fig. 46 was designed. The construction and arrangement of the different parts of this system is such that the apparatus is constantly adapted to the rate of flow of the material to be distilled, which is 40 centimetres ( $15\frac{3}{4}$  inches per second). The apparatus was distinguished (1) by its heating being adjusted by a steam regulator; (2) by the method of regulating the feeding of the

liquids to be distilled ; (3) by its wash-heater, with great heating surface, which utilised the heat of the alcohol vapours to raise the temperature of the cold wash entering the apparatus ; (4) by froth preventers, which secure products which are less acid and free from entrained impurities ; (5) by the tubular refrigerator, the interior arrangement of which reduces the condensation water required to one-half ; (6) by the special arrangement of the column plates, with great bubbling surface, where each litre of liquid to be distilled is subjected to a sheet of vapour representing in the larger apparatus 656 feet. *Working the column.*—(1) Set wash and cold water pumps to work to fill top reservoirs. (2) Fill the refrigerator D with cold water. (3) Fill the wash-heater C and also the plates of column A. (4) Close the water tap 3 and wash-feed tap 2. (5) Turn on steam gradually to heat all the plates of the column, and expel, without bumping, the air contained in the wash-heater and refrigerator. (6) When alcohol flows from test-glass E, open the refrigerator tap 3. (7) Open gradually the wash-feed tap 2. (8) Here a difficulty occurs. It is necessary to find a suitable rate of wash-feed, not too great, which would stop the production of alcohol at the test case, and, on the other hand, not great enough to maintain the product at the right strength. It is determined by the feed-tap and its dial indicator. (9) To satisfactorily determine the point it is necessary that the wine reservoir be always full to the same level. It must therefore be constantly fed by the pump, and the overflow from the reservoir must return with the aspiration of the pump. (10) The heating steam should be cautiously applied at the outset until the alcohol reaches the test safe, when the steam regulator acts. (11) To stop working, close tap 2, then shut off steam. If the stoppage is from Saturday to Monday, before starting again let the steam act a little longer, so as to expel all the alcohol.

*Test-glasses.*—Savalle's gauge test-glass (Fig. 46) measures with great precision the actual output of a distilling or rectifying apparatus. The principle of its construction depends upon the flow of liquids through a thin partition. Its essential part is shown in Fig. 47. The alcohol from the condenser enters by the

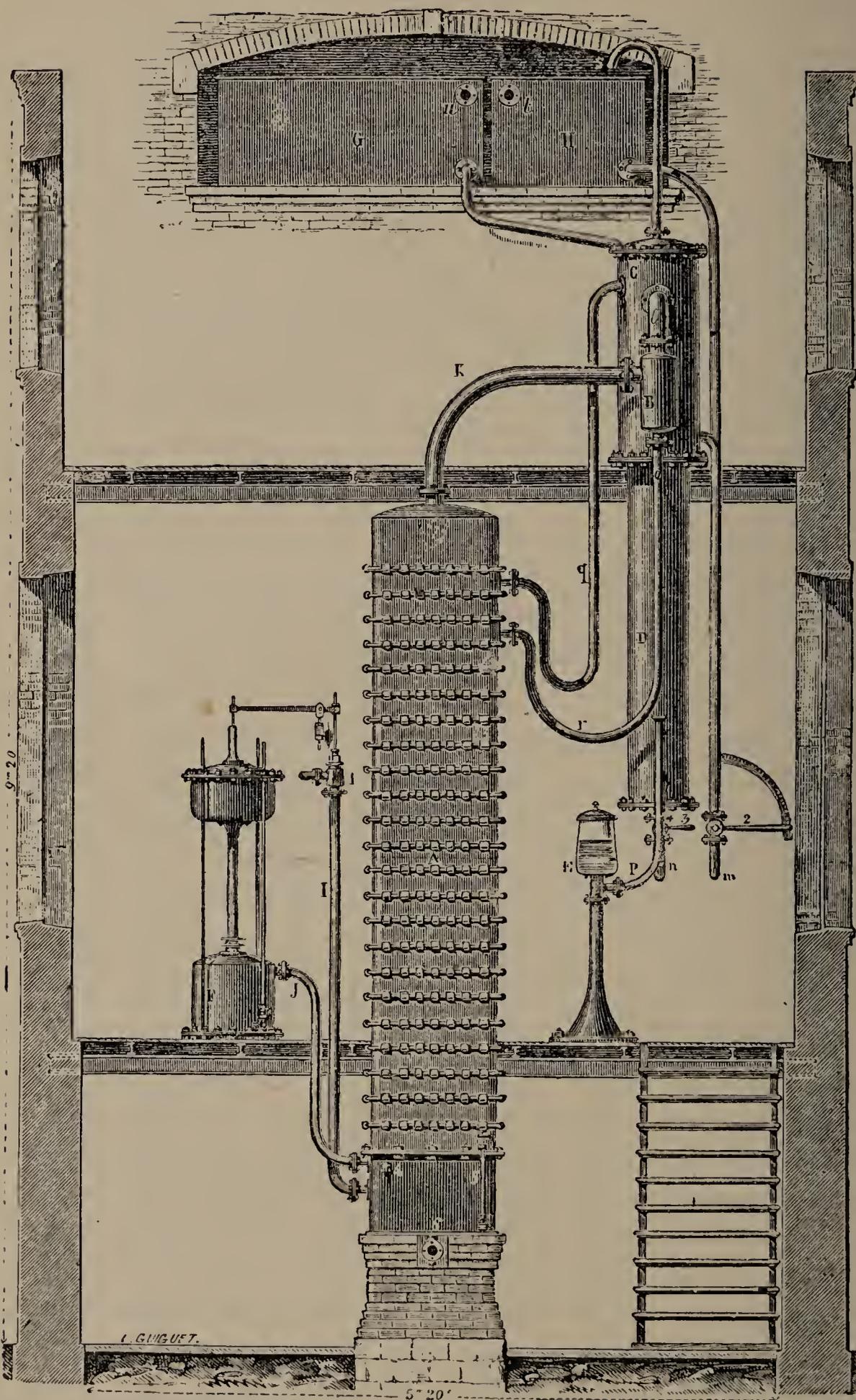


FIG. 46.—Steam distilling column (SAVALLE). A distilling column to distil 8800 gallons wash in twenty-four hours. A, rectangular plate column; B, froth preventer; C, wash heater; D, refrigerator; E, test-glass; G, water tank; H, wash tank; I, J, steam regulator.

bent tube, rises by the tube C into the crystal test-glass, and escapes by a fixed orifice F made in the graduated tube. The level of the alcohol rises in the test-glass until the discharge by the graduated orifice exactly balances that entering the test-glass. The least variation in the output of the apparatus alters the level in the test-glass, and this is determined by noting the oscillations of the level by means of the graduated tube. The outflow orifice is determined; thus the level of the liquid marking 15, an outflow of 100 litres per hour, corresponds to an outflow

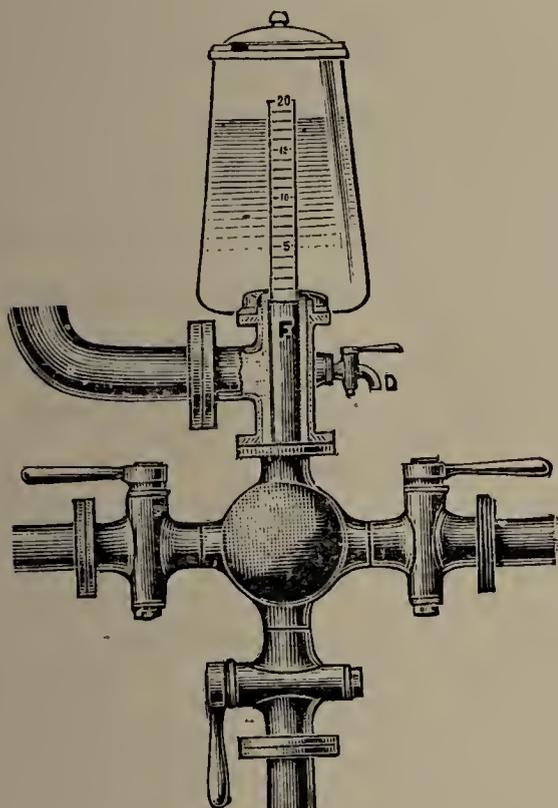


FIG. 47.—Savalle's Test Apparatus.

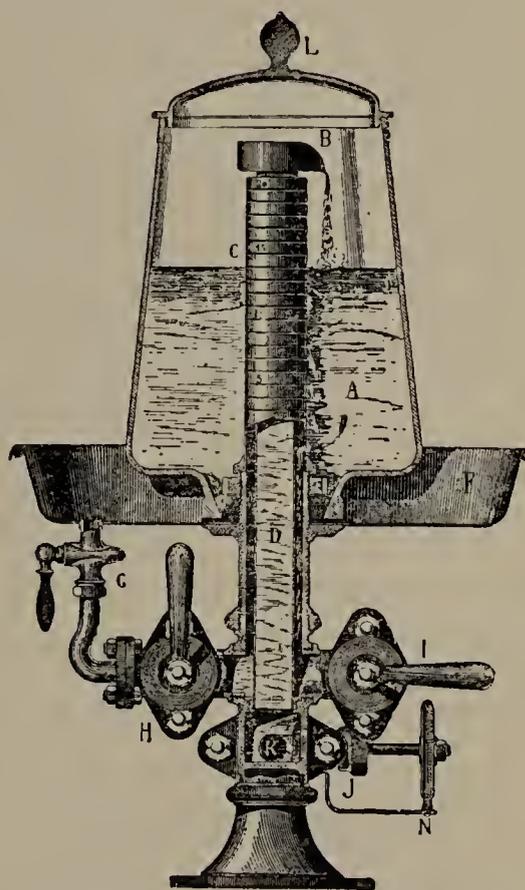


FIG. 48.—Test-glass for measuring density and rate of flow of alcohol from condenser (E. BARBET).

section of 28 sq. mm. It is therefore easy to determine the right section. A series of graduated alcoholometers with a range of 30 degrees of a length of 14 cm. are used, so that their movement may not be impeded by the dimensions of the test-glass. When this instrument is applied to the rectifier, the alcohol flows from the graduated tube into the spherical reservoir. The various taps below are used for the outflow of "moyen gouts," bons gouts, etc.

*Barbet's test-glass tap* (large pattern) (Fig. 48), used for the discharge exit for the pure or pasteurised alcohol, is an improved

form of the classic test gauge glass. The alcohol, the flow of which is regulated by a tap, ascends the central tube CD, then runs away by the spout B of the upper funnel. When the exit tap is closed, the level of the alcohol gradually rises in the globe A. Now this globe is graduated, from the lower to the upper mark being 5 litres. The time is noted when the level is at the lower mark, and again when the volume is exactly 5 litres. The difference gives the time required for 5 litres to accumulate, from which the hourly flow is deduced; that done, the exit tap is in turn regulated so as to keep the level constant at the upper mark. So that if by any chance the alcohol passing through the entrance tap varied, warning is at once given by the change in the level of the alcohol in the globe. Finally, the arrangement of the globe and the manner in which it is mounted on the test-glass are such that the whole of the globe may be emptied and rinsed out. Barbet's small model, intended for the extractions of first and last runnings "mauvais gouts," is much smaller, the principle, however, being identical.

In old models the alcoholic liquid arrives by the annular part and issues by the orifice at the base of the central tube. The top of the test-glass, in default of renewing, heats and yields erroneous indications by alcoholometer and hydrometer. In Barbet's model two concentric tubes are used, the central tube being capped by a small movable discharge lug, by which the flow of alcohol can be at any moment controlled. Delivered from the top the alcohol issues from the bottom of the test-glass by a hole pierced in the bottom of the exterior tube, which bears equidistant marks. The level assumed by the alcohol in the globe gives the measure of the hourly flow. The outside tube is divided so as to allow the test-glass to be emptied when the liquid is not sufficiently fit, and the shape admits of this being done completely, whilst in the usual models the projection made by the tightening screw hinders the expulsion of sand or mastic, which frequently contaminate the test-glass. In countries where the Excise require rigorous sealing-up of the test-glass, the cap of the cover is made of bronze, and it can be joined to a circle of bronze fitted to the upper ring of the globe.

For extracting, the fusel oil taps are also fixed to the test-glasses, whether the products be soluble in water  $40^{\circ}$ - $50^{\circ}$ , or insoluble therein  $75^{\circ}$ - $80^{\circ}$ . The working pressure being constant in the apparatus at all stages, it follows that the outflow of oils is also constant, like that of the pasteurised and non-pasteurised alcohol. Finally, the exit of the residual water at the bottom, or the exhausted wash when wash is being directly rectified, is entirely automatic, requiring no supervision except when the apparatus is to be stopped, or when it is feared the exhaustion is insufficient. In many factories they do not hesitate to push the extractions of non-pasteurised as far as 15-20 per cent., so as to make a cheaper alcohol than the pasteurised, which constitutes the superior brand. But when this secondary quality is not required, this test-glass is eliminated, and the non-pasteurised enters directly into the middle of the concentration trunks purifier. This arrangement is shown in Barbet's 1900 models, sometimes exclusively and sometimes co-existent with the non-pasteurised test-glass. In the first instance, a slight practical difficulty had to be overcome. In fact, a large proportion of non-pasteurised cannot be conveniently returned to the purifier; this alcohol has been driven off from the high strength alcohol in the rectifier, and the pasteurised should only constitute the complement of the non-pasteurised, so that the total corresponds with the hourly output of the apparatus. Thus, take a rectifier the capacity of the plates and condenser of which correspond to a normal flow of 400 litres per hour. This flow includes pasteurised and non-pasteurised. Therefore, if 100 litres be withdrawn per hour from the non-pasteurised, the pasteurised must be reduced to 300 litres. So long as the non-pasteurised is collected as a second-class marketable product, the output of the apparatus is a purified phlegm which contains, associated with pure alcohol, only water and the less volatile products of the last runnings. This is a great step in advance, because nothing further has to be done than to separate the pure alcohol, on the one hand, from the remainder, all that is less volatile than itself, viz., water and oils. The last problem is still analogous to the previous one. It is a little more delicate,

but it is quite legitimate to predict the solution of advance by means of an appropriate continuous apparatus.

Adopting the principle of the division of labour, the fractional separation of the two great classes of impurities may be effected by two consecutive but conjoint operations, both acting in concert and continuously. There are three principal classes of substances in phlegm : (1) The aggregate of all those substances which are more volatile than alcohol, or the first runnings. (2) Pure ethylic alcohol. (3) The aggregate of all those substances which are less volatile than alcohol, or the last runnings. Therefore the whole science and practical skill of the distiller should be concentrated and brought to bear upon the subject so as to make only these three kinds. All other kinds are bastard lots. He ought, moreover, so to arrange matters that the first and last runnings are reduced to a minimum volume. Just as we possess distilling columns that yield highly concentrated alcohol at the outset, in a similar manner the preliminary purification of the phlegms should be so conducted that it yields as far as practicable only first runnings of a maximum degree of concentration. Owing to the great mutual affinity which subsists between the first runnings and the alcohol, the task is not so simple as the separation of alcohol from the wash. It may even be affirmed that there is a technical difficulty to overcome. More or less promising solutions of the problem may be arrived at, but the actual result must be attained. The second operation, rectification properly so called, must be conducted on exactly similar lines. The condenser is not an analyser.

*Fire-heated distilling and rectifying column.*—For small distilleries or farms which do not possess a steam boiler, the purifying column may be constructed as shown in Fig. 49. By aid of the system of invariable flow (presently to be described), the slight variations due to fire heat are remedied, and the sharpness of the separations leaves nothing to be desired. Economy in heating is assured by the use of a heat recuperator R, and the pasteurised alcohol may at will be blended with the œnanthic vapours in the vessel P, as in the case of steam-heated wash. Alcohol of any desired strength may be obtained. In starting, no extraction is

pasteurised until the first runnings worm safe tap marks the desired strength. In a very short time the apparatus will have reached the point, because this requires only a very feeble stock

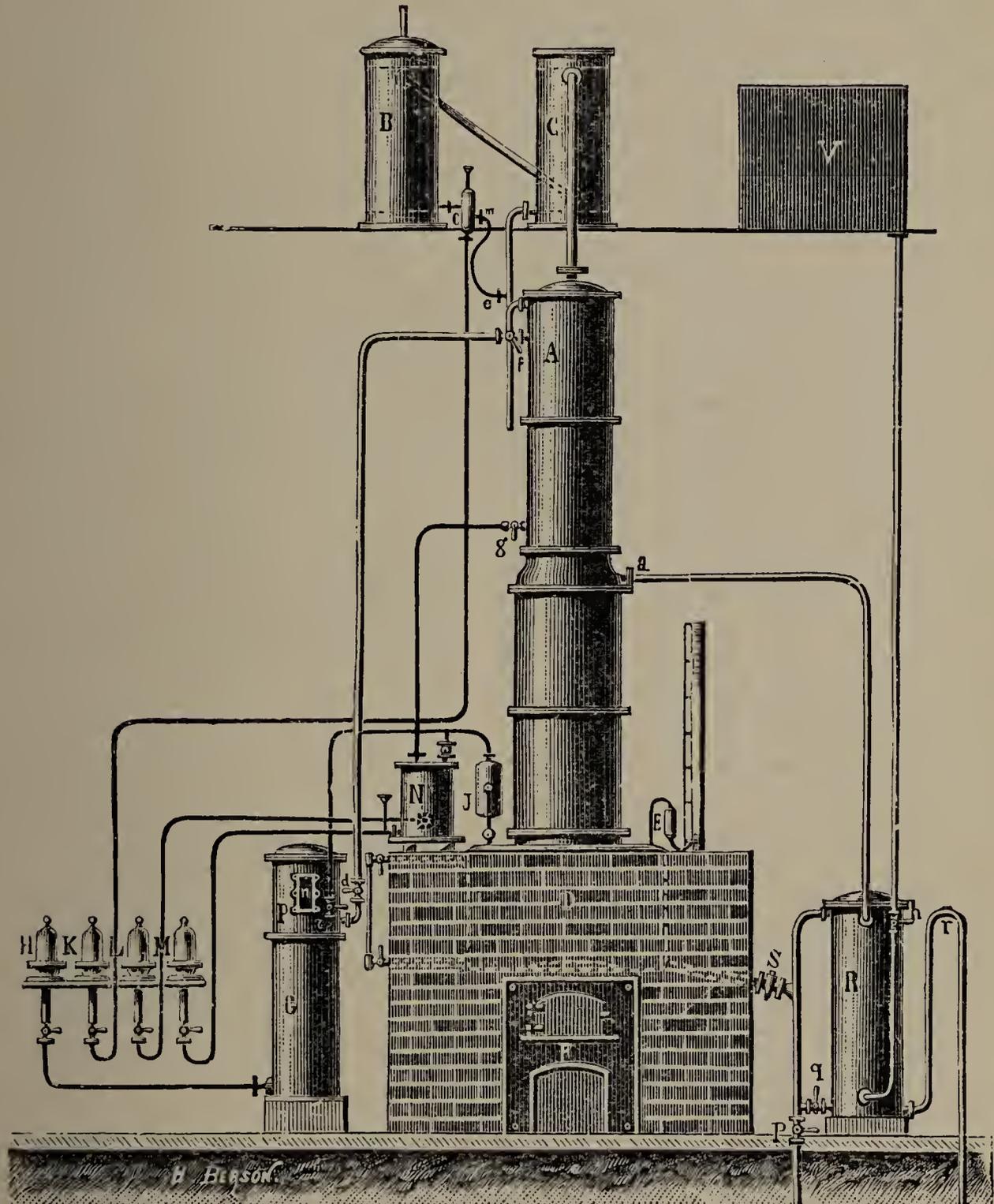


FIG. 49.—Fire-heated distilling and rectifying column. A, rectifier; B, condenser; C, refrigerator; H, pasteurised test safe; K, first runnings test safe; L, last runnings test safe; M, exhaust test safe; R, forewarmer; S, vinasse exit; V, wash tank; a, wash entrance (E. BARBET).

of alcohol on the plates. If it be desired to reach  $94^{\circ}$  or  $95^{\circ}$ , it will take rather a long time to get ready, possibly more than an hour, during which time the alcohol entering as wash accumulates

and grades itself on the upper plates owing to the total retro-gradation. The interior working of this still is explained below.

*Invariable regulation of flow.*—Pasteurisation will be better understood by a diagrammatic illustration introducing invariable regulation of flow to reduce extraction of ethers as far as necessary

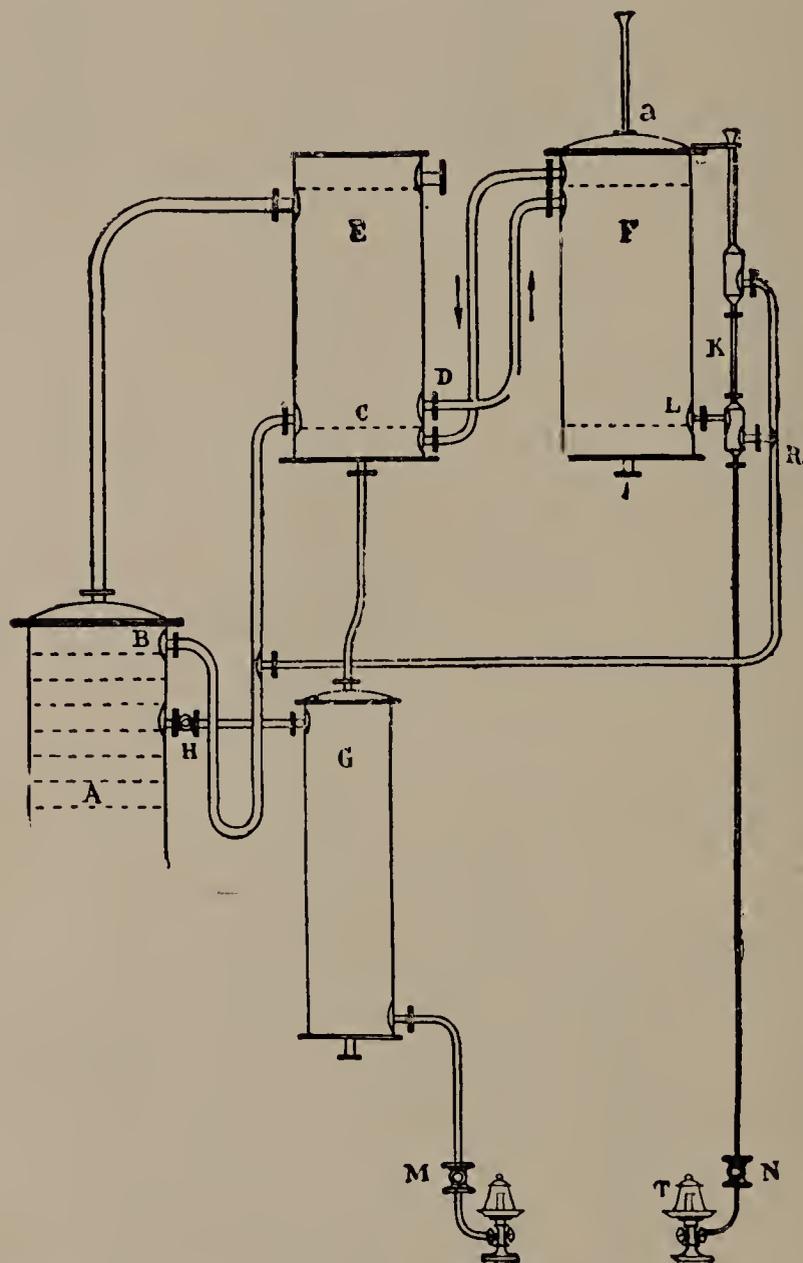


FIG. 50.—Invariable regulation of flow (E. BARBET) A, Rectification plates; E, condenser; F, refrigerator; G, refrigerator pasteurised; H, tap for extraction of pasteurised alcohol; K, air escape; L, exit of cold alcohol; M, tap regulating flow of pasteurised; N, tap regulating invariable flow; P, test-glass; R, return to retrogradation or excess of first runnings; T, first runnings test-glass

at T. The refrigerator F is on the same floor as the condenser E. The cooled first runnings issue by L, with its test-glass T and regulating tap N interposed at the entrance to the latter, just as there is a regulating tap M to the test-glass P of the pasteurised alcohol, on the alcohol descending pipe connected with the

retrogradation from the condenser E. If the apparatus is at work, the tap N being quite shut, the ethers ascend the pipe NK, through the tube R, to mix with the retrogradation re-entering the rectifier A at B. With rather more water than formerly, rectification still proceeds as before, even though there be no alcohol outflow. Open N to get 5 per cent. initial flow. Open fully pasteurisation tap H and regulate flow from P by tap M to 95 per cent. of front flow from T. The apparatus is now regulated, or one-third may be drawn from T and two-thirds from P, or equal parts or any other proportions, without appreciable variation in the amount of water used, or of pressure on regulator. In spite of small variations of level in the water-tank, working is very regular. If the water feed diminishes slightly, condenser E gives less retrogradation; but as cooler F is too strong (it is so in all plants), cooling of the alcohol is still assured. As all the alcoholic vapour is condensed, and as flow from T cannot vary owing to tap N, the excess of alcohol may re-enter the apparatus, thus completing almost mathematically the deficiency in retrogradation from E. The analysis of crude aqueous alcoholic vapours in the condenser is thus insignificant, and the test-glass alcohol in no way differs in composition from that of retrogradation from E. This plan, with the aid of the variable pressure regulator, enables the flow and play of the column to be modified during rectification either to alter the quality of the rectified spirit or when the phlegms are changed. Pasteurisation is thus a new method of expelling ethers, a new process of continuous fractionation applicable to other products, *e.g.*, petroleum. In a continuous column fed regularly with retrogradation, the flow being constant, each plate is charged with liquid of constant composition corresponding to the boiling-point of the liquid on that plate. As many extractions can thus be made as there are different liquids to be isolated, or of special mixtures on given plates. The table on next page shows the purifying capacity of pasteurisation.

From very impure products an alcohol is thus produced containing only one-sixtieth of the impurity of that flowing simultaneously from the first runnings tap. By adopting

TABLE XXV.—SHOWING RESULTS BY ANALYSIS BY PERMANGANATE AT DIFFERENT PHASES OF RECTIFICATION OF BAD QUALITY PHLEGMS (CRUDE SPIRIT) IN A CONTINUOUS RECTIFIER.

Duration of Decolorisation.		Duration of Decolorisation.	
Pasteurised Alcohol.	First Runnings.	Pasteurised Alcohol.	First Runnings.
1 min. 30 secs.	2 secs.	8 min. 45 secs.	10 secs.
3 „ 0 „	3 „	13 „ 30 „	13 „
4 „ 45 „	5 „	15 „ 0 „	19 „
6 „ 0 „	6 „	17 „ 0 „	22 „
7 „ 0 „	8 „	—	—

pasteurisation the ethers may be extracted without a purifier in the form of vapour, which is driven upwards from plate to plate until it enters another column or passes to a condenser, where we shall leave it for the time being and pass to the steam regulator.

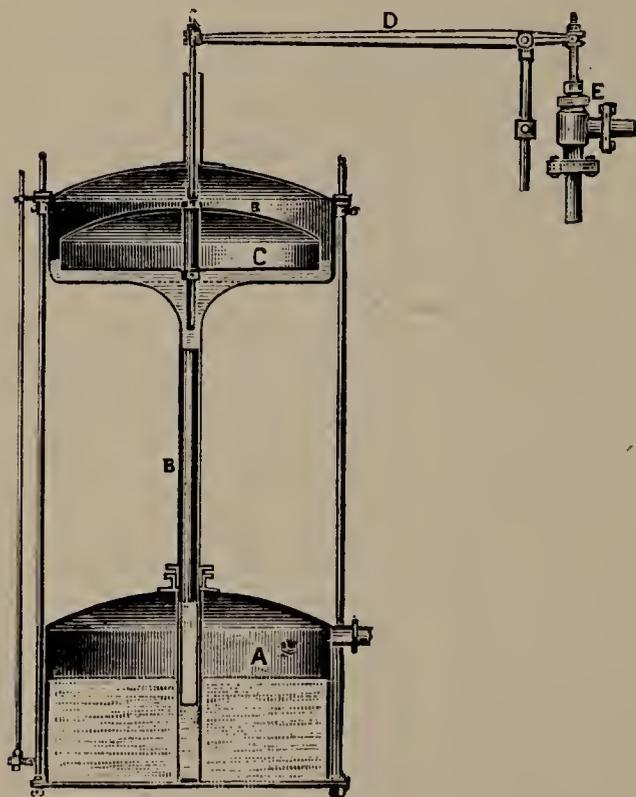


FIG. 51.—Steam regulator (SAVALLE).

*Savalle's steam regulator.*—The essential part of this apparatus is a copper float C, capable of moving in the chamber B, and controlling by the differential lever D the balanced steam valve E. This float exercises in this way on the rod of the valve a pressure equal to 400 kilogrammes, capable of overcoming all accidental resistances. The principle of

this regulator is quite easily understood. Cold water is run into the tank A, forming the foundation up to the level of the pipe F, which acts as an overflow and communication pipe between the tank and the steam reservoir, the pressure of which is to be regulated. The upper part of this tank forms an air-cushion between the steam under pressure and the layer of water; under

the influence of the pressure the water rises in the lower tank, raises the float C, and causes the lever which controls the steam valve to act. The pressure can be regulated in this way to a centimetre of water, *i.e.*, about  $\frac{1}{1000}$  of an atmosphere.

*Barbet's steam regulators.*—Barbet was the first to draw attention to the importance, even with intermittent rectifiers, of being able to vary the working pressure without disturbing the distillation in any way. In intermittent rectification, towards the end, when there is hardly any alcohol left in the still, it is desirable to increase the supply of steam, without which final exhaustion and expulsion of oils are retarded. Now Savalle's regulator is essentially fixed, and every change in the working pressure effects quite a transformation, which entails, in the first place, the stoppage of the plant. The benefit of being able to vary the pressure during the course of the rectification has been so much recognised that Savalle, Egrot, Crepelle, Fontane, and Guillaume have invented steam regulators intended to meet this requirement. None of these, however, solve the problem in such a practical and simple manner as that of Barbet's, which was also first in the field. His method is an improvement on the Savalle rectifier. On the lower receiver he placed two small taps, connected with the pipe which communicates the pressure. Hence the expense is insignificant, and the device can be adapted to all existing regulators. The working is very simple; if more pressure be desired the first tap *a* is opened; more pressure still, the second is opened, and *vice versa* to return to lower pressures. The explanation is equally simple to understand when the two taps *a* are closed, the working pressure is H. By opening the first tap the level in the lower vessel descends to the extent of *h*, but the upper level does not move, therefore the working pressure becomes  $H + h$ . By opening the second, the pressure becomes  $H + h + h$ . The heat being more and more energetic, the plant can do more work. Thus there are only three working pressures at command. His competitors have tried to improve on this by varying the pressure from centimetre to centimetre, which is not good practice, because it is impossible to perceive any change in the working by a variation of 3 or 4 centimetres in the pressure.

It requires at least 8-10 centimetres to produce a tangible effect. Thus variation from centimetre to centimetre had led to unpleasant if not dangerous mechanical complications. All that is

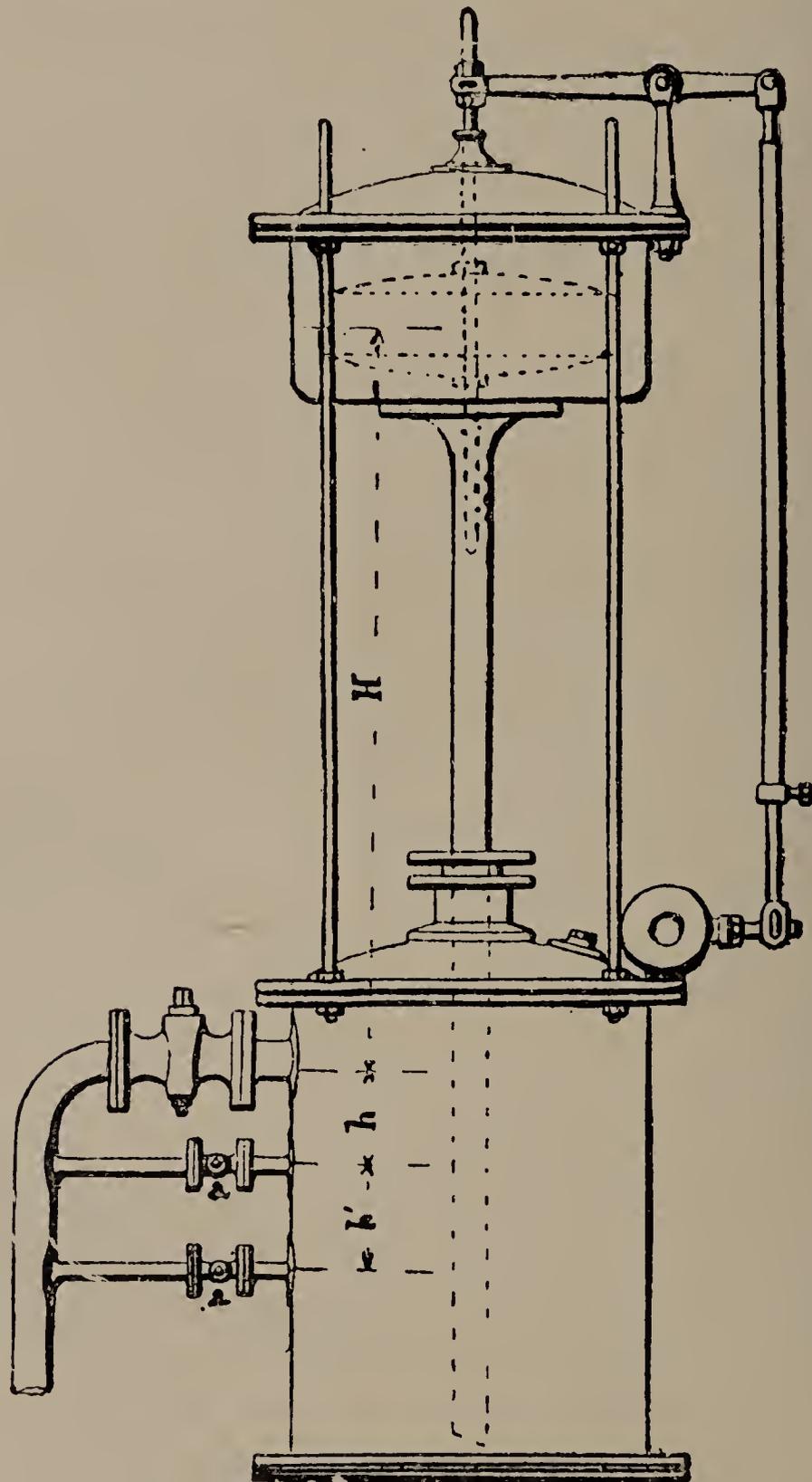


FIG. 52.—Steam regulator (E. BARBET).

required to regulate the rectification speed to correspond to the progress of brewing, is to be able to tell the distiller to go at a slow, a fair, or at a quick speed. By regulating the speed of distillation to a greater or less extent the speed of manufacture

is kept to its proper limit with the greatest of ease. Barbet has also improved the regulator itself. First of all, replacing the balanced valve of Savalle by a flap valve, the latter requiring less force to work it, so that the regulator need not be so wide. The wear and tear of the flap valve is very slow, whereas in the balanced valve the rush of the steam hollowed out the grooves, which soon wore out the valve. Besides, it is easier to regulate the flap valve than the balanced valve, the upper reservoir of the regulator rising and falling more regularly, the rod which works the flap valve being sheathed. A pressure screw adjusts the entrance of the rod into the sheath to the desired extent. Finally, Barbet's model is intended to prevent the expulsion of liquid, which sometimes occurs and is a source of great trouble. The upper float is fitted with two small lateral studs fixed in the arm of a lever. For the free disengagement of air, a pipe is fitted to the upper part, but this pipe may be 50 cm. long, so that it would require quite an extraordinary pressure to cause the liquid to be expelled through the orifice of this pipe. This new type of regulator is very trustworthy; it is less liable to get jammed than the old type, the long screwed rod of which was not always perfectly centred. In continuous rectifiers, where strong pressure is often applied,

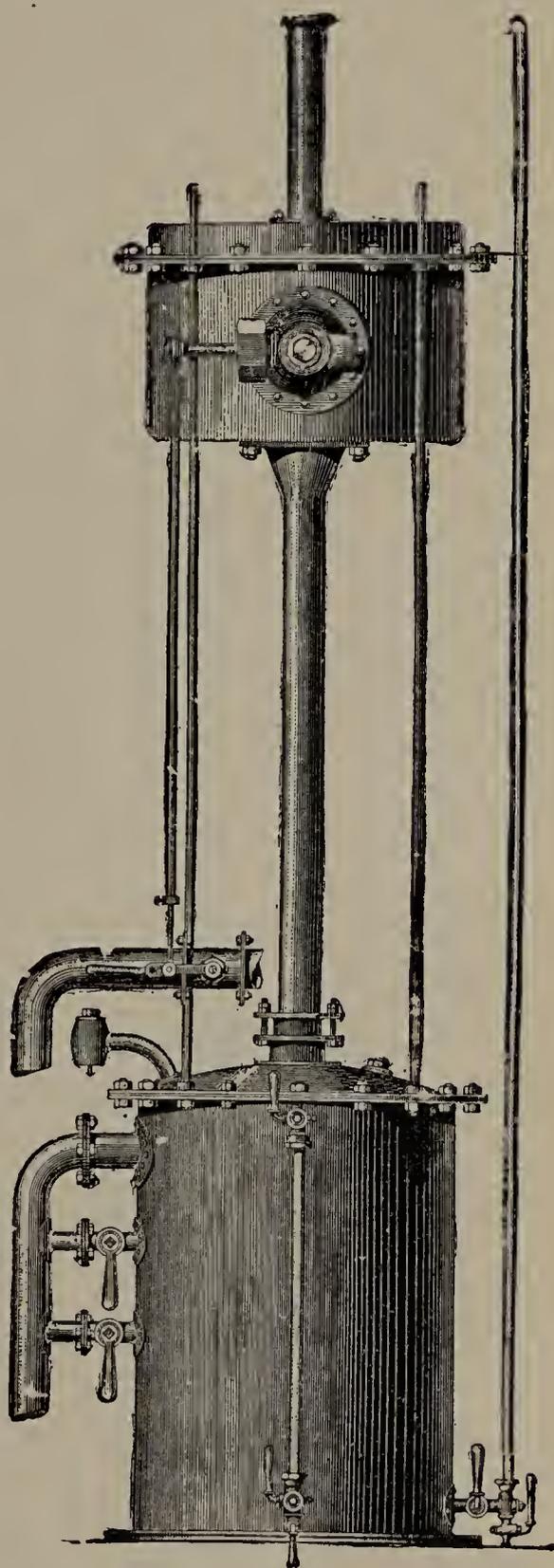


FIG. 53.—Steam regulator  
(E. BARBET).

it is important to be protected from priming not only on account of the loss of alcohol, but because of the risk of fire. But this is not to be feared with the new model. As to the functions of the plates, condensers, as will be seen in the sequel, have no appreciable effect on the elimination of impurities, *i.e.*, on the rectification of alcoholic vapours. They are not analysers, as the Germans term them. The whole work of fractionation and of assortment is accomplished on the plates by the aid of the retrogradation or the condensed liquid which falls back on to the plates from the condenser, this acting as a refining washing liquor. The plates are the sole seat of rectification, and it matters little whether the washing liquor comes from tubular, serpentine, horizontal, or vertical homothermes, or counter-current condensers. The only important thing is the manner in which the plates utilise this purifying agent. The best form of plate is that which, with the minimum of retrograded alcohol, yields the most clearly defined fractionation; because the more one is obliged to condense the alcohol at the condenser to get the right strength and purity, the more heat and water is consumed. The more perfect the plate and the fewer the number required, the greater is the economy in the cost of the plant, and the less the height of the building. The question of the capacity of the plates to produce 96.5 per cent. alcohol as a minimum is an important one, for they cannot be multiplied indefinitely, especially in an agricultural distillery. The owners will demur at the height of the buildings; besides, the cost would be excessive. The number of plates in distilling plant is usually in great excess. Theoretically, four plates should be sufficient to exhaust the wash without increasing the steam. The main obstacle to the use of a minimum number of plates arises from the habitual imperfection of the bubbling of the vapour through the liquid. With long round caps, with smooth rims, boiling is quite tumultuous, the vapours being evolved as enormous bubbles. These only come into contact with the liquid at their periphery. All the vapour in the centre of the bubble bursts on the surface without being utilised. So far as analytical capacity is concerned, nothing appeared up to 1896 to be better than Savalle's perforated plates, which atomise

the vapour in the alcoholic liquid, and thus impart the maximum facility of exchange between a weak and a strong vapour. Unfortunately, these plates run the risk of being discharged at any moment by the least variation in pressure, and such a change in pressure is made each time the feed is modified. Finally, by continued use, especially with acid wines or washes, the holes become enlarged and the apparatus no longer works normally.

Barbet's plates consist of a large number of quite small equidistant caps of hammered copper, around which the wash is divided and circulates easily. The developed length of the line

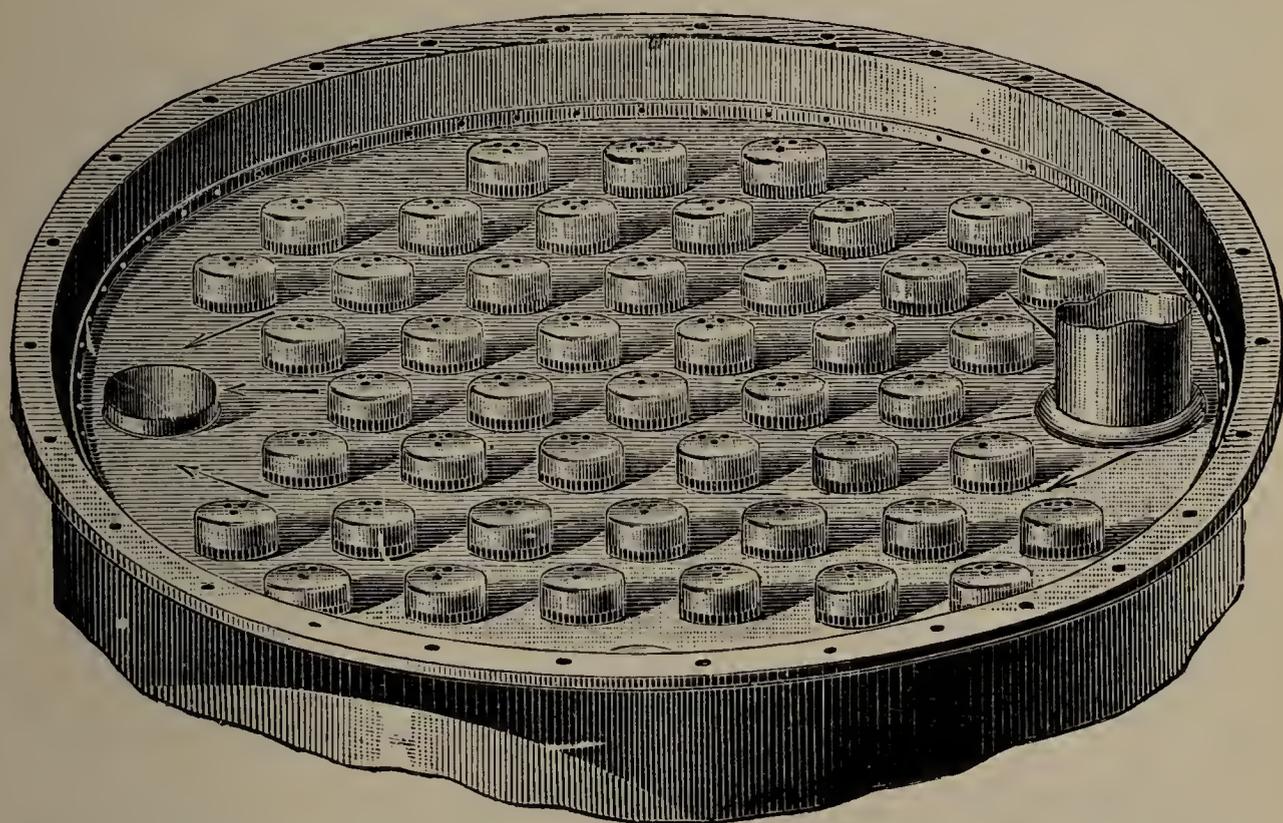


FIG. 54.—Distilling column cross section, showing circulation of wash on plate, and bubbling of alcoholic vapours through comb-slit caps (E. BARBET).

of ebullition or bubbling, compared with that of the older patterns, is increased considerably. But what differentiates this system from others is the fact that the circumference of the caps is divided by a large number of *saw cuts*, so that they resemble long-toothed combs. The vapour, imprisoned under each cap, streams through these combs, thus securing perfect atomisation and a maximum utilisation. With the old perforated plates, the drops of liquid were projected, vertically, against the upper plate. Here the jets of vapour are horizontal, and collide against each other, to form a much more regular and tranquil emulsion, and

without entrainment of the liquid, in the path of the vapour. In fact, these caps cannot be constructed except by machine. There is, therefore, perfect regularity and absolute uniformity in the sections for the passage of the vapour, and in the dips, and so on. To control the efficacy of his plates, Barbet made several precise tests at the base of the trunks reserved for rectification. He fixed a tap at the level of a certain number of plates (Nos. 2, 3, 4, 5, 7, 11, and 17), so as to draw off a sample of the liquid from the upper overflow. Then, the apparatus having been perfectly regulated, during several hours previously, as regards feed, indicated by the thermometer controlling the working, samples of sufficient size to be tested by the control alcoholometer were drawn off as rapidly as possible.

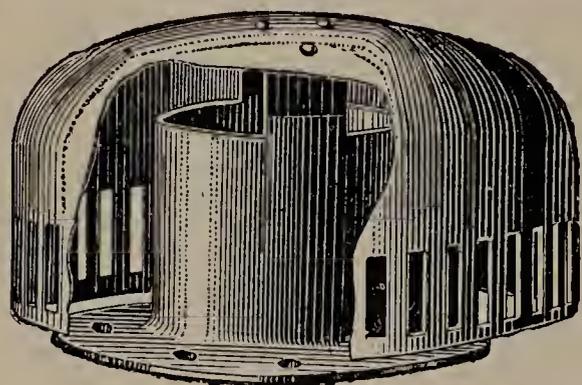


FIG. 55.—Comb-slit cap dipping into liquid alcohol through which vapour of alcohol bubbles, on plate of distilling column (E. BARBET).

The first two plates are affected by the proximity of the feed, therefore taking No. 3 as a starting-point, it was  $19.1^\circ$  deficient in actual working capacity. According to Sorel's tables, the vapours disengaged from a liquid of  $19.1^\circ$  ought to have a strength of  $65.6^\circ$ , therefore, if the plates are working perfect and analysis is effected according to theory, the plate

immediately above should have a strength of  $65.6^\circ$ . In its turn, the liquid on plate 4, having a density of  $65.6^\circ$ , should yield a vapour of  $80.1^\circ$ , and so on. The following figures were obtained in the above test :—

No. of Plate.	Degree G. L.	Tables G. L.	No. of Plate.	Degree G. L.	Tables G. L.
2	12.4	—	7	87.9	89.1
3	19.1	—	11	93.3	93.9
4	60.2	—	17	95.5	—
5	77.4	78.24	—	—	—

If No. 4 had been taken as the point of departure instead of No. 3, the tables would have given  $78.24^\circ$ , whereas  $77.4^\circ$ , or one

degree less, was obtained. Taking 5 as the point of departure, the tables yield theoretically  $85.2^\circ$  for No. 6, and  $89.1^\circ$  for No. 7, whilst the test yielded  $87.9^\circ$ , average loss of  $0.6^\circ$  per plate. Finally, starting from No. 7 with  $87.9^\circ$ , consecutive calculation shows that No. 8 should mark  $90.6^\circ$ ; No. 9,  $92.2^\circ$ ; No. 10,  $93.3^\circ$ ; No. 11,  $93.9^\circ$ . The four plates have had exactly the same effect as three theoretical plates, since No. 11, with  $93.3^\circ$ , corresponds exactly with what No. 10 should give. The coefficient of capacity has therefore attained 75 per cent. From No. 5 to No. 7 it may be said that only  $10.5^\circ$  have been gained instead of  $11.7^\circ$ .  $\frac{10.5}{11.7} = 90$  per cent. of the theoretical capacity. The

same calculation would give 88.4 per cent. from plate 3 to plate 4, and 95.3 from plate 4 to plate 5. These are very satisfactory yields. As comparative experiments, those of Agde, in 1889, may be quoted: No. 3,  $81.3^\circ$ ; No. 8,  $90^\circ$ ; No. 15,  $95^\circ$ ; No. 21,  $95.5^\circ$ . It thus took eighteen plates to raise the strength from  $81.3^\circ$  to  $95.5^\circ$ , whilst with the new plates only twelve were required. In making calculations like the above by comparison with the theoretical increase in strength, it is found that, starting with No. 3 plate at  $81.3^\circ$ , it is the 10th plate which ought theoretically to show  $95.5^\circ$ ; that is, with seven plates instead of twelve. Capacity, 58.3 per cent. Sorel gives the following: "Ascent of degrees found in the plates of a rectifying apparatus, Savalle system, with rectangular plates." In the middle of the operation, in full normal working, Savalle found the following results:

No. of Plate.	Degree G.L.	No. of Plate.	Degree G.L.
4	87.5	24	94.6
9	92.0	29	95.0
14	93.3	34	95.3
19	94.0	—	—

If these plates had, starting from 4, followed the theoretical rule of increase in strength, there would have been on No. 5,  $90.41^\circ$ ; No. 6,  $92.1^\circ$ ; No. 7,  $93.3^\circ$ ; No. 8,  $93.9^\circ$ ; and No. 9,  $94.5^\circ$ . Instead of that, it is the 24th plate which registers  $94.6^\circ$ , and not

No. 9. Twenty plates have therefore been required instead of five, according to theory, hence the yield is only 25 per cent. Besides, ten plates were required to rise from  $87.5^{\circ}$  to  $93.3^{\circ}$ , whilst only four plates were required at Eprunes to rise from  $87.9^{\circ}$  to  $93.3^{\circ}$ , and twenty plates to rise from  $93.3^{\circ}$  to  $95.3^{\circ}$ , whilst only six were required at Eprunes to rise from  $93.3^{\circ}$  to  $95.5^{\circ}$ . The yield of the new plates is therefore two and a half to three times better, which constitutes a considerable improvement. Again, with washes indicating only  $1.25^{\circ}$ , Barbet found the following alcoholic strengths at the different test taps:—

Pasteurised . . . .	$96.8^{\circ}$ at $15^{\circ}$ C.	Actual capacity, $96.8^{\circ}$
First runnings not pasteurised . . . .	$97.6^{\circ}$ at $18^{\circ}$ C.	„ $97.0^{\circ}$
Last runnings, 5 plate . . . .	$80.0^{\circ}$ at $21^{\circ}$ C.	„ $78.2^{\circ}$
First runnings from purifier . . . .	$96.6^{\circ}$ at $20^{\circ}$ C.	„ $95.6^{\circ}$

The above results indicate very clearly the power of the concentration plates, for there were only ten to secure this result with extremely weak wash. Barbet also tested the expenditure of water and steam by the apparatus. All the water, hot or tepid, was run into a small tank of 250 litres capacity. In seven minutes 195 litres of water ran in at a temperature of  $46^{\circ}$ , equivalent to 1670 litres per hour. The cold water had a temperature of  $7^{\circ}$  C., the water had therefore gained  $39^{\circ}$ . The apparatus put through in the same time 2500 litres of wash at  $1.25^{\circ}$ , and yielded 31 litres of alcohol, calculated to  $100^{\circ}$ . Finally, the spent wash issued from the recuperator at  $83^{\circ}$ . By making calorimetrical calculations, it will be found that the expenditure of steam was 366 kilos, say 14.64 kilogrammes per hectolitre of wash and 67 kilogrammes of water. These two numbers are respectively half that which the German columns expend, according to Maercker; now the latter columns only yield an impure phlegm at  $90^{\circ}$ , whilst that produced by Barbet's apparatus yields an excellent rectified alcohol at 96.5 per cent., sold at a premium for quality. Barbet's cap-comb plates possess the powerful analytical capacity of the old perforated plates, but with the advantage that they are no

longer capricious, and are incapable of emptying themselves on the slightest provocation. Continuous rectification may be stopped for several hours, then restarted without any trouble, because each plate has retained its liquid and is ready instantly to resume its role. Barbet's plates possess other advantages upon which it will be well to insist. When through use the holes of the ordinary perforated plate enlarge, this disturbs the regular working of the apparatus, more pressure being required to maintain the liquid on the plate, hence a quite useless expenditure of steam.

If the pressure be not increased the liquid drains away, and in the end it is necessary to replace all the plates. With Barbet's plates nothing of this nature occurs. Even if the slits become a little wider by use, the subdivision of the vapour will be effective for many years. And if one day it be necessary to remedy matters, all that requires to be done is to remove the caps, and not the plates, nor the central vents of the caps. The expense is therefore very limited. As compared with the old plates with round or long caps, Barbet's plates, in addition to analytical capacity, have other very appreciable practical advantages. Whatever may be the daily production demanded of an apparatus, the same proportion can be maintained between the following different elements.

1. *Developed length of the line of bubbling.* Let  $A$  be the daily production, experience of Barbet's apparatus has demonstrated that each comb-cap corresponds to a daily production equal to  $a$ . Therefore the number  $n$  of comb-caps to use is determined in a precise manner and without error by the equation  $A = na$ . However, the difficulty of spacing large caps round or long, never allows this proportion, the necessity of which is evident.
2. *Area of the plate and stock of liquid which it contains.*—This proportion can always be maintained. If  $n$  small caps are fixed on an area  $S$ , then  $2n$  caps can always be encased in double the area  $2S$ .
3. *Section of passage for the circulation of liquids between the caps from one overflow pipe to the next.* This is a condition which is very rarely fulfilled by the old cap plates. It follows that the thickness of the layer of liquid is never uniform, and that the caps near the overflow do more work than those which are in proximity to the chute of the overflow from

above. Barbet's caps are arranged in such a pentagonal style, that the liquid is forced to spread itself, uniformly throughout the intermediate spaces. 4. *Section of passages and outlets for the vapour, both by small vents and slits.* This depends on the fact that the number of caps is strictly proportional to the capacity demanded. From a constructional point of view, the advantages are more striking still. All the caps are made mechanically, and are thus perforce identical. They are turned in the lathe, in a uniform manner, and the slits are made in a mortice machine. The regularity of construction is absolute; finally, the constructor has the advantage of being able to make beforehand the fittings for these caps, and is thus in a position to construct the apparatus in a minimum of time. The central chimneys, by a special equipment, are also made by mechanical stamping. It follows, therefore, that over the whole of the plate, the dips are absolutely uniform, and that is a condition of good working which had never previously been obtained, certainly not with long caps. Supposing the plates of the apparatus, once mounted in the factory, were not absolutely horizontal—for instance, were 10 millimetres out of level—with the old caps the bubbling would be suppressed, as far as those most deeply immersed were concerned. Only a portion of them would work; whilst with Barbet's long slit there are, in the one case, 35 millimetres of length of slit which will be utilised, and 25 millimetres in the other; but all the caps will work, and the irregularity will be of little importance.

*Working of thick washes.*—For the working of thick washes Barbet makes a comb-slit cap of a special type. The cap is conical in its upper part, so that no dregs can be deposited thereon. Moreover, the cap emerging very little above the liquid, the latter by the tumult of boiling constantly washes the cone and prevents it getting clogged. This cap is prolonged till it comes in contact with the plate, to prevent the pellicles of bran from penetrating under the cap. In the same way, the slits are very fine. During the working of the apparatus there is no danger of penetration, but it is when the column is stopped that it is necessary to protect the interior from obstruction. Moreover, a column, working

thick wash, ought never to be stopped without being fed with water, to displace all the muddy liquids both in the wine-heater and on the plates. Working thus, difficulties are avoided. With thick wash there are in the columns points on the plate which are more subject than others to become obstructed with dregs. This being due almost always to the bad design of the plates, and because of recesses or backwaters, in which there is no bubbling, hence the decantation and thick deposition of solid matter. The great superiority of Barbet's plates lies in the fact that owing to the small diameter of the caps no point in the plate escapes the bubbling. When a pair of observation glass panes are placed above one of the comb-cap plates, the absolute regularity of the bubbling is easily seen without violence or projections on the upper plate. Owing to the great length of

the comb slits, the deep layers of the liquid are affected, thus allowing of a large stock of liquid being left on each plate without inconvenience, imparting a perfect stability to the working of the apparatus in the most simple, natural, and infallible manner. The way in

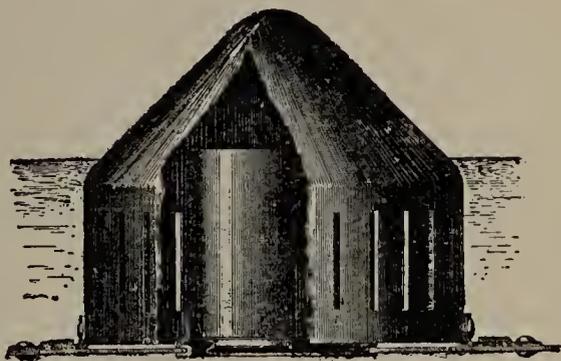


FIG. 56.—Non-obstructable comb-slit cap for thick washes (E. BARBET).

which the truncated comb-caps are fixed to the plate renders them removable at will for cleaning the interior. There are no bolts or rivets to undo. Once cleaned and put in its place the tongue is lowered with a slight tap from a hammer, after which the cap cannot get displaced. It may even be applied to cast-iron columns by a simple letting in of the vent into the body of the plate.

Barbet's tubular condensers are of bronze or copper, without solder. Responsible makers now construct them in no other way. They are furnished with a raised cast-iron pedestal which holds a large stock of liquid; the water or the wash enters in the centre, the entrance pipe is on the side and above the floor, so that the connection joint may be easily made. This joint is too often placed under the floor of the condenser, about 20 feet above

the test-glass floor, *i.e.*, in an inaccessible or dangerous position. The height of the pedestal admits of one or two observation-holes

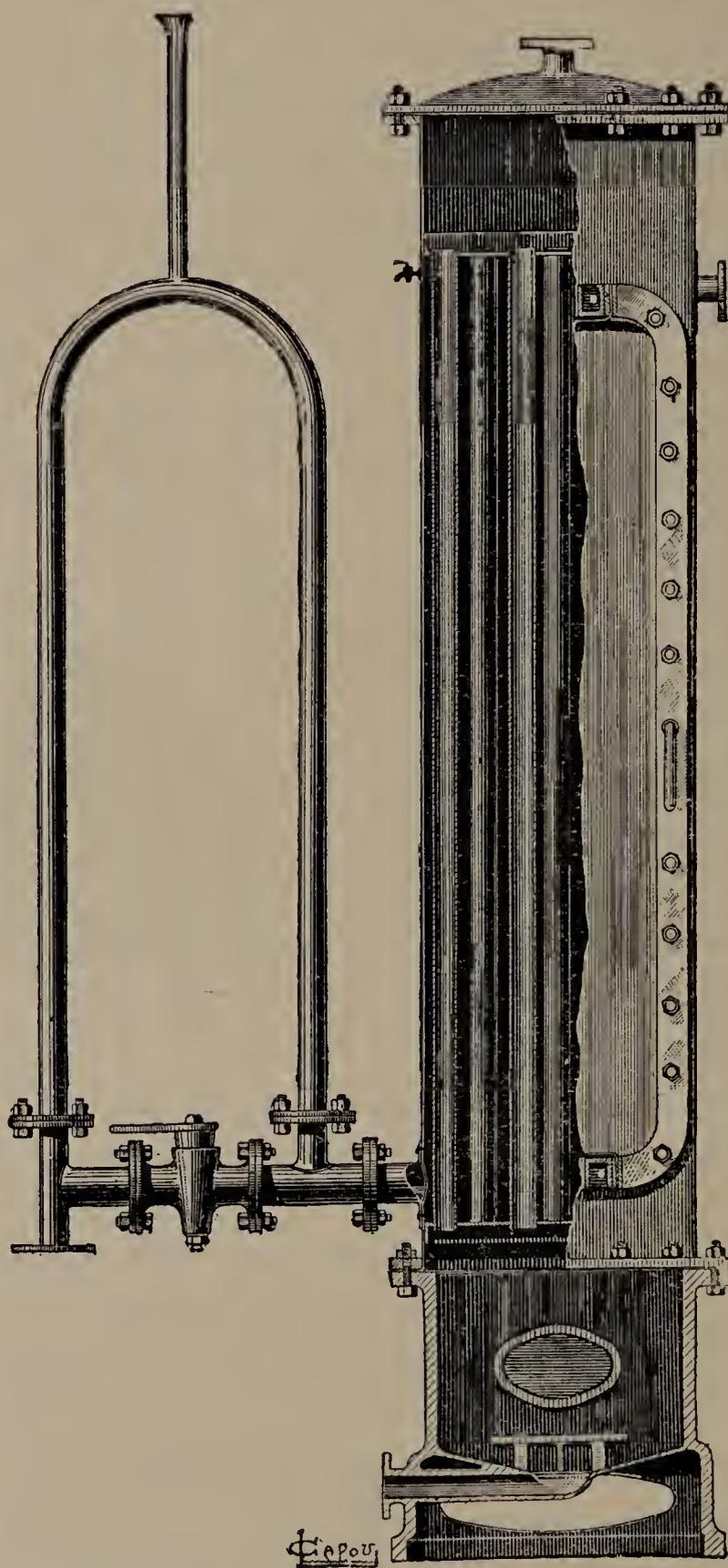


FIG. 57.—Tubular vessels, condensers, recuperators, etc. (E. BARBET).

being inserted, according to the diameter of the tubular vessel, wide enough for any one to enter the dudgeon to fix a tube if need be, or a leaking tube which can be plugged. It is desirable

to provide for the cleaning of the outside of the tubes of the tubular bundle, *e.g.*, in the case of recuperators, or even in the

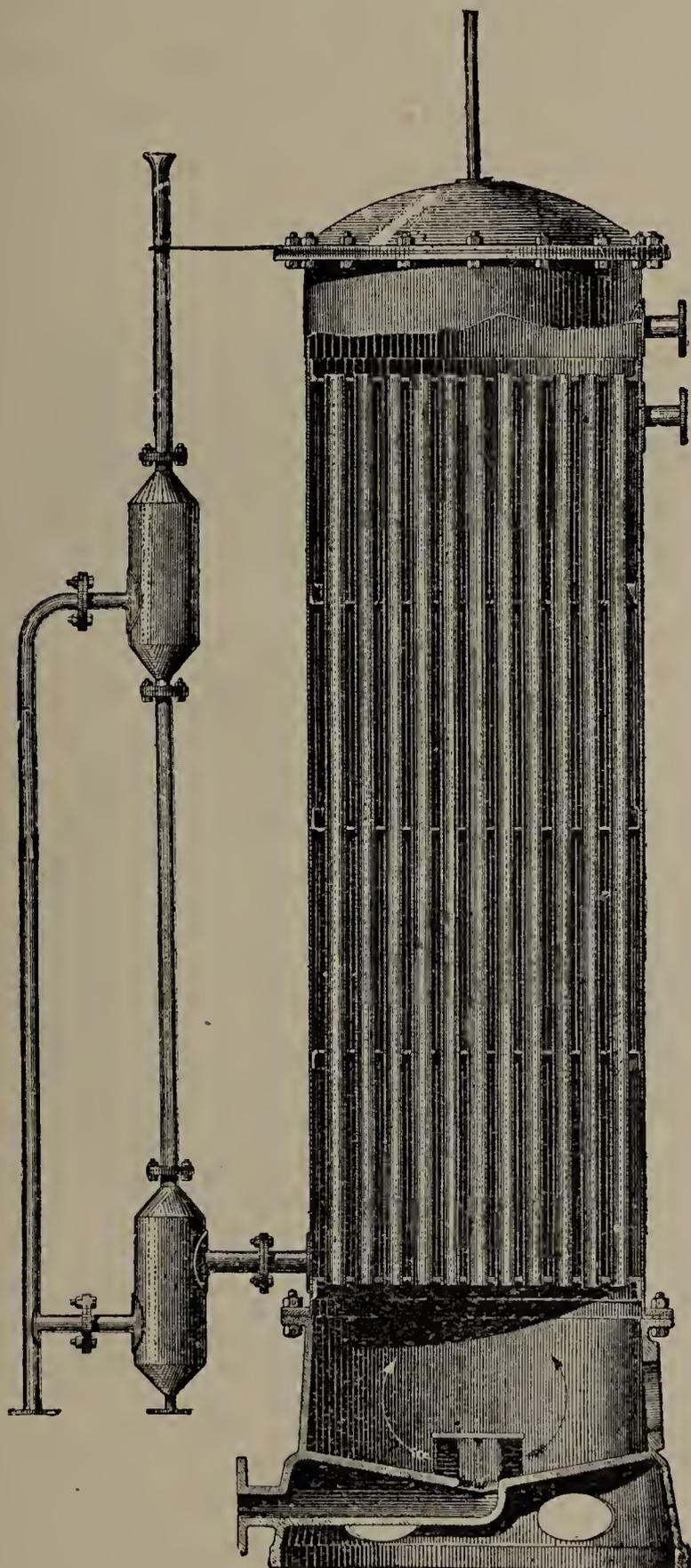


FIG. 58.—Tubular vessels, condensers, recuperators, etc. (E. BARBET).

case of wine-heaters, through which weak alcohol vapours pass. Not only is froth entrained, but also volatile acids and gases, which corrode the copper and cover it with saline encrustations.

In Barbet's tubular vessels the whole bundle is removable, that is, capable of being lifted out of its envelope. The wash enters by the upper pipe, which branches into a central pipe of the tubular plate. At the bottom the second tubular plate is independent of the envelope; it connects with a bomb-shaped bottom furnished with a manhole for cleaning purposes. The wash now changes its direction, spreading into the tubes of the

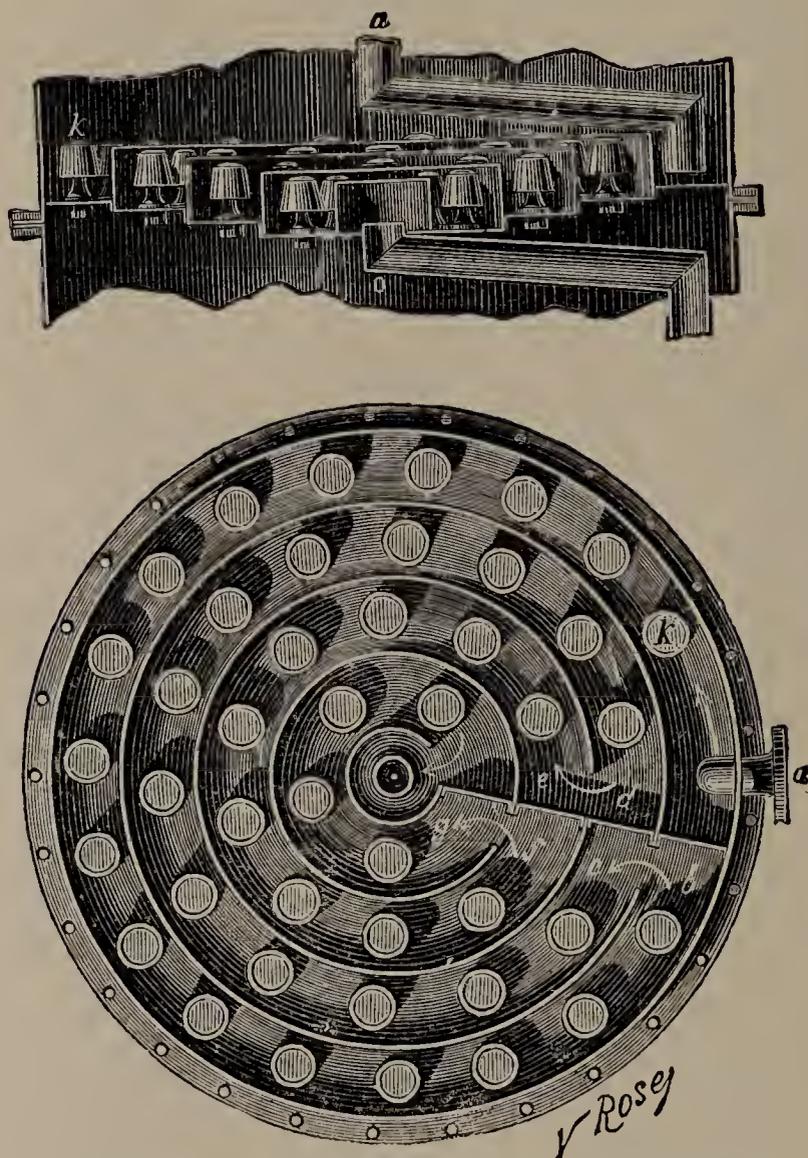


FIG. 59.—Vertical and cross section of distilling column, showing cascade arrangement of plates and caps (EGROT and GRANGÉ).

tubular bundle, and becoming heated as it ascends, issuing from the top of the tube. The methodical circulation of the spent wash is in the opposite direction. This enters by an upper pipe, is cooled as it descends, and issues by a pipe at the bottom. A reascending tube ensures that the tubular vessel remains full. Fig. 59 shows the arrangement of Egrot & Grangé's plates. The liquid descends from the upper plate through the pipe *a*, traverses

in the direction of the arrows the exterior ring *ab*, descends into *c*, and traverses *c d* in an opposite direction. Finally, reaching the centre of the plate *o*, this liquid descends on to the plate below, where it recommences a similar circulation. The surface of the plate is therefore utilised so that the wash descends by a very long circuitous route ; moreover, by the cascade arrangement the

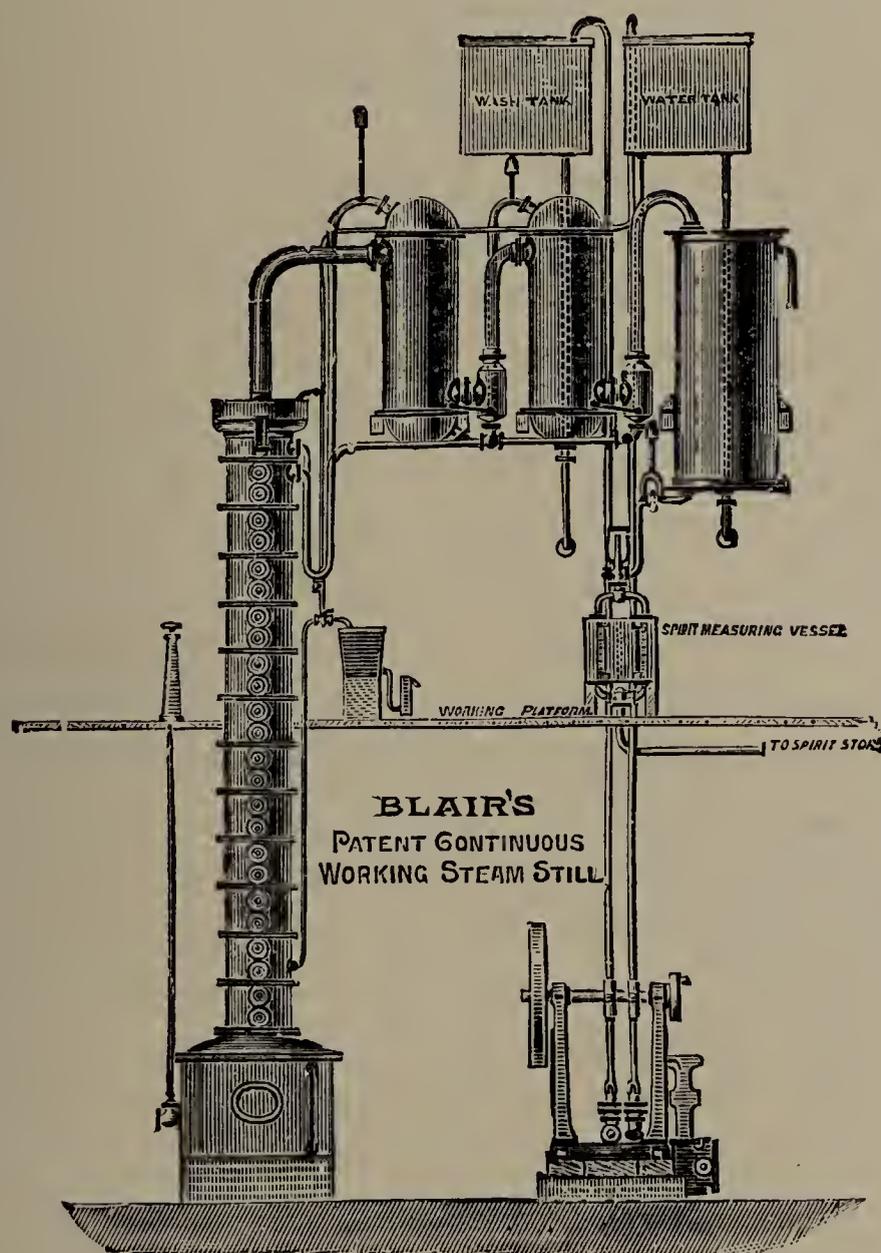


FIG. 60.—Blair's continuous working steam still. A, still kettle, with steam coils B, patent analysing and rectifying column ; C, D, patent rectifying wash-heaters ; E, refrigerator or condenser (BLAIR, CAMPBELL, & MCLEAN, Glasgow).

level of the flow is very regular over the whole of this long circuitous route. Again, the numerous small "boilers" *k* fixed in the path of the liquid split it up and agitate it so that the whole liquid mass is well exposed to the action of the ascending vapours.

"Blair's" continuous working steam stills.—Fig. 60 shows "Blair's" patent continuous working steam still, constructed

specially to produce high-class rum or spirit of best aroma and good quality, continuously, in one operation. By means of this still all the spirit is entirely exhausted from the wash, and a good spirit produced, free from impurities, but retaining its aroma and flavour. The still shown will continuously produce spirit from good wash up to 40° Cartier (95·9° G.L.), or at lower strengths than this as desired. If weaker spirit only—say 25° to 30° Cartier (67·7°-79·1° G.L.)—be required, the vessel D is dispensed with, and the column made somewhat shorter. The still is constructed almost entirely of copper and brass, and consists of a steam chamber surmounted by a distilling and rectifying column, with all internal fittings and mountings, patent rectifying wash-heaters, with spirit separators, spirit refrigerator, spirit test case, sampling apparatus, and all mountings and connections to make the foregoing complete. For small sizes of stills direct fire instead of steam may be applied. An essence box can be adapted to give special flavouring to the spirit if required.

*Inclined distilling column.*—Guillaume's distilling column differs from previous types used hitherto. All distilling columns belong to one or other of two types—(a) plate columns, or (b) full columns (those working full of the wash to be distilled). Each type has its advantages and disadvantages. The plate column distils much more economically, because each plate has its own expansion chamber, in which the alcoholic vapours are freed from any dregs brought in the train of the wash, and because the regular exhaustion of the wash and the enrichment of the vapours is efficiently accomplished therein. But this type, says Guillaume, is defective in distilling thick washes, because obstructions which arrest the circulation of the wash are very frequent. In full columns the wash presses directly from top to bottom on all the liquid mass in circulation, as well as on the discharge of the spent wash, the exit regulator of which ensures the permanent level of the wash in the upper part of the column. It will easily be seen that obstructions may be avoided by this arrangement; but then distillation becomes more costly, because the emulsion produced by the steam in the mass injuriously affects to a considerable extent the regularity of the exhaustion of the wash and

the enrichment of the vapours. The vapours disengaged are therefore poorer in alcohol, while the risk of inefficient exhaustion of the spent wash is greater, both items entailing an abnormal waste of steam. The advantages of the one type offset those of the other. Guillaume, in constructing his inclined column, aimed at combining the merits of both, eliminating, at the same time, their defects. In this apparatus (Fig. 61) the bottom of the

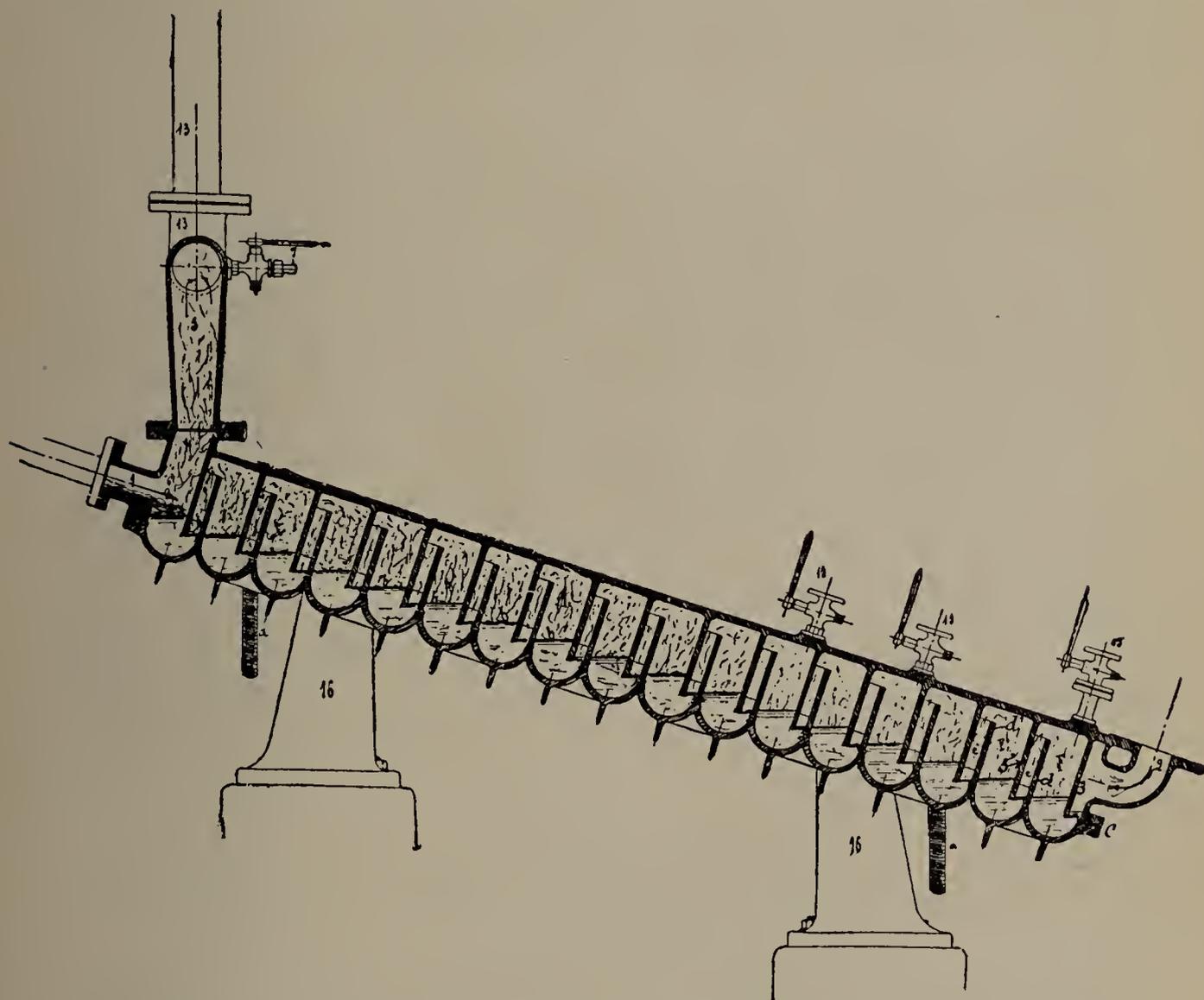


FIG. 61.—Guillaume's "inobstructable" inclined column, longitudinal section (EGROT and GRANGÉ).

column consists of a continuous channel in which the wash circulates freely in one continuous section and slope. The hydrostatic pressure is exerted from top to bottom without loss of pressure or interruption, so as to force this circulation. The wash to be distilled enters at the upper portion of this inclined column, and the spent wash issues from the bottom by means of the extraction regulator B. The steam for heating enters through

2, passing through the partitions 3, 3<sup>1</sup>, from chamber to chamber, bubbling regularly through the wash. After each period of bubbling, it is arrested in a chamber which retains the entrained liquid portions, and it finally reaches the upper chamber 4 and the dome 5. The crude alcohol vapours then pass to the wash-heater (*chauffe vin*) if it be desired to produce weak spirits, or to an appropriate concentration trunk if strong spirits or directly rectified alcohol be desired. This column thus consists of two parts only—(1) the inclined or sloped bottom, used for the regular

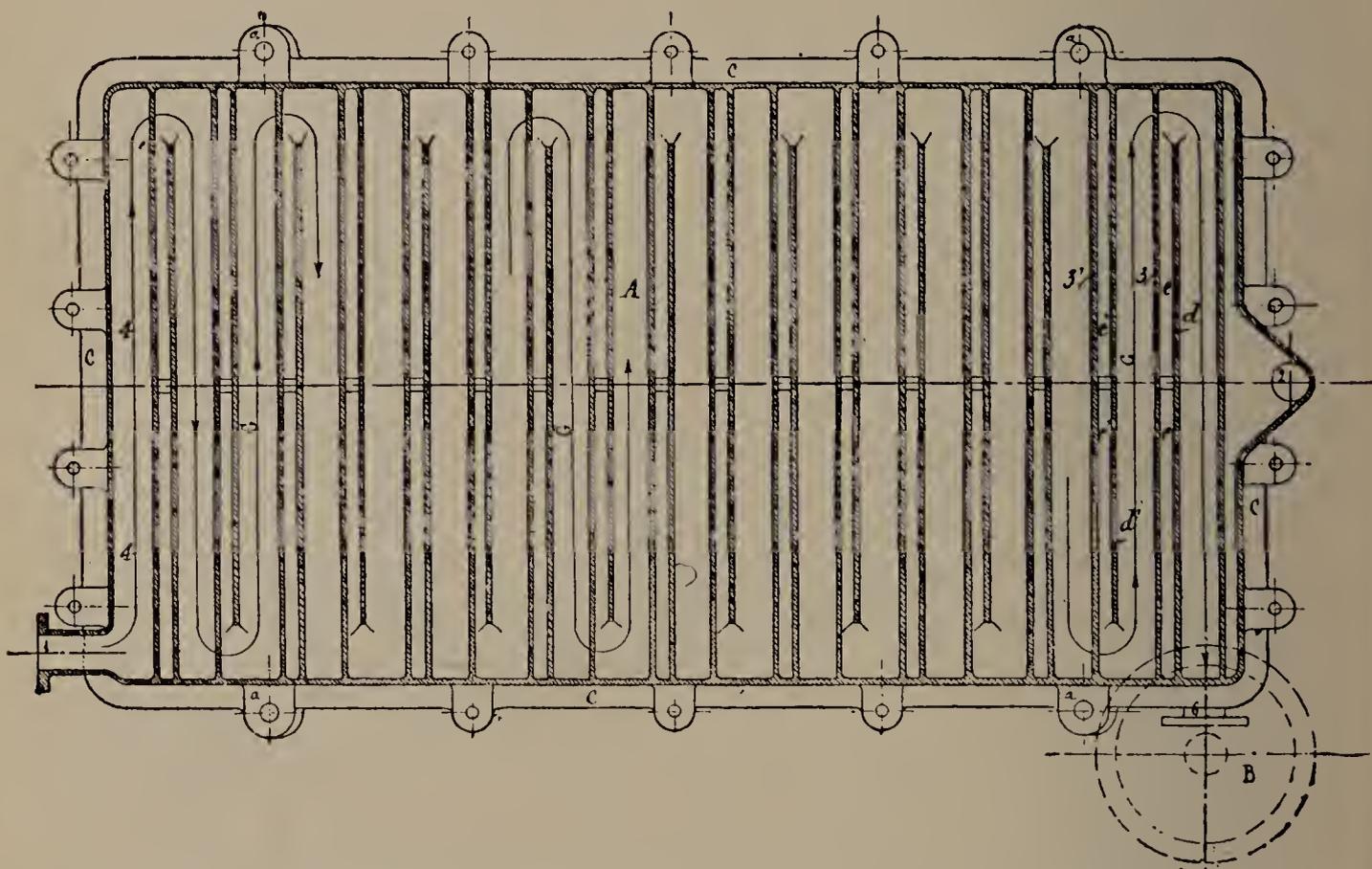


FIG. 62.—Guillaume's "inobstructable" inclined column, ground plan (EGROT and GRANGÉ).

circulation of the wash, consisting of a continuous channel; and (2) the top, used as vapour trap and bubbling-cap chambers. It thus combines the good features of full columns and plate columns. Moreover, in order to inspect the whole of the interior, all that requires to be done is to unscrew the single chief joint C, and to lower, so as to leave the bottom suspended on the vertical rods A. Lowering and raising into position again are thus done very rapidly. But copper columns which are light do not require this arrangement. The use of this "inobstructable" column is said to be particularly adapted for thick potato and grain washes.

Moreover, it also possesses very appreciable advantages in the working of limpid washes on account of its extreme simplicity and the ease with which it can be lowered, its compactness and low height, etc. The apparatus is generally heated by *direct open* steam, except in particular cases where it is not desirable to add the condensed water to the spent wash issuing from the apparatus ; for example, in distilling molasses, which have to be concentrated afterwards to extract the salts.

Escape steam can, however, be utilised. In order to utilise the escape steam, an escape steam "balloon" is installed having a capacity proportionate to the size of the plant. All the steam of the distilling apparatus is taken from this balloon. An admission regulator of direct steam into this latter ensures regular feeding when the escape steam is insufficient, or in case of the stopping of the engine. This balloon is fitted with a loaded safety valve, which allows the escape of steam when the interior pressure exceeds  $\frac{1}{2}$  kilogramme per square centimetre, and the steam entrance regulator is so arranged that the steam cannot enter the balloon until the pressure falls below 450 grammes, and then just in proportion to keep it at that pressure. By this arrangement the distilling column absorbs the escape steam first, and only takes automatically direct steam when the escape steam becomes insufficient.

*Surface heating of the apparatus.*—When the wash must not be diluted, surface heating has to be resorted to, using the arrangement shown in Fig. 63. The spent wash issuing from column A flows into the evaporator J, where it is boiled by a tubular surface condenser, itself heated by steam. The unevaporated *vinasse* issues automatically, regulated as to quantity by the wide open siphon C. The steam is admitted by the tap *d*, and its output is regulated by the valve which follows this tap, and which is acted upon by the regulator O. The regulation of the heating of the plant is done automatically by Guillaume's automatic steam regulator, if the plant be heated by steam ; or if by naked fire, by an automatic regulator acting on the steam entrance. By Guillaume's regulator the pressure at the bottom of the distilling column can be varied by hand and the consumption of

steam regulated according to the work to be performed ; that is to say, only what steam is necessary to exhaust the wash and no more is used.

The discharge of the spent *vinasses* is effected by means of a floating extractor, which is indispensable when working with thick potato and grain, etc., washes, or by an extractor siphon, which is simpler and to be preferred if liquid substances are being

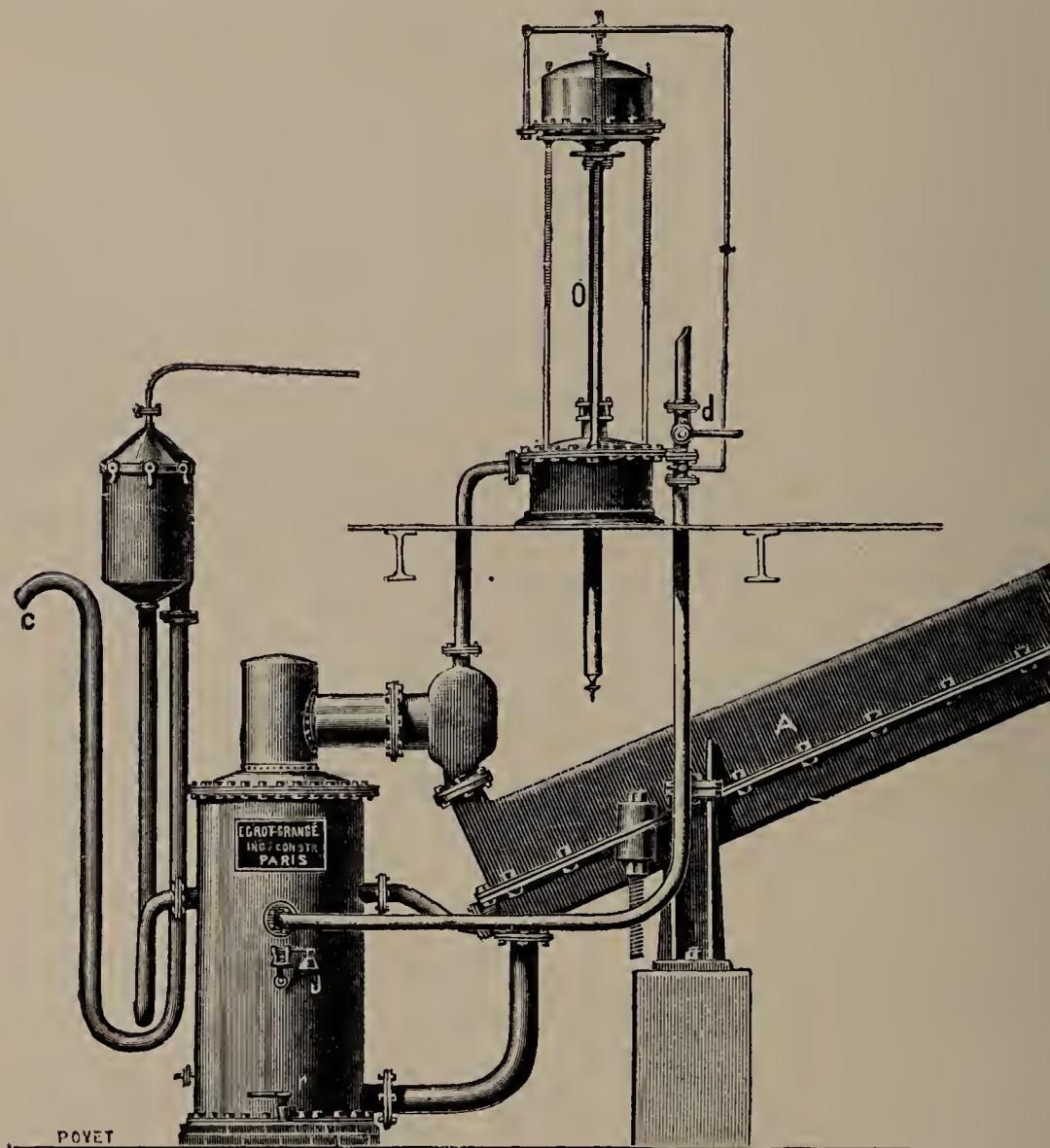


FIG. 63.—Guillaume's plant for surface heating of wash to be distilled which cannot be diluted by open steam (EGROT and GRANGÉ).

distilled. The apparatus used in working limpid liquids is furnished with a wash feed regulating tank supported on the apparatus itself. Finally, the constructors supply a very solid iron foundation, on which all parts of the apparatus are mounted in such a way that no support requires to be fixed on the spot, and each component having its place marked on the support, fitting-up is rendered very easy. The inclined column, it is said, replaces

the old columns which comprised a great number of superimposed plates, and which occupied a great height. There is no need for manholes for cleaning purposes, the bottom of the column being readily released in a few minutes and the interior laid bare for inspection.

The type M<sup>a</sup> of this distilling column (Fig. 64) is intended to produce raw spirits of 60°-70° G.L. (22°-26° Cartier). The inclined column A can be used to distil all sorts of liquids, limpid or thick, wine, *piquettes*, ciders to make common brandy, cane juice for rum or tafias, grain wash for making raw spirits, or wash from potatoes, beets, Jerusalem artichokes, etc. Certain alterations are made on the column, the wine-heater, and the wash-extractor in the case of thick washes. The advantage claimed for this apparatus is its great working capacity and small compass, which renders the fitting-up easy and simplifies supervision. The exhaustion of the liquids distilled is absolute. It is economical because it minimises loss of alcohol and requires less steam in proportion to the diminished size of the cooling surfaces, and that without prejudice to the economy of the system already described. This column can be worked by any labourer on the spot. In cleaning there is no need for manholes. The inside is accessible in a few minutes by releasing the big joint by which the whole plate is let down below the column. In larger-sized columns the bottom is lowered by a special arrangement as far as desired by turning the nuts placed on four screw bolts. To raise it into position the same tightening screws are turned in an opposite direction, so that the bottom of the column reassumes its original position, and the joint may be again made by fixing the bolts. The liquid or wash to be distilled, which comes from a tank placed above the apparatus, is fed by a ball valve in the regulating tank K, from which a pipe leads to the tap *m*, which regulates the output of the apparatus. The wash reascends into the wash-heater, in which it is heated as it ascends; it then descends through a pipe (bent at the top) into the top of the inclined column, in the circuitous descent of which it is exhausted, and finally it is discharged through the siphon tube C after passing through the extractor D. The exhaustion of the *vinasse* is constantly

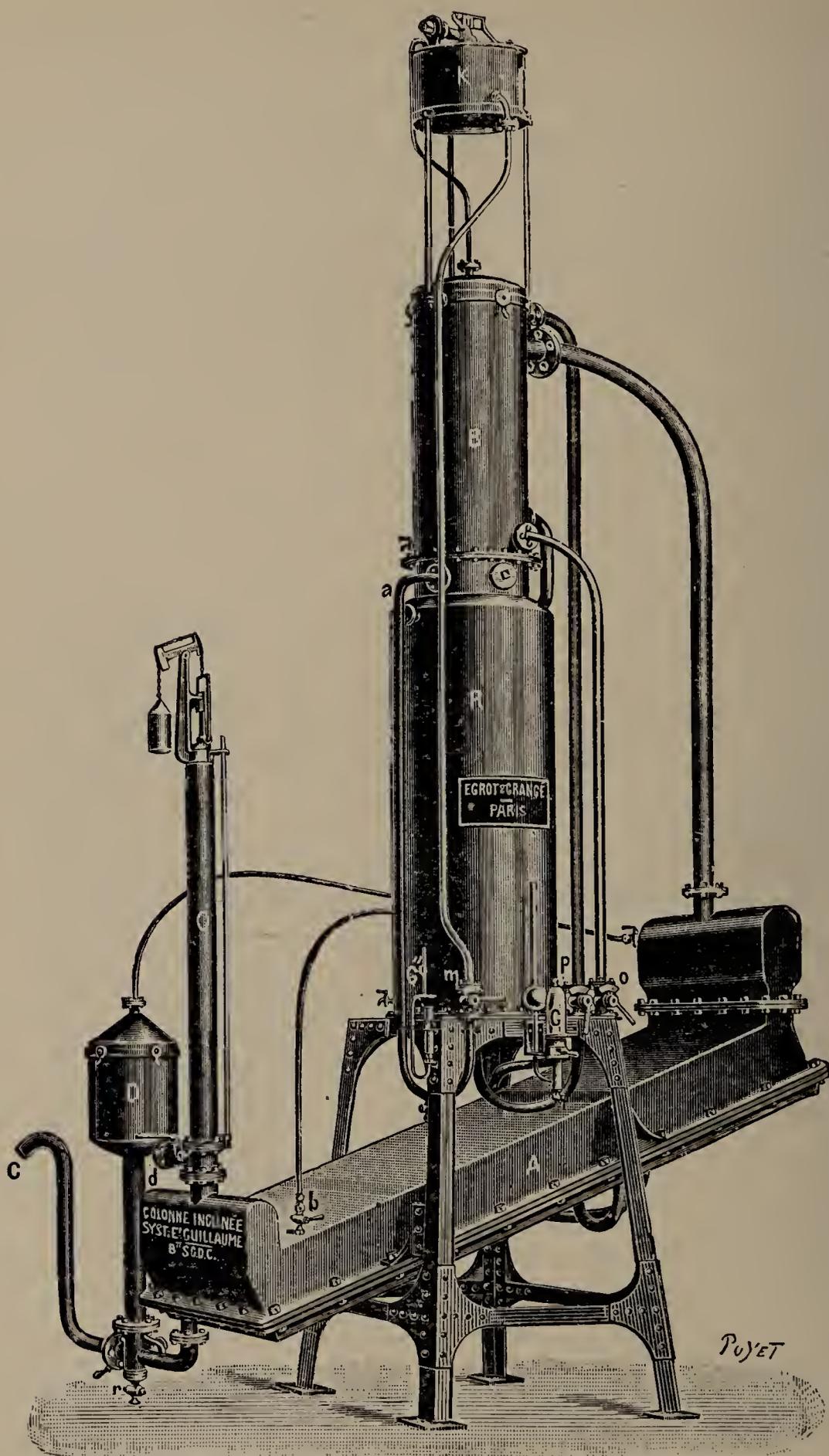


FIG. 64.—Guillaume's distilling column, Type Ma. A, distilling column; *a*, entrance of wash into wash-heater; B, wash-heater; *b*, exhaust test-tap; *c*, exit of spent wash; D, spent wash extractor; *d*, steam valve; G, alcohol test-glass; G'', exhaust test-glass; K, feed regulating tank; *m*, wash regulating valve; O, steam regulator; *o*, valve for regulating strength of spirit; *p*, water entrance tap; R, refrigerator; *r*, exit tap under extractor D (EGROT and GRANGÉ).

verified by the test-glass G", through which the liquid formed by the condensation of the vapour from the spent wash may be inspected. This vapour is brought by pipe from *b*.

The steam enters the bottom of the inclined column, rises from one division to another, becoming charged with alcohol from the descending wash. The alcoholic vapours issuing from the inclined column are condensed in the wash-heater B, then in the refrigerator R underneath. Finally, the alcohol is collected at the test-glass G. Heating is regulated automatically by *o*, which acts on the entrance steam valve. The alcoholic strength is regulated by opening the tap O more or less. By a slight alteration, in distilling weak washes, water may be dispensed with, and the wash alone used for condensation.

According to the raw materials used and the final quality aimed at, the result to be attained by distillation-rectification varies from one distillery to another. Take, for example, an existing distillery with rectifying plant desirous of improving the quality of its spirits without double rectification, which would entail much loss and great expense in fuel. It would not be a question of attaining perfection at the outset, as the operation would be completed by existing rectifiers. Elimination of the roughest impurities; expulsion of ethers and oils as far as practicable would suffice. This is done in the column shown in Fig. 65. There is no continuous purifier. Ethers are eliminated by "pasteurisation" on top of the rectifying plates. By the invariable regulation of flow the extraction of ethers is effected to as small or as great an extent as desired. The oil extraction identical with that of the continuous rectifier must be placed underneath the rectifying trunks and not below the feed. Being a high strength column, is it more economical than low strength distillation followed by rectification? Not only is it not more costly than ordinary columns, but it may even consume less steam, especially when the nature of the liquor being distilled allows a forewarmer to be used. Again, further rectification in the intermittent rectifier will yield a much larger proportion of *bon gout*; there is both economy in fuel and improvement in the spirit. The pasteurised alcohol it yields is chemically very pure.

It attained 96 per cent. at Clastres decolorised permanganate in forty-five minutes and even more. One hour five minutes as a maximum at the exit from the test-glass, that is a chemical purity not attained by double rectification. Were analysis the only criterion of the market value of alcohol, this column would be perfect. But pasteurised alcohol retains traces of odours of origin more perceptible since there are no others to mask them. In the distillation of wine, cider, fruits and cane sugar, the odours of origin are very precious, distinguishing brands and affording evidence of genuineness highly appreciated and esteemed by consumers. But in distilling beets, the odour of the distillate, however weak it may be, is a drawback which must be eliminated either by continuous purification, at low strength, or, finally, by continuous or even intermittent rectification. The pasteurised phlegm, filtered or not, being of very great purity, further rectification has few impurities to eliminate. It no longer produces *mauvais gouts*, but *moyens gouts* only. With continuous rectifiers these are in very minimum proportion. Here it is not rectification but distillation that furnishes the concentrated *mauvais gouts* to be

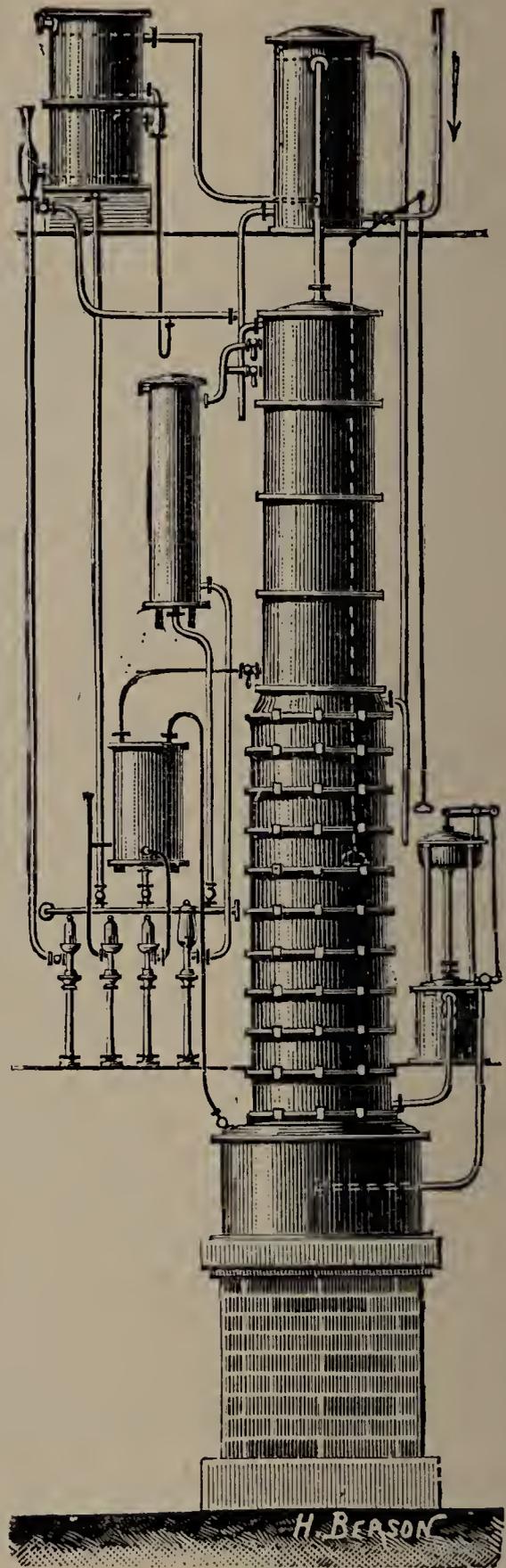


FIG. 65.—High strength rectifying column, model No. 3 (E BARBET).

sold for denaturation. It is better, therefore, to produce them at the outset of maximum strength. The oils being extracted between 40 per cent. and 60 per cent. are not marketable at that strength, and therefore they must be concentrated. This is effected automatically by a small column closely resembling Barbet's laboratory apparatus.

The oils extracted from the distilling column enter half-way up the additional apparatus, the upper plates of which effect a summary purification. The pasteurisation from this additional column yields oils at 90°-94° very bad, containing only traces of ethylic alcohol, whilst the latter ascends to the condenser and refrigerator, from whence it descends into the exhaustion plates of the rectifying column. The ethers may be concentrated by diminishing the flow of first runnings, but that would affect the quality of the pasteurised, whilst with the additional apparatus in Fig. 66 the pasteurised is improved. At the condenser exit the vapours, instead of going direct to the refrigerator, are concentrated in a small plate column underneath the refrigerator. It is solely in these plates that the first runnings settle; moreover, the retrogradation from the first condenser is itself very impure. The pasteurised derived in a direct line from it is itself so much the better, and the flow of ethers may without detriment be diminished to a minimum, 2 or even 1 per cent., according to the impurities in the wash.

*Suppression of "mauvais gout" rectifiers.*—The concentration of oils and ethers is important to agricultural distilleries which do not rectify, or for rum or brandy distilleries, as it does away with the necessity of installing a special rectifier for all the *mauvais gouts*, whilst furnishing 96-97 per cent. of *bon gout premier jet*. For large distilleries likewise the *mauvais gout* rectifier is also dispensed with. Some distillers have no low produce apparatus, but return all the *mauvais gouts de tête et de queue* from the rectifier to the wash. This, however, is not to be recommended since either the column does not completely exhaust the wash, and the oils disappear with the *vinasses*, a loss pure and simple, or exhaustion by the column *n* is complete, as it ought to be, but the oils are evaporated afresh and contaminate the

phlegms. It then becomes impossible to produce a suitable alcohol by rectification, whilst with rectifying columns the return of the *moyens gouts* of rectification is quite legitimate, the column being provided with special outlets to both first and last runnings products. Accumulation therefore becomes impossible, and the

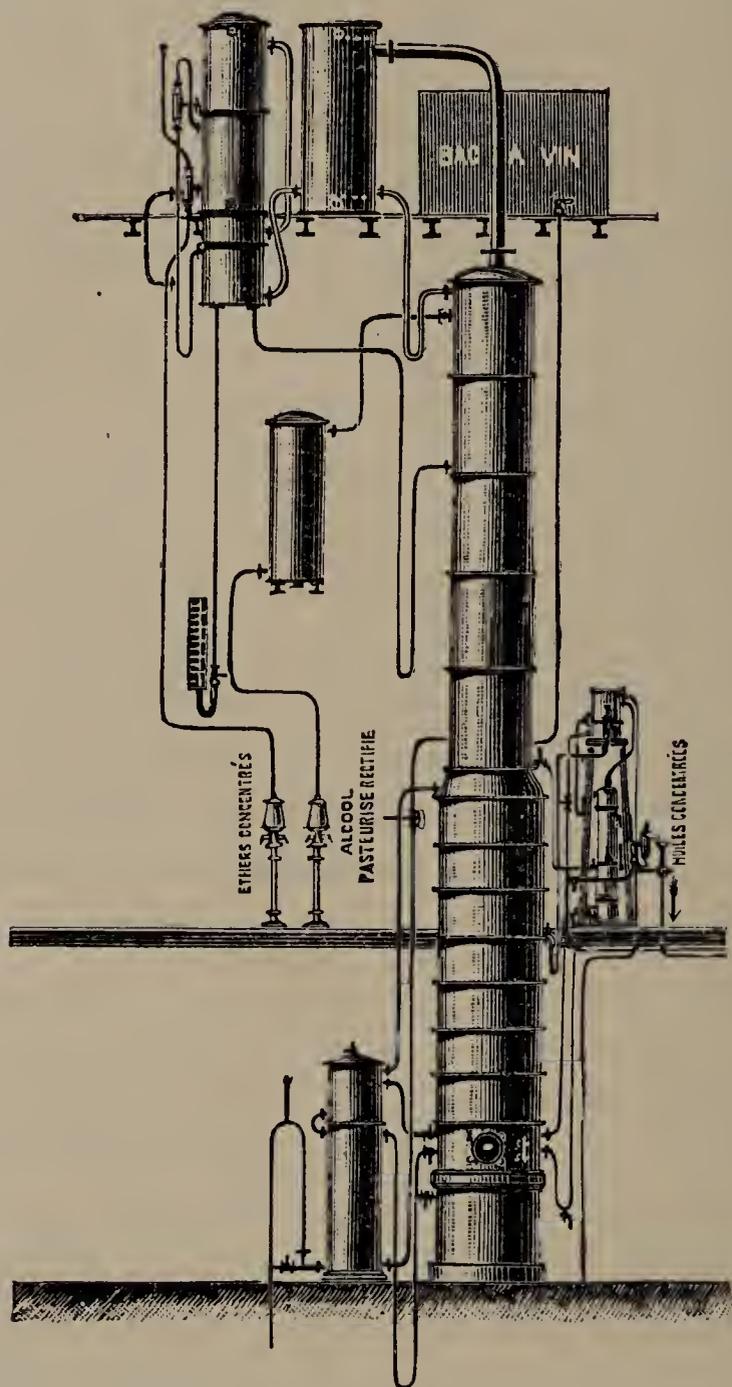


FIG. 66.—Continuous rectification column, with apparatus for concentrating oils  
(E. BARBET).

distiller is freed from the constant worry of indefinite repassages. The manufacture is continuously resolved without loss into pasteurised *bon gout*, and *mauvais gouts* to sell. One meets distillers with old plant who boast of never selling *mauvais gouts de tête*, *i.e.*, they use these products up by repassing them. This is a manufacturing loss, and therefore wrong.

*Messrs. Blair, Campbell & McLean's Single-column Rectifying Still.*—The single-column rectifying still of Messrs. Blair, Campbell & McLean (Fig. 67) is simple in construction and easily worked. The distillation being carried on by means of steam, which is passed into the lower portion of the still. Alcohol and water vapour pass up through the rectifying column, which is divided into compartments, into a rectifier, from which the impurities pass back to the still head, the purified alcohol passing through a condenser to the storage tank.

*Twin column.*—Take a distillery to be erected, say a beet distillery, *working only three months in the year*; an agricultural and not a commercial company, the owners desiring to make alcohol as good as possible, but aiming at making permanent customers with high-grade alcohol. It is desired to work economically to avoid traces of original odour, which are a defect of the preceding simple rectifying column; low-strength purification is resorted to, which easily eliminates any such impurity. The column is double or twin. In the first part purification from ethers is effected; in the second, which constitutes rectification proper, pasteurised alcohol is obtained at  $97^{\circ}$ , and the oils are separated in the concentrated condition. The apparatus thus *seems* identical with the German or English twin columns, but it differs from them essentially by *the first column possessing, above the feed, some rectifying plates, as well as a condenser and refrigerator. In the German columns, the alcoholic vapour of low strength issues from the top of the first column, to be rectified in the second. Here it is not the vapour but a purified boiling phlegm which passes from the first to the second column,* and this modification is vitally important as regards purification. The alcoholic vapours at about  $40^{\circ}$ , produced by the exhaustion plates, ascend to the upper plates, where a sorting of alcoholic liquors, of increasing strength, is effected as in the preceding apparatus. A very small volume is extracted so as to get it very concentrated. The retrogradation from the condenser is thus itself very impure, but that is no disadvantage, because it is no longer intended to extract high strength pasteurised alcohol. The retrogradation is allowed to descend to the base of the rectifying plates, and, in this long

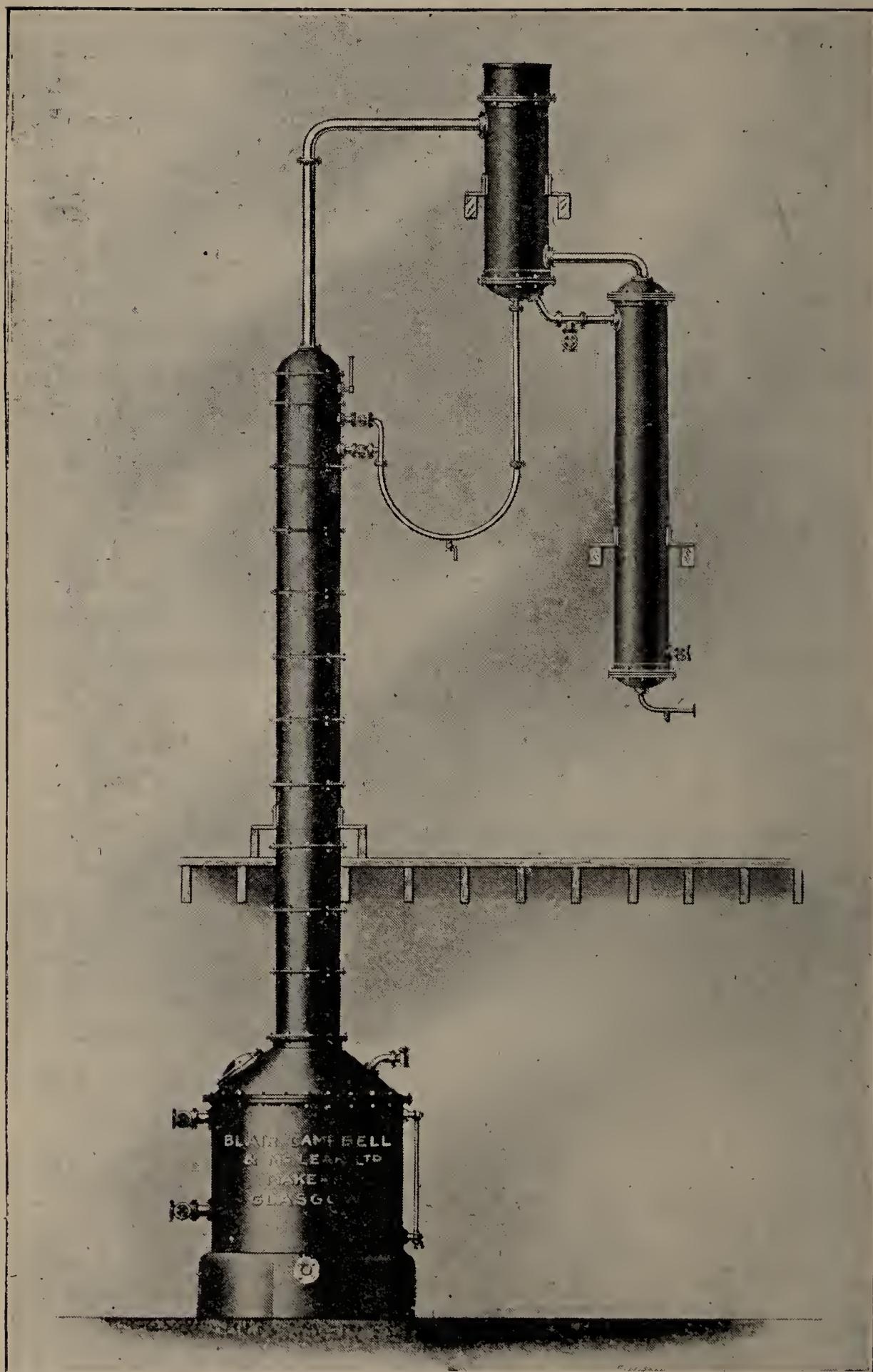


FIG. 67.—Messrs. BLAIR, CAMPBELL & McLEAN's single-column rectifying still.

methodical passage, it has ample time to become thoroughly pasteurised, *i.e.*, to lose all trace of first runnings impurity. The whole retrograded liquid is discharged through E. This liquid is perforce of the strength of the vapour disengaged from the wash at the feed plate, diminished by the small quantity resulting from the extraction of the ethers. Take this vapour to be 40° G.L., and that 2½ per cent. of ethers are extracted for each hectolitre at 40°, one litre in the state of ethers will be drawn off. The strength will therefore be brought to 39°. It contains the whole of the *last* runnings impurities, but is free not only from first runnings impurities, like all pasteurised, but also from odours. After having vaporised the latter by boiling the wash, they cannot be stopped by the liquid of the first rectifying plates, for they are too slightly soluble in dilute boiling liquors of about 40°. It is only on the upper plates that they might be partially redissolved by alcohol at 95°, but without injurious accumulation, owing to the permanent outlet furnished by the extraction of ethers. A purified phlegm, fit for supplementary rectification in the twin, issues from E. The phlegm passes through a closed special type test-glass H to ascertain the strength and rate of production. The strength is, moreover, shown by the boiling-point registered by the gauge T. The second column is like a continuous rectifier with fresh pasteurisation. Fig. 68 shows an oil concentrator like that described above adapted thereto. The twin rectifying column occupies by itself the whole distilling-room. The 4 or 5 per cent. of *moyens gouts* extracted from the second column return to the first. So only extra pure alcohol issues on the one hand, and ethers and oils are reduced to a minimum volume on the other.

As to the elimination of fusel oil, etc., Barbet, as far back as 1881, attempted to solve the problem by using a thermometer in the lower part of the apparatus, where he thought the oils ought to occur. This was much too low down, and being unable to regulate his plant, he took the opportunity of placing a small tap above the feed plate on the side opposite to the entrance of the phlegm, to permanently maintain a stream of liquid, the strength of which was shown by an alcoholometer. By maintaining

constant alcoholic strength on the feed plate, the working of the apparatus was regulated. Barbet attempted in this way

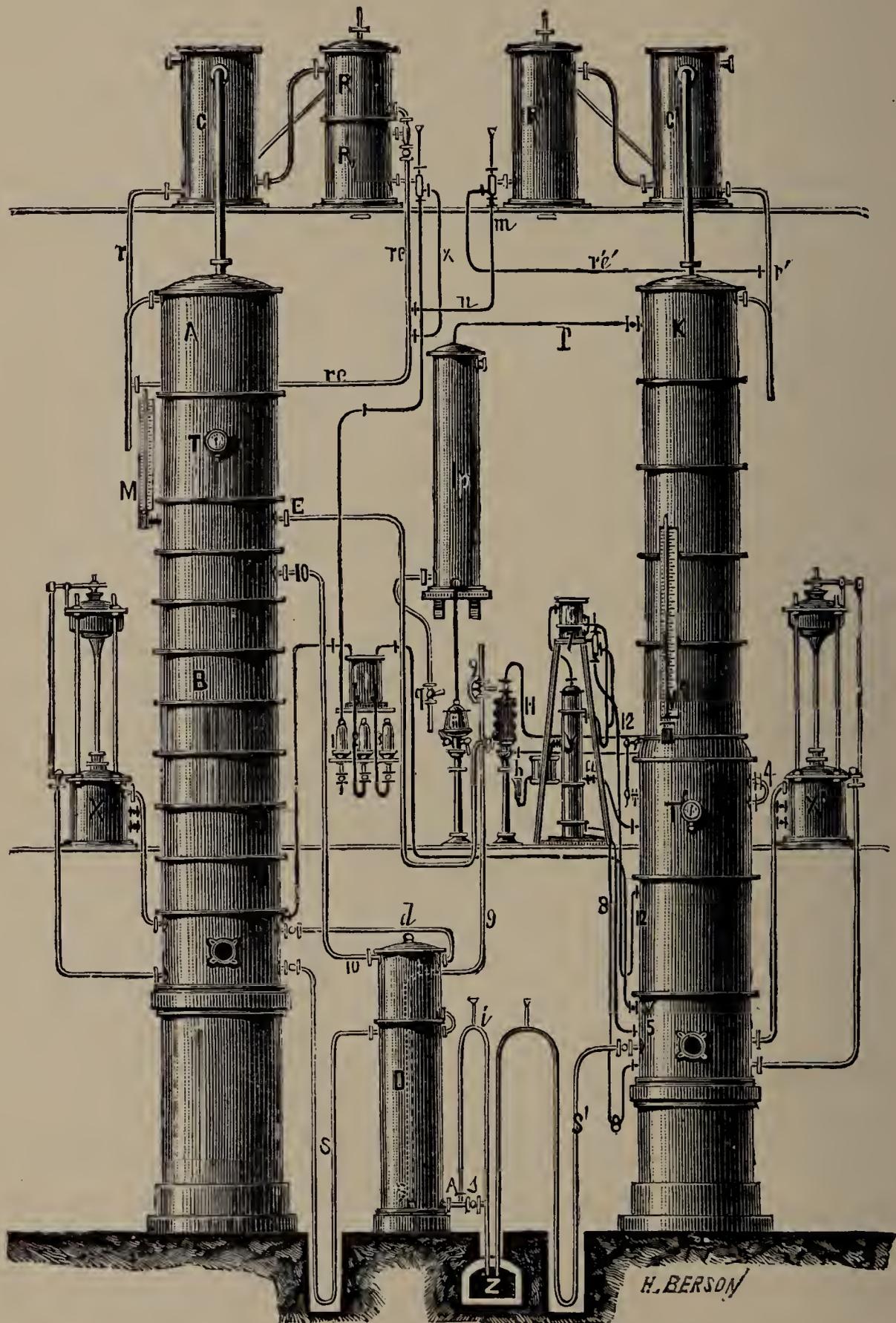


FIG. 68.—Twin rectifying column (E. BARBET).

to eliminate the odours from the last runnings, which were due, he imagined, to the fact that the exhaustion rose too high up the

apparatus without his being aware of it. The extraction of this sample of the liquor rendered the service desired; at the same time, it was a revelation. Instead of getting a liquor analogous to the phlegm, a vile product issued, full of amylic oils, demonstrating that in this zone of the plant there was an accumulation of fusel oil which refused to issue at the bottom with the residual water. After extraction had proceeded for some time, the product assumed its normal state as regards fusel oil. The apparatus was gradually desaturated, and the problem of the continuous elimination of these impurities was solved. It was, however, highly improbable that the stage where the phlegm was diluted with the rectification reflux was the best to obtain a maximum amylic extraction.

Barbet made extractions from the plate immediately above, then from the plates beneath, and ended by recognising that the zone of maximum contamination was two or three plates below the feed plate, on a plate where the normal strength of the liquid was  $40^{\circ}$  to  $50^{\circ}$ . Hence, the continuous extraction of amylic oils at  $40^{\circ}$  to  $50^{\circ}$  serves to extract these impurities and also to regulate the feed tap of the rectifier. That level in the apparatus is precisely the spot where the least difference in the exact rate of feeding has the most rapid reaction on the strength. This being the most sensitive stage, Barbet installed a dial thermometer T at this level, as a complementary control visible at a distance. The distiller regulates his feed by its indications, because he is warned by it sooner than by his fusel test-glass. It is not difficult to establish equilibrium between the inflow and the outflow. The plates of the rectifying column being charged with liquid, all these distinct reservoirs added together represent a very imposing bulk of alcohol which constitutes the fly-wheel of the operation, and it is interesting to know that, when the feed is completely cut off, *bon gout* still flows for two and a half hours, so great is the bulk of liquid to be exhausted. In all continuous apparatus the stock *facilitates* regulation. No objection has been raised against the triple effect in sugar works, under the pretext that it was difficult to regulate the respective feed taps of each vessel, so as to suffice for the output of the factory, and to maintain in each vessel both

a suitable gravity and the level of the liquid best adapted to good working. Owing to the stock, these adjustments are made very easily. Certainly it is good policy to increase the "fly-wheel" of the apparatus, and, in 1893, Barbet did so by increasing the depth of liquid on each plate in the bottom parts of the rectifier: Barbet's comb-shaped bubbling caps lend themselves readily to this increase in the height of the liquid. In 1894 he went further still in the model of rectifying column installed at Clefs, which contained a real phlegm reservoir, which can be seen under No. 12 (Fig. 68), the level of the gauge glass. This reservoir receives the purified phlegm coming from the first column pipe E and closed test-glass H, and the reflux from the rectifying trunks K. From there the mixture redescends to the lower plates by the tap 4 and the elbow pipe. The tap was so regulated as to keep the reservoir half-full, and, according as the thermometer T' showed access of alcohol or exhaustion, the tap 4 was adjusted so either to further charge the reservoir or to allow it to empty. That gave time to correct the production of phlegm from the first column by a slight retouch of the wash entrance tap 9. In a word, a reservoir was available which could momentarily absorb the excess of alcohol or restore it at the will of the operator.

Barbet afterwards found that this precaution was superfluous, since the increase of the stock on each plate was quite sufficient to ensure regular working. On the other hand, Guillaume, in his accumulating receiver V (Figs. 74, 75, and 76), has accumulated the stock, and, according to Barbet, in such a way as to exaggerate the inconveniences of the system by introducing a useless complication. Far from regulating the working, he has, according to Barbet, introduced a permanent cause of irregularity and inconvenience.

For rectifying phlegm, wine, or fermented wash, the methods of working are slightly different, but there is nothing to prevent a continuous rectifier of wash being used in rectifying phlegms. The reciprocal is not possible, because wash or wine requires larger surfaces than phlegms. Take first of all the rectification of phlegms. The complete and radical solution consists in using

an isolated column for the amylic products. The last runnings purification column E E<sup>1</sup> (Fig. 69), fed by the refluxes from the

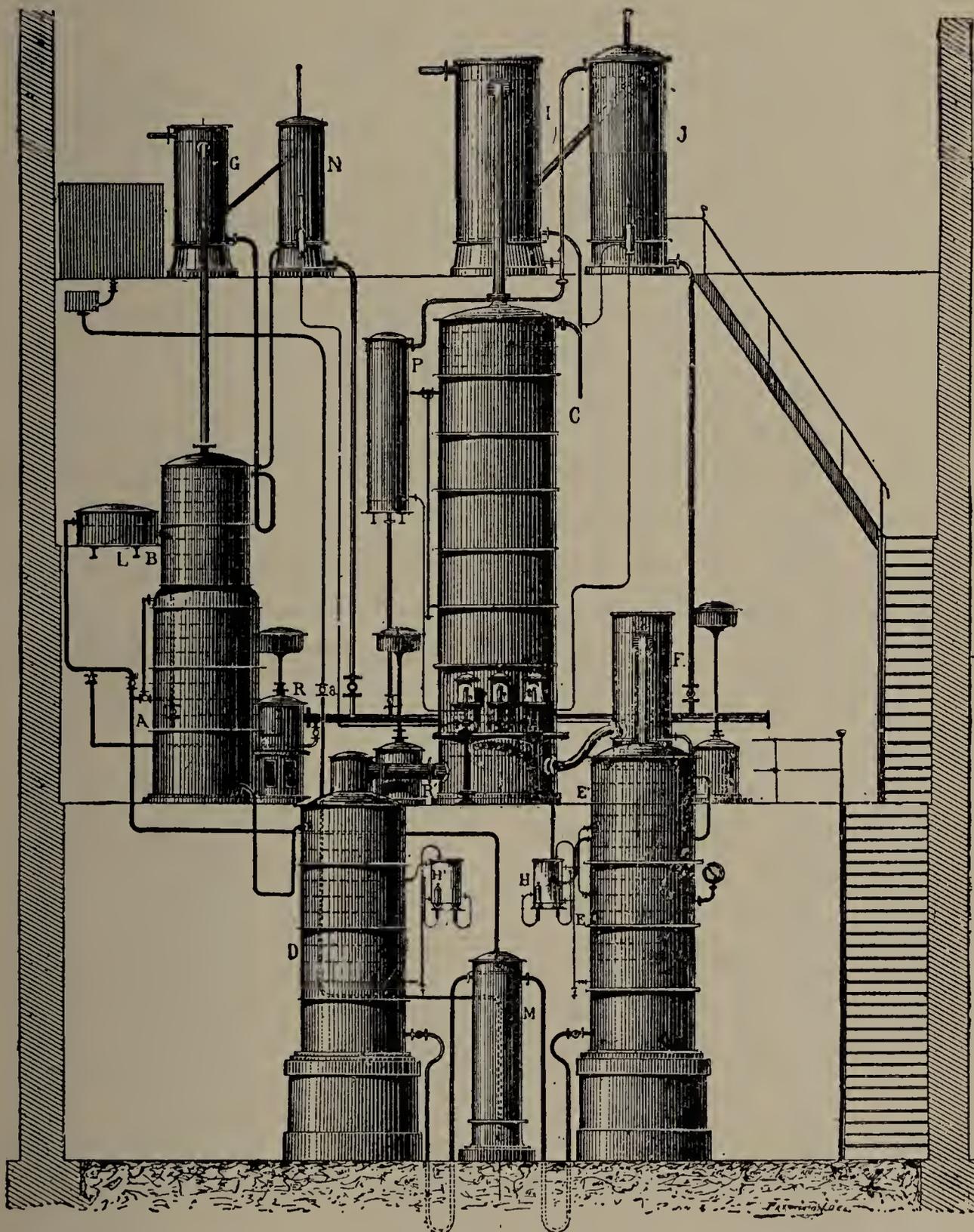


FIG. 69.—Continuous rectifier of raw spirit (phlegm), exhausting by two distinct columns both the purified phlegm and the amylic refluxes from the rectifying plates (E. BARBET).

rectifier, and by these alone, then possesses, like every other continuous distilling column, its exhaustion plates E, those of concentration E<sup>1</sup>, its condenser F, and its steam regulator. As

to the purified phlegms, they also have their separate plates of exhaustion D, and their steam regulator R.

Now the refluxes from the rectifier are much more impure than the purified phlegm. It is therefore better to purify and refine them apart. The model represented here exhausts the purified phlegm in the column D, and the refluxes in the column E E<sup>1</sup>. The plates E exhaust the refluxes, and the plates E<sup>1</sup> concentrate the last runnings, which is effected by a special supplementary condenser F. An appropriate extraction is made of the last runnings impurities, and the high strength alcoholic vapours which issue from the condenser F, being purified by the supplementary refining, pass to the bottom of the rectifier C, and mix with the vapour of the purified phlegm. The results obtained by this complementary purification are excellent. The alcohol has been compared favourably with that furnished by the same phlegm after two intermittent rectifications, each preceded by very energetic filtration over wood charcoal. All Russian refineries of alcohol using Barbet's rectifiers have been authorised by ministerial decision to dispense with filtration through charcoal.

By the continuous rectification of wash, merchantable alcohol may be obtained very pure on analysis. But such alcohols always retain traces of odours imbibed by the too intimate contact of the alcohol at high strength with the fermentation gases. This drawback is very much accentuated when badly fermented washes are rectified directly, the fermentation of which has generated gases which prolonged boiling is alone capable of expelling from the wash, such as sulphuretted hydrogen, for example. This is obviated, and alcohols obtained, not only pure on analysis, but irreproachable as regards both taste and smell, by exhausting the wash totally, in a first column, in which also first and last runnings products are separated. The alcohol purified by pasteurisation passes into a second column, where, after dilution with hot water, it is thoroughly purified by live steam. The product thus obtained, doubly purified as regards first and last runnings, is run into a complete rectifying column, where it is concentrated at great strength and perfectly purified.

It follows that the pasteurised alcohol is absolutely pure and perfectly free from odour or taste which would betray its origin.

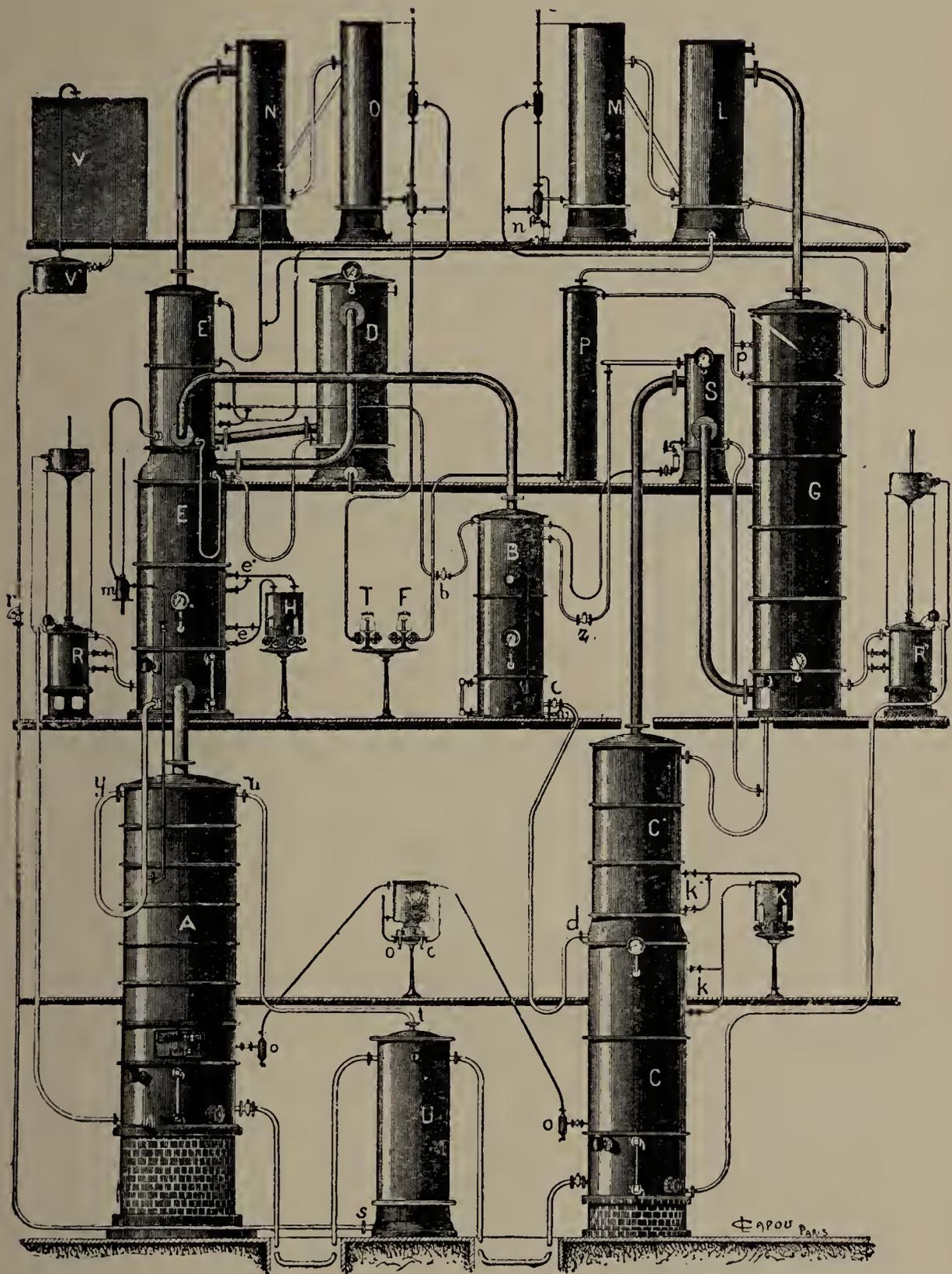


FIG. 70.—Plant for continuous indirect rectification of wash. V, feed tank (wash); V<sup>1</sup>, feed regulator; A, E, E<sup>1</sup>, rough purifying columns; N, O, condenser and refrigerator; H, oils test-glass for rough purifier; T, first runnings from rough purifier; B, purifier; d, feed from purifier to rectifier C, C<sup>1</sup>; G, rectifier; U, recuperator forewarmer; L, condenser; M, refrigerator; P, refrigerator of pasteurised alcohol; F, test safe; K, fusel oil test safe (E. BARBET).

The plant consumes but very little more coal than the direct rectifiers.

The exhaustion column C (Fig. 71) is surmounted by a series of

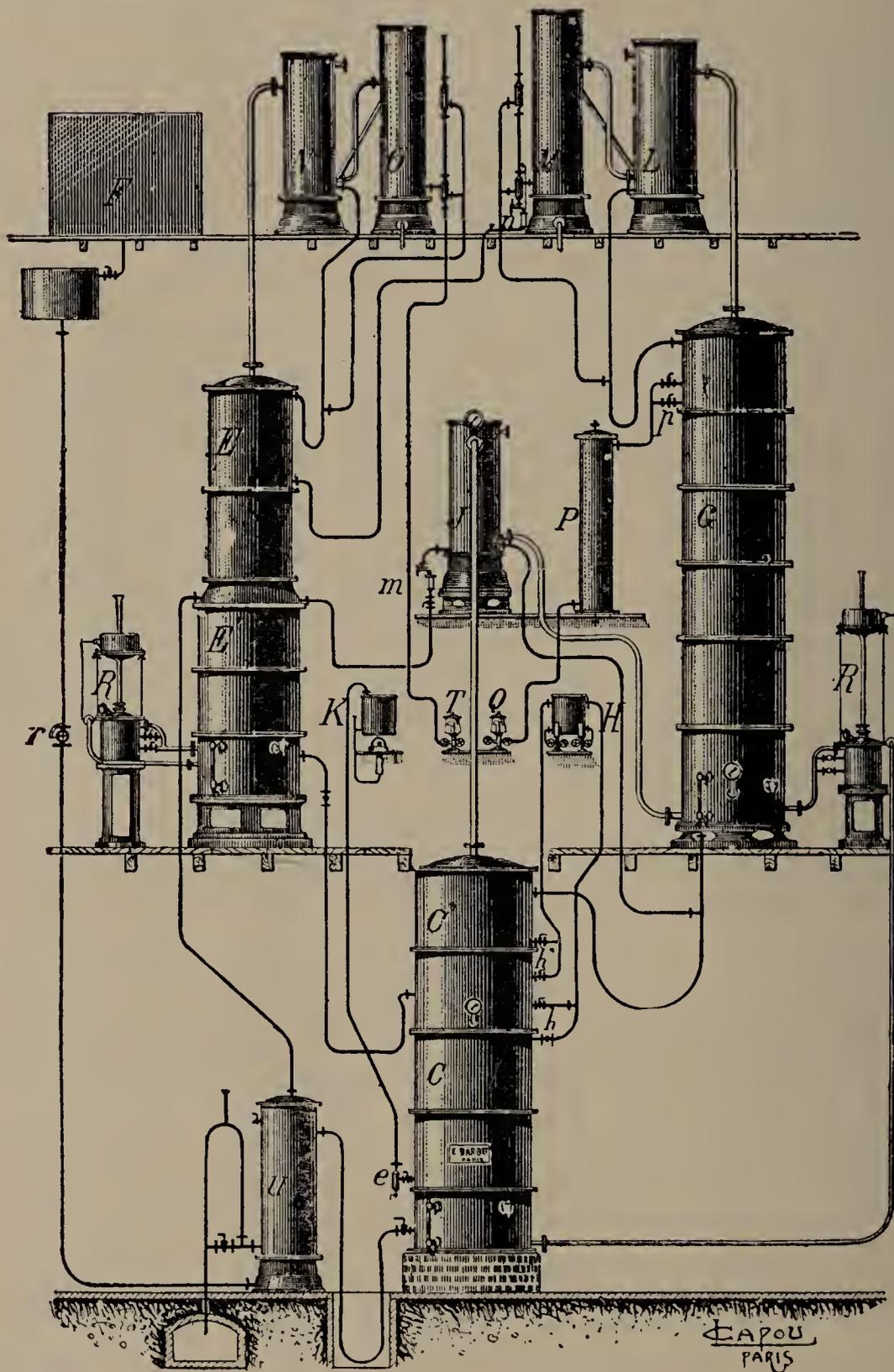


FIG. 71.—Continuous rectification of raw spirit (phlegm); F, feed tank with feed regulator; U, forewarmer; R, R<sup>1</sup>, steam regulators; E, E<sup>1</sup>, preliminary purifier; N, condenser; O, refrigerator for first runnings; J, special condenser; G, rectifier; C, C<sup>1</sup>, last runnings rectifier; P, refrigerator for pasteurised alcohol; T, test safe for first runnings; Q, test safe for pasteurised alcohol; K, exhaust test safe; H, test safe for oils (E. BARDET).

plates C<sup>1</sup>, and a special condenser J. These new adjuncts form a complete last runnings purifier. They cleanse the vapours emitted by the exhaustion of the purified phlegm and by the refluxes from the rectifier G. The oils are extracted at *h h*<sup>1</sup>. Very pure alcohol results.

*Blair, Campbell & McLean's Double-Column Still.*—Fig. 72 shows a double-column continuous rectifying still made by Messrs. Blair, Campbell & McLean, the distillation and rectifying columns being shown in partial section. A is a supply tank which delivers the fermented wash by gravity through the float regulating tank B to the preheater C, in passing through which it is heated by the exhaust hot waste liquor. It then passes through pipe D into the upper chamber of the distilling column and falls from one chamber to another, meeting in its passage a current of steam introduced through the automatic regulator F. The distillation column is divided into compartments by means of concentrating plates through which there are a number of holes covered by “bells,” through which the vapour passes into the liquid standing on the plates, thus evaporating off the alcohol. The exhausted hot wash passes out from the bottom of E through the preheater C, where its heat is utilised. The vapour of alcohol and water passes into the lower part of the rectifying column H, in which water gradually condenses on the plates, the lighter spirit passing out at the top into the rectifier J to the condenser K, where it liquefies and passes down through a test-glass to a receiver. Pure alcohol passes from the pipe M to the cooler N, and through the test-glass O to the receiver. The impure alcohol from the plates of the rectifying column gradually passes downwards, loosing some alcohol on the way, and runs by the pipe L back into the column E, where it is redistilled. The heavier fusel oils pass out of the rectifying column to the condensing tank, from which they are drawn off and returned to the still. The working of the still is practically automatic, the flow of fermented wash and the supply of steam being adjusted by the controlling devices. so that once started it requires very little attention. It yields nearly pure neutral alcohol of 95 to 97 per cent. strength in one operation.

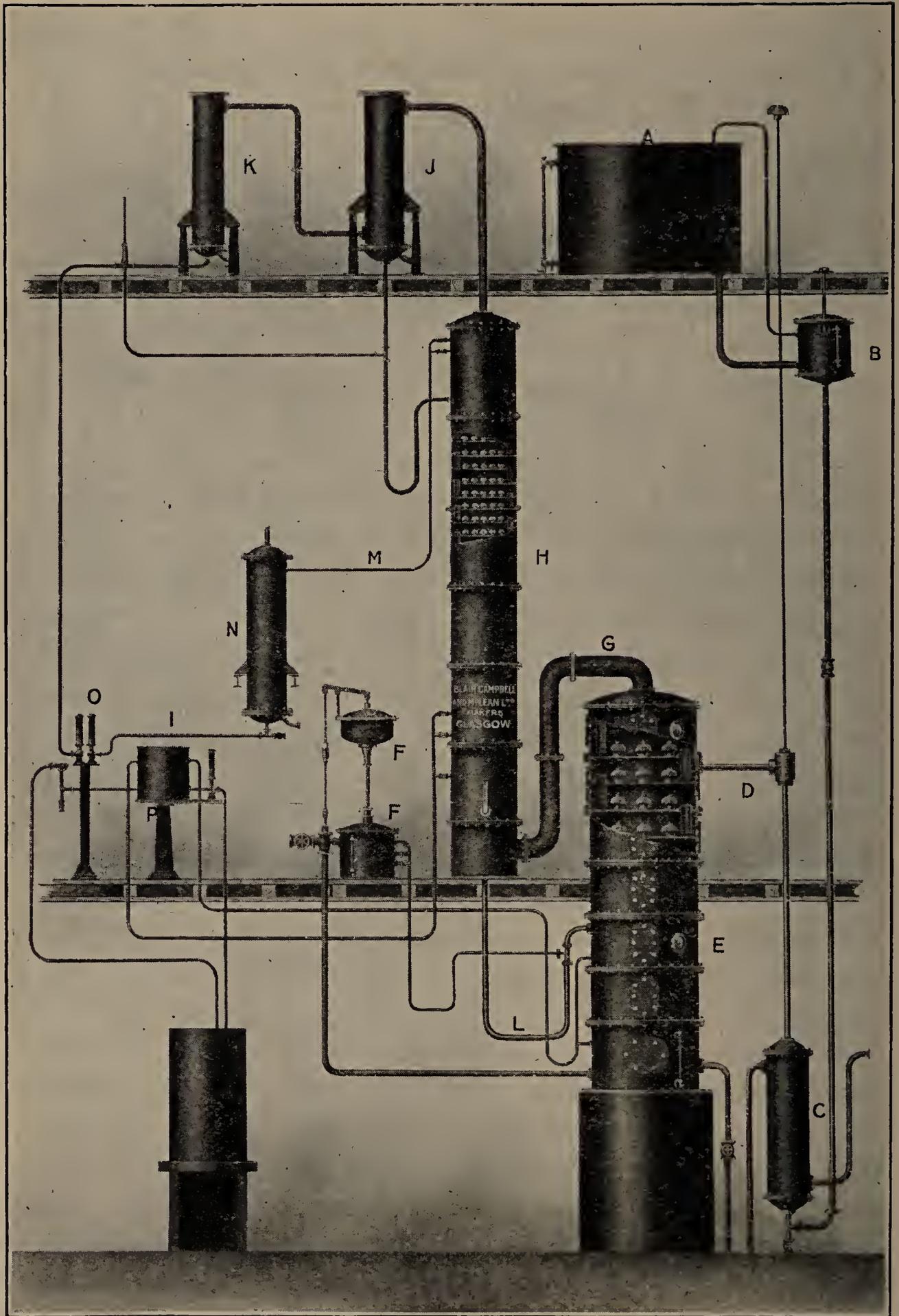


FIG. 72.—Messrs. BLAIR, CAMPBELL & McLEAN's double-column still.

*Messrs. Blair, Campbell & McLean's Triple-column Continuous Working Still.*—Fig. 73. In this apparatus the fermented wash is pumped up into the wash supply tank A, from which it flows to a float regulating tank L, which regulates its flow to the separating column C, through the wash preheater B, heated by the spent wash from the still D. In C the liquor parts with the aldehydes and ethers, which pass up the pipe into the rectifier H and through the condenser I, passing through the regulating tester T to a storage tank. C is divided into compartments by plates perforated with holes or “bells,” through which the vapour blows into the liquid remaining on them, the liquor as it accumulates falling down from one compartment to the other, meeting in its course a current of steam delivered through the automatic regulator S. From the separating column C the liquor flows into the still D, also divided into compartments, in which it passes from one to the other, finally passing out through the preheater B, where it heats up the inflowing wash. Steam passes into D through the automatic regulator S1, and meeting the liquor evaporates off all the alcohol and heavier spirits, which pass into the boiling column rectifier D1, in which the heavier oils which condense pass back to C, while the purer alcohol goes to the rectifying column E, and thence to the rectifier F and cooler G, from whence it passes through the cooler V to a rectifying tester T1, and then to the storage tank.

The production of good quality rectified alcohol, *i.e.*, at least “commercial,” prior to Guillaume’s researches, required, he says, two distinct operations: (1) Distillation, usually in continuous stills, consisting solely in extracting the alcohol from the wash, to produce a more or less pure spirit of a greater or less strength, but which it was always necessary to rectify afterwards, so as to obtain an alcohol which could be marketed as fine. (2) Rectification in intermittent or continuous rectifiers. Continuous rectifiers have already taken the place of the intermittent form to a considerable extent. It is evident they possess all the advantages of continuous over intermittent distillation; steam economy; economy in handling and working; initial economy in installing. Continuous rectification eliminates the intervals in intermittent

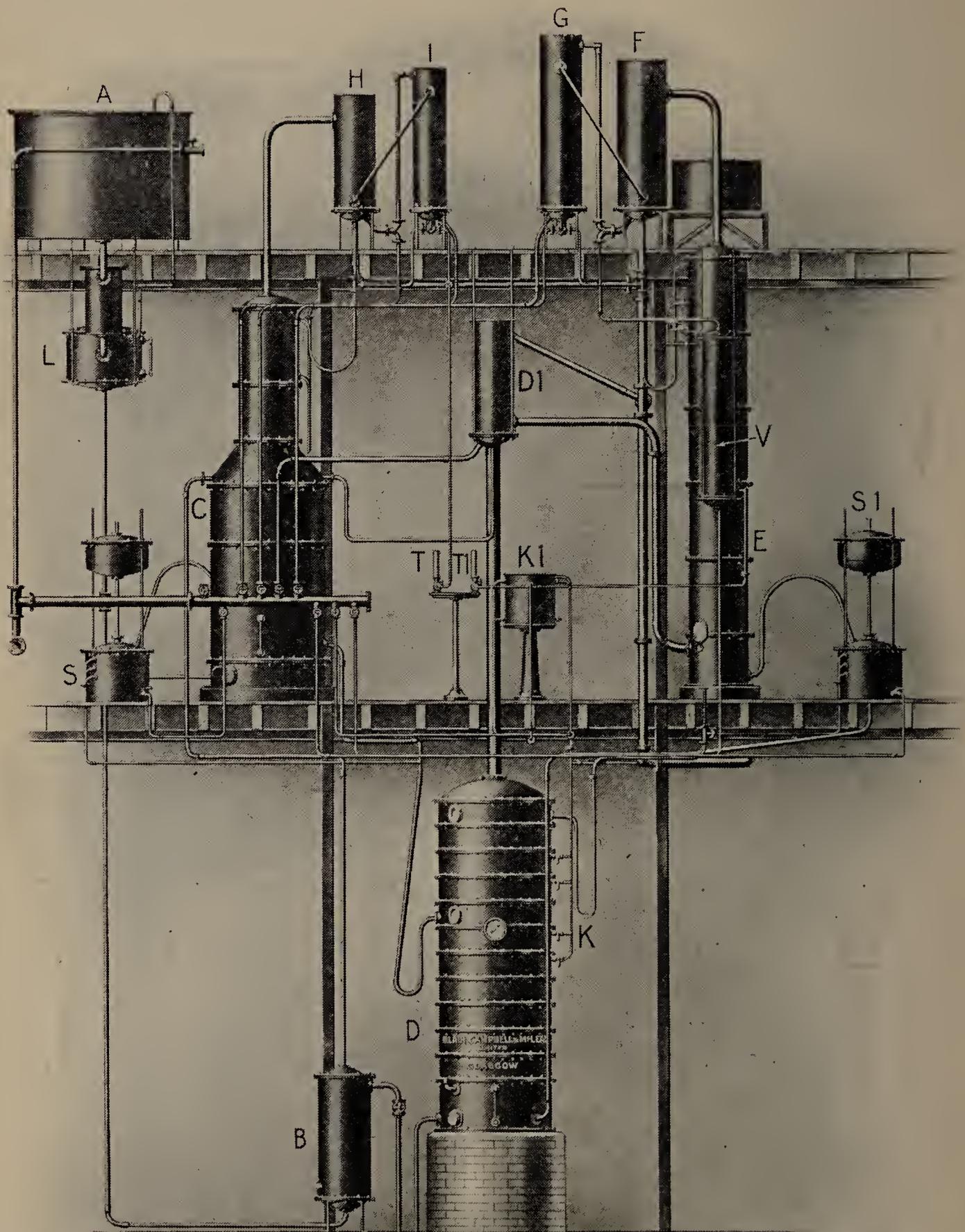


FIG. 73.—Messrs. BLAIR, CAMPBELL & McLEAN's treble-column still.

rectification when the worm safe yields middling quality products only. With continuity of distillation and rectification the products once classified on the plate columns remain constantly fractionated into—(1) foreshot products ; (2) fine tasted, finished alcohol ; (3) after-products. These three products run respectively to their individual worm safes, and always in the same proportion once the latter is regulated. It is thus easy at a low estimate to get 90 per cent. of very pure fine alcohol, and 10 per cent. for the aggregate of the foreshot and after-products, and that at the very outset and without redistilling. The coal bill is thus reduced to a very great extent (1) from the continuity of the operation ; (2) from the utilisation of the heat of the spent wash ; (3) from the permanent regularity and routine established ; but (4) more especially owing to the elimination of the numerous redistillations incidental to intermittent plant. The continuous distillation-rectification of wash is more economical and profitable than independent continuous rectification, because it produces rectified alcohol directly of very good quality in the above proportions, starting directly with the distillation of wash without any intermittent operation. To the above advantages must be added the elimination of all cooling, of warehousing, and intermediate manipulation of phlegms with loss of alcohol.

The chief difficulty which, according to Guillaume, his predecessors had not surmounted was the irregular working of plant of this nature. The least hitch, he says, caused the classification of products in each part of the plant to be disturbed. Each variation in heating, in the alcohol, in the feeding, in the condensation, destroyed the regularity of the classification on the plates, causing the alcohol issuing from the worm safes to vary likewise. Again, owing to the want of fixity in the strength of the alcohol intended to be rectified, one is never sure of the wash being regularly exhausted of alcohol. These drawbacks were common up to recently to all continuous columns working up to great strength. With the latter, if the quantity of alcohol extracted at the gauges does not correspond exactly at each moment with the amount introduced into the columns, there is continual irregularity in the working of the column. If the quantity

flowing out of the gauge glasses be less than the quantity fed into the rectifier, immediately the column is surcharged in strength; and as the plates on the top, already of very great strength, cannot be charged further, this surcharge must be absorbed by the lower plates, so that if matters be not at once remedied the non-exhaustion makes itself felt very quickly, and the surplus of alcohol at once flows away with the spent wash. Guillaume states that in important continuous plant other than his own, there are losses of alcohol in the spent wash amounting to more than 8 per cent. in normal industrial working, as letters from interested parties showed. And this state of affairs will remain until remedied—almost always to fall into the opposite extreme—the flow of alcohol to the gauges when the feed has not been diminished in the necessary proportions. If, now, the amount flowing to the gauges is greater than the feed, the column must discharge itself gradually of the exact deficiency produced by the excess of flow. The plates then become exhausted successively from the foot of the column upwards, so that if the plate where the strength was, say  $40^{\circ}$ , was at the moment the 12th plate, it becomes first the 13th, then the 14th, the 15th, the 16th, the 17th, etc., continuing to ascend thus higher and higher until the flow from the gauges is exactly diminished or the feed increased proportionately. To get back to the normal classification on each corresponding plate, the correction to be made has to be exceeded, which causes matters to fall into the opposite extreme, and so on. It will thus be seen that there is a series of continuous fluctuations on the plates; at one time, when the column is overloaded with alcohol, the excess soon descending to escape by the wash outlet; or, again, the strength diminishing on the lower plates, the seat of the maximum concentration of the fusel, etc., oils ascends towards the top, thus approaching nearer and nearer the spot where the ethylic alcohol ought to remain absolutely exempt therefrom, and the series of plates which succeed this maximum concentration of fusel oils, contaminated in decreasing proportion, ascend in the same way. Hence the great difficulty and danger in managing continuous columns for great strengths up to now, and the almost absolute

impossibility of getting perfect products regularly. It is impossible to regulate the flow of alcohol to the gauges by hand so that it corresponds at each instant with the feed. But Barbet (p. 258) contradicts this. By taking the vapours directly from the distillation of the wash resulting from good normal fermentation, more than 90, and up to 93 and 94, per cent. of rectified spirit of great purity, and 6 to 10 per cent. for the aggregate of foreshot and after-products, are obtained by the first distillation and without re-passing. Guillaume guarantees the total loss to be lower than 1 per cent. The installation expense is less by the smaller space occupied by simplification of piping and diminution in the number of reservoirs. The mildness and mellowness of the alcohol produced by Guillaume's appliances he claims as incomparable. He asserts that it is the repeated and prolonged heating, the redistillations, which imparts dryness and hardness to alcohol. Now, with direct distillation-rectification these defects are reduced to a minimum, in a single operation, without any redistillation, the spirit only remaining a very short time in the apparatus A. Large numbers of installations have already been made, and are said to be working satisfactorily.

*Guillaume's direct distillation-rectification Type C, Agricultural* (Fig. 74).—A, inclined distillation column; a, tank for wash to be distilled; b, cold water tank; C, rectification column; D, final purification column; e, water feed tap; I, wash-heater; K, condenser; K<sup>1</sup>, gas refrigerator; N, distillation gauge; O, *bons gouts* and first runnings refrigerator; Q, last runnings refrigerator; R, *vinasse* extractor; r, siphon for *vinasse* discharge; S, steam regulator; s, tap and pipe for leading phlegms to distilling column; UU<sup>1</sup>, water regulator; u, extraction taps for intermediate impurities; V, accumulating receiver; v, last runnings extraction tap; X, exit test-glasses of *bon gout* alcohol; Y Y<sup>1</sup> Y<sup>2</sup>, test-glasses for first runnings, last runnings, and intermediate products; Z, exhaustion of spent wash test-glass. It is claimed by the constructors that: This apparatus is easily worked, and expends no more steam than an ordinary distilling column. It consists of (1) the distilling column A. The figure shows that described on pages 243 *et seq.*, but this organ may be of another

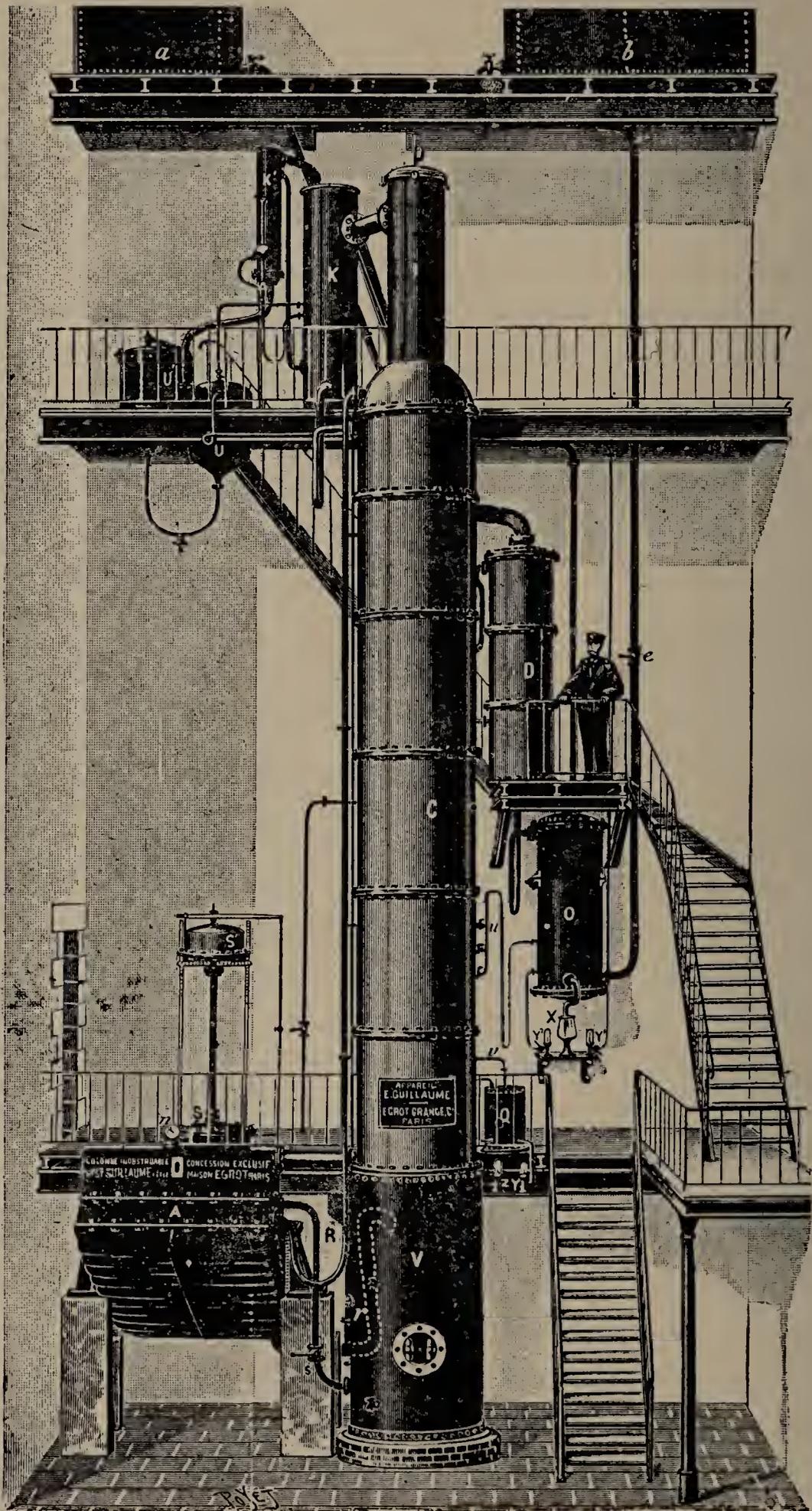


FIG. 74.—Direct distillation-rectification (GUILLAUME, EGROT, and GRANGÉ)  
Type C, Agricultural.

type than that prescribed. Any plate column may be utilised, but then it would not be possible to return the retrogradation from the rectifying column C, on account of the great height which it would then be necessary to impart to the distillery building, and it is necessary to provide in that case a special exhaustion column for this retrogradation. This column is shown in Fig. 75 (p. 272).

(2) The concentration column C, with the accumulating receiver V, and surmounted by its condensers K I, in which the extraction of the first and last runnings and oils is effected which flow in a continuous fashion from the test-glasses Y Y<sup>1</sup> Y<sup>2</sup>. The accumulating receiver V, filled with the retrogradation liquid from the rectifying column, assures regularity and stability in working.

(3) The final purifying column D, and the refrigerator O. This final purification column submits the finished alcohol to a real redistillation, which extracts from it the last traces of first runnings products. After cooling in O, the pure alcohol flows into the test-glass X. It is claimed that in these types C of Guillaume's apparatus the rectified alcohol is of very good quality, and even sold at a premium varying according to local circumstances and at a guaranteed strength of 96°-97° G.L., the alcohol being obtained in one single operation in the proportion of about 90 per cent. of the whole alcohol produced, and with, so to speak, no loss in rectification, which latter is always guaranteed under 1 per cent. It is worked by farm labourers formerly quite unacquainted with distillation. They become quite expert at working it in a few days.

*Guillaume's system of direct distillation-rectification Type B, Industrial* (Fig. 75). A, distilling column; a, tank for wash to be rectified; B, low strength purification column; B<sup>1</sup>, column for concentration of first runnings products; b, water tank; C, rectifying column; D D<sup>1</sup>, final purification column; e, water feed tap; h, steam tap for distilling column; I, wash-heater, steam tap purification column; J, condenser; P, gas refrigerator; L, condenser; L<sup>1</sup>, gas refrigerator; O, *bon gout* alcohol and first runnings products refrigerator; p p<sup>1</sup>, pressure indicators; Q, last runnings refrigerator; R, spent wash extractor; r, spent wash exit; S, steam (distillation) regulator; s, tap and pipe for

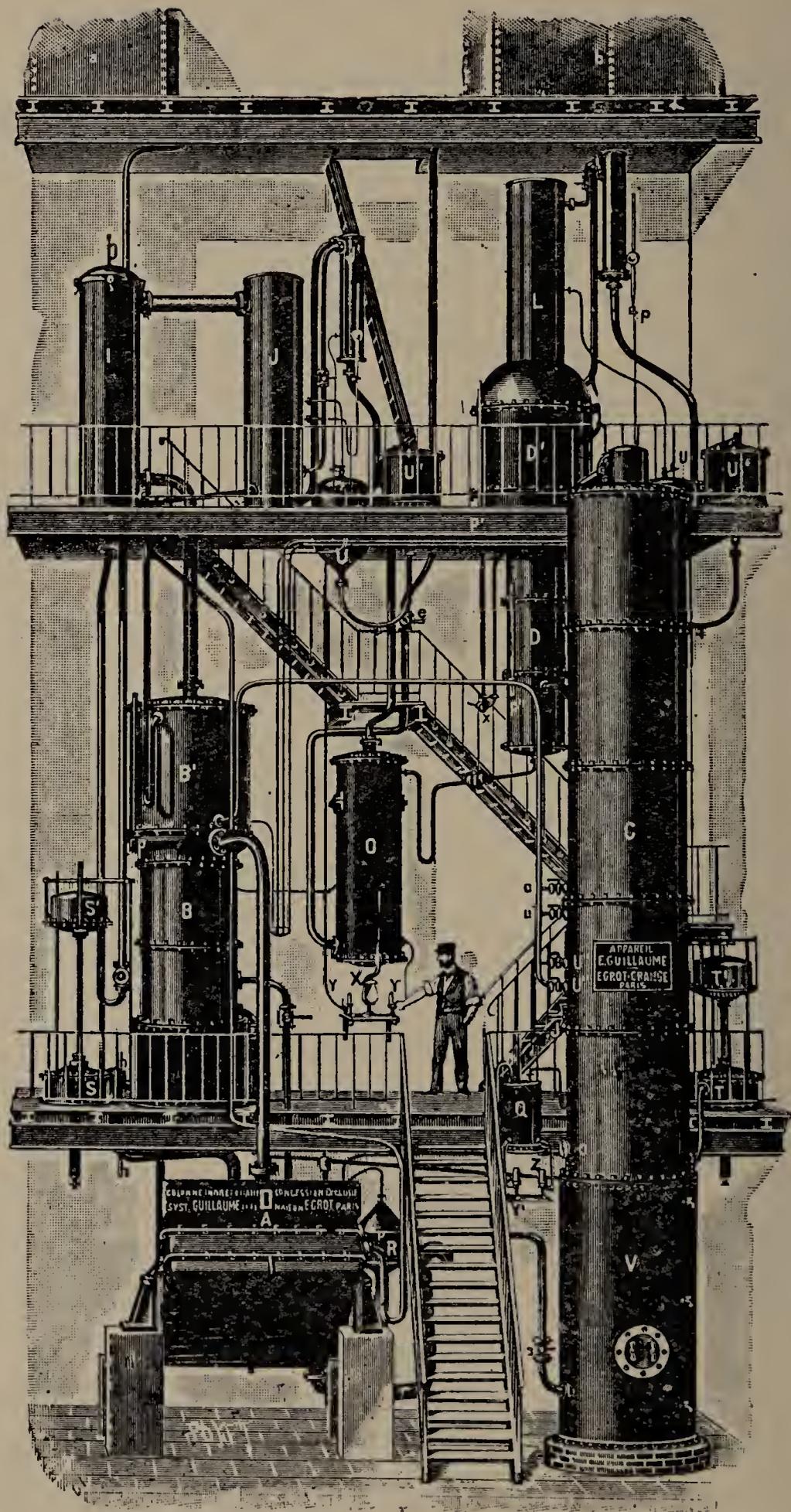


FIG. 75.—Direct distillation-rectification (GUILLAUME, EGROT, and GRANGÉ)  
Type B, Industrial, for beets, molasses, grain, potatoes.

leading wash to distilling column ; T, steam (rectification) regulator ; U U<sup>1</sup>, water regulators ; *u u*<sup>1</sup>, return from rectification to first runnings products ; V, accumulating receiver ; X, *bon gouts* test-glass ; Y, first runnings test-glass ; Y<sup>1</sup>, last runnings test-glass ; Z, test-glass for determining degree of *exhaustion of spent wash*. According to the constructors of this plant, the preceding type C (Fig. 74) does not suit a distillery whose chief aim is the production of alcohol of very high quality capable of competing with the great French and foreign brands. The type B is particularly designed for that purpose. The proportion of *bon gouts* of high quality obtained as first distillate by this apparatus varies with the substance treated, but is often over 92 per cent. of the total alcohol obtained. The bad flavoured first and last runnings are collected apart. The arrangement differs very appreciably from the preceding type. As they issue from the distilling column A the crude alcoholic vapours pass up into the concentration column B B<sup>1</sup>. The major part of these products is extracted at the bottom of B<sup>1</sup>, whilst the first runnings are concentrated in the upper part and extracted from the condensers I J. The whole of the part B of this purifier placed below the entrance of the vapour from the distilling column completes the purification from first and last runnings by a real partial redistillation of the retrogradation from the part B<sup>1</sup>. The purified alcoholic liquors pass to the rectifying column proper C and to the accumulating receiver V. The process is finished by removing the last runnings in V, whilst on the top the concentrated alcohol is sent to the final purifying column D D<sup>1</sup>, to be there freed from first runnings products, which are reformed in the course of working.

*Guillaume's continuous rectifier, Type S.*—Fig. 76, *a*, tank for raw spirit to be rectified ; *b*, water tank ; B, first runnings products concentrating column ; C, rectification column ; D D<sup>1</sup>, final purification column ; E, exhaustion column ; F, low wine (to be rectified) feed tap ; H, recuperator (raw spirit heated by hot spent wash) ; K, condenser ; K<sup>1</sup>, gas refrigerator ; *k*, steam pipe ; O, refrigerator ; *o*, gauge ; *pp*, pressure gauges ; Q, last runnings refrigerator ; T, steam regulator ; V, accumulating

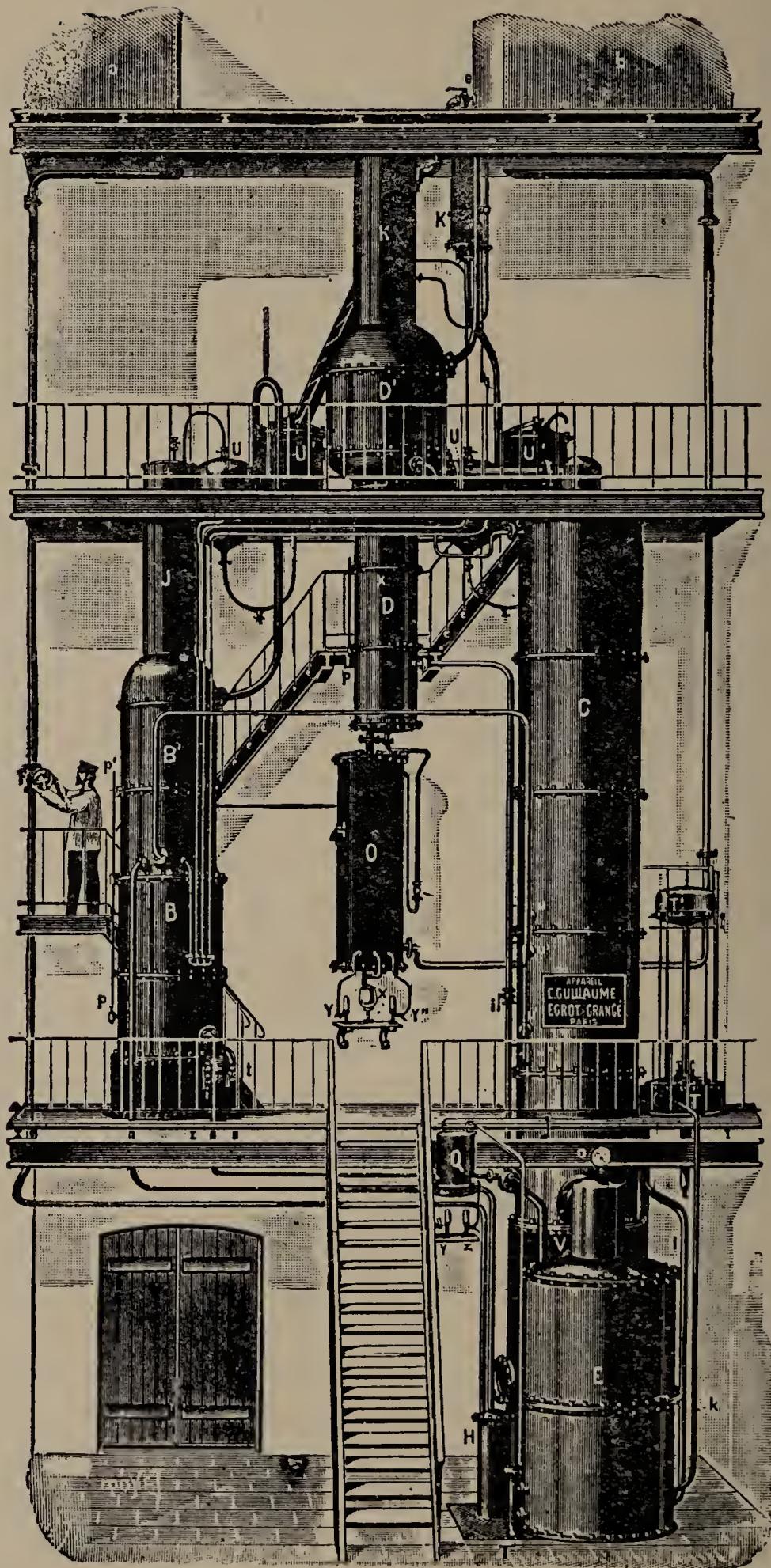


FIG. 76.—Continuous rectifier Type S (GUILLAUME, EGROT, and GRANGÉ).

receiver. According to Guillaume, his continuous rectifier type S is based on the same principles and comprises almost the same organs as the direct distillation-rectification apparatus type B (Fig. 75) previously described. There is no distillation column, and the purification column which received the phlegms in the state of vapour from the distilling column is fed with them in the liquid state. Owing to the absence of the distilling column into which the retrogradations from the rectifier are returned, the continuous rectifier comprises an exhausting column E, from the bottom of which the spent liquor flows to the drum. The problem of complete rectification is less difficult to solve than that of the production of rectified alcohol from wash. Regularity in working is in that case more easily attained, for if the quality of phlegms may vary, their strength is uniform. Guillaume says the instability of working of the plant formerly in use has rendered the adoption of this principle very slow. Instability is denied by Barbet, though less advantageous than direct distillation-rectification as far as economy is concerned, continuous rectification none the less presents great advantages over the ordinary methods, yielding from 90 to 94 per cent. of *bon gout* alcohol in a single operation, with great economy in fuel compared with intermittent rectification. Elimination of leakage in rectification, great facility in working, complete stability of strength and quality of the good alcohol flowing from the test glass are ensured security for the perfect exhaustion of the spent wash from the apparatus. Of course, any system of prior purification used in intermittent rectification, whether by chemical treatment or by filtration, may be adopted with continuous rectification, but such costly operations are unnecessary with Guillaume's apparatus. Rectifiers have lost their interest, says Guillaume, since direct distillation-rectification yields products equal to those from independent rectifying plant. The literature of this controversy is far too voluminous to be further dealt with here.<sup>1</sup>

<sup>1</sup> For the theory of fractionation and further information on distillation plant see Stephane Musfarand, *Jour. Soc. Chem. Ind.*, 1920, 299 T. The following recent patent specifications may also be consulted: E. A. Barbet, *French Pats.*, 343, 488, Apl. 16, 1904; 428, 299, June 17, 1910, 470, 242, June 10, 1913, and addition Apl. 25, 1914; 478, 885, June 9, 1914, and addition Oct. 19, 1914; 478, 946, Oct. 19, 1914; E. Guillaume, *French Pat.*, 403, 385, May 26, 1909.

G. Renaix has taken out a patent (French Pat. 428,012, June 11th, 1910) for the rectification of alcohol, the principle of the process being that a high vacuum is maintained throughout the distillation and condensing plant, the claim being made that the flavour of the products is materially improved thereby.

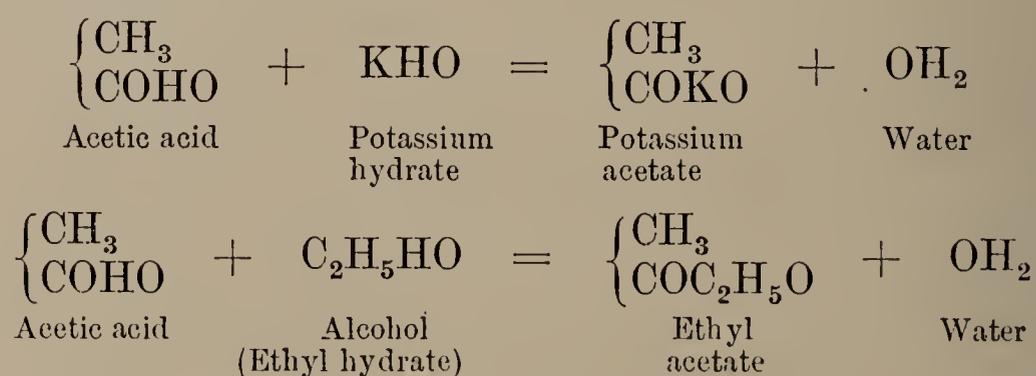
## CHAPTER XII.

### THE MANUFACTURE AND USES OF VARIOUS ALCOHOL DERIVATIVES, ETC.

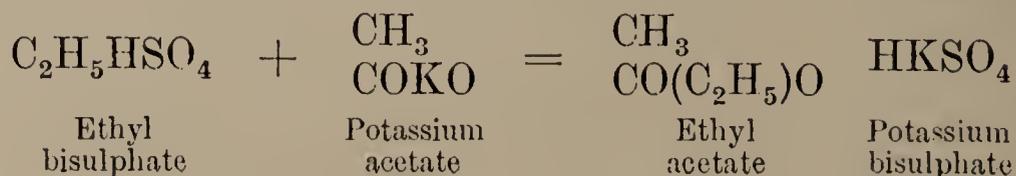
*Alcohol* is the raw material of numerous compounds used in industry, many of which are almost of equal importance as itself. Want of space prevents our dealing with any but the more important compounds. Of the higher alcohols we can only treat of *amyl alcohol* and its most important derivative, *amyl acetate*. Allied to ethyl alcohol are the spirituous products *methyl alcohol* and *acetone*, obtained from wood spirit, a constituent of the wood tar and crude pyroligneous acid obtained by the destructive distillation of wood. *Ether*.—Amongst the substances directly derived from alcohol into which the compound ethyl enters as an essential constituent, ether occupies the first place. Germany and France each produces eight to ten times as much ether as Great Britain. This is a serious matter as affecting the manufacture of smokeless powders. The ether is erroneously called sulphuric ether, since sulphuric acid is used in producing it. Ether is used alone as a solvent in a great number of instances, also in conjunction with other solvents, particularly alcohol, for dissolving many commercial products, more especially nitro-cellulose. It was used as an anæsthetic prior to chloroform, and is still used for the same purpose. Its use in refrigeration (cold storage) is well known. *Ethyl chloride*.—By the action of hydrochloric acid gas on alcohol, ethylic chloride is obtained. It is a liquid boiling at  $12.5^{\circ}$  C., often used as an anæsthetic, but several fatal accidents have recently resulted from its use. *Ethyl bromide* and *iodide* are produced by acting on alcohol with bromine and iodine respectively in presence of amorphous phosphorus. These two heavy liquids are used in making intermediate products in coal-tar colour manufacture, which constitute the starting-points of

at least 100 distinct dyes, each of which is a well-defined chemical compound. *Chloroform*.—This liquid, the use of which as an anæsthetic is well known, is manufactured in large quantities by heating alcohol with a solution of calcium chlorohypochlorite (*chloride of lime*). *Chloral*.—By the direct action of chlorine on alcohol, chloral is formed, and the latter treated with an alkali is decomposed into formic acid and chloroform. Both are well-known medicinal agents. *Bromoform*.—By the action of bromine on alcohol, bromoform is produced. It is prescribed as a remedy for coughing. *Iodoform*.—By the action of iodine on alcohol, the well-known antiseptic iodoform is formed. *Paraldehyde*, *sulphonal*, and *urthane* are three other substances directly derived from alcohol all used as anæsthetics or soporifics.

*Ethereal salts*—*Haloid ethereal salts*—*Esters*—*Compound ethers*.—These compounds correspond to the metallic oxides, to the metallic salts of the halogen acids, and to the metallic salts of the oxyacids. The acids from which they are derived may be either mineral or organic ; but the base must always be organic. Ether corresponds to potassium oxide. The haloid ethereal salts—ethyl chloride, ethyl bromide, ethyl iodide—correspond to potassium chloride, bromide, and iodide. The ethereal salts are produced by reactions analogous to those employed for the preparation of metallic salts, thus :—



But as the hydrates of the organic radicals do not react with acids so energetically as potassium hydrate, it is often advisable to employ the acid in the form of a potassium salt, and the radical as a sulphoacid ; thus, with acids of the acetic series :—



SULPHURIC ETHER (Ethyl Ether).—Ordinary ether  $\left. \begin{matrix} \text{C}_2\text{H}_5 \\ \text{C}_2\text{H}_5 \end{matrix} \right\} \text{O}$ —  
 molecular weight, 74; boiling-point,  $34.8^\circ \text{C}$ .; flash point,  
 $-20^\circ \text{C}$ .—is prepared by the action of sulphuric acid on ordinary  
 (ethyl) alcohol. On a small scale the process is conducted as  
 follows: Nine parts of concentrated sulphuric acid and five parts  
 of alcohol are carefully mixed together, and, after cooling, heated  
 to a temperature of  $90^\circ \text{C}$ ., and a continuous stream of alcohol is

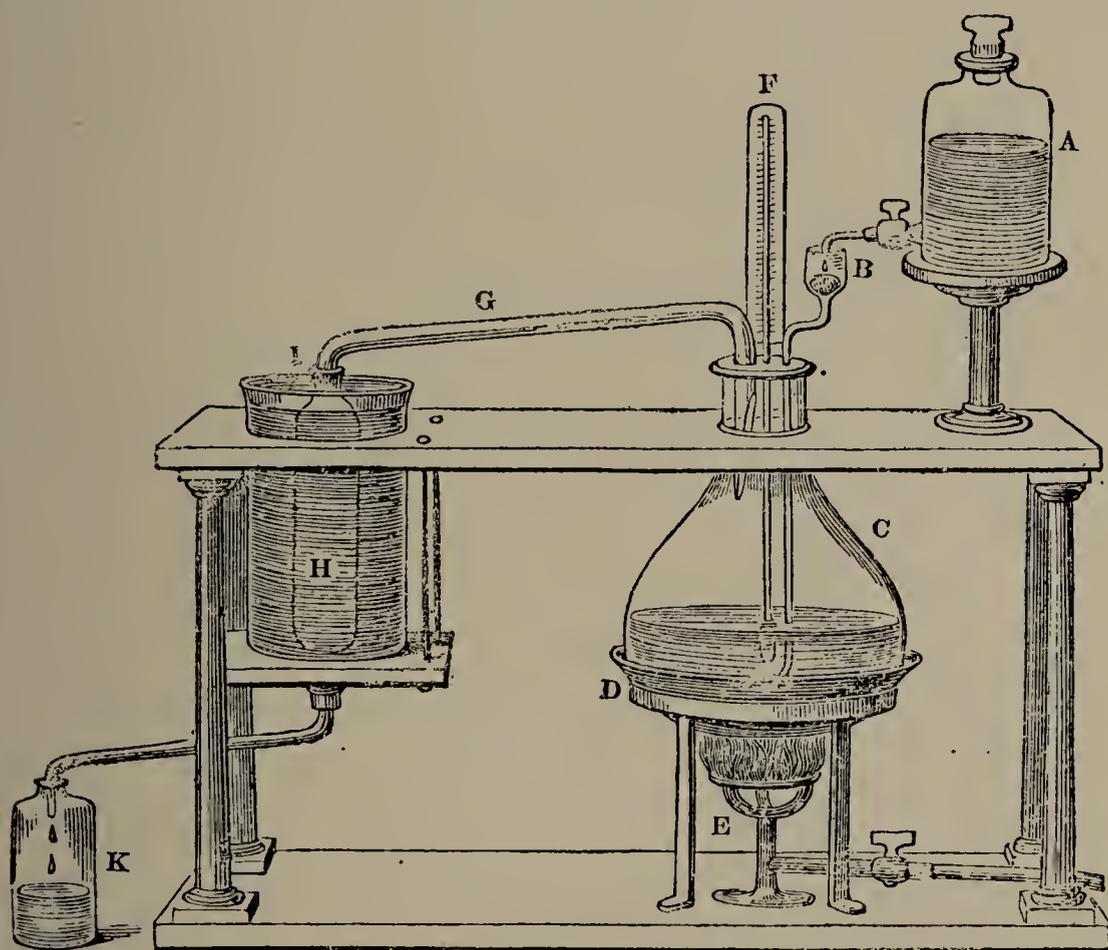
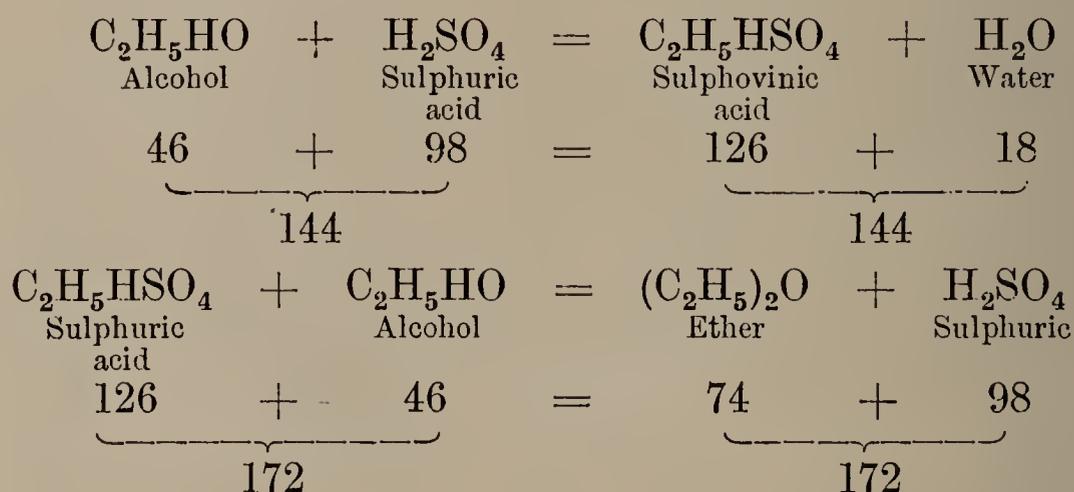


FIG. 77.—Laboratory apparatus for preparation of ether. A, alcohol vessel; B, tap and funnel regulating feed; C, etherifier; D, sand bath; E, gas burner; F, thermometer; G, tube leading vapour to condenser; H, condenser; I, refrigerator; K, receiver.

caused to flow into the mixture. The flow of alcohol is regulated so that the mixed liquid is always maintained at the same height in the etherifying vessel. The temperature gradually rises to  $136^\circ$  to  $137^\circ \text{C}$ ., and remains constant as long as there is production of ether. Working in this manner, a mixture of ether and water distils over and is collected in a well-cooled receiver. At the same time a little alcohol, and, if the operation is carried too far, a little sulphurous acid also passes over. The ether thus produced generally contains an oily product, from which it may

be freed by adding caustic potash and allowing it to stand for twenty-four hours. The ether is then decanted, washed with water to remove alcohol, and repeatedly rectified over calcium chloride. To obtain it exceptionally pure, a small piece of sodium kept in contact with it for some time will remove the last traces of alcohol and water. The tube leading the vapours from the generating vessel to the condenser should be drawn out to a point in the former, and a thermometer dipping into the boiling liquid serves to indicate the temperature. The reaction takes place in two continuous stages. The sulphuric acid and alcohol first form sulphovinic acid and water. The sulphovinic acid reacting on the fresh supply of alcohol forms ether and regenerates sulphuric acid, while the latter reacting on fresh alcohol reforms sulphovinic acid, and so on.



Thus the same cycle of reactions goes on indefinitely with the same quantity of sulphuric acid. The sulphuric acid unceasingly regenerated is always the same, but in forming sulphovinic acid it reacts continuously upon fresh supplies of alcohol in such a manner that the sulphovinic acid existing at one moment is not the same as that which existed before, or will exist afterwards.

Ether is a colourless light mobile liquid with a characteristic agreeable fragrant ethereal odour. Its taste is first burning, then cooling. It is very volatile and inflammable, burning with a brilliant flame; it does not redden litmus, but becomes slightly acid by the absorption of oxygen and the formation of acetic acid from contact with the air in imperfectly stoppered bottles. When pure its specific gravity at 15.5° C. is about .720 under the

normal pressure. At  $-31^{\circ}$  C. it congeals, forming brilliant white plates. Ether is miscible in all proportions with alcohol, carbon disulphide, chloroform, wood spirit, and benzol; thirty-six parts of pure ether dissolve one part of water, increasing thereby its density from  $\cdot720$  to  $\cdot723$  at  $15\cdot5^{\circ}$  C.; nine parts of water dissolve one part of ether. From its solution in ether the water may be completely removed by carbonate of potash, provided the ether be free from alcohol and otherwise pure. When completely free from alcohol and water, ether has no action on dry tannic acid, which, if either of these be present, liquefies to a thick syrupy fluid. Ether freely dissolves essential oils, nitro-cellulose, most of the fatty and resinous substances, alkaloids, and in general all substances rich in carbon and hydrogen. Its vapour mixes rapidly with air forming with the oxygen contained therein a mixture which explodes most violently in proximity to an incandescent body. Shaken with an equal bulk of water in a small graduated cylinder, ether should not lose more than one-fifth of its volume. Blue litmus paper, when immersed in both strata in the cylinder should remain unaltered, as also when a small quantity of the ether is evaporated in a porcelain capsule until reduced to a few drops and then tested with litmus paper; a slight acid reaction would indicate acetic acid, and, in crude ether, possibly sulphurous or sulphuric acid; the acid reaction may also be caused by traces of ethyl sulphate; traces of this and other ethyl or amyl ethers or alcohols are also indicated when about half an ounce of ether is evaporated from a flat porcelain capsule by causing the fluid to flow to and fro; when the ether is evaporated, the surfaces of the capsule should be covered with a deposit of moisture without taste or smell, and without any oily appearance. Alcohol and ether mix in all proportions, and, as above stated, the ether of commerce generally contains alcohol, which affects its density and its boiling-point: the means of abstracting the alcohol have been before mentioned. *Hoffman's anodyne liquor*, and the *spirit of ether* of Pharmacy, are such alcoholic solutions. When water, alcohol, and ether are shaken together, the mixture separates into two layers, each of which contains the three liquids; in the uppermost there is excess of

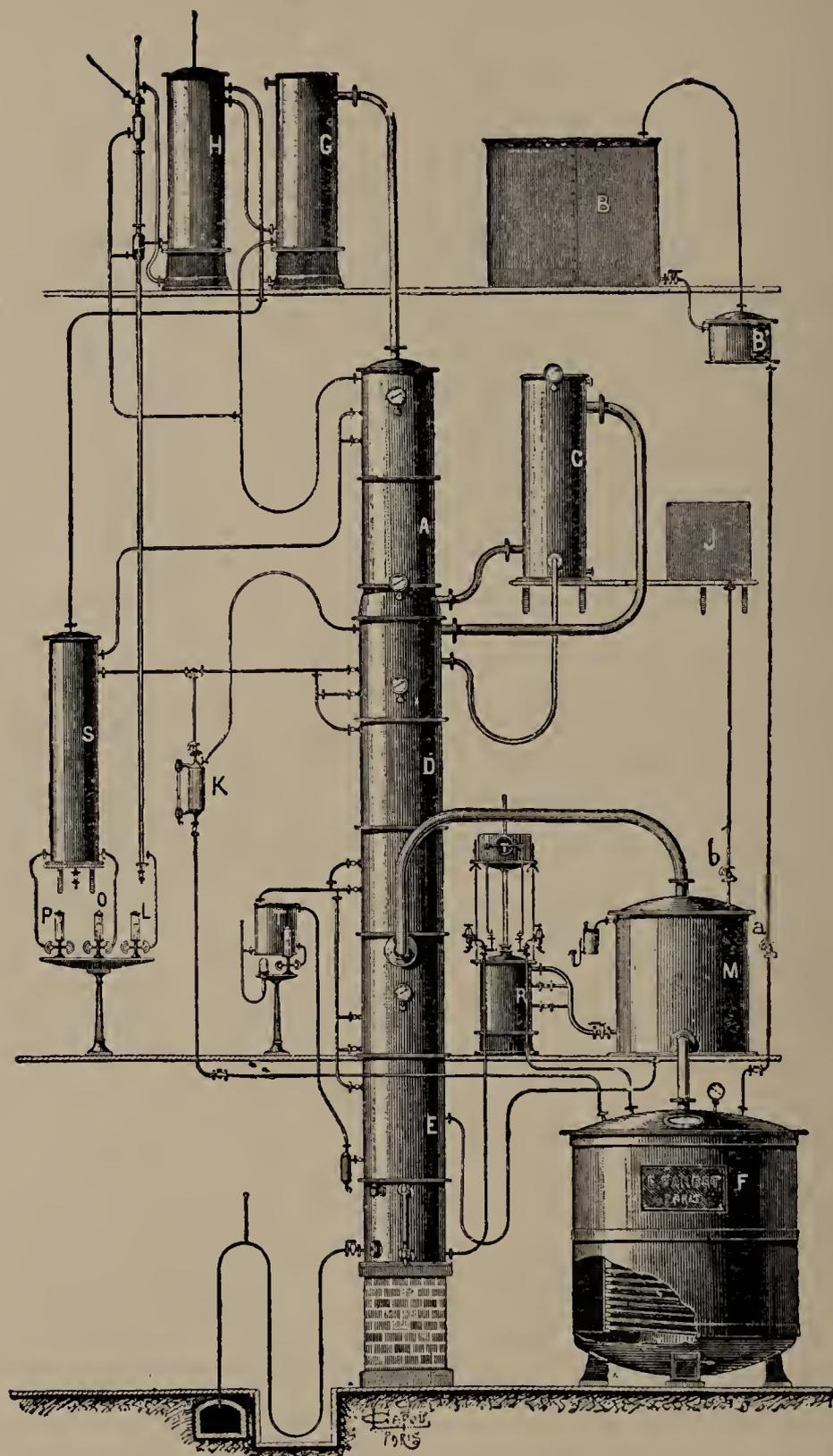


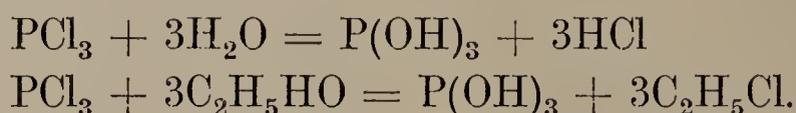
FIG. 78.—Plant for manufacture of ether (E. BARBET). B, alcohol feed tank; B<sup>1</sup>, regulating tank; *a*, regulating tap; F, coil heated etherifier (H<sub>2</sub>SO<sub>4</sub> fed in through lid manhole); J, caustic soda tank; K, alcohol extraction (returned to F); M, saturator (excess of acid); *b*, caustic soda tap; R, steam regulator; A, D, E, rectifier; C, special condenser; G, condenser; H, refrigerator; S, ether and alcohol condenser; P, ether test-glas; O, alcohol test-glass; L, first runnings test-glass; T, oils and exhaustion.<sup>1</sup>

<sup>1</sup> See also E. Barlet et Fils et Cie., *Eng. Pat.* 100,406, May 2, 1916.

ether, and in the lowermost excess of water. By the further addition of alcohol, the specific gravity of the upper layer may be brought to 0.82, and that of the lower to 0.92, after which an increase of alcohol produces a homogeneous mixture. The production of ether has become a most important industry, large quantities being required for manufacturing purposes (*e.g.*, smokeless powder, artificial silk, etc.) and for refrigerating purposes. For most, if not for all, of these purposes, ether made from ordinary methylated spirit is quite suitable. But inasmuch as it requires much more than a gallon of strong spirit to produce a gallon of ether, the price of spirit is manifestly a consideration of primary importance to this industry. *Richardson's ether* is a very dangerous solution of hydrogen peroxide in ether. It is not, therefore, a real ether. *General caution.*—Too great care cannot be exercised, whether in manufacturing, storing, or handling of ether. If ether takes fire in a vessel, it should be at once covered, and not opened until the contents are thoroughly cool. The heat of burning ether vaporises ether so rapidly that when mixed with air an explosion is bound to occur. Flash point,  $-4^{\circ}$  F., or 36 “degrees of frost.”

*Ethyl chloride* ( $C_2H_5Cl$ ); boiling-point,  $11^{\circ}$  C. ( $57.8^{\circ}$  F.).—*Sweet or dulcified spirit of salt* was a favourite preparation with the older chemists; they conceived it to possess some peculiar solvent powers on the salts of gold. It was also used in medicine: it was prepared in various ways, either by distilling a mixture of alcohol and hydrochloric acid, or of chloride of sodium, sulphuric acid, and alcohol. Its preparation by the action of alcohol upon chloride of tin was first described by the Marquis de Courtanvaux in 1768 (*Mem. de l'Acad. Royale des Sciences*, v. 19). *Ethyl chloride* may be obtained by subjecting to careful distillation a concentrated solution of hydrochloric acid gas in alcohol; or a mixture of one part of alcohol, one of sulphuric acid, and two of fused and finely powdered chloride of sodium; or a mixture of chloride of antimony, or of chloride of tin, and alcohol. Groves leads hydrochloric acid gas into the heated alcohol containing half its weight of zinc chloride in solution. The chlorides of phosphorus may also be used for the replacement of the HO of

alcohol  $C_2H_5HO$  by  $Cl$ , since they react with alcohol in an analogous manner to their action on water :



In all these cases, ethyl chloride passes over ; it should first be transmitted through warm water, by which the accompanying acid and alcohol are abstracted, and its vapour may then be condensed by conducting it through a cold tube, and receiving it in a bottle surrounded by ice and salt. The amount of ethyl chloride made in Germany is not differentiated in the same way as ethyl bromide, which has a special denaturant of its own, viz., itself.

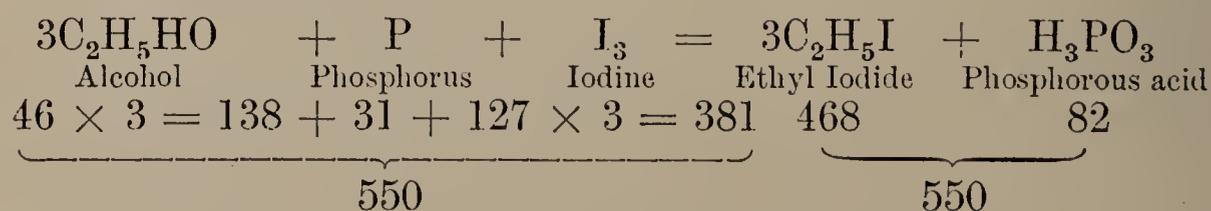
*Ethyl bromide* ( $C_2H_5Br$ ) is the bromine compound corresponding to ethyl chloride and iodide : it is made in an analogous manner to ethyl iodide.

*Ethyl iodide* ( $C_2H_5I$ ) ; molecular weight, 156. 1 litre of ethyl iodide vapour weighs 78 criths ( $78 \times 0.0896$  grammes). Sp. gr., 1.9464. Boils at  $72.2^\circ C$ .

*Preparation.*—Ethyl iodide may be prepared in an analogous manner to ethyl chloride, viz., by saturating alcohol with hydriodic acid gas and distilling, but it is more easily prepared by the action of phosphorus and iodine on alcohol. The phosphorus and the iodine mutually interacting to form hydriodic acid ( $HI$ ) and phosphorous acid  $H_3PO_3$ . The proportions are :

	Brande.	Frankland.	Wurtz.	Joly.	Theoretical.
Iodine . . .	4	2	23	1	381
Alcohol . . .	10	5	35	1	138
Phosphorus . .	2.5	1	7	0.2	31

It will be seen that authorities differ greatly as to the relative proportions. According to the equation representing the reaction, these should be as follows :

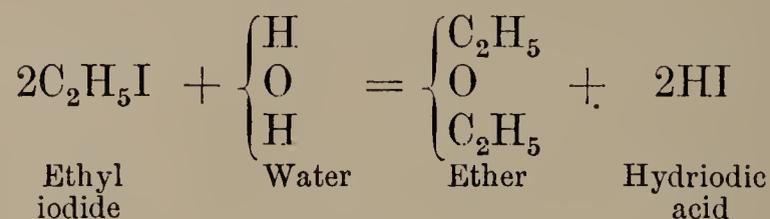


Wurtz used the apparatus described below, and the propor-

tions given under his name above. The distilling flask is surmounted by a conical glass vessel filled with the iodine mixed with fragments of glass. A bent tube is adapted to the neck of the conical vessel, which connects with an ascending Liebig's condenser. On placing the flask in the water-bath and heating, the alcohol boiled, ascended the conical vessel and dissolved the iodine. The alcoholic solution of iodine fell back into the flask, where the iodine and phosphorus decomposed the alcohol with formation of ethyl iodide and phosphorous acid. When all the iodine disappeared from the conical vessel, and the liquid in the flask was completely decolorised, the cone-shaped vessel was removed, and the liquid distilled on the water-bath as long as anything passed over. The product of the distillation being mixed with water, the ethyl iodide collects at the bottom of the aqueous liquid. If it be coloured by an excess of iodine, the latter is removed by a weak solution of caustic potash. The ethyl iodide is then dehydrated over calcium chloride and rectified. A more usual method of preparation is to mix the iodine and the alcohol in a retort or flask, and gradually add the amorphous phosphorus, digesting for some hours with a reflux condenser, and then distilling and rectifying as before. Ethyl iodide recently prepared is a neutral colourless liquid, but it becomes rose-coloured, owing to liberation of free iodine after standing some time in diffused daylight. Heated with water to  $100^{\circ}$  C., it is resolved, like methyl iodide, into alcohol and hydriodic acid (Berntsen). It is not easily inflammable, but when dropped on red-hot charcoal it diffuses a purple vapour. It is decomposed when passed through a red-hot tube, and among the products is an unctuous matter containing iodine. It is sparingly soluble in water, but readily so in alcohol and ether. Potassium does not decompose it. Alkalis, nitric acid, and chlorine slowly act upon it; sulphuric acid rapidly decomposes it. It decomposes silver oxide energetically in the cold, forming ether and silver iodide. It acts similarly by decomposition on other silver salts to form compound ethers and silver iodide, *e.g.*, it at once yields, even in the cold, a yellow precipitate of silver iodide with nitrate of silver. It is decomposed in contact with a great number of

metals,  $C_4H_{10}$  being liberated with the formation of iodides. Under suitable conditions the nascent ethyl may unite with the metals to form organo-metallic compounds.

According to Frankland, ethyl iodide, when heated with water in a sealed tube, produces ether and hydriodic acid :



*Ethyl acetate.*—Acetic ether, so extensively used, *inter alia*, as a solvent in smokeless powder-making, occurs naturally with other organic acetates in both wine and vinegar, contributing to their odour and flavour. It was discovered as far back as 1759 by the Count de Lauraguais (*Mem. Acad. Par.*). The methods of manufacture, the materials and their relative proportion vary somewhat. It is generally made by distilling acetic acid or a metallic acetate with sulphuric acid and alcohol, using one or other of the following formulæ :

	A	B	C	D	E	F	G	H
Acetic acid . . .	—	—	—	—	—	—	63	—
Potassium acetate	3	—	—	—	—	—	—	—
Sodium acetate . .	—	6	6	10	—	—	—	$12\frac{3}{8}$
Lead acetate . . .	—	—	—	—	6	32	—	—
Sulphuric acid . .	3	9	15	7	3	10	17	10
Alcohol . . . . .	2	3.6	6	8	2	9	100	10

The sulphuric acid is usually mixed with the alcohol in a vessel surrounded by ice, and when cold the mixture is poured on the acetate or the acetic acid, and the whole distilled on a sand bath. In process A the distillate is mixed with  $\frac{1}{5}$  part of sulphuric acid, and by careful redistillation acetic ether equal in volume to the alcohol may be obtained. The product of the operation contains free acid and alcohol. The distillate consists, in fact, of a mixture of acetic ether, ether, acetic acid, and alcohol ; sulphurous acid is also generally present. The distillate is therefore agitated with a solution of chloride of calcium, to which a little milk of lime

has been added ; the former eliminates alcohol, the latter neutralises the acidity when lead acetate is used. It is also purified by agitating it with water and adding carbonate of soda as long as any effervescence ensues. The ether which separates is then dehydrated by means of chloride of calcium, and distilled, and the ether, which first passes over, is collected. When the boiling-point rises to  $165^{\circ}$  F., pure acetic ether is obtained. Acetic ether is a colourless, limpid, very volatile, and highly inflammable liquid, with a very pleasant ethereal fruity odour, of density  $0.9072 \left( \frac{15^{\circ} \text{C.}}{15^{\circ} \text{C.}} \right)$  (Perkin),  $0.89$  ; (Dumas and Boullay), vapour density,  $3.03$ . It boils at  $77.5^{\circ}$  C. (Perkin). It burns with a yellowish flame, and acetic acid is developed by its combustion. Water dissolves about one-seventh of its weight of this ether, and the solution is decomposed by potash, giving rise to an acetate, and to alcohol. Ammonia has no action upon it. It is soluble in all proportions in alcohol and in ether. Acetic ether is rapidly absorbed by a mixture of quicklime and caustic potash ; on the application of heat, hydrogen is evolved, and acetate of potash remains (Dumas and Stass, *Ann. der Pharm.*, xxxv. 162). It has been analysed by Dumas and Boullay (*Poggend. Ann.*, xii. 440 and *Ann. Ch. et Ph.*, xxxvii. 15), and by Liebig (*Poggend.* xxvii. 616), with the following results :

		Calculated.	Liebig.	Dumas and Boullay.
Carbon	. . 4 = 48	54.55	54.820	54.47
Hydrogen	. . 8 = 8	9.09	8.755	9.67
Oxygen	. . 2 = 32	36.36	36.425	35.86
Acetic ether	. . . . 88	100.00	100.000	100.0

Acetic ether is intermediate in danger between absolute alcohol and ethyl ether. It is not quite so extremely dangerous as ordinary ether. Hence it replaces the latter as a solvent when it is required to minimise the risk.

*Ethyl nitrite.*—*Nitrous ether* ( $\text{C}_2\text{H}_5\text{NO}_2$ ).—The production of an ethereal fluid by the mutual action of nitric acid and alcohol is said to have been remarked by Paracelsus, and afterwards by Kunckel, but it was forgotten till rediscovered by Navier in 1742

(Navier and Geoffroy, *Mém. de l'Acad. de Paris*, 1742). It was subsequently studied by Thenard (*Mém. d'Arcueil*, i. 75 and 359), and later still by Dumas and Boullay (*Ann. Ch. and P.*, xxxvii. 19). Many processes have been published for the preparation of this ether (see Dumas, *Chim. App. aux Arts*, v. 553; and Thomson, *Inorg. Chem.*, ii. 317), among them the following, due to Thenard, is the least objectionable: Introduce into a sufficiently capacious tubulated retort equal weights of alcohol (sp. gr., 0.820) and of nitric acid of commerce (sp. gr., 1.30), and connect it with five Woulfe's bottles, the first of which is empty, and the remaining four half-filled with a saturated solution of salt in water. Apply a gentle heat to the retort, till the liquid begins to effervesce, then withdraw the fire, when the vapour passing through the bottles—which should be kept cold by a mixture of ice and salt—deposits the ether on the saline solution. In performing this experiment the retort should be more than one-sixth filled with the mixture of acid and alcohol, and cold water should be at hand to cool it if required, in order to subdue the violence of the effervescence. The alcohol should be first poured in, and then the acid, and not mixed by agitation. If the materials are warm, the acid fuming, and the alcohol of proper strength, the action often begins immediately, with a crackling noise, escape of air-bubbles, and great effervescence, so that, notwithstanding the size of the retort, its contents are very apt to pass over into the first receiver, and it is often burst; this may generally be prevented by applying a wet cloth to the retort. The tubes through which the vapour is to pass should be wide ones, for, in consequence of the suddenness and abundance of its extrication, it requires a ready means of escape; indeed, the whole process requires careful management and caution, and is most successful when conducted upon rather a small scale in a large retort, with from 2 to 4 ounces of alcohol and acid, for instance. When the effervescence has entirely ceased, the residue in the retort is found to be equal in bulk to less than one-third of the material employed: the first bottle contains an acid mixture of alcohol, water, and nitrous ether; but the bulk of the ethereal product is found upon the cold saline solution of the second bottle; a little also passes

into the third bottle. The ethereal products are collected, digested with powdered lime, and redistilled into a receiver cooled by ice : not more than ten parts of *rectified ether* are usually obtained from 100 parts of the mixture of acid and alcohol. Besides the ether, many other products are the result of this operation, such as nitrogen and its oxides, nitrous acid, carbonic acid, and traces of acetic acid and acetic ether : oxalic acid is sometimes found in the contents of the retort. Liebig prepared nitrous ether as follows : 1 part of starch is heated in a retort with 10 parts of nitric acid, sp. gr., 1.3. The retort is connected by means of a long glass tube with a tubulated bottle containing a mixture of 2 parts of alcohol, sp. gr., 0.835, with 1 part of water, kept cool by being wrapped in a wet cloth ; the other opening of this bottle is connected by means of a tube with a condenser, to which a receiver is attached. When the nitrous acid produced in the retort passes through the diluted alcohol, it decomposes it so as to form nitrous ether vapour, which is condensed and ultimately collected in the receiver ; it is purified by being shaken with water, and is then dehydrated by chloride of calcium. *Nitrous ether* has the following properties : It has a slightly yellow tint and a peculiar odour, which when much diffused is not unlike that of ripe apples ; its specific gravity is 0.880 at 40° F. (0.947 at 60° F., Liebig ; 0.909, Favre). It is extremely volatile, boiling under normal pressure at a temperature of about 70° F., so that at summer heat it is apt, on removing the stopper of a bottle containing it, to evaporate rapidly, and even to enter into spontaneous ebullition : dropped upon the hand it instantly disappears and excites great cold. The specific gravity of its vapour is 2.568 ; or experimentally, 2.627 (Dumas and Boullay). It is very inflammable, burning with a yellowish flame, and leaving no perceptible residue ; when recently prepared it has no action on litmus, but in a few days it becomes perceptibly sour, especially in the presence of moisture and light. Mixed with water, part is dissolved and another part decomposed, forming nitric acid, and giving off nitrous gas ; mixed with solution of potash it soon forms potassium nitrate, alcohol, and traces of potassium acetate. It is without action on ammonia.

Owing to its dangerous nature, it must be highly diluted with alcohol before use. The ultimate components of this ether are :

				Per cent	Dumas and Boullay.
Carbon	.	.	2 = 24	32.00	32.69
Hydrogen	.	.	5 = 5	6.67	6.85
Oxygen	.	.	2 = 32	42.67	41.46
Nitrogen	.	.	1 = 14	18.66	19.00
			—	—	—
Nitrous ether	.	.	75	100.00	100.00

*Ethyl nitrate* ( $C_2H_5NO_3$ ).—Milon succeeded in obtaining a true ethyl nitrate, and preventing the formation of nitrous acid in a mixture of nitric acid and alcohol by the addition of nitrate of urea ; 1 volume of pure nitric acid, sp. gr. 1.401, and 2 volumes of alcohol, sp. gr. .842 (being nearly equal weights), are mixed with a proper proportion of nitrate of urea, one or two parts of the latter sufficing for 120 to 150 parts of the mixture ; the operation succeeds best upon the small scale, for instance, 4 or 5 ounces of the mixture : it should be gently heated, and about seven-eighths distilled over, in which case the operation proceeds quietly, and without that violence which occurs in the absence of urea ; the nitrate of urea remains nearly intact, and may be repeatedly used. The first product is weak alcohol, soon followed by nitric ether, which is recognised by a peculiar odour, and which, on the addition of a little water, falls in the form of a dense liquid ; it is purified by washing with an alkaline solution, then left for a day or two in contact with fragments of chloride of calcium, and distilled (*Ann. Ch. et Ph.*, 3ème Sér., viii. 233). Nitric ether has a peculiar sweet odour distinct from that of nitrous ether ; its taste is sweet and slightly bitter ; its density, 1.112 at 62° F. ; its boiling-point, 185° ; it decomposes at a temperature a little above this ; it burns with a very white flame ; it is not decomposed by caustic potash, except in alcoholic solution, in which case crystals of nitrate of potash without any nitrite, are formed : it is insoluble in water, but soluble in alcohol, from which a little water immediately precipitates it ; it is decomposed by nitric acid.

*Œnanthic ether*.—The so-called œnanthic ether of Liebig and

Pelouze, obtained from the oil of the Marc brandy of France, has been shown by Faget and Fischer to consist of a mixture of the ethyl ethers of caproic and caprylic acids. The product met with on the market under the name of Artificial Essence of Cognac is generally a mixture of the ethyl ethers of different acids. The pelargonate of ethyl in particular possesses the real cognac odour. Pelargonic acid is obtained by the oxidation of oil of rue. It is etherified by passing a current of hydrochloric acid gas through its alcoholic solution. The œnanthic acid obtained by oxidation of œnanthol may also be etherified. In fact, the ethers prepared from the fatty acids of coconut oil (caprylic, caproic, and capric), are used as a basis for artificial essence of cognac.

CHLOROFORM ( $\text{CHCl}_3$ ).—This important product was discovered in 1831 by MM. Soubeiran and Liebig. By distilling chloral mixed with lime and water, or with caustic potash solution, they obtained a liquid which, when shaken with sulphuric acid and then separated and rectified over barium hydrate, yielded a dense fluid, viz., chloroform. The term chloroform refers to the constitution of formic acid ( $\text{HCOHO}$ ), so that regarding it from that point of view, chloroform is a trichloride of formyl (Brande). But it is better to regard it as marsh gas,  $\text{CH}_4$ , the old formene in which three atoms of hydrogen have been replaced by chlorine.

*Preparation.*—Ten parts of chloride of lime and 3 parts of slaked lime are stirred up with 60 parts of warm water. The milky fluid thus obtained is placed in a capacious retort, in which it should not occupy more than one-third the space at the most; 2 parts of alcohol are then added, and the whole heated. Towards  $80^\circ \text{C}$ . a very energetic action ensues, causing a very considerable frothing up. The heat is then withdrawn. Distillation commences and continues for a time without further heat. As the reaction ceases, it is again heated to carry over the final product, and when this has no longer the sweet taste of chloroform the operation is stopped. In the receiver are found two or three parts of liquid in two layers. The lower dense layer is chloroform, mixed with alcohol, and coloured yellow by an excess of chlorine, The upper portion is a rather milky mixture of alcohol, water,

and chloroform. The chloroform is decanted, washed with water, then with a solution of carbonate of potash, and rectified over calcium chloride, and again distilled.

Chloroform is a dense, colourless, volatile, very mobile liquid with an agreeable, ethereal, sweet odour. The specific gravity of pure chloroform is about 1.50 at 15.5° C. In this state of purity it is subject to decomposition by exposure to air and light, but a slight addition of ethyl alcohol protects it therefrom, and medicinal chloroform contains 2 or 3 per cent. of ethyl alcohol, which lowers its density from 1.496 to 1.480. Its taste is first sharp, then cool and sweetish. It does not act upon litmus and is not readily inflammable; but when a wick is saturated with chloroform and ignited it burns with a greenish flame, giving off pungent fumes containing hydrochloric acid. It is very volatile even at ordinary temperatures, producing by rapid evaporation great cold, but leaving neither a residue nor a film of moisture, nor any unpleasant odour when wholly evaporated by the warmth of the hand while causing the chloroform to flow to and fro in a porcelain basin. Its boiling-point is about 140° F.; vapour density, 4.2. When its vapour is respired it soon induces insensibility, in the same way, but more rapidly and effectually, than ether vapour; hence its use in the performance of painful operations, as originally suggested by Sir James Simpson, of Edinburgh (*Pharm. Journ.*, vii. 277 and 313). Poured on water, the greater part sinks in globules, which are of a milk-white appearance when the chloroform is not perfectly free from alcohol. It is so little soluble in water that 3 drops added to 9 ounces of distilled water and well shaken did not wholly disappear, though they imparted a strong odour to the liquid. It boils at 61° to 62° C. Ten parts of chloroform dissolve in 7 of rectified spirit, 1 part in 1½ of ether, and 1 in 200 of water. Its specific gravity is 1.490. It is miscible in all proportions with absolute alcohol, ether, benzol, carbon, disulphide, and essential and fatty oils, and is a powerful solvent for resins, beeswax, acting on vulcanite, and dissolving caoutchouc, gutta-percha, paraffin, camphor, mastic, elemi, tolu, benzoin, and copal; amber, sandarac, and lac are only partially soluble. Contrary to statements of Dumas, Taylor found that it did not

perceptibly dissolve sulphur and phosphorus. It dissolves iodine and bromine, forming deep red solutions. A few drops of chloroform shaken with an aqueous solution of iodine or bromine removes either of those substances, and the chloroform falls to the bottom of the vessel, acquiring a red colour, the depth of which is proportional to the quantity of either substance present. Chloroform floats on concentrated sulphuric acid, which is only darkened by it at a boiling temperature, when the chloroform is rapidly dissipated in vapour. It slowly decomposes nitric acid in the cold; but at a higher temperature deoxidation is rapid, and nitrous acid is evolved. It scarcely affects a solution of iodic acid, which acquires after a time only a faint pink colour. It has no bleaching properties; it does not decompose iodide of potassium, nor does it dissolve gold either by itself or when boiled with concentrated nitric acid. When nitrate of silver is added to it there is no precipitate, the chloroform merely acquiring that milky opacity which it has when dropped into distilled water. When the vapour of chloroform is passed over copper or iron heated to redness, it is decomposed, a metallic chloride results, and carbon is deposited, but according to Liebig no inflammable gas is evolved. It is not decomposed by potassium—some bubbles of hydrogen are sometimes evolved, but it may be distilled over the metal without change. Caustic alkalis do not decompose it, except after long boiling, when it is entirely converted into chloride and formate of potassium. Chloroform consists of :

				Calculated.	Dumas.
Carbon	.	.	1 = 12	10.04	10.24
Hydrogen	.	.	1 = 1	0.84	0.83
Chlorine	.	.	3 = 106.5	89.12	88.93
			—	—	—
Chloroform	.	.		119.5	100.0
				100.0	100.0

*Storage.*—Chloroform should be stored in the dark, in cool cellars, in glass vessels. As it does not burn without a wick, it is comparatively safe. Its vapours are non-explosive.

*Qualitative tests for impurities.*—*Chlorine.* (1) A test-tube is rinsed out with ammonia and then a few spots of chloroform dropped into the bottom of the tube. White fumes of ammonium chloride would indicate chlorine. (2) On agitation with zinc

iodide and starch solution, no blue coloration should appear. *Chlorine Compounds*.—Shake 20 c.c. with 15 c.c.  $\text{H}_2\text{SO}_4$  in stoppered tube washed with the acid prior to the test. No coloration must occur to the acid in less than an hour.

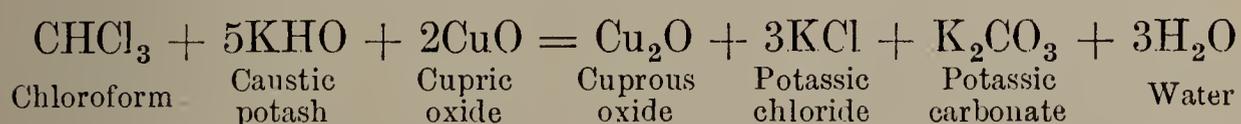
*Ethylene chloride*,  $\text{C}_2\text{H}_4\text{Cl}_2$  (oil of Dutch chemists).—Fused potassium hydrate is dissolved in absolute alcohol in a dry test-tube, the clear part decanted into another dry test-tube, and a little of the chloroform added. No reaction occurs unless the chloroform contains ethylene dichloride, when a rise in temperature will be registered by a thermometer dipping into the liquid with simultaneous evolution of gas and formation of a crystalline precipitate of potassium chloride.

*Test for alcohol*.—As medicinal chloroform always contains about 2 or 3 per cent. of alcohol, an examination for an admixture of alcohol by delicate tests would obviously be out of place. The density, the percentage decrease in volume when shaken with water, and the property of chloroform to form a perfectly clear and transparent mixture with sweet oil of almonds, which it will not do if it contains more than 5 or 6 per cent. of alcohol, afford sufficient evidence of the quality of chloroform in regard to the alcohol test. A chloroform with a density less than 1.48, at  $15.5^\circ \text{C}$ ., which yields a turbidity with oil of almonds, and causes an appreciable rise of temperature when shaken in a dry test-tube with an equal volume of concentrated sulphuric acid, cannot be regarded as officinal.

*Tests for the detection of alcohol in chloroform*.—(1) Strong sulphuric acid, to which a little potassium bichromate has been added, shaken with an equal bulk of chloroform, will turn green in presence of alcohol. (2) Two volumes of chloroform and one volume of concentrated sulphuric acid are mixed in a bottle closed by a glass stopper; after repeated agitation the bottle is allowed to stand for a few hours; the liquid is then carefully diluted with about an equal bulk of water, the supernatant aqueous layer is decanted into a beaker, and so much of a mixture of pure barium carbonate in water added, with constant stirring with a glass rod, as completely to neutralise the acid, so that, after gentle warming, the cooled liquid does not redden blue litmus-paper;

it is then passed through a moist filter, and the filtrate tested with diluted sulphuric acid. If the chloroform contained traces of alcohol, this would have given rise to the formation of ethyl-sulphuric acid (sulphovinic acid), and subsequently to soluble barium ethyl-sulphate, contained in the filtered solution, and which would be precipitated by sulphuric acid as barium sulphate. Consequently, the occurrence of a white precipitate will be evidence of the presence of alcohol. (3) A mixture of two volumes of the chloroform with five volumes of water is warmed, in a test-tube, to from 30° to 48° C.; after violent agitation for a few minutes, the liquid is passed through a moist filter, and to the filtrate is added a little solution of iodine in potassium iodide; potassium hydrate is then gradually added, until the colour of the liquid disappears. After twelve hours' standing in a conical glass, a crystalline deposit of iodoform will have formed if alcohol be present; the crystals may be recognised under the microscope, when the deposit is carefully removed by means of a small pipette from the lowest point of the conical glass, and transferred to a glass slip. (4) Koninck uses a solution of potassium permanganate in saturated barium hydrate, in the event of alcohol being present reduction occurs, the red colour turning green. Commercial chloroform is freed from alcohol and water by agitation, with double its volume of concentrated sulphuric acid, neutralised with potassium carbonate and then rectified.

*Quantitative determination of chloroform.*—Baudrimont has based a process for the quantitative determination of chloroform on its reducing action on Fehling's solution :



*German commercial brands of chloroform.*—There are on the German market, besides the German officinal chloroform, various other brands of chloroform, e.g., “Chloroform Anschutz,” chloroform *ex-chloral*, chloroform Pictet. The chloroform from chloral is very pure, but it is nearly always equalled in purity by the officinal. The chloroform Pictet, prepared by crystallisation at 70° C. and below 100° C., is likewise in a high state of

purity, but that from the purest chloral hydrate equals it in that respect, but both decompose in the absence of the small amount of alcohol required to preserve them. Anschutz salicylide chloroform is prepared from a crystalline compound of salicylide

$C_6H_4 \left\{ \begin{array}{l} CO^1 \\ O^2 \end{array} \right\} 4$ , with chloroform, the compound formed being

$C_6H_4 \left\{ \begin{array}{l} CO^1 \\ O^2 \end{array} \right\} 4, 2CHCl_3$ , the chloroform acting as water of crystal-

lisation. By simple distillation the chloroform can be recovered in a chemically pure state.

*Manufacture of chloroform from acetone.*—Even manufacturers of acetone chloroform use a certain quantity of alcohol. Chloroform needs a little spirit to help in preserving it. There is always  $\frac{1}{2}$  per cent. in that prepared in England, and up to 1 per cent. in Germany. If the chloroform is not made from spirit, naturally spirit has to be added to it. It is made in Britain from acetone, and the pure alcohol added to it. British makers have competed with Germany for some foreign orders, and have got the orders; but the duty places them at a disadvantage to the extent of that 1 per cent. of alcohol. In this connection it is very important if it is the fact, to know that chloroform is made from acetone, because then the question arises, Where are we at a disadvantage in our exportation price? It is not all made from acetone. In Scotland a great deal is made from pure alcohol, and a great deal also from methylated spirit. Chloroform is exported, and no rebate is granted.<sup>1</sup>

*Bromoform* ( $CH_3Br$ ) is the bromine compound which corresponds to chloroform. It has a density of 2.902 at 15° C. It sometimes occurs associated with chloro-bromoform ( $CHBr_2Cl$ ) and carbon tetrabromide ( $CBr_4$ ) in the residual liquor from bromine rectification. It is made by acting on acetone with bromine, with simultaneous action of potash, or by action of alkalis on bromal. The pure liquid is colourless, solidifying at 8° C. It has been prescribed for diphtheria, and as an anæsthetic.

*Iodoform* ( $CHI_3$ ).—This substance which represents methylic

<sup>1</sup> Excise Committee Report and evidence.

iodide ( $\text{CH}_3\text{I}$ ), in which two atoms of hydrogen are replaced by iodine, was discovered by Serrulas. Dumas ascertained its composition. Bouchardat examined its properties. It is produced when iodine reacts in presence of an alkali or an alkaline carbonate on numerous organic products, *e.g.*, wood spirit, alcohol, ether, dextrine, gum, albuminous matter. It may be prepared by dissolving 2 parts of crystallised carbonate of soda in 10 parts of water; 1 part of alcohol is added, and then (small portions at a time) 1 part of iodine is introduced into the liquid heated to  $60^\circ\text{-}80^\circ$ . Iodoform separates in crystals; the liquid is filtered, again brought to  $60^\circ\text{-}80^\circ\text{ C.}$ , and 2 parts of soda crystals and 1 part of alcohol are then added; then a rapid current of chlorine is injected through the liquid, with constant stirring, and a fresh quantity of iodoform is obtained. It is best obtained by boiling in a long-necked flask a mixture of 60 grains of iodine, 50 of carbonate of potash, and 60 of alcohol diluted with 3 parts of water: the boiling is continued till the colour of the iodine has disappeared; on diluting with water the iodoform falls, and only requires washing with water (Mohr, *Ann. der Pharm.*, xxix. 12). Iodoform crystallises in yellow nacreous, hexagonal tables, possessing an odour of saffron and a density of 2.05. It melts between  $115^\circ\text{ C.}$  and  $120^\circ\text{ C.}$ , and is volatilised in part without decomposition. It distils over with the vapour of water. It is insoluble in water, but dissolves in ether, volatile oils, and fatty acids. Heated with an alcoholic solution of sodium ethylate, it forms methylenic iodide,  $\text{CHI}_2$ . It reacts on acetate of silver with formation of argentic iodide and liberation of carbonic acid.

*Chloral* (Trichloroacetaldehyde,  $\text{CCl}_3\text{CHO}$ ).—The mutual action of *chlorine and alcohol* was originally inquired into by Scheele and Westrumb; it afterwards engaged the attention of the principal chemists, who expounded the theory of etherification, and later on was investigated by Liebig and by Dumas (*Ann. Ch. et Ph.*, xlix. 146, lvi. 113), and by Stadeler (*ibid.*, 61, 101). The resulting product was originally termed *heavy muriatic ether*; the term *chloral* (referring to chlorine and alcohol) was applied to it by the last-mentioned chemists. Chloral is obtained by passing a large quantity of carefully dried chlorine in a continuous current

through anhydrous alcohol ; the alcohol is at first kept cold, but when the first action is over it requires to be gradually warmed to about  $60^{\circ}$  C. during the whole operation, which lasts several hours ; hydrochloric acid gas is evolved, and must be allowed to escape. The current is continued until the action is complete, and no more chlorine is absorbed by the syrupy liquid. Liebig found that several days were required to complete this action upon a quantity of alcohol amounting to about 8 ounces. The product, chloral alcoholate, of this operation is mixed with twice its bulk of sulphuric acid, and after digestion for several hours at  $60^{\circ}$  C., is decomposed into alcohol and chloral ; the latter separates as an oil and is subjected to careful distillation ; a limpid oil-like liquid passes over, which is to be heated in an open flask till its boiling-point attains about  $93^{\circ}$  C. ( $200^{\circ}$  F.). It should then again be distilled off sulphuric acid, and finally rectified off a small quantity of fresh quicklime, the distillation being performed in a bath of salt and water. Some authorities rectify the oily liquid from  $\text{H}_2\text{SO}_4$  directly over calcium carbonate. It cannot be prepared, as might be inferred from its composition, viz., that of a chlorinated aldehyde, by treating acetaldehyde with chlorine unless water be present together with calcium carbonate to neutralise free HCl, since butyric chloral is the result when dry materials are employed. Chloral is a transparent colourless liquid with an oily appearance ; its specific gravity at  $18.3^{\circ}$  C. ( $65^{\circ}$  F.), is 1.502 ; its boiling-point =  $206^{\circ}$  F. ; and the density of its vapour about = 5.0. It has an irritating odour, is almost tasteless, somewhat caustic in its action upon the skin, soluble in water, neutral, and its solution is not affected by nitrate of silver. If, instead of dropping the chloral into water, and heating it to effect the solution, it be put into contact with a few drops of water, the liquids combine to form a white crystalline solid, heat being evolved ; and when a few drops of chloral are put into a flask containing humid air, small groups of crystals gradually form upon its interior ; these are *hydrate of chloral*. When chloral is poured upon sulphuric acid, and left for a time, it forms a white substance, which Liebig calls *insoluble chloral*. Chloral dissolves iodine, bromine, sulphur, and phosphorus. When its vapour is

passed over heated lime or baryta, those bases become incandescent, carbonic oxide is evolved, and metallic chlorides mixed with carbon remain. This sometimes happens in rectifying chloral over quicklime. The hydrated alkaline oxides decompose chloral. Nitric acid is almost without action upon it. Chloral consists of :

					Dumas.	
Carbon	.	.	2	24	16.27	16.6
Hydrogen	.	.	1	1	0.68	0.7
Oxygen	.	.	1	16	10.81	10.8
Chlorine	.	.	3	106.5	72.24	71.9
Chloral	.	.		147.5	100.00	100.00

*Hydrate of chloral* consists of 1 molecule of chloral and 1 of water. It occurs in the form of colourless, semi-transparent, crystalline plates, or crystals, of a peculiar ethereal odour and pungent taste. Exposed in a dry test-tube to a gentle heat, by dipping the tube into hot water, chloral hydrate fuses at about 58° C., and solidifies again when cooled to 15° C.; at about 95° C. it boils, and is partly resolved into chloral and water, which, however, combine again, and form a crystallising deposit in the cooler parts of the tube; at a higher temperature it is wholly volatilised without combustion. Chloral hydrate is soluble in about half its weight of cold water, and freely in both alcohol and ether, but only sparingly soluble in *cold* chloroform, in carbon bisulphide, or in oil of turpentine. Its aqueous solution is neutral, and when slightly acidulated with diluted nitric acid gives no reaction with solution of argentic nitrate, nor upon subsequent addition of ammonia; but upon heating this mixture, decomposition takes place with effervescence, and with the formation of argentic chloride and metallic silver, the latter coating the walls of the tube. When the aqueous solution is acidulated with dilute sulphuric acid, and faintly tinged with a few drops of solution of potassium permanganate, no decoloration should take place within a few hours. Concentrated sulphuric, nitric, and hydrochloric acids dissolve chloral hydrate with decomposition, but without colour, and without the evolution of coloured vapours. Solutions of the alkaline hydrates decompose it, when heated, into soluble formates and chloroform.

Ammonium sulphide dissolves chloral hydrate, with the evolution of heat, forming a turbid, reddish-brown liquid ; the same reagent produces, in concentrated as well as in diluted solutions of chloral hydrate, a yellow coloration, which becomes dark brown, forming, with the separation of sulphur, a reddish-brown compound, gradually when cold, immediately upon warming. Chloral alcoholate is a reagent for resins.

*Antipyrin.*--A febrifuge produced by the action of aceto-acetic ether on phenyl hydrazin. Antipyrin was quoted in evidence before the 1904-5 Excise Committee as an instance of very large profits made out of one of these preparations by the patentees, £60,000 a year being made by Messrs. Meister, Lucius, and Brüning, the patentees, for several years while the patent ran. The patent has now expired, and it is quite probable that English manufacturers would take up the manufacture of the product, as it would be a profitable undertaking, if they could obtain pure alcohol at a sufficiently low price. Alcohol is by far the most important substance used in the making of that compound. The following paragraphs are condensed from the evidence given before the Excise Committee. Where alcohol enters to a very large extent into these preparations, then, quite apart from the duty, a substantial difference in the price of the alcohol, as, for instance, the difference between the price of methylated spirit and unmethylated spirit, would of itself be a heavy handicap against them undoubtedly, but not quite so heavy a handicap as in the case of the colouring matters. These pharmaceutical products are produced in smaller quantities and bear higher profits. They are not cut quite so fine, and the competition is not so keen, as in the case of the colouring matters. Consequently the difference in the price of alcohol would not be of the same importance as in the case of the colouring matters, but it must always be a hindrance.

In the case of antipyrin, the wood naphtha in the methylated spirit would be perceptible in the finished product, if methylated spirit were used. It is not so much the methyl alcohol in the methylated spirit which is objectionable, but the other impurities—the acetones, etc.—which are present, and which would be very

detrimental indeed. Aceto-acetic ether is necessary in the manufacture of antipyrin. It certainly would be made in this country if manufacturers could obtain acetic ether at practically the nett price, that is without the duty. Acetic ether functions in the manufacture of aceto-acetic ether and in the manufacture of antipyrin, antipyrin being the condensation product of phenyl hydrazine and aceto-acetic ether. Neither acetic ether nor aceto-acetic ether could be made for such a purpose from methylated spirit. It is necessary that it should be in a pure state, because neither a satisfactory yield nor a satisfactory product would be obtained if the attempt were made to produce aceto-acetic ether from crude acetic ether. The methyl alcohol and the acetones in methylated spirit would prevent the formation of commercially pure aceto-acetic ether. With 10 per cent. of wood spirit, there would be about 7 per cent. of methyl alcohol and about 3 per cent. of acetones in the methylated spirit. There is a very large percentage of acetones in crude wood naphtha, but that is a matter entirely within the control of the methylator. There is no obligation to use such an amount of acetones or to use a crude wood naphtha which contains a considerable amount of acetones and ketones. The regulations define very accurately the amount of methyl alcohol and of acetone and of the other substances which may be used. Therefore it is a matter which in that respect is entirely under the control of the user. But the new regulations provide for the use of pure alcohol in cases like antipyrin.

*Amyl alcohol*,  $C_5H_{12}O$ ; molecular weight, 87.81.—Scheele was acquainted with this substance in its impure state as fusel oil. Ordinary or fermentation amylic alcohol is one of the eight possible alcohols of the formula  $C_5H_{12}O$ , and is the main ingredient of the fusel oil in the last runnings from the rectification of alcohol. Dumas determined its composition in 1834. Cahours in 1837 pointed out its analogy to ordinary alcohol, an analogy which was confirmed by the researches of Dumas and Stas, but more especially by those of Balard. The fusel oil, which is the last product to come over in the rectification of alcohol (either from malt, wine, potatoes, or beetroot), consists principally of amyl alcohol.

Amyl alcohol is invariably present in fermentation alcohol, but exactly how it is formed is unknown. With regard to this problem it may be stated that views have been put forward which are somewhat conflicting.

O. Emmerling<sup>1</sup> states that the formation of amyl alcohol is due to anærobic bacteria, for instance, *Granulobacter butylicum*, *Bacillus orthobutylicus*, and the amylic ferment of Péreire and Guignard, the products formed in mashes of boiled potatoes and wheat grist being propyl, butyl, and amyl alcohol, together with hydrogen, carbonic acid and butyric acid. According to the patent of E. A. Mislin and L. Lewin,<sup>2</sup> certain bacteria obtained from the must of prunes are capable of converting sugar or maltose into amyl alcohol, and they make use of a ferment thus obtained for fermenting a mash of potato or grains, to which 1 to 2 per cent. of alkali is added, the fermentation occupying 120 to 150 hours, and the amyl alcohol being subsequently distilled off at a temperature of 136° to 140° C.

B. Rayman and K. Kries,<sup>3</sup> on the other hand, came to the conclusion that amyl alcohol is not the product of bacterial action, but is due to the action of the yeast on certain constituents of the mash. In the fermentation of glucose no amyl alcohol is formed, but in mashes of raw barley and brewers' grains it is always present, and they ascribe its formation to be due to the fermentation of the carbohydrates known as "furfuroids" (furfurol-yielding products), which are always present in these materials.

Windisch<sup>4</sup> is also of this opinion, that the amyl alcohol is formed by culture yeast acting upon certain fermentable sugars, the products of the hydrolysis of polysaccharides contained in the raw materials. He found it to be always present in fermented barley malt, but not when pure sugar was employed.

F. Ehrlich,<sup>5</sup> as the result of his experiments, came to the same conclusion as A. Müller in 1870, that fusel oil is produced by the action of yeast on the albuminous constituents of the wort, and

<sup>1</sup> *Ber.*, 1904, 37, 3535-3538.

<sup>2</sup> *French Pat.*, 354,807, May 16, 1905.

<sup>3</sup> *Woch. f. Bran.*, 1904, 21, 25-27.

<sup>4</sup> *Z. Spiritusind.*, 1904, 27, 311.

<sup>5</sup> *Z. Ver. deutsch. Zucker-Ind.*, 1905, 539-567. *Jour. Soc. Chem. Ind.*, 1905, 683.

that sugar is not the primal material from which it is formed. In his view, the two amyl alcohols contained in fusel oil are produced from leucine and isoleucine, the other higher alcohol and a proportion of the fatty acids being derived also from other bodies belonging to the amino-acid group. He found that when using a pure culture of yeast and pure sugar solution, no fusel oil was found, but under similar conditions a solution containing leucine yielded inactive amyl alcohol, while a solution containing iso-leucine yielded  $\alpha$ -amyl alcohol, the amount of alcohol formed being practically proportional to the amino-acid destroyed.

Commercial fusel oils of different degrees of purity are to be found on the market. Ordinary commercial contains only about 30 per cent. of pure amyl alcohol. To obtain it in a pure state, the fusel oil is shaken with hot milk of lime, decanted, dried over calcium chloride, and rectified, that which passes over between  $128^{\circ}$  and  $132^{\circ}$  C. being collected apart. The portion distilling over below  $128^{\circ}$  C. contains butyl alcohol. Five c.c. mixed with concentrated sulphuric acid should give no more than a faint yellow or reddish colour. Commercial amyl alcohol is coloured black to blackish brown by sulphuric acid. Amyl alcohol, colourless when mixed with sulphuric acid, can be obtained only by repeated treatment with concentrated sulphuric acid, and absolutely pure amyl alcohol can only be produced by decomposing pure amyl sulphate. Amyl alcohol is a clear, colourless liquid without action on litmus-paper, which turns brownish by age; it possesses a disagreeable odour, and the vapour is most irritating to the throat and lungs, causing persistent coughing, and preventing its use to the extent that its superior solvent capacities for resins, etc., warrant. Rabatte uses it as a potash solvent in oil analysis, and technically to neutralise free acid in rosin oil. It has a burning taste, and burns with a white smoky flame. It boils at  $132^{\circ}$  C. Its density at  $15^{\circ}$  C. is 0.8184. It dissolves in alcohol and ether, carbon disulphide, essential and fatty oils, but is sparingly soluble in water—1 in 39 at  $16.5^{\circ}$  C., or 1 in 50 at  $13^{\circ}$ - $14^{\circ}$  C., but the solution becomes milky at  $50^{\circ}$  C. (Balbiano). It dissolves in all proportions in acetic acid diluted with its own bulk of water. It deviates the plane of polarisation to the left,

but to a different extent, according to its source. Ordinary amyl alcohol is a mixture in varying proportions of two isomerides, one of which is inactive and the other levo-rotary. By converting

TABLE XXV A.—COMPOSITION OF FUSEL OIL (WINDISCH) PER CENT. BY WEIGHT.

	Potato Fusel Oil.	Potato Fusel Oil free from Water and Alcohol.	Rye Fusel Oil.	Rye Fusel Oil free from Water and Alcohol.
Water . . . . .	11.61	—	10.150	—
Ethyl alcohol . . . . .	2.76	—	4.020	—
Normal propyl alcohol . . . . .	5.87	6.854	3.170	3.690
Isobutyl alcohol . . . . .	20.85	24.350	13.530	15.760
Amyl alcohol . . . . .	58.88	68.760	68.530	79.850
Free fatty acids . . . . .	0.009	0.011	0.137	0.160
Esters of fatty acids . . . . .	0.017	0.020	0.262	0.305
Furfurol, etc. . . . .	0.004	0.005	0.018	0.021
Hexyl alcohol . . . . .	—	—	0.114	0.133
Terpene . . . . .	—	—	0.028	0.033
Terpene hydrate . . . . .	—	—	0.041	0.048
	100.000	100.000	100.000	100.000

the mixture into amyl sulphuric acids and neutralisation with barium carbonate, the two isomeric alcohols can be separated, since the barium salt of the active alcohol is two and a half times more soluble in water than that of the inactive alcohol. Amyl alcohol does not take fire by mere contact with a flame, and when dropped on paper does not leave a permanent greasy stain. Ten grammes evaporated on the water-bath should leave no residue.

*Fusel oil.*—Some interesting particulars with regard to fusel oil are given in the "Oil, Paint, and Drug Reporter," U.S.A., April 20th, 1912.

In an enquiry as to the feasibility of the manufacture of fusel oil in Cuba, it appears from a report from the U.S. Consul at Havana that, although about 1,000,000 gallons of alcohol of all kinds was distilled from molasses annually in Cuba, no attempt had been made to recover the fusel oil. At that time the value of the molasses was about 3½ cents per gallon, and no difficulty

was found in disposing of it. In 1910 the exports of molasses from Cuba were valued at 1,477,756 dollars, the United States taking 60 per cent. of this and Great Britain practically the whole of the remainder. The principal uses of fusel oil are in the manufacture of explosives and artificial fruit essences. The United States Bureau of Manufactures give the following particulars with regard to its production: In the United States there were in 1911 seventeen molasses distilleries working; the molasses fermented and distilled amounting to 41,014,190 gallons, yielding 21,634,257 gallons of spirit. The maximum yield of fusel oil from proof spirit amounts to 0.2 per cent. In the United States in 1910 the production of fusel oil totalled 110,792 gallons, a figure which had only once previously been exceeded, in 1907, when it reached 124,709 gallons. Imports of fusel oil into the United States amounted in the fiscal year 1910 to 4,953,952 lb., valued at \$598,199, and in 1911 to 5,231,252 lb., valued at \$842,916. More than half of this came from Russia and Germany.

G. Heinzelmann<sup>1</sup> has examined several samples of fusel oil from various sources, and found them to contain from 52 to 77.8 per cent. of amyl alcohol; their specific gravities varied between 0.8385 and 0.8495. Fusel oil from sulphite-cellulose alcohol was characterised by an odour of turpentine, and by the high proportion of residue left on distillation at 132° C. Thus in the distillation of 1500 c.c. of such alcohol, the residue amounted to 148 c.c., whereas the fusel oils from yeast factories and the distillation of potato spirit yielded only 41 to 55 c.c.

*Amyl acetate.*—There are several possible isomeric acetates of amyl. The commercial amyl acetate known to chemists as iso-amyl acetate,  $(\text{CH}_3)_2\text{CH} \cdot \text{CH}_2\text{CH}_2\text{O} \cdot \text{C}_2\text{H}_3\text{O}$ , is a colourless mobile liquid with a pleasant aromatic, but somewhat suffocating, odour, recalling that of Jargonelle pears. It *may* be prepared by heating a mixture of iso-amyl alcohol with concentrated sulphuric and acetic acid,  $\text{CH}_3 \cdot \text{CO}_2\text{H} + \text{SO}_4\text{H}_2 + \text{C}_5\text{H}_{11}\text{OH} = \text{CH}_3 \cdot \text{CO}_2\text{C}_5\text{H}_{11} + \text{H}_2\text{SO}_4 + \text{H}_2\text{O}$ , but it is better to replace acetic acid by an alkaline acetate. In that case it may be prepared by distilling a mixture of 1 part of amyl alcohol, 1 part of strong sulphuric

<sup>1</sup> *Z. Spiritusind.*, 1913, 36, 14.

acid, and 2 parts of dried potassium acetate. The distillate is agitated with water, the upper layer of amyl acetate is run off, shaken up with a strong solution of sodium carbonate, again run off, dried over calcium chloride, and redistilled. Dried potassium acetate may be replaced by fused sodium acetate. The fused sodium acetate is prepared by heating the crystallised salt in an enamelled basin of wrought-iron, aqueous fusion occurs at about 100° C., then the salt solidifies, to again melt at about 320° C. As soon as the salt is completely transformed into a clear liquid, the basin is covered and allowed to cool. When its temperature has cooled to 50°-60° C., the product is pulverised. It is preserved in a stoppered bottle, the mouth of which is suitably lubricated. *Preparation.*—Into a 300 c.c. flask surmounted by a vertical condenser there are introduced :

Acetate of sodium, dry crushed . . . . . 30 grammes  
to which are added a mixture of sulphuric acid and amyl alcohol,  
prepared beforehand, in the proportion of :

Amyl alcohol . . . . . 30 grammes  
Sulphuric acid (concentrated) . . . . . 60 grammes

and the whole is heated on the sand-bath for about one hour. The product is then run into an excess of cold water, and the acetate of amyl formed is separated by decantation, washed with dilute soda and water, dried over calcium chloride, and distilled. The yield is 80-90 per cent. The amyl acetate of commerce contains other isomeric acetates of amyl. It is manufactured in a similar manner to the laboratory processes already described, but the pure amyl alcohol is replaced by the ordinary rectified fusel oil, consisting chiefly of a mixture of active and inactive primary iso-amyl alcohol ; also acetate of lime is often used instead of the acetate of potassium or sodium. As the ether always retains a little amyl alcohol, if it is desired to free it from this impurity it is treated with acetic acid diluted with its own weight of water, which dissolves the alcohol but does not act on the ether. The persistent choking smell of some samples of commercial amyl acetate may be due to unchanged amyl alcohol, and it might be advisable to attempt to purify them in this way. Acetate of

amyl is less dense than water, in which it is insoluble ; it boils at  $133^{\circ}$  C. ; sp. gr., 0.857. It dissolves in all proportions of ether, and in both amyl and ethyl alcohols. Its solution in ethyl alcohol is extensively used under the trade name of Jargonelle pear essence for flavouring confectionery. Amyl acetate freely dissolves resins, camphor, and a solution of pyroxylin in amyl acetate is used as a lacquer, although the persistent smell of the crude acetate stands somewhat in the way of its more extended use. The flame of acetate of amyl has been suggested as a photometric standard.

*Methyl alcohol* ( $\text{CH}_3\text{HO} = 32$  ; B.P. =  $66.6^{\circ}$  C.) may be obtained in the pure state from wintergreen oil, which consists of acid methyl silicylate  $\text{C}_6\text{H}_4(\text{OH})\text{CO} \cdot \text{OCH}_3$  by distillation over potash, whereby potassium salicylate is formed and methyl alcohol distils over  $\text{C}_6\text{H}_4(\text{OH})\text{CO} \cdot \text{OCH}_3 + \text{KHO} = \text{C}_6\text{H}_4(\text{OH})\text{CO} \cdot \text{OK} + \text{CH}_3\text{HO}$ . The product is very pure. *Wood spirit*.—Crude pyroligneous acid, in which P. Taylor, in 1819, first discovered wood spirit (methyl alcohol, wood naphtha), contains 1 per cent. of wood spirit. The crude pyroligneous acid is redistilled in copper stills heated externally by hot gases, or internally by a steam coil, with steam at 25 lb. pressure. Cast-iron stills may be used instead of copper ones, but less advantageously. The tar, deposited at this stage, is run off whilst still hot. The first runnings, or 20 per cent. of the whole distillate, consists of crude dilute wood spirit (methyl alcohol), a volatile liquid extensively used in methylating, or, as they call it on the continent, “denaturing,” ordinary ethyl alcohol for industrial purposes. As the methyl alcohol distils, a certain amount of tar, which is held in solution, is left behind in the still. The crude, weak naphtha is neutralised and repeatedly rectified with quicklime, sometimes with the acetic acid distillate added. The addition of lime also holds back tarry matters and converts methyl acetate into methyl alcohol. The clear liquid separated from the oil which floats on the surface, and from the sediment, is treated with a small quantity of sulphuric acid, which absorbs ammonia and precipitates tarry matters, and is redistilled over quicklime. The vapours given off during both the distillation

of the wood and its redistillation are inflammable and explosive, and great care must be exercised in the process. The final rectification is done in Coffey's stills (Figs. 43-45), or by Barbet's continuous process. Barbet has adapted the plant shown in Fig. 78 for the rectifying of wood spirit. The tar does not adhere to the comb-slit caps of his rectifying columns.

The ordinary wood spirit of commerce is rarely pure. When mixed with water, it becomes milky, and an oily layer forms on the top, consisting of different bodies insoluble in water. The insoluble portion being separated, and the clear aqueous liquid distilled, methyl alcohol passes over first, and is rectified over quicklime. These processes are long and tedious, and only give imperfect results. When wood spirit is mixed with a fourth of its volume of olive oil, the latter combines with the impurities, and on distillation of the purified product a comparatively pure methyl alcohol is obtained. A surer process is to distil the wood spirit with oxalic or citric acid so as to obtain a crystallisable ether (methyl oxalic ether). The latter is decomposed by distilling with water (after a previous purification by expression between folds of filter paper). The wood spirit thus obtained is rectified over quicklime. Wood naphtha can be made miscible with water by diluting until complete precipitation is effected, and then shaking with molten paraffin, cooling with continued agitation, filtering and redistilling. The paraffin may be revived by steaming, and used repeatedly. Pure methyl alcohol is a colourless, mobile liquid, with a spirituous odour. The empyreumatic odour of the wood spirit of commerce is due to impurities. Its density at 0° C. is 0.8142. It boils at 66.5° C. under the ordinary atmospheric pressure. However, its boiling-point varies within rather wide limits (60°-65°), according to the nature of the sides of the distilling vessel. Methyl alcohol burns with a non-luminous flame, and hence may be burnt in a spirit lamp. It mixes in all proportions with water, alcohol, and ether, and dissolves certain resins and fatty and volatile oils; its deportment in this respect being similar to ordinary ethyl alcohol, although its solvent action on gum resins is often different. Thus some gums are insoluble in ordinary alcohol, and also in wood spirit,

but in a mixture certain proportions of these two may dissolve the gum, yet it often happens that an excess of either of the alcohols precipitates the resin from solution.

The following have been detected as amongst the natural constituents of crude wood spirit : (1) Acetone, at least 3-4 per cent. methyl ethyl ketone ; (2) higher ketones, methyl propyl ketone, allyl methyl ketone, allyl ethyl ketone ; (3) aldehydes ; (4) dimethyl acetal, methyl ethyl acetal, di-ethyl acetal, and methyl propyl acetal ; (5) allyl alcohol ; (6) propyl aldehyde ; (7) dimethyl furfuran ; (8) methyl formate and (9) acetate ; (10) crotonic and (11) angelic acids ; (12) pyroxanthine ; (13) traces of ammonia and (14) methylamine. There are 360,000 gallons of wood naphtha used in this country annually, of which only a small proportion is produced from home-grown raw material, the greater proportion being imported from Canada, the United States and other countries where wood is abundant. All hard woods, particularly oak and beech, are suitable for distilling ; elm is not a good wood for the purpose, and pinewood is less suitable. This alcohol, especially when crude, is more volatile, and its vapours more readily inflammable than those of ethyl alcohol ; and, owing to the high acetone content of crude spirit, its vapour is explosive at the ordinary temperature. Flash point, 32° F.

*Methyl chloride.*—This compound is best obtained by heating a mixture of 2 parts of chloride of sodium, 1 of methyl alcohol, and 3 of sulphuric acid ; a gas is evolved, which is washed with water, and which is pure methyl chloride. Any impurities are abstracted by the water. The gas is collected in tubes filled with mercury. This gas is not condensable at 0° ; exposed to intense cold it condenses to a liquid which boils at — 22° C. It is colourless, has an ethereal odour, and sweet taste, and burns with a greenish-white flame. Water at 60° F. dissolves 2.5 volumes. It forms with water a hydrate crystallisable at +6° C. It is very soluble in alcohol. It is quite neutral, and gives no precipitate with solution of nitrate of silver, resembling in these respects ethyl chloride. Its density is 0.9523 at 0° C. Its chief source is beet distillery residues. When chloride of methyl is passed

through a red-hot tube, it is resolved into hydrochloric acid, marsh gas, ethylene, and other hydrocarbons. It burns with a yellow flame, and is condensed by chlorine under exposure to light. Heated with caustic potash, chloride of methyl evolves hydrogen, and formate of potash is formed, which by further decomposition yields carbonate of potash. In ordinary daylight chlorine produces no change on chloride of methyl, but in the direct rays of the sun the following compounds are successively formed, namely,  $\text{CHCl}_2$ ;  $\text{CHCl}_3$ ; and  $\text{CCl}_4$ .

The components of chloride of methyl are :

Carbon	.	.	1	12	23.76	} = Methyl	1	15	29.70
Hydrogen	.	.	3	3	5.94				
Chlorine	.	.	1	35.5	70.30		Chlorine	1	35.5
			—	—	—		—	—	—
Chloride of methyl				50.5	100.00			50.5	100.00

*Methyl bromide* ( $\text{CH}_3\text{Br}$ ).—A colourless liquid prepared by acting on methyl alcohol with bromine in presence of phosphorus. It contains 53.7 per cent. of combined methyl calculated to methyl alcohol.

*Methyl iodide* ( $\text{CH}_3\text{I}$ ) is prepared in a similar way to methyl bromide. The necessary quantity of iodine is dissolved in the alcohol, and then run in a stream into a still provided with an agitator, and containing vitreous phosphorus. The methyl alcohol and iodine pass in a continuous stream into the still, and methyl iodide ( $D = 2.199$ ; B.P. =  $43.8^\circ \text{C}$ .) issues in a continuous stream from the condenser.

The ethereal salts of methyl alcohol, methyl acetate, methyl nitrate, and methyl sulphate are made by processes so analogous to the corresponding ethyl salts, that we need not occupy further space with a description of their manufacture. Suffice it to say that methyl acetate has been suggested to replace *amyl acetate* as a solvent for nitro-cellulose in the facing of cotton to resemble silk. The smell of the former, although pleasant in small quantity, is too persistent.

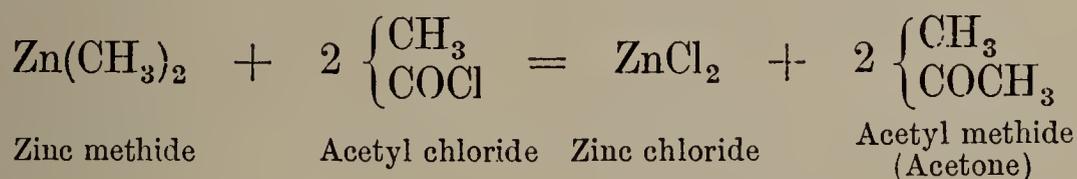
ACETONE (*Pyroacetic spirit*—*Pyroacetic ether*—*Dimethylketone*—*Propanone*) was known in the Middle Ages. It was obtained so far back, at least, as the sixteenth century by the distillation

of *Salt of Saturn* (lead acetate). It was obtained by Derosné (*Ann. de Chim.*, lxxiii. 267) by the destructive distillation of cupric acetate. Chenevix obtained it from other acetates. Subsequently it was examined by Macaire and Marcet (*Ann. of Phil.* N.S., viii. 69), and by Liebig and Dumas, who established its composition in 1832. Gerhardt regarded it as acetyl methide. It has, in fact, been prepared by replacing the chlorine of acetyl chloride by methyl. The relationship between acetic acid, acetyl chloride, and acetone is that acetyl chloride :

$\left\{ \begin{array}{l} \text{CH}_3 \\ \text{COCl} \end{array} \right.$  is acetic acid  $\left\{ \begin{array}{l} \text{CH}_3 \\ \text{COHO} \end{array} \right.$ , in which the hydroxyl is replaced by

chlorine. In acetone  $\left\{ \begin{array}{l} \text{CH}_3 \\ \text{COCH}_3 \end{array} \right.$  it is replaced by methyl. The

experiment was carried out by Pebal and Freund, who in 1861 effected the synthesis of acetone by treating zinc-methide with acetyl chloride :



Acetone, in fact, belongs to a class of bodies termed generically ketones. Frankland regarded acetone and other ketones as derived from the fatty acids by the substitution of the hydroxyl of the latter by a monad alkyl radical ; they thus resemble the aldehydes in constitution :

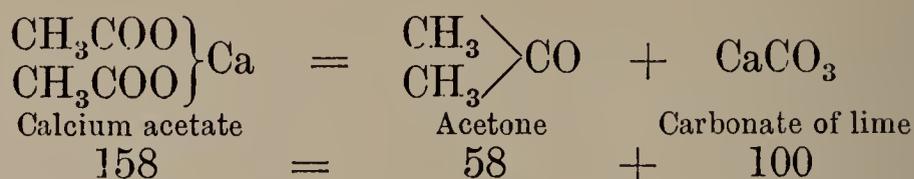


The ketones are also correctly represented as compounds of carbonic oxide with monad alkyl radicles ; that is, they contain the carbonyl group CO linked on both sides with an alcohol radical :

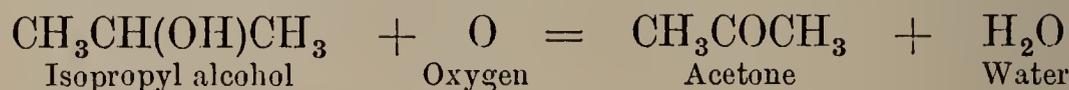


This composition is illustrated by the reaction which takes

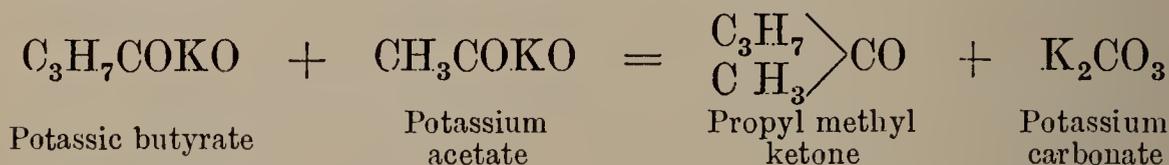
place when acetone is produced by the dry distillation of acetate of lime :



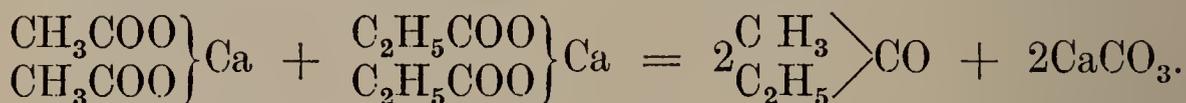
Acetone may also be produced by the oxidation of isopropyl alcohol, which thus loses 2 atoms of hydrogen :



By distilling together the salts of two different fatty acids, ketones containing two different alkyl radicles are obtained :



In the same way, if a mixture of the acetate and propionate of calcium be distilled, methyl ethyl ketone is produced :



*Laboratory methods of preparing acetone on a small scale.*—When dry acetate of lime is carefully distilled in a *fireclay retort*, it yields a considerable proportion of this product. The vapours disengaged are condensed in a well-cooled receiver, and the crude distillate is rectified with a small quantity of bichromate of potash and sulphuric acid. It may be freed from water and any remaining traces of empyreumatic oil by repeated rectification over chloride of calcium. Acetone is also formed during the dry distillation of anhydrous acetate of lead (Wohler). Zeise gives the following as the most suitable process for the preparation of acetone. One part of dry quicklime and 2 parts of crystalline acetate of lead are pulverised and mixed, and introduced into a retort, or iron bottle (4 of lead acetate to 1 of lime—Wurtz). After a time the lime becomes hydrated at the expense of the water of crystallisation of the acetate, and much heat is evolved. The retort is then adapted to a receiver immersed in a freezing mixture, or in ice, and heat is gradually applied till it becomes

red-hot. The crude product is a mixture of acetone, water, and two oily products ; it is redistilled in a bath of chloride of calcium, and the distillate is then again poured upon chloride of calcium, and after some days is poured off, and three-fourths of it distilled over. Acetone may also be prepared from sugar, starch, gum, etc., by distillation with eight times their weight of powdered quicklime. It is, however, accompanied in this case by phorone—an oily liquid separable by water, in which it is insoluble. It is also produced by heating citric acid with potassium permanganate (St. Gilles, *Jahr uber de Fort chemie*, 1858, 585). Acetone is also one of the products formed in the destructive distillation of wood (Volckel, A, 80, 310) and of citric acid (Robiquet). It would be best prepared by destructive distillation of barium acetate at a moderate heat, but barium acetate is costly. Hence acetate of lime is generally used for the preparation of acetone on the large scale, but the temperature required is greater, and the distillate is contaminated with impurities such as *dumasin*, an isomeride of mesitylene oxide. Acetone can also be manufactured from crude wood spirit, of which it is an important constituent, by the continuous rectification plant, now in use both in France and Canada, shown in Fig. 79. The raw product contains about 25 per cent. acetone, and the plant yields acetone of 95-98 per cent. purity. The arrows show the circulation of the liquid or vapour. As acetone boils at  $55.5^{\circ}$  C. and methyl alcohol at  $66.5^{\circ}$  C., there is a difference of  $10^{\circ}$  C. between their boiling-points, which suffices for fractionation. The product thus obtained can finally be purified by treatment with sodium or potassium bisulphite (thereby taking advantage of the fact, first discovered

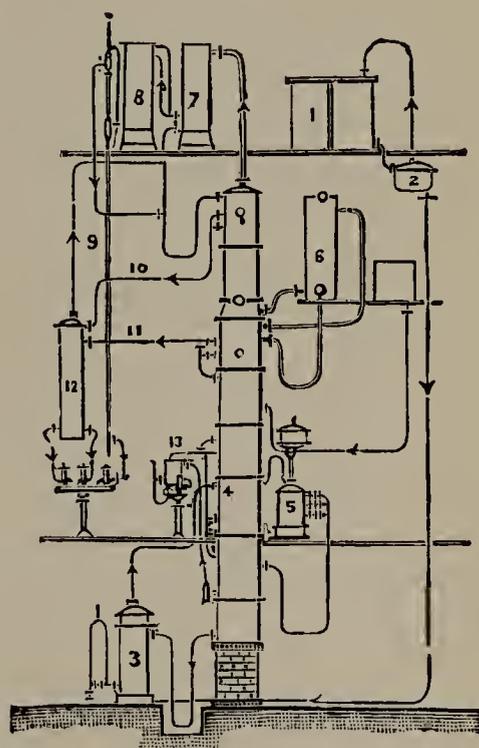


FIG. 79.—Acetone still (E. BARBET, Paris). 1, Feed; 2, feed regulator; 3, forewarmer; 4, entrance of crude raw material; 5, steam regulator; 6, special condenser; 7, condenser; 8, refrigerator; 9, first runnings; 10, pasteurised acetone; 11, pasteurised methyl alcohol; 12, refrigerator; 13, oils and exhausts.

by Limpricht, that acetone combines like aldehyde with alkaline bisulphite to form crystalline compounds), recrystallising the crystalline compound, and then treating the crystals with and distilling over carbonate of soda lye. Acetone is used for cleaning galvanised iron, in spirit varnish manufacture, as a solvent for resin, fats, and oils. In the purification of crude anthracene, in the manufacture of chloroform, and in the process of producing artificial silk, and, above all, in smokeless powder manufacture. Acetone requires similar precautions in use to benzol.

*Ketone oils.*—The salts which the higher acids of the acetic series produce with alkalies yield on distillation the higher homologues of acetone : the so-called ketone oils used as solvents.

## CHAPTER XIII.

### STATISTICS AND THE USES OF ALCOHOL IN MANUFACTURES, ETC.

*Denaturants.*---The high duties which are imposed on potable spirits make it imperative to supply for manufacturing and general purposes a cheaper kind, which, in order to satisfy the requirements of the Inland Revenue authorities, has been rendered unfit for drinking purposes as nearly as possible without affecting its properties for the uses to which it is to be applied. The addition of such materials is termed "methylating" or "denaturing," the materials added being known as "denaturants."

The choice of a suitable denaturant is not a simple problem, no single substance known fulfilling all the requirements.

Sir J. J. Dobbie, in a report submitted to the Alcohol Motor Fuel Committee, published by permission of H.M. Petroleum Executive,<sup>1</sup> defines the characters desirable in an ideal denaturant, these being :—

1. It should impart a taste or smell sufficiently obnoxious to render the alcohol unfit for drinking purposes even after dilution, sweetening, or flavouring.
2. It should not be easily eliminated by filtration, distillation, or other process which could be readily applied or is in common use in manufacturing operations.
3. It should be of such a nature as to be easily and with certainty detected, even when present in minute quantity.
4. It should mix readily with alcohol and form a mixture possessing essentially the same properties as pure alcohol, and the mixture should be quite as suitable for use in manufacturing operations as the undenatured spirit.
5. Its cost should not add materially to the price of the spirit.

<sup>1</sup> *Jour. Soc. Chem. Ind.*, 1920, 86 R.

The substance which most nearly fulfils these requirements is crude methyl alcohol or wood spirit, which is used in this country and most parts of the British Empire, in the United States, and also in most European countries.

Previous to 1902, all alcohol manufactured in this country was dutiable, except what was known as methylated spirit, which contained 10 per cent. of wood spirit and was used in the preparation of varnishes and polishes, and for burning purposes. In many manufacturing operations this spirit could not be employed, owing to its pronounced odour, which adhered to the products manufactured from it. Also, in certain cases—for instance, in the manufacture of certain aniline dyes and the preparation of pure ethyl derivatives—crude methyl alcohol could not on any account be employed; hence in these cases duty-paid alcohol, which was very expensive, had of necessity to be used. Another objection to the use of the wood spirit is that, since it is about twice the price of pure alcohol, its addition raises the price of the denatured spirit proportionally.

Naturally, this was regarded as a powerful deterrent to chemical industry, particularly the manufacture of fine chemicals and dyes in this country, and several manufacturers, notably the late Mr. Thomas Tyrer, urged the matter so forcibly that the Government passed an Act in 1902 which allowed more or less latitude in the choice of denaturants to suit particular industries.

Section 128 of the Excise Act of 1880 authorised the use of methylated spirit consisting of 90 parts of alcohol and 10 parts of wood spirit, free of duty.

At the present time two kinds of methylated spirit are manufactured :

(a) *Ordinary methylated spirit for use in manufacturing operations or industrial methylated spirit.*—Consisting of 95 parts of ordinary ethyl alcohol, 60 to 66 O.P. (91 to 95 per cent. of real alcohol) and 5 parts of wood naphtha of an approved type.

(b) *Mineralised methylated spirit.*—For burning, cleaning, and general domestic and other purposes. Consisting of 95 parts of ordinary ethyl alcohol as above, 5 parts of wood naphtha, and an

addition to each 100 gallons of the mixture of  $\frac{3}{8}$  of 1 per cent. of mineral naphtha (sp. gr., 0.800 to 0.830) of approved type.

No duty is charged on British spirits used for methylation, and foreign and colonial spirits to be used for the same purpose are exempt from the ordinary taxes.

Methylated spirit can be made only by distillers, rectifiers, or methylators who have to pay an annual licence of £10 10s. Certain regulations have to be complied with as to the amount of spirit manufactured at one time, storage, and disposal. The industrial methylated spirit can only be sold by methylators to persons licensed by the Board of Inland Revenue to receive and use it. Methylators are not allowed to sell a greater quantity than 50 gallons, nor a less quantity than 5 gallons at one time to a retailer. Smaller retailers are allowed to purchase from the latter in quantity of not more than 1 gallon at a time, and are allowed to sell not more than that quantity at one time. Retailers of this spirit pay an annual licence of 10s. Since 1918 the addition of a small quantity of methyl violet, sufficient to colour the alcohol, has been authorised as an extra precaution.

The ordinary methylated spirit mixes with water without becoming turbid, but the mineralised methylated spirit yields a permanent milky emulsion which is particularly nauseous to the taste.

One disadvantage of the denaturation of alcohol as at present practised is the increased cost, due to the methylating agents, and also for supervision.

The increase in cost due to the denaturant in industrial methylated spirit was in 1906 0.77d. per gallon, and for mineralised methylated spirit, 1.6d. per gallon.<sup>1</sup>

The table on p. 318 shows the total increase in cost of denatured alcohol for the years 1906 and 1919 for comparison.

In order to obviate the disadvantages arising from the use of methylated spirit in certain manufacturing industries, provisions were made in Section 8 of the Finance Act of 1902 for the Commissioners of Customs and Excise to authorise the use of ethyl and methyl alcohol denatured with substances other than wood

<sup>1</sup>Sir J. J. Dobbie, *Jour. Soc. Chem. Ind.*, 1920, 86 R.

TABLE XXVI.

*Mineralised Methylated Spirit.*

	Increase due to denaturant.	Per bulk gallon.		Difference.
		Selling price of 600 P. alcohol (91 per cent).	Selling price of denatured spirit.	
1906 . . .	1.60d.	1s. 4d.	1s. 7d.	0s. 3d.
1919 . . .	5.16d.	7s. 2½d.	9s. 6d.	2s. 3½d.
<i>Industrial Methylated Spirit.</i>				
1906 . . .	0.77d.	1s. 4½d.	1s. 6d.	0s. 1½d.
1919 . . .	2.66d.	7s. 0¾d.	8s. 7d.	1s. 3½d.

naphtha, to suit particular requirements. This concession has been a boon to manufacturers, but certain regulations have still to be complied with, and the cost of supervision has to be borne by the users.

The following methods of denaturing have already been authorised :<sup>1</sup>

1. *In making coal tar dyes—*

(a) Methyl alcohol, mixed with 0.05 per cent. of mineral naphtha.

(b) Ethyl alcohol, mixed with 2 per cent. of nitrotoluol or 2 per cent. of nitrobenzol.

2. *For the manufacture of xylonite and similar substances—*

Ethyl alcohol mixed with :

(a) One pound of camphor and 1 pint of coal-tar naphtha (toluol) per gallon ;

or (b) one pint of toluol per gallon ;

or (c) one pound of camphor per gallon ;

or (d) 0.05 per cent. of bone oil ;

or (e) 0.5 per cent. of petroleum.

3. *For the manufacture of fulminates—*

<sup>1</sup> Sir J. J. Dobbie, *Jour. Soc. Chem. Ind.*, 1920, 86 R.

Ethyl alcohol mixed with 10 per cent. of the spirit from a previous operation and 0·025 per cent. of bone oil.

4. *For coconut oil refining* (temporary)—

Ethyl alcohol mixed with an equal volume of ordinary methylated spirit.

5. *For making electric lamps*—

Ethyl alcohol mixed with 1 lb. of red phosphorus per gallon.

Undenatured alcohol is allowed to be used by universities, colleges, and other public institutions for research and teaching purposes, under certain prescribed conditions.

Wood naphtha and other denaturing agents must conform to certain specifications, and must be approved by the Principal of the Government Laboratory after they have been tested.

In those cases where denaturing with wood spirit is proved to the satisfaction of the Commissioners of Customs and Excise to render the spirit unsuitable or detrimental for use in any art or manufacture, the Commissioners have power to grant the use of duty-free spirit under other conditions, which will be explained if application is made to them in writing.

As a temporary measure, owing to the shortage of wood naphtha, the composition of methylated spirit manufactured on and after June 1st, 1918, was prescribed as follows :—

*Industrial Methylated Spirit.*

(a) *Ordinary*—

Wood naphtha . . . . .	2	per cent.
Mineral naphtha . . . . .	$\frac{1}{2}$	„
Alcohol . . . . .	$97\frac{1}{2}$	„

(b) *Special*—

Wood naphtha . . . . .	3	per cent.
Alcohol . . . . .	97	„

*Mineralised Methylated Spirit.*

Wood naphtha . . . . .	5	per cent.
Alcohol . . . . .	95	„

with the addition of  $\frac{1}{2}$  per cent. of mineral naphtha and 0·025 oz. of aniline dye (methyl violet) to every 100 gallons of the mixture.

In France the general denaturing process consists in adding to 100 litres of ethyl alcohol, 10 litres of wood spirit of at least 90°, containing 25 per cent. of acetone and 2.5 per cent. of impurities pyrogénées. This wood spirit or "methylene regie" has a specific gravity of 0.830 (58° O.P.), and contains about 65 per cent. of methyl alcohol.

This denatured spirit is used in the manufacture of varnishes, solid extracts, solidified spirits, plastic substances, alkaloids, fulminate of mercury, transparent soap, insecticides, etc.

For lighting and heating this spirit must be further denatured by the addition of 0.5 per cent. of heavy benzene, distilling between 150° and 200° C., and for the manufacture of finish by the addition of 4 per cent. of gum resin.

As ordinary methylated spirit cannot be used in certain industries, the Ministry has authorised the employment of other denaturing agents. These are special for each manufacture or general for products or classes of products already approved on the advice of the Consultative Committee, or have to be approved by the Minister. Quite a number of formulæ have already been approved.

In Germany undenatured spirit duty free can only be used in State or municipal hospitals, and scientific institutions, and in the manufacture of smokeless powders, fuses, and fulminates.

Spirit for manufacturing and general purposes is either completely or partially denatured, according to the circumstances of the case.

There are two methods authorised for complete denaturing :  
(1) An admixture with every 100 litres of alcohol of 2½ litres of a mixture composed of 4 parts of wood naphtha and 1 part of pyridine bases. In the event of the odour of this spirit proving objectionable, the addition of 50 grammes of lavender or rosemary oil may be made, to disguise the smell of the pyridine bases, but this is seldom done, probably on account of the expense.

This spirit is employed mainly for heating and lighting purposes, and but rarely for manufacturing operations, except in the making of cheap varnishes.

(2) An admixture with every 100 litres of alcohol of 1¼ litres

of the above denaturing mixture, and, in addition,  $\frac{1}{4}$  litre of a solution of methyl violet together with benzol, the quantity of the latter varying from 2 to 20 litres.

This spirit is authorised for use only for agricultural and motor engines.

Quite a number of special formulæ for incomplete denaturing have been approved, but for general use on a large scale in manufacturing operations, "wood spirit" denatured alcohol is allowed to be manufactured and employed under special conditions. This spirit consists of 100 litres of alcohol of not less than  $90^{\circ}$  ( $58^{\circ}$  O.P.) and 5 litres of wood naphtha.

In the United States the Internal Revenue authorities have authorised two formulæ for complete denaturing, and quite a number for partial denaturing applicable to special processes. The formulæ for complete denaturing are similar to those already given, viz:—

1. Alcohol, 100 gals. ; wood spirit, 10 gals. ; benzine,  $\frac{1}{2}$  gal.
2. Alcohol, 100 gals. ; wood spirit, 2 gals. ; pyridine, bases,  $\frac{1}{2}$  gal.

Completely denatured alcohol may be sold without restrictions. Users of this spirit consuming not more than 50 gallons per month do not require a permit, but manufacturers who require more than 50 gallons per month must obtain a permit from the collector of Inland Revenue in the district where the business is situated. In those cases where the completely denatured alcohol is unsuitable, application has to be made for a special formula, and certain regulations as to storage and use have to be complied with by those who have obtained special permits.<sup>1</sup>

The quantities of spirits issued duty free in Great Britain for the years (ending March 31st) 1907-12 were as follows, the figures being taken from the Annual Reports of the Commissioners of Inland Revenue and the Government Chemist :

<sup>1</sup> U.S. Internal Revenue Circular, 1907.

TABLE XXVII.—DUTY FREE SPIRIT ISSUED IN GREAT BRITAIN.

	Proof Gallons.					
	1907	1908	1909	1910	1911	1912
Mineralised methylated spirit for domestic use. }	2,338,000	2,339,782	2,395,306	2,302,091	2,407,578	2,529,544
Industrial methylated spirit for manufacturing purposes. }	3,717,000	4,115,593	3,884,739	3,859,158	4,323,452	4,853,581
Spirits otherwise denatured for manufacturing purposes. }	432,000	473,509	439,038	519,818	622,782	630,167
For use in Universities, Hospitals, etc. }	4,000	4,323	4,815	4,951	5,193	6,063
Total.	6,491,000	6,933,207	6,723,898	6,686,018	7,359,005	8,019,355

The Finance Act of 1902 allowed the use of specially denatured alcohol for certain industrial purposes, and also for use in universities, hospitals, etc., under special permits, the increase in the demand for this class of spirits being shown by the following figures :

TABLE XXVIII.—CONSUMPTION OF DENATURED ALCOHOL IN GREAT BRITAIN.

Year ending March 31.	Proof Gallons.		
	For use in Manufactures.	For use in Universities, Hospitals, etc.	Total.
1903 . . . . .	341	358	699
1904 . . . . .	206,452	2,272	208,724
1905 . . . . .	267,706	2,680	270,386
1906 . . . . .	354,516	3,314	357,830
1907 . . . . .	431,898	4,017	435,915
1909 . . . . .	439,038	4,815	443,853
1910 . . . . .	519,818	4,951	524,769
1911 . . . . .	622,782	5,193	627,975

The production and distribution of spirits in Great Britain for the year ending March 31st, 1916, were as follows :—

	Proof Gallons.
Used for methylating . . . . .	9,539,547
„ in arts and manufactures . . . . .	1,165,148
„ for fortifying wines, ship's stores, etc. . . . .	544,555
Exported . . . . .	9,186,002
Detained for home consumption (potable) . . . . .	28,949,519
Distilled . . . . .	49,135,199
Allowed for deficiencies, etc. . . . .	6,503,080

TABLE XXIX.—IMPORTS OF SPIRITS INTO GREAT BRITAIN.

Year.	Proof Gallons.		
	Total Imports.	Not for Methylation.	For methylation and for use in arts and manufacture on which differential duty has been paid.
1900 . . . . .	1,429,910	1,225,614	61,708
1901 . . . . .	2,001,994	1,456,184	380,555
1902 . . . . .	3,184,729	1,622,260	1,422,809
For arts or manufacture . . . . .	—	—	228
1903 . . . . .	1,907,099	1,476,712	411,949
For arts or manufacture . . . . .	—	—	1,764
1904 . . . . .	465,713	417,016	15,145
For arts or manufacture . . . . .	—	—	2,801
1905 . . . . .	239,265	203,176	24
For arts or manufacture . . . . .	—	—	3,577
1906 . . . . .	451,108	383,791	—
For arts or manufacture . . . . .	—	—	3,433

The amount of industrial methylated spirit employed in Great Britain, and the uses to which it is put, are given in the Annual Reports of the Government Chemist. For the years ending 31st March, 1911 and 1912, these were as follows :

TABLE. XXX.—INDUSTRIES IN WHICH ALCOHOL IS USED AND AMOUNT CONSUMED.

Nature of Manufacturing Operations or other purpose for which the Industrial Methylated Spirit is used.	Quantity used.	
	1911 Gallons.	1912 Gallons.
Manufacture of finish for sale . . . . .	369,283	419,080
Manufacture of varnishes, polishes, or lacquers for sale . . . . .	940,186	1,030,262
Manufacture of stains, paints, enamels,, etc., for sale . . . . .	61,176	63,242
Manufacture of varnishes, finish, stains, polishes, lacquers, etc., or for polishing purposes in manufacturers' own workshops . . . . .	294,343	316,863
Manufacture of felt and other hats . . . . .	123,430	128,001
Manufacture of celluloid, xylonite, and similar substances . . . . .	24,003	28,729
Manufacture of oilcloths, leather-cloths, pegamoid, and similar substances . . . . .	151,184	173,203
Manufacture of linoleum and similar substances . . . . .	948	398
Manufacture of smokeless powders, fulminates, and other explosives . . . . .	20,327	21,134
<i>Carried forward</i> . . . . .		

TABLE XXX.—Continued.

Nature of Manufacturing Operations or other purpose for which the Industrial Methylated Spirit is used.	Quantity used.	
	1911 Gallons.	1912 Gallons.
<i>Brought forward</i> . . . . .		
Manufacture of soap . . . . .	144,884	160,166
Manufacture of electric light filaments . . . . .	13,151	11,835
Manufacture of electric cables . . . . .	4,762	1,832
Manufacture of incandescent mantles . . . . .	18,176	33,169
Manufacture of ether . . . . .	192,878	212,058
Manufacture of chloroform . . . . .	9,627	7,912
Manufacture of ethyl chloride and bromide . . . . .	747	652
Manufacture of solid medicinal extracts . . . . .	56,720	58,438
Manufacture of alkaloids and fine chemicals . . . . .	22,868	33,856
Manufacture of embrocations, liniments, and lotions . . . . .	32,230	33,302
Manufacture of surgical dressings . . . . .	8,461	7,400
Manufacture of capsules and other medicinal appliances . . . . .	1,846	1,190
Manufacture of hair washes . . . . .	19,770	26,787
Manufacture of cattle medicines . . . . .	2,324	2,031
Manufacture of plant washes, insecticides, and sheep dips . . . . .	9,630	10,763
Manufacture of aniline and other dyes—solids . . . . .	874	1,515
Manufacture of aniline and other dyes—liquids . . . . .	4,382	1,526
Manufacture of fireworks and matches . . . . .	5,207	4,785
Manufacture of photographic plates and papers and other photographic purposes . . . . .	42,294	43,425
Manufacture of steel pens . . . . .	3,264	2,739
Manufacture of silk, crape, and embroidery . . . . .	9,698	7,341
Manufacture of artificial flowers, etc. . . . .	4,435	4,695
Manufacture of rubber . . . . .	1,223	921
Manufacture of artificial silk . . . . .	—	—
Manufacture of ships' compasses, spirit levels, etc. . . . .	952	1,433
Manufacture of inks . . . . .	2,558	438
Manufacture of collodion . . . . .	8,302	5,331
Manufacture of disinfectants . . . . .	220	1,866
For dyeing and cleaning operations in laundries and dye-works . . . . .	46,992	45,394
For textile printing . . . . .	11,115	8,416
For preservation of specimens in museums and hospitals . . . . .	6,850	6,562
For educational and scientific purposes in colleges and schools . . . . .	6,150	6,435
For analytical and scientific purposes in the labora- tories of analysts, works chemists, etc. . . . .	4,836	6,577
For hospitals, asylums, and infirmaries . . . . .	43,662	47,216
For electrotyping and printing . . . . .	553	1,450
For miscellaneous uses . . . . .	8,573	7,671
For Admiralty dockyards and War Office arsenals and workshops, chiefly for varnishes and polishes . . . . .	13,541	20,455
Total . . . . .	2,748,563	3,008,514

The following is an alphabetical list of uses to which alcohol may be put, and of the arts, crafts, and industries in which alcohol is an important factor, with reference letters and key showing the function of alcohol in the particular industry :

*Key.*—(a) The alcohol acts as a solvent for resins, or both dyes and resins, the solution of which is applied to articles in this industry (varnishes, varnish stains, lacquers, antifouling compositions, insulating compositions, etc.). Lacquers are thin and dilute, and most generally applied to metals. Spirit varnishes are glossy paints in which varnish formed as above plays the part of vehicle.

(b) Alcohol is a vehicle for flavouring fruit essences.

(c) As in (a), but as solvent for waterproof and airproof materials in addition.

(d) Alcohol acts by bringing substances into solution and as a volatile vehicle and diluent.

(e) The alcohol acts as a preserving medium or remedial agent *per se*.

(f) The alcohol acts as the raw material, the purifying medium, the coagulant, or as the solvent vehicle in which the reaction takes place, or the medium by which the finished article is used or applied.

(g) The alcohol acts as a solvent vehicle for an aseptic or antiseptic agent or disinfectant.

(h) The alcohol acts as the volatile solvent for a dye generally insoluble in water.

(i) The alcohol acts as a solvent for a stiffening agent, insoluble in water.

(j) The alcohol acts as a solvent vehicle for nitro-cellulose.

(k) The alcohol acts as a solvent vehicle for scents and perfumes.

(l) The alcohol acts as a solvent vehicle for a salt which imparts a coloured flame to burning alcohol.

(m) The alcohol is used in adjusting these instruments.

(n) The alcohol is used as a solvent for coating to protect steel.

(o) The generator of cold, the ether which produces sterilisation is an alcohol derivative.

(p) The alcohol acts as a solvent for the impurities.

(q) The alcohol acts as vehicle for incorporating agents, or as a necessary constituent of the mixed solvent.

(r) The alcohol solution of resin acts as a vehicle for the vitrifiable pigment.

(s) The alcohol acts as vehicle for the flux.

(t) The alcohol acts as a solvent, volatile or otherwise.

(u) The alcohol is used in candle polishing, or as a vehicle for the wick-curing chemicals.

(v) The alcohol acts as a combustible fuel. There is also a "solidified" alcohol used as a fuel. In blackboard slating the alcohol is fired to produce a dull surface.

(u) The alcohol acts as a volatile vehicle for the tan.

(x) The ignited vapour of alcohol renders the mantle incandescent.

(y) Alcohol is the main raw material.

(z) Alcohol acts as a motive power for machinery.

- A
- Accoutrements, military  
(a) (c) (h).  
Accroides varnish (a).  
Acetic acid and acetates  
(f).  
Acetic ether (f).  
Aconitine (f) (p).  
Aerated waters (b) (k).  
Agricultural implements  
(a).  
Airproof vessels (c).  
Alkaloids (f).  
Alkanet root (d).  
Aluminium enamel (a).  
    " printing  
    ink (a).  
Amber varnish (d).  
Anatomical specimens (e).  
Aniline dyes (f).  
Antipyrin (f).  
Antiseptics (g).  
Aquaria (c).  
Articles of vertu (a).  
Artificial camphor (f) (p).  
    " ebony (a) (h).  
    " flowers (a) (h).  
    " grass (a) (h).  
    " jasmine (f).  
    " leather (j).  
    " malachite (a)(h).  
    " musk (f).  
    " veroli (f).  
    " perfumes (i).  
    " shellac (t).  
    " silk (f).  
    " stone (a) (h).  
    " teeth (a).  
    " tortoiseshell (a).  
Astronomical instruments  
(a).  
Atropine and its salts (f).  
Automobiles (a) (j).
- B
- Badges (a) (h) (r).  
Bags, canvas (a) (h).  
Bags, leather (a) (h).  
Balance, chemical (a).  
    " spring (a).  
Balloons (c).  
    " toy (c).
- Balls (a).  
Bangles (a).  
Barrows (a).  
Baskets (a).  
Bedsteads (a).  
Bells, hand (a).  
Benzoin tincture (d).  
Bicycles (a).  
Bird stuffing (g).  
Black enamel (a).  
    " varnish (a).  
Blackboy gum varnish (a).  
Blacking (d).  
Blasting powders (f) (j).  
Blue dyes (h).  
    " enamel (a).  
    " lacquers (a).  
    " varnish (a).  
Boat building (a).  
    " models (a).  
    " painting (a).  
Bookbinding (a).  
Boot polishes (h).  
Boots (h).  
Botanical specimens (e).  
Boxmaking (a).  
Brass instruments (a).  
    " ornaments (a).  
    " polishing (t).  
Brassfounding (a) (p).  
Brazing (s).  
Brewer's glaze (a).  
Bridles (a).  
Brilliantine (h).  
Bronze printing ink (a).  
Bronzing (a).  
Brown dyes (a).  
    " enamels (a).  
    " lacquers (a).  
    " stains (a).  
    " varnishes (a).  
Brushes (a).  
Buckets (a).  
Burns (e).  
Butyric ether (y).
- C
- Cabinetmaking (a).  
Cagemaking (a).  
Candelabra (a).  
Candlemaking (a).  
Canes (a).
- D
- Canisters (japanning) (a).  
Canoe building (a).  
Cantharidine (f).  
Canvas (c).  
Caoutchouc (f).  
Capsules (a).  
Carbolic acid (g).  
Carpenters' tools (a).  
Carriage decoration (a).  
Cartridge case making (c).  
Case, show, etc. (a).  
Cattle medicines (g).  
Celluloid (j).  
Cement (t).  
Chandeliers (a).  
Chatelaines (a).  
Chemical analysis (f) (t).  
    " cleaning (p).  
    " processes.  
Cherrywood stains (a).  
Choral hydrate (y).  
Chlorodyne (e).  
Chloroform (y).  
Church illumination (v).  
Clarionette (a).  
Cleaning (p).  
Clinometer (a).  
Clockmaking (a).  
Cocaine hydrochloride (f).  
Cochineal tincture (h) (t).  
Cold storage (o).  
Collodion (j).  
Compasses (m).  
Copal varnish (t).  
Copperplate (a).  
Copying presses (a).  
Corkscrews (a).  
Corsetmaking (n).  
Cosmetics (e).  
Cotton dyeing (h).  
Cotton treating with nitro-  
cellulose (j).  
Crape stiffening (i).  
Curling tongs heaters (v).

- Drapery (*h*).  
 Drawers (*a*).  
 Drums (*a*) (*c*).  
 Dry-rot preventives (*g*).  
 Dye manufacture (*f*).  
 Dyeing (*h*).
- E**
- Eau de Cologne (*k*) (*y*).  
 Edible fats (*p*).  
 Electric light (*z*).  
 Electrical instruments (*a*).  
 Electrical lamp filaments (*j*).  
 Electrodes for storage batteries.  
 Embalming (*g*).  
 Emulsions (photo) (*j*).  
 Enamelling (*a*).  
 Enamel paints (*a*).  
 Engine models (*a*) (*z*).  
 Engines, motor power—fuel in Serpollet's steam (*z*).  
 Engraving (*a*).  
 Eserine and its salts (*f*).  
 Etching (*a*).  
 Ether (*f*).  
 Ethyl bromide (*f*).  
 Ethyl chloride (*f*).  
 Ethyl iodide (*f*).  
 Explosives (*j*) (*q*).
- F**
- Fabrics (*c*) (*h*) (*i*) (*j*).  
 Feathers (*h*).  
 Fiddles (*a*) (*h*).  
 Finish (*a*).  
 Fireworks (*l*).  
 Floor polish (*a*).  
 Fluxes (*s*).  
 Frescoes (*a*).  
 Fruit essences (*b*).  
 Fuel (*v*).  
 Fulminates (*f*).  
 Furniture (*a*).
- G**
- Gallic acid (*d*) (*p*) (*t*).  
 Garment cleaning (*p*).  
 Gas brackets (*a*).
- Gas-pipe deposit remover (*t*).  
 Gauges (*a*).  
 Glass enamelling (*r*).  
 „ gilding (*a*) (*f*).  
 „ painting (*a*).  
 „ staining (*a*).  
 Glazes, coloured (*r*) (*s*).  
 „ glass (*r*) (*s*).  
 „ porcelain (*r*) (*s*).  
 Globes (*a*).  
 Gloves (*h*) (*w*).  
 Gold gilding and enamelling—china, glass, pictures (*r*) (*f*).  
 Gramophones (*a*).  
 Green dyes, stains, enamels, lacquers, varnishes (*a*) (*h*).  
 Grinding machinery (*z*).  
 Guaiacol carbonate (*f*).  
 Guitar making (*a*).  
 Gutta percha (*a*).  
 „ varnishes (*a*).
- H**
- Hair pins (*a*).  
 „ wash (*g*).  
 Halls, public illumination of (*x*).  
 Hammer handles (*a*).  
 Hard spirit varnish (*a*).  
 Harness compositions (*t*).  
 Hat dyeing (*h*).  
 „ making (*h*).  
 „ stiffening (*h*).  
 Heliotropine (*f*).  
 Hemp dyeing (*h*).  
 House decoration (*a*).  
 Horn (*a*).  
 Hosiery (*h*).  
 Hospital purposes (*e*).  
 Hydrometers (*m*).  
 Hydroquinone (*f*).
- I**
- Ice making (*o*).  
 Illumination (*x*).  
 Implements (*a*) (*j*).  
 Incandescent light (*f*) (*a*) (*j*).  
 Inkstands (*a*).
- Insecticides (*g*) (*h*).  
 Insulators (*a*).  
 Iridescent varnishes (*a*).  
 Ironmongery (*a*).  
 Ironwork (*a*).
- J**
- Japanning (*a*) (*h*).  
 Jewellery gilding (*a*), brazing, enamelling (*r*) (*s*).
- L**
- Laboratory (*d*) (*f*).  
 Lace (*j*).  
 Lacquers (*a*).  
 Lakes (*f*).  
 Lamps (*a*) (*v*) (*x*).  
 Lantern projections (*x*).  
 Latticework (*a*).  
 Laundry irons (*v*).  
 Lavatories (*a*).  
 Lead acetate (*p*).  
 Lead pencils (*a*).  
 Leather dyes (*h*).  
 „ enamels (*a*).  
 „ stains (*h*).  
 „ tannings (*l*).  
 „ varnishes (*a*).  
 Lens (mountings) (*a*).  
 Light (*x*).  
 Lincrusta Walton (*a*).  
 Liniments (*g*).  
 Linen dyeing (*h*),  
 „ treating (*j*).  
 Linoleum (*a*).  
 Liquid fuel (*v*).  
 Lithophane (*a*).  
 Lithography (*j*).  
 Looking-glasses (*a*) (*f*).  
 Luminous paints (*a*).
- M**
- Machinery (*z*).  
 Magnets (*a*).  
 Mahogany stains (*a*).  
 Malachite stains (*a*) (*h*).  
 Mantles, incandescent (*x*).  
 Maps (*a*) (*j*).  
 Marquetry (*a*) (*j*).  
 Masks (*a*).  
 Mats (*h*).

- Mats varnish (*a*).  
 Medicine.  
 Mercerising (*j*).  
 Metal plate work (*a*) (*z*).  
 Metalochrome (*a*).  
 Metals (*a*).  
 Microscopes (*a*).  
 Military uniform and service equipments (*a*) (*c*).  
 Mixing machinery (*z*).  
 Models (*a*).  
 Mordant (*a*).
- N
- Negatives (*j*).  
 Nets (*h*) (*w*).  
 Nitrous ether (*y*).  
 Nubian blacking (*a*).
- O
- Oil extraction (*t*).  
 „ refining (*p*) (*t*).  
 Orange dyes (*h*).  
 „ lacquer (*a*).  
 „ stains (*a*).  
 Outdoor illuminant (*x*).  
 Overmantels (*a*).
- P
- Paint cleaning (*p*).  
 „ machinery (*z*).  
 „ making (*t*).  
 „ removing (*p*).  
 „ restoring (*h*).  
 Paper (*a*) (*j*).  
 Paraldehyde (*f*).  
 Parquet floors (*a*).  
 Pedestals (*a*).  
 Pegamoid (*j*).  
 Percussion-caps (*f*).  
 Perfumery (*b*) (*f*) (*q*).  
 Phenacetin (*f*).  
 Phenazone (*f*).  
 Pharmacy (*b*) (*d*) (*e*) (*f*) (*g*) (*h*) (*k*) (*q*).  
 Pianos (*a*).  
 Picture cleaning (*p*).  
 „ framing (*a*).  
 „ gilding (*a*).  
 „ painting (*a*).  
 „ restoring (*p*).  
 Pilocarpine and salts (*f*).
- Picric acid (*f*) (*t*) (*h*).  
 „ „ stains (*t*) (*h*).  
 Pipe and pipe cases (*a*).  
 Printing (*a*) (*t*).
- R
- Rackets (*a*).  
 Red dyes (*h*).  
 „ enamels (*a*).  
 „ stains (*a*).  
 „ varnishes (*a*).  
 Ribbons (*h*).  
 Ropes (*i*).  
 Rubber goods finishing (*t*) (*p*).
- S
- Saddlery (*h*) (*a*).  
 Salicylates (*f*).  
 Sandalwood (*t*) (*f*).  
 „ lake (*t*) (*f*).  
 Santonin (*f*).  
 Sauces (*b*).  
 Scalds (*c*).  
 Scent (*k*).  
 School bags (*a*) (*j*).  
 „ furniture (*a*).  
 „ black-boards (*a*) (*h*) (*v*).  
 Shaving soaps (*g*).  
 Ship building (*a*).  
 „ painting (*a*).  
 „ bottom compositions (*d*).  
 Signalling, coloured flames (*l*).  
 Silk (*h*).  
 Shop fittings (*a*) (*y*).  
 Skin tanning (*w*).  
 Skins curing (*w*).  
 Slate enamel (*a*).  
 Soap manufacture (*t*).  
 Solidified spirit (*v*).  
 Soporifics (*f*).  
 Spectroscopes (*a*).  
 Spinning textile (*z*).  
 „ wheels (*z*).  
 Spittoons (*a*).  
 Stains, black (*a*).  
 „ blue (*a*).  
 „ green (*a*).  
 „ yellow (*a*).
- Stains, mahogany (*a*).  
 „ walnut (*a*).  
 Steel pens (*a*).  
 Stereoscopes (*a*).  
 Stethoscopes (*a*).  
 Street illumination (*x*).  
 Stoves (*v*).  
 Sulphonal (*f*).  
 Surgery (*g*) (*e*).  
 Surveying instruments (*a*).  
 Syringes (*a*).
- T
- Tan extraction (*t*).  
 Tannic acid (*t*) (*v*).  
 Tanning leather (*u*).  
 Tapestry (*h*).  
 Tartan (*h*).  
 Thermometers (*m*).  
 Thymol (*f*).  
 Timber preservation (*e*).  
 Tin decoration (*a*).  
 „ soldering (*s*).  
 Tinacidine (room disinfectant) (*g*).  
 Tinctures — arnica, benzoin, cantharides, cochineal, iodine (iron, turmeric, etc.) (*t*) (*q*).  
 Tinned goods, japanning (*a*).  
 Tobacco (*t*).  
 Toilet soaps (*t*).  
 Toys (*a*).  
 Tramcars (*a*) (*j*).  
 Transparent soaps (*t*) (*g*) (*h*) (*k*).  
 Trunks (*a*) (*g*).  
 Turmeric (*h*).  
 Turnery (*a*).  
 Turnips, etc., preservation for show (*g*).
- U
- Uniforms, accoutrements, and equipments (*a*) (*d*) (*h*).
- V
- Vanilla (*f*).  
 Varnish (*a*).  
 Vinegar (*y*).

Village illumination ( <i>x</i> ).	Weaving ( <i>z</i> ).	X
Violet dyes, enamels, lac-	White lead ( <i>p</i> ).	Xyloidin ( <i>j</i> ).
quers, varnishes, stains	Winnowers (fanners) ( <i>z</i> ).	Y
( <i>a</i> ) ( <i>h</i> ).	Wood carving ( <i>a</i> ).	Yacht building ( <i>a</i> ).
Violin varnish ( <i>a</i> ) ( <i>h</i> ).	„ dyeing ( <i>h</i> ).	Yacht decoration ( <i>a</i> ) ( <i>j</i> ).
	„ “enamelling” ( <i>a</i> ).	Yellow dyes ( <i>h</i> ).
	„ staining ( <i>a</i> ).	„ enamels ( <i>a</i> ) ( <i>j</i> ).
	„ work machinery ( <i>z</i> ).	„ lacquers ( <i>a</i> ) ( <i>j</i> ).
	Wool dyeing ( <i>h</i> ).	„ stains ( <i>a</i> ) ( <i>j</i> ).
Waterproofing ( <i>a</i> ).	Wool fat ( <i>p</i> ).	„ varnishes ( <i>a</i> ) ( <i>j</i> ).
Water-pumping ( <i>z</i> ).		

Alcohol is most extensively used where it first acts as a solvent for the raw material, so as to bring it into a condition fit for use, and is permanently retained in the preparation as the solvent vehicle by which the article may be used or applied in any desired degree of consistency. Alcohol thus figures as a raw material in chemical industry mainly by acting as a solvent for organic matters which are insoluble in water. As a solvent, alcohol is the anti-thesis of water. A resin dissolves in alcohol, it will not dissolve in water; water precipitates the resin from its alcoholic solution. Gum arabic dissolves in water; it will not dissolve in alcohol. Alcohol, in fact, precipitates the gum from its alcoholic solution, and advantage is taken of this principle to purify gums. A large number of organic substances dissolve in alcohol, and to a greater extent than in water. *Anatomical specimens*.—The ordinary methylated spirit is used in Britain. In Germany 1 litre of commercially pure methyl alcohol and 1 litre of petroleum benzine are used to denature 100 litres of alcohol for this purpose. In Belgium 500 grammes of nitro-benzol, 500 grammes camphor, or 1½ litres of methyl-ethyl-ketone are used to denature 100 litres of alcohol, and in 1903 they so denatured 1100 gallons at 50 per cent. strength. *Antiseptics*.—Alcohol is the raw material for the manufacture of many antiseptics, *e.g.*, iodoform. *Artificial flowers*.—Alcohol is used as a vehicle for the dye to dye paper, and as a vehicle for the scent to perfume.

*Bedstead enamels*.—Almost pure alcohol, with ½ litre of turps per 100 litres, is used in Germany as a solvent for resin. *Bismarck brown*.—Much alcohol is used in dissolving this dye, and in dissolving resins to form the spirit varnish stain which produces imitation mahogany. *Blacking*.—Blacking figures in the British Inland

Revenue returns as one of the products in which methylated spirit is used. It is used as a solvent for some of the ingredients in liquid blacking, or as a vehicle for a scent. *Black lead*.—These remarks apply also to black lead. *Brassfounding*.—This figures in British returns as a methylated spirit consuming business, as a vehicle for the lacquer for brass, or as varnish for foundry patterns.

*Calico printing* is also an alcohol consuming industry, possibly as a solvent for dyes. *Candlemaking*.—Industrial alcohol is consumed possibly in treatment of wicks, or polishing of candles, or as a solvent for dyes, etc. *Castor oil*.—Alcohol has been experimentally tried in Marseilles on the large scale as a medium or solvent by which to extract castor oil from the bean. The experiments are said not to have been attended with success, possibly from want of attention to details. Otherwise, there is no reason why castor oil should not be economically extracted from the castor oil bean by alcohol. *Celluloid*.—In this case alcohol acts as an ingredient of the solvent mixture used to dissolve the cellulose. In France 5069 hectolitres (111,518 gallons) of alcohol were used in this industry in 1901, only about one-half of that used in 1899. In Germany 493,636 gallons of alcohol were used in this industry in 1903. The denaturant in Germany is 1 kilogramme of camphor, or 2 litres of turpentine, or  $\frac{1}{2}$  litre of benzol, per 100 litres. *China manufacture*.—Alcohol is used as a solvent for the varnish vehicle used to paint the vitrifiable pigments on the china goods. *Coal-tar colour works*.—In coal-tar colour works it is extensively used as the starting-point of the manufacture of ethyl-aniline, from which so many coal-tar colours are derived, and as a purifying, extracting, and crystallising medium, and each German coal-tar colour factory is said to use 10 to 60 metric tons of alcohol per annum, but, of course, much of it is recovered and used again. *Corsetmaking*.—This figures as an unmineralised methylated spirit consuming industry in the Excise returns for 1901, to the extent of 590 gallons, possibly as a vehicle for stiffening agents. *Crape*.—These remarks also apply to crape. This industry, with silk and embroidery manufactures, consumed 8434 gallons of unmineralised methylated spirit in 1901.

*Diastase.*—The preparation of this enzyme or soluble ferment is apparently pursued on a commercial scale in France by the aid of alcohol. Pure alcohol is used, and is added to a solution of malt. The operations are conducted in closed vessels under Excise supervision. *Drying.*—Alcohol as it evaporates carries away the last traces of water in its train. In drying ethereal extracts, especially oils, it is invaluable in expelling the last traces of water and thus preventing bumping. Water may even be displaced from a substance by a great head of alcohol under pressure, as in making smokeless powders. *Dyeing, etc.*—Again, a somewhat recent development is the dyeing of delicate tints by alcoholic solutions of dye-stuffs. The objection to ordinary water in dyeing is that the salts in solution, especially lime, affect the tints. A weaker solution of a colour can be struck on to delicate fabrics from alcohol than from water. *Dyes.*—Alcohol is used either directly or indirectly in the manufacture of a large number of coal-tar dyes and other organic preparations, including victoria blues, brilliant green, patent blue, rhodamines, chryso-phenine, tartrazine, dianisidine base, auramine, benzopurpurin, rhodines, indazurines, direct violet, direct blues, tartrachromine, dioxyrubine, phenocyanine, antipyrine, ethyl chloride, phenacetine, salipyrine, dermatol, salol, heliotropine, vanilline, kryofine, guaiacal carbonate, isosafrol, nitrophenetol, phenetidine.<sup>1</sup>

Alcohol acts as an extracting and purifying agent. *The edible fat industry* may be mentioned, in which the alcohol dissolves the impurities, floats to the top, and the impure alcoholic liquid may be syphoned off and the alcohol distilled for re-use. *Enamel paints spirit.*—Alcohol is used as the solvent for the resins, and the solution of the resin in the alcohol as the vehicle for the paint or enamel. *Engines, Driving of.*—Alcohol is not used to drive engines in Britain. In Germany 648,010 gallons were used in alcohol-driven engines in 1903. *Electric lamp filaments.*—Methylated spirit is employed in the making of filaments in incandescent electric lamp manufacture. *Electrodes for electric storage batteries.*—The German Excise give a method for denaturation of alcohol for this industry, but it is difficult to see the function of

<sup>1</sup> Thos. Tyrer. *Jour. Soc. Chem. Ind.*, 1904, 221.

the alcohol unless it be as a vehicle for an insulating medium. *Electrotyping*.—The British consumption of unmineralised methylated spirit for this purpose was 128 gallons in 1901. *Embrocations, linaments, lotions, cattle, and other medicines*.—The British consumption of methylated spirit for 1901 in the preparation of these, mostly proprietary, articles was 15,410 gallons. *Essential oils*.—Alcohol is used as the solvent vehicle or medium for purification, and as a diluent for sale.

*Fireworks*.—Many salts when dissolved in alcohol impart exceedingly bright colours to its non-luminous flame, *e.g.*, common salt colours it yellow; cupric chloride, bright green; boric acid and barium chloride, pale green; strontium chloride and lithium chloride, bright red; potassium chloride, violet. Both the alcohol and the salts should be pure to get the best results. *French polishing*.—Both crude methyl alcohol (*i.e.*, wood spirit) and crude ethyl alcohol (methylated) are used as vehicles, solvents, and diluents for the solution of dyes and resins used in this industry. Crude wood spirit is bad from a hygienic point of view. *Fruit essences*.—This is an extensive industry in which alcohol figures largely. These substances are alcoholic solutions of aromatic products used in the making of sweets. Some of them are made from fusel oil, *i.e.*, from the last runnings in the rectification of raw spirit, and it is interesting to note that in the development of this new industry the German distillers are at times able to sell their residuals at a higher price than their main product. *Furniture polishes* (household revivers).—Alcohol enters into the composition of some of these, though more frequently turpentine is the solvent, as turps dissolves beeswax, but alcohol only acts as a very partial solvent. Alcohol has, however, the advantage of being a solvent for dyes.

*Gas-pipe deposit remover*.—This is used in Russia and Switzerland, where the intense cold in winter renders deposits of naphthalene of frequent occurrence. Possibly the addition of a little camphor to the alcohol would aid the solvent action of the alcohol. *Gilding*.—Alcohol forms a medium for the reduction of the gold chloride. *Glycerophosphate of lime*.—The French Excise authorities publish a method for denaturing alcohol for use in this

industry. The alcohol is added to the glycerophosphate dissolved in an aqueous mixture of ammoniacal salts and ammonia. The work is done in closed vessels under the supervision of an officer at the manufacturer's expense. *Gum resins.*—See under Varnish. Of true gum resins only the resinous ingredient is soluble in alcohol.

*Hatmaking.*—Both crude methyl alcohol (wood spirit) and unmineralised but methylated spirit are extensively used as solvents. The British industry consumed 121,104 gallons of unmineralised spirit in 1901. The French, 9218.

*Indiarubber.*—1. *Use of alcohol as a coagulant for the latex.* According to Morisse, “1 volume of 90 per cent. alcohol coagulates 6 volumes of latex, yielding a superb rubber of brilliant whiteness, and yellowing but slightly on ageing, but the high cost of alcohol and its feeble coagulating power puts it out of the reckoning.” This was written in 1889, when alcohol was much dearer than it is now. Unfortunately the latex must be treated on the spot. Under favourable circumstances, however, alcohol should be the coagulant *par excellence* for indiarubber latex, and where rubber is produced in plantations it may be advantageously used. By the aid of a still, much of it might be recovered and used again. It is evident that as alcohol coagulates the rubber in the latex, rubber is insoluble in alcohol. 2. *Finishing of rubber goods.* The use of alcohol for this purpose is possibly to remove the sulphur efflorescence, or as a vehicle for a scent to disguise the smell of coal-tar naphtha, and of sulphuretted hydrogen, the latter from the vulcanisation process. *Ink manufacture.*—This industry figures amongst those in which unmineralised methylated spirit was used in 1901. The probabilities are that the alcohol was used as an ingredient in spirit varnish or spirit stain used as a marking ink, or as an aid to reduce silver nitrate. *Insecticides.*—The function of the alcohol is as a solvent for nicotine.

*Laboratory.*—The uses of alcohol in the laboratory are numerous. 1. It is the solvent *par excellence* for all organic substances which do not dissolve in water. Ammoniated alcohol is a better extracting agent than alcohol alone, where a coal-tar is “struck” on to an inorganic base. 2. It may replace water in saponification

with many advantages, both the solid caustic alkalis (potash and soda) being soluble therein. 3. As a precipitating medium, it is only necessary to refer to the precipitation in presence of alcohol of dilute lead solutions by sulphuric acid, and the estimation of potash and ammonia by platinum chloride. The use of alcohol in the separation of barium from strontium and calcium is well known. We need not dwell on these minor uses of alcohol. The Auer incandescent lamp burning the vapour of alcohol is of use in polarimetry where gas, etc., is not available. The new alcohol lamps prove invaluable in such situations. There is also a great future for alcohol motors in laboratories. *Lanolin* is a mixture or emulsion of purified wool fat with about 20-25 per cent. of water, which has the peculiar property of taking up water like a sponge, hence it is an excellent medium for the preparation of salves and ointment. The wool fat is extracted from the crude wool or grease by petroleum spirit. Alcohol is used in its purification; no less than 21,824 gallons of methylated spirit were used in Germany for this purpose in 1903. The method of denaturing is to add 5 per cent. of petroleum. *Lincrusta Walton*.—In the case of pegamoid the function of alcohol is, of course, to aid in dissolving the nitro-cellulose, but the function of alcohol, both in linoleum making and lincrusta Walton is obscure, possibly it is used as a solvent for coloured rosins, or as a finishing spirit varnish to impart a gloss. In any case, 21,128 gallons of unmineralised methylated spirit were so used in Britain in the three industries in 1903.

*Mahogany stain*.—Enormous quantities of alcohol in the form of finish, ordinary methylated spirit, or unmineralised methylated spirit, are used as a vehicle in the staining of wood to imitate mahogany. There are no available statistics. (See under *Bismarck brown*.) *Museums*.—Spirit varnish is largely used in show-case making, and in polishes for the same, but also in preserving natural history specimens. In addition, it is used in the varnishes employed for coating delicate specimens to protect them from the action of the air and preserve them from decay, and as an ingredient of insecticides. It is of frequent use in taxidermy.

*Oil refining.*—Alcohol is used as a solvent for removal of free fatty acids and other impurities. In 1901, 1150 gallons of unmineralised methylated spirit were used for *paint cleaning*, according to the Excise authorities. Most likely the alcohol in question was used owing to its solvent power, or as a vehicle for some of the substances which dissolve dried paint, viz., carbolic acid, etc. *Perfumery.*—Alcohol is a constituent of the great bulk of perfumes now sold. It acts as a vehicle for the perfume. It also acts as a diluent, since perfumes in their natural state are often far too strong to be pleasant. Alcohol, moreover, is the raw material for many perfumes, such as acetic ether, butyric ether.

Enormous quantities of alcohol are consumed in the *perfumery* industry. A special phase of the perfume industry is the absorption of the perfume of flowers by fats, and the extraction of the absorbed perfume by alcohol. This is the reverse of the principle of the purification of animal fats, to render them edible, to which reference has already been made. In the former case the alcohol is used to remove the unpleasant-smelling impurities, and is recovered for future use. Here it is not only used to recover the scent, but also to act as the permanent vehicle and diluent therefor. The perfume industry has attained great developments in Germany. Although Germany entered into the competition for this trade without a single advantage in her favour, even its climate being against it, and not at all kindly disposed to odoriferous flowers and plants, yet, strange to say, it has been in this rigorous, unfriendly climate that the chemically pure odoriferous principles of essential oils have each in their turn been successively isolated; amongst others, *citral* from essence of lemon, *anethol* from essence of anise, and *menthol* from essence of peppermint, *geraniol* from geranium oil, and so on. But to crown these triumphs, several perfumes have been evolved synthetically, all by German chemists. Both natural and synthetic perfumes come upon the market as alcoholic solutions.

*Photography.*—Alcohol is used in photography in various ways. 1. *In collodion manufacture as a solvent.* The pyroxylin or nitro-cellulose is steeped in alcohol for twenty-four hours, but

there is an disadvantage in adding at the same time 10 per cent. of ether. A gallon and a half of collodion suffices to cover 500 square feet of collodion paper. The price of collodion would be very much reduced if it could be made from duty-free spirit; the present price of absolute alcohol is against this. 2. In the process of photography for stripping and drying negatives, alcohol is used for the purpose of stripping negatives and drying dry-plate negatives. A large quantity is consumed where the dry-plate process is used, when it is required to get the negatives dried quickly. Methylated spirit is not suitable for this purpose. It is used, however, but often spoils negatives. In the art of photography alcohol appears mainly as an ingredient of the solvent mixture for nitro-cellulose in the making of collodion and celluloid, and of the mixtures used to dilute it. Throughout the trade a great deal of collodion is used that is made with methylated spirit, but few commercial collodions are made entirely with it because they give too much trouble. *Colour photography.*—Alcohol figures largely in photography in colours, *e.g.*, Lippman uses, *inter alia*, in the making of gelatine plates, an alcoholic solution of cyanine, and moistens each plate with alcohol before washing it. Again, before use, the sensitised film is washed in a solution of absolute alcohol 100, nitrate of silver 0.5, glacial acetic acid 0.5.

*Resins.*—There are several solvents for resins, but alcohol and its derivatives are *par excellence* the solvents to be employed in making spirit varnishes, lacquers, etc. This is one of its principal uses. *Sheep dips.*—Possibly the 450 gallons of methylated spirit used in 1901 were for the extraction of nicotine from tobacco. *Silk manufacture.*—This industry, together with crape and embroidery manufacture, consumed 834 gallons of alcohol in 1901, mainly for stiffening. *Silvering mirrors: Martin's process.*—No. 1 Solution: 1, Dissolve in the cold 4 grammes of crystallised nitrate of silver in 100 c.c. of distilled water. 2. Dissolve 6 grammes of nitrate of ammonia in 100 c.c. of distilled water. Allow the solutions to stand for some time, then mix them. The solution should be tested in the manner given below. No. 2 Solution: Dissolve 25 grammes of sugar in 250 c.c. of water. Add 3 grammes of tartaric acid, boil about 10 minutes; *add*,

after cooling, 50 c.c. of 90 per cent. alcohol, and make up the volume to 500 c.c. No. 3 Solution: Dissolve 10 grammes of pure caustic potash in 100 c.c. of water. Clean the glass plate to be silvered (1) with nitric acid, (2) with a mixture of equal parts of solution 3 and alcohol, then the glass is washed, standing it in the distilled water. One hundred c.c. of No. 1 solution are run into a test-tube, then poured into a saucer or plate according to the size of the mirror; 50 c.c. of No. 2 solution are run into a second test-tube, and 50 c.c. of No. 3 solution added. The mixture is shaken and run into solution No. 1. The glass, which has remained under water, is brought rapidly into the liquid and kept at  $\frac{1}{2}$  centimetre from the bottom of the dish, taking care to agitate it gently. If the solutions have been properly made, the transparency of solution No. 1 is not altered when the mixtures of solutions No. 2 and 3 are added. *Soldering.*—Some 660 bulk gallons of alcohol were denatured in Belgium in 1903 for dissolving the resin used for soldering metal boxes. Not only is alcohol used as a flux in soldering, but it also constitutes the heating agent in new and improved brazing and soldering alcohol lamps, which burn a mixture of alcohol vapour and air instead of coal-gas and air, and is also a constituent of the lacquer.

*Spirit stains.*—Alcohol is a solvent and vehicle for the dye and resin. *Soap.*—In the manufacture of transparent soap, the soap is made in the ordinary way first, then it is shredded, and the natural water of it is dried out; then the soap is dissolved in methylated spirit, and part of the methylated spirit is distilled off. The resultant soap and spirit is then cast into blocks and cut up. It has to be kept for a long time to enable the spirit to evaporate at normal temperatures. As the soap is kept, it becomes more and more transparent. Soap made with methylated spirit is quite as transparent as that made with pure spirit. The French use certain scents which cannot be used in the concentrated state, but only in the form of solution, which are practically put out of reach in this country. They are mostly synthetic perfumes of slight solubility. Small quantities of soap have been made with duty paid spirit, but there is no appreciable

difference in the transparency. The alcohol is used mainly in connection with *transparent* soap. Alcohol has certain influences on the physical state of the soap which tend to make it a superior article. The real object of using alcohol is to eliminate the uncombined alkali and fat, and also to inhibit its tendency to crystallise or curd, which renders it opaque. Soap separates from solution in alcohol in a truly colloidal state, and is therefore more homogeneous. *Sugar: Use of alcohol in extraction of sugar from molasses.*—Again, alcohol has long been used to extract the impurities from lime sucrate, in the recovery of sugar from molasses. The alcoholic processes of extracting sugar from molasses have, however, nearly all been abandoned. There, nevertheless, remain a few applications of Manoury's system in Germany and Russia.

*Tannin.*—Alcohol is largely used abroad in the extraction of tannic acid from tanning materials. *Tanning leather.*—An alcoholic solution of tannic acid is used as a quick and effective vehicle in tanning. Tannic acid is soluble in alcohol, and alcohol is used to extract the tannin from gall nuts and other products.

*Varnish making.*—We now come to the spirit varnish industry, founded on the solubility of the raw resin in alcohol. Many resins are more or less completely soluble in alcohol, such as sandarac, shellac, common rosin, grass tree gum, and the oleoresin turpentine. Some varieties of manilla resin are soluble, others not. White hard spirit varnish and brown hard spirit varnish are alcoholic solutions of resins. By adding an alcoholic solution of Bismarck brown to a brown hard spirit varnish, a mahogany stain is obtained, and so on. Lacquers are more dilute solutions of resins, etc., than the spirit varnishes, and they may be tinted green by an alcoholic solution of brilliant green, yellow by a similar solution of chrysoidine, blue by an alcoholic solution of spirit blue, and black by nigrosine. *Vinegar making.*—Vinegar is made in Britain from malt, which is first mashed and fermented to alcohol, and there is now no tax on malt. France converted in 1901, 50,576 hectolitres of alcohol, or about 1,112,672 bulk gallons, directly into vinegar. Germany, in 1903, 3,624,588

gallons ; Switzerland, in 1903, 60,980 gallons ; Belgium, in 1903, 240,548 gallons.

*Waste products from distilleries, utilisation of.*—Amongst products obtained from distillery residuals may be mentioned *acetal* and *furfurol*, both of which are used in coal-tar colour manufacture. *Butyric ether* is also another product obtained from distillery residuals, and has the odour of pine-apples. Methyl chloride is obtained from beet distillery spent wash. Amyl acetate is produced by appropriate treatment of the fusel oil. We need not enlarge further on this part of the subject here, as it has already been dealt with.

## CHAPTER XIV.

### THE USES OF ALCOHOL FOR INCANDESCENT LIGHTING, HEATING, AND MOTIVE POWER.

*The principles of alcoholic illumination.*—Two systems of lighting by alcohol are in vogue: (1) Imparting luminosity to alcohol by carburetting it. Substances rich in carbon are added to the alcohol, *e.g.*, coal-tar distillates, petroleum or shale oil naphthas, capable of imparting to alcohol the luminosity which it lacks. (2) Rendering certain earths incandescent by the ignition of the vapour of alcohol previously mixed with air in contact with them.

The enrichment of alcohol with liquids capable of burning with highly luminous flames was the object of numerous researches about the middle of the nineteenth century. Spirit of turpentine, for instance, burns with a highly luminous but very smoky flame. By mixing it with alcohol the latter became luminous, and the tendency of the spirit of turpentine to burn with a sooty flame, if not entirely eliminated, was greatly diminished. But these efforts were made rather with the object of eliminating the smoke from the flame of the spirit of turpentine, than with the view of invoking the aid of spirit of turpentine, etc., to render alcohol luminous. We are now using hydrocarbons, which burn in a similar manner to spirits of turpentine, to render alcohol luminous without even distilling the mixtures. The first attempts to produce "burning oil" from a mixture of alcohol and hydrocarbons were made about 1832. At that period turpentine and tar distillers vied with each other in producing illuminants known in Britain as "camphine," and in France as "gasogene." The British camphine, which differed essentially from the French gasogene, was often simply a more or less well-rectified spirit of

turpentine. The French gasogene was a mixture in predetermined proportions of ethyl alcohol with wood spirit, spirit of turpentine, naphtha, shale oil, etc. Both French and British patents specified, as was natural, the alcohol being free from water, otherwise the mixtures would separate into as many layers as there were ingredients in the mixture. It was also insisted upon that the mixture should be distilled prior to use. The light afforded by the combustion of these fluids in suitable lamps—camphine and gasogene (vapour) lamps—was white and agreeable. The inventors all claimed this method of illumination as suitable for general use, but particularly so for distilleries. Amongst the numerous patents for this method of illumination, that of Ludersdorf of Berlin, dated 1834, is the most simple. Ludersdorf used a mixture of four volumes of 95 per cent. alcohol and one volume of rectified spirit of turpentine. His lamp was so arranged that the liquid sucked up by a wick placed in a suitable tube is vaporised by the heat of the flame, the vapour thus produced burning with a fine white flame. Moreover, this mixed fluid, although yielding a less intense light than that yielded by the use of turpentine alone, was more easily managed and less liable to smoke owing to its lower carbon content. Furthermore, the inherent difficulty of the diminution of capillarity in the wick incidental to the combustion of highly carbonaceous products due to the deposition of carbon in the pores of the wick was obviated, the lamp being lighted at the outset by burning a little alcohol in a cup fixed round the tube containing the wick, so as to vaporise the mixture brought up by capillarity. The burner tube containing the wick descends almost to the bottom of the reservoir. It is contracted at the top and terminated by a metallic knob, at the base of which are a row of perforations. Some alcohol is burned in a cup surrounding the wick tube, so as to vaporise the fluid raised by the wick. As the vapour issues from the perforations, it ignites, and the flame heats the knob. The lamp once lit, the heat conducted from the knob continuously vaporises the illuminant. An outer tube forms an annular air-space which surrounds the upper part of the burner tube, and thus prevents the spirit in the reservoir becoming overheated.

*Rendering a mantle of the rare earths incandescent by the ignition of the vapour of alcohol.*—The alcohol is first vaporised, and the vapour is burned in a Bunsen burner covered by an incandescent mantle on the Auer principle, or by some similar mantle. This latter method is a development of the principle on which the limelight is based. The intense luminosity of the limelight is due to the flame of an oxyhydrogen burner impinging on quicklime, the intense heat rendering the lime incandescent. The ordinary incandescent gas mantle is rendered intensely luminous by coal-gas, which is burnt in admixture with excess of air in a Bunsen burner. This mixture of coal-gas and air burns with a non-luminous flame, the heat generated being so intense as to render the mantle incandescent. If the mantle were made of less intractable material it would fuse, and the heat would be expended in fusing it and keeping it in a state of fusion. But as the heat is not dissipated in fusing the mantle, the material of which it is composed being infusible, the heat is converted into light. The heat must be sufficient to maintain the mantles continuously incandescent for the light to be continuous, and this is done by burning the proper amount of the mixture of coal-gas and air. To pass from a mixture of coal-gas and air to a mixture of alcohol vapour and air was merely a step. Incandescent alcohol lamps were, in fact, the natural sequence of incandescent gasburners. But as a matter of history that is not quite correct. It would be more correct to say that incandescent gas lighting was the tardy sequel to the invention, about 1847, of a system of incandescent lighting in general by the use of alcohol in particular. Frankenstein, the editor of a trade journal at Gratz, about that time published a series of articles on a peculiar system of lighting of his own invention, by which he imparted great luminosity to the colourless, or rather non-luminous, alcohol flame. A report, published in 1848, gives a detailed description of Frankenstein's method. It is based on the known fact that certain bodies, especially the alkaline earths, when heated to incandescence, emit a very intense light. The inventor used a lamp with a round wick, burning alcohol with a colourless flame. In this flame he introduced a gauze cone, or a cone of any other appro-

priate tissue, steeped in a paste composed of lime, magnesia, water, and gum arabic. The preparation of the paste, the method of shaping the tissue cone, steeped in this paste, the drying, and other particulars, are all described with many details, so that the reader sees at a glance the forerunner of the Auer or Welsbach mantle, which, however, differ materially from that of Frankenstein by the substitution of the rare earths for the alkaline earths, and by the use of salts decomposable by heat, in place of oxides. Whether Frankenstein's invention had any temporary success is unknown. But he certainly had a great many followers, as the numerous English and German patents for this style of lighting testify. But it was not until 1895 that a really practical method of illumination was evolved by Auer's mantle, and in improved lamps which yield a fine light with a moderate consumption of methylated spirit. There are several links in the chronological chain of the history of incandescent lighting. There is first the well-known oxyhydrogen limelight, then Frankenstein's alcohol lime-magnesia light. Then the coal-gas Bunsen burner, then Auer's mantle of salts of the rare earths, the prelude to the ignited vapour of alcohol mixed with air rendering a similar mantle incandescent. Having now briefly summarised the principles of these systems of incandescent lighting, leaving incandescent electric lighting out of account for the present, as being the result of electrical phenomena pure and simple, let us pass to the consideration in detail of the system which concerns us for the time being, namely, the study of the different indoor and out-of-door applications of illumination by the ignition of a mixture of the vapour of alcohol and air rendering an infusible white tissue of the rare earths luminous. By means of alcohol it is possible to fit table lamps with the incandescent light. The incandescent light was formerly practically possible only with gas, although paraffin lamps have been made for an incandescent mantle. Since the introduction of alcohol lamps, it is stated that one firm alone sold 53,000 of these lamps in three months in Germany. The general principle of their construction is as follows: The alcohol is vaporised by a small heating vessel—often so minute as to consist of a mere tube—charged either by (1) capillarity, or

(2) pressure: charging by capillarity involves cotton wicks dipping into the reservoir; charging by pressure necessitates placing a charging reservoir on the burner, or the air contained in the alcohol reservoir may be compressed by means of a pump fixed on the exterior—occasionally the necessary pressure is produced by the expansion of the air contained in the reservoir by the heating of the metal parts of the lamp. Auer causes the alcohol in the reservoir of the lamp to rise by means of a series of wicks towards a small vessel heated by the flame of a small night-light wick fed from the same reservoir. In this way the alcoholic vapour is made to issue with considerable force through the small cone of the burner, and with it a current of air, the alcohol-charged air, on reaching the upper part of the jet, burning with an extremely hot, non-luminous flame, which immediately renders the Auer mantle composed of the rare earths incandescent. It has been urged against this method that the lamp requires to be lighted twice, first the small wick, and then the mantle, and that it wastes a small quantity of alcohol in heating a larger; once started, however, it burns regularly and very cheaply, but, as will be seen, it is somewhat dangerous, and does not admit of the use of glass or porcelain containers.

On the same principle as the Auer, the Continental Nouvelle Co. of Paris manufacture a burner styled the *bec préféré*, in which the vaporiser, in which the alcohol is volatilised, is fed with alcohol by means of wicks. The alcohol vapour issues from the injector, draws in an exactly regulated current of air, and thus reaches the burner. The vaporiser is heated by a night-light wick shielded from draughts by a metal jacket. The wick of this small jet may be regulated at will by raising or lowering a hooked tube to a greater or less extent. To light the lamp, the hooked tube is lowered to the bottom, the small jet is lighted, and in a minute or so the lamp is lighted by holding a light of some kind at the upper end of the glass chimney. The mantles hitherto used for alcoholic lamps are those made for use with coal-gas, without regard being paid to the difference in temperature between an alcohol burner and a gas-burner. Good mantles such as the Auer mantles are made by means of a solution of the nitrates of the

rare earths, consisting of 99 per cent. thorium and 1 per cent. cerium. This formula was adopted as the result of the experiments of Landolt and Hintz, who found that mantles of the same dimensions, steeped in solutions of the same concentration, gave, according to the relative proportion of oxide of cerium, the following luminous intensity estimated in carrels of 9.62 bougies :

TABLE XXXI.—LUMINOUS INTENSITY OF INCANDESCENT MANTLES.

Thorium.	Cerium.	Carrels.	Thorium.	Cerium.	Carrels.	Thorium.	Cerium.	Carrels.
99.1	0.1	1.8	99	1	7.8	95	5	4.4
99.8	0.2	4.5	98	2	6.8	90	10	1.2
99.5	0.5	6.8	97	3	3.5	85	15	1.0
						80	20	1.0

The effect of the Auer mantle on the luminosity of an alcohol flame is very remarkable. M. Sorel has demonstrated that an alcohol flame burning freely is so feebly luminous that it is necessary to burn 100 grammes to produce one French candle-power per hour. But if a mantle be placed in the flame of previously vaporised alcohol, the consumption diminishes to about 2 grammes. An alcohol flame burning freely expends per French candle-power per hour, 6 grammes of carburetted alcohol, and barely 1 gramme of carburetted alcohol burning on a mantle. Carburetted alcohol compared against methylated spirit, *ceteris paribus*, has therefore greater luminosity, and thus less of it is consumed to get the same amount of light. This is due to the difference in their composition. Illumination is generally the more economical the more intense it is. The unit of luminosity costs less in a very powerful lamp than in a domestic lamp. It has been demonstrated that the pressure with which the alcohol arrives at the injector increases the intensity and diminishes the expense per unit of light, owing to the fact that the jet of burning alcohol is projected more perfectly and brings into a more constricted space the amount of air to be burnt, making a column of flame analogous to a blow-pipe flame. If the mantle has been well chosen, it exactly encloses the flame, and the mantle thus

assumes its maximum luminosity. It follows, therefore, that for illumination on the large scale it is desirable to employ carburetted alcohol, and at the present price of benzine this can be done economically. It is desirable to use powerful lamps so as to get a cheaper unit of light. Pressure lamps are, in fact, best, since provided with well-fitting mantles they produce the maximum of luminosity. But in houses where carburetted alcohol cannot be burnt for fear of producing a smoky flame, where an intensity of 25-30 bougies cannot be exceeded, and where a pressure reservoir is not available in spite of the few farthings extra cost per night, it is better to use methylated spirit. Incandescent alcohol lamps without previous vaporisation, in which quick lighting is a feature, consist simply of a paraffin oil Argand lamp with a round wick, which is lighted directly, and by a bayonet arrangement there is fixed above the flame the Auer mantle and the glass chimney. The wick is raised to a certain height so that the flame strikes the mantle, the latter immediately becoming incandescent, the burner being so constructed as to ensure a supply of air all round the flame. The first lamp based on this principle was exhibited in Berlin in 1902 by Aschner, but acted only moderately well. But other makers succeeded in doing better, and at the Paris Exhibition of 1902, two models of lamps on this principle were exhibited by Schuster and Baer of Berlin. One of these, made entirely of copper, is furnished with an *open* unobstructed channel through the axis of the alcohol reservoir, thus ensuring a good current of air through the interior of the flame, which, increasing the heat of the latter, renders the mantle still more vividly incandescent. The second model had a glass reservoir without a central air channel.

Jean Delamotte's alcohol vapour lamp (Figs. 80, 81) has neither heating jet nor recuperator. The alcohol is drawn into the heater A (Fig. 81) by the cotton wicks C, within the tubes B. A is heated by small flames coming from the Bunsen D, and the alcoholic vapour from A descends by the tube *a* to the injector *b*. The Bunsen D is placed in *c* above the injector *b*, it is held in P, supported against the rim of the heater A. It is lighted by heating the Bunsen D by means of a "topette" dipped in alcohol,

the gas escaping by the small orifice *o* first becomes alight itself, warms the heater *A*, then lights the mixed gases above the orifice of the Bunsen *D*, the mantle *Z* becoming incandescent and soon extremely bright. To extinguish the lamp the injector *b* is closed by the key *M*. Light is applied by the "topette" inserted for the purpose into the funnel; the tail end of the "topette" being



FIG. 80.

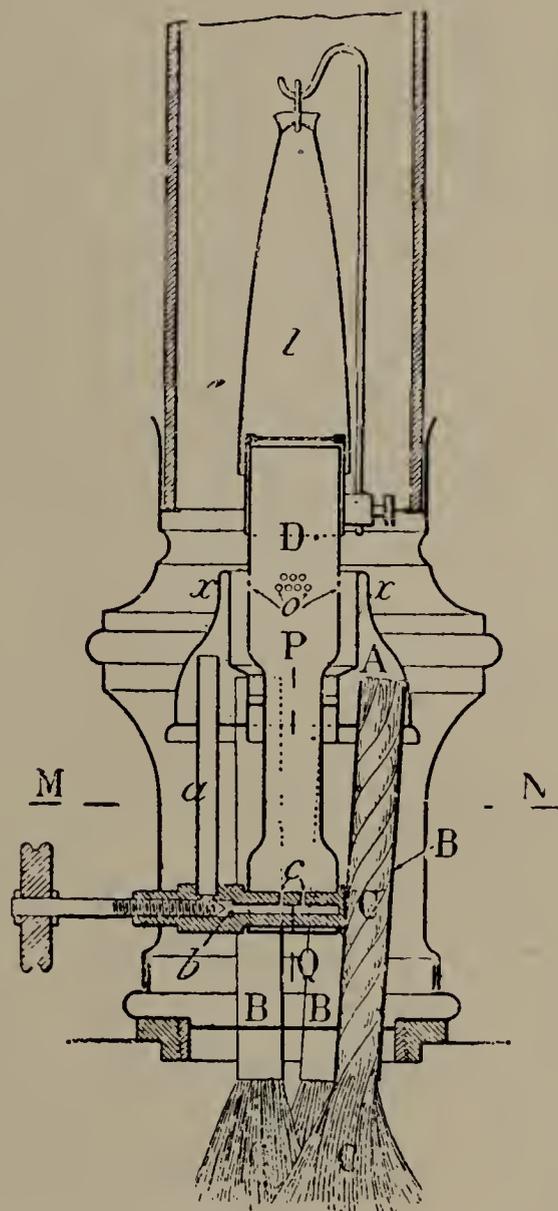


FIG. 81.

Alcohol vapour lamp on incandescent principle for domestic illumination (essential parts) (DELAMOTTE, Paris).

rested in the small recess. The alcohol drawn up continuously by the wicks into the heater is vaporised, the gas formed in this way ascends into the upper part of the heater and redescends by the small tube *C* as far as the holes of the injector, through which it issues and passes into the Bunsen burner, drawing in the requisite amount of air for its complete combustion on the grating at the

exit of the Bunsen. The lamp lights itself by a single application of a flame without the necessity of bringing the flame above the glass. On the upper part of the Bunsen are three groups of small holes through which a portion of the gas is derived. This gas burns with a blue flame, and heats the upper part of the heater, and thus induces vaporisation of the alcohol. On the upper part of the Bunsen are three copper studs to keep the Bunsen in its place, and care has to be taken to place the Bunsen in a vertical position over the injector, and to insert it at the bottom. The notch at the bottom of the Bunsen is intended for this purpose. To extinguish the lamp the milled head is turned to the right. In two or three seconds it is given two turns to the left, so as to open it, and then it is left so; the tap of the lamp should always be left open, it is only closed for a moment to extinguish the light.

*The mantle.*—Take the mantle from its box, being careful not to squeeze it in the hand. It is far better not to touch it, but to place it on the burner, by the small cotton thread attached to it. To put the mantle Z on the burner D, hook it on to the rod, then fix the latter in the socket, pressing the screw seen in Fig. 81. The mantle fixed, the socket is placed on the Bunsen. The first time the mantle is used it should be inflamed before lighting the lamp, thus: The mantle is placed on the burner and a flame applied to its upper part, the thin layer of collodion with which it is covered so as to protect it before being brought into use being instantaneously burnt.

*Wicks.*—These last for a long time and are easily renewed; they only require to be pulled to remove them. To insert the new wick, seize with flat pincers the metal rod with which each is furnished and insert wick well at bottom.

The working arrangements of the “Monopole” lamp for indoor and outdoor illumination are shown in Fig. 82. The alcohol in the cylindrical reservoir *a* descends through *c* into another small, lower receptacle, into which the extremities of tubes lined with asbestos dip; these suck up the alcohol, which is then vaporised by the heat of the mantle, the vapour passing through the tubes *k k* to *l*, where it deposits the condensed drops, mixes with the air drawn in from the exterior, and burns under the mantle. The use of the small reservoir *d d* in communication with the principal reservoir

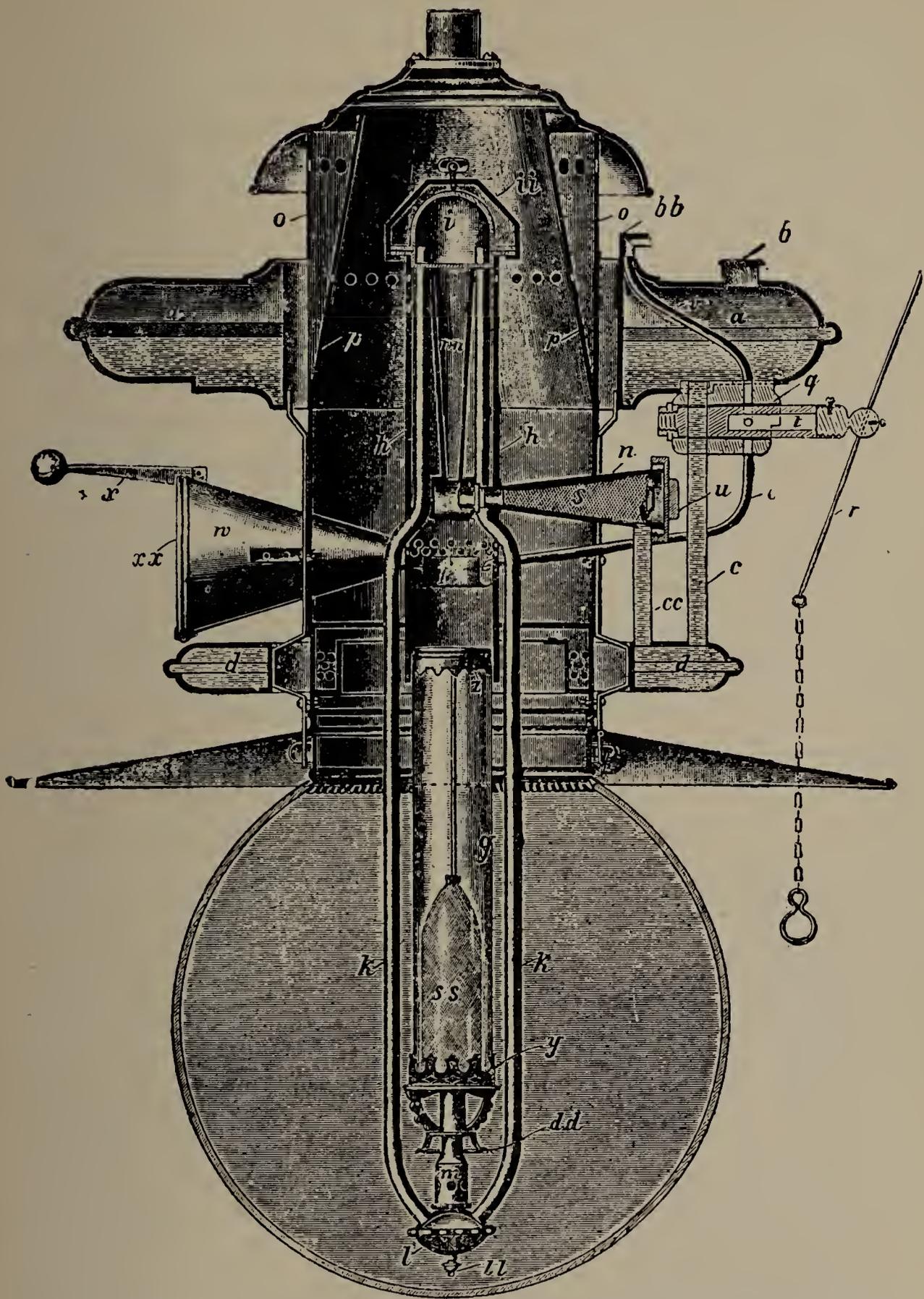


FIG. 82.—Monopole lamp for indoor and outdoor illumination (DELAMOTTE, Paris).  
*a*, reservoir; *b*, aperture for filling; *c*, feed; *d*, regulator; *e*, pipe leading alcohol to *f*; *f*, receptacle for lighting; *g*, lamp glass; *h*, cylinder prolonging lamp glass; *i*, vaporiser; *k*, alcoholic vapour pipe; *l*, asbestos receptacle; *m*, burner; *n*, asbestos cone holder; *o*, protective envelope; *p*, apartment entering into *o*; *r*, tap lever; *s*, asbestos cone; *t*, filling orifice; *v*, air pipe; *w*, lighting funnel; *x*, shutter for *w*. (Illuminating capacity, 80 to 150 French candle-power and upwards).

*a a* is to ensure the continuity of the flame, by automatically regulating the feeding of the alcohol into the vaporisers by hydrostatic pressure. A disadvantage of this arrangement is that it involves the dismantling of the upper part of the lamp when it is necessary to change the lining of the vaporisers N N. A small model is also made which consumes 56 c.c. of alcohol with a light of 30 bougies.

The use of petroleum lamps in projecting lanterns presents many inconveniences, of which all public lecturers are aware. In order to avoid the bad smell and the smoke, alcohol lamps have been invented for use in halls not provided either with gas or electricity. These alcohol lamps are fitted with an Auer mantle, rendered incandescent by a mixture of the vapour and air. The chief defect of these mantles is their great fragility. The conical form of the mantle, moreover, causes a great portion of the light produced to be lost; in fact, more than half the luminous rays emitted by the incandescent tissue does not fall on the lens of the condenser: they are therefore not utilised. The substitution of an incandescent sheet for an incandescent mantle obviates this, and has also another advantage, the price of six sheets is almost the same as that of a single mantle, and the lifetime of one is at least equal to that of the other. Henceforth the lecturer will not be interrupted by frequent renewal of the fragile tissue, the expense becoming insignificant and the manipulation more simple. There is no danger in working the apparatus. The capacity of the reservoir R (Fig. 83) has been calculated so as to feed the lamp in a continuous manner and with maximum intensity for at least two hours. Ordinary methylated spirit is run in through the opening covered by the screw stopper *b*, which is fitted with a pipe from an indiarubber bulb. By capillarity of the wick in the tube T, the alcohol from the reservoir R rises in A, where it is volatilised, first by the heat of the combustion of a little alcohol previously poured into the cup *c*, and afterwards by the flame of the jet B, fed by the vapour produced in A, led by the tube *t*, and mixed with the air entering by the orifice O. The air compressed by the indiarubber bulb cannot pass by the tube *t*. It simply forces the ascension of the alcohol up the wick. It will

be seen that no communication is possible between the flame of the jet B and the alcohol of the reservoir R ; if the stopper *b* is screwed in its place, the mixture of air and alcohol vapour contained in B cannot become inflamed nor any explosion occur. If the alcohol be made to ascend too rapidly in A, owing to too strong pressure of the indiarubber bulb, volatilisation may be incomplete, and a few drops of alcohol may become entrained into the tube *t*, producing a cracking sound in O, where they become inflamed.

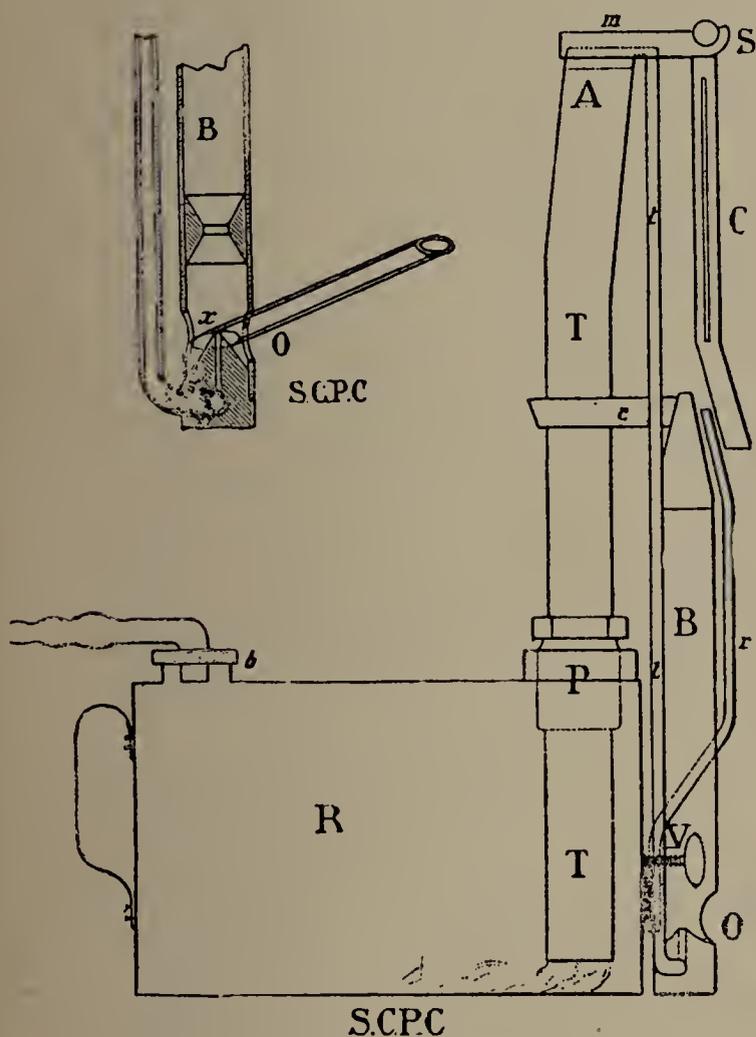


FIG. 83.

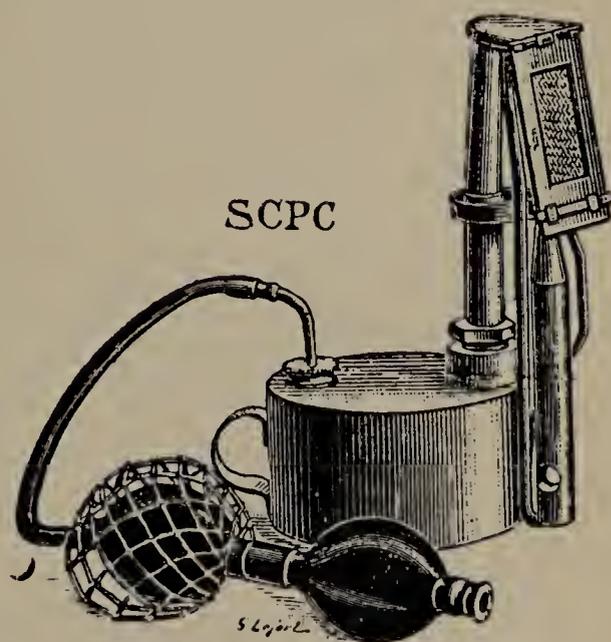


FIG. 84.

Alcohol vapour incandescent lamps, for lantern demonstrations, with flat incandescent film instead of conical mantle. Fig. 84, general view of lamp, Fig. 83, section. The top section to the left shows the method of extinguishing.

They may even be projected by the jet B on to the tissue of the frame C, thus damaging it. This is avoided by pressing the indiarubber bulb gently. It is detached from the stopper *b* at the first crack ; besides, it must not be adjusted to the tube of the stopper until the alcohol in the cup is burnt out and the flame of the jet B has become regular, which occurs in two or three minutes. If the profile of this flame be examined, it appears to

form a very regular blue sheet, one side of which impinges on the tissue placed in the frame C, and the upper extremity of which heats strongly the metallic piece *m*, and consequently the space A, where the vapour of alcohol is formed. To produce the maximum luminosity, the flame should occupy a determined place neither too near nor too far away from the tissue to be rendered incandescent. This position is easily found by manipulating the screw V, by which the frame C may be moved nearer to or further from the flame. The frame C is easily removed, opened, shut, replaced, or hung on S without difficulty. The sunk part of the frame C is intended to receive one of the sheets of collodion drawn from a case containing a small supply, and which can be despatched by post. It is useless to inflame the tissue before lighting the lamp; this operation is done only by the flame rising from the cup *c* or issuing from the tube B. To extinguish the lamp, all that is required is to prevent the current of alcohol vapour from ascending to the jet B. To do this the extremity *x* of a glass tube, or a tube made out of a goose-quill or a toothpick, is kept for a few seconds on the orifice O. On no account must the flame be blown out: blowing on the jet B, the tissue of the frame would fly away as dust. This tissue may be used on several occasions when the lantern has not to be transported to another locality. *Method of working.*—1. Regulate the height of the tube T. This tube slides in a stuffing-box P. It must be raised or lowered so that the centre of the empty rectangle of the frame C is at the same height as the centre of the condensor. This operation is done once for all for the same apparatus. 2. Almost completely fill the reservoir B with ordinary methylated spirit, rescrew the stopper *b* tightly. On the first occasion, this should be done half an hour beforehand; it is necessary for the wick in the tube T to become completely soaked. 3. Fill the frame C and fix in position. 4. Three-quarters fill the cup *c* with alcohol, inflame, and wait the finish of the combustion. 5. Moisten the end of the tube of the indiarubber bulb, adjust it to *b*, and inflate it moderately. 6. Regulate the screw V. 7. Place the lamp in the lantern so as to give the maximum of brilliancy to the disc projected on the screen. 8. Slide and adjust the mantle frame.

*Alcohol heating apparatus.*—Polo's alcohol vapour furnace (Figs. 85, 86) is a type of this class of stove. Other forms differ only in detail. It consists of a cylindrical reservoir, on the peri-

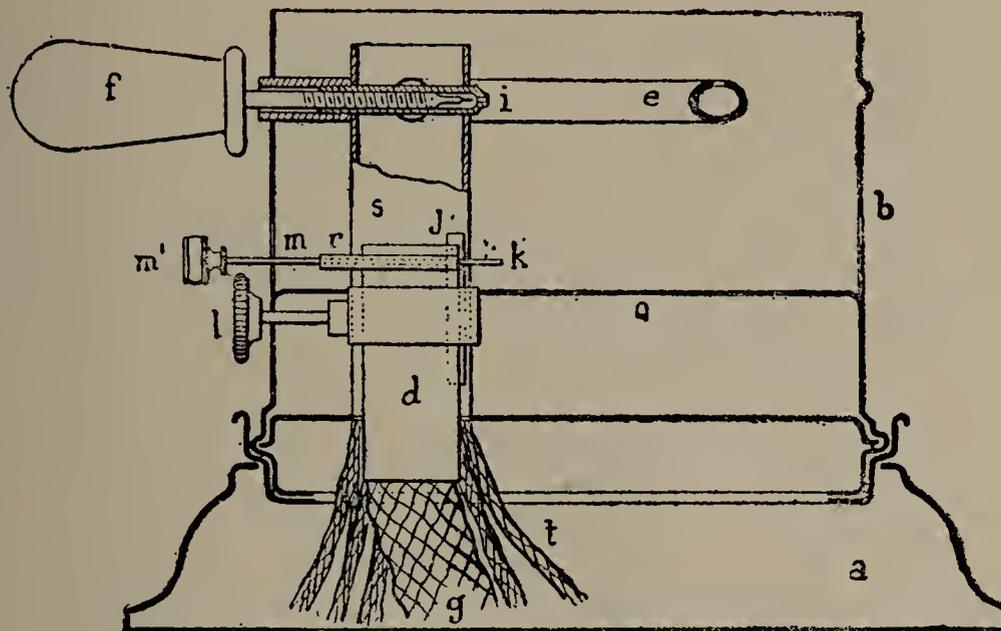


FIG. 85.—Polo's alcohol vapour heater (section).

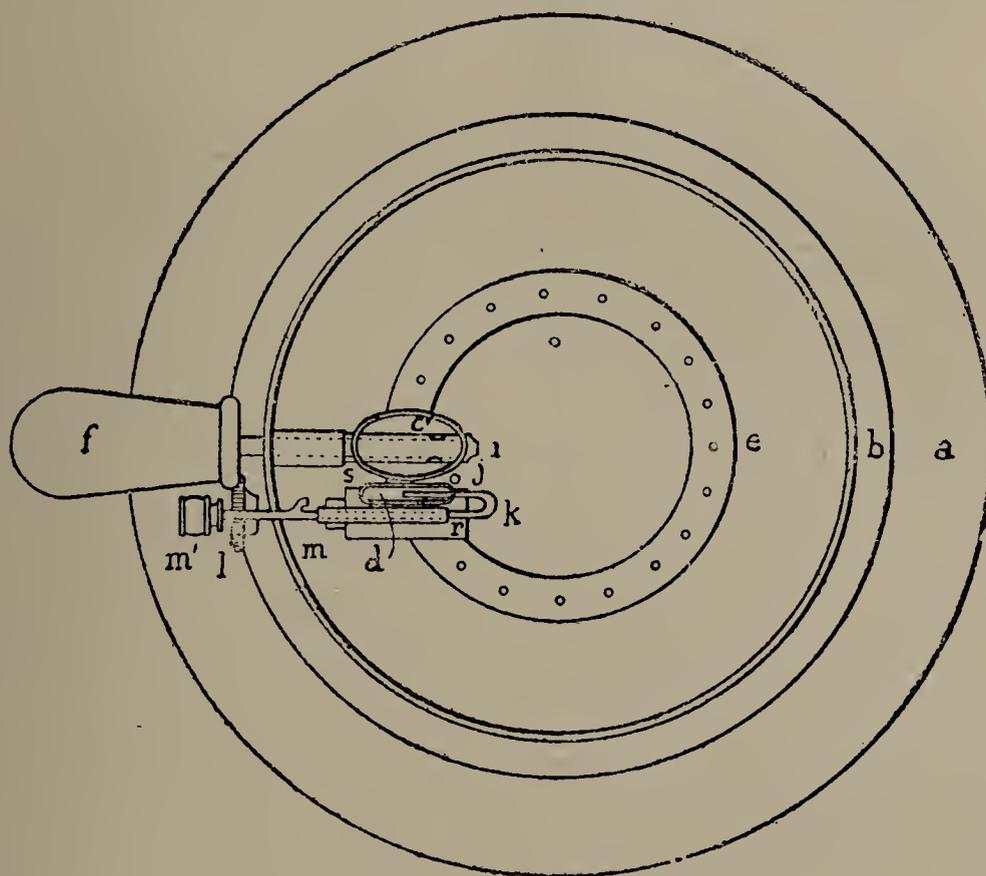


FIG. 86.—Ground plan.

phery of which is mounted a flat brass socket *s*, containing an asbestos wick *g*, fed by a cotton wick *t*, and fitted in its upper part with an injector *l*, directed towards the centre of the furnace and regulated by *f*. On heating the upper part of the wick, the

alcohol therein evaporates and escapes through the injector in the form of vapour. This jet is received in a brass tube S, arranged almost horizontally so as to form a Bunsen burner feeding a perforated ring which touches the flat brass socket *s*, already referred to, on both its flat sides. The furnace is lighted by heating the extremity of the wick *g* by means of a small jet *j* placed against the socket, which is extinguished as soon as the ring burner is lit. The requisite evaporation heat is maintained by two jets of flame in the ring itself directed against the flat socket. The burner is regulated by turning the handle *f*, the

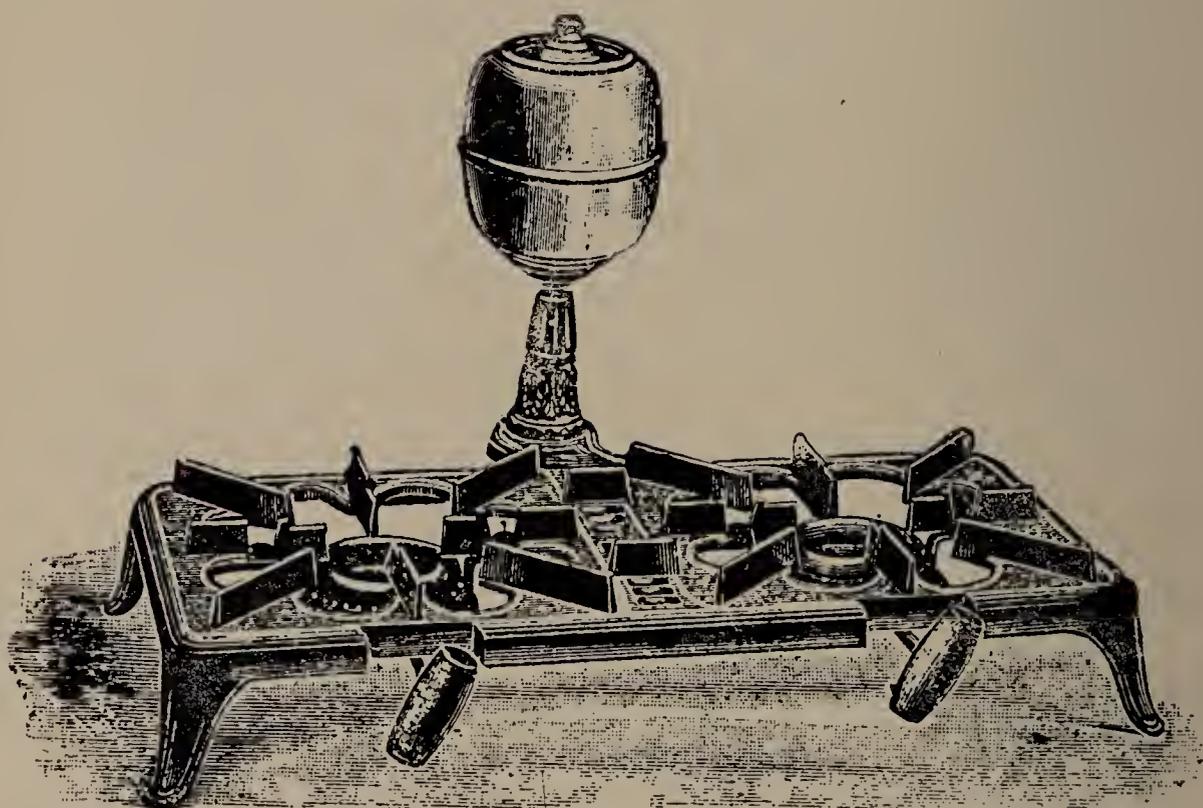


FIG. 87.—Twin alcohol vapour heater, for cooking and other purposes.

vaporisation of the alcohol depending on the size of the orifice of this tap and the height of the flame coming from it. According to Lindet, all these heaters, with or without wicks, with simple vaporisation and admixture with air, consume in bringing a litre of water to boiling-point almost the same amount of methylated spirit, viz., 30-35 grammes of 90 per cent. methylated spirit. The time required to do this, however, varies in different heaters, depending on the power of the furnace. In any case, vaporisation heaters, especially those in which the alcohol vapour is mixed with air, are better to regulate and produce a flame easily directed under the vessel which it is desired to heat.

Amongst sundry appliances heated by alcohol may be enumerated—soldering and brazing blowpipes, iron heaters, plate-heaters, frizzing and curling tongs, stoves for living-rooms, bedrooms, greenhouses, camp stoves, etc. Fouillard's brazing and soldering blow-pipe (Fig. 88) is especially useful when it is desired to produce a very high temperature quickly, as in brazing or soldering operations very often obligatory in the case of agricultural machines and implements. The principle of all these blow-pipes or brazing-lamps is the same, they consist essentially of a Bunsen burner fed by 50 per cent. carburetted alcohol from

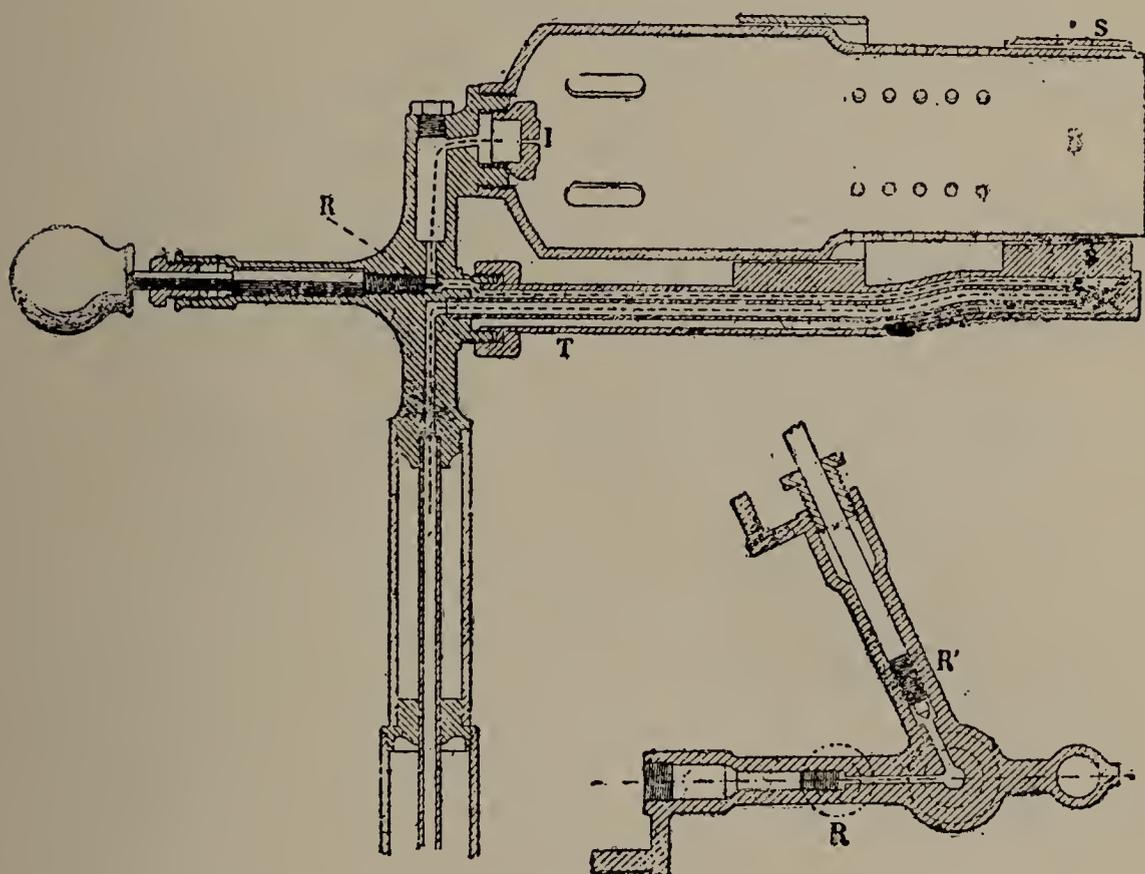


FIG. 88.—Fouillard's alcohol vapour soldering blowpipe.

a reservoir under a pressure from a hand-pump of 1-3 kilogrammes. The vaporisation of the alcohol is brought about by a preliminary heating, then by a special piece of metal to recuperate the waste heat, a nozzle tap regulates the intensity of the flame, which rapidly becomes very hot. The reservoir may be either independent or self-contained. Room and carriage stoves are made of many sizes and designs.

*Alcohol motors.*—The widespread use of motors on the internal combustion principle is due to the undoubted advantages which they possess over steam-driven engines, more especially in the

case of machinery which is driven intermittently. The four-cycle type of engine characteristic of all explosive motors was invented in 1862 by Beau de Rochas, but it is more generally known as the Otto cycle, *i.e.*, (1) aspiration of the explosive mixture ; (2) compression of this mixture ; (3) inflammation, explosion, and expansion producing the propelling force ; (4) expulsion of the burnt gases. 1. In the first forward movement of the piston the air carburetted by the gaseous or liquid fuel is aspirated, and the return backward stroke of the piston compresses the explosive

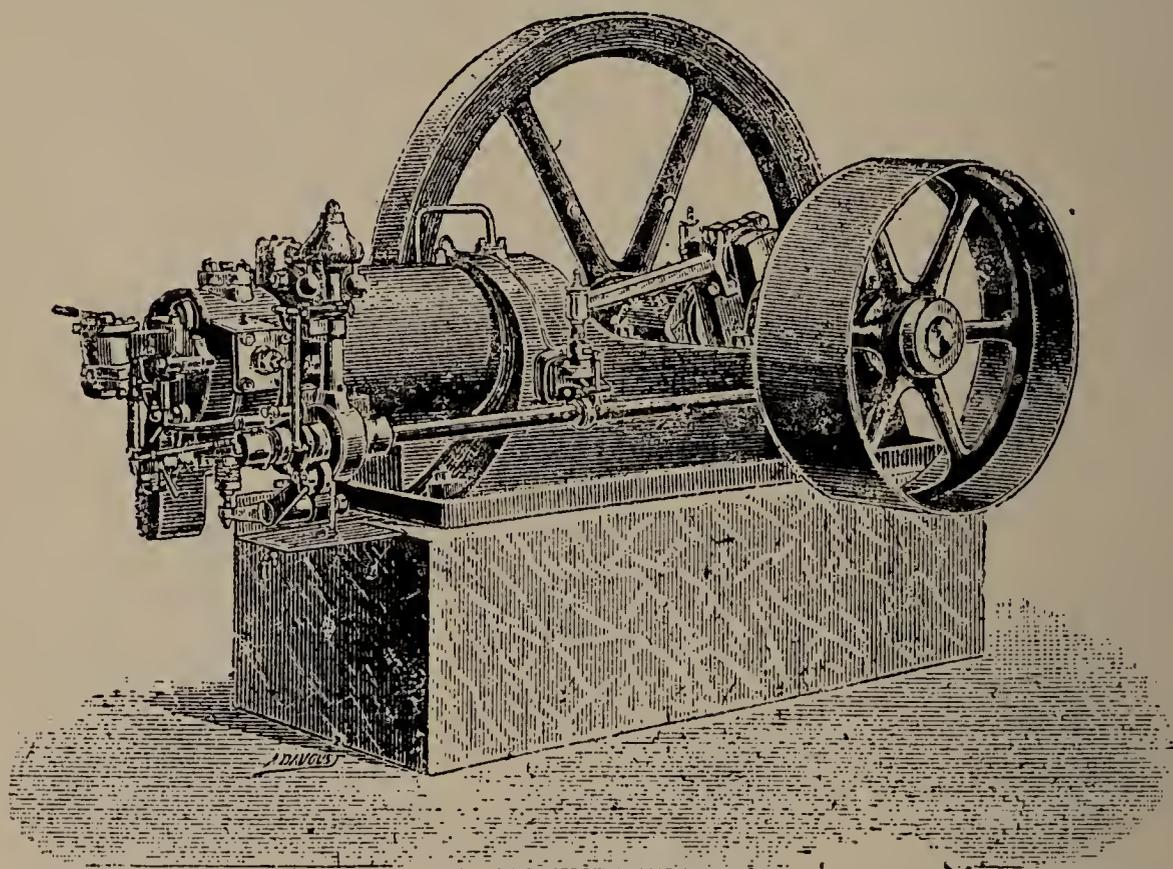


FIG. 89.—Alcohol vapour motor engine, Charon type. (There are also vertical engines of the same type.)

mixture. At the end of that course then the mixture is ignited by a special arrangement which explodes the gas. As a result of the expansion the piston is driven forward to do useful work, returning afterwards to drive out the spent gases produced by the combustion. Of the four motions of the piston in its cycle—*aspiration*, *compression*, *expansion*, and *escape*—only one of these does useful work, the three others result from the revolution of the fly-wheel. The aspiration and compression phases are reproduced every two turns of the fly-wheel, the entrance and escape valves are worked by cams fixed on a shaft revolving half as fast

as the fly-wheel, and connected to the latter by suitable gearing. Gas and petrol motors on the four-cycle principle have been in use for some time, but alcohol-driven motors of this type are more recent. One reason for this is that the calorific intensity of petrol is 10,000 calories, whilst methylated spirit, even slightly

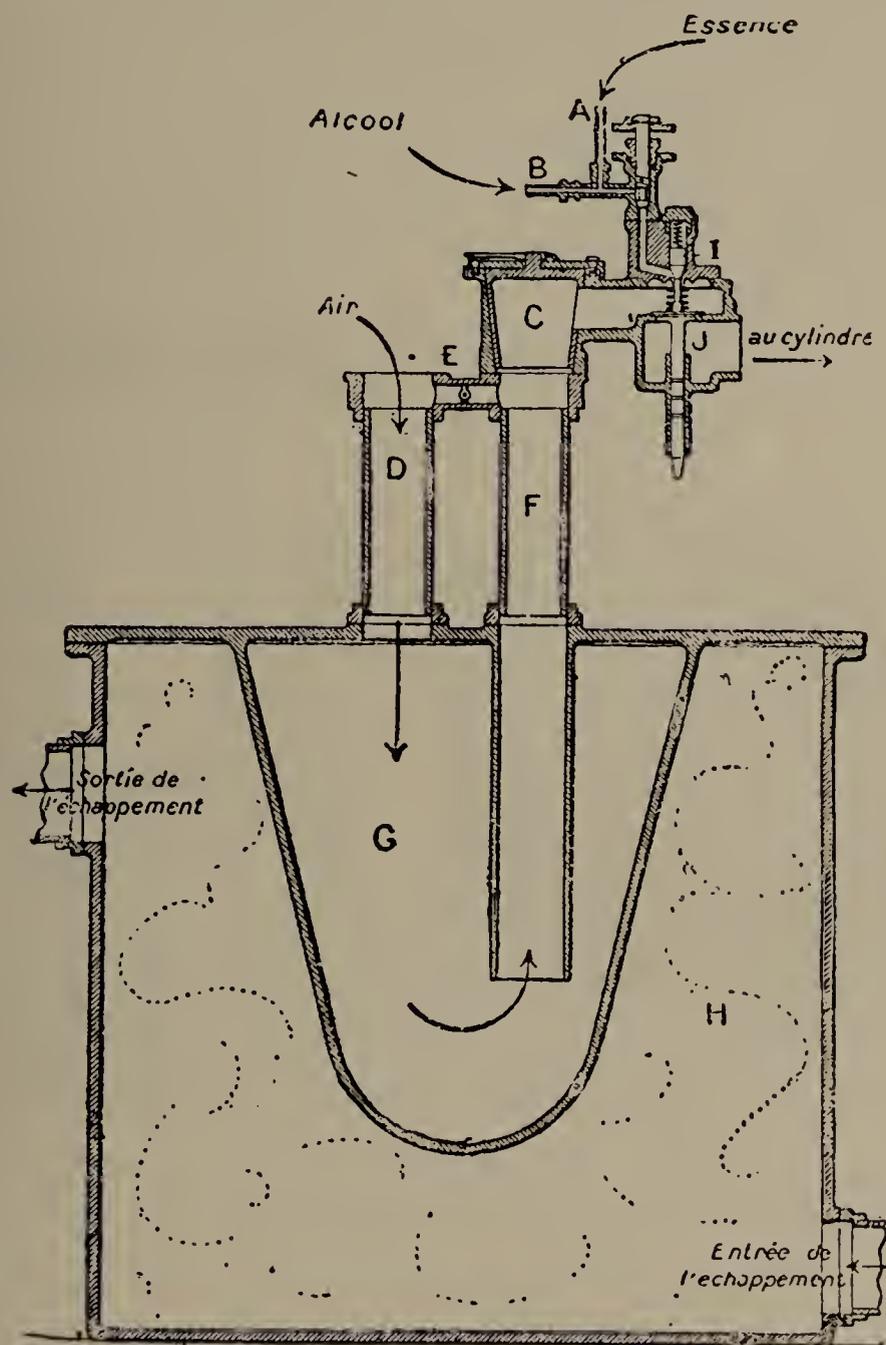


FIG. 90.—Section of feeding and escape arrangements of type of alcohol engine shown in Fig. 89. A, entrance of petrol for starting; B, entrance of alcohol; C, hot air valve; D, cold air entrance; E, temperature regulator; F, hot air entrance pipe; G, air heating chamber; H, escape gas chamber; I, liquid feed valve; J, feed and atomiser valve.

carburetted, has a calorific intensity of only 6000 calories; hence it was deduced theoretically that petrol was more economical than alcohol. Hence, in searching for new outlets for industrial alcohol, the domain of domestic heating and lighting was first explored and exploited. But in that domain the question of

economy and expense is often a secondary one to that of hygiene and general comfort. The first attempts to substitute alcohol for petrol for generating power were made in a petrol engine constructed by Grob of Leipsic, capable of working also with alcohol. Hartmann reported on the relative advantages of alcohol: combustion was more complete and the products were inodorous, but it consumed 839 grammes of alcohol against 426 grammes of petroleum per horse-power hour. More recently, trials were made at the Berlin Fermentation Institute under the auspices of the German Government. The alcohol used contained

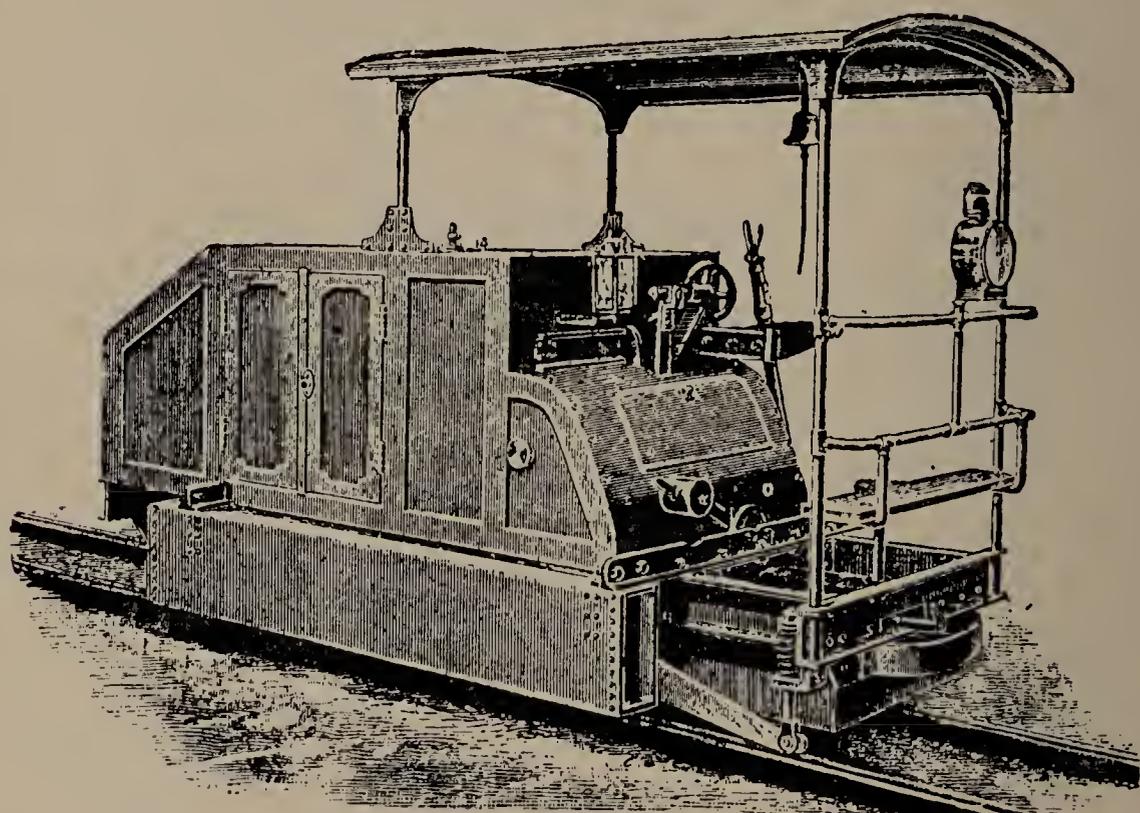


FIG. 91.—Alcohol engine in use on light railways.

85-90 per cent. by weight, say, 89.5-93.5 G.L., *i.e.*, the alcohol which the German distillers produce directly in their own distilleries. Slaby, of the Charlottenburg Polytechnic, about the same time tested a five horse-power motor fitted with a Petreano evaporator, and obtained per horse-power hour a consumption of 550 grammes of alcohol of 86.2 per cent. (90.4 G.L.), say, an efficiency of 24.6 per cent. In March, 1897, Haak tested a six horse-power Koerting system petroleum spirit motor internally fitted up for the use of alcohol, which generated a force of 9.93 horse-power with a consumption per horse-power hour of 390 grammes of alcohol of 93 per cent. by weight (94.4 G.L.). The

engine tested was fitted with a vaporisation chamber intercalated between the fuel atomiser and the entrance valve, a vaporiser heated by the escape gases in direct communication with the alcohol and air valves. After these experiments an alcohol engine was erected in the Berlin Fermentation Institute, which was used for many subsequent tests. Carburation by benzol and its effect on the horse-power was first studied. The results obtained by Goolich with 86 per cent. alcohol by weight (90.2 G.L.), with 5, 10, 15, 20, 25, and 30 per cent. of benzol by weight, are given in the following table. The reason for studying the effect of benzol was connected with its use as a denaturant. The price of benzol at 16s. the hectolitre, or 9d. per gallon, is less than that of the general German denaturant 2 per cent. of wood spirit and  $\frac{1}{2}$  per cent. of pyridine bases.

TABLE XXXII.—COMPARATIVE EFFICIENCY OF CARBURETTED AND PURE ALCOHOL AS MOTOR POWER GENERATORS (GOOLICH).

Benzol Per Cent. by Weight.	H.P.	Consumption Per Horse-power hour.	Economy in Fuel compared with Pure Alcohol.	Benzol Per Cent. by Weight.	H.P.	Consumption Per Horse-power hour.	Economy in Fuel compared with Pure Alcohol.
0	8.809	420	0	20	8.819	370	12.0
5	8.862	405	3.6	25	8.887	386	8.0
10	8.888	386.5	8.0	30	8.880	390.5	7.0
15	8.867	381.5	9.0				

The inference from the above results is that 20 per cent. carburation gives the maximum efficiency. Meyer (Charlottenburg Polytechnic) made analogous tests with an alcohol-driven Otto locomobile of 14 horse-power, his results, given in Table XXXIII., confirming those of Goolich.

The first alcohol-driven locomobile (of Oberursel construction) of 15 horse-power was tested in February, 1900, 21.8 horse-power being generated with a consumption of 410 grammes of 88 per

TABLE XXXIII.—COMPARATIVE EFFICIENCY OF PURE AND CARBURETTED ALCOHOL AS MOTOR POWER GENERATORS.

Load.	Alcohol (Pure) 86.7 Per Cent. by Weight 90.7 G.L.		With 9.12 Per Cent. Benzol.		With 14.3 Per Cent. Benzol.		With 20.95 Per Cent. Benzol.	
	H.P.	Consumption of Pure Alcohol Per Horse-power hour.	H.P.	Consumption of Pure Alcohol Per Horse-power hour.	H.P.	Consumption of Pure Alcohol Per Horse-power hour.	H.P.	Consumption of Pure Alcohol Per Horse-power hour.
Full .	16.14	445	16.00	422 gr.	15.90	388 gr.	16.09	375 gr.
Normal	13.78	463	14.07	433 „	14.09	412 „	14.18	385 „

cent. alcohol carburetted with benzol in the proportion of 20 per cent. The above and similar tests prove that alcohol, either pure or carburetted, can be advantageously and economically employed as a motive power. The combustion of carburetted alcohol is perfect, and the work is comparatively more cleanly and less dangerous than with petroleum spirit. The consumption of carburetted alcohol varies from 360 to 420 grammes per horse-power hour. Alcohol motors compared with petroleum spirit motors do 25 per cent. more work. There is no difficulty in starting alcohol motors. Owing to the composition of alcohol and the injurious products incidental to its incomplete combustion, it is necessary that both vaporisation and combustion be complete. The piston rings and the valves of the alcohol-driven engine of the Berlin Fermentation Institute, after working three consecutive years, were found, when it was dismantled in 1903 for inspection, to be in excellent condition. To find the practical result, *i.e.*, the work accomplished compared with the calories contained in the liquid fuel, it must be borne in mind that alcohol carburetted to the extent of 20 per cent., *i.e.*, 80 litres of 90 per cent. alcohol and 20 litres of benzol, has a density of 0.844 and a calorific intensity of 6.633 calories per kilogramme, or 5.598 calories per litre; the

mechanical equivalent of heat being 427 kilogrammes, the complete combustion of 1 kilo. of this alcohol producing 6.633 calories

will give  $\frac{6.633 \times 427}{3.600 \times 75} = 10.49$  horse-power. But in actual practice

1 horse-power consumes 400 grammes of alcohol, or 2.5

horse-power for 1 kilo of alcohol, hence  $\frac{25 \times 100}{10.49} = 23.8$  per cent.

efficiency. This result is certainly very high, since the best steam engines give an efficiency of only 13 per cent. According to Musil, petroleum spirit gives 14 to 18; petroleum (lamp oil), 13; steam, 13; gas, 18 to 31; alcohol on an average, 24.8. This demonstrates the greater value of the alcohol-driven engine not only over a steam-driven engine, but over that driven by any other liquid fuel. This comparison is, of course, with intermittent steam-driven engines that require heating to get up steam before they can be used. There is no comparison with a stationary engine working continuously, against which the alcohol-driven engine does not compete. We shall soon see that still better results have been obtained with alcohol-driven engines. But before going further, it may be of interest to explain how the efficiency of an alcohol-driven motor is tested.

*Testing the working capacity of an alcohol motor.*—The following is an abstract of a report made by Perisse on an alcohol motor (*i.e.*, engine) of the Charon (Economique) type. The brake used in testing acted on the plate of a bascule through the intermediary of a vertical rod fixed to the extremity of the lever of the brake. The bascule being balanced, the effective work in horse-power units was ascertained by the formula :

$$\frac{P}{60 \times 75} = 0.001396 P_n L,$$

in which L represents the length of the lever; P, the weight indicated by the index of the bascule diminished by the tare of the brake, the actual weight of the rod which acts on the plate and arm of the lever; number of revolutions per minute, *n*.

L = 1.0 metre. Tare of brake, 5 kilogrammes.

Density of the 50% carburetted alcohol, Electrine Leprêtre 0.839

„ „ 8% „ „ „ „ „ 0.840

„ „ pure alcohol, Moto-Schnick Leprêtre 0.838

The horizontal motor type Economique, No. 3002, of 15-18 horse-power, specially designed for alcohol, had the following characteristics :

Course, 360 mm.	Capacity of compression chamber, 2.65 litres.
Diameter, 230 mm.	Height of reservoir, 1.95 metre.
Ratio of course	Diameter of flywheel, 1.9 metre.
to diameter, 1.56.	Width of fly-wheel, 130 mm.
Speed, 200 revolutions.	Diameter of brake pulley, 0.50 metre.

Function and regularity were excellent. Starting was effected without any difficulty with carburetted alcohol and with pure alcohol. The distribution pipes were tested several times, either after running several hours or after several days without cleaning, and neither oxidation nor encrustation were found. The general results are embodied in the table on opposite page.

There are two points to be considered in changing from a petrol-driven motor. There is, first, the carburettor, and, second, the increased compression. The full advantage is not obtained from the alcohol without an increased compression ; that requires an exceedingly simple alteration, merely placing on the end of the piston or cylinder head a blank to fill up so much space. There would, therefore, be no serious difficulty in adapting the present petrol engine to work with alcohol. In France, petrol cars have been adapted for alcohol by merely altering the carburettor ; the modern petrol engine has a very much higher compression than such engines used to have. In any case, there is no difficulty in adapting the carburettor, for the simple reason that petroleum itself can be used in some of the carburettors that are now commonly running. The Cremorne carburettor works quite well with petroleum. Such efficiency is now obtainable with agricultural motors that the volume of alcohol required would be practically identical with that of petrol. If high-speed motor engines can be built with the same degree of efficiency, a motor car may be built which will run as many miles on a gallon of alcohol as it will on a gallon of petrol. Such a result has been

TABLE XXXIV.—ACTUAL TESTS MADE BY PERISSE TO DETERMINE THE EFFICIENCY AND CAPACITY OF AN ALCOHOL-DRIVEN ENGINE, ILLUSTRATING THE VARIOUS POINTS TO BE DETERMINED AND SCHEDULED.

	A	B	C	D	E	F	G	H	I	J	K
50% carburetted											
Empty . . . . .	208	20	—	1.000	3.000	—	—	—	—	—	2.517
Half load . . . . .	205	20	84	1.450	4.350	35	30	8.58	0.507	0.425	—
Load . . . . .	203	20	—	1.850	5.550	—	50	14.16	0.391	0.328	—
Full load . . . . .	205	10	—	1.160	6.960	—	65	17.68	0.393	0.330	—
80% carburetted—											
Empty . . . . .	207	30	—	1.675	3.350	—	—	—	—	—	2.814
Half load . . . . .	205	16	85	1.237	4.640	—	30	8.58	0.540	0.453	—
Full load . . . . .	199	10	—	1.062	6.372	—	60	16.62	0.383	0.322	—
Pure alcohol . . . . .											
Empty . . . . .	213	10	40	0.825	4.950	—	—	—	—	—	4.148
Half load . . . . .	200	10	—	1.075	6.450	—	35	9.77	0.660	0.553	—
Full load . . . . .	200	20	99	2	6	—	52	14.51	0.413	0.346	—
” 50% carburetted	—	—	90	—	—	—	—	14.65	0.392	0.329	—

A, average number of revolutions per minute.  
 B, duration of test in minutes.  
 C, number of explosions per minute.  
 D, consumption in litres of alcoholic liquid during test.  
 E, hourly consumption in litres.  
 F, weight registered by the balance.  
 G, net load on the brake in kilogrammes.  
 H, horse-power developed.  
 I, consumption in litres per horse-power hour.  
 J, consumption in kilogrammes per horse-power hour.  
 K, consumption per hour in kilogrammes.

obtained by perhaps four or five firms. A few years' practice might possibly lead to a greater mileage with alcohol than with petrol. A larger number of calories can be got from a given cylinder capacity with alcohol than with petrol. It would be

safer to back an alcohol motor racer specially designed for alcohol than a petrol motor, as a larger number of calories in a given cylinder capacity can be converted into work in an alcohol engine than in a petrol engine. This is important, as every motor car builder desires to turn out an engine which for its size and weight will give the largest possible output of work, and their efforts go more in that way than towards an absolute efficiency in consumption. For tropical countries there is no doubt that alcohol would be of enormous advantage, because the volatility and explosive risk of petrol is great, and there are many objections to the use of paraffin oil. There is, of course, no reason why petrol should not be used in hot countries as long as care is taken. The Indian Government have relaxed their restrictions very largely; and allow petrol to be used in India where it was not allowed to be used eight or ten years ago; but it is a fact that, unless considerable precautions are taken, the mileage on petrol, registered from one week's end to another in India, generally comes out very low, because of the evaporation. For this reason motor riding is distinctly a pastime for rich people in India. Therefore, from that point of view alone, if this country is to build motor cars for the colonies, it is even now desirable that we should be experimenting with alcohol engines. The mere fact, so encouraging to know, that it is largely used in Egypt in British-made alcohol engines is a sign of the times, and shows that it is a desirable fuel in hot countries. We may take it that the German and other owners of agricultural alcohol engines are interested in obtaining alcohol at as cheap a price as possible; probably similar agricultural engines would become largely used in this country if alcohol were obtainable at the same price. The conditions are, however, somewhat different in this country to those in Germany, since coal-gas is so widely used here. There is no place of any reasonable size in this country but has a coal-gas supply, or where coal is not easily available for motive power.

If we analyse the results of alcohol-driven engines given in engineering literature, and the reports as to the appearance of the machinery after it has been used for some time with alcohol, one of the most serious objections is the extraordinary corrosion

which, it is alleged, in some cases takes place. When there is a very high degree of corrosion, alcohol-driven engines being seriously damaged in that way, the fault is to be attributed either to badly rectified alcohol, on the one hand, or imperfect combustion on the other.

The use of alcohol for motors in Germany, in spite of the advantages mentioned, is not yet very extensive, but the reason is not far to seek. The agencies for the distribution of motor alcohol are not so complete as the distributing facilities for petrol, since petrol is sold through all the agencies instrumental in distributing ordinary paraffin oil, they being largely controlled by the same people. Again, it would not pay the big motor manufacturing firms to put down plant and make templates for machines specially designed to run with alcohol, which would only be sold in Germany, where the alcohol is comparatively easily obtainable, and which would be useless outside the boundary of their own country. These cars would be useless here, because if built to work to the best advantage with the aid of alcohol, they would work at such high compression that they could not be used with petrol at all. The use of alcohol must become pretty general in various countries because these big manufacturers, who look to a European sale and not merely to one in their own country, will not go to the expense of building motors for use in one country only. In addition to locomobiles, alcohol-driven engines are used for driving light machinery, spinning and weaving machinery, wood-working machinery, driving dynamos for electric light, pumping, and all sorts of light machinery work. Several engines of this type constructed in Paris are actually at work in different continental countries.

*Motor fuels.*—The report of the Fuels Committee of the Motor Union of Great Britain and Ireland, published in July, 1907, contains a considerable amount of information on present and prospective sources of motor fuels. The evidence collected by this Committee was to the effect that the mineralised methylated spirit in common use in this country is in no way unsuitable or detrimental for such purpose.

Alcohol has one great advantage over benzol and petrol, that

the raw material can be raised annually and in almost unlimited amount. The only question to be considered is that of price. At that time alcohol of 64 O.P. strength could be produced at 11½d. per gallon, but the cost of denaturing and supervision brought the cost up to 2s. Petrol was then half that price, and there could be no question of competition.

Section 30 of the Report reads : “ Any question, therefore, of the use of spirit for motor vehicles will be one of price, and as the present price of petrol is about half the price of methylated spirit, we think that close investigation of the matter may be delayed until such time as there may be an approximation between the price of petrol and spirit sufficient to create a practical alternative of choice between the two.”

It has been stated that alcohol could be produced from peat at 3d. per gallon, and if that were the case the problem would be solved, but this estimate has not been confirmed.

With regard to denaturing, the report says that a denaturant which will satisfy the requirements of the Excise authorities, and also those of the motorist, should possess the following characters :

1. The substance used should add to the energy latent in the fuel (Holden).

2. In any case, the denaturing substance should pass away with the products of combustion, and must not leave any objectionable residue that might corrode or soot up the passages (Holden).

3. Acetone (in methyl alcohol) should be absent, as being the source of acetic acid if combustion is incomplete (Ormandy).

4. Benzol is an advantage, 2·5 per cent. preferably, to replace some of the methyl alcohol (Ormandy).

5. The addition of a minute quantity of methyl alcohol is a very considerable additional security against improper use (Ormandy).

6. The cost of separating out ethyl alcohol from the denatured spirit should exceed the price of potable spirit as sold in this country.

Comparing alcohol with petrol as a motor fuel, the report enumerates the following advantages in favour of the former.

*Safety.*—In case of conflagration the flame of alcohol can be extinguished with water, but that is not the case with petrol, which is liable to spurt in flame in all directions. The flash-point of alcohol is higher (*i.e.*, 60° C.) than that of petrol (as low as 10° C.), and hence storage of alcohol is the safer.

*Thermal efficiency.*—The thermal efficiency of alcohol is much higher than that of petrol, due to the fact that a much smaller volume of air is required for complete combustion, thus the thermal efficiency of alcohol may be as high as 35 per cent. (28 per cent.<sup>1</sup>), whereas that of petrol is below 20 per cent. (16·5 per cent.<sup>1</sup>).

*Calorific value.*—The calorific value of alcohol is lower than that of petrol. Absolute alcohol gives 12,600 B.Th.U. ; methyl alcohol (sp. gr., 0·820), 11,300 ; petrol (sp. gr., 0·722), 19,300 to 20,300. Other determinations may be quoted here: Petrol (sp. gr., 0·684), 20,923 B.Th.U. ; methylated spirit, 11,160<sup>1</sup> ; methylated alcohol, 10,620 B.Th.U. ; methylated alcohol with 50 per cent. of petrol, 14,200 ; crude American oil, 19,630 ; refined American oil, 19,880.<sup>2</sup>

*Practical limit of compression.*—The practical limit of compression with alcohol is about 200 lb. per square inch, as compared with 80 lb. per square inch for petrol. The explosion pressure of alcohol is therefore much higher than with petrol and its efficiency correspondingly greater.

*Complete combustion.*—Alcohol is much more efficiently burnt than petrol, this being due to the smaller range in the distillation temperatures, *e.g.*, 80° to 100° C., whereas petrol has a much larger boiling range at all temperatures between 50° and 150° C., allowing in the case of alcohol the degree of carburation to be more accurately adjusted.

*Propagation of flame.*—In the case of alcohol the propagation of flame is much slower than with petrol, the pressure on the piston being more uniform throughout its stroke.

*Odour.*—When alcohol is employed there is practically no smell in the exhaust.

<sup>1</sup> *Times Eng. Supp.*, Jan. 15, 1913.

<sup>2</sup> *Brewer's Jour.*, 1905, 41, 681.

*Flexibility.*—Alcohol vapour mixed with air will explode when the percentage of the former varies between the limits 4 and 13 ; petrol vapour, on the other hand, has a much lower range, from 2 to 5 per cent. This allows more flexibility in the engine running on alcohol.

The defects of alcohol are :—

*Corrosive effect.*—The general opinion is that pure alcohol shows no corrosive effect on the cylinder or tubes, and that when this is observed it is due to impurities in the denaturing agent employed. This difficulty can, however, be overcome by adjusting the carburation so as to ensure complete combustion.

*Starting cold.*—Some difficulty is encountered when starting cold with alcohol, but if a proportion of benzol be mixed with the spirit, which will no doubt be a common practice, this drawback will disappear. Improvements in the carburettor will also probably solve the difficulty.

*Vaporisation.*—Alcohol requires a certain amount of heat to vaporise it, estimated at  $5\frac{1}{2}$  per cent. of the total heat of combustion, whereas petrol will burn without any external aid. Since, however, much heat is lost in the exhaust, this could be employed in a properly constructed engine for vaporising the alcohol without interfering with its energy producing powers.

The use of mixtures of alcohol and benzol, or alcohol and petrol, for motor purposes appears to be a distinct advantage, the vapour pressures of such mixtures being much lower than that of alcohol alone. Benzol will mix in all proportions with commercial alcohol, but petrol does not mix so readily, and if 5 per cent. or more water be present in the alcohol the mixture separates into two layers. The composition of the mixture may vary within wide limits. J. Novel<sup>1</sup> states that the best mixture consists of alcohol 80 parts, acetone 10 parts, and petrol 10 parts. Such a mixture, it is claimed, can be employed in the present types of internal combustion engines without any alteration to the carburettor, the engine starting readily from the cold, even at as low as 10° C.

<sup>1</sup> *French Pat.*, 380, 310, July 27, 1907.

W. Hempel<sup>1</sup> gives the following as the respective heats of combustion of motor fuels: Benzine (petrol), 9500-10,500; pure benzene, 10,260; commercial 90 per cent. benzol, 9550-10,000; pure alcohol, 7402; 95 per cent. alcohol, 5875; and pure naphthalene, 9628.3 calories per kilo. He informs us that in Berlin all motors have been adapted to burn alcohol. He expresses the opinion that alcohol denatured with 2 to 20 per cent. of benzol is much more suitable as a fuel for motor engines than the usual denatured alcohol containing wood spirit or pyridine.

K. Dietrich<sup>2</sup> suggests quite a number of mixtures as suitable for use as motor fuels. These are given in the following table:

TABLE XXXV.—MOTOR FUELS.

	1	2	3	4	5	6	7	8	9	10	11
Alcohol (95 per cent.) .	70	—	70	—	90	90	70	—	—	—	—
Alcohol (90 per cent.) .	—	50	—	50	—	—	—	50	—	—	—
Benzol . . . . .	30	30	—	—	—	—	—	—	—	—	—
Commercial acetone .	—	20	—	20	—	—	30	50	—	1	—
Benzine . . . . .	—	—	30	30	—	—	—	—	1	—	—
Ether . . . . .	—	—	—	—	10	10	—	—	—	—	10
Naphthalene . . . . .	—	—	—	—	—	1	—	—	—	—	1
Petroleum . . . . .	—	—	—	—	—	—	—	—	2	3	90

According to Dietrich, all these mixtures require preliminary heating. Alcohol is liable to produce rust in the engine, but this can be obviated by adding 1 litre of motor oil to 100 litres of any fuel containing it.

Dietrich also states that the following mixtures could be satisfactorily used in a Lyma carburettor after suitable regulation of the tubes: (1) Alcohol (95 per cent.), 4 volumes; 90 per cent. benzol, 1 volume. (2) Alcohol (95 per cent.), 4 volumes; benzol, 1 volume; 200 grammes naphthalene being dissolved per litre in the latter. (3) Alcohol (95 per cent.), 4 volumes; light coal tar oil, 1 volume, also containing 200 grammes of naphthalene per litre. In a long trial run with No. 1 mixture, 18 litres were

<sup>1</sup> *Z. angew. Chem.*, 1914, 27, 521-522.

<sup>2</sup> *Ibid.*, 1914, 27, 543-544.

found equal to 15 litres of ordinary petrol. The use of a solution of acetylene in acetone or in alcohol is also suggested as possible. Acetone dissolves thirty-one times its volume of acetylene, and alcohol six times its volume. Crude products are not suitable for use as motor fuels, but only those which have been purified with sulphuric acid and alkali.

O. Mohr<sup>1</sup> expresses the opinion that only simple mixtures should be employed, such as : Alcohol 50 parts, benzene 50 parts ; or alcohol 50 parts, benzene 25 parts ; and petrol 25 parts. These have been tried and have given satisfactory results, the latter being most suitable, since it gives no crystalline deposit in the winter time. Naphthalene is not to be recommended, since it tends to deposit. The addition of a small quantity of ammonium perchlorate (0.5 per cent.) to alcohol, although suggested by some, is not to be recommended, since chlorine compounds are generated on ignition, which will lead to corrosion.

W. R. Ormandy<sup>2</sup> describes tests which have been made with two types of modern petrol engines running on mixtures of alcohol and benzene in the proportions of 1 to 1 and 2 to 1, which yielded power equal to that obtained when petrol was employed. The same author<sup>3</sup> gives the results of tests with a four-cylinder Mawdsley engine having a cylinder of 90 mm. bore and 130 mm. stroke, with compression space of about quarter total cylinder capacity. The fuels employed were petrol, benzol, and mixtures of methylated spirit and benzol. The results were as follows :

TABLE XXXVI.—RELATIVE POWER OF MOTOR FUELS.

Fuel used.	Relative power.	Relative volume of fuel used.
Petrol (sp. gr., 0.710)	100	100
Benzol	98.25	84.5
Benzol, 1 part ; methylated spirit (sp. gr., 0.815), 1 part	99	96.3
Benzol, 1 part ; methylated spirit, 2 parts	92	108.9
Benzol, 1 part ; methylated spirit, 3 parts	91.5	124.5

<sup>1</sup> *Z. angew. Chem.*, 1914, 27, 558-559.

<sup>2</sup> *Jour. Roy. Soc. Arts*, 1914, 62, 234-235.

<sup>3</sup> *Jour. Gas Lighting*, 1913, 580.

In these experiments two qualities of benzol were employed, 90 per cent. benzol (sp. gr., 0.885) and 50-90 per cent. benzol (sp. gr., 0.875), no appreciable difference being noted. Further experiments proved that "straight run" naphtha (80°-160° C.) could also be employed. In the tests recorded the engine was run at 1000 revolutions per minute, but in another case it was found practicable to run at 2000 revolutions. The jacket of the induction pipe was kept at 160° F.

According to a "Times" report, a cheap motor fuel could be prepared by dissolving 10 per cent. of crude benzol in spirit at a cost not exceeding 6d. per gallon for manufacturing charges, which would be quite suitable for all motors at present in use, with little or no alterations to the latter.

The effect of various denaturing agents on the value of alcohol for motor purposes is illustrated by experiments conducted by A. Lecomte<sup>1</sup> with motor alcohol from different countries, the results being the averages of tests for maximum power developed, power developed at half speed, and the amount of alcohol consumed per horse-power. The order of efficiency is as follows: Austrian, German, Italian, Russian, Swiss, French. Austrian spirit contains no acetone, German motor spirit contains 0.5 per cent. of wood spirit and 0.25 per cent. acetone; Russian spirit, 10 per cent. wood spirit, 5 per cent. acetone; Swiss spirit, 5 per cent. of wood spirit and 2.2 per cent. of acetone; French spirit, 7.5 per cent. of wood spirit and 2.5 per cent. of acetone. As the result of these trials the author comes to the conclusion that wood spirit lowers the calorific value of the alcohol, which is what one would expect.

In Belgium the following formula for motor spirit has been recommended: Alcohol (90 per cent.), 100 litres; methyl-ethylketone (containing about 0.2 per cent. of higher ketones), 2.5 litres; benzene, 1 litre.

A motor fuel known as "Natalite" has, during the last few years, been made in South Africa. This fuel is stated to be composed of 55 per cent. of alcohol and 45 per cent. ether, with apparently a little ammonia. A certificate was issued by the

<sup>1</sup> *Bull. Assoc. Chim. Sucr. Dist.*, 1905, 23, 386-390.

Royal Automobile Club<sup>1</sup> recording the result of a trial with this fuel, in which it is shown that the consumption of fuel in an official trial was at the rate of 16·4 miles per gallon, or equal to 26·2 ton miles, there being no difficulty in starting the engine at any time. An official trial has also been carried out by "The Engineer" in comparison with petrol, in which it was found that the fuel consumption in both cases was practically the same. The engine worked quite freely with "Natalite" after a slight reduction of air supply to the carburettor had been effected, and no difference in the running of the engine could be detected when this fuel was used in place of petrol. It is claimed that the fuel actually brings about the removal of any deposit which may previously have been formed, and that it has no tendency to cause corrosion.

This fuel can be manufactured in quantity in existing distilleries, and it was proposed to manufacture it on a very large scale in South Africa.

It was also reported in the "Times Trade Supplement," June 19th, 1920, that a company had been formed in Australia to manufacture "Natalite" in Papua, where 100 square miles of territory had been acquired for the raising of the raw product, described as "plants and trees," which are capable of yielding 73 gallons of alcohol per ton. The company proposed to erect a plant capable of turning out 5½ million gallons of "Natalite" per annum, and to extend this eventually to a capacity of 18 million gallons. The price of this motor spirit would, it is stated, be about 2s. per gallon. Some idea of the nature of the raw material may be gathered from the fact that the company intended to form co-operative undertakings throughout the Commonwealth to induce farmers to undertake the cultivation of sorghum, which gives a yield of 80 gallons per ton. If these schemes materialised, it was believed that Australia would have sufficient motor fuel for its needs, and would be independent of imported petrol, which in 1920 amounted to £2,500,000 in value.

It appears that experiments have been in progress also in this country, with a view to the manufacture of a motor spirit from

<sup>1</sup> *Chem. Trade Jour.*, June 19, 1915.

alcohol by the Distillers' Company, Limited, the huge whisky concern, which has an aggregate capital of £8,000,000. At a recent annual meeting of this company the managing director informed the shareholders that a plant had been erected at their Hammersmith distillery for the production of this spirit, and that it was regularly supplied from there to motor owners, that steps were being taken for the construction of similar plants at their distilleries in Liverpool and Glasgow. Although no definite information has been given as to the composition of this fuel, the reporter gathered that it was made by mixing whisky with another material of Indian and Ceylon origin, and that it could be obtained at about half the price of petrol.

In considering the materials most suitable for the manufacture of alcohol cheaply for industrial and fuel purposes, it is necessary to take into account many factors, such as abundance of supply of raw material, cost of same, yield of alcohol, proximity to consumers or to shipping ports, efficiency of plant, and cost of denaturing and supervision.

A. P. H. Desborough<sup>1</sup> discusses the question of available supplies and cost of production of alcohol from various materials. The synthetic production of alcohol from calcium carbide, upon which great hopes were centered, does not appear to have been a successful venture. The Lonza Swiss factory contracted to supply alcohol, manufactured from this material, to the Swiss Federal Government at the equivalent of 1s. per gallon, but were unable to make it pay. The production of alcohol from coke-oven gases at the Skinningrove Iron Company's works worked out at about 2s. per gallon.

The liquors from the sulphite cellulose pulp manufactories form a raw material which costs practically nothing, but the yield of alcohol is small, and the cost of production comparatively high. In a recent report of the Honorary Advisory Council for Scientific and Industrial Research for Canada, it is stated that if the liquors from the whole of the pulp mills in Canada were thus dealt with, the yield of 95 per cent. alcohol would amount to at least 5,000,000 gallons, obtainable at a cost of about 1s. 6d.

<sup>1</sup>*Jour. Soc. Chem. Ind.*, 1920, 302 R.

per gallon. The production of alcohol from wood waste suffers from certain disadvantages, the raw material being obtainable only in very inaccessible regions, the cost of carriage to a distillery is almost prohibitive, the amount of acid required for conversion is considerable, and, besides, there is the residual cellulose, amounting to about 50 per cent. of the original wood, to deal with in a wet condition. Against this may be set the advantages that the wood waste is obtainable in enormous quantities, that its cost is extremely low, and that the residue may be burnt under the boilers and thus consumed, if not utilised in other ways. Peat and seaweed have been suggested, and even tried on a small scale, but the results so far obtained are not encouraging.

The author bases his calculation on alcohol selling at, say, 3s. 2d. per gallon. In order to place alcohol on the market at this price the manufacturer would have to sell at 2s. 2d. per gallon, leaving 1s. for distribution and other charges. The cost of manufacturing alcohol by fermentation in a modern factory he estimates at 9d. per gallon, including depreciation of plant.

The costs may therefore be set off thus :

COST OF PRODUCING AND DISTRIBUTING 1 GALLON OF 95 PER CENT. ALCOHOL.

	<i>s.</i>	<i>d.</i>
Raw material . . . . .	1	1
Cost of production of alcohol . . . . .	0	9
Interest on capital . . . . .	0	4
Distribution and other costs . . . . .	1	0
	<hr/>	
	3	2

The production of alcohol from fermentable carbohydrates averages 120 gallons per ton of pure material, and therefore the highest price which could be paid for such material is £6 10s. per ton delivered at the distillery. Taking into account the percentage of fermentable carbohydrates in the various raw materials, the highest price it is possible to pay for these is given in the table on opposite page.

From this table it will be seen that at the prices usually ruling for potatoes, mangolds, and such materials, the manufacture of alcohol could not be attempted with any hope of a profit.

Raw materials.	Maximum price payable per ton.
Fermentable carbohydrates (100 per cent.) .	£6 10 0
Jerusalem artichokes . . . . .	1 3 0
Potatoes . . . . .	1 3 0
Mangolds . . . . .	0 11 0
Sugar mangolds (13 per cent. sugar) . . . . .	0 17 0
Maize . . . . .	4 8 0
Rice . . . . .	4 17 0

The author states that the problem of the production of cheap alcohol has been under investigation at the Royal Naval Cordite Factory, Holton Heath, for some time, latterly in conjunction with the Department of Scientific and Industrial Research. The results were not at the time sufficiently advanced to allow of any useful conclusion being drawn from them but that progress in certain directions had been made. Several acres of heath land had been brought under cultivation and planted with artichokes. A new kind of beet, comparing very favourably with the sugar beet, which had been introduced into France by M. de Vilmorin for the manufacture of alcohol, had been planted, and a study was being made of a tuberous plant—a *Helianthus* (*Polymnia edulis*) which grows in the Andes at an altitude of 5000 feet, and produces tubers from  $\frac{1}{2}$  to 2 lb. in weight, and comparable in sugar content to mangold. The production of alcohol from wood was a problem also under investigation, from the point of view of converting cellulose into sugars by the agency of some particular organism, on the principle of the Boulard process.

The problem has also been discussed by A. M. Breckler,<sup>1</sup> who states that the total production of alcohol in the United States in 1914 was 182,000,000 proof gallons, which is equivalent to not more than 5 per cent. of the total consumption of petrol. Grain being the chief raw material employed in the manufacture of alcohol, in order to produce sufficient alcohol from this source to entirely replace petrol it would be necessary to consume 16 per cent. of the total cereal production, or 26 per cent. of the maize crop. During the five years previous to 1917, alcohol

<sup>1</sup>*Jour. Ind. Eng. Chem.*, 1917, 8, 612-15.

manufactured from maize cost 17·5 cents per proof gallon, and during 1917 the cost, it was estimated, would be 25 cents per proof gallon, or 45 cents per gallon for 90 per cent. alcohol, with no prospect of any considerable reduction in price, so that alcohol from this source could not compete with petrol. "Black strap" molasses forms a cheap source of alcohol, and some years ago it could be obtained at 2 cents per gallon at the refinery, but in 1917 the price was 12 to 14 cents per gallon for a poorer article. The cost of alcohol from "black strap" molasses, say, at 5 cents per gallon, would be about 10 cents per gallon, but the total available material in the United States and Cuba would produce no more than 162,000,000 gallons of alcohol, or only about 5 per cent. of the total required as motor fuel. Two plants were in operation in the United States for the production of alcohol from sawdust, and three plants were manufacturing alcohol from sulphite cellulose waste liquors, but these were favoured by the high price of alcohol.

In a report of the Executive Committee of the Australian Advisory Council of Science and Industry, 1916-17, it is stated that in Australia the most economical source of alcohol is sugar molasses, but if the whole of this were employed it would yield only about 4 million gallons of alcohol per annum, which is not nearly sufficient to replace the petrol imported, amounting to about 17 million gallons. The production of alcohol from molasses could, however, be largely increased, since out of a total of about 50,000 tons only about 12,500 tons is at present used for this purpose, the remainder being to a great extent wasted. The price of alcohol made from molasses was at that time about 1s. 9d. per gallon in Melbourne.

With regard to the production of alcohol in Australia from waste materials, such as waste timber, grass tree, prickly pear, waste fruit, and straw, the Committee do not see any prospect that alcohol could be prepared from such materials at anything like a reasonable figure.

The other alternative, therefore, is the growing of special crops for the purpose, those which offer most promising results being maize, wheat, barley, potatoes, and beet.

In considering the relative advantages of these materials for the fermentation industry, the yield of alcohol is most important, therefore the Committee have collected information from several sources on this point, and give the following as the approximate yield of alcohol under working conditions :

Yield of alcohol 95 per cent. strength per ton of 2240 lb.

Maize . . . . .	80-83	gallons
Barley . . . . .	65-70	„
Wheat . . . . .	80-85	„
Potatoes . . . . .	16-24	„
Beet . . . . .	12-16	„
Apples and pears . . . . .	9-14	„
Peaches and apricots . . . . .	9-13	„
Grass tree . . . . .	12	„
Sawdust (soft woods) . . . . .	20	„
Grapes . . . . .	18	„
Molasses . . . . .	65-70	„

The materials employed in Australia for denaturing alcohol to comply with the Excise regulations are 2 per cent. of wood naphtha,  $\frac{1}{2}$  per cent. of pyridine, and  $\frac{1}{2}$  per cent. of mineral naphtha, the cost of these denaturants being 1.74d. per gallon. The Committee proposed in place of these the addition of 1 per cent. of pyridine, which would cost 0.9d. per gallon, and approached the Comptroller-General of the Department of Trade and Commerce, in order to urge this change, but the proposal was not considered feasible.

Neither wood spirit nor pyridine are, however, suitable as denaturants for alcohol intended for use as a motor fuel. For this purpose W. J. Rowe<sup>1</sup> suggests the addition of benzol, petrol, or the redistilled oil obtained in the Pintsch gas producers, any one of which would be preferable as denaturing agents. This author points out that wheat is quite out of the question as a source of alcohol, and that potatoes for this purpose would have to be supplied at no more than 23s. per ton. The working expenses, irrespective of the restrictions of the Revenue authorities, he places at about 5d. per gallon on the average.

Molasses is one of the best and cheapest sources of alcohol, owing to its high content of sugar, and the fact that it needs no

<sup>1</sup> *Bull.*, No. 8, Dept. of Chem., South Australia.

conversion such as starch requires, but is ready to be fermented immediately after dilution. In this connection the article by T. H. P. Heriot<sup>1</sup> is of great importance. This author, after comparing the advantages of alcohol over petrol as a motor fuel, deals very fully with the production of alcohol, more particularly from molasses. The advantage of molasses as against starch is that in the former case the raw material is a waste product from another industry—sugar manufacture—the cost of raising, harvesting, and transporting the cane or beet crop having been already debited to the sugar, whereas with starch the cost incidental to its raising and production are entirely borne by the starch itself. Quoting Peck, of Hawaii, who states that “if the 21 million gallons of molasses produced in these islands in 1913 had all been converted into alcohol, over 9 million gallons of 90 per cent. alcohol would have been produced. A bushel of corn yields 2·8 gallons of 90 per cent. alcohol, and the average yield of corn is 30 bushels per acre. Therefore to produce 9 million gallons of alcohol 3,248,000 bushels of corn would be required, representing a crop from 108,000 acres, an area only 5000 acres less than that from which the 21 million gallons of molasses were derived, together with the main crop of 50,000 tons of sugar shipped.”

In a similar way Heriot calculates that a bushel of potatoes yields 0·73 gallon of alcohol. An average crop of potatoes being 20·5 bushels per acre, 9 million gallons of alcohol would require the crop from 60,000 acres, which is 53 per cent. of the area required in Hawaii for producing the equivalent of molasses and the additional 50,000 tons of sugar.

Molasses is in some countries a veritable waste product. If large distilleries were erected in sugar producing countries this waste product could be utilised in the manufacture of cheap alcohol for use in motors, and would go far to supply the needs of the community in this important commodity. Reviewing the present sources of molasses, Heriot gives the following particulars relating to cane and beet molasses :

<sup>1</sup>*Jour. Soc. Chem. Ind.*, 1915, 336.

## PRODUCTION OF CANE MOLASSES.

	Million gallons.	Per cent. on sugar made.
Hawaii . . . . .	16	19.5
Java . . . . .	52	25.4
Cuba . . . . .	90	27.3
Queensland . . . . .	6½	24.4
Egypt . . . . .	1½	22.0
	Average .	23.7

The total amount of cane molasses available annually is therefore 1½ million tons. Analyses are given showing the composition of molasses from various sources, the average percentage of sugar being 57.7 (calc. as glucose). The 1½ million tons of molasses would therefore represent 865,500 tons of sugar. Now sugar will yield, theoretically, 51.1 per cent. of its weight of alcohol, but owing to various causes—incomplete fermentation, bacterial action, and losses occurring during fermentation—the yield is lower than this. Investigations by Deer and Peck showed that 83 per cent. of the apparent sugars present in molasses could be converted into alcohol. Five or 6 per cent. changes into glycerol, and about 6 per cent. is unutilisable, probably due to unfermentable substances which are estimated as sugars. Making allowances for these, the production from 865,500 tons of molasses would be about 367,000 tons of alcohol.

In the case of beets, the lowest yield of sugar is obtained in France, that is, 13.18 per cent., and the highest in Germany, viz., 16.32 per cent., the average yield for the five years ending in 1911 in ten European countries being 15.38 per cent. The amount of molasses produced varies from 3 to 4 per cent. on the weight of the roots, which is equivalent on the lowest estimate to 19.5 tons of molasses per 100 tons of commercial beet sugar produced. Since beet molasses contains 48 to 53 per cent. of sucrose, or an average of 50, and the theoretical yield of alcohol 53.8 per cent. of the contained sucrose, an 83 per cent. yield of this on a production of 8 million tons of beet sugar, or 1.56 million tons of molasses, would be 348,000 tons of absolute alcohol.

The total possible production of alcohol from cane and beet

molasses would therefore be in the neighbourhood of 715,000 tons, or 200 million gallons.

The author gives the following comparative data for the cost of alcohol from various sources :—

	Gallons of absolute alcohol per ton.	Percentage of theoretical yield.	Cost of raw material per gallon 90 per cent. alcohol.
Cane molasses (57.7 per cent. total sugars) . . . . .	69	83	<i>Nil</i> to 8d.
Beet molasses (50 per cent. sucrose)	63	83	<i>Nil</i> to 8d.
Potato (20 per cent. starch) . . . . .	30	77	7½d.
Beetroot (15.4 per cent. sucrose) . . . . .	20	87	1s. 1d.
Maize (60 per cent. starch, plus 8 per cent. malt) . . . . .	84	87	11½d.
Rice . . . . .	80 to 90	—	—
Wood sawdust . . . . .	28 to 45	—	—
Nipa palm sap (14-16 per cent. sucrose) . . . . .	5.6 per cent. by vol.	74	6d.

The author states that the total cost of rum from Demerara landed in England is 1s. per gallon. In the United States the cost of producing 95 per cent. alcohol from molasses, exclusive of the cost of the raw material, and without taking into account the recovery of the nitrogen and potash, is 8 cents per gallon; in Demerara it is produced at 5 cents per gallon, but this does not include any allowance for fuel, which presumably is the crushed cane; in Hawaii the cost is 10.2 cents; and in Cuba, 10 cents per gallon. The working costs are therefore approximately 5d. per gallon.

This is an important problem in sugar producing countries where alcohol could be substituted for imported petrol and the excess sent to other countries; but, as shown by the author, if the problem was effectively dealt with, much could be done in this direction in Europe, Germany being an example of what is possible by scientific organisation and co-operation. In 1903 potato spirit was retailed in Germany at 7d. per gallon, and specially designed motors were placed on the market for using alcohol as a fuel. According to Brachvogel, one firm in Berlin alone had orders for over one thousand of these motors, intended to be used for the following purposes: Agricultural operations,

TABLE XXXVII.—ALCOHOL AS A MOTOR POWER

*Scientific Data bearing upon the Combustion in Motor Cylinders of Petrol, Benzine, Alcohol, Methylated Spirit, Methyl Alcohol, and Wood Spirit.*

	Formulae.	Mole- cular weight.	Boiling point.	Volume of 1 kilog. of vapour in cubic metres.	1 Kilogramme requires for com- plete combustion		
					Oxygen in kilog.	Oxygen in cubic metres.	Air in cubic metres.
<i>Hydrocarbons of the Saturated Series (Paraffins) C<sup>n</sup>H<sup>2n+2</sup>.</i>							
Pentane . . .	C <sub>5</sub> H <sub>12</sub>	72	36	0.3400	3.555	2.4855	11.950
Hexane . . .	C <sub>6</sub> H <sub>14</sub>	86	68.5	0.2595	3.535	2.4715	11.858
Heptane . . .	C <sub>7</sub> H <sub>16</sub>	100	98	0.2232	3.520	2.4610	11.832
Octane . . .	C <sub>8</sub> H <sub>18</sub>	114	125	0.1957	3.508	2.4529	11.795
Nonane . . .	C <sub>9</sub> H <sub>20</sub>	128	130	0.1744	3.500	2.4472	11.766
Decane . . .	C <sub>10</sub> H <sub>22</sub>	142	161	0.1572	3.493	2.4422	11.741
Undecane . . .	C <sub>11</sub> H <sub>24</sub>	156	194.5	0.1431	3.487	2.4380	11.721
Dodecane . . .	C <sub>12</sub> H <sub>26</sub>	170	214.5	0.1313	3.482	2.4345	11.704
Tredecane . . .	C <sub>13</sub> H <sub>28</sub>	184	234	0.1213	3.478	2.4317	11.691
Tetradecane . . .	C <sub>14</sub> H <sub>30</sub>	198	252	0.1127	3.475	2.4295	11.681
Pentadecane . . .	C <sub>15</sub> H <sub>32</sub>	212	270	0.1052	3.472	2.4275	11.671
Hexadecane . . .	C <sub>16</sub> H <sub>34</sub>	226	287	0.0988	3.468	2.4247	11.659
Heptadecane . . .	C <sub>17</sub> H <sub>36</sub>	240	303	0.0930	3.466	2.4233	11.650
Octadecane . . .	C <sub>18</sub> H <sub>38</sub>	254	317	0.0878	3.464	2.4219	11.644
<i>Benzenic Hydrocarbons.</i>							
Benzine . . .	C <sub>6</sub> H <sub>6</sub>	78	80.4	0.2862	3.077	2.1514	10.343
Toluene . . .	C <sub>7</sub> H <sub>8</sub>	92	111	0.2426	3.130	2.1883	10.521
Xylene . . .	C <sub>8</sub> H <sub>10</sub>	106	137	0.2105	3.169	2.2156	10.652
<i>Alcohols and Derivatives.</i>							
Methylic alcohol	C <sub>1</sub> H <sub>4</sub> O	32	64.4	0.6975	1.500	1.0489	5.042
Ethylic alcohol	C <sub>2</sub> H <sub>6</sub> O	46	78.4	0.4852	2.087	1.4591	7.015
Acetone . . .	C <sub>3</sub> H <sub>6</sub> O	58	56.4	0.3848	2.207	1.5430	7.419
<i>Mixtures.</i>							
1 litre Methylic Alcohol at 90° G.L. weighs 0.8339 kilog. and contains :							
0.71965 kilog. . .	CH <sub>4</sub> O	—	—	0.5019	0.6429	—	3.628
0.11425 kilog. . .	H <sub>2</sub> O	—	—	0.1410			
For 1 kilog. . .		—	—	—	0.7709	—	4.346
1 litre Methylene (Crude Wood Spirit) at 25 per cent. by vol. of acetone (A).							
0.750 litre methylic alcohol at 90°	}	0.8269	0.4822	0.5600	2.712	1.495	4.207
= 0.6254 kilog. . .							
0.250 litre acetone = 0.2015 kilog. . .							
For 1 kilog. . . . .			—	0.6893	—	—	5.100
1 litre Ethylic Alcohol at 90° G.L. weighs 0.8339 kilog. and contains :							
0.71065 kilog. C <sub>2</sub> H <sub>6</sub> O . . . . .	}		0.3448	0.4969	—	—	4.985
0.12327 kilog. H <sub>2</sub> O . . . . .							
For 1 kilog. . . . .			—	0.5959	—	—	5.997
1 kilog. Alcohol denatured (A) equals 1.200 litre				0.6173	—	—	5.942
1 kilog. alcohol denatured (A) carburetted equals	}			0.3953	—	—	8.218
1.171 litre . . . . .							

544 ; pumping engines, 88 ; creameries, 63 ; electric light installations, 52 ; woodworking machinery, 45 ; flour mills, 40 ; bakeries, 33 ; motor trucks, 30 ; boats, 30 ; and the remainder for general use in generating power.

In 1904, however, the potato crop in Germany failed, with the result that the price of alcohol rose to 1s. 3d. per gallon, which was higher than that of petrol, even after paying the import tax.

This fluctuation in the yield of crops in temperate climates is a very serious hindrance to the manufacture of alcohol at a competitive price.

THE END.

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