INDUSTRIAL ALCOHOL

J. G. McINTOSH

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

BY

CANADA: COPP CLARK CO. LTD., TORONTO

UNITED STATES: D. VAN NOSTRAND CO., NEW YORK

1907

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BY

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"THE TECHNOLOGY OF SUGAR" ETC.
LECTURER ON MANUFACTURE AND APPLICATIONS OF INDUSTRIAL ALCOHOL AT
THE POLYTECHNIC, REGENT STREET, LONDON

WITH SEVENTY-FIVE ILLUSTRATIONS
AND TWENTY-FIVE TABLES

LONDON: SCOTT, GREENWOOD & SON
19 LUDGATE HILL, E.C.

CANADA: COPP CLARK CO. LTD., TORONTO
UNITED STATES: D. VAN NOSTRAND CO., NEW YORK
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PREFATORIAL NOTE

THE Author's thanks are due to Messrs. Blair, Campbell, & M'Lean, Monsieur Barbet, Messieurs Egrot, Grangé et Cie., and Herr Stade and others, for illustrations and explanations of the most recent and modern installations of distillery plant and appliances equipped according to their respective systems. The Author also expresses his indebtedness to numerous authorities in different branches of the special literature of the subject.

J. G. M.

LONDON, *February* 1907.

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INDUSTRIAL ALCOHOL

CHAPTER I

ALCOHOL AND ITS PROPERTIES

1. Definition :—*Ethylic 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 97 per cent. strength may be obtained free from ethers and aldehydes on the one hand, and fusel oil (amylic alcohol, etc.) on the other.[1] Alcohol being more volatile than water, when a mixture of the two is distilled the alcohol comes over first, mixed with a certain quantity of water. The vapour is more rich in alcohol than the boiling liquid generating it. When these vapours are condensed just as they are given off by the liquid subjected to distillation, a spirituous liquor is collected, the strength of which thus depends upon how far 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 vapours, on the other hand, will rise and ascend further and further away from the source of heat towards the cooler portions, and where the more condensable vapour of water cannot follow them. 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 than the portions which condense further away. Only this latter portion is collected for sale. The more watery portions

[1] See footnote [1], p. 13.

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" abounds in aldehydes and ethers, that of the last runnings in fusel oil. In the continuous rectification distillation columns there is (1) continuous feed of raw spirit and continuous separate elimination therefrom of (*a*) ethers and aldehydes, (*b*) pure alcohol, (*c*) fusel oil, and (*d*) water.

2. *Properties of alcohol.*—Alcohol is a liquid with a vinous 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 bodies, with the exception of the fatty oils, which it only dissolves 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 extensive and important solvent. Its solvent action on resins, balsams, camphor, etc., is well known. As met with in commerce it invariably contains more or less water. The term *absolute* alcohol (9) is really more relative than absolute. Absolute alcohol, that is, alcohol free from water, even if it were capable of being prepared, would, the moment it came in contact with ordinary air, absorb water from it. A given bulk of alcohol weighs much less than a similar bulk of water. An imperial gallon of water weighs 10 lbs. An imperial gallon of absolute alcohol weights 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 so long as the liquid contains only pure alcohol and water (Tables I.–III.). Alcohol is highly 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 but little 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. We have said that the products of the ordinary combustion of alcohol are carbonic acid and water. The weight of the water considerably exceeds that of the alcohol burnt, thus:—

$$\underset{\text{Alcohol}}{C_2H_6O} + \underset{\text{Oxygen}}{O_6} = \underset{\text{Carbonic Acid}}{2CO_2} + \underset{\text{Water}}{3H_2O}$$

$$\underbrace{46 + 96}_{142} \qquad \underbrace{88 + 54}_{142}$$

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

3. *Destructive distillation products 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 got by T. de Saussure, who passed alcohol through a red-hot porcelain tube; there was deposited on its interior a little charcoal, a volatile crystalline substance (naphthaline), and a brown empyreumatic oil; a gas was evolved, the specific gravity of which was 0·856. This gas consists of a mixture of hydrocarbides (methane, ethane, acetylene, benzene), carbonic oxide and hydrogen, and a great number of pyrogenated hydrocarbides. When alcohol vapour and oxygen are mixed in proper proportions and fired by an electric spark, a violent explosion ensues, and carbonic acid and water are the results. One volume of alcohol vapour requires three volumes of oxygen for its perfect combustion, and the result is 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 (CrO_3) loses half its oxygen, and is reduced to sesquioxide of chromium (real chrome green), Cr_2O_3.

4. When alcohol is burned 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 less, and aldehydic and acetic compounds are formed. There are many substances which communicate *colour* to the flame of alcohol; from boracic acid and the soluble salts of baryta it acquires 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 saline bodies, forming, as it were, a substitute for 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 of them 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. It combines 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 decom-

poses 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, strontia, and magnesia. A mixture of alcohol and nitric acid is without action upon carbonate of potash, but it acts powerfully on carbonate of lime, and of strontia, and slowly on carbonate of soda, baryta, and magnesia. Alcoholic solutions of acetic, and of tartaric acid, decompose none of the carbonates: a similar solution of citric acid decomposes carbonate of potash and of magnesia, but not carbonate of baryta, strontia, or lime; and the alcoholic solution of oxalic acid decomposes carbonate of strontia, of lime, and of magnesia, 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, strongly acid. It is difficult to suggest an explanation of these statements.

5. 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 a 240th part at its boiling-point, and retaining a 320th 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 the alkalis. Potassium and sodium slowly decompose alcohol at common temperatures; heated with it, they evolve hydrocarbides. Caustic potash and soda are soluble in alcohol, and it is sometimes resorted to as a means of the purification of those alkalis; after a time, however, they begin to act upon each other, and complicated changes ensue; alkaline carbonate is formed, and carbonaceous matter is evolved on the application of heat; by their slow mutual action, acetic acid, a resin, and a species of brown extractive appear to be formed. Ammonia and its carbonates are soluble at common temperatures in alcohol: it also absorbs a large quantity of ammoniacal, and of several other gases. Lithia, baryta, strontia, and lime are almost insoluble in alcohol, even in their hydrated states; so also are the fixed alkaline carbonates: their sulphides are 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 alcoholised compounds above mentioned are produced. Thus there are definite 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; hence the use often made in the analysis of mixtures of salts, of the separative power of alcohol. Zeise studied the mutual action of chloride of platinum and alcohol. He showed the existence of a peculiar class of salts, of which 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 species of fuel, but we are almost debarred from its use by its high price; and for the same reason many manufactures, in which alcohol is an essential agent, cannot be productively carried on in this country. Its solvent powers, in regard to resins, oils, and other organic products, have been elsewhere noticed: its medicinal and pharmaceutical employment 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 products of many coal-tar colours, and hundreds of other substances. See Chaps. IX. and XI.

6. *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 $\frac{12}{13}$ths of an equal measure of distilled water." The temperature of the distilled water is not specified, but there can be no doubt that it also is referred to as at 51°. 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 + 64·5 = 164·5 proof strength.
164·5 × 19·8 ÷ 100 = 32·571
taken as 32·5 gallons at proof.

The facility with which the hydrometer can be used is such as

to render it the best adapted for determining the strength of alcohol by the Excise. 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 speaks for itself. "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."[1]

7. *Heat developed by and contraction ensuing on mixing alcohol and water.*—Equal mixtures of alcohol (D = 0·825) and water each at 10° C. (= 50° F.) register, when suddenly mixed, a temperature of 21·1 C. (= 70° F.), and a mixture of equal measures of proof spirit and water each at 10° C. (= 50° F.) registers, under like conditions, 15·6° C. (= 60° 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° 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° C. (59° F., Rudberg). From this point the contractions

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.

[1] 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. 14–16.

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.

8. 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 C_2H_6O = 46 and 3 molecules of water = 54. The absolute amount of the contraction varies with the temperature, according to Tralles, at 4° C. (39° F.) it amounts to 3·97, at 11° C. (52° F.) to 3·77, at 18° C. (64° F.) to 3·60, and at 37° C. (100° F.) to 3·31.

9. *Absolute alcohol.*—Absolute alcohol is prepared by rectifying the alcohol of commerce by substances which take up water. Quicklime is the substance most generally used. The alcohol is digested with a large quantity of quicklime 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, caustic baryta, made by decomposing the nitrate.

10. 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.

11. 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 of the lowest of the tables and 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, having been managed with a considerable air contact, 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 tables were all too high for the present time. Table III. gives results of his reinvestigation. Metallic sodium has been 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.

$$\left.\begin{matrix} Et \\ Na \end{matrix}\right\} O_2 + H_2O = NaHO + EtHO$$

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 sec. 16).

12. The phlegms furnished by the best distilling columns are far from being a mixture of water and pure alcohol. 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, furfurol. 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 aldehydes, essential oils, etc. etc. If some of these bodies by their being present impart to the liquors a savour 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.

13. *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 may be detected by adding a small amount of the alcohol to be tested to a saturated alcoholic solution of liquid paraffin. Slight traces of water render the liquid 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) The first runnings impurities may be recognised and roughly estimated by aid of the reaction of 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. 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.

14. 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*) *Determination of ethers.*—Boil 100 c.c. of the distilled alcohol for an hour along with 20 c.c. of decinormal potash, 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. (*b*) *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 ethylic aldehyde at $\frac{1}{10000}$, 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 × 0·050.

(*c*) *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 aldehyde phosphate. 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 sulphuric acid at specific gravity 1·84 (168° Tw.) according to the known method, 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.

(*d*) *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 grm. 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 furfurol, 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.

15. *Separation of the aldehydes—Girard and Rocques' method.*—The authors dissolve in 200 c.c. of alcohol, at 50°, 3 grms. of metaphenylen-diamine hydrochloride, and boil for half an hour with an ascending condenser. The liquid takes a pale yellow colour. It is let 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

takes 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

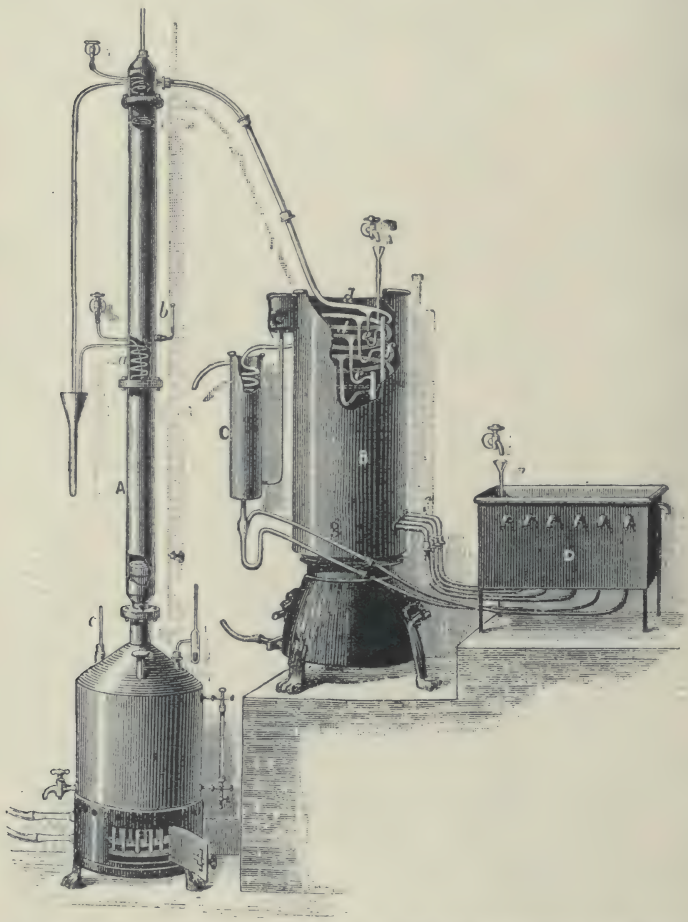


FIG. 1.—Laboratory Rectifier (SOREL). A, rectifying column; *a*, auxiliary refrigerator; *b*, thermometer; B, condenser No. 1 regulated to 77°·5 C.; *dd*, coil of 10 spirals, each with S tube (*e*) for products condensed therein; *f f*, separate collectors of four different lots from *e* (viz. 1 and 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).

those given by alcohol at 75°, to which known quantities of pure amylic alcohol have been added.

16. 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, baryta, sodium, etc., although adequate to remove the water, fail to affect the aldehyde, and it cannot be eliminated by fractionation.[1] The problem of its removal has been attacked recently by L. W. Winkler in the following manner (*Ber.* 1905, xxxviii. 3612):—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 grms. 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 grms. 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 is 99·9 per cent. pure. 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

[1] 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. 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 Morin'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 B C is not an analyser, as generally believed. The analysis is effected on the plates (Figs. 46 and 50).

with the following results:—Sp. gr. 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 m.m.), 78°·20 (754·9 m.m.), and 78°·29 (757·8 m.m.); therefore a difference of 1 m.m. pressure causes a change of 0·034° in the boiling-point. Winkler mentions two rather curious facts which he has observed in the course of his work. 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. 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 CORRESPONDENCE BETWEEN THE SPECIFIC GRAVITY AND PER CENTS. OF ALCOHOL OVER AND UNDER PROOF AT 60° F. (URE).

Specific Gravity.	Per Cent. over Proof.	Specific Gravity.	Per Cent. over Proof.	Specific Gravity.	Per Cent. over Proof.	Specific Gravity.	Per Cent. 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

TABLE II.—*Continued.*

Specific Gravity.	Per Cent. over Proof.	Specific Gravity.	Per Cent. over Proof.	Specific Gravity.	Per Cent. over Proof.	Specific Gravity.	Per Cent. under 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.	Per Cent. under Proof.	Specific Gravity.	Per Cent. under Proof.	Specific Gravity.	Per Cent. under Proof.	Specific Gravity.	Per Cent. 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	31·1	·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 combinations 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—		
	4° C. = 39°·2 F.	15° C. = 59° F.		15°·6 C. = 60° F.		25° C. = 77° F.		15°·60 C. = 60° F.	25° C. = 77° F.	
	True.	Apparent.	Corrected for Expansion of Glass.	Apparent.	Corrected for Expansion of Glass.	Apparent.	Corrected for Expansion of Glass.	True.	Apparent.	Corrected for Expansion of Glass.
100	0·80257	0·79348	0·79326	0·79301	0·79279	0·78537	0·78496	0·79350	0·78591	0·78573
99	0·80579	0·79667	0·79645	0·79618	0·79595	0·78847	0·78806	0·79669	0·78901	0·78882
98	0·80875	0·79965	0·79943	0·79893	0·79870	0·79131	0·79089	0·79967	0·79205	0·79186
96	0·81467	0·80555	0·80533	0·80509	0·80486	0·79744	0·79702	0·80558	0·79801	0·79782
92	0·82593	0·81680	0·81658	0·81631	0·81607	0·80865	0·80823	0·81684	0·80915	0·80896
88	0·83649	0·82751	0·82728	0·82699	0·82675	0·81929	0·81886	0·82755	0·81982	0·81963
84	0·84681	0·83770	0·83747	0·83719	0·83695	0·82953	0·82909	0·83775	0·83007	0·82987
80	0·85683	0·84773	0·84750	0·84718	0·84694	0·83959	0·83915	0·84779	0·84021	0·84001
76	0·86655	0·85742	0·85718	0·85699	0·85674	0·84937	0·84892	0·85749	0·84991	0·84971
72	0·87600	0·86702	0·86678	0·86649	0·86624	0·85895	0·85850	0·86711	0·85953	0·85933
68	0·88516	0·87655	0·87631	0·87607	0·87582	0·86847	0·86801	0·87665	0·86905	0·86885
64	0·89479	0·88625	0·88601	0·88578	0·88552	0·87832	0·87786	0·88636	0·87884	0·87863
60	0·90401	0·89549	0·89524	0·89556	0·89530	0·88766	0·88719	0·89561	0·88821	0·88800
56	0·91297	0·90452	0·90427	0·90405	0·90379	0·89687	0·89640	0·90465	0·89745	0·89724
52	0·92177	0·91349	0·91324	0·91309	0·91283	0·90597	0·90549	0·91365	0·90653	0·90632
48	0·93045	0·92231	0·92206	0·92187	0·92160	0·91489	0·91441	0·92247	0·91547	0·91525
44	0·93875	0·93082	0·93056	0·93045	0·93018	0·92361	0·92313	0·93101	0·92427	0·92405
40	0·94655	0·93901	0·93875	0·93865	0·93838	0·93217	0·93168	0·93923	0·93275	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			

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

Volume.	Weight.	Volume.	Weight.	Volume.	Weight.	Volume.	Weight.
Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	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 VI. ALCOHOLIC CONTENT OF BOILING LIQUID AND VAPOUR OF AQUEOUS ALCOHOL AT DIFFERENT BOILING-POINTS (GRÖNING).

Temperature of Vapour. Degrees.	Alcohol by Volume in Boiling Liquid.	Alcohol by Volume in Distillate.	Temperature of Vapour. Degrees.	Alcohol by Volume in Boiling Liquid.	Alcohol by Volume in Distillate.
C.	Per Cent.	Per Cent.	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			

TABLE VII. SHOWING THE CONTRACTION OF ALCOHOL IN COOLING DOWN FROM ITS BOILING-POINT IS GIVEN BY DUMAS AS FOUNDED ON 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 VIII. AMOUNT OF WATER TO ADD TO AN ALCOHOL OF GIVEN STRENGTH.

	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 an 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 IX. BOILING-POINT IN DEGREES C. OF ALCOHOL OF DIFFERENT STRENGTHS (NOYES AND WARFRE).

Per Cent.	Boiling Point.	Per Cent.	Boiling Point.	Per Cent.	Boiling Point.	Per Cent.	Boiling Point.	Per Cent.	Boil'g 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 metadiamido benzol 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 cc. of sample into test-tube, add 0·02 grammes of either carbolic acid, naphthol α, naphthol β, resorcin, hydroquinone, phloroglucine, pyrogallol, guaicol, thymol, gallic acid. The reagent dissolved, pour 1 cc. 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.

CHAPTER II

CONTINUOUS FERMENTATION AND STERILISATION IN INDUSTRIAL ALCOHOL MANUFACTURE

1. THE manufacture of industrial alcohol comprises: Saccharification, Fermentation, Distillation, Rectification. *Saccharification.*—Sucrose, glucose, and maltose[1] are either directly capable of undergoing fermentation, or can be transformed into a sugar capable of undergoing fermentation, by a soluble diastase, *invertine*, secreted by the alcoholic ferment. These do not, therefore, require any special initial 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 (*a*) by the action of dilute acids (5 lbs. of acids per 100 lbs. of grain) acting under a pressure of 5 kilogrammes per sq. c.m., or (*b*) by the diastase of malt at 12° C. (53°·6 F.); about 25 lbs. of malt are used for 100 lbs. of starch to be saccharified. (See Chaps. IV. and V.)

2. *Fermentation.*—The transformation of all the above saccharine matters into alcohol is effected by an *organised* ferment belonging to the numerous class of saccharomyces. The great difficulty of industrial fermentation is to produce a sufficient vegetation of the right and 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 that quantity be too small, reproduction is stopped, and the fermentative power soon disappears. If too 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,

[1] For a detailed description of these sugars, densities of their solutions, etc., see the author's *Technology of Sugar*, 2nd ed.

but no more, and to use up completely the ferment produced. The presence of other figured ferments must be avoided. They not only use up, in waste, some of the sugar treated, but produce bodies, 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 anaerobic, do not take kindly to the presence of oxygen. They may thus be dealt with by oxidising or oxygenising 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 1 litre titrates 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; as 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 matters 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 cause the ferment in a regression stage to secrete disassimilation products injurious to the quality of the alcohol.

3. 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 *pied 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 albumenoid 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 titrating as much as 18, and even 22 per cent. of maltose, and the percentage of sugar is never let 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 permissible extent its fermentative power without propagating itself beyond measure. Moreover, it must not be allowed to reach the regression 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.

4. Yeast is also used in the fermentation of beet juice, but from a different point of 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 albumenoids, 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 4 hours. In the case of beet juice treated in this way the fermentation process lasts 24 hours, with molasses 48 hours, with grain saccharified under pressure, or for thick grain or potato worts saccharified by malt, 72 hours.

5. 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" (*pied de la cuve*) without any intermediate prolification 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 formalised water, and all the valves possess a small vessel in which the packing is protected by antiseptic water. The prolific propagation of the yeast is intensified and its fermentation strength increased by doing 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. 2), 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 4 to 6 aeration plates, on which the liquid forms a very thin layer about 2 c.m., say, $\frac{3}{4}$-inch thick. The liquid of the lower reservoir is continuously raised on to the upper plate by means of a steriliser 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 sort of small elongated tubular vessel which only 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 splits up into a series of air-bells which occupy the whole width of the pipe, and which are separated one from another by rings of liquid (*liquid pistons*). If the entrance 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 aerobiose 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.* a 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 immerged form of the mucedinæ would be produced, the mycelia would divide and assume the form of oval globules, like yeast, and yield alcohol.

6. 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 C D E. H is a small vessel containing formalised water 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

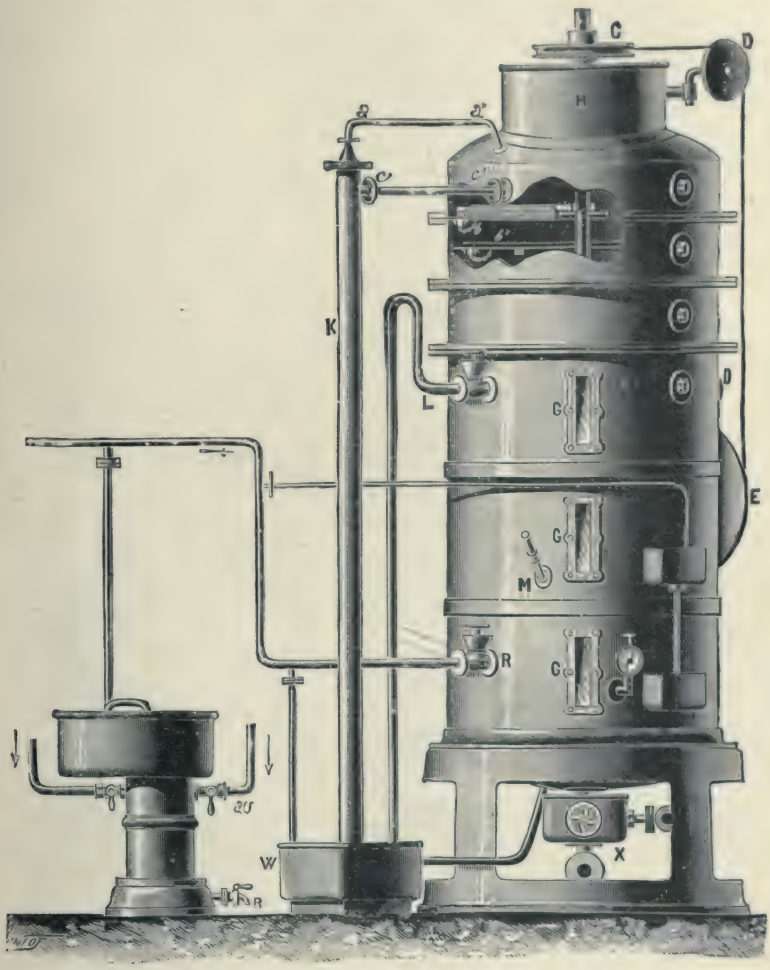


FIG. 2.—Plant for cultivation of pure ferments (aerobic cultivation) (E. BARBET, Paris).

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

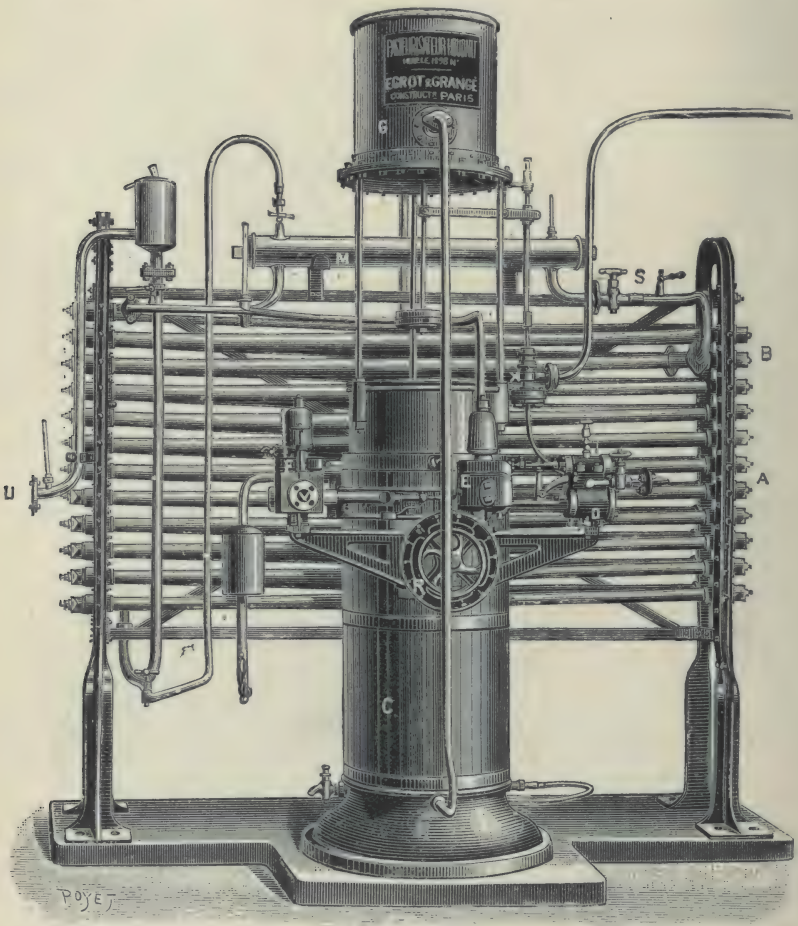


FIG. 2.—Forewarmer steriliser (EGROT and GRANGÉ). A, B, jacketed tubular pipes: B, heating; A, cooling.

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. 3 shows Egrot's combined steriliser and forewarmer.

7. Before the time of Pasteur, everything was empirical (rule of thumb); whilst at the present day, Pasteur's methods, which he himself had already applied to brewery and vineyard practice, are beginning to be adopted in industrial distilleries. By their means the distiller can regulate his fermentation processes, whereas formerly he was completely at their mercy. 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 had 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 albumenoids, phosphates, and salts, it became 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 an altogether preponderating influence being attributed to the strain of ferment, and 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 counter to its natural and favourite food, it is none the less true that there is some change in its mode of existence, and this change resolves itself into a modification in the nature and *bouquet* of its secretions.

8. 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 secretions, Pasteur determined 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 secretions, the simultaneous generation of which is incontestable. Now, in brandy, it is not the alcohol which has the commercial value, the whole value of brandy rests most decidedly in the perfumed secretions, peculiar to this form of fermentation. The alcohol is simply the vehicle for the perfumed secretion. If we cultivate a grand 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 the sum of the characteristic aromas of wine, because the culture medium has altered the digestive secretions of the ferment. If in the residual lees from the distillation of white wine we place 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 tartaric acidity, the same albumenoids, saline or 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 on the one hand, and the old malt whisky distillery and those which produce the so-called natural brandies on the other hand. One hundred years ago, as far as natural brandies are 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.

9. On the dawn of industrial distillation, seeking its raw material amongst cheap agricultural products, such as raw grain, potatoes, beets, Jerusalem artichokes, or sugar residuals, treacle or molasses, 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 fermentations by yeast, to carry continuous distillation into practical effect, and to purify the product afterwards (because it in no way resembled natural brandy) by rectification, filtration, chemical or electrical treatment, addition of perfumes, etc. All improvements, however, have only served to widen still further the gulf separating the two kinds of distilleries: the old distillery retaining its old supremacy quite intact, whilst the industrial distillery had no other ambition than to make cheap routine products—in one word, alcohol. It strove to eliminate all smell which would betray its origin, and to approach neutrality, and chemical purity, as far as possible. But this neutrality itself only shows that qualities and grades have been renounced, and that the least possible amount of defects is alone aimed at in plain spirits (Chap. IV. sec. 12).

10. To produce spirits resembling natural brandy, Barbet commences by treating potatoes or beets so as to extract their starch or sugar by the usual methods. He saccharifies the starch by the following method. He uses normal sulphuric acid, in the proportion of 1 to $1\frac{1}{2}$ per cent. on the starch, first boiling

in the open air to dissolve the starch, then charging into a copper autoclave at a pressure not exceeding 1 to 1½ kilogrammes per square centimetre at the most. Duration about one hour (Chap. II. sec. 1). The other processes are: saturation by chalk (the syrup is extremely pale); filtration through a filter press and through animal charcoal, and also, if necessary, through wood charcoal. It must be rendered colourless and inodorous. In the composition of 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 are absolutely interdicted. The most convenient manner of reproducing fruit must, consists in using the vinasse from fruit distillation. It may seem difficult at first sight, to those engaged in the industry on the large scale, to procure such musts in sufficient quantity for daily use; but first of all it should be borne in mind that the original must can serve at least five times, and even more, it being taken for granted that certain special heating precautions are observed during distillation. The principal danger to be avoided is caramelisation, therefore 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 it will be seen that his process has nothing subversive in it. He employs other vegetable acids and other vinasses. Certain precautions, certain turns of the wrist, render this process far from laborious, and it is the one to which the manufacturer should devote all his care and attention.

11. 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 in aerobiose culture, so as to yield the maximum activity and vitality to the ferment in very acid musts. The purity and the high vinosity of the three-six[1] are in direct proportion to the organic acidity of the musts. Moreover, he claims that his system of pure ferments and aerobiose 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 fraction of alcohol is sacrificed, in order to produce enough ferment, as it is the source of the desired aroma. Therefore, at the end

[1] *Trois-six* is a term applied in France to 90 per cent. alcohol, because 3 volumes of it mixed with 3 volumes of water yield a mixture marking 19° Cartier.

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 with aerobiose, so as to make reproduction as prolific as is 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, prevent infection from all kinds of bacteria. It is very difficult to produce forced ageing of spirits after or during distillation. These are very delicate culinary questions, and success is not often attained. Barbet gets over the difficulty in a great measure by ageing the fermented liquor before distillation by prolonged heating of the fermented wash in a series of vessels, PP^1 P^2 P^3, Fig. 3*a*. By pumping the fermentation gases into P^3, 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.

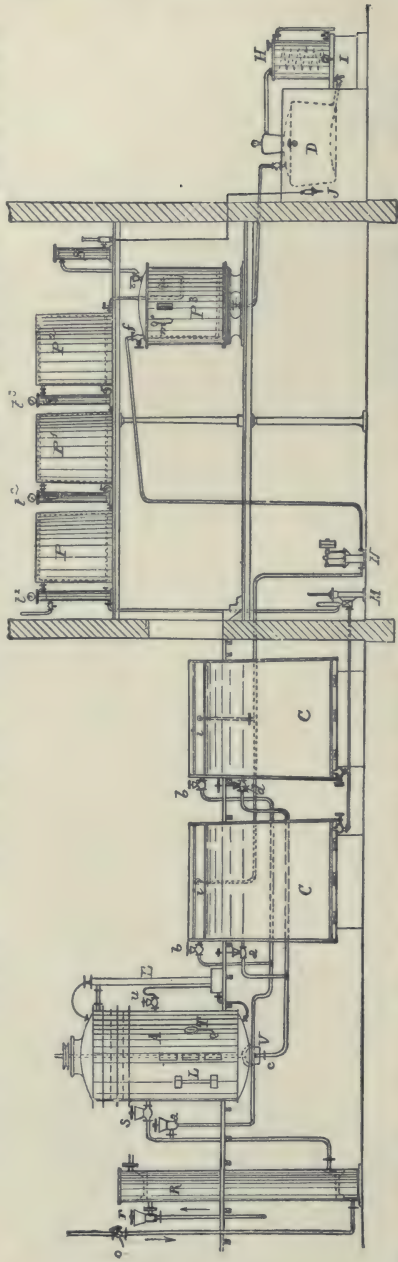


FIG. 3*a*.—Plant for aseptic fermentation, capture of fermentation gases, ageing by prolonged heating and distillation of spirit (E. BARBET). *A*, ferment vessel; *C C*, fermentation tuns; *K*, steriliser; *M*, fermented wash pump; *N*, fermentation gas pump; *i i*, fermentation gas pipes; t^1 t^2 t^3 calorisators; P P^1 P^2 P^3, closed vessels; P^3, autoclave to stand pressure of CO_2 from *C C*; *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).

CHAPTER III

THE MANUFACTURE OF INDUSTRIAL ALCOHOL FROM BEETS

1. *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 bodies, such as certain acids, asparagin, etc., as shown in accompanying table.

TABLE X. COMPOSITION OF SUGAR BEET AND SUGAR-BEET JUICE.

Water, 79 to 84·5 per cent.
Dry matter, 15·5 to 21·0 per cent., of which—
  Soluble in water, 11·5 to 17·0 per cent.
  Insoluble in water, 4 to 5 per cent.
Ingredients of juice—
  I. Water . . 80 per cent.
  II. Dry matter . 20 ,,
    A. Sugar . . . 15 per cent.
    B. Non-saccharine matter 5 ,,
      (*a*) Ash, 0·8.
        (1) Incombustible salts—
          Potassium, sodium, rubidium, vanadium, calcium, magnesium, iron and manganese, combined with chlorine, sulphuric, phosphoric, silicic, and nitric acids.
        (2) Salts transformed by combustion into carbonates. The same metals combined with oxalic, citric, malic, and succinic acids.
      (*b*) Nitrogenous matter, 1·60 per cent.
        Proteins (albumen, etc.).
        Plasmatic substances.
        Asparagin ($C_4H_8N_2O_3$), and various amides.
        Betaine ($C_5H_{11}N_2O_2 + H_2O$).
        Glutamine ($C_3H_5(NH_2)(CONH_2)CO_2H$).
        Leucine ($NH_2C_5H_{10}CO_2H$).
        Tyrosine ($C_6H_4(OH)(C_2H_3)(NH_2)CO_2H$).

(*c*) Non-nitrogenous bodies, 1·0.
Raffinose ($C_{18}H_{32}O_{16} + 5\ H_2O$).
Arabinose ($(C_6H_{10}O_5)^n$).
Dextrine ($(C_6H_{10}O_5)^n$).
Soluble pectic substances.
Chlorophyll.
Chromogene.
Fat.
Coniferin ($C_{16}H_{22}O_8 + 2\ H_2O$).
(Vanillin, $C_8H_8O_3$).
(*d*) Cellulose bodies, 1 per cent.; pectic, 0·6 per cent., and colouring substances.
(*e*) Cholesterin ($C_{26}H_{42}OH + Aq$).

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

2. *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 more are the 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. 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 passed to the saccharimeter,[1] there are few agricultural distilleries where the instrument is in current use. It is, however, met with pretty often, but, when not of a very primitive form, or completely out of working order, it is generally regarded as an object of curiosity, a luxury, in which the distiller has indulged to gratify himself. It is used, occasionally, in the course of a "campaign," for example, when very high or very low beets are being put through hand, 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, it matters little; what it is desired to establish is, that the distiller has an immediate interest in keeping an exact account of the percentage of sugar in his beets, if it were only to ascertain whether the alcohol extracted corresponded 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 such and such a field, are more saline, 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

[1] *I.e.* to the polariscope, see the author's *Technology of Sugar.*

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 more pure, and their density is 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 got by him, due to nothing else, will be more considerable 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°, the sugar corresponds well with that generally accepted, viz. twice the density. This coefficient decreases in proportion as the density becomes lower, so that a beet, at 4°, for example, rarely reaches 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 in 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.

3. 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. 4. The beets are brought by truck to the foot of the archimedean screw elevator, which lifts them up 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

FIG. 4.—Screw elevator for beets—Beet washer—Stone eliminator—Cup elevator (EGROT and GRANGÉ). (Sec. 3.)

machines (Fig. 8, H), from whence they descend into the conveyor which feeds the diffusers (Fig. 5; Fig. 8, I).

Feeding the washing machines.—The cheapest way of feeding the beet washers is *by hydraulic carriers*, a narrow channel or mill-lade 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 pitched into this stream, as their density is only slightly greater than 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 swim 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 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 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 slope 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.

4. *Beet-slicing machinery.*—The essential organ 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 of a certain height, 20 to 40 inches, the weight of the beets above, pressing on those below, and which rest immediately above the plate, acting as an automatic pusher, causes 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 8 to 10 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 used up, 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 (Chap. III. sec. 16). 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 belts and pulleys. 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 shift. The diameter of the plates is very variable—the average is 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 revolve more slowly than smaller ones. Plates of 5 feet in diameter make 100 to 120 turns, so that 6½-feet plates need only make 60 to 90 turns for their circumference to have travelled the same space in the same time as the 5-feet plates.

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 into small sections the slices cut off by the knife. (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 zigzag form at an angle of 60°. 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 is thus altogether 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 the mark of its shape on the beet, when the next arrives, it passes into the same furrow, it 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 catch more or less as the slice is to be larger

or smaller. The knives are sharpened on very hard steel discs, which revolve rapidly on their axis, 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. It is a big job, which 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.[1]

5. *Extraction of beet juice by maceration.*—About 1830, Mathieu de Dombasle (1777–1843), a celebrated French agronomist, 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 its juice from the sugar beet without pulping, rasping, or grating the beet, and without hydraulic presses. The beets were cut into thin slices by a rotary machine. The slices were then transferred to the No. 1 of a series of casks arranged in the form of a battery, the juice of No. 1 being run on to No. 2. The beet slices were macerated in No. 1 for about an hour with about their own bulk of water, at a temperature of about 212° F. After this treatment, the liquid—having now acquired a density of 2° Baumé, sp. gr. 1·014—was run off into No. 2 containing fresh beet slices. From the No. 2 it was run into No. 3, and so on until it had passed through the 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 prevent cooling, it was reheated in its passage through the casks, and the maximum amount of sugar possible was thus extracted. The exhausted slices scarcely contained any sugar. But 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, as the juice obtained by it 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

[1] See author's *Technology of Sugar.*

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 carbonatation (alternate treatment of beet juice by lime and carbonic acid) paved the way for diffusion in sugar works, its adoption by distilleries followed as a natural sequence. It was first used in a distillery by E. Barbet at Telques in 1880.

6. *A diffusion battery* consists of a series of eight to fourteen cylindrical vessels arranged consecutively, and called *diffusers*. They 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 *calorisator*, keeps the liquid always hot. Again, taps are so arranged that water may be run into each diffuser, instead of juice. There is also a tap 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 starts to act 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, as 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, such as it is, is then run into the diffuser next to it, being reheated in its passage. The saccharine liquid is now in contact with fresh beet slices, whose juice is of greater density than its own, *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, like certain white wines after they have gone wrong.

7. *Simplified diffusion.*—The system of simplified diffusion

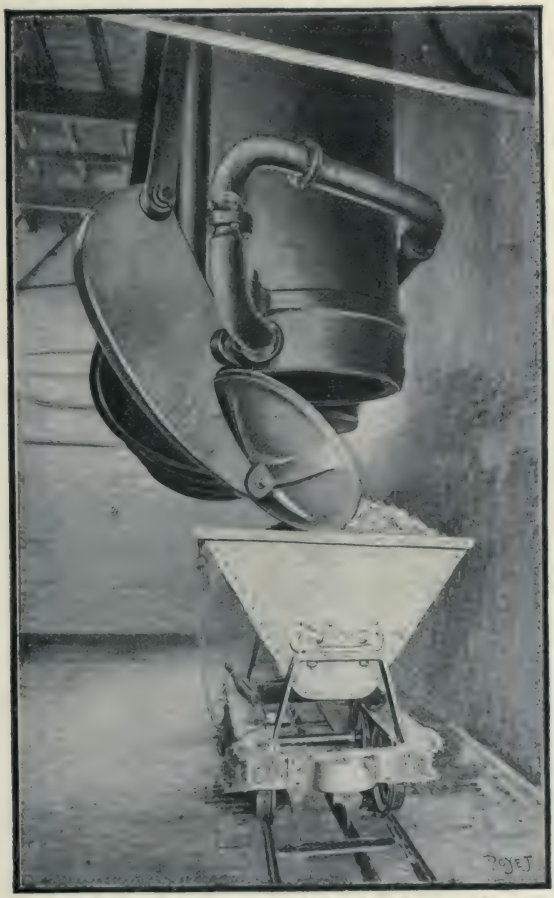


FIG. 5.—Beet diffusers (bottom part) showing doors and method of discharge of spent slices into lateral tilting trucks on rails *en route* for spent pulp silo (Fig. 10) (EGROT and GRANGÉ).

installed in the distillery of M. Postel (Fig. 8), consists of 6 diffusers in line; they have a total 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, the widest diameter at the bottom. The bottom (Fig. 5) of each diffuser is

entirely constituted by the discharge door, so that that being opened the whole contents of the diffuser are discharged directly into a truck underneath. The opening (Fig. 5) and the closing of this door is manipulated from above by the man in charge of the diffusion (Fig. 8, H, I); for each diffuser a single tap enables 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 pressed 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 and 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. 3).

8. A common fault, in distillery working, whatever method of extraction be adopted, consists in exaggerating too far the proportion of juice withdrawn per kilogramme of beets. This over-extraction is made for fear of not sufficiently exhausting the slices; but, very often, the volume of juice withdrawn may be very materially reduced without the sugar increasing in the pulp, on that account. The three points which determine exhaustion must be better supervised: (1) the state of division of the pulp or slices and the regularity of the same; (2) the temperature in the macerators or diffusers; (3) the length of contact. Very often it is tried to make good, by increasing the proportion of juice withdrawn, a defect in one of the above three factors. 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, which it would then be imprudent to further diminish, exactly as in a beet-sugar works. The advantages to be gained from this reduction in volume of extracted juice are the same from all points of view, an increase in the output, and reduction of the general expense. The further treatment of the juice, whether it be evaporated or distilled, is on a smaller bulk, hence economy in full. The distiller, in 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. Besides, the expense of acid is less, and the only thing to supervise is the exhaustion of the pulp. But to repeat, over-extraction is perfectly useless, if extraction has been well done, or rather it is only a costly palliative of imperfect extraction.

9. *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 1 to 8 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 fact. It is this latter 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, except in those factories which wish to produce strong leavens from pure ferments. Now the method adopted at Dammard differs essentially from these three methods. It is real continuous

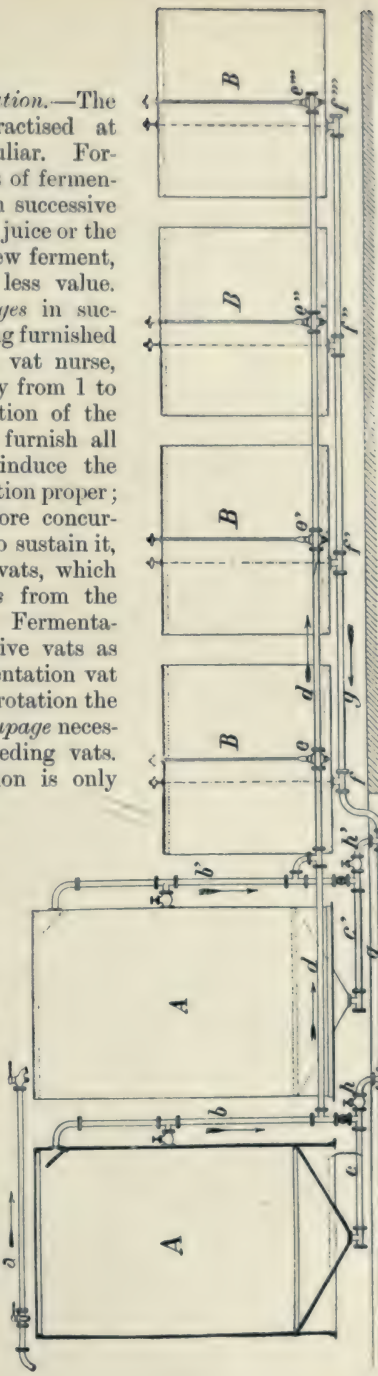


FIG. 6.—Diagrammatic illustration of Fermentation Department of Dammard Beet Distillery (Fig. 8, M, N, O) (EGROT and GRANGE). AA, chief fermentation tuns; B, "Cuves de chute"; *a*, juice pipe; *b b' c c'*, draw-off pipes; *d*, feed pipe to B B B B; *e e' e'' e'''*, feed taps; *f f' f'' f'''*, exit taps; *g*, pipe to distillery; *h*, pipe to discharge A (either vessel) into *g* (if need be).

fermentation effected constantly in the same vat, that being 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 existing piping does not admit of it. To send the fermented wort to the distillation, a continuous draw off is made from the fermentation vat, and the rate of flow is so regulated as to maintain always in the latter the right 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* in which the fermentation subsides, and from which it passes to the direct distillation rectification plant. At Dammard Distillery, fermentation is performed in this way, in two twin vats A A 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 4 *cuves de chute* B B B B of much smaller capacity. It may be added that three of these suffice after working for 15 days and at the full capacity of the factory. Fermentation is very fine, very active, and very complete, being maintained at 29° Centigrade in the fermentation vats without ever going beyond 30°, and falling towards 26°–27° in the *cuves de chute.*

10. *Antiseptic fermentation.*—Fermentation is often started by stirring a quantity of beer yeast into the beet juice, then, when fermentation has made a good start, letting the juice flow on to the foots thus formed; when in full swing several fermenting tuns are kept at work simultaneously by starting a fresh one when the tail one is full. That is working by "coupage," or mixing of the tuns, almost exclusively used in distilleries working by beer yeast. In some distilleries the use of beer yeast has been given up on account of its doubtful purity. Starting from the principle that to obtain a really pure ferment it is necessary that the point of departure, that the yeast itself be perfectly pure, select ferments are used and 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" (Fig. 2) and ferment tuns of different capacities. Besides, the precautions to take 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 epochs 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 all contamination by extraneous germs is avoided, contamination from those contained in the juice itself cannot be prevented. If the proportion 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 hotbeds for microbes. This contamination will make itself manifest in a more apparent manner the further the juices have themselves gone wrong. Some distillery specialists therefore urge that the system of true aseptic fermentation can never be applied integrally in the working of beets.

11. There is therefore, they argue, some justice in the remark, "Why force us by costly plant and elaborate precautions to refrain from introducing foreign ferment into our leaven, if it only be to mix the same leavens 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 altogether its initial purity up to the "chute," it will always be less imperfect than in the system of mixing the tuns, where the contamination, even momentarily, in one tun may affect all which succeed it until the fermentation 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, nothing prevents the difficulty being 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 action against contamination, but other agents, and amongst them hydrofluoric acid fluorides, possess this power in a high degree. Generally, when fermentation is effected in antiseptic washes, a start 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. In the following experiment, it was that of 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, 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 grammes per litre. Attempts were made to increase this proportion, but the fermentation slackened, and became weak, and the use of fluoride had to be suspended for several hours. The same results were got by repeating the experiment, and the dose had to be

restricted to the above, otherwise the leaven itself was embarrassed by the presence of that 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 hardly live in such a media; 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 enormous doses the most active bacteria are rendered powerless.

12. 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. One might almost say "the reverse," if cleanliness was not always a commendable feature. 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, from the point of view of fermentation mishaps which it suppresses by destroying the cause, it gives absolute security in working.

13. *Aseptic fermentation in beet distilleries.* — Barbet's plant (Fig. 7) meets all the objections sraised in sections 11 and 12 above. AA are the measuring tanks of the diffusion or maceration juice. The whole of the juice is sterilised at a temperature bordering on 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 seizes by methodical exchange the heat of the sterilised juice issuing from C, and entering by the valve R into the tubular vessel; the very methodical refrigerator V completes the refrigeration of the juice. The tubular vessels before use are carefully sterilised by steam (valve J on the recuperator), as well as all the connection pipes, the effect of the steam is completed by the injection of a little formol instantaneously diffused by the steam in 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-air gutters, 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

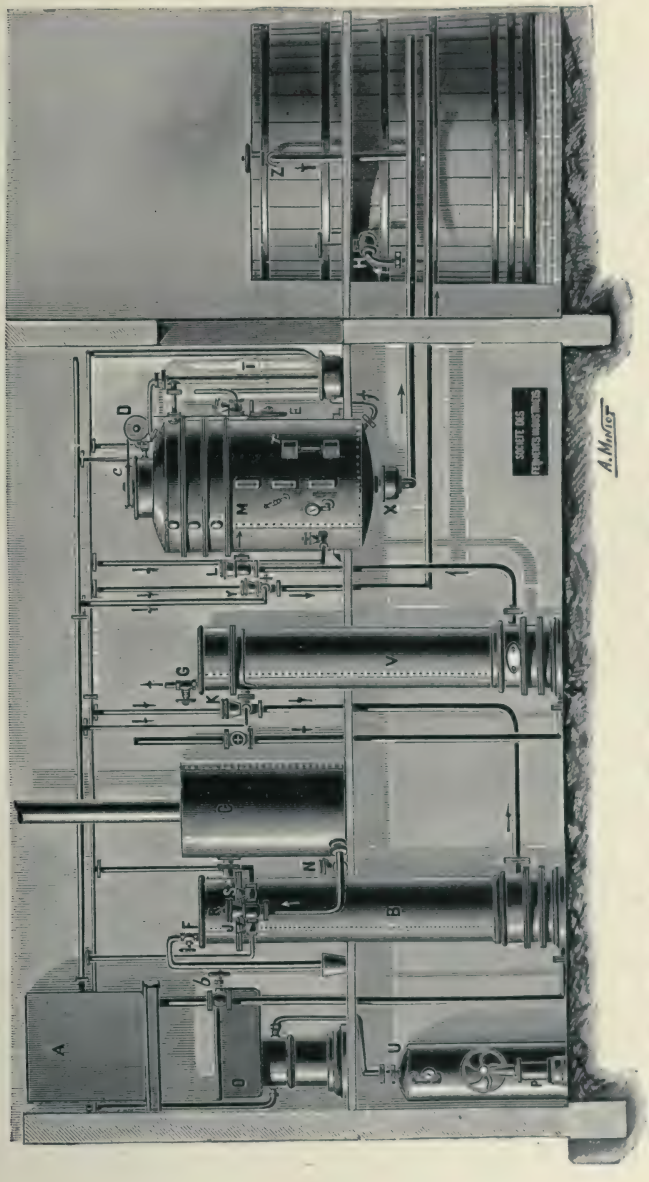


FIG. 7.—Plant for preparing pure aerobiose yeast for aseptic fermentation of beet juice (E. BARBET).

the juice to be made at as high a density as may be desired, whilst now it is rather difficult to ferment at 4°. Beet juice becomes as docile as molasses, which ferments at much higher densities. Now, as the expense of distillation is proportional to the volume of the wash, there is great advantage in economising one or two tuns per day, which effects much more economy than sterilisation entails expenses. 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 in distillery working if desired (Fig. 8). Half the acid is economised owing to sterility of the juice and its smaller volume; the juice does not require such strong protection against bacteria. Another advantage consists in not putting any acid at all in the diffusers and diffusing with water if desired, as 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 start of the season, the first "pied de levain" can no longer refuse to work. It is well to bear in mind 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 4–6 hours are required to form each batch of yeast, consequently 4–6 batches of yeast are produced per 24 hours. If there are many tuns, there will be only one batch of yeast 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 wrought regularly, yielding the advantages just summarised. They have been able particularly with pure yeast to bring the juice quickly, and feed the tun rapidly without killing the fermentation, which would infallibly be the case with juice not freed from its saccharogenic diastase.

14. Fig. 8 shows a general view of the interior structural arrangements, and the correlation of the individual machines, stills, and other organs 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 as nearly in an automatic manner as practicable.

I. MOTOR 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.

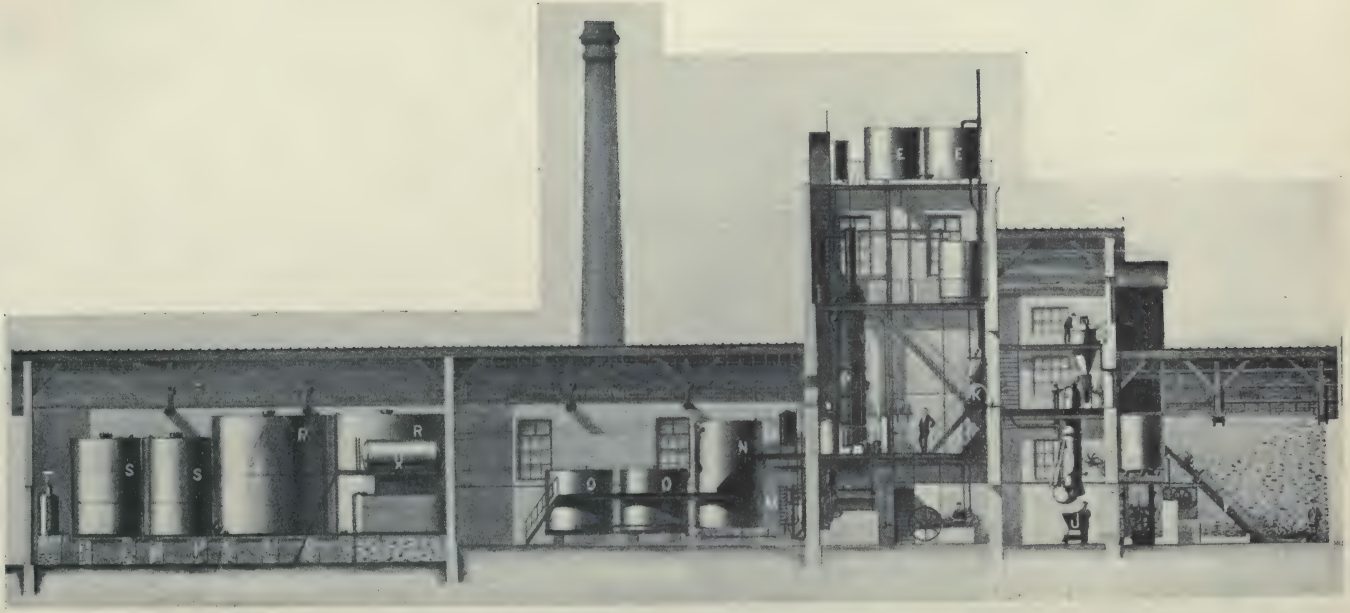
III. WASHING AND UN-STONING OF THE BEETS—BEET-SLICING MACHINES.—F, Elevator which lifts the dirty beets fed into it at the

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 and Grangé's simplified diffusion, 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 prior to fermentation. M, Combined cooler and heater, to cool the vinasse to 75°–80° 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° C. the fresh juice prior to fermentation. This combined cooler and heater (sec. 7) is of the multiple and removable tubular bundle type of the type (Fig. 3) used by Egrot and Grangé 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 more easy to manage; it produces, on the one hand, and as principal product, a perfectly rectified alcohol, marketable forthwith as such 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 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



Rectified alcohol warehouse. Continuous fermentation. Continuous direct distillation rectification. Beet-slicing, simplified diffusion. Beet-washing elevator.

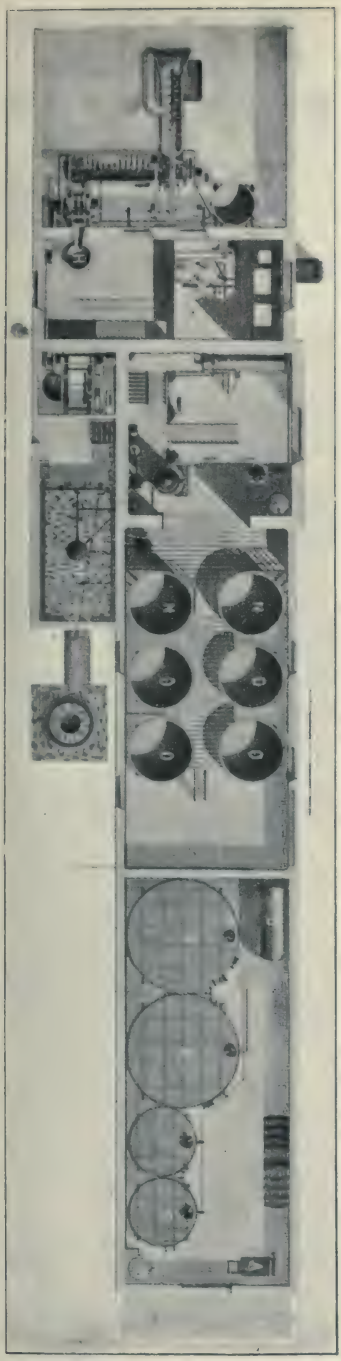


FIG. 8.—Agricultural distillery (beets) installed on the farm of Dammard, belonging to M. Potel, farmer, near La Ferté Milon (Aisne), France, on Guillaume, Egrot, and Grangé's system. Capable of treating 50 tons of beets in 24 hours. Interior view showing structural arrangements, with ground plan of same. (Details are given in sec. 14.)

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.

15. 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 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

FIG. 9.—View of rear part of beet distillery, showing silos, mud-settling ponds from washers (EGROT and GRANGÉ). (See Fig. 4 ; Fig. 8, G.)

steadings. It will be seen from Figs. 10 and 11 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.

16. *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 to 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

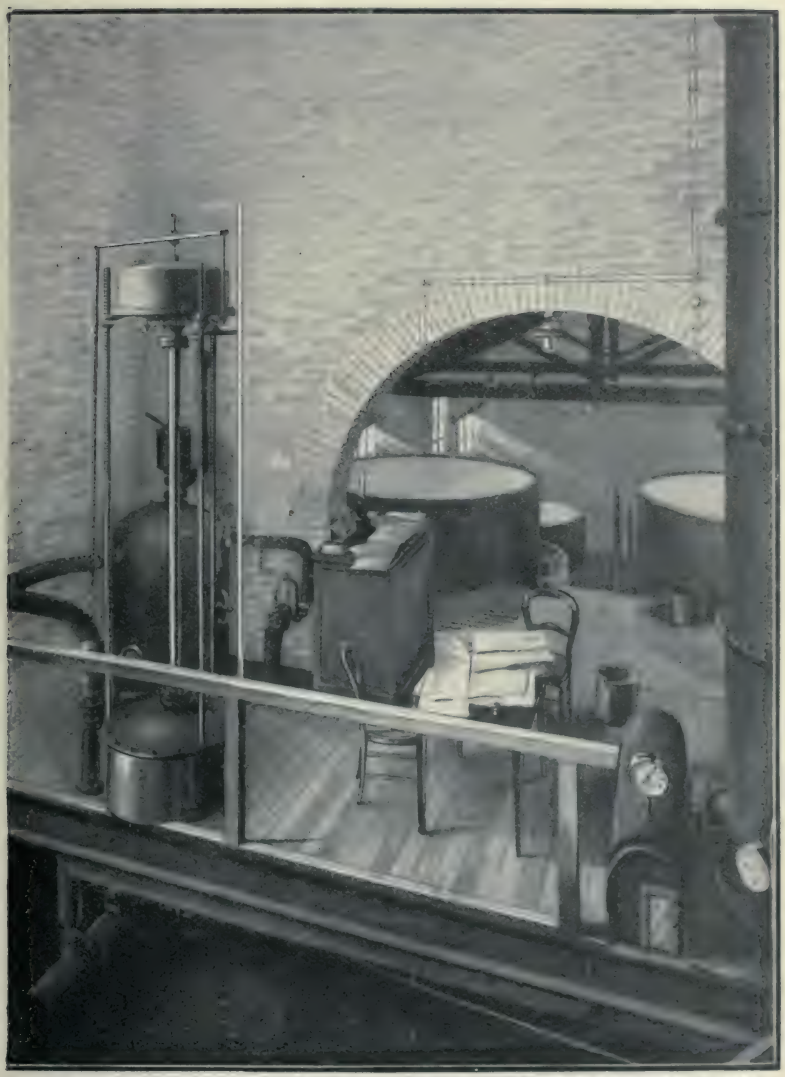


FIG. 10.—Point from which foreman distiller controls plant (shown in Fig. 8), showing Guillaume's steam regulator and dial thermometers of (*a*) inclined distilling column, (*b*) bottom of rectifier (EGROT and GRANGÉ).

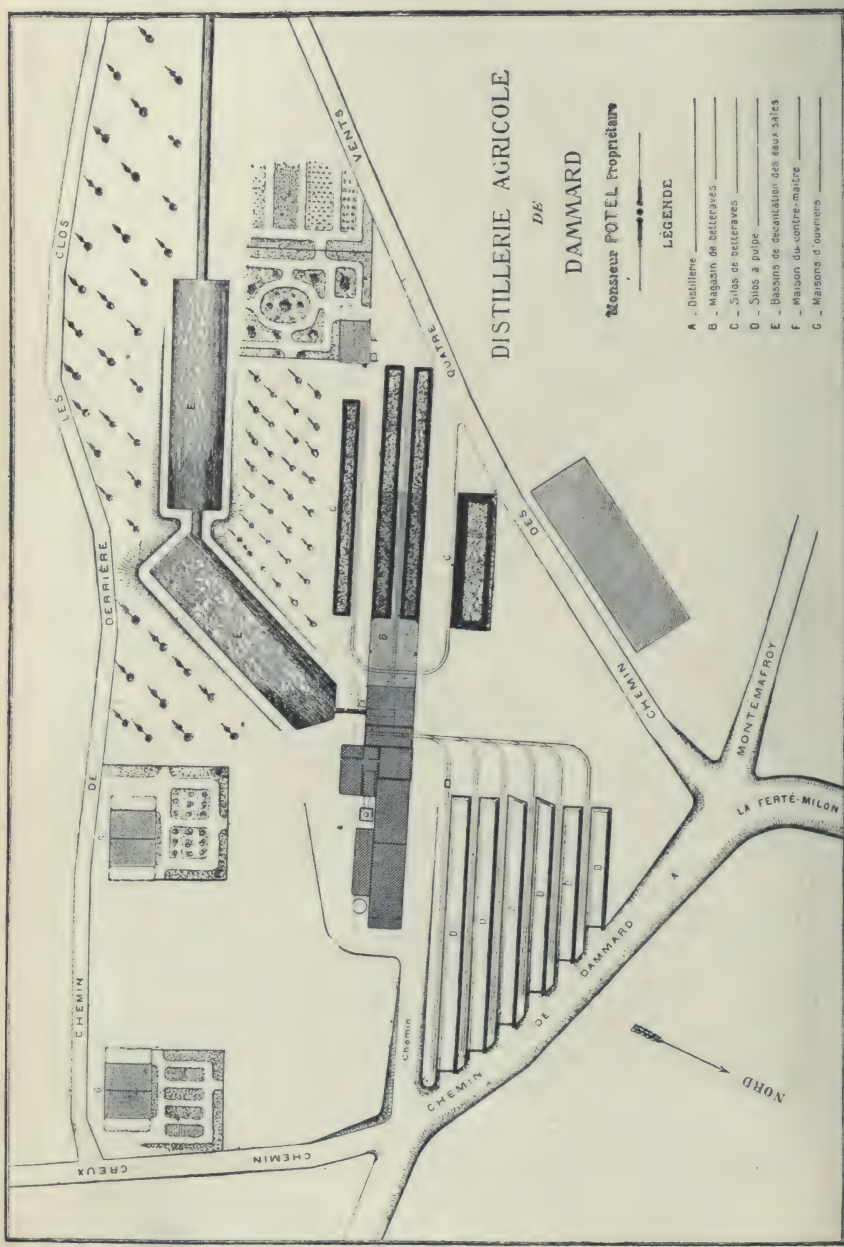


FIG. 11.—General plan of an agricultural distillery and its surroundings (EGROT and GRANGÉ). A, distillery; B, beet store;

FIG. 12.—Exterior view of beet distillery (Fig. 8), 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 (BERGOT and GRANGE).

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° (say, 8½ lbs. of coals per gallon of absolute alcohol).

17. *Application of Barbet's pure fermentation process to the*

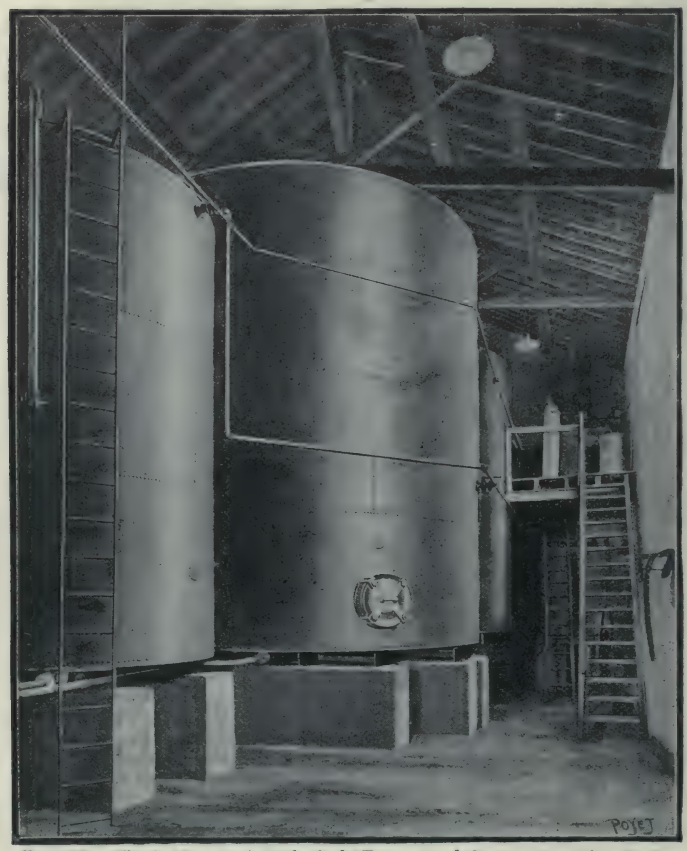


FIG. 13.—Tanks for storing alcohol (EGROT and GRANGÉ). (See R, S, Fig. 8, sec. 13.)

distillation of beet molasses.—The first idea would be to work in an analogous manner to that for beets (sec. 13, Fig. 17), replacing the 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 the sterilisator would actually be brought to the boil so as to denitrate the molasses. But denitration is not accomplished well in dilute washes, because the acidity is not strong enough. The process is therefore modified as follows. At A are two dilution vats to 28°–30° Bé, with a little water and the whole of the sulphuric acid required for fermentation. *b* is a regulating feed tank. B is the recuperator (forewarmer). The molasses heated to about 80° C. by the fore-

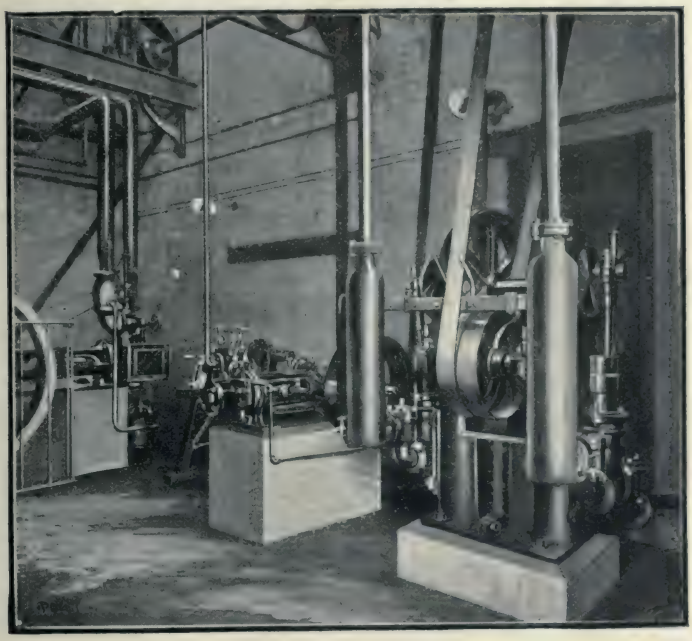


FIG. 14.—Pumps and motor power arrangements (EGROT and GRANGÉ). (See C, Fig. 8, sec. 13.)

warmer enters the continuous denitrator C, where it is boiled 15 to 20 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 sort of mixer before entering the thinning vessel. The dilution temperature

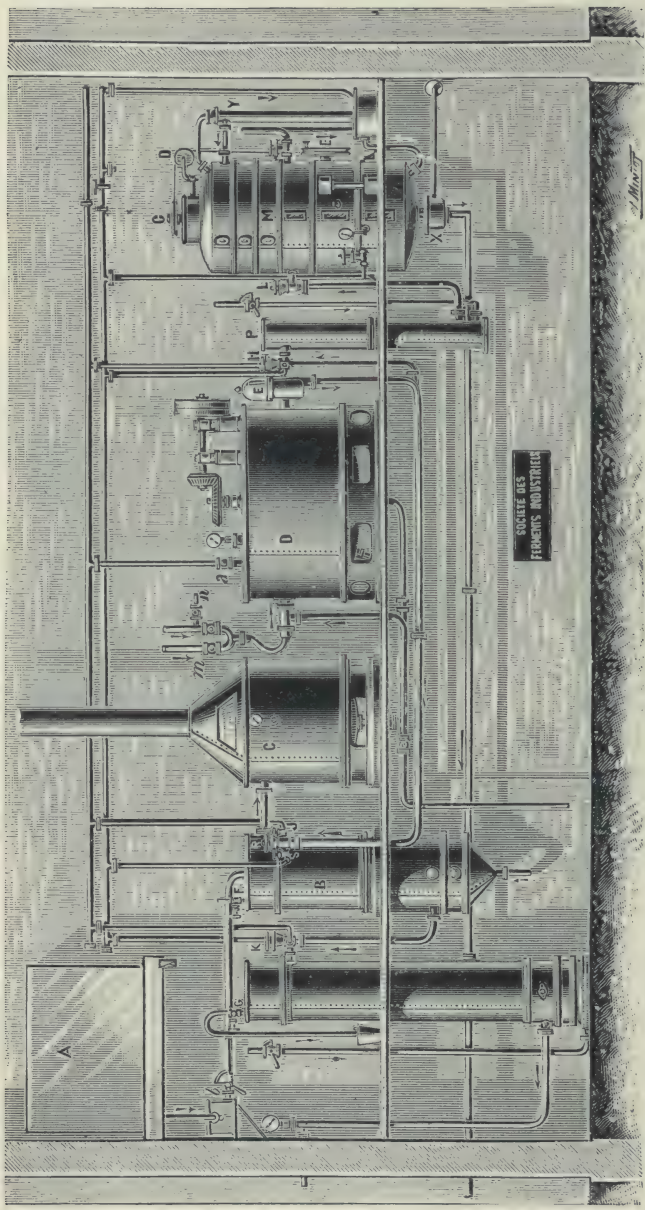


FIG. 15.—Plant for aseptic fermentation of beet-sugar molasses (E. BARBET). A, tanks to dilute to 28–30° B. ; *l*, regulating feed tank ; B, forewarmer ; C, continuous denitrator and steriliser ; D, mixer to dilute with hot water or hot spent wash ; *m*, spent wash tap ; *n*, hot water tap ; E, test-glass ; H, regulating valve ; M, pure yeast producer (Fig. 2) ; P, cooler for yeast apparatus ; V, refrigerator to left of B.

is about 80° C. It may be increased a little by injecting steam. But, in fact, owing to the presence of acid, the temperature of effective sterilisation is not very high, and the 80° effect a practically efficient purification for the short duration of industrial fermentation. At the exit is a test case 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 tuns. For the yeasts M direct draw-offs of wash are made on the mixer D, which for the occasion are reheated to 97°–98° 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 same activity of fermentation enables the same end to be obtained 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 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.

18. Instead of heating the distilling column by direct high pressure steam, a small triple or even double effect system working under pressure is 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 obtained gratuitously. With beets, Barbet's beet diffusion with the spent wash terminated by aqueous diffusion enables the pulp to be pressed, and thus produce 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 practic-

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

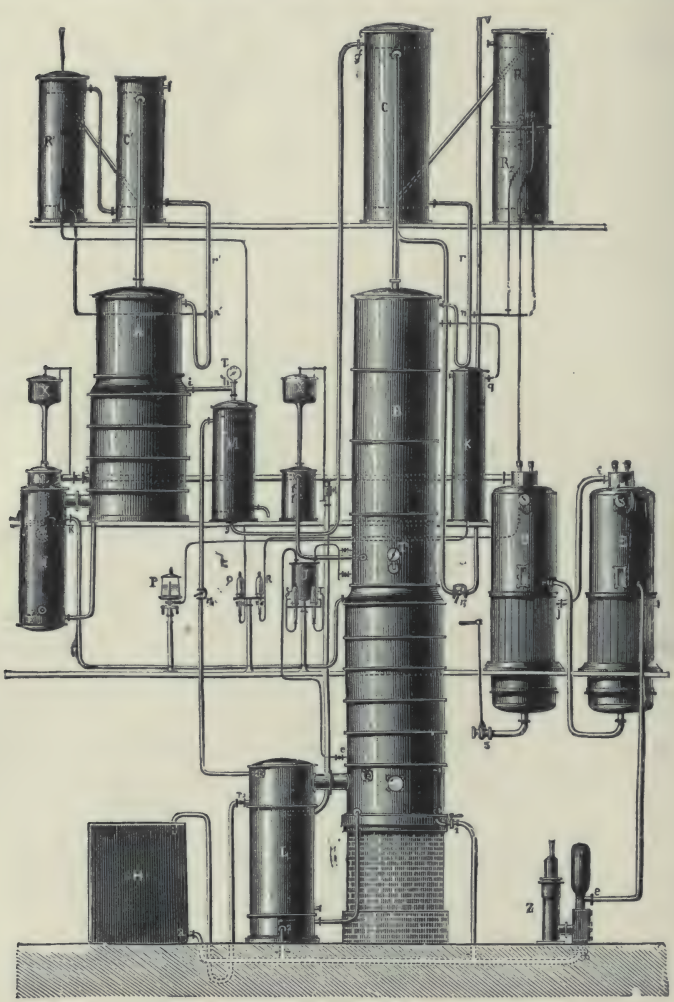


FIG. 16.—Direct distillation rectification plant (heated by steam from triple effect used to evaporate vinasse). A, preliminary purifier; B, rectifier; C C′, condensers; D E E, multiple effect vinasse evaporators; K, pasteurised alcohol condenser; R R′, refrigerators (E. BARBET).

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, less aqueous, is better for cattle, especially in the case of potatoes. Freight is three times less, and a stock of food is available for many months after the season is over.

19. *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. 8 contravenes our Excise laws in every direction.

CHAPTER IV

THE MANUFACTURE OF INDUSTRIAL ALCOHOL FROM GRAIN

1. The cereals (corn, grain) are the fruit or seed 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 seed 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 benighted districts. The grain is then separated from the straw by threshing and from the outer envelopes by winnowing.

2. *Storage of grain, and its liability to damage during storage.*—Grain is capable of being damaged in many ways during storage, whether from the attacks of insects or the development of fungi, or even from the germination of the seed itself. It is preserved by covering it with substances capable of killing all living germs, whether by depriving it of moisture, air, or heat. The chief cereals are wheat, barley, oats, rye, maize, and rice.

Table XI. 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
Gum and sugar .	3·8	4·2	5·7	11·3	2·9	1·6
Albumenoids .	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
Total . .	100·0	100·0	100·0	100·0	100·0	100·0

3. THE MANUFACTURE OF ALCOHOL FROM GRAIN BY SACCHARIFICATION BY TORREFIED OR KILN DRIED MALT.—*Malt* is barley which has been made to germinate to a certain extent, after which the process is stopped by heat. In germinating grain there is developed a small quantity of a white, insipid, nitrogenous substance termed *diastase.* Diastase is a soluble ferment, and it possesses the property of causing starch, which is naturally insoluble, to ferment and become soluble—*i.e.* by being changed into dextrine and maltose (sec. 5). The barley is steeped in cold water for about 50 hours at a constant temperature of 14°–15° C., and is then made into a heap, or *couch*, upon the malt-floor—a floor of slate or cement—until it germinates. Here it absorbs oxygen, and evolves carbonic acid; its temperature augments, and then it is occasionally turned, to prevent its becoming too warm. In this process the *radicle* lengthens, and the *plumule*, called by the malsters *acrospire*, elongates; and when it has nearly reached the opposite extremity of the seed, its further growth is arrested by drying at a temperature slowly elevated to 150° F. or more. This slight torrefying 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; when, therefore, malt is intended for the brewer or distiller, care should be taken not to heat it too much, otherwise its saccharifying power will be diminished, as shown below.

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

	80° C.	100° C.	120° C.
Maltose	37·01	52·44	51·32
Dextrine	14·92	18·49	19·35
Lactic acid	6·56	0·49	0·31
Soluble albumenoids . . .	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

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, of which

1·5 is carried off by the steep-water.
3·0 dissipated on the floor.
3·0 roots separated by cleansing.
0·5 waste.

8·0

The system of pneumatic malting more especially in vogue on the Continent is for the moment beyond the limits of this treatise, and falls to be dealt with more appropriately in a treatise on potable alcohol. According to Cameron, from 3 to 6 per cent. of the weight of 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 contained a peculiar substance, insoluble in hot water, which he called *hordein*, and which, during malting, is diminished in quantity, and 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."

4. 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—*dextrine*, 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 barbarous 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 molasses or starch, or unmalted corn, by mixing them with from 10 to 50 per cent. of malted grain.

5. *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, and 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 altered. 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.

6. The manufacture of alcohol from grain, etc., naturally divides itself 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 Spirit.
1 quarter of barley malt yields	18
1 „ „ malt grain „	20
1 cwt. „ sugar „	10
1 „ „ molasses „	7½
1 ton „ beetroot „	15

Ure quotes:—

	Alcohol of Specific Gravity 0·9427 $\frac{6}{11}$ of British Proof Spirit in lbs.
100 lbs. wheat	40–45
100 „ rye	36–42
100 „ barley	40
100 „ oats	36
100 „ buckwheat	40
100 „ maize	40

or the mean of the whole at 40 lbs., say, $4\frac{1}{4}$ 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 $20\frac{1}{4}$ lbs. of the mixed grist, or very nearly 5 proof gallons per 100 lbs. 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 lbs. of the mixed grist; and sometimes, in favourable seasons, he asserts that working with high class corn the produce even exceeds the $17\frac{1}{4}$ lbs. mixed grist, producing 1 gallon of proof spirit. Ure gives the following proportions as used by some experienced Scotch distillers, 250 bolls, containing 6 bushels each, being used for a mashing consist of—

25	bolls	oats	weighing	284	lbs. per boll,	or $47\frac{1}{3}$	lbs.	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 boll weighing 291 lbs., 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 lbs. 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 weekly production from that quantity is between 19,000 and 20,000 gallons of proof spirit per week. At 50 lbs. per bushel, a yield of 20,000 gallons per 1000 qrs. of grain exactly corresponds to 5 proof gallons per 100 lbs. of grain.

7. Barley, therefore, is the predominant grain used in distilleries for making potable spirit in Britain. But maize and molasses are the chief raw material 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 merely mixed with a certain amount of barley malt to induce the conversion of the starch of whatever grains may be used 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 sorts of grain instead of using all of one sort, 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, however, is malted grain, a much more limpid wort is got than that obtained 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 product at the same cost of materials to the distiller. When, besides malt, barley is the only other grain used, 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 peculiarly adapted for mixing with wheat to keep the meal open in the mashing.

8. *Mashing.*—Raw grain and unmalted barley are ground to meal by millstones, but malt is simply crushed between rollers. To facilitate drainage of the mass some oat husks are added, *i.e.* if as much as 87·5–90 per cent. of barley be 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 (Fig. 17). 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 tested every half-hour during mashing, it will be found to become gradually sweeter, to all appearance thinner, but in reality more dense. The wort is drawn off from the grain whenever it has reached its maximum density, which seldom exceeds 150 lbs. per barrel, *i.e.* $\frac{360+150}{360} = 1{\cdot}42$, 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 everything soluble, 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 upon a fresh quantity of meal and malt. With the above proportions of malt, raw grain, and water, the first infusion may have a strength = 20 per cent. = sp. gr. 1·082, or 73 lbs. per barrel, the second of 50 lbs. per barrel or 14 per cent., and the two together

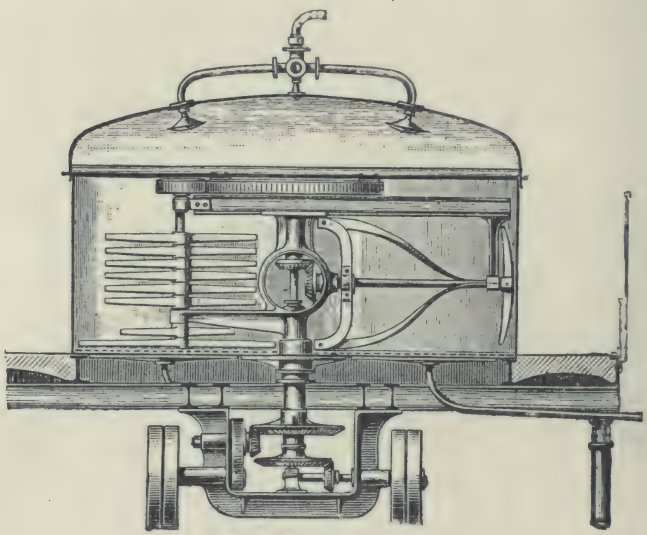


FIG. 17.—Mash tun.

would have a strength of 61·2 lbs. per barrel = 17 per cent., sp. gr. 1·070. But direct experiments on a larger scale show that no more than four-fifths of the soluble saccharo-starchy matter of the worts is decomposed in the best regulated fermentation of the distiller from raw grain. For every 2 lbs. 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 lbs. of such alcohol, it will take twice 4·6 lbs. of saccharine matter to produce the said gallon. To these 9·2 lbs. actually converted into alcohol, one-fifth, *i.e.* 1·84 lbs., must be added, which will raise to 11·04 lbs. the amount of solid matter employed in producing a gallon of above spirits.

9. *English infusion process—Factors of hydration*[1] (GRAHAM).

TABLE XIII. INFLUENCE OF TIME.
(Malt, 100. Water, 1000. Temperature, 145° F.)

No. of Malt.	Duration of Mash.	Maltose.	Dextrine.	Total Sugars.	No. of Malt.	Duration of Mash.	Maltose.	Dextrine.	Total Sugars.
	Hrs.								
1	½	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	½	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	½	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 XIV. 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	59·65
,,	150	48·61	13·83	62·44	,,	160	39·90	18·06	57·96

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 2 hours at the respective heats.

TABLE XV. 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[2]	35·70	16·18	57·88

[1] For influence of kiln-drying temperature, see Table XII. p. 63.

[2] 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 XVI. INFLUENCE OF NATURE AND RATIO SUBSTITUTE OF MALT, OR PLUS SUBSTITUTE, 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	IV.	T	50·87	14·43	65·30
	H	57·69	1·73	59·42		U	48·10	17·05	65·15
II.	I	47·62	17·38	65·00		V	45·11	19·88	64·99
	J	46·15	18·48	64·63		W	49·99	18·94	68·93
	K	42·50	22·11	64·61		X	46·87	19·98	22·07
	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.

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 albumenoids of barley; with rice boiling is essential.

10. *Manufacture of alcohol from grain by saccharification by green malt.*—Formerly the distillation of fermented grain wash 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 improvements 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 wrought 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 and in the method of working and use of sulphurous acid, the constructors claim that the yield obtained may be increased to 36 litres of alcohol per 100 kilos of maize,[1] and 10 to 12 litres of alcohol at 100 per cent. per 100 kilos of potatoes,[2] 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 one kilo of meadow hay. To produce 5 tons of dry dregs in 24 hours, 4 filtering tanks are required. These dregs may also be transformed by hydraulic presses into dry edible cakes in the proportion of 25 per cent. of its weight, and into oil of maize in the proportion of 2 per cent.

11. *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 with certain additions to their existing plant for making alcohol 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 lbs., or well on to 20 per cent. more.

A *cuiseur saccharificateur*, or boiler saccharifier, is shown in Fig. 19, in which by appropriate treatment the starch in grain or corn may be transformed into glucose. The entire grain is submitted in presence of water to a pressure of 3 atmospheres; then, when the grains are completely transformed into paste, acid is forced into the boiler from the globular vessel A by steam pressure from the boiler. Saccharification is complete in 15 to 20 minutes. This plant is made in all sizes, according to the scale on which the operations are conducted. A

[1] Say, 3·6 bulk gallons of 100 per cent. alcohol per 100 lbs., or 6·3 proof gallons.
[2] Say, 1·2 bulk gallons of 100 per cent. alcohol, or 2·1 proof gallons per 100 lbs.

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 lbs. of maize 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

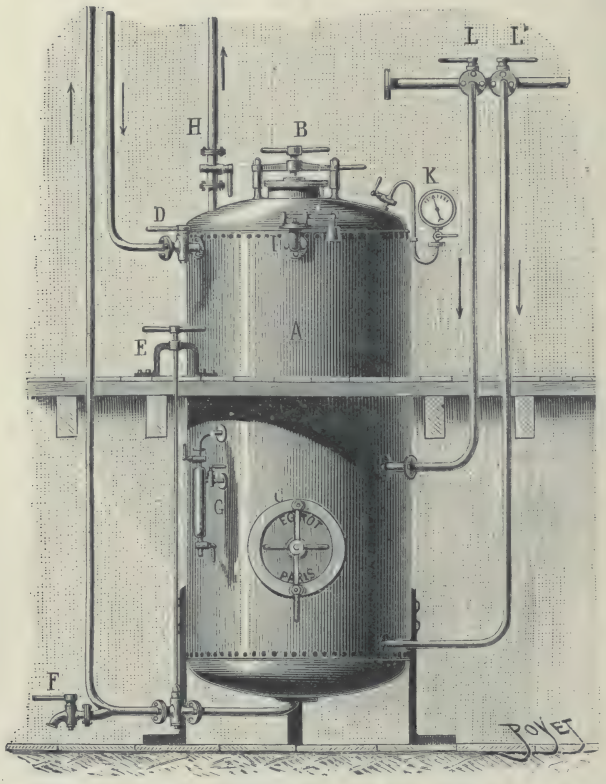


FIG. 18.—Digester for saccharification of grain, etc., with acid under pressure (EGROT and GRANGÉ). A, Digester; B, C, manholes; D, feed pipe; E, rod for manipulating bottom discharge valve; F, sampling pipe; H, discharge pipe; K, pressure gauge; LL, steam pipes.

increased to 3 atmospheres. This pressure is kept up for 2½ hours, and the state of the paste is then ascertained. When the paste is well formed, concentrated muriatic acid is run in the proportion of 2½ per cent. on the weight of the maize being treated. Saccharification takes 25 minutes, and the worts obtained in this way are very pale. The yields in glucose and dextrine are respectively 68 and 1·75 per

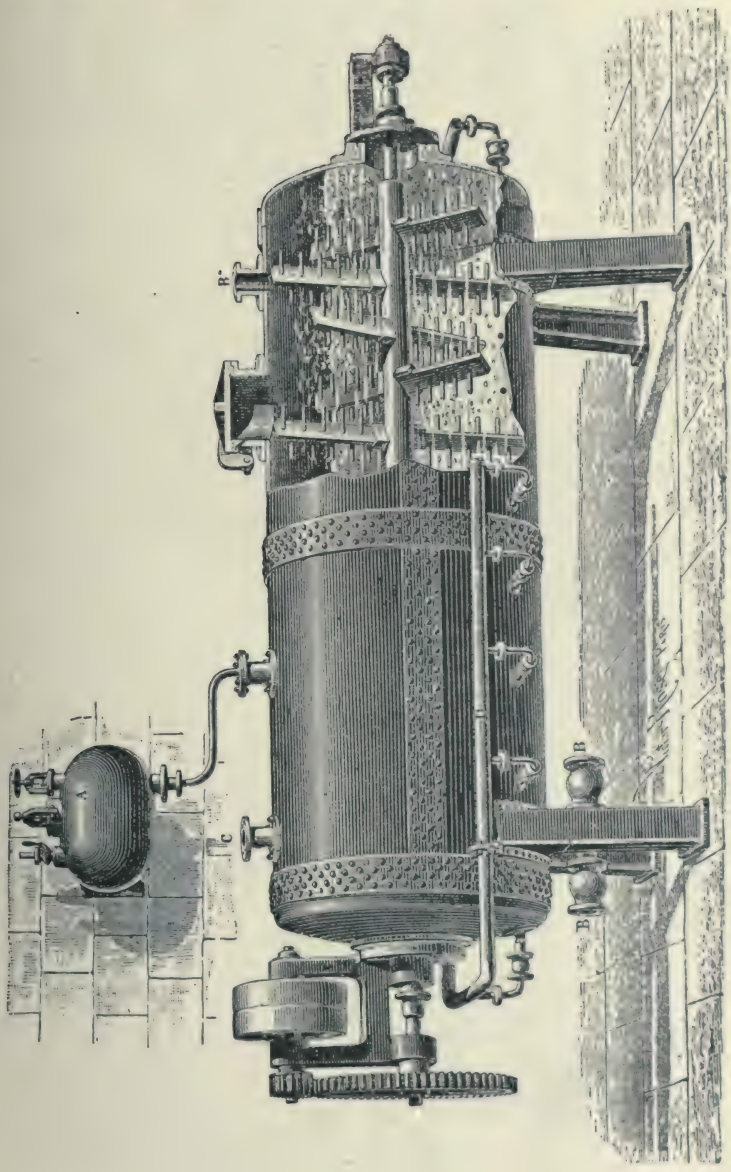
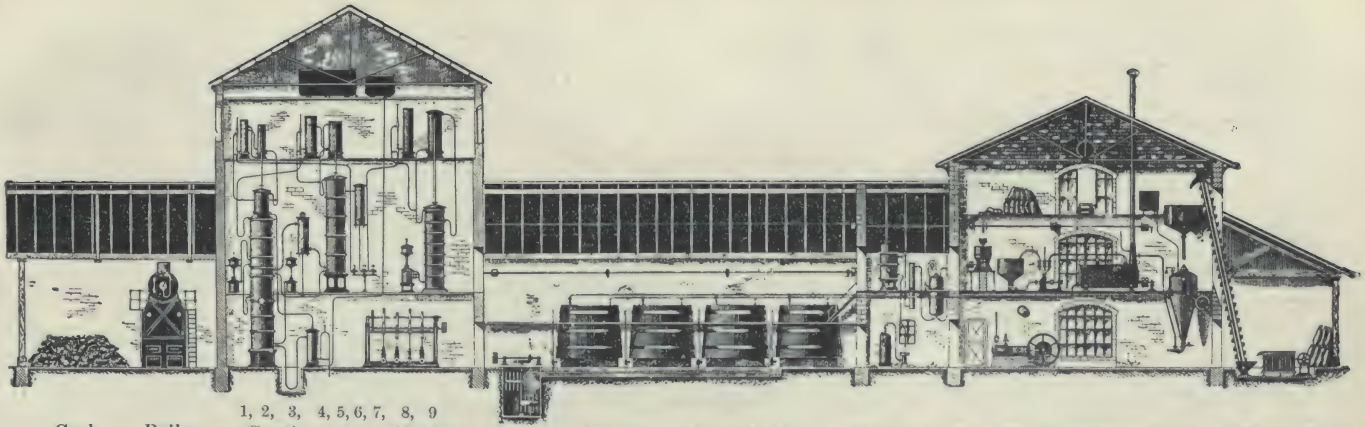


FIG. 19.—Horizontal mixing saccharifier of grain by acids (WAREIN and DEFRANCE).



Coal.

Boiler.

Tubular boiler.

1, 2, 3, 4, 5, 6, 7, 8, 9
Continuous rectification distillation department.

1 Refrigerator, steam regulator.
2 3 Condenser; purifying column, first runnings (aldehydes and ethers) eliminator.
4 Steam regulator.
5 Cooler, special condenser recuperator or forewarmer.
Water tank, condenser, and rectification column.
6 Special condenser, test vessels in glass cases, with floating hydrometer.
7 8 Reservoir for raw spirit from column.
9 Refrigerator steam regulator.
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.

Air sterilizer.
Pure aerobiose ferment vessel —Air vessel.
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.

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

FIG. 20.—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.

cent., and 3½ British imperial gallons of rectified alcohol of 90° are obtained per 100 lbs. of maize. The distillation residuals (dregs) passed through the filter press are dried and pressed in a hydraulic press so as to extract the oil which they contain.

The weight of oil obtained per 100 lbs. of maize is as follows:—Oil 2·3 lbs., or, say, 1 quart, leaving 10 lbs. of maize cake.

The oil is sold to soap makers at about 18 shillings a cwt., and the cakes are sold *as manure* at about £3, 10s. a ton.

Maize, rice, etc., and 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.*

12. The spirit produced in Britain for methylation is substantially the same as that produced for consumption. At one time a different material may be used, but at any moment the distiller might be forced to use the same. Distillers who produce only for methylation generally use molasses, or very largely use 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 has been affected by the price of sugar. It has risen rapidly, so that it might at any moment become prohibitive for this purpose. Less plant is used 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. As to the question whether the spirit produced for methylation could be used for consumption by being blended but 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.

The cost of *plant* on Barbet's principles, as shown in Fig. 20, to treat 50 tons of grain daily, or say 2000 quarters per week, with a yield of 3·6 bulk gallons of alcohol calculated to 100 per cent. per 100 lbs. of grain, and an hourly consumption of 6½ to 7 metric tons of steam, say one ton of coal per hour, or about one-half ton coal per ton of grain, working continuously day and night, Sunday and Saturday, is £14,000. But, owing to our Excise laws, the cost of both plant and buildings is about trebled, and continuous working is forbidden.

CHAPTER V

THE MANUFACTURE OF INDUSTRIAL ALCOHOL FROM POTATOES

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

Table XVII. Showing Composition of Unimproved Varieties of Potatoes.[1]

Kind 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 very greatly indeed. Davy obtained from 18 to 20 per cent. Körte obtained, as the mean result of the examination of 55 varieties of potato, a percentage of 24·9 solid matter; the average of the starch was 11·85 per cent. Those potatoes keep best in which starch is most abundant; but the starch diminishes, probably passing into gum and sugar, by keeping: thus, from the same variety of potato, Payen obtained 17·2 per cent. of starch in 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 wrought wonders. Vauquelin examined 47 kinds of

[1] This table is given to show by contrast the increase in starch content effected by pedigree selection in the present-day distillery potatoes. See p. 82.

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-producing element of potatoes.

2. Alcohol may be produced from potatoes either directly or indirectly. By the first or direct method the starch of the potato is fermented without having been previously separated from the potato as starch, 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 less 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 pap 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 more readily, in less amount. Beets or carrots were said to improve the flavour and increase the quantity of the spirit. After the fermentation was pushed to its fullest extent the wash was distilled in the ordinary way. In Siemens' process (which was said to produce half as much spirit again as the ordinary plant used in Germany), and which was at one time used in Denmark, 3 to 4 tons of potatoes were steamed a little over 212° F., then mashed in the steaming vessel by a revolving iron cross and warm water in quantity to form a thin paste and rendered slightly alkaline by $1\frac{1}{4}$ lbs. of caustic potash added. The resulting starch paste 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 in 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—198 imperial gallons of spirit. Cadet stated that 800 lbs. of potatoes yielded 30 lbs. of spirit, costing the distiller 36 francs, and selling for 48. From 50 kilogrammes (110 lbs.) 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° 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 some short time from 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 seven or eight 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°, 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° gravity was obliged to be used, otherwise the worts at the gravity of 40° 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.

3. At the present time alcohol is largely distilled from potatoes 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 (Fig. 17, Chap. IV. sec. 8), 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. 41 and 42). 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, and 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, and the quantity of spirit distilled is 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 little 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 on 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. Out of the total number of agricultural distilleries in the German Empire there are not more than some 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: and 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 potatoes they would realise £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 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 Gährungsgewerbe 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 is about 20s. per ton. The yield of alcohol from a ton of potatoes may be taken at about 25 gallons of pure alcohol or about 44 proof gallons.

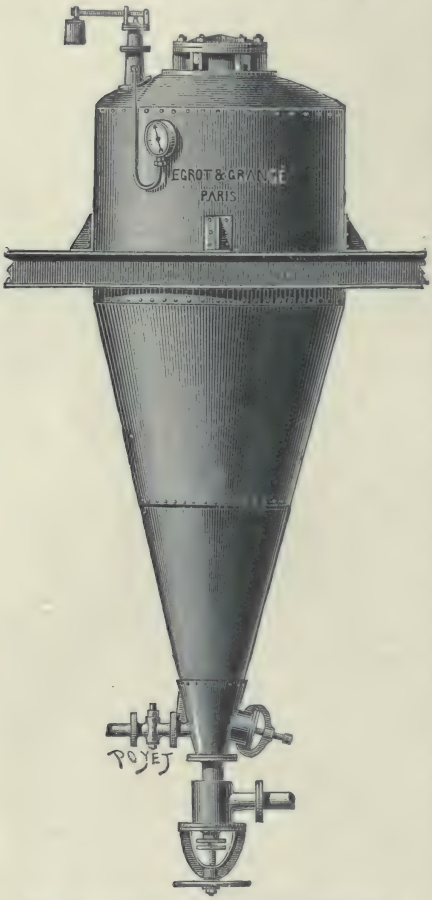


FIG. 21.—Autoclave for saccharification, by malt, of potatoes under pressure (EGROT and GRANGÉ).

4. 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 in Germany of 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 is fairly fertile in many parts, 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 people who send the potatoes may be paid according to their weight. It is a weight recorder very much like the weighbridges used for automatically weighing corn. It tips itself and cuts off the supply when charged with a certain amount, be it 1 ton or 3 tons. The spirit as it leaves these agricultural distilleries is not at all 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; say, 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. So the actual yield from a cwt. of potatoes is 2½ proof gallons, or, roughly, two-thirds of the theoretical yield. Several of the distilleries near Munich get from 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 just 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.

5. 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 is roughly 27s. per imperial ton—a high price, but potatoes were 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 are at times in Berlin even cheaper than 20s. per ton for alcohol purposes. The price of potatoes for domestic consumption is usually somewhat higher.

6. 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 one farmer will have a distillery. In Saxony a large farmer may have his own distillery. A single farm in Germany is sometimes able to keep a distillery supplied. Some of the large estates in Bohemia and Austria

possess distilleries. But, as a general rule, in Bavaria it takes more than one farm, or even estate, to keep a distillery going. In East Prussia the farms are large, and that is where potatoes are mainly grown, owing to the sandy nature of the soil, and there one 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 percentage of starch. That is estimated by a not very exact method in some cases, but by means of the specific gravity, and then by using Maercker's formula, so that if one person sent in potatoes containing 20 per cent. of starch, the price would be regulated accordingly. A certain standard is thus taken as the basis, and for every unit per cent. of starch more, a certain amount more is paid per metric ton or centner. In one distillery visited by Dr. Hinchcliff, besides barley, they were using a mixture of imported maize and potatoes, 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 sorts grown for household consumption, but still 7 tons is considered a satisfactory yield. About 6 tons per statute acre is something like an average crop in Britain, and about 4 tons per statute acre in Ireland, but it is not considered a good crop under 7 tons.

7. The quality of the potato for distillery purposes has risen of late years, those having a high starch content have been chosen for planting. The duty is not imposed with the object of encouraging the production of a high class quality, but rather with the idea of encouraging potato culture. The duty, in fact, is not imposed in relation to the weight of the raw material that is dealt with in an agricultural distillery, but is only imposed with regard to the output of alcohol; but the capacity of the mash-tun is also taxed, so that it is to their advantage, to a certain extent, to put in as concentrated a mash as possible, and hence to put in a potato that has as high a starch content as possible. At the Alcohol Exhibition in Vienna, several varieties of potato were exhibited that had been tested at some of the experimental stations and found to contain over 22 per cent.; 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 dextrine. The relative quantities of maltose and dextrine should be as wide as possible, and in that respect not only the starch content, but also the character of the potatoes as regards the dextrine, has been improving. There is a prevalent misunderstanding anent possibility of converting diseased potatoes directly into alcohol. But this can only be done by first separating the starch from the unsound portion of the potato. But the introduction of a potato starch stage to work up diseased potatoes *indirectly* into alcohol would be a very doubtful economy.

CHAPTER VI

THE MANUFACTURE OF INDUSTRIAL ALCOHOL

From Surplus Stocks of Wine, Spoilt Wine, Wine Marcs, and from Fruit in general

1. 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 sarmentose plant which flourishes in moderately warm climates, and which comprises several varieties, termed *cepages*. The quality of the wine depends not only on the variety, but also on the soil, its exposure to the sun or aspect, the manure, the nature of the ferment, and the care exercised in the vinification. 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 so used 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 is 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, off-hand, when the juice is saccharine.

2. *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 fermentation is favourised 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

TABLE XVIII. 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 at 100°.	Extracts Vacuo.	Ash.	Tartar.	Reducers as Glucose.	Sulphate of Potash.	Acidity as H^2SO^4
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 *blanc*, 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	1·68	3·49	0·91	0·25	7·05
,, *blanc*, 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	4·88
Loire-Inf. Nantes *blanc*, 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
,, *blanc*, 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, Andrinople, 1878 .	11·4	22·9	29·6	2·47	2·06	5·00	0·71	3·10
Piquette, lixiviation of, 1883, marcs, Midi .	5·9	17·9	20·8	4·68	3·59	traces	2·75	4·07

ceased, the liquid is drawn off whilst it is still mild, and the transformation of the sugar into alcohol finished in casks. The skins left behind retain a certain amount of saccharine juice, and constitute the *marc*, which, 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, the result is *piquette* (sour wine). The casks of wine must always be kept full, but open at the bung hole, so that the evaporation surface is not too great, 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 be used, the process is almost 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 pellicle containing the colouring matter should not rest in contact with the liquid after fermentation sets in.

3. *Barbet's process of cultivating pure wine ferment.*—The application of Pasteur's principles to wine presents special difficulties. Up to now all that has been done is to despatch to wine-growers when wanted a few bottles of ferment germs, leaving the culture thereof to the wine-growers themselves when a second crop of ferment enables them to set their fermentation tuns. But all such delicate preparatory operations 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 its 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 pure ferment germs, but a small factory supplying each vineyard, and, for each vat, a pure copious leaven in full activity.

Fig. 22 shows a plan of a factory of this nature, which is supposed to be annexed to an existing wine-cellar. The necessary grape juice is supplied by the wine-press to the pure leaven. 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

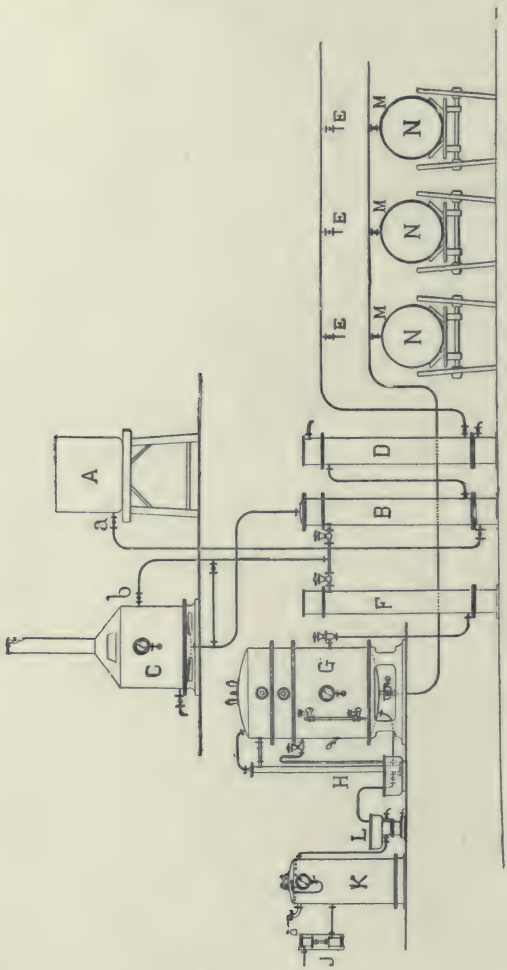


FIG. 22.—Plant for production of pure aseptic ferments for fermentation of wine.

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 sterilisator 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 *b*, 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 leaven apparatus G, in which 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 recommended by Pasteur is thus realised. The air sterilisator 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 without any delicate manœuvring. J is the steam pump to compress the air. K is the compressed air receiver. The tubular vessels B D F are fitted with sterilisable tubular cases. N N N are galvanised iron casks tinned inside. They are intended for transporting the pure leavens into the other wine-cellars in the neighbourhood. 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 leaven drawn from apparatus G. Fermentation is allowed to go on, and the carbonic acid gas is not allowed to escape, only the casks possess a safety valve, gauged to 1 kilogramme per square centimetre. When the casks reach the wine-cellar for which they are intended, they are in full fermentation and under a pressure, which will be sufficient to elevate the leaven into the vats, to be leavened, without requiring a pump. This simple manœuvring avoids contaminating the leaven. Fermentation develops instantaneously in the vats, thus ensuring that no mishap occurs in fermentation—mishaps which occurred too frequently by the old methods. If there be in the region in which this system is being wrought two or three very distinct kinds of wine which require different strains of leaven to be used, it will suffice to have in the works two or three apparatus G, each with its own strain of leaven, all the remainder of the plant remaining as before. The continuous sterilisation of the juice is a perfect instrument for use afterwards in pasteurising the juice drawn from it. A double advantage is thus got from the same plant.

4. 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 with the too rapid increase in its annual production. The annual production of these two districts have exceeded the French annual consumption by 440 million gallons. This unsaleable merchandise, deprived, by fiscal enactments, of all chance of exportation, as such, must needs find a new outlet. The only outlet possible is 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, if its wine is not exportable, as such, its brandy is not affected by the same impossibility. It must not be forgotten, in fact, that 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 somewhat unscrupulous merchants blended the Charentais product, decimated by the phylloxera, with du Nord alcohol; and now, when abundance has once more returned, the French do not imagine it to be 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 see that their brandies are not generally esteemed, neither by direct customers nor by wholesale merchants, and that the latter prefer du Nord alcohol for making their cheap blends. If the industrial raw material is afflicted with an original unpleasant odour, on the other hand, it cannot be denied that its fermentation is surrounded with the greatest of care, and that it engenders few fermentation impurities. If we place in parallel 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. There is often found in wine alcohols a piquant odour, recalling burnt paper or burnt caoutchouc. It is acrolein, due to fermentation at far too high a temperature. This defect is almost general in Algerian spirits. Often, again, the dominant odour is of sulphurous acid, and sometimes to an unimaginable extent. If a solution of baryta be poured into this alcohol, an abundant precipitate not only of sulphite of baryta 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, it has formed very bad 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 determined, 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, methylic or ammoniacal compounds analogous to those given off by over-ripe cheese or fish. None of these grave 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 worry over purifying branyd by distillation. The apparatus must be combined in such a way that the plant can produce a sound and proper brandy even from the worst wines. Seek, then, the origin of the acetic, sulphuretted, and sulphurous ethers, the obnoxious presence of which has just been

determined. Chemistry tells us that ethers are formed each time that boiling alcohol of great strength comes in contact with an acid, mineral or organic, liquid or gaseous. Acetic ether arises from the acetic acid in the piqués 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 was of a rose colour.

5. 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. 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 been determined, which, under the influence of the acidity of the wine, disengage sulphuretted hydrogen. Acetic acid, sulphurous acid, sulphuretted hydrogen are three volatile acids, which on distillation are disengaged from the wine, and rise up into the upper parts of the distilling column at the same time as the alcohol becomes stronger and stronger, till the moment arrives when the strength of the alcohol is such as to enable it to seize hold of the acids which accompany it, and combine therewith, under 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.

6. 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 energetically 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 ablutions, 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. 25).

The scrubbers are almost always effective when rectified alcohol is desired, because it is necessary to obtain absolute neutrality of odour. When it is desired to make brandy, the reaction is less indispensable, in so far as the great vinosity of the product has a sufficient aroma in general to mask completely small defects.

7. *By-products.*—The vine contains cream of tartar, the price of which in the crude state is twice greater than that of alcohol; it is economically extracted by the same multiple effect processes as are used in sugar works (Fig. 25). Wine also contains a notable proportion of glycerine, 5–9 lbs. per 100 gallons, worth about £40 per ton. The glycerine in a day's turnover of 2200 gallons 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 working of the marcs, from the pressing, ought also to be industrialised, so as not only to extract all the spirit, but also to obtain cream of tartar and tartaric acid, then the wine-distiller ought to separate the seeds, which are 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 leavens, at the moment of pressing. Centralise all the marcs for distillation. Profit by use of apparatus for sterilisation of juice for pure leavens 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, sorgho, 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. 24, 25) would have work for nine months out of twelve, and consequently would be in excellent condition to maintain a capable technical staff and have 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 privilege of the Bank of France and that of the Bank of Algeria were only renewed on condition of placing an extremely important amount of capital at the disposal of agricultural enterprises of this nature.

8. *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 beck P (Fig. 23). 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, gratuitously, 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 3 or 4 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, taking its 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 through the plates E to the still D. There it finds a still, so capacious that the boiling of the vinasse is prolonged for 3 hours at least, allowing the volatile fatty 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 in intimate contact with the vapours of the wine, and to etherify them. But as these acids are not very volatile, it was to be feared that 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 fatty 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 beck 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 also be pumped to the wine beck 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 left 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 fatty 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, 2^1, or 2^2 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,

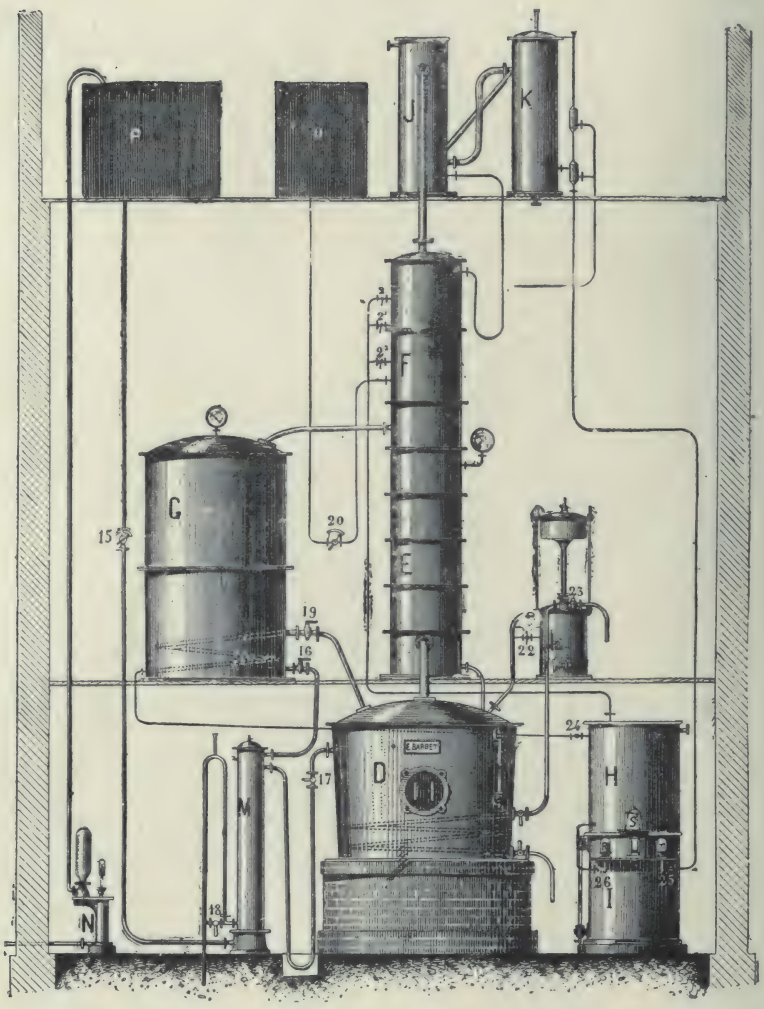


FIG. 23.—Plant for distillation of wine. D, still; E F, rectifier; G, wine boiler; J, condenser; K, refrigerator; M, wine forewarmer; N, pump; O, fatty acid liquor; P, wine tank (E. BARBET).

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

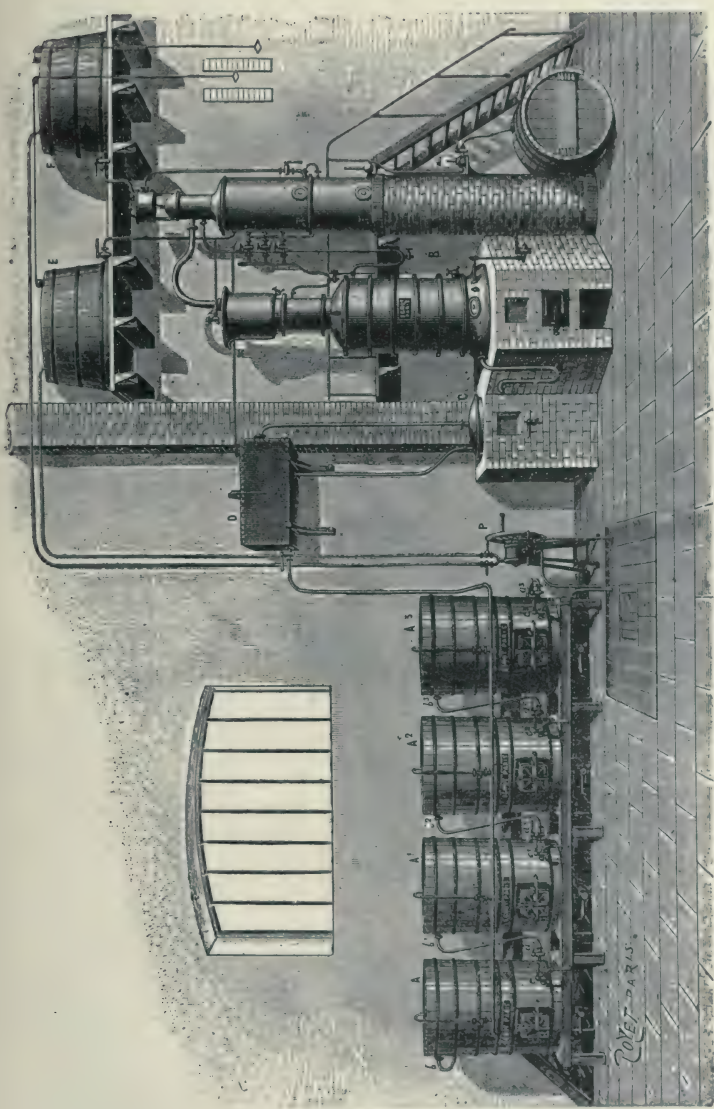


FIG. 24.—Plant for manufacture of alcohol marc brandy, from marc of wine (EGROT and GRANGÉ). A A^1 A A^3, maceration vats; a a^1 a^2 a^3 b b^1 b^2 b^3, connection pipes conveying juice obtained by maceration from the bottom of the one macerating tun to the top of the next; C C^1 C^2 C^3, taps for drawing off macerated juice; P, pump; D E F, maceration juice tanks; B, continuous still; G, rectifying column.

eliminate, it is drawn from the upper tap 2, whilst by the lower tap 2^2 a slight extraction is made 1 to 2 per cent. of volatile oils. It is there they concentrate. The plant is provided with a steam regulator.

9. In large establishments where the supply of marc to be distilled 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 on to 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 chased 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, and No. 2 becomes the first. Afterwards No. 2 will be the last, and No. 3 the first, and so on. This methodical maceration yields good results: it furnishes high strength piquette and completely exhausts the marcs. The piquette is pumped to D E F, and continuously distilled.

10. Some wines, owing to fermentation errors, as already mentioned, are very defective, which ordinary distillery plant is powerless to remedy. Special measures are necessary to cope with the difficulties they present. It is more especially the acid gases that hinder distillation; sulphurous acid and sulphuretted hydrogen ascend the column and combine with the spirit to form very bad ethers, which it is impossible to destroy or eliminate afterwards. The remedy, therefore, consists in not allowing these gases to rise into the upper stages of the column. It is necessary, in fact, to seize 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 of fixed salts without any action on the spirit itself, whilst it absorbs sulphuretted hydrogen and ammoniacal compounds. In fact, this vapour cleansing is done after the style of Woolf's bottles in

laboratories. If the reagent vessels are outside the column, the cleansed vapours return thereto. A supplementary condenser is

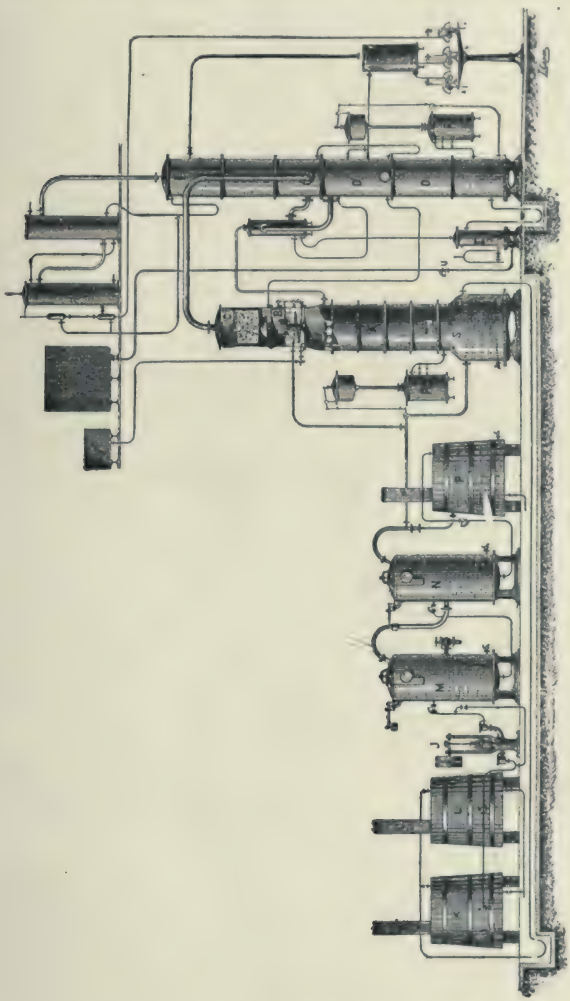


FIG. 25.—Plan for producing 96 per cent. rectified wine alcohol and recovering cream of tartar by multiple effect evaporation, the distilling steam coming from multiple effect apparatus (E. BARBET).

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. 25 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 only requires a low building, so that it meets the wants of the greater number of distillers in the South of France. From the time 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 bad elements. No precaution must be neglected, because the wines used for alcohol making are generally the worst of all. 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^1 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^1. 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. As at the same time the elimination of the odours of the marc by the special column D D^1 is perfect, a 96 per cent. "bon gout" alcohol, quite fresh and pure, is obtained. The vinasse is run alternately into K and L, and the tartaric acid saturated by potassium carbonate. The clarified liquid is pumped into M N, heated by live steam from 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, and into which the vinasse from N (No. 2) is allowed to run continuously. After this fresh and last 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 go further, and the concentration thus obtained utilised to extract the glycerine. The cream of tartar mother liquors contain, in fact, a sufficient quantity of glycerine as to render the rational extraction easy and economical. Such work entails the addition of special supplementary plant, and, in particular, plant for the use of superheated steam and distillation in vacuo; but the product obtained being of great value, the capital sunk yields a very profitable return, which distillers should not overlook (Chap. III. sec. 18, etc.).

11. *Fruit distillation on a small scale.*—We in Great Britain are so accustomed to regard the distillation of fermented liquors as a large scale operation only, that we are apt to lose sight of the fact that, abroad, distillation is carried on by the peasantry, small farmers and proprietors, more especially in France, on what is often a very small scale indeed. This they are enabled to do by more liberal Excise laws, which permit a farmer or market gardener to distil his own

produce with the most unexacting of restrictions. An idea of the trifling nature of these restrictions may be gathered from the fact that perambulating stills are carried 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 fruit 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 hygienic measure if jam manufacturers were allowed to distil for industrial alcohol purposes such fruit as now reaches them in an unfit state for human food. This would be a special advantage to such jam manufacturers as 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 he would get for it as damaged fruit. In France the scriptural injunction "Let nothing be wasted" is carried out in its integrity. In Britain, one manufacturer seems to vie with another as to how profligate he can be not only in the use of his raw materials, but in the amount of residual products he can send down the drain.

12. Fig. 26 shows 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 refrigerator R being filled with water, the furnace fire is lit, and the noise of the liquid boiling in the still is soon heard. When the distillation is going to commence, 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. More or less water is run into the rectifier 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., it is collected apart and the distillation urged. 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 be not desired, or if aromatic waters are distilled, water is not run into the rectifier. The still then works like an ordinary still. The operation finished, the still head is taken off, and 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, all that has to be done is to raise the handle H without having to touch the bolt K, which shuts by itself.

It is suitable for distillers, spirit dealers, dispensers, chemists, per-

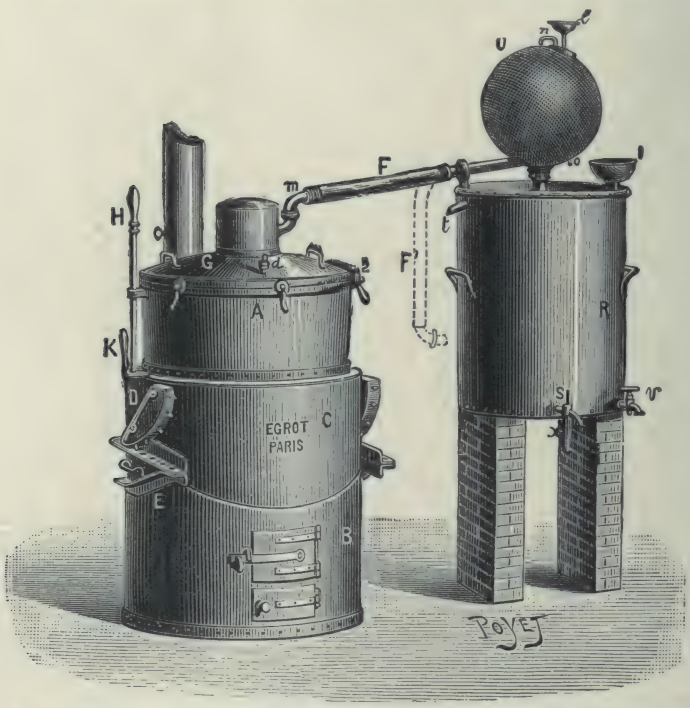


FIG. 26.—Fire heated portable tilting still for small scale distillation of wine, fruit, etc. A, body of still; B, wrought-iron furnace; C, part of furnace fixed to body of still; D, cam; E, rail on which D rolls; G, still head; *d*, screw box; Z, bolt; *m*, goose neck joint; F, goose neck during tilting; H, handle; K, lever bolt; U, spherical rectifier; *l*, water funnel; R, refrigerator; I, funnel; V, discharge tap; S, worm safe; *x*, test-glass (EGROT and GRANGÉ).

fumers, amateurs, etc. For distilling solid matters, metallic baskets may be used to prevent burning by 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.

13. 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 bodies, the second fairly pure ethyl alcohol, the

FIG. 27.—Egrot's still, with water bath for various purposes (EGROT and GRANGÉ). A, flat-bottomed pan capable of being used for various purposes; B, water bath, may be used as round bottom pan; C, still head. This plant may serve :—

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	

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 smelt strongly of fusel oil, and had 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

portion, which after dehydration by potassium carbonate weighed 352 grammes, gave—

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

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. Grammes.	II. 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 bases	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

1. 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 from 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 skimmings and washings of sugar pans. When molasses is diluted with 20 times its weight of water, and when the mixture has cooled to 78° F. and 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 inherent to cane juice, and dissipated at the high temperature employed in the production of molasses. This product is called *Tafia* in the West Indies, *Cana* 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, just as if intended for sugar manufacture (Figs. 28, 29). It is generally strained to remove bagasse débris, and then fermented. The natural ferment is found in great numbers on the exterior waxy surface of the cane. The cane-sugar ferment differs from other European ferments. The alcoholic ferments of Venezuela are 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 rapidly and in good condition with "chutes" at zero. The alcoholic ferment can stand strongly acid liquors expressed as sulphuric acid. Media in which European ferments would be paralysed, the Venezuellean ferment can stand very well. The reproduction of the alcoholic ferment is effected at a temperature between 30° and 36° C. (86°–96°·8 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 like mineral acids,

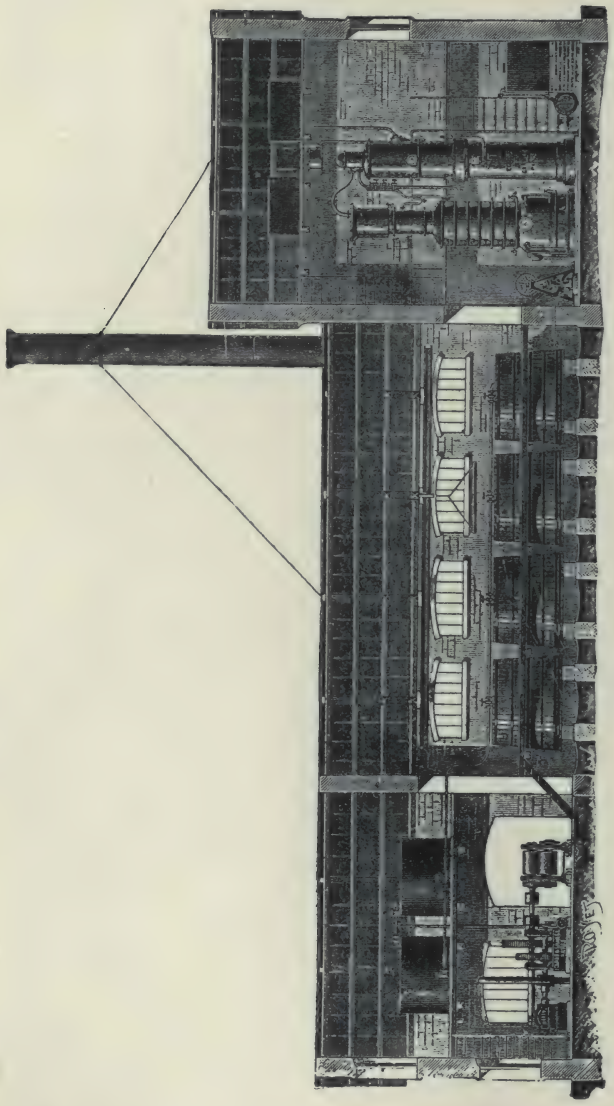


FIG. 28.—Rum distillery (EGROT and GRANGÉ). Front view of interior.

but they stand certain organic acids very well. Bakers' yeast ferments with great activity in lactic acid media equivalent to 6–7

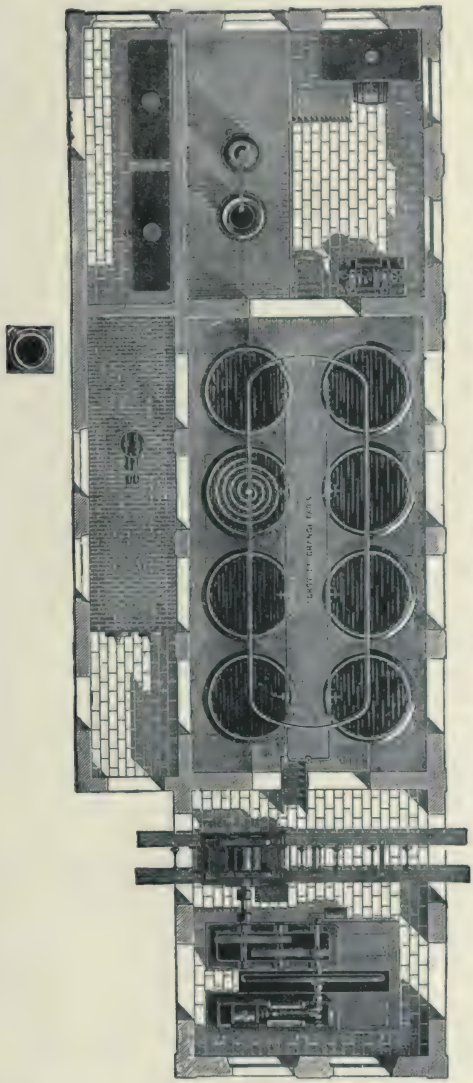


FIG. 29.—Rum distillery (EGNOT and GRANGÉ). Ground plan of Fig. 28.

grammes of sulphuric acid per litre (*i.e.* 6–7 lbs. of sulphuric acid per 100 gallons), and wine ferments in certain years have a tartaric acidity exceeding an equivalent of 7–9 grammes of sulphuric acid per

litre (7–9 lbs. per 100 gallons).[1] The hotter the wash the more need it has of a certain acidity (preferably organic, with the exception of volatile fatty acids) 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 hotbed of bacterian disease.

2. Sometimes it is a film or web of mycoderms 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 there is no remedy but strong acidity, so long as such acidity is not so strong as to be injurious to the proper ferment itself. In cane juice stringing like thickened oil, says Delafond, I have found the viscose 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 viscose ferment does not develop in a strongly acid media. The addition of sulphuric acid stops viscous fermentation in active evolution, and as it lives on levulose, this ferment is not propagated until the alcoholic ferment has commenced its chemical work of splitting up sugar. The *acetic* ferment may be contended against by avoiding the heating of the juice and by realising as *anaerobic* a fermentation as possible. In regard to other bacteria, the acidity of the juice acts best. The best way of securing cheap acidity in the colonies 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*; it is a question of trial. Another way of checking too energetic fermentation is the rational use of antiseptics, refrigeration of the wort, previous sterilisation, etc. The refrigeration of the wort is not often practicable for want of fresh water, if carelessness, bad air, polluted water, and general filthiness 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 while 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 from a financial point of view, even though all the exhaust steam were utilised and the very best rectifying apparatus employed. Under such circumstances it cannot be wondered that the percentage of the theoretical 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

[1] Chap. VI. sec. 2, Table XVIII.

(but fallacious) figure, and the yield from the molasses appears to be satisfactory.

3. *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 separated from one another, not arranged in masses or in form of long bands. Rum ferment remains unchanged as 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 starch and dextrine solutions, there appears in about 48 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 been going on slowly or the wash has been too much exposed to the air. The rum ferment not only differs in structure from the beer ferment, its products and behaviour 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 slackens, 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. Of course, this means pure sugar, and does not apply to liquids such as molasses. 6. The rum ferment both in its proper form and that of mycelium separates a diastase which converts saccharose into glycose (STADE).

4. In Jamaica and some of our colonies, says Ure, 50 gallons of spent wash or lees are mixed with 6 gallons of molasses, 36 gallons of sugar-pan skimmings, a substance rich in aroma, and 8 gallons of water, in which mixture there is about one-twelfth part of solid saccharum. The fermentation is seldom complete in less than 9 days, and most commonly it requires from 12 to 15, 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 fallen to the bottom, 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 after this period the better, to prevent loss of alcohol by the acetous 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 lbs., and contain nearly one-eighth of their weight of

sugar = 1575 lbs., which should yield nearly its own weight of proof spirit, whose bulk is $\frac{1575}{0{\cdot}92} = 171{\cdot}2$ 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 1 gallon of sweets should yield 1 gallon of spirits, and hence the above 16,666 gallons should have afforded the same bulk of rum.

Dr. Ure also quotes an experiment he made as follows:—150 lbs. of West Indian molasses were dissolved in water and mixed with 2 gallons of yeast weighing exactly 20 lbs. 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 up from 80° to 90° F. during the last two days by means of a steam-pipe, to favour the fermentation. The product of spirits was 11·35 gallons. Now, 150 lbs. of the above molasses were found to contain of solid matter, chiefly uncrystallisable, 112 lbs., so the yield was in accordance with the above statement.

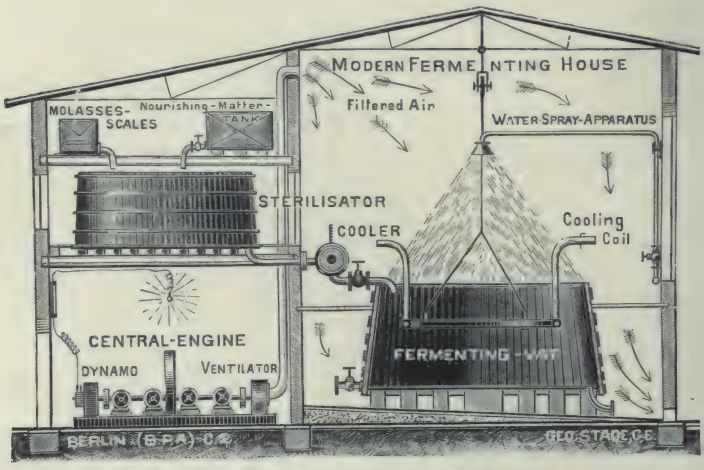


FIG. 30.—Modern fermenting room (STADE).

5. More than one million tons of cane-sugar molasses are produced annually, sufficient to make 132,000,000 gallons of spirit of 60 per cent. strength. The sixth part of it is not made, because, especially in the French colonies, they have, as a rule, given up making

rum, or Tafia as they call it, and even, it is alleged, run their molasses into the sea or into streams.

6. *Composition of molasses.*—The analysis of molasses is complicated by the co-existence of a great number of sugars and different reducers, saccharose, dextrose, levulose, mannose, glutose, raffinose, caramel, etc. The choice and ratio of the defecation agent greatly influence the results.

TABLE XIX.—ANALYSES OF CANE-SUGAR MOLASSES.

	Java (Prinsen Geerligs).			Thompson Average Analyses of Molasses of Different Sources.
	Extreme Limits.		Average.	
	From	To		
Crystallisable sugar . Calculated by inversion	5·7	44·3	35·3	26·34
Reducers	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 matter . . .	—	—	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 a molasses is no safe guide as a basis of estimation of 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
Reducers	19·33	16·90
Total sugar calculated as uncrystallisable . .	60·48	58·53
Total by direct inversion of the molasses . .	59·96	58·56

On inspection of these analyses, No. 1 ought to yield more than No. 2, but direct experiment yielded 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 $3\frac{1}{2}$ per cent. more. Direct fermentation method is indispensable. The acidity of the molasses is first determined so as to calculate what sulphuric acid has to be added to get a wash with 2 grammes of acidity per litre before fermentation. Then a well-sterilised 2 litre flat-bottomed flask is taken, tared, and 200 grammes of molasses weighed into it. Eight hundred grammes of water are added to make the fermentation test on the base 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 10 to 15 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 neutral 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 every six hours for instances. 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_2 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 liquid 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—

$$v = \frac{p}{d} = \frac{1021{\cdot}1}{1018 \times 0{\cdot}99916} = \frac{1021{\cdot}10}{1017{\cdot}1} = 1004 \text{ c.c.}$$

The 500 c.c. taken for distillation correspond to 200 grammes × $\frac{500}{1004}$ = 99·601 grammes of molasses. To get the exact alcoholic strength, saturate exactly the 500 c.c. of wort, for without that the volatile fatty acids distilled would cause a slight loss of alcohol, push 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

7. With pure ferments it is nowise 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 left to itself. Fermentation is spontaneous, *i.e.* the germs in the air sow themselves in the liquid. At the end of the season, as germ spores are rare in the air, rule-of-thumb processes, justified by strange theories, are resorted to by the foremen. Sulphuric acid, lime, 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, and yields more lactic 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 and filled with dilute sterilised molasses. Fermentation soon sets in, and a leaven with an indigenous ferment adapted both to the high temperature and to the composition of the culture "bouillon" 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 make the wash of the same average strength, and to make sufficient wash for the night-shift. It is the fermentation antechamber, and there the wash left to itself is contaminated, so much the more because the vat is rarely emptied and still more rarely washed and asepticised. The wash ought to be sent to the fermentation tuns as soon as 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 get the right density. Such plant is in use in Austria to dilute the crude phlegm before filtration or rectification.

8. In spite of its concentration, cane-sugar molasses often decomposes so much in the casks as to shoot out the bungs and even the bottoms of the casks with disengagement of bad smelling gases. Such molasses are often so acid that by dilution they yield a wash with an acidity of 3 grammes per litre. All volatile fatty acids are antiseptic and hinder fermentation; they may even stop fermentation in favour of 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 by an equipment quite identical with that used in the "denitration" of beet molasses. The most simple process is Barbet's continuous sterilisation process. The copper boiling and aeration pan is of such a capacity that the molasses previously diluted to 25°–30° Baumé

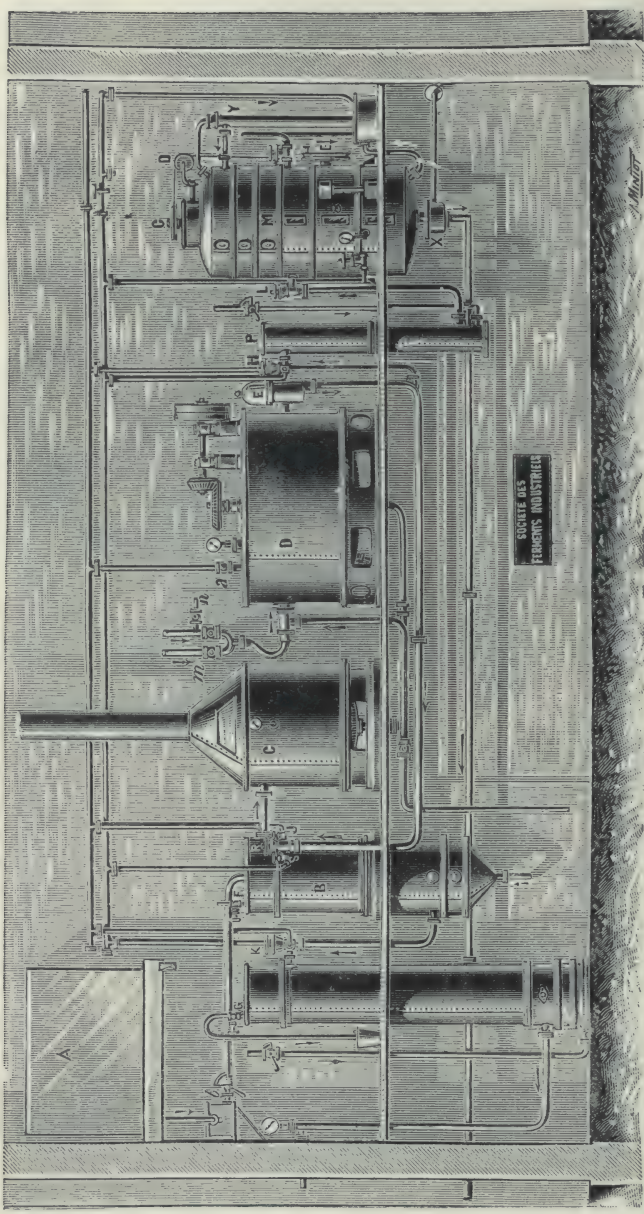


FIG. 30a.—Plant for aseptic fermentation of cane-sugar molasses (E. BARBET). A, tanks to dilute to 28–30° B.; *b*, regulating feed tank; B, forewarmer; C, continuous denitrator and steriliser; D, mixer to dilute with hot water or hot spent wash; *m*, spent wash tap; *n*, hot water tap; E, test-glass;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

remains there for over a quarter of an hour. The molasses before entering is methodically heated by the heat of the issuing boiling molasses, so that the plant requires little or no steam. Next to the heat recuperator a tubular refrigerator brings the sterilised masses back to a good heat for fermentation. It may then be diluted to 9° or 10° Baumé, so as to send the liquid to 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 thinned down, as above, to about 30° Baumé, and if need be a small dose of sulphuric acid is added so as to liberate fatty acids. The thinning down 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 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 red 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 smell. When the molasses is of good growth and of good quality, the perfume of the acid water is not unpleasant; it is then preserved 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 expulsion 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 *pro rata*. As the molasses come from the sterilisator C, the boiling mass which is at 30° B runs into a supplementary thinning pan D before being sent to the recuperator B, and this supplementary thinning 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.

9. According to circumstances, the thinned 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 is lower the greater amount of free acid in the wash. Thus natural wines are pasteurised as low as at 67° C. (152°·6 F.), because their tartaric acidity is such that suitable asepticism is got at this temperature. With molasses wash 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 thinner D, the capacity of which ensures to the wash a stay of 20 minutes at a high temperature, there is a special test-glass E which attests 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 fermentation temperature. 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 of 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 leavened by 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 thinning vessel A is dispensed with, and there is then only one recipient for the sterilisator C and the thinner D. In it the molasses is first thinned down, boiled, the boiling spent wort then added to bring the thinning to the right density for fermentation. Thinning is done by hand.

10. *Pure ferments for cane spirit.*—Several chemists have attempted for some years to propagate 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 leaven, 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, about 36 to 40 hours for molasses wash. The stumbling stone of almost all these previous attempts was the preparation of pure leaven from the ferments furnished by laboratories. Starting 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 beck of 20 or 30 hectolitres, and finally it was this leaven which served as "yeast" to 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 no 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 leavens charged with a mass of quite vigorous cells. There is only one pure leaven apparatus under way closed and tested at a

kilogramme of pressure. At the outset it is sterilised by steam at 115° C. (239° F.) for several minutes, then the excess of pressure is relaxed, sterilised air is introduced, and 8 to 10 hectolitres of wash sterilised in a vessel on one side and cooled in the refrigerator. Cold or tepid water circulates 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 introduced and aerated briskly to prolifically push 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 play. It consists in cultivating the ferment in 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. It at the same time accentuates in the leaven the faculty of developing spores. In a word, when a leaven is fatigued it is revivified by surface cultivation in a wash in as shallow a layer as possible. Pasteur realised this very simply 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 borrows from 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. Take a reservoir A (Fig. 30*b*) 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 compressed 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 much 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 sending the air to the bottom of the wash, which utilises the oxygen of the air very badly, Barbet utilises 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, a safety valve. It is likewise fitted with brass brushes on each plate, so that when desirable the leaven may be detached

from the plate and forced to descend into the mass of leaven underneath, and from thence to the fermenting tuns. When the leaven is

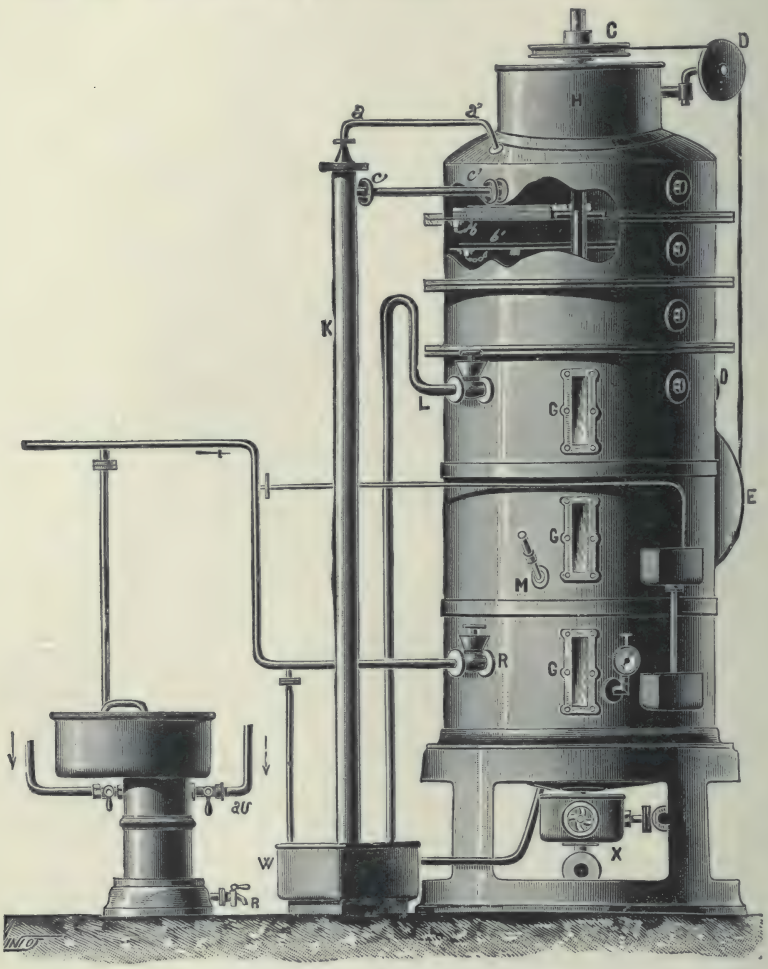


FIG. 30*b*.—Plant for producing pure ferment for fermentation of cane-sugar molasses (E. BARBET).

ripe, only two-thirds or more of it are taken for the fermenting tuns. The remainder is used to perpetuate the fermentation in the apparatus which has been filled with fresh wash, sterilised and cooled. Fresh

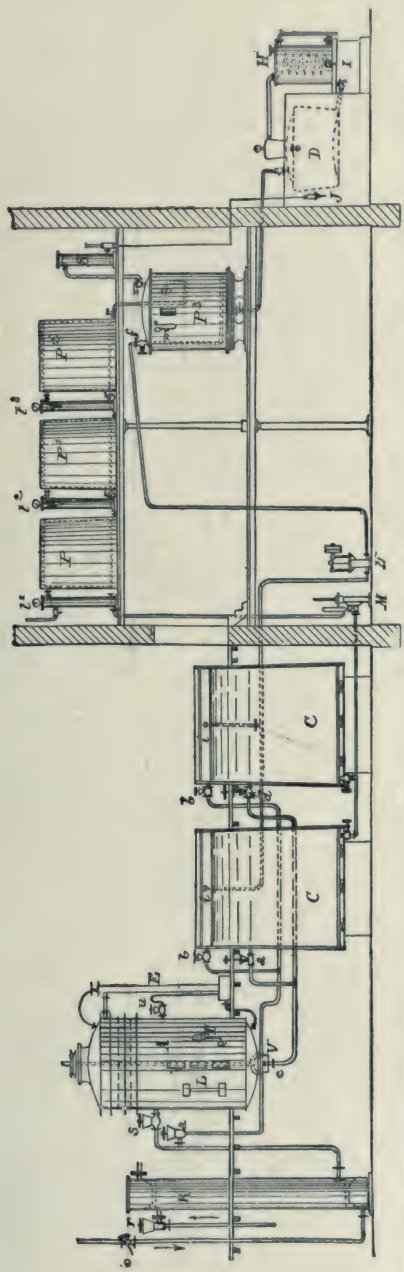


FIG. 31.—Plant for aseptic fermentation, capture of fermentation gases, ageing by prolonged heating and distillation of rum (E. BARBET). *A*, ferment vessel; *C C*, fermentation tuns; *R*, steriliser; *M*, fermented wash pump; *N*, fermentation gas pump; *i i*, fermentation gas pipes; t^1 t^2 t^3, calorisators; P P^1 P^2 P^3, closed vessels; P^3, autoclave to stand pressure of CO_2 from *C C*; *E*, condenser for u nabsorbed fermentation gas; *D*, still; *H*, condenser; *I*, test safe; *J*, test safe for perfumed liquid run into still or distilling column (E. BARBET).

leaven for the fermenting tuns may thus be got every 5 or 6 hours. All the accessories are so designed as to prevent all interior contamination whatever, 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 start with a fresh culture once more. Molasses alone would not be an excellent culture media 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.

11. *Continuous fermentation.*—The pure leaven plant just described makes a batch of leaven every 6 or even every 5 hours, which corresponds to 4 or 5 tuns per day. If the tuns be small and numerous, one batch of leaven can do for 2 or 3 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 leaven, 2 or 3 large copper autoclaves are placed fitted with 1 or 2 aerobiose 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 extensively inoculated by the addition of the pure leaven, the emulsifier and the "aerobiose" treatment stimulate fermentation, and the liquid from B constitutes in its turn a more bulky source of leaven than what 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^1 in proportion as the remainder of the sterilised juice is fed into it. In B^1 the same phenomena occur as in B. The "aerobiose" 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 according to 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. In 24 to 36 hours it can be distilled. So much is this the case, that the tuns are too powerful. That is not, however, an evil, because it gives a certain elasticity of pace to the manufacture, because a certain amount of interval before distillation seems to improve the perfume of the rum.

12. *The saline matter of cane-sugar molasses.—Aerobiose* leavens being very vigorous, a large proportion of spent wort may be allowed to enter the wash in the tuns without any fear of the accumulation of salts and organic matters arresting the fermentation. The spent wort becoming richer in salts, the potash salts may 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. Take 25,000 kilogrammes of molasses as 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 say 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 only be sent to the potash evaporators 500 hectolitres containing exactly the potash of 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 of all sorts does not act prejudicially on the fermentation and the yield. Take the two Egyptian molasses 1 and 2 previously mentioned. In the distilled vinasses thin down respectively a fresh quantity of No. 1 and No. 2 in such a proportion 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 enormous 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 got.

	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. For 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. But beet molasses as sent to be incinerated only contain about 3 per cent. of ashes, but here is a liquid much more rich in salts and which ought to be profitably evaporated and incinerated. Pellet gives numerous analyses of cane molasses ashes. Generally there is a large proportion of insoluble, sometimes even 50 per cent., consisting principally of carbonate of lime silica and carbon due to the abuse of lime in the fermentation process. The carbonate of potash oscillates on account of the insoluble from 10 to 35 and even 40 per cent. If it be considered that with the least concentration in the triple effect bringing the spent wort to 11° Baumé, it may be incinerated without any expense in coal, the extraction of potash salts, therefore, will yield a great profit whatever price fuel may be in the colonies. The most simple triple effect system for this purpose is Barbet's, 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 *auto-evaporable* in the furnace, *i.e.* the combustion of the organic matters incinerated suffices to finish the evaporation of the water without expense of fuel, except when the potash furnaces are lit up. Neither in the triple effect nor in the furnace are there any expenses for coal. The potash salts are thus got gratuitously.

13. *Distillation with purification and ageing.*—With good quality molasses and fermentation as described, little has to be done but to distil, say, in the classical alembic of Père Labat, or, say, in still shown in Fig. 33. The ageing of the wort (Fig. 31) prior to distillation dispenses with ageing in the distillery plant. But routine and resistance to new fermentation methods has to be taken into account as well as 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 only utilising the pot-still proper and the condenser. A heater for the wash is only added when it 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 a play of taps direct 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 retrogradation, a methodical classification 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, and separates 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 rôle of ageing by compelling, in virtue of the great alcoholic strength, the fatty acids to combine with the alcohol to form fruity ethers and perfumes.

14. *Rectification of rum.*—Rum is as easily rectified as other crude alcohols from beet or molasses, but it is evident that this operation is facilitated the less impure and strong smelling the rum. Pure fermentation is therefore more necessary in this instance than for mere 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 low 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. Stade makes, in addition to the plant illustrated in Fig. 32, a simple distilling column, a description of which will be given first. This distilling column type A is identical with Fig. 32, except that it has no dephlegmation column. The wash regulating tank is open at the top, and a pipe from its bottom 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 is appropriated to the distillation proper. The remaining 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 correct 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 water boxes screwed to the sides, and as

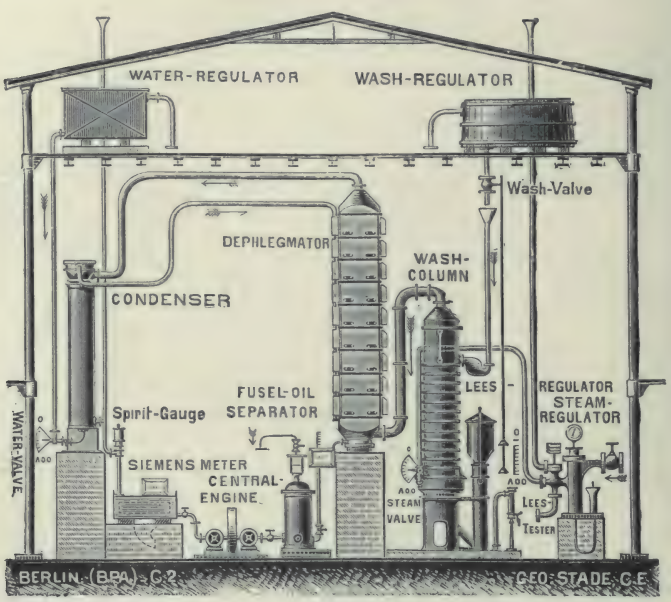


FIG. 32.—Rum distillation and rectification plant (STADE, Berlin).

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.

15. *Preservation and transport of molasses.*—At the outset it was mentioned that the cane molasses now produced ought to yield 132,000,000 gallons of spirit of 60 per cent. strength yearly. This is far from being done, and that is due chiefly to the fact that in Cuba, Java, Louisiana, two-thirds of the molasses are run into the sea; some factories try to burn them. Sugar factories give

up distilling them on the spot, such have been the bad yields in quality and quantity; moreover, there being no outlet or market on the spot, the rum casks must be transported to them, and rum casks are very dear. Tropical climates do not lend themselves to the transport and preservation in good condition of empty casks, the sun dislocated them, and in spite of re-coopering and steeping them in water the damage is considerable, and the returned empties are a great expense. More often than not, the value of the cask is at least double of that of the molasses it contains. Molasses distilleries should be installed at the seaports, then cheap transit of the molasses thereto, and their warehousing and preservation, must be easily secured. 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 diminish the store 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 again available sooner. Before running the molasses into the mould, the latter is lined with a sheet of packing paper. The molasses is run in, and, when set, dumped out. The molasses is thus packed in the paper, which hinders them from sticking together in transit. It protects the somewhat hygroscopic molasses from humidity. Transit of such molasses loaves becomes an extremely simple matter: manipulation is identical with sugar loaves, there is no dead weight to transport except the paper, and that is negligible compared with the casks, which often exceeds 2 per cent. The concentration itself of the molasses ensures a very appreciable economy in freight, because it removes 10 to 12 per cent. of water; there is only 88 to 90 per cent. of the original weight. In colonies deprived of roads, transit was done formerly on the backs of mules. Moreover, the molasses cakes may be stored in ordinary warehouses instead of immense wrought-iron tanks. Moreover, there is no need to fear the deterioration of the molasses which frequently takes place, so far as to be incapable of being fermented, or even to burst the casks. Owing to its concrete condition and complete solidification, it is impossible for it to go wrong, the more so as the mass has been sterilised by the boiling which has solidified it. It may be preserved indefinitely.

N.B.—The coffey type of still described in Chap. VIII., Figs. 39–37 is extensively used in the distillation of alcohol from the sugar cane in the British Colonies. also fire-heated stills like Fig. 33, p. 123,

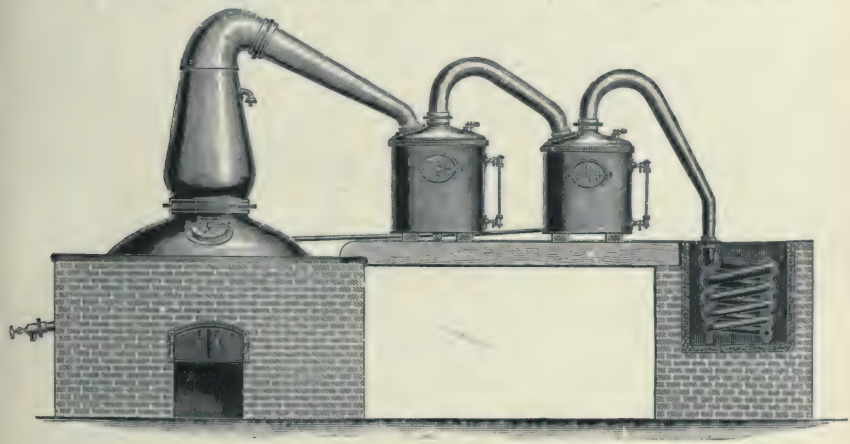
CHAPTER VIII

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

1. *Water and fuel.*—The purer the water for distillery purposes the better, and the distiller could not do better than consult the English edition of De la Coux' *Industrial Uses of Water.* Each kind of water must be treated differently in order to purify it, and to discuss such processes would lead us too far. Some distillers are accustomed at the end of the season to take into account, after each spell of working, what it has cost them in the form of working expenses to produce a hectolitre of alcohol. This total is, moreover, subdivided under the headings "wages," "acid," "coal," and so on; even "lubricants" have a heading. This classification is highly useful, for each year the distiller can judge by comparison with previous runs the cause of the increase or decrease in general expenses, and take the necessary measures to remedy any increased expenditure thus divulged. Now, if we compare the cost of manufacture detailed in this way, as determined in different distilleries, nothing is more surprising than the enormous differences under the heading of coal. Some distilleries use per hectolitre of alcohol produced a good third more than their neighbours. 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 every sugar work strictly analyses the coal, coke, limestone, and all purchases generally, 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 calorie ought to be the same. That *should* be so, but it is not always so, and distillers are known to pay very often dear for indifferent coal with much ash. This precaution is sometimes very useful, it is never superfluous. (*b*) More or less complete retention and utilisation of the heat generated. Here, again, there are great variations from one distillery to another. Sometimes it is a bad stoker who rakes his furnaces too much about at one time, and loses as black smoke the heat he should generate; again, it is the fire bars that are defective, producing ashes with much

imperfectly burnt cinders; later on, it is the boilers themselves, the obsolete pattern of which does not allow complete using up of the heat generated. Or it may be the hard water, which deposits a hard layer of encrustation in the interior of the boilers, which, gradually increasing, prevents the complete utilisation of the heat generated. (*c*) Equipment. It is a fact that certain machines require and use up much heat to produce the same useful effect as more improved plant. (*d*) Greater or less economy in using the heat. In beet distilling the volume of juice withdrawn may be reduced; on the other hand, much of the hot vinasse is used over again, so much heat gained or saved. It is well to remember in regard to heat, "Nothing is lost, nothing is created." The distiller convinced of the truth of the last clause of that maxim knows only too well that the first does not apply in his case to his out-of-pocket expenses.

2. *Plant.*—This point merits great consideration. Some distilleries



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 still head without intervention of the two intermediate retorts.)

FIG. 33.—Fire-heated still with fractionating apparatus on Woulfe's bottle principle (BLAIR, CAMPBELL, & M'LEAN, Glasgow).

have obsolete or worn-out plant, and the examination of the question of plant to replace it the most advantageously is an important one. The first stills used for the distillation of fermented liquors were *intermittent pot-stills*. The pot-still was a sort of 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 still communicated with a condenser consisting of a copper worm suitably cooled. Later on (about 1800) attempts were made to *fractionate* the vapours by the intervention of vessels on the Woulfe's bottle principle, as shown in Fig. 33. This latter type of still is most commonly used in Jamaica in the West Indies generally. But the old intermittent pot-still (with or without intervening vessels between it and condenser) of this type is only now used for the production of certain potable spirits of superior quality, rum, whisky, etc., the value of which is due to the high estimation in which the aroma of the alcoholic vapours retained by the distillate from the appropriate fermented liquors distilled therein is held by connoisseurs.

3. If a mixture of pure alcohol and water be heated in a pot-still under the action of the heat from the furnace, the contents of the pot-still boil. As alcohol is more volatile than water, the steam given off in the beginning consists of vapours rich in alcohol (Table VI.). Then, as the contents of the pot-still become exhausted of alcohol, they become more and more aqueous, until, finally, the whole of the alcohol is eliminated by boiling, 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 larger proportion of the less volatile water. But when, say, 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 over; and, lastly, there comes over a substance less volatile than alcohol, the fusel oil. But these can only be completely separated from each other by appropriately constructed fractionating stills, the introduction of the two intervening Woulfe's bottles was a preliminary attempt at such fractionation. It 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 pot-still pure and simple 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, an alcohol of the desired strength is obtained. The fermented wash, in the first instance, consists of (1) non-volatile or very slightly volatile bodies: such as—mineral salts, proteins, yeast, glycerin, succinic acid, lactic acid; (2) volatile bodies—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 so far a liquid mixture of alcohol and water. Now, as pure alcohol boils at 78°·3 C., whilst water boils at 100° C. under a barometric pressure of 760 millimetres, it will at once be seen that the more rich the wash is 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 this same wash just as if a current of gas were injected through the liquid at the same temperature. Steam is therefore entrained, and so much the more abundantly as the temperature approaches 100° 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 good condensing medium, the temperature of which is lower than the boiling-point of the less volatile, but not so low as the boiling-point of the more volatile, the vapour of the less volatile liquid will be condensed whilst the more volatile retains its elastic gaseous form.

4. Again, as the temperature at which the fermented wash boils increases as its alcoholic strength decreases, the proportion of steam in the aqueous alcoholic vapour becomes greater until the boiling-point of water (100° C.) is reached, the vapour then contains no more alcohol. When the mixed vapours are led through a pipe laid in water at a temperature under 100° C., but not so 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 C. (176° F.). The same phenomenon occurs with the pot-still. The aqueous vapour is 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 is liquefied. (The cold water enters by the bottom and leaves by the top of the trough containing the worm, hence the lowest spiral is exposed to the coldest water and the top spiral to the most hot. The whole of the water and much of the other impurities which passes over with the alcohol are thus found in the condensation products of the pot-still pure and simple.)

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, then they consist entirely of water and no alcohol. Therefore, as the temperature at which water boils is 100° C. (212° F.), and that at which alcohol boils is 78° C. (176° F.), if a mixture of these two liquids be distilled, the mixture will boil at an intermediate temperature proportional to the percentage of each liquid in the mixture, and the boiling-point of the liquid being distilled is an index 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. Hence, as previously mentioned, intermittent

pot-still distillation is now only used in the preparation of whisky, rum, brandy, and such like.[1]

5. *The use of soap in alcohol distilling.*[2]—As the use of soap, if empirical, was brought prominently to the front 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 suited to the size of the factory. The retorts, he says, were provided with a rotary chain for preventing the lees from adhering to the bottom of the still, which, unless prevented, would deposit and become charred from the heat and communicate a disagreeable taste to the spirit. Previous to distillation about 1 lb. of soap was added to every hundred 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, and were very weak, not averaging more than 63° below proof on Dicas' hydrometer. The remainder of the spirituous product of the 8000 gallons was received in another vessel for a further distillation. The singlings were re-distilled or doubled in the second still, and the spirit 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 mixed with the muddy part of the first distillate, water added, and the whole redistilled. Very weak singlings were 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 muddy, 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 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 "eviscerated" by the husks of the grain. A self-regulating bath in some distilleries was put in the capital of the still. The then common Scotch stills had the capital

[1] There are many varieties of pot-stills of very large capacity used in the manufacture of whisky and rum, and the makers of the still shown in Fig. 33 and other distillery constructors list 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.

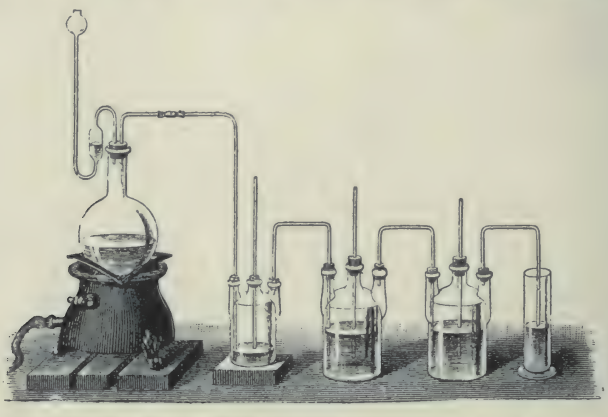
[2] Soap as a froth preventative is now obsolete, so also are mechanical froth preventatives like B, Fig. 40.

15 to 20 feet high to prevent the wash from boiling over into the worm; it was customary to strike the capital from time to time with a rod, and from the sound emitted it is inferred whether it be empty, partially filled, or in danger of an overflow, in the latter case the fire is withdrawn or damped by means of a spout 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 liquor 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 liquid 60 per cent. O.P. was obtained even in the first distillation and at a comparative saving of fuel, time, and labour, while the use of soap was unnecessary. But 67 O.P. is now easily got.

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 saponaceous flavour, which is occasioned by its fatty particles being carried over mechanically in the vapour and dissolved in the alcoholic liquid. Muspratt goes on to explain the manner in which the soap acts to prevent the charge running foul, as follows. During fermentation and subsequent transference of the wort into the still, small portions of acetic acid are generated which decompose part of the soap, setting free the oily compound, (fatty acids), which then rise 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. It should be used, if at all, in such proportion as to exactly neutralise the free acetic acid, and the still hardly seems the right place in which to do this if it 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 here it is oil that is used, not soap. Unless the alcoholic liquor and the soap solution be titrated and neutralised in exact proportion, an excess of soap solution is bound to occur as often as not, and woe betide the distiller whose still begins to prime with an excess of soap solution in it. If the soap solution be intended to prevent frothing, slight excess will accentuate the evil, and frothing and priming will occur with redoubled fury. Excessive heating is not always the cause of priming with soapy liquors or liquors with a tendency to froth. Very often the still acts imperfectly or refuses to act at all, when suddenly it commences to prime, and even if the heat be instantaneously withdrawn, the distiller can only look on whilst the still empties itself through the worm. At least such was the author's experience with non-alcoholic alkaline soapy solutions in a jacketed steam-still, and he has no reason to doubt a repetition

of the same phenomenon with soap in excess in a spirit still. Soap is therefore a most dangerous ingredient to put into a still. Happily, with continuous rectifiers, soap is unnecessary, and such enormous bulks of liquid are not now subjected to heat. Moreover, the passing of the acid alcoholic vapour through Barbet's marble or limestone scrubbers eliminates all acidity.

6. The first great step in advance was made in modern 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 principle for saturating water with gaseous vapours in a series of Woulfe's bottles had been known for a long



A B C D E

FIG. 34.—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.

time prior to this discovery, but no one had hitherto thought of applying their principles to the vapours evolved on distillation, until Edouard Adam, an illiterate workman from Rouen, settled at Nîmes, near Montpelier, attended a course of chemical lectures at Montpelier, and listened to a discussion on the utility of Woulfe's apparatus. The idea apparently 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 chase 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." He obtained a patent for his invention on 29th May 1801, and soon afterwards, as a result of his success

on the small scale, was able to set up a magnificent 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 on 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 given quantity thereof to boil by the transmission through it of the vapour from the same liquid. His efforts were crowned with success; he obtained at the very outset spirit at 33° Cartier instead of brandy. In six hours he distilled in one of his stills 400 velts of wine, say, 679 imperial gallons, from which he got by a single distillation 58–60 velts, say, 100 gallons of 90 per cent. spirit. This apparatus was arranged so that the vapours rising from the cucurbit passed into a series of egg-shaped vessels 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 sent its vapour more and more highly charged with spirit into a series of smaller empty vessels, where they deposited in transit their most aqueous portion, the phlegm of the distilleries, the amount of which diminished in each successive 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 Montpelier telling everybody of the surprising results of his invention. The result was that several rivals sprung up, more especially Isaac Solimani, Professor of Chemistry in Montpelier, and he obtained, about the same time as Adam's first patent, a patent involving much the same principle, but his patent was dated somewhat later than Adam's, viz. in July 1801. So Solimani's claim of priority fell through. Several other adaptations of the still and condenser to Woulfe's principle were introduced, Berard's, patented on 16th August 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 described 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 were infringements on his rights, and the law-suits to which he exposed those using any new invention prevented the use of any other improved form of still than his own.

After realising a handsome fortune by his own distillery and the proceeds of his patent, he became so involved in lawsuits in which he ultimately lost the day, that expenses and costs reduced him to complete penury, in which he died. But some at least of Adam's compatriots revere his memory. Girardin especially credits him with endowing the South of France with an industry which has returned her many millions. But be that as it may, notwithstanding the many good points of the stills of Adam and also of those of his rivals, they each and all had 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 not so complex and more easily managed, yet it consumed more fuel than either, owing to it being necessary to frequently discharge and recharge the still.

7. *History of continuous distillation, continuous rectification and continuous distillation* cum *simultaneous rectification.*—The first to attempt the construction of a continuous still was Baglioni. His attempts, however, were not attended with any great success, but 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 it. Armand Savalle also took the matter up, first in collaboration with Blumenthal and Derosné, and 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 his work 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, and successively created what are claimed to be the most improved types. Desiré Savalle was succeeded by Albert Savalle, the grandson, as 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. But the latter has 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 seems to be easily dismantled for cleaning purposes. Though hampered by unwise Excise laws, Blair of Glasgow has also designed continuous stills, which work well.

8. *Continuous Distillation.* — Let us first of all examine Derosné's method of continuous distillation, whose plant is shown

in Fig. 35. The plant consisted of seven principal parts, viz. the boilers, the distilling column, the rectifying column, the condenser and wine warmer, the refrigerator, the vat where the wine is contained, and the vessel which determines the flow of wine into the apparatus. Of these A and B are the boilers encased in masonry or brickwork, and receiving directly the action of the flame playing beneath them. The fire is applied under A, and the extra heat is communicated to B by the flue passing under it on its way to the chimney. In the copper A, the *vinasse*, or spent wine, is finally exhausted of all its alcohol. C is the column of distillation; D, the column of rectification; E, the wine-heating condenser; F, the refrigerator; G, a vessel supplying *vinasse* to the cooler F, and feeding itself at the same time by means of a ball stop-cock placed in the vessel H; H, wash reservoir; I, tube of communication conducting the alcoholic vapours of the rectifying column D up into the flat worm of the wine-heater E; *a*, stop-cock of discharge of the alembic A: when the operation goes on, the spent *vinasse* runs off continually by the stop-cock; *b*, a glass tube to show the height of the liquor in A; *c*, a safety-valve; *d*, a stop-cock for passing the vinasse from the alembic B 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 column of distillation; *i*, a stop-cock for emptying the wine-heater at the end of an operation; *l l*, two tubes fitted to the wine-heater E, of which the first descends into the last compartment of the rectifier, whence

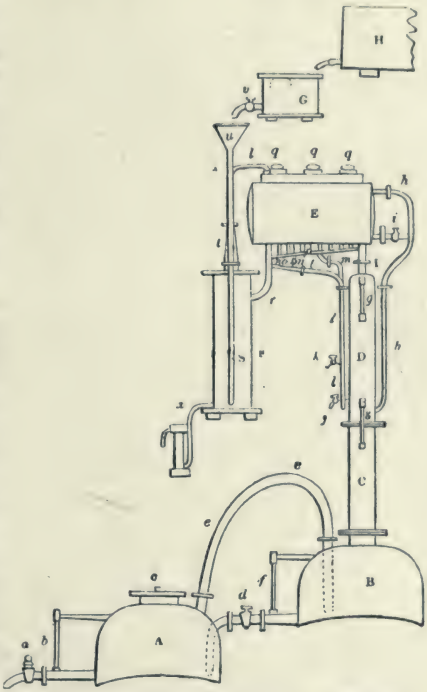


FIG. 35.—Derosné's continuous distillation plant.

it rises to the fifth; and the second tube descends to the third compartment, whence it rises above the second. At the curvature of each of these two tubes a stop-cock, *l* and *k*, is 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 slanting 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 it be wished to obtain a very strong spirit, 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 had 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 balances), with spaces between, each alternate pan having the convex side turned reversely of the preceding one, for the purpose of prolonging the cascade descent of the vinasse through C, and exposing it more to the heating action of the ascending vapours; the edges of these pans are, moreover, furnished with projecting spiculæ of copper wires, to lead off the liquor from their surfaces in a fine shower. The interior of the rectifier column D is mounted 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 either end; *p p p* is a general tube, 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*, peep-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. 39 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.

9. *Continuous working steam stills on Coffey's patent principle* (sec. 11).—This apparatus is constructed to produce alcohol continuously at 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

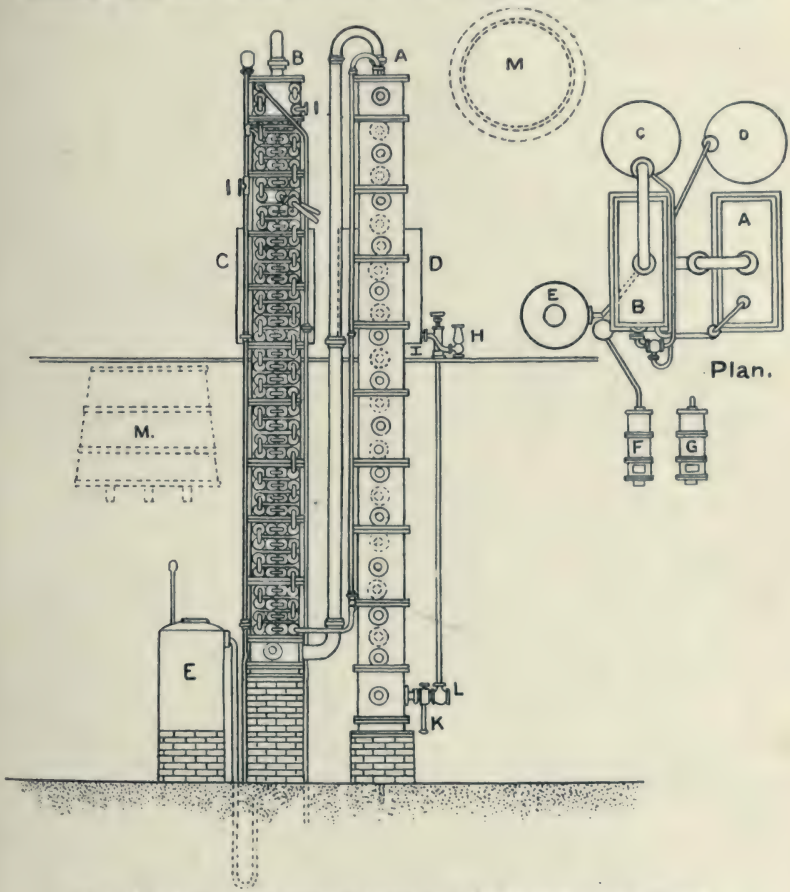


FIG. 36.—Continuous working steam-still Coffey type copper frames (BLAIR, CAMPBELL, & M'LEAN, Glasgow).

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; 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 certainly economical, both as regards fuel and water, and will produce strongest spirit continuously, entirely exhausting all the spirit from the wash. Two steam-pumps of the horizontal type are usually supplied, one for

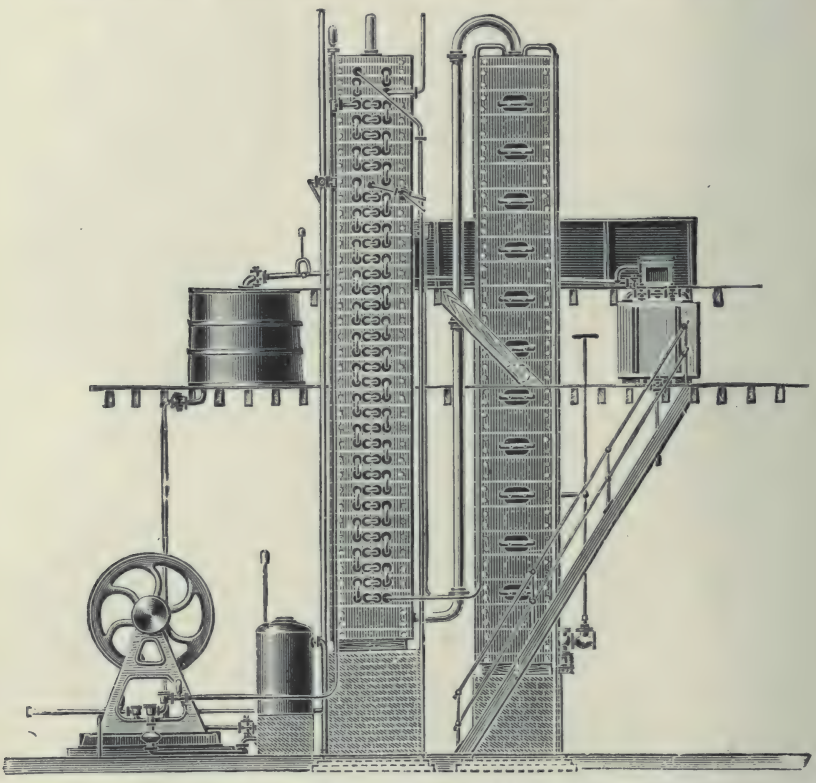


FIG. 37.—Continuous working steam-still Coffey type wood frames (BLAIR, CAMPBELL, & M'LEAN, Glasgow).

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 (supplied of wood on the spot). Fig. 37 represents Blair, Campbell, & M'Lean's continuous working steam

still, on "Coffey's" principle, with all latest improvements, guaranteed to produce absolutely pure spirit of best quality at 43° 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. Both columns are 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, and the necessary tank for worm is of cast-iron; also 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 complete the still. 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 claimed to be the most economical in the market, both as regards fuel and water, and will, it is further claimed, produce the strongest alcohol, entirely exhausting all the 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 can also be constructed with the columns built entirely of copper. (Fig. 36).

10. *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. 36. By aid of the system of invariable flow (15) 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 that only requires a very feeble stock of alcohol on the plates. If it be desired to reach 94° or 95°, 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 retrogradation. The interior working of this still is explained in sections 14–20.

11. *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 eliminating the 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 old stills, though it possesses

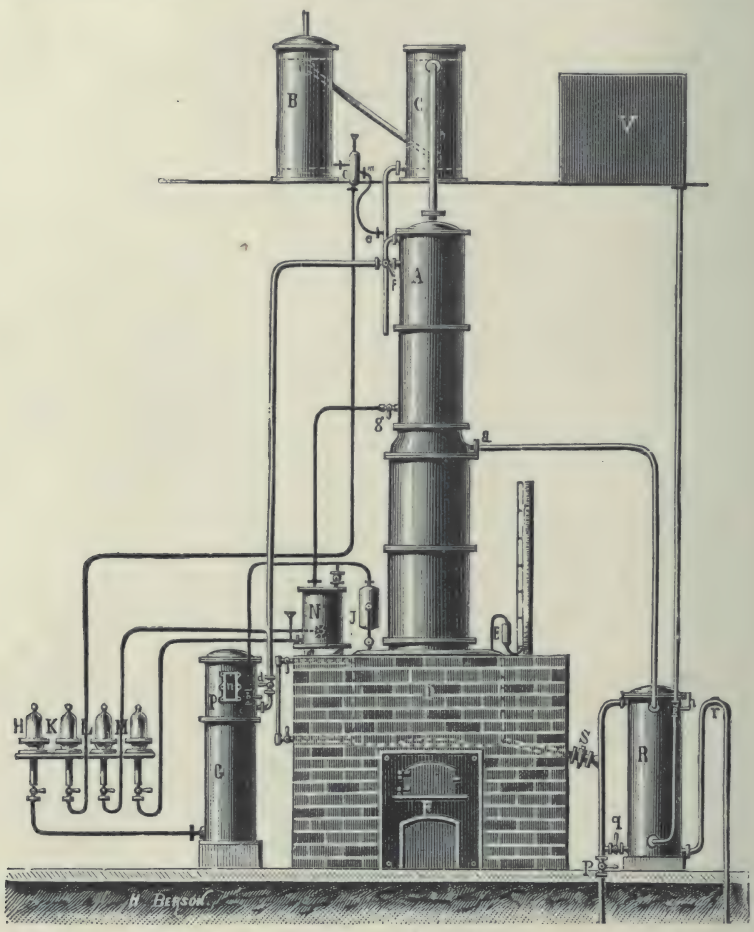


FIG. 38.—Fire-heated distilling and rectifying column. A, rectifier; B, condenser; C, refrigerator; H, pasteurised test safe; K, first runnings test safe; L, last runnings test safe; M, exhaust test safe; R, forewarmer; S, vinasse exit; V, wash tank; *a*, wash entrance (E. BARBET).

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. Its main structure consists of a series of wooden planks, 5 or 6 inches thick, fixed over one another, the joints being covered, or the whole being lined with sheet copper; so that the apparatus resembles a great 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 rectifying 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

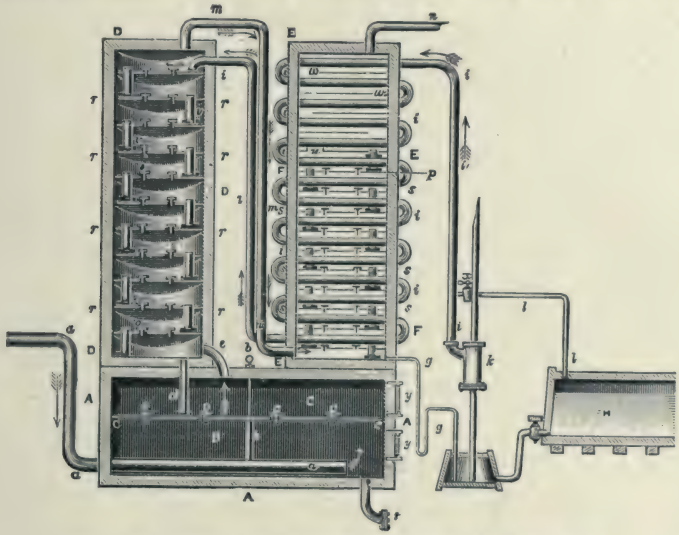


FIG. 39.—Coffey's still, section showing working (sec. 11).

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 a T-shaped valve *o o o*. The wash rectifier D is divided by the plates *r r*, of a like drainer construction, into 12 chambers, and the feint rectifier F F into 10 chambers by similar plates *s s s*. These orifices are so narrow as to allow the passage of the rising vapour, but to prevent the downward passage of the liquid resting on the plates, which passes downwards through the adjunct tubes, viz. *d* into the wash collector B, *v* into the rectifier D, and likewise into the dephlegmator F, passing from each upper into the next under chamber. When the steam pressure is too strong, the valves *o o* give it vent.

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 nicely, so as just to feed the tube *i*, allowing the excess to return through the stop-cock *x*, and the tube *l*, into the wash cistern H. The tube *i* enters into the uppermost partition of E, forming 7 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 plate, and thus gives 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 it has risen up in it to the upper orifice of the glass tube *y*, the valve *b* is to be opened to allow it to flow off into B through the tube *b*. Here into 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*. It rushes through it and carries off the spirit from it through the small orifices of 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 plate *r r*. Whilst the steam meets the wash in every chamber and becomes more spirituous the higher it mounts, it at the same time 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. It here pursues a like path 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 on by the pump with fresh wash 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

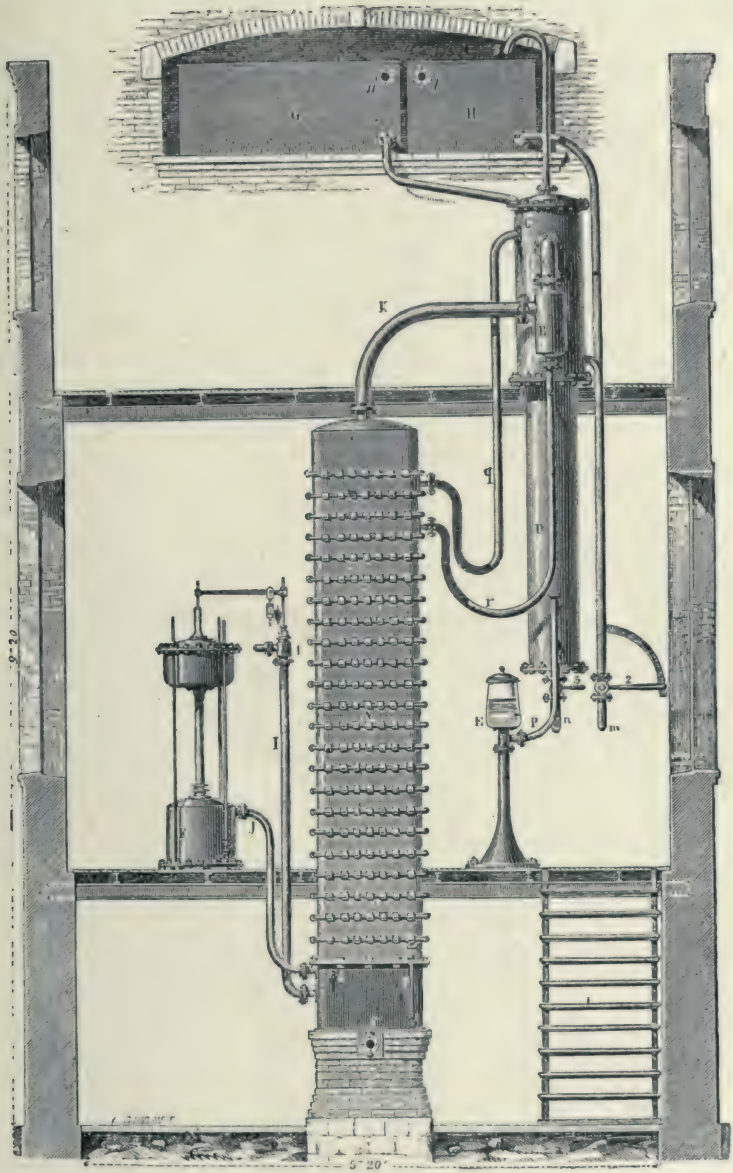


FIG. 40.—Steam distilling column (SAVALLE). A distilling column to distil 8800 gallons wash in twenty-four hours. A, rectangular plate column; B, froth preventer; C, wash heater; D, refrigerator; E, test-glass; G, water tank; H, wash tank; I, J, steam regulator.

the junction tube there (which is larger) through the annexed tube sideways at *p* into the refrigerator not shown in the figure.

12. In the first distilling columns constructed by Savalle about 1870, the plates were perforated ones. They were cheap and simple, and, when new, wrought perfectly; but the perforation of the plate through which the vapours passed, 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 by the wear and tear of the plates. To cope with this evil the plant shown in Fig. 40 was designed. The design and arrangement of the different organs 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 of great heating surface which utilised the caloric of the alcohol vapours to heat the cold wash entering the apparatus; (4) by froth preventers, which secure less acid products free from entrained impurities; (5) by the tubular refrigerator, the interior arrangement of which reduces the condensation water required by 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 (3) and wash-feed taps (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, so that it be not too great and stop the production of alcohol at the test case, and that, on the other hand, it be 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 stop steam. If from Saturday to Monday before starting again, let steam act a little longer, so as to expel all alcohol.

13. *Test-glasses.*—Savalle's gauge test-glass measures with great precision the actual output of a distilling or rectifying apparatus. The principle of its construction rests on the flow of liquids through

a thin partition. Its essential part is shown in Fig. 41. The alcohol from the condenser enters by the 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 this 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 section of 28 sq. mm. It is therefore easily to

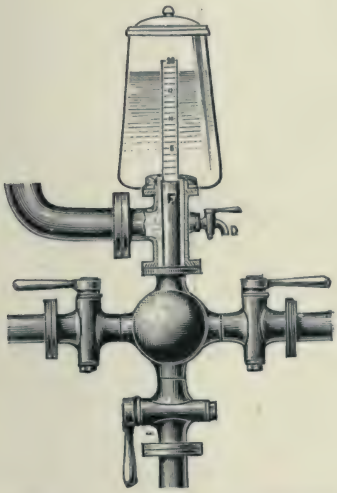


FIG. 41.—Savalle's Test Apparatus.

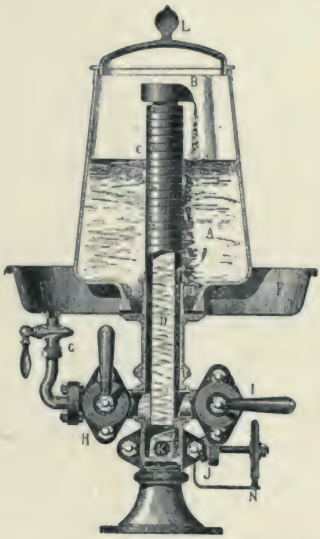


FIG. 42.—Test-glass for measuring density and rate of flow of alcohol from condenser (E. BARBET).

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 play 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.

14. *Barbet's test-glass tap* (large pattern), 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 flows away by the

beck B of the upper funnel. When the exit tap is closed, the level of the alcohol gradually rises in the globe A. Now that globe is graduated, from the lower to the upper mark is 5 litres. The time is noted when the level is at the lower mark, and again when the 5 litres is exactly completed. The difference gives the time required for 5 litres 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 of level in 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 top to bottom. Barbet's small model intended for the extractions of first and last runnings "mauvais gouts" is much smaller, the principle is 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, and the central tube is 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 lid 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 taps are also fixed to the test-glasses whether the products be soluble in water 40°–50°, or insoluble therein 75°–80°. 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. Finally, the exit of the residual water at the bottom, or the exhausted wash when wash is being directly rectified, is entirely automatic and requires 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 wanted, this test-glass is suppressed, and the non-pasteurised enters directly in 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. That 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 in 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 conjunctive 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 only make these three sorts. All other sorts are bastard or mongrel lots. He ought, moreover, so to act that his 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 only yields as far as practicable 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 wash. It may even be affirmed that there is a technical difficulty to overcome. More or less promising solutions of the problem may be got, 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.

15. *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 at T. The refrigerator F is on the same floor and level 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

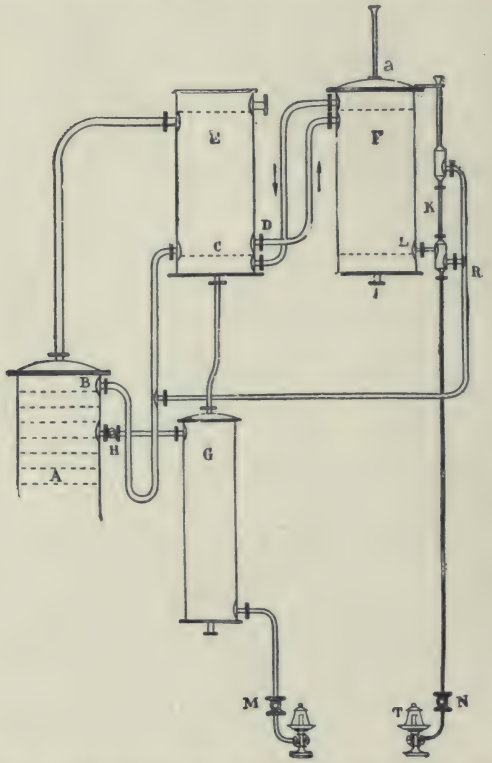


FIG. 43.—Invariable regulation of flow (E. BARBET). A, Rectification plates; E, condenser; F, refrigerator; G, refrigerator pasteurised; H, tap for extraction of pasteurised alcohol; K, air escape; L, exit of cold alcohol; M, tap regulating flow of pasteurised; N, tap regulating invariable flow; P, test-glass; R, return to retrogradation or excess of first runnings; T, first runnings test-glass.

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 at work—the tap N be 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 $\frac{1}{3}$ may be drawn from T, and $\frac{2}{3}$ from P, or equal or any other proportions, without appreciable variation in amount of water used, nor of pressure on regulator. In spite of small variations in level of water-tank, working is very regular. If water feed diminishes slightly, condenser E gives less retrogradation; but as cooler F is too strong (it is so in all plant), cooling of the alcohol is assured anyhow. As all the alcoholic vapour is condensed, and as flow from T cannot vary owing to tap N, the excess of alcohol must 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 and, with the aid of the variable pressure regulator, the flow and play of the column can be modified during rectification either to alter the quality of the rectified, 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, and flow constant, each plate is charged with liquid of constant composition corresponding to boiling-point 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. Table shows purifying capacity of pasteurisation.

TABLE 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′ 30″	0′ 02″	8′ 45″	0′ 10″
3′	0′ 03″	13′ 30″	0′ 13″
4′ 45″	0′ 05″	15′	0′ 19″
6′	0′ 06″	17′	0′ 22″
7′	0′ 08″		

From very impure products an alcohol is thus produced sixty times more pure than that flowing simultaneously from the first runnings

tap. By adopting pasteurisation the ethers may be extracted without a purifier as 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.

16. *Savalle's steam regulator.*—Its essential organ is a red copper float C, playing in the beck 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 of 400 kilogrammes, capable of overcoming all accidental resistances. The principle of this regulator is quite easily understood. Cold water is run into the beck A, forming the foundation up to the level of the pipe F, acting as an overflow and communication pipe between the beck and the steam reservoir, the pressure of which is to be regulated. The top of this beck forms an air-cushion between the pressure steam and the layer of water; under the influence of the pressure the water rises in the lower beck, 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, say, $\frac{1}{1000}$ of an atmosphere.

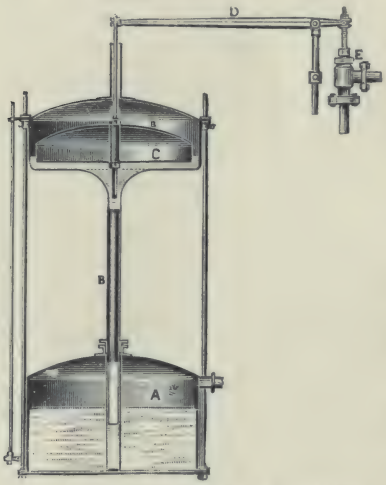


FIG. 44.—Steam regulator (SAVALLE).

17. *Barbet's steam regulators.*—Barbet was the first to call attention to the importance, even with intermittent rectifiers, of being able to vary the working pressure without disturbing it in any way. In intermittent rectification, towards the end, when there is hardly any alcohol in the still, it is desirable to apply more steam, without which final exhaustion and expulsion of oils are retarded. Now Savalle's classical regulator is essentially fixed, and every change in the working pressure effects quite a transformation, and 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 meeting this requirement. But none of them solve the problem in such a practical and simple manner as Barbet, who was also the first in the field. His method is a simple improvement on the Savalle rectifier, on the lower receiver of which he has placed two small taps connected with the pipe which

communicates the pressure. Hence the expense is insignificant, and it can be adapted to all existing regulators. The working is equally

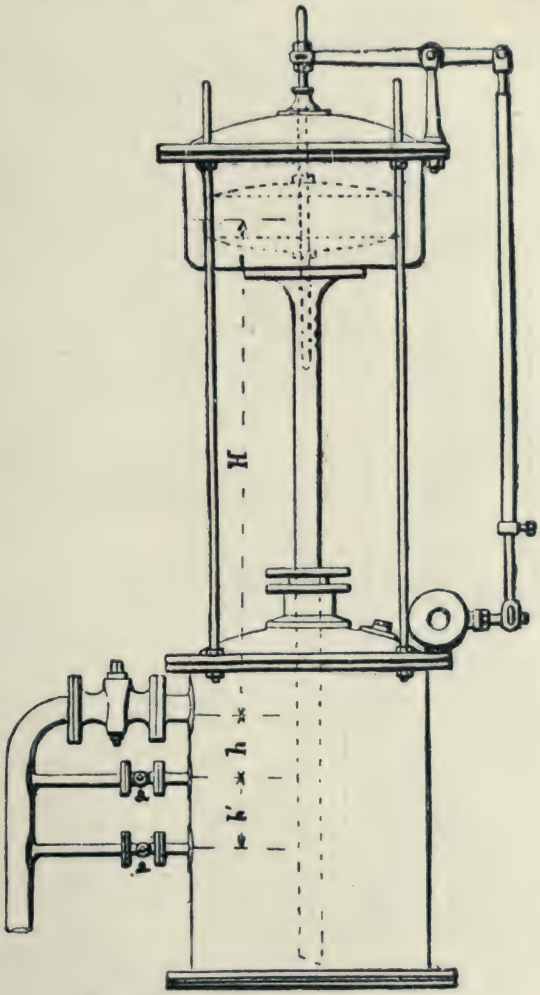


FIG. 45.—Steam regulator (E. BARBET).

simple. If more pressure be desired, the first tap *a* is opened. More pressure still, the second is opened, and *vice versâ* to return to lesser 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. Barbet's rivals have tried to do better. He has only three working pressures at command. They have varied it from centimetre to centimetre, which is not practice but hollow theory, because it is impossible to perceive the actual modification produced in the working by 3 or 4 centimetres of variation in the pressure. It requires at least 8–10 centimetres to have a tangible effect. Now variation from centimetre to centimetre has led to unpleasant if not dangerous mechanical complications. All that is wanted so as to regulate the rectification speed by the stage of progress of the brewing, is to be able to tell the distiller to go at a slow, a fair, or at a quick speed. By prolonging these speeds to a greater or less extent the speed of manufacture is followed with the greatest of ease. Barbet has also improved the regulator itself. First of all, he has replaced the balanced valve of Savalle by a flap valve; the latter requires 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, whilst in the balanced valve the rush of the steam hollowed out the grooves which soon wore out the valve. Besides, it is more easy to regulate the flap valve than the balanced valve, so as to be able to raise or lower easily the upper reservoir of the regulator; the rod which

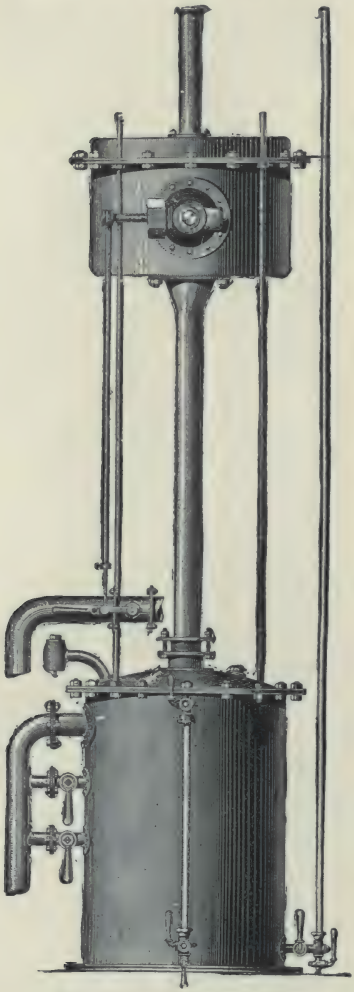


FIG. 45*a*.—Steam regulator (E. BARBET).

works the flap valve is 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 drives inexperienced distillers crazy. 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 well enough centred. In continuous rectifiers, where the pressure is often applied at a rather strong stage, it is important to be protected from priming not only on account of the loss of alcohol itself, 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 analysis 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 aid of the retrogradation or the condensed liquid which falls back on to the plates from the condenser, which acts 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 plate is that which, with the minimum of retrograded alcohol, yields the most decisive and well-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 price of the plant and the less the height of the building. The question of the capacity to be imparted to the plates to obtain 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 to the height of the buildings, besides the cost would be excessive. The number of plates in distilling plant is usually grossly exaggerated. Theoretically, four plates should be sufficient to exhaust wash without increasing the steam. The main obstacle to this simplification of the number of plates arises from the habitual imperfection of the bubbling of the vapour in 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 by their periphery. All the vapour in the centre of the bubble bursts on the surface without having been utilised. So

far as analytical capacity is concerned, nothing up to 1896 could have been desired better than Savalle's perforated plates, which molecularise 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 enlarge, and the apparatus no longer works normally.

Barbet's plates consist of a great number of quite small equidistant caps of hammered copper, around which the wash is

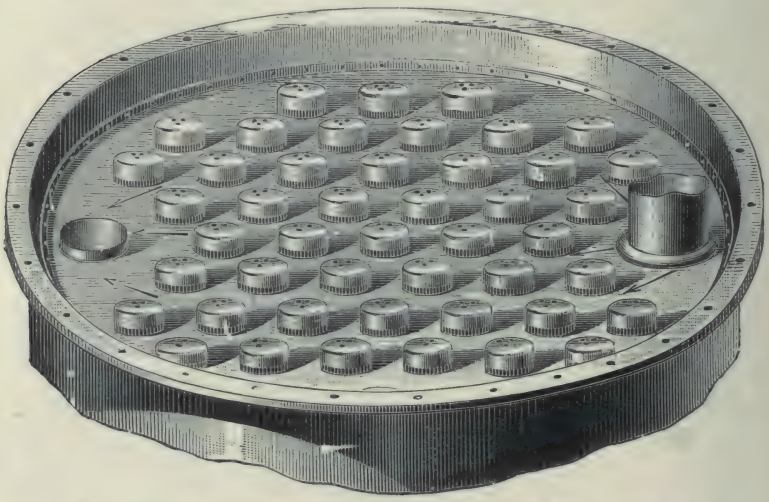


FIG. 46.—Distilling column cross section, showing circulation of wash on plate, and bubbling of alcoholic vapours through comb-slit caps (E. BARBET).

divided, and circulates easily. The developed length of the line 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* which make them in a way look like long-toothed combs. The vapour, imprisoned under each cap, is finely laminated through these combs, thus securing perfect molecularisation, 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 vesicular 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, as testified by the thermometer controlling the working, samples of sufficient size to be tested by the control alcoholometer were drawn off as rapidly as possible.

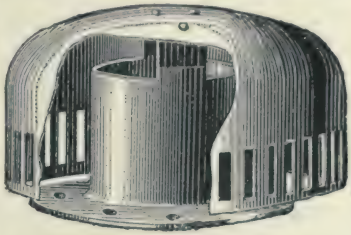


FIG. 47.—Comb-slit cap dipping into liquid alcohol through which vapour of alcohol bubbles, on plate of distilling column (E. BARBET).

The first two plates are affected by the proximity of the feed. Taking No. 3 as a starting-point, it was 19°·1 deficient in actual working capacity. According to Sorel's tables, the vapours disengaged from a liquid of 19°·1 ought to have a strength of 65°·6. Therefore, if the plates are perfect, if analysis is effected according to theory, the plate immediately above should have a strength of 65°·6. In its turn, the liquid on plate 4, having a density of 65°·6, should yield a vapour of 80°·1, and so on. The following figures were got by 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, whereas 77°·4 or one degree less was obtained. Taking 5 as the point of departure, the tables yield theoretically 85°·2 for No. 6, and 89°·1 for No. 7, whilst the test yielded 87°·9, average loss of 0°·6 per plate. Finally, starting from No. 7 with 87°·9, consecutive calculation shows that No. 8 should mark 90°·6; No. 9, 92°·2; No. 10, 93°·3; No. 11, 93°·9. The 4 plates have had exactly the same effect as three theoretical plates, since No. 11 with 93°·3 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 have been gained instead of 11°·7. $\frac{10°\cdot5}{11°\cdot7} = 90$ per cent. of 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, 1889, may be quoted: No. 3, 81°·3; No. 8, 90°; No. 15, 95°; No. 21, 95°·5. It thus took 18 plates to rise from 81°·3 to 95°·5, whilst with the new plates only 12 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 it is the 10th plate which ought theoretically to show 95°·5; that is, with 7 plates instead of 12. 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 got 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 got on No. 5, 90°·41; No. 6, 92°·1; No. 7, 93°·3; No. 8, 93°·9; and No. 9, 94°·5. Instead of that it is the 24th plate which registers 94°·6, and not No. 9. Twenty plates have therefore been required instead of 5 according to theory, hence the yield is only 25 per cent. Besides, 10 plates were required to rise from 87°·5 to 93°·3, whilst only 4 plates were required at Eprunes to rise from 87°·9 to 83°·3, and 20 plates to rise from 93°·3 to 95°·3, whilst only 6 were required at Eprunes to rise from 93°·3 to 95°·5. The yield of the new plates is therefore 2½ to 3 times better, which constitutes a considerable improvement. Again, with washes titrating only 1°·25, Barbet got the following alcoholic strengths at the different test taps:—

Pasteurised . . .	96°·8 at 15° C.	Actual capacity 96°·8
First runnings not pasteurised . . .	97°·6 at 18° C.	,, 97°·0
Last runnings, 5 plate .	80°·0 at 21° C.	,, 78°·2
First runnings from purifier . . .	96°·6 at 20° C.	,, 95°·6

The above results give a striking idea of the power of the concentration plates, for there were only 10 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 7 minutes 195 litres of water ran in at a temperature of 46°. The cold water was at 7° C. The water had gained 39°. That made 1670 litres per hour. The apparatus put through in the same time 2500 litres of wash at 1°25, and yielded 31 litres of alcohol, calculated to 100°. Finally, the spent wash issued from the recuperator at 83°. By making all calorimetrical calculation, it will be found that the expenditure in steam was 366 kilos, say, 14·64 kilogrammes per hectolitre of wash and 67 kilogrammes of water. These two numbers are respectively the half of that which the German columns expend according to Maercker; now these columns only yield an impure phlegm at 90°, 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 of being no longer capricious and of being 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 rôle. Barbet's plates possess other advantages upon which it will be well to insist. When through use the holes of the perforated plate enlarge, that disturbs the regularity of working of the apparatus. It requires more pressure to maintain the liquid on the plate, hence a quite useless expenditure of steam.

If the pressure be not increased, the plates become partly displenished. In the end it is necessary to replace all the plates. With Barbet's plates nothing of this nature occurs. Even when the slits become a little wider by use, the molecular division of the vapour will be effective for many years. And if one day it be necessary to remedy matters, all that has 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 regards 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 spreading large caps, round or long, never allows this proportion, the necessity of which is

evident to be respected. 2. *Area of the plate and stock of liquid which it contains.* This proportion can always be respected. If n small caps are fixed on an area S, then 2 n caps can always be encased in double the area 2 S. 3. *Section of passage for the circulation of liquids between the caps from one overflow pipe to the next.* That 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 quincunx style, that the liquid is forced to spread itself uniformly throughout their winding maze. 4. *Section of passages and outlets for the vapour both by small vents and slits.* That depends on the fact that the number of caps is strictly proportional to the capacity demanded. From a construction 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 mathematical 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, so as to be able to construct the apparatus in a minimum of time. The central chimneys, owing to a special equipment, are also made by mechanical stamping. It follows, therefore, that over the whole of the plate, the dips are perforce absolutely uniform, and that is a condition of good working which has never been obtained, especially with long caps. Supposing the plates of the apparatus, once mounted in the factory, are not absolutely horizontal, suppose they are 10 millimetres out of level, with the old caps the bubbling would be suppressed, as far as those most deeply immersed were concerned. Only the other portion of them would work. Whilst with Barbet's long slit there are 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 a small matter.

18. 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 dirty. 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, annoyance is avoided. With thick wash there are in the columns points on the

plate which are more subject than others to get obstructed with dregs. That is 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 resides in the fact that owing to the small diameter of the caps no point in the plate escapes the bubbling. When a pair of peep-hole glass panes are placed above one of the comb-cap plates, the absolute regularity of the bubbling is easily seen without any violence or projections on the upper plate. Owing to the great length of the comb slits, the deep layers of the liquid are brought into play, thus allowing of a large stock of liquid being left on each plate without inconvenience, thus imparting a perfect stability to the working of the apparatus in the most simple, natural, and infallible manner. The way in which the truncated comb-caps are fixed to the plate renders them removable at will to clean the interior. There are no bolts nor rivets to undo. Once cleaned and put in its place the tongue is lowered with a slight tap from a hammer and 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.

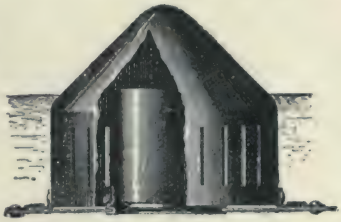


FIG. 48.—Non-obstructable comb-slit cap for thick washes (E. BARBET).

19. Barbet's tubular condensers are of bronze or red 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 peep-holes being inserted according to the diameter of the tubular vessel, wide enough for one to enter the dudgeon to fix a tube if need be, at any rate 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 case of wine-heaters, through which weak alcohol vapours pass. Not only is froth entrained, but volatile acids and gases, which corrode the copper and cover it with saline encrustations. In Barbet's tubular vessels the whole bundle is movable, 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 cleaning manhole. The wash now changes its direction and

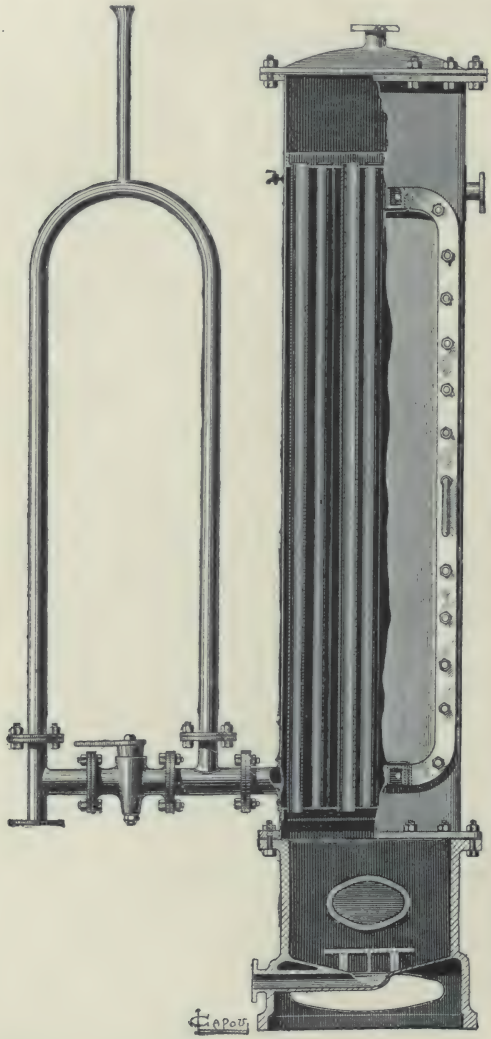


FIG. 49.—Tubular vessels, condensers, recuperators, etc. (E. BARBET).

spreads into the tubes of the tubular bundle, becoming heated as it ascends. It isuses from the top of the tube. The methodical

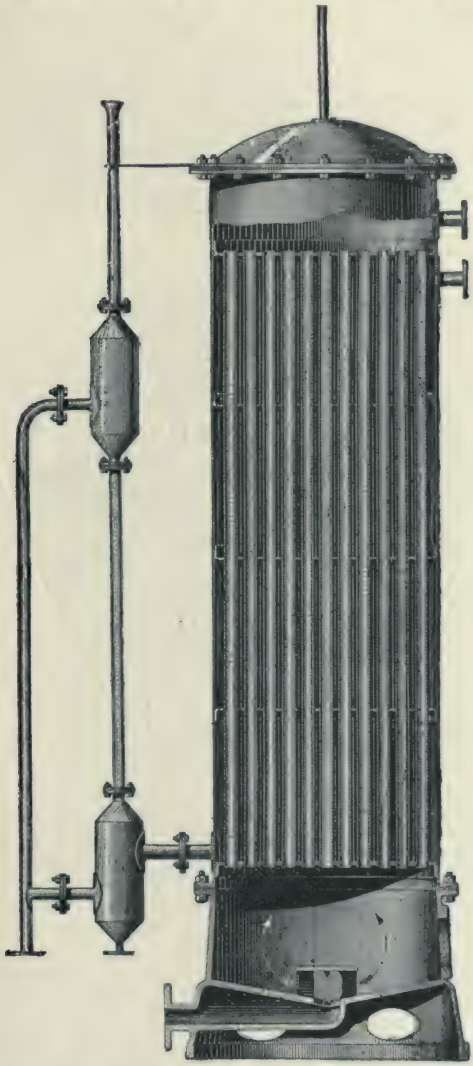


FIG. 49*a*.—Tubular vessels, condensers, recuperators, etc. (E. BARBET).

circulation of the spent wash is in the opposite direction. It enters by an upper pipe, is cooled as it descends, and issues by a pipe at the bottom. A reascending tube forces the tubular vessel to remain

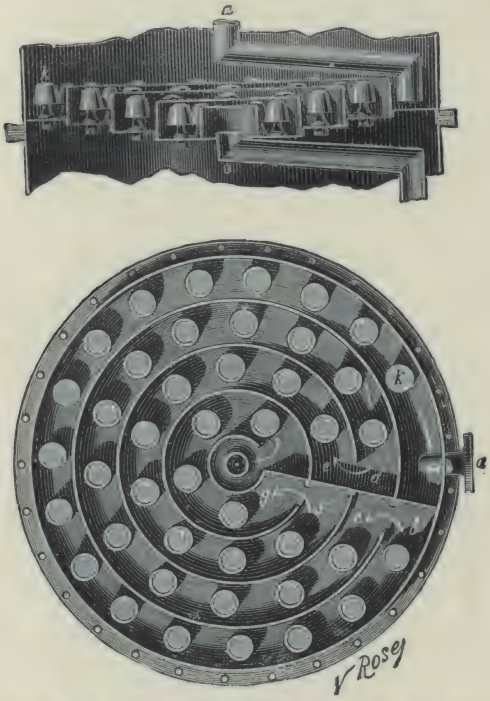


FIG. 50.—Vertical and cross section of distilling column, showing cascade arrangement of plates and caps (EGROT and GRANGÉ).

full. Fig. 50 shows the arrangement of Egrot and Grangé's plates. The liquid descends from the upper plate through the pipe *a*, traverses in the direction of the arrows the exterior ring *a b*, 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 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.

20. "*Blair's*" *continuous working steam stills.*—Fig. 51 shows "Blair's" patent continuous working steam still, constructed specially to produce high-class rum or spirit of best aroma and good quality,

FIG. 51.—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, & M'LEAN, Glasgow).

continuously, in one operation. By this still all the spirit is entirely exhausted from the wash, and a good spirit obtained, free from impurities, but retaining its aroma and flavour. The still, as shown, will produce spirit continuously 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.

21. *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, where 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 well 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, and the risk of bad exhaustion of the spent wash is greater, both items which entail an abnormal waste of steam. The advantages of the one type debar those of the other. Guillaume, in constructing his inclined column, aimed at combining the merits of both, and to eliminate their defects. The bottom of the 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, nor interruption, so as to force this circulation. The wash to be distilled enters by the top portion of this inclined column, and the spent wash issues from the bottom by means of the extraction regulator B. The heating steam enters through 2, passes through the partitions 3, 3^1, 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; 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 circulation of the wash, consisting of a continuous channel; and (2) the top, used as vapour trap and bubbling-cap chambers. It combines the good features of full columns and plate columns. Moreover, in order to inspect the whole of the interior, all that has 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

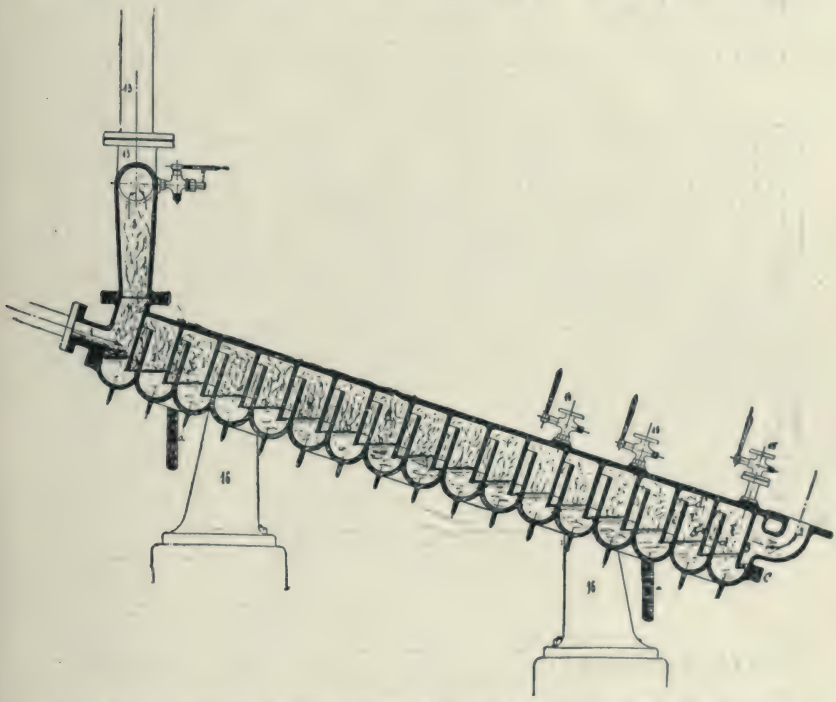


FIG. 52.—Guillaume's "inobstructable" inclined column, longitudinal section (EGROT and GRANGÉ).

again are thus done very rapidly. But copper columns which are lighter 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. So as to employ escape steam, an escape steam "balloon" is installed of a volume 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

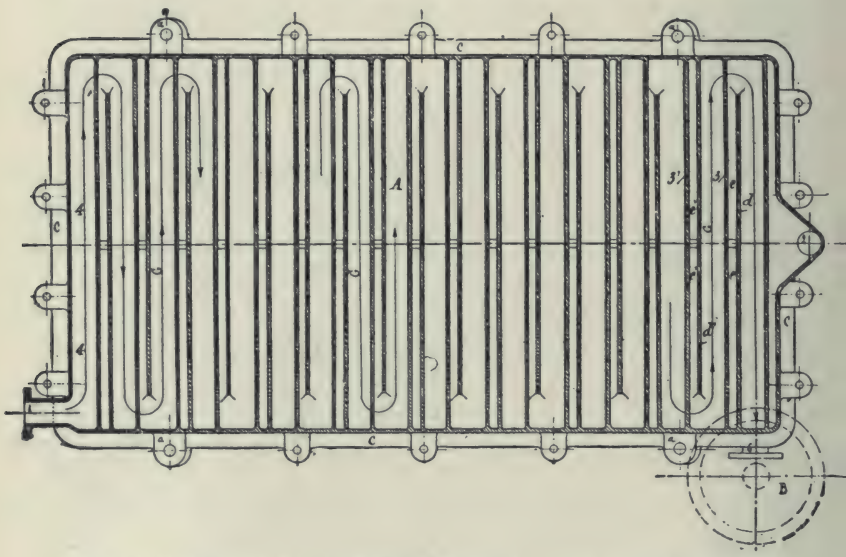


FIG. 53.—Guillaume's "inobstructable" inclined column, ground plan (EGROT and GRANGÉ).

when the interior pressure exceeds ½ kilogramme per square centimetre, and the steam entrance regulator is arranged so that the steam cannot enter the balloon until the pressure descends to 450 grammes, and that just in proportion to keep it at that pressure. By this arrangement the distilling column uses up the escape steam first of all, and only takes automatically direct steam when the escape steam becomes insufficient.

22. *Surface heating of the apparatus.*—When the wash cannot be diluted, surface heating has to be resorted to, using the arrangement shown in Fig. 54. The spent wash issuing from column A flows into the evaporator J, where they are boiled by a tubular

surface, itself heated by steam. The unevaporated vinasse issues automatically, regulated as to quantity by the wide open siphon C. The heating steam is admitted by the tap *d*, and its output is regulated by the valve which follows this tap, and which is wrought by the variable regulator O. The regulation of the heating of the plant is done automatically by Guillaume's automatic steam regulator, if the

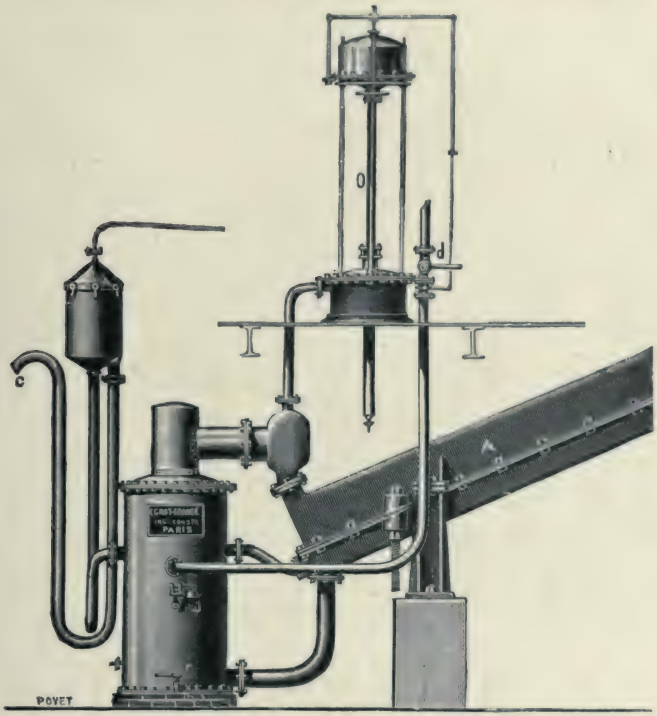


FIG. 54.—Guillaume's plant for surface heating of wash to be distilled which cannot be diluted by open steam (EGROT and GRANGÉ).

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 to the work to be done; that is to say, only to use what steam is necessary to exhaust the wash and no more.

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 more simple and preferable if liquid substances are being distilled. The apparatus used in working limpid liquids is furnished with a wash entrance regulating tank supported on the apparatus itself. Finally, the constructors supply a very solid iron foundation, on which all the parts of the apparatus are mounted in such a way that no support has to be fixed on the spot, and, each organ 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 further need for cleaning manholes, the bottom of the column being capable of being undone in a few minutes and the interior laid bare for inspection.

23. The type M^a of this distilling column (Fig. 55) is intended to produce raw spirits of 60°–70° G.L. (22°–26° Cartier). The inclined column A can 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. 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 under a small compass, which renders the fitting up easy and simplifies supervision. The exhaustion of the liquids distilled is absolute. It is economical because it suppresses loss of alcohol and expends 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 first on the spot. In cleaning there is no need for manholes. The inside is accessible in a few minutes by undoing 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 4 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 discharged through the siphon tube C after passing through the extractor D. The exhaustion of the vinasse is constantly verified by the test-glass G″, through which the liquid flowing from the condensation of the vapour from the spent wash may be inspected. This vapour is brought by pipe from *b*.

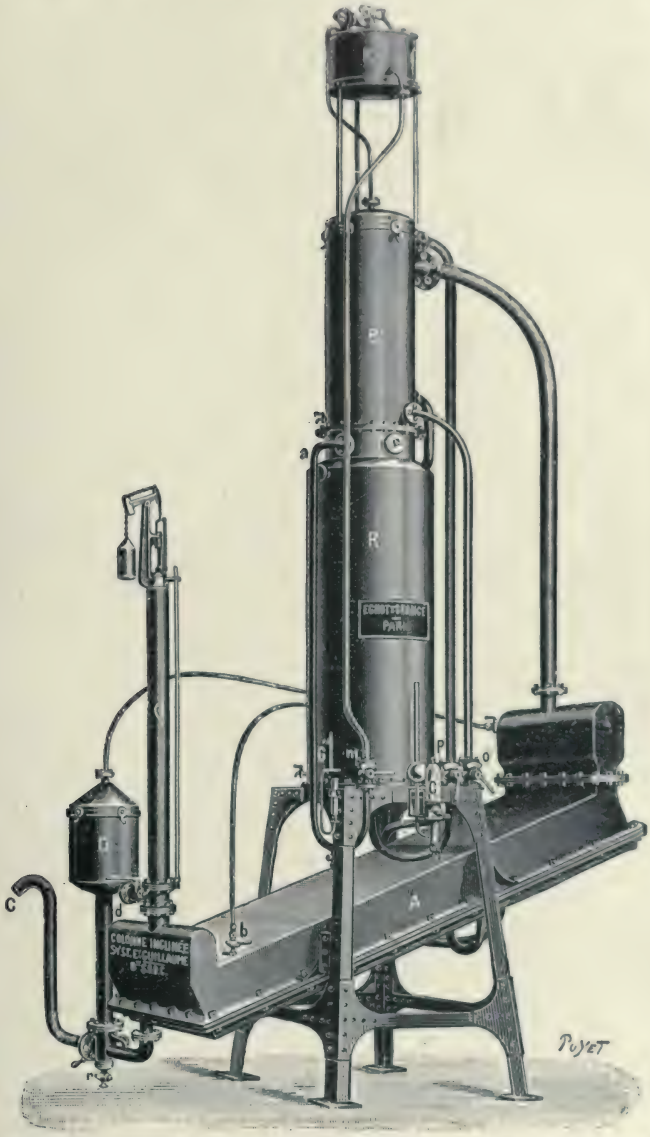


FIG. 55.—Guillaume's distilling column, Type Mª. A, distilling column ; *a*, entrance of wash into wash-heater ; B, wash-heater ; *b*, exhaust test-tap ; *c*, exit of spent wash ; D, spent wash extractor ; *d*, steam valve ; G, alcohol test-glass ; G″, exhaust test-glass ; K, feed regulating tank ; *m*, 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).

The heating 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.

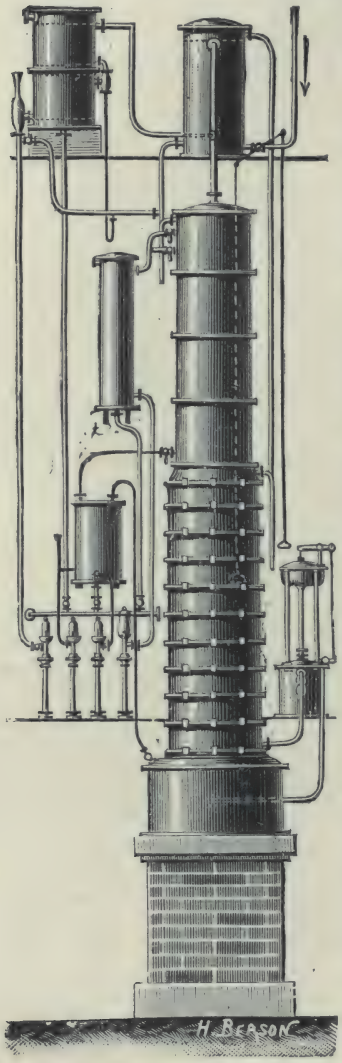


FIG. 56.—High strength rectifying column, model No. 3 (E. BARBET).

24. According to raw materials, and final quality aimed at, the result to be attained by distillation-rectification varies from one distillery to another. Take an existing distillery with rectification plant desirous of improving the quality of its spirits without double rectification, which would entail too much loss and too great an 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 column shown in Fig. 56. 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 minimum 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 produces is chemically very pure. It attained 96 per cent. at Clastres decolorised permanganate in 45 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 trace of odours of origin more perceptible, as there are no others to mask them. In distillation of wine, cider, fruits, cane sugar, the odours of origin are very precious, distinguishing brands affording evidence of genuineness highly appreciated and esteemed by consumers. But in distilling beets, the subsistent odour, 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 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. They must be concentrated. This is effected automatically by a small column much resembling Barbet's laboratory apparatus.

25. 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, and only containing 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. 57 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

with impunity be diminished to a minimum 2 and even 1 per cent., according to the impurities of the wash.

26. *Suppression of "mauvais gout" rectifiers.*—The concentration of oils and ethers are important to agricultural distilleries which

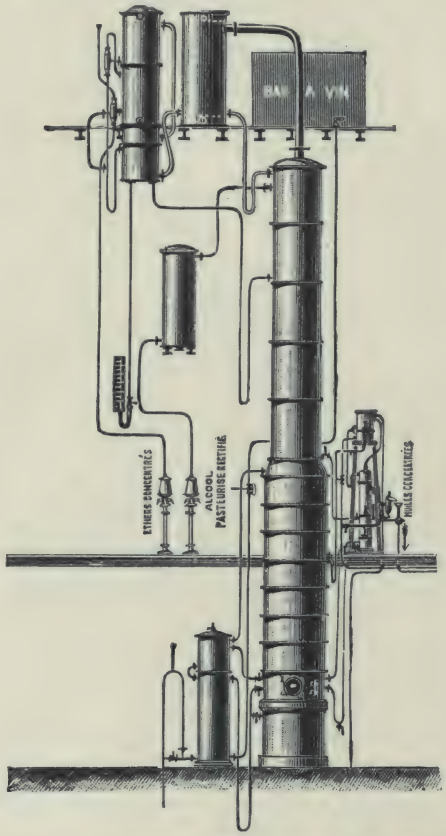


FIG. 57.—Continuous rectification column, with apparatus for concentrating oils (E. BARBET).

do not rectify, or for rum or brandy distilleries, for they economise the installation of 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 suppressed. Some distillers have no low produce apparatus, and return all the *mauvais gouts de tête et de queue* from the rectifier to the wash. But

that is bad working, for of two alternatives, 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, and then the oils are evaporated afresh and soil the phlegms. It then becomes impossible to produce a suitable alcohol by rectification, whilst with rectifying columns nothing is more legitimate than the return of the *moyens gouts* of rectification, the column giving a special outlet to both first and last runnings products. Accumulation therefore becomes impossible, and the 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 many distillers with old plant who boast of never selling *mauvais gouts de tête*, *i.e.* they use these products up by repassing them. It is a manufacturing loss, and therefore wrong.

27. *Twin column.*—Take a distillery to be erected, say a beet distillery, *only working three months a year*, an agricultural, and not a commercial company, desiring to make alcohol as good as possible, but aiming at making permanent customers with high grade alcohol. It desires 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°, and the oils are separated in the concentrated condition. The apparatus thus *seems* identical with the German or English twin columns. 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°, produced by the exhaustion plates, ascend the upper plates, where an assortment of alcoholic liquors, by 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 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 $2\frac{1}{2}$ per cent. of ethers are extracted for each hectolitre at 40°, one litre in the state of ethers will

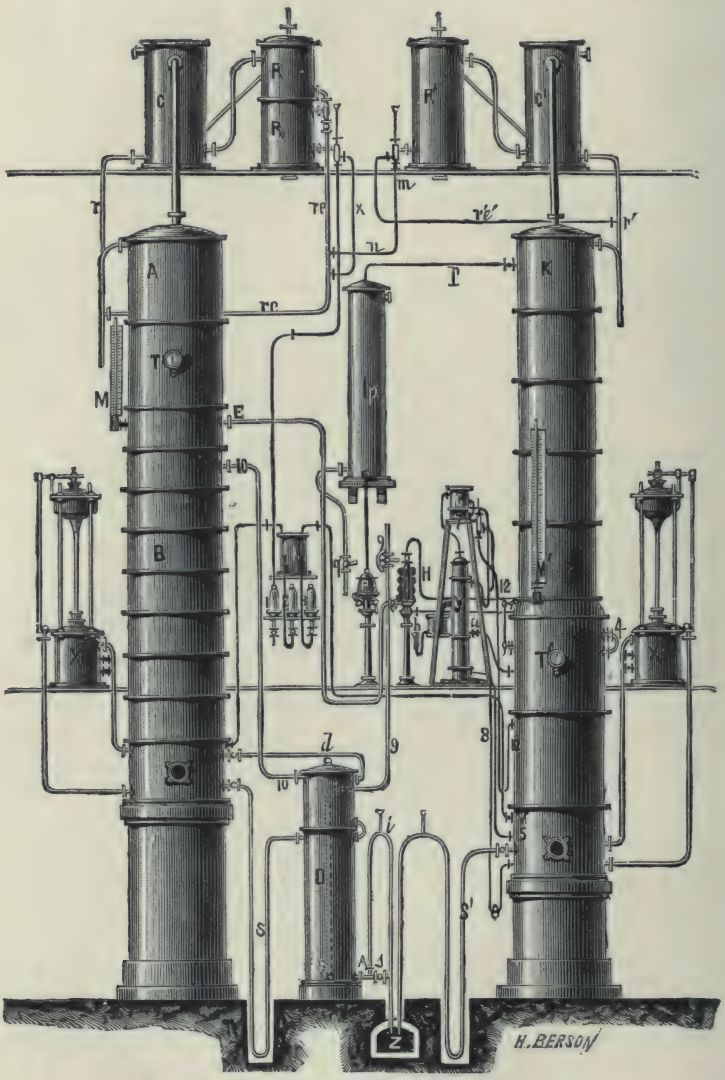


FIG. 58.—Twin rectifying column (E. BARBET).

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 thalpotasimeter T. The second column is like a continuous rectifier with fresh pasteurisation, the Fig. 58 shows that an oil concentrator like that described above has been 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, so far back as 1881, attempted to solve the problem by reserving the use of a thermometer in the lower part of the apparatus, where he thought the oils ought to occur. It was much too low down; 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 perforce regulated. Barbet thought in this way to cut short 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 of it. But it was a revelation at the same time. Instead of getting a liquor analogous to the phlegm, a horrible product issued, rotten with amylic oils, demonstrating that in that zone of the plant there was an accumulation of fusel which refused to issue at the bottom with the residual water. After extraction had gone on for some time, the product assumed its normal state as regards fusel. The apparatus was gradually desaturated; the problem of the continuous elimination of these impurities was solved. Only it was 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 underneath, and ended by recognising that the zone

of maximum infection was two or three plates below the feed plate, on a plate where the normal strength of the liquid was 40° to 50°. Hence, the continuous extraction of amylic oils at 40° to 50° both serves to extract these impurities and 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. It is the most sensitive stage. Barbet therefore installed a dial thermometer T at this level, as a complementary control visible from afar. 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 are charged with liquid, and all these distinct reservoirs added together represent a very imposing bulk of alcohol which constitutes the fly-wheel of the operation, to which it may be added that, when the feed is completely cut off, "bon gout" still flows for $2\frac{1}{2}$ hours, so great is the bulk of stock 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. 58), 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 leave the reservoir half-full, and, according as the thermometer T′ showed excess of alcohol or exhaustion, the tap 4 was adjusted so as either to let the reservoir get further charged or to let it empty itself. 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, there was a reservoir 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, and that 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. 62, 63, and 64, has set himself to

exaggerate the stock, and, according to Barbet, he has done so in such a way that whilst hyperbolising the inconveniences of the system, he has only introduced a useless complication. Far from regulating the working, he has, Barbet alleges, introduced a permanent cause of irregularity and inconvenience.

28. Whether it be required to rectify 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 to rectify 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^1, Fig. 59, fed by the refluxes from the rectifier, and by these alone, then possesses, like every other continuous distilling column, its exhaustion plates E, those of concentration E^1, 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^1. The plates E exhaust the refluxes, and the plates E^1 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 suppress filtration through charcoal.

29. By the continuous rectification of wash, merchantable alcohol may be obtained very pure on analysis. But such alcohols always retain trace of source odours caused by the too intimate contact of the alcohol at high strength with the fermentation gases. This drawback is very much in evidence 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

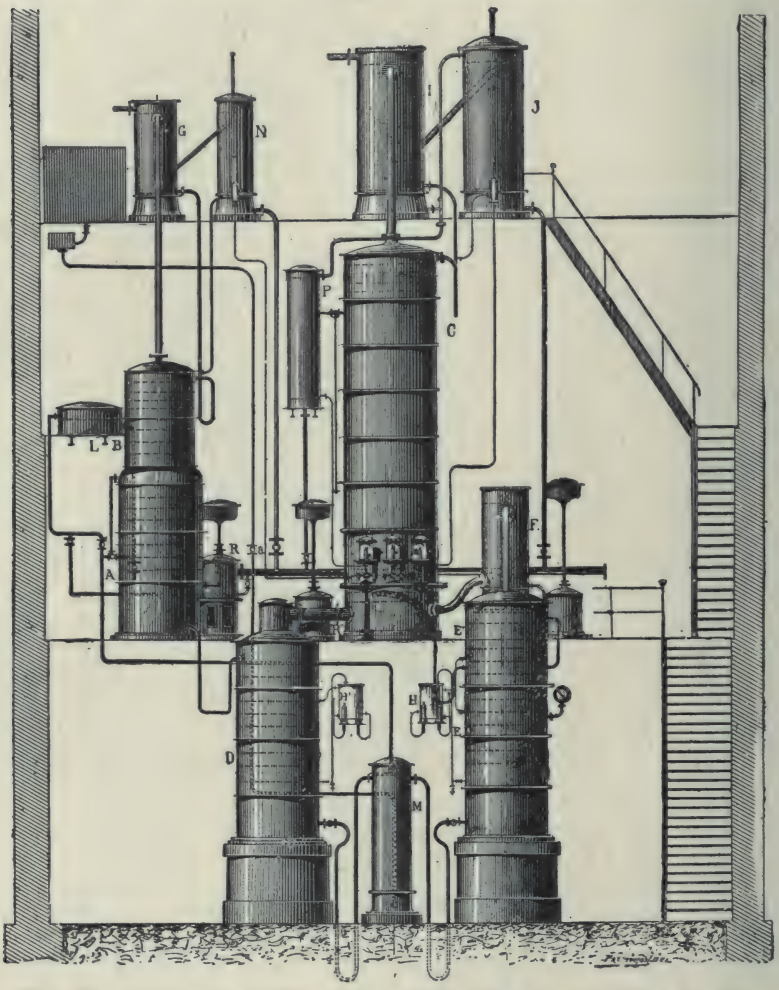


FIG. 59.—Continuous rectifier of raw spirit (phlegm), exhausting by two distinct columns both the purified phlegm and the amylic refluxes from the rectifying plates (E. BARBET).

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

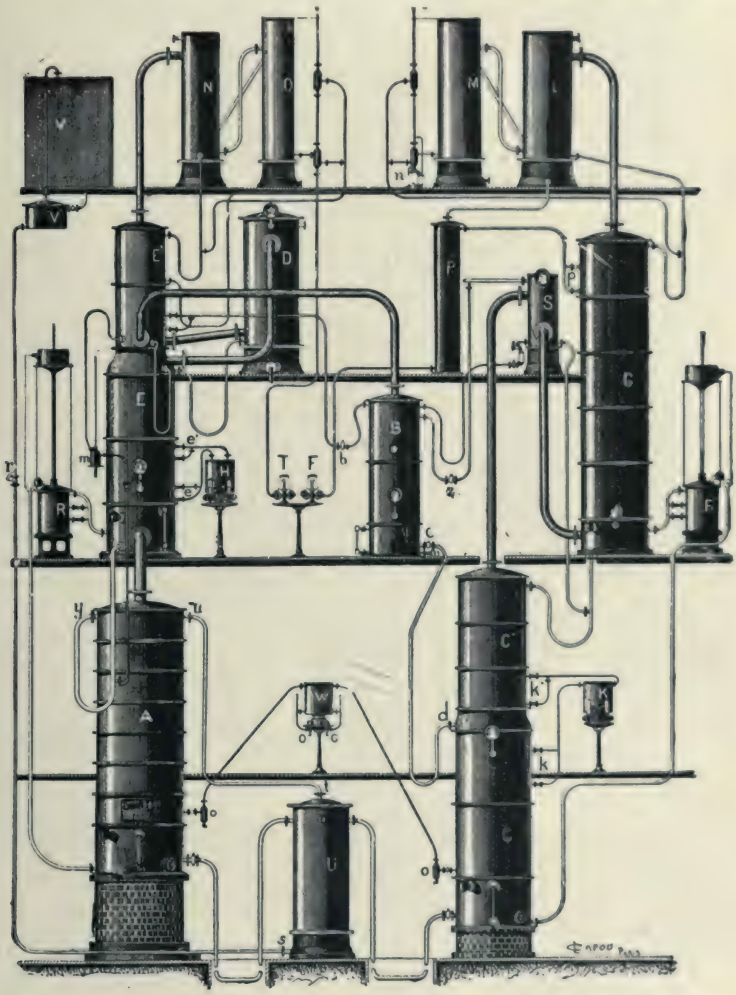


FIG. 60.—Plant for continuous indirect rectification of wash. V, feed tank (wash); V^1, feed regulator; A E E^1, rough purifying columns; N O, condenser and refrigerator; H, oils test-glass for rough purifier; T, first runnings from rough purifier; B, purifier; *d*, feed from purifier to rectifier C C^1; G, rectifier; U, recuperator forewarmer; L, condenser; M, refrigerator; P, refrigerator of pasteurised alcohol; F, test safe; K, fusel oil test safe (E. BARBET).

and perfectly free from odour or taste to betray its origin. The plant consumes but very little more coal than the direct rectifiers.

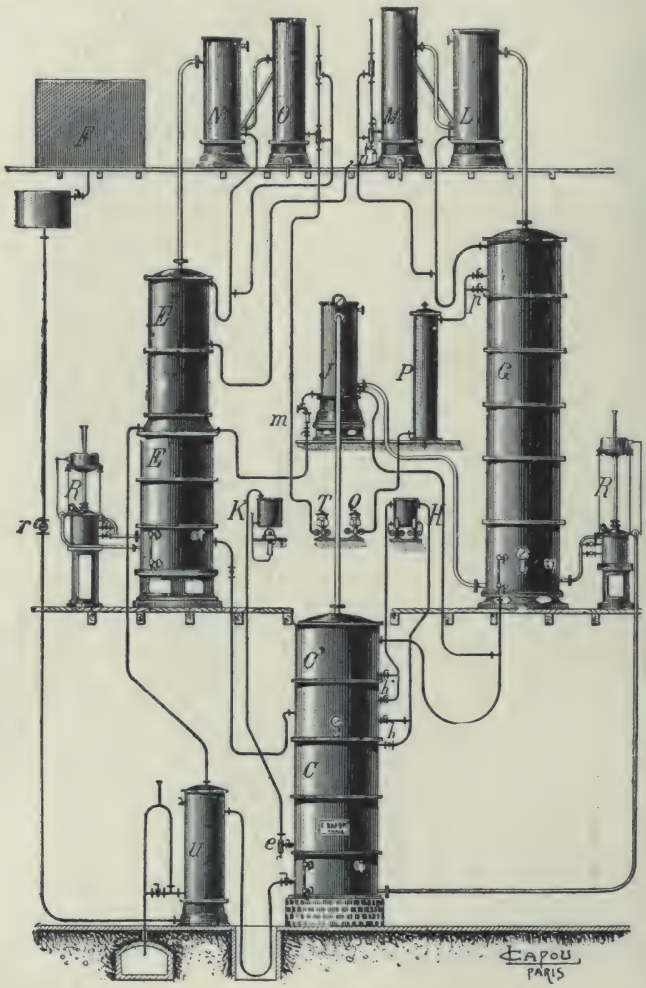


FIG. 61.—Continuous rectification of raw spirit (phlegm); F, feed tank with feed regulator; U, forewarmer; R R^1, steam regulators; E E^1, preliminary purifier; N, condenser; O, refrigerator for first runnings; J, special condenser; G, rectifier; C C^1, last runnings rectifier; P, refrigerator for pasteurised alcohol; T, test safe for first runnings; Q, test safe for pasteurised alcohol; K, exhaust test safe; H, test safe for oils (E. BARBET).

The exhausting column C (Fig. 61) is surmounted by a series of plates C^1, and a special condenser J. These new organs 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^1. Very pure alcohol results.

30. The production of good quality rectified alcohol, *i.e.* at least "commercial," required prior to Guillaume's researches, he says, two distinct operations: (1) Distillation, generally in continuous stills, consisting solely in extracting the alcohol from the wash, so as 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 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 easy thus 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.

31. 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 disarranged. 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 now to all continuous columns working up to great strength. With the latter, if the quantity of alcohol extracted at the gauges does not correspond exact at each instant 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, all at once 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 bad 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 show. And the state of affairs will last 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°, 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, soiled 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 (sec. 27)

contradicts all 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 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 repassing. 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 impart 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.

32. *Guillaume's direct distillation-rectification Type C, Agricultural* (Fig. 62).—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^1, gas refrigerator; N, distillation thalpotasimeter; 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; U U^1, 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^1 Y^2, 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 in § 21 *et seq.*, but this organ may be of another 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. 64, p. 183. (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^1 Y^2. 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

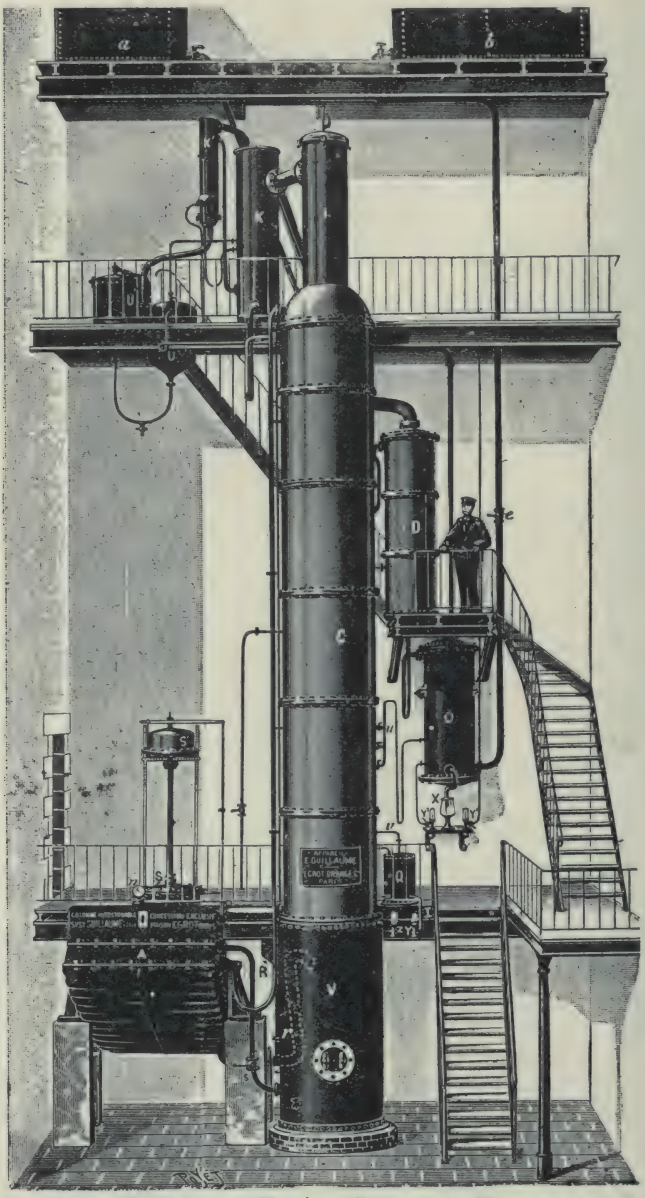


FIG. 62.—Direct distillation-rectification (GUILLAUME, EGROT, and GRANGÉ) Type C, Agricultural (sec. 32).

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 greater or less premium 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, and which is always guaranteed under 1 per cent. It is wrought by farm labourers formerly quite unacquainted with distillation. They become quite expert at working it in a few days.

33. *Guillaume's system of direct distillation-rectification Type B, Industrial* (Fig. 63). A, distilling column; *a*, tank for wash to be rectified; B, low strength purification column; B^1, column for concentration of first runnings products; *b*, water tank; C, rectifying column; D D^1, 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^1, gas refrigerator; O, "bon gout" alcohol and first runnings products refrigerator; *p* p^1, pressure indicators; Q, last runnings refrigerator; R, spent wash extractor; *r*, spent wash exit; S, steam (distillation) regulator; *s*, tap and pipe for leading wash to distilling column; T, steam (rectification) regulator; U U^1, water regulators; *u* u^1, return from rectification to first runnings products; V. Accumulating receiver; X, "bons gouts" test-glass; Y, first runnings test-glass; Y^1, 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. 62) 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 *bons gouts* of high quality obtained as first distillate by this apparatus varies with the substance treated, it 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^1. The major part of these products is extracted at the bottom of B^1, 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^1. 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

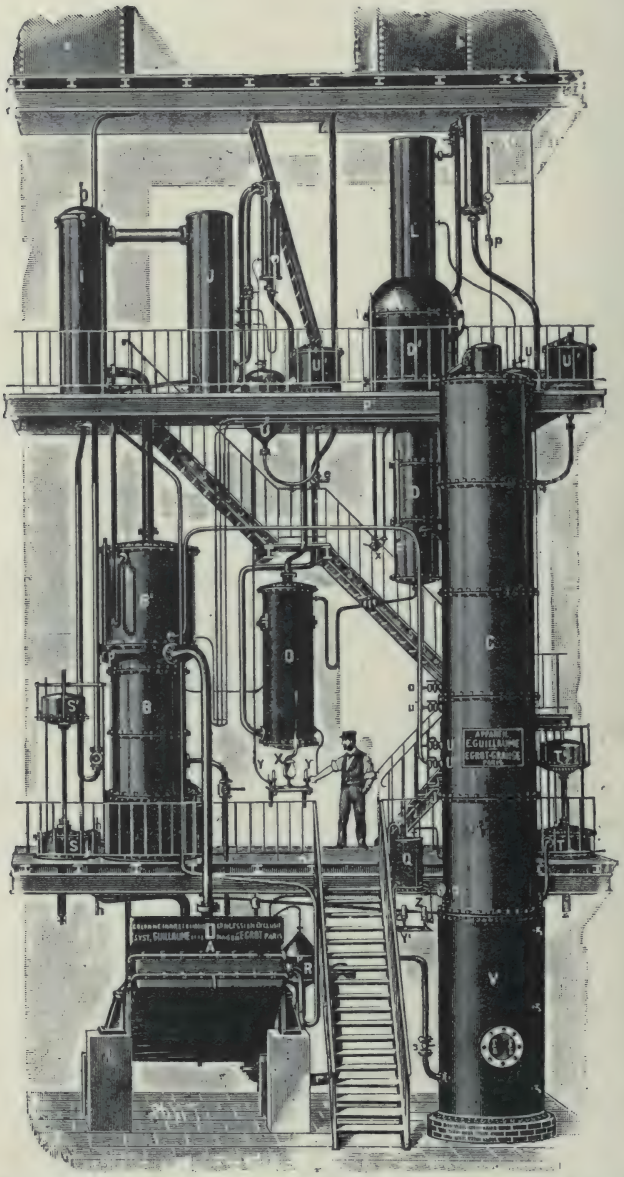


FIG. 63.—Direct distillation-rectification (GUILLAUME, EGROT, and GRANGÉ) Type B, Industrial (sec. 33), for beets, molasses, grain, potatoes.

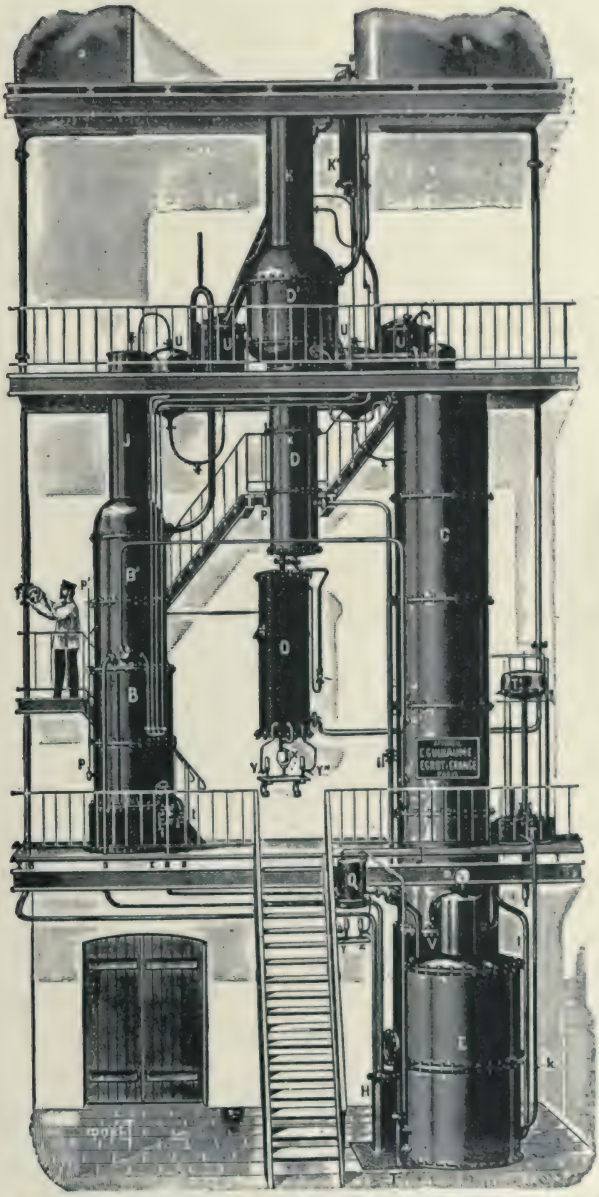


FIG. 64.—Continuous rectifier Type S (GUILLAUME, EGROT, and GRANGÉ) (sec. 34).

to the final purifying column D D^1, to be there freed from first runnings products which are reformed in the course of working.

34. *Guillaume's continuous rectifier Type S.*—Fig. 64, *a*, tank for raw spirit to be rectified; *b*, water tank; B, first runnings products concentrating column; C, rectification column; D D^1, 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^1, gas refrigerator; *k*, steam pipe; O, refrigerator; *o*, thalpotasimeter; *pp*, pressure gauges; Q, last runnings refrigerator; T, steam regulator; V, accumulating 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. 63) 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 retrogradation 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 obtained, 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: obtaining from 90 to 94 per cent. of "bon gout" alcohol in a single operation, 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. Absolute 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.

CHAPTER IX

THE MANUFACTURE AND USES OF VARIOUS ALCOHOL DERIVATIVES, ETC.

1. *Alcohol* is the parent of numerous compounds used in industry, many of which are almost of equal importance with itself. Want of space prevents us dealing with any but the more important compounds. Of the higher alcohols we can only treat of *amylic alcohol* (15) and its most important derivative, *amylic acetate* (16). Allied to ethylic alcohol are the spirituous products *methyl alcohol* (17) and *acetone* (18) obtained from wood spirit, a constituent of the wood tar and crude pyroligneous acid obtained by the destructive distillation of wood. *Ether* (3).—Amongst the substances directly derived from alcohol into which combined ethyl enters as an essential constituent, ether occupies the first place. Germany and France each produces 8 to 10 times as much ether as Great Britain. This is a serious matter as affecting the manufacture of smokeless powders. The ether is called sulphuric ether, as sulphuric acid is used in producing it. Ether is used by itself as a solvent in a great number of instances, also in conjunction with other solvents, particularly alcohol, as a solvent for many commercial products, more especially nitro-cellulose. It was used as an anæsthetic prior to chloroform, and is still used for the same humane purpose. Its use in refrigeration (cold storage) is well known. *Ethylic chloride* (4).—By the action of hydrochloric acid gas on alcohol, ethylic chloride is obtained. It is a liquid boiling at 12·5° C., often used as an anæsthetic, but several fatal accidents have recently resulted from its use. *Ethylic bromide* (5) *and iodide* (6) are produced by acting on alcohol with bromine and iodine in presence of amorphous phosphorus. These two heavy liquids are used in making intermediate products in coal-tar colour manufacture, which are the starting-points of at least 100 distinct dyes, each of which is a well-defined chemical compound. *Chloroform* (10).—This liquid, the humane use of which as an anæsthetic is well known, is manufactured in large quantities by heating alcohol with a solution of calcic chlorohypochlorite (*chloride of lime*). *Chloral* (13).—By the direct action of chlorine on alcohol, chloral is formed, and the latter treated by an alkali is decomposed into formic acid and chloroform. Both are well-known medicinal agents. *Bromoform* (11).—By the action of bromine on alcohol, bromoform is produced. It is prescribed as a remedy for

coughing. *Iodoform* (12).—By the action of iodine on alcohol, the well-known antiseptic iodoform is formed. *Paraldehyde, sulphonal, and urethane* are three substances all used as anæsthetics directly derived from alcohol.

2. *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 oxysalts of the acids. 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—ethylic chloride, ethylic bromide, ethylic 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:—

$$\begin{cases}\mathbf{C}H_3\\ \mathbf{C}OHO\end{cases} + KHO = \begin{cases}\mathbf{C}H_3\\ \mathbf{C}OKO\end{cases} + \mathbf{O}H_2$$

Acetic acid — Potassic hydrate — Potassic acetate — Water

$$\begin{cases}\mathbf{C}H_3\\ \mathbf{C}OHO\end{cases} + \mathbf{C}_2H_5HO = \begin{cases}\mathbf{C}H_3\\ \mathbf{C}OC_2H_5O\end{cases} + \mathbf{O}H_2$$

Acetic acid — Alcohol (Ethylic hydrate) — Ethylic acetate — Water

But as the hydrates of the organic radicals do not act upon acids so energetically as potassic hydrate, it is often advisable to employ the acid in the form of a potassic salt, and the radical as a sulphoacid; thus, with acids of the acetic series:—

$$\mathbf{C}_2H_5HSO_4 + \begin{matrix}\mathbf{C}H_3\\ \mathbf{C}OKO\end{matrix} = \begin{matrix}\mathbf{C}H_3\\ \mathbf{C}O(\mathbf{C}_2H_5)O\end{matrix} \quad \mathbf{H}KSO_4$$

Ethyl disulphate — Potassium acetate — Ethyl acetate — Potassium disulphate

3. SULPHURIC ETHER.—Ordinary ether $\left.\begin{matrix}C_2H_5\\ C_2H_5\end{matrix}\right\}O$—molecular weight, 74; boiling-point, 34·8° C.; flash point, – 20° C.—is prepared by the action of sulphuric acid on ordinary (ethylic) alcohol. On a small scale the process is conducted as follows: 9 parts concentrated sulphuric acid and 5 parts of alcohol are carefully mixed together, and, after cooling, heated to a temperature of 90° C., and a continuous stream of alcohol is 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° to 137° 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 body, from which it may be freed by adding caustic potash and allowing it to stand for twenty-four hours. The ether is decanted, washed with water to remove alcohol, and repeatedly rectified over calcic chloride; if it must be exceptionally pure, a small piece of sodium 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

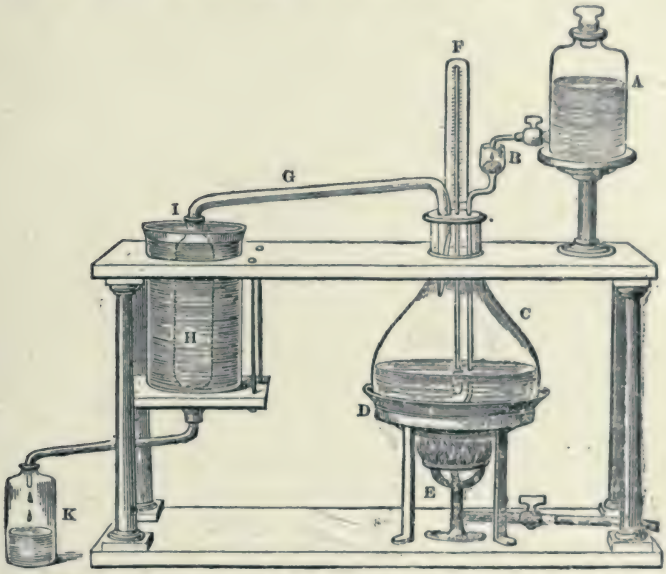


FIG. 65.—Laboratory apparatus for preparation of ether. A, alcohol vessel; B, tap and funnel regulating feed; C, etherifier; D, sand bath; E, gas burner; F, thermometer; G, tube leading vapour to condenser; H, condenser; I, refrigerator; K, receiver.

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, and the latter reacting on fresh alcohol reforms sulphovinic acid, and so on.

$$\underset{\text{Alcohol}}{C_2H_5HO} + \underset{\text{Sulphuric acid}}{H_2SO_4} = \underset{\text{Sulphovinic acid}}{C_2H_5HSO_4} + \underset{\text{Water}}{H_2O}$$

$$\underbrace{46 + 98}_{144} = \underbrace{126 + 18}_{144}$$

$$\underset{\substack{\text{Sulphuric}\\\text{acid}}}{C_2H_5HSO_4} + \underset{\text{Alcohol}}{C_2H_5HO} = \underset{\text{Ether}}{(C_2H_5)_2O} + \underset{\text{Sulphuric}}{H_2SO_4}$$

$$\underbrace{126 + 46}_{172} \qquad \underbrace{74 + 98}_{172}$$

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 of 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° C. it congeals, forming brilliant white plates. Ether is miscible in all proportions with alcohol, carbon disulphide, chloroform, wood spirit, and benzol; 36 parts pure ether dissolve 1 part of water, increasing thereby its density from ·720 to ·723 at 15°·5 C.; 9 parts of water dissolve 1 part of ether. From its solution in ether the water may be completely removed by, say, 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, liquesces to a thick syrupy fluid. Ether freely dissolves essential oils, gun cotton, most of the fatty and resinous substances, alkaloids, and in general all substances rich in carbon and hydrogen. Its vapour mixes rapidly with air and forms 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 ethylic or amylic 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

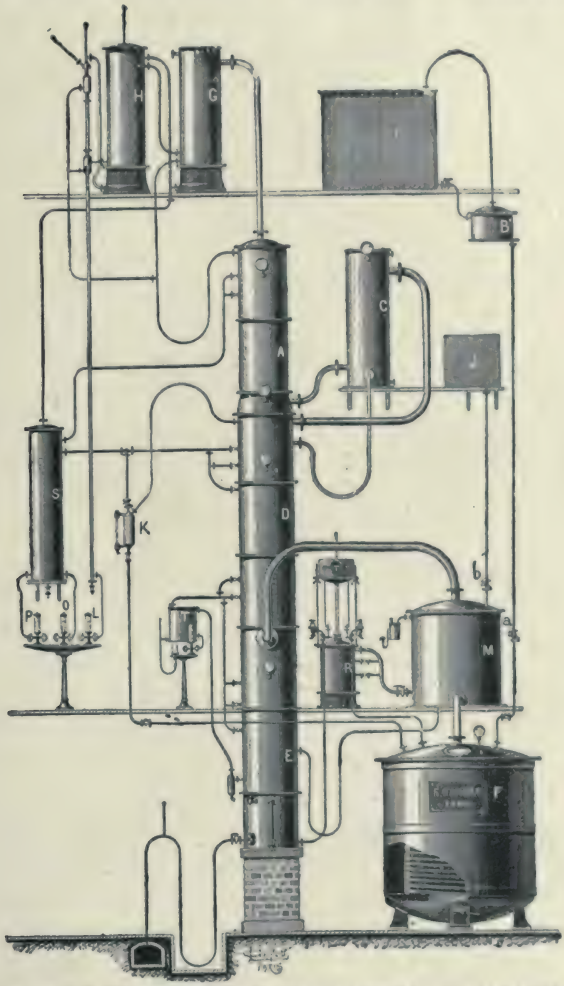


FIG. 66.—Plant for manufacture of ether (E. BARBET). B, Alcohol feed tank; B^1, regulating tank; *a*, regulating tap; F, coil heated etherifier (H_2SO_4 fed in through lid manhole); J, caustic soda tank; K, alcohol extraction (returned to F); M, saturator (excess of acid); *b*, caustic soda tap; R, steam regulator; A D E rectifier; C, special condenser; G, condenser; H, refrigerator; S, ether and alcohol condenser; P, ether test-glass; O, alcohol test-glass; L, first runnings test-glass; T, oils and exhaustion.

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 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 moment 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 shut, and not opened until 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° F., say 36 "degrees of frost."

4. *Ethyl chloride* (C_2H_5Cl); boiling-point, 11° C. (57°·8 F.)—*Sweet or dulcified spirit of salt* was a favourite preparation with the old chemists; they conceived it to possess some peculiar solvent powers in regard to 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 (*Mém. de l'Acad. Royale des Sciences*, v. 19). *Ethylic chloride* may be obtained by subjecting to careful distillation a concentrated solution of hydrochloric acid gas in alcohol; or a mixture of 1 part of alcohol, 1 of sulphuric acid, and 2 of fused and finely powdered chloride of sodium; or a mixture of chloride of antimony, or of chloride of tin, and alcohol. Groves leads HCl 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, as they react with alcohol in an analogous manner as with water—

$$PCl_3 + 3\ H_2O = P(OH)_3 + 3\ HCl$$

$$PCl_3 + 3\ C_2H_5HO = P(OH)_3 + 3\ C_2H_5Cl$$

In all these cases, ethyl chloride passes over; it should first be transmitted into warm water, by which its adhering 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.

5. *Ethylic bromide* (C_2H_5Br) is the bromine compound corresponding to ethylic chloride and iodide: it is made in an analogous manner to ethylic iodide.

6. *Ethylic iodide* (C_2H_5I); molecular weight, 156. 1 litre of ethylic iodide vapour weighs 78 criths ($78 \times 0{\cdot}0896$ grammes). Specific gravity, 1·9464. Boils at 72·2° C.

Preparation.—Ethylic iodide may be prepared in an analogous manner to ethylic 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 phosphorous and the iodine mutually interacting to hydriodic acid (HI) and phosphorous acid H_3PO_3. The proportions are—

	Brande.	Frankland.	Wurtz.	Joly.	Equation.
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:—

$$\underset{\text{Alcohol}}{3\,C_2H_5HO} + \underset{\text{Phosphorus}}{P} + \underset{\text{Iodine}}{I_3} = \underset{\text{Ethylic Iodide}}{3\,C_2H_5I} + \underset{\text{Phosphorous acid}}{H_3PO_3}$$

$$\underbrace{46 \times 3 = 138 + 31 + 127 \times 3 = 381}_{550} \qquad \underbrace{468 \qquad 82}_{550}$$

Wurtz used the apparatus described below, and the proportions 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 falls back into the flask, where the iodine and phosphorus decompose the alcohol with formation of ethylic iodide and phosphorous acid. When all the iodine has disappeared from the conical vessel, and the liquid in the flask is completely decolorised, the cone-shaped vessel is removed, and the liquid distilled on the water-bath as long as anything passes over. The product of the distillation being mixed with water, the ethylic 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 ethylic iodide is then dehydrated over calcium chloride and rectified. A more usual method of pre-

paration 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. Ethylic 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° C., it is resolved like methyl iodide into alcohol and hydriodic acid (Bernthsen). It is not easily inflammable, but when dropped on red-hot charcoal it diffuses 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 only slowly act upon it, sulphuric acid rapidly decomposes it. It decomposes argentic 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 formation of iodides. Under suitable conditions, the nascent ethyl may unite with the metals to form organo-metallic compounds.

According to Frankland, ethylic iodide, when heated with water in a sealed tube, produces ether and hydriodic acid—

$$\underset{\text{Ethylic iodide}}{2\,C_2H_5I} + \underset{\text{Water}}{\left\{\begin{matrix} H \\ O \\ H \end{matrix}\right.} = \underset{\text{Ether}}{\left\{\begin{matrix} C_2H_5 \\ O \\ C_2H_5 \end{matrix}\right.} + \underset{\text{Hydriodic acid}}{2\,HI}$$

7. *Ethylic 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, and contributes to their odour and flavour. It was discovered as far back as 1759 by the Count de Lauraguais (*Mém. 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 (cp. sec. 2), using either of the following formulæ :—

	A	B	C	D	E	F	G	H
Acetic acid . .	—	—	—	—	—	—	63	—
Potassic acetate .	3	—	—	—	—	—	—	—
Sodic acetate .	—	6	6	10	—	—	—	12⅜
Plumbic 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, as a rule, is 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 where 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 put aside. When the boiling-point rises to 165° 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 $\left(\frac{15^\circ\ C.}{15^\circ\ C.}\right)$; Perkin, 0·89; Dumas and Boullay, vapour density 3·03. It boils at 77°·5 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 . .	1	=	88	100·00	100·000	100·0

Acetic ether is intermediate in danger between absolute alcohol and sulphuric ether. It is not quite so extremely dangerous as ordinary ether. Hence it replaces the latter as a solvent when the risk is too great.

8. *Ethylic nitrite—Nitrous ether* ($C_2H_5NO_2$).—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*, 1. 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 (specific gravity, 0·820) and of nitric acid of commerce (specific gravity, 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 liquor begins to effervesce, then withdraw the fire, and 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 acids 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 cracking 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 not be too small, for, in consequence of the suddenness and abundance of its extrication, it requires a ready means of escape; indeed, the whole process requires much 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 10 parts of *rectified ether* are usually obtained from 100 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 sometimes is 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, and 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 (*Liebig's Condenser*). 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 mean 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 very 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 recent, 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, a part is dissolved, and another part decomposed, forming nitric acid, and giving off nitrous gas; mixed with solution of potash it soon forms potassium nitrite, 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 . .	1	=	75	100·00	100·00

9. *Ethylic nitrate* ($C_2H_5NO_3$).—Millon 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, as upon 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 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 ethylic ethers of caproic and caprylic acid. The product met with on the market under the name of Artificial Essence of Cognac is generally a mixture of the ethylic 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 cocoa nut oil, caprylic, caproic, and capric are used as a basic for artificial essence of cognac.

10. CHLOROFORM ($CHCl_3$).—This important body 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 baryta, yielded a dense fluid, viz. chloroform. The term chloroform refers to the constitution of formic acid (HCOHO), so that looking at it from that point of view chloroform is a trichloride of formyl (Brande). But it is better to regard it as marsh gas, CH_4, the old formene in which three atoms of hydrogen have been replaced by chlorine.

Preparation.—10 parts of chloride of lime and 3 parts of slacked lime are stirred up with 60 parts of warm water. The milky fluid thus obtained is placed in a capacious retort, and ought not to fill it more than one-third at the most; 2 parts of alcohol are then added, and the whole strongly heated. Towards 80° C. a very energetic action ensues, causing a very considerable frothing up. The heat is then withdrawn. Distillation commences and continues of its own accord. As the reaction ceases, it is again heated to carry on 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 a more or less liquid form 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 of an agreeable, ethereal, aromatic 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 percentage of ethylic alcohol protects it therefrom, and medicinal chloroform contains 2 or 3 per cent. of ethylic alcohol, so as to lower in 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 by 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 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 an extensive 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 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 bodies, 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 of potassium and formiate of potassa. 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 . . .	1		119·5	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 aqua 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 cc. with 15 cc. H_2SO_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, $C_2H_4Cl_2$ (oil of Dutch chemists).—Fused potassium hydrate is dissolved in absolute alcohol in 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 oil of Dutch chemists, 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 a sufficient evidence of the quality of chloroform in regard to alcohol test. A chloroform

with a density less than 1·48, at 15°·5 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 let 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 by a glass rod, as completely to neutralise the acid, so that, after gentle warming, the cooled liquid does not change 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 is 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 about 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 iodinised potassium iodide; liquor potassæ is then gradually added, until the colour of the liquid disappears. After 12 hours' standing in a conical glass, a crystalline deposit of iodoform will have taken place, 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.

$$CHCl_3 + 5KHO + 2CuO = Cu_2O + 3KCl + K_2CO_3 + 3H_2O$$

Chloroform	Caustic potash	Cupric oxide	Cuprous oxide	Potassic chloride	Potassic carbonate	Water

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 $C_6H_4 \left\{\begin{matrix} CO^1 \\ O^2 \end{matrix}\right\} 4$, with chloroform, the compound formed being $C_6H_4 \left\{\begin{matrix} CO^1 \\ O^2 \end{matrix}\right\} 4, 2CHCl_3$, the chloroform acting as water of crystallisation. By simple distillation it can be examined 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 a half per cent. in it 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 disadvantaged 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.[1]

11. *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 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.

12. *Iodoform* (CHI_3).—This body, which represents methylic iodide (CH_3I), of which two of hydrogen are replaced by iodine, was discovered by Serrulas. Dumas determined its composition. Bouchardat examined its properties. It is produced when iodine reacts in presence of an alkali or an alkaline carbonate on numerous organic bodies, *e.g.*, wood spirit, alcohol, ether, dextrine, gum, albumenoid

[1] Excise Committee Report and evidence.

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°–80°. Iodoform separates in crystals; the liquid is filtered, again brought to 60°–80° 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 potassa, 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 teriodide 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 of density 2·05. They melt between 115° C. and 120° C., and are volatilised in part without decomposition. It distils over with the vapour of water. It is insoluble in water and dissolves in ether, volatile oils, and fatty acids. Heated with an alcoholic solution of sodium ethylate, it forms methylenic iodide, CHI_2. It reacts on acetate of silver with formation of argentic iodide and liberation of carbonic acid.

$$CHI_3 + 3AgO = CO_2 + H_2O + 3AgI.$$

British makers of fine chemicals make iodoform. They crystallise from methylated spirit. It is very fortunate that iodoform smells so nasty, or people would complain of the British make as against the Germans', because there is a very distinct flavour of methylated spirit about it, even after all possible purification (Howard).

13. *Chloral* (Trichloracetaldehyde, CCl_3CHO). — 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° 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° 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° C., 200° 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 H_2SO_4 straight away 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, of a greasy aspect; its specific gravity at 18·3 C., 65° F., is 1·502; its boiling-point = 206° F.; and the density of its vapor 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 the contact of a few drops of water, the liquids combine into a white crystalline solid, and heat is 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 to itself, 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	1	147·5	100·00	100·00

Hydrate of chloral consists of 1 atom 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 down to 15° C.; at about 95° C. it boils, and is partly resolved into chloral and water, which, however, combine again, and

form a crystalline 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 gives no reaction when slightly acidulated with diluted nitric acid, with diluted solution of argentic nitrate, nor upon subsequent addition of aqua ammoniæ; 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 diluted 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 formiates 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 colour reagent for resins.

14. *Antipyrin.*—Febrifuge produced by action of aceto-acetic ether on phenyl hydrazin. Antipyrin was quoted before the 1904–5 Excise Committee as an instance of very large profits made out of one of these preparations by the patentees. A very large profit was made at one time on antipyrin. £60,000 a year was 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 that product, as it is a profitable product, if they could have alcohol sufficiently pure and sufficiently low-priced. Alcohol is by far the most important body used in the making of that compound. The following paragraphs are condensed from the evidence 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 weight as in the case of the colouring matters, but it must always be a hindrance.

Taking 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 and so on—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 get acetic ether at practically its short price, that is without the duty. Acetic ether functions in the manufacture of antipyrin and in the manufacture of aceto-acetic ether. Antipyrin is the methylated condensation product of phenyl hydrazine and aceto-acetic ether. Aceto-acetic ether could not be made for such a purpose from methylated spirit. Acetic ether in a pure state cannot be made 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 it were tried to make aceto-acetic ether from crude acetic ether. Pure acetic ether is necessary. There is the methyl alcohol and the acetones in methylated spirit which would prevent the formation of commercially pure aceto-acetic ether. Take it on the 10 per cent. of wood spirit, there would be somewhere about 7 per cent. of methyl alcohol and about 3 per cent. of acetones. 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, 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. There is no obligation to have, say, three-tenths of the amount acetone. 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.

15. *Amylic alcohol*, $C_5H_{12}O$; molecular weight, 87·81.—Scheele was acquainted with this body 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 with 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 body to come over in the rectification of alcohol (whether from malt, wine, potatoes, or beetroot), consists principally of amylic alcohol. Amylic alcohol is invariably present in fermentation alcohol, but how formed is unknown. Commercial fusel oils of different degrees of purity are to be found on

the market. Ordinary commercial only contains about 30 per cent. of pure amyl alcohol. To obtain it pure, the fusel oil is shaken with hot milk of lime, decanted, dried over calcium chloride, and rectified, that which passes over between 128 and 132° C. is collected apart. The portion distilling over below 128° C. contains butylic alcohol. Five c.c. mixed with concentrated sulphuric acid should only give a faint yellow or reddish colour. Commercial amylic alcohol is coloured black to blackish brown by sulphuric acid. Amylic alcohol, colourless when mixed with sulphuric acid, can only be obtained by repeated tedious treatment with concentrated sulphuric acid, and quite pure amyl alcohol can only be got by decomposing pure amyl sulphate. Amylic 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 extent 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° C. Its density at 15° 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° C. will stand 1 in 50 at 13°–14° C., and the solution becomes milky at 50° C. (Balbiano). It dissolves in all proportions in acetic acid diluted with own bulk of water. It deviates the plane of polarisation to the left, but to a different extent, according to its source. Ordinary amylic alcohol is a mixture in varying proportions of two isomerides, one of which is inactive and

TABLE XXI.—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	100·00

the other levo-rotary. By converting mixture into amyl sulphuric acids and neutralisation with barium carbonate, the two isomeric alcohols can be separated, as the barium salt of the active alcohol is $2\frac{1}{2}$ times more soluble in water than that of the inactive alcohol. Amylic 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.

16. *Amylic acetate.*—There are several possible isomeric acetates of amyl. The commercial amyl acetate known to chemists as iso-amyl acetate, $(CH_3)_2CH.\ CH_2CH_2O.\ C_2H_3O$, is a colourless mobile liquid with a pleasant aromatic, if somewhat suffocating, odour, recalling that of Jargonelle pears. It *may* be prepared by heating a mixture of iso-amylic alcohol with concentrated sulphuric and acetic acid, $CH^3.\ CO^2H + SO^4H^2 + C^5H^{11}OH = CH^3.\ CO^2C^5H^{11} + H^2SO^4 + H^2O$, 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 amyl alcohol, 1 part strong sulphuric acid, and 2 parts 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 down 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 amylic alcohol prepared beforehand in the proportion of—

Amylic 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 more pure amyl alcohol is replaced by the ordinary rectified fusel oil, consisting chiefly of a mixture of active and inactive primary iso-amyl alcohol, and acetate of lime is often used instead of the acetates of potassium or

sodium. As the ether always retains a little amylic alcohol, if it be desired to free it from such, 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° C. Sp. gr. 0·857. It dissolves in all proportions in 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.

17. METHYLIC ALCOHOL ($CH_3HO = 32$; B.P. = 66·6° C) may be obtained in the pure state from winter green oil, which consists of acid methyl salicylate $C_7H_4O_3HCH_3$ by distillation over potash, whereby potassium salicylate is formed and methyl alcohol distils over $C_7H_4O_3HCH_3 + KHO = C_7H_4O_2HK + CH_3HO$. 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 (25 lbs. steam). 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 (methylic alcohol), a volatile liquid extensively used in methylating, or, as they call it on the Continent, "denaturing," ordinary ethylic alcohol for industrial purposes. As the methylic 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 keeps back tarry matters and converts methylic acetate into methylic 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. 36, 37), or by Barbet's continuous process. Barbet has adapted the plant shown in Fig. 60 (p. 175) 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 turns 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 revivified 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 between 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 ethylic 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 if a mixture of 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) methylic formiate 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 140,000 gallons are produced from home-grown raw material. It is not a case of getting over a crude product from America, or some place, and purifying it here. There is a certain quantity which comes over from America, and is purified here, but the above 140,000 gallons are distilled from wood, in this country.

There is no wood-distilling done in Ireland. A gentleman from Bradford started works in Ireland, but after he got the concern into working order the men struck for higher wages, and the enterprise was discontinued. All hard woods, chiefly oak or beech, are suitable for distilling; elm is not a good wood for the purpose, and pinewood is worse. This alcohol, especially when crude, is more volatile, and its vapours more readily inflammable than those of ethylic alcohol; and, owing to the high acetone content of crude spirit, its vapour is explosive at the ordinary temperature. Flash point, 32° F.

18. *Methylic 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, of 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 carbides. 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 formiate 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, $CHCl_2$; $CHCl_3$; and 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	70·30
Chloride of methyl	1	50·5	100·00		1	50·5	100·00

19. *Methyl bromide* ($CH_3Br.$).—A colourless liquid prepared by acting on methyl alcohol with bromine in presence of phosphorus. It contains 53·7 of combined methyl calculated to methyl alcohol.

20. *Methyl iodide* (CH_3I) is prepared in a similar way to methyl bromide. The necessary quantity of iodine is dissolved in alcohol, and then run in a stream into a still provided with an agitator, and containing vitreous phosphorus. The methylic alcohol and iodine goes in a continuous stream into the still, and methyl iodide (D = 2·199; B.P. = 43·8° C.) issues in a continuous stream from the condenser.

21. The ethereal salts of methylic 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 as a solvent to replace *amylic acetate* as a solvent for nitro-cellulose in the facing up of cotton to resemble silk. The smell of the former if pleasant is too persistent.

22. ACETONE (*Pyroacetic spirit—Pyroacetic ether—Dimethyl-ketone—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.* lxiii. 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), by Liebig and Dumas, who established its composition in 1832. Gerhardt regarded it as acetylic methide. It has, in fact, been prepared by replacing the chlorine of acetylic chloride by methyl. The relationship between acetic acid, acetylic chloride, and acetone is that acetylic chloride—

$\left\{\begin{matrix}CH_3\\COCl\end{matrix}\right.$ is acetic acid $\left\{\begin{matrix}CH_3\\COHO\end{matrix}\right\}$, in which the hydroxyl is replaced by chlorine. In acetone $\left\{\begin{matrix}CH_3\\COCH_3\end{matrix}\right.$ 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 by acetylic chloride—

$$\underset{\text{Zinc methide}}{Zn\,(CH_3)_2} + \underset{\text{Acetylic chloride}}{2\left\{\begin{matrix}CH_3\\COCl\end{matrix}\right.} = \underset{\text{Zinc chloride}}{Zn\,Cl_2} + \underset{\text{Acetylic methide (Acetone)}}{2\left\{\begin{matrix}CH_3\\COCH_3\end{matrix}\right.}$$

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—

$\left\{\begin{matrix}CH_3\\COHO\end{matrix}\right.$	$\left\{\begin{matrix}CH_3\\COH\end{matrix}\right.$	$\left\{\begin{matrix}CH_3\\COMe\end{matrix}\right.$
Acetic acid	Acetic aldehyde	Acetone

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—

$$\underset{\text{Acetone}}{Me - CO - Me}$$

This composition is illustrated by the reaction which takes place when acetone is produced by the dry distillation of acetate of lime—

$$\underbrace{2\,CH_3 - COOca}_{\text{Calcium acetate}} = \begin{matrix} CH_3 \\ CH_3 \end{matrix}\!\!>\!CO + CaCO_3$$

Calcium acetate		Acetone		Carbonate of lime
158	=	50	+	100

Acetone may also be produced by the oxidation of isopropyl alcohol, which thus loses 2 atoms of hydrogen—

$$\underset{\text{Isopropyl alcohol}}{CH_3CH(OH)CH_3} + \underset{\text{Oxygen}}{O} = \underset{\text{Acetone}}{CH_3COCH_3} + \underset{\text{Water}}{H_2O}$$

By distilling together the salts of two different fatty acids, ketones containing two different alkyl radicles are obtained.

$$\underset{\text{Potassic butyrate}}{C_3H_7COKO} + \underset{\text{Potassium acetate}}{CH_3COKO} = \underset{\text{Propyl methyl ketone}}{\begin{matrix} C_3H_7 \\ CH_3 \end{matrix}\!\!>\!CO} + \underset{\text{Potassium carbonate}}{K_2CO_3}$$

In the same way, if a mixture of the acetate and propionate of calcium be distilled, methyl ethyl ketone is produced.

$$CH_3 - COOca + CH_3CH_2COOca = \begin{matrix} CH_3 \\ C_2H_5 \end{matrix}\!\!>\!CO + CaCO_3$$

Laboratory methods of preparing acetone on small scale.—When dry acetate of lime is carefully distilled in a *fireclay retort*, it yields a considerable portion 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 best process for the preparation of acetone. One part of dry quicklime and 2 parts of crystalline sugar of lead are well 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 gradually applied till red-hot. The crude product is a mixture of acetone, water, and two oily bodies; it is redistilled in a water-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 produced 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 such like impurities as *dumasin*, an isomeride of mesityle 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. 66*a*. 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 C. and methyl alcohol at 66°·5 C., there is a difference of 10° C. between their boiling-points, which suffices for fractionation. The product thus obtained can finally be purified by treatment with sodium or potassium bisulphates (thereby taking advantage of the fact first discovered by Limpricht, that acetone combines like aldehyde with alkaline bisulphite to form crystalline compounds), crystallising 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 as benzol.

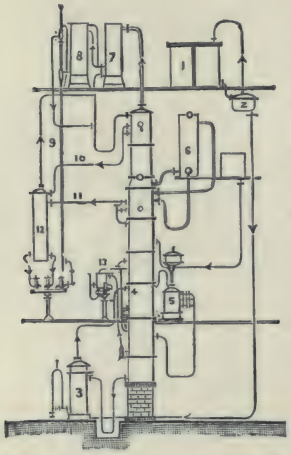


FIG. 66*a*. — Acetone still (E. BARBET, Paris). 1, Feed; 2, feed regulator; 3, forewarmer) 4, entrance of crude raw material; 5, steam regulator; 6, special condenser; 7, condenser; 8, refrigerator; 9, first runnings; 10, pasteurised acetone; 11, pasteurised methyl alcohol; 12, refrigerator; 13, oils and exhausts.

23. *Ketone oils.*—The salts which the higher homologues of acetic acid produce with alkaline salts yield on distillation the higher homologues of acetone: the so-called ketone oils used as solvents.

CHAPTER X

THE USES OF ALCOHOL IN MANUFACTURES, ETC.

1. The following is an alphabetical list of uses to which alcohol may be put, and of arts, crafts, and industries in which alcohol is an important factor, with reference letters and key showing the function of the alcohol in the industry.

2. *Key.*—(*a*) The alcohol acts as 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 vehicle for flavouring material fruit essence.

(*c*) As in (*a*), but solvent for waterproof and airproof materials in addition.

(*d*) Alcohol acts to bring substances into solution and as volatile vehicle and diluent.

(*e*) The alcohol acts as 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 acting agents, or as a necessary constituent of the mixed solvent.

(*r*) The alcohol solution of resin acts as vehicle for vitrifiable pigment.

(*s*) The alcohol acts as vehicle for flux.

(*t*) The alcohol acts as solvent volatile, or otherwise.

(*u*) The alcohol is used in candle polishing, or as vehicle for wick-curing chemicals.

(*v*) The alcohol acts as combustible fuel. There is also a "solidified" alcohol used as a fuel. In blackboard slating the alcohol is fired to produce a dull surface.

(*w*) The alcohol acts as a volatile vehicle for the tan.

(*x*) The ignited vapour of alcohol renders mantle incandescent.

(*y*) Alcohol is main raw material.

(*z*) 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*).
,, neroli (*f*).
,, perfumes (*k*).
,, shellac (*t*).
,, silk (*f*).
,, stone (*a*) (*h*).
,, teeth (*a*).
,, tortoiseshell (*a*).
Astronomical instruments (*a*).
Atropine and 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*).
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.
Clarionette (*a*).
Cleaning (*p*).
Clinometer (*a*).
Clockmaking (*a*).
Cocaine hydrochloride (*f*).
Cochineal tincture (*h*) (*t*).
Cold storage (*o*).
Collodion (*j*).
Compasses (*m*).
Copal varnish.
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*).

D

Decoration and decorative industries generally (*a*).
Disinfectants (*f*) (*g*).
Dog collars (*a*).
Dominoes (*a*).
Dragon's blood (*h*).
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*),
,, tanning (*t*).
,, 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*).
,, mahogany (*a*).
,, walnut (*a*).
Steel pens (*a*).
Stereoscopes (*a*).
Stethoscopes (*a*).
Street illumination (*x*).
Stoves (*v*).
Sugar (from molasses) (*p*) (*t*).
Sulphonal (*f*).
Surgery (*g*) (*e*).
Surveying instruments (*a*).
Syringes (*a*).

T

Tan extraction (*t*).
Tannic acid (*t*) (*u*).
Tanning leather (*w*).
Tapestry (*h*).
Tartan (*h*).
Thermometry (*m*).
Thymol (*f*).
Timber preservation (*e*).
Tin decoration (*a*).
,, soldering (*s*).
Tinacedine (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 (*x*).
Violet dyes, enamels, lacquers, varnishes, stains (*a*) (*h*).
Violin varnish (*a*) (*h*).

W

Waterproofing (*a*).
Water-pumping (*z*).
Weaving (*z*).
White lead (*p*).

Winnowers (fanners) (*z*).
Wood carving (*a*).
,, dyeing (*h*).
,, "enamelling" (*a*).
,, staining (*a*).
,, work machinery (*z*).
Wool dyeing (*h*).
Wood fat (*p*).

X

Xyloidin (*j*).

Y

Yacht building (*a*).
Yacht decoration (*a*) (*j*).
Yellow dyes (*h*).
,, enamels (*a*) (*j*).
,, lacquers (*a*) (*j*).
,, stains (*a*) (*j*).
,, varnishes (*a*) (*j*).

3. 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 such organic matter as is insoluble in water. As a solvent, alcohol is the antithesis of water. A resin dissolves in alcohol, it will not dissolve in water; water precipitates the resin from its alcoholic solution. Gum 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. Far more 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 commercially pure methyl alcohol and 1 litre petroleum benzine are used to denature 100 litres of alcohol for this purpose. In Belgium 500 grammes 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 many antiseptics, *e.g.* iodoform. *Artificial flowers.*—Alcohol is used as vehicle for dye to dye paper, and as vehicle for scent to perfume.

4. *Bedstead enamels.*—Almost pure alcohol, ½ litre of turps per 100 litres, is used in Germany as solvent for resin. *Bismarck brown.*—Much alcohol is used in dissolving this dye, and in dissolving resins in the alcoholic solution 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. No doubt it is used as a solvent for some ingredient 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, possibly as vehicle for lacquer for the brass, or as varnish for foundry patterns.

5. *Calico printing* is also an alcohol consuming industry, possibly as solvent for dye. *Candlemaking.*—Industrial alcohol is consumed possibly in treatment of wicks, or polishing of candles, or as 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.*—Here 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 ½ 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 over 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.

6. *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 close 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 more weak solution of a colour can be struck on to delicate fabrics from alcohol than from water—the two solvents act differently; suffice it to say, spirit penetrates more uniformly into the interstices of the fibre without manipulation than water, and does so more evenly and brings all the affinity of the fibre for the dye into play by a far more intimate contact than is possible with water.

7. In those instances where 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 engine-driving 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 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, liniments, lotions, cattle, and other medicines.*—The British consumption of methylated spirit for 1901 in the preparation of these often proprietary articles was 15,410 gallons. *Essential oils.*—Alcohol is used as the solvent vehicle medium, purification and crystallisation agent and diluent for sale.

8. *Fireworks.*—Many salts when dissolved in alcohol impart exceeding bright colours to its non-luminous flame, *e.g.* common salt colours it yellow; cupric chloride, bright green; boracic acid and barium chloride, pale green; strontium chloride and lithium chloride, bright red; potassium chloride, purple. Both alcohol and salts should be pure to get 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 solutions 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. They are made from fusel oil derivatives, *i.e.* from the last runnings in the rectification of raw spirit, and, curiously enough, 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 a very partial solvent indeed. Alcohol has, however, the advantage of being a solvent for dyes.

9. *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 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 permanent supervision of the Excise at the manufacturer's expense. *Gum resins.*—See under Varnish. Of true gum resins only the resinous ingredient is soluble in alcohol.

10. *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.

11. *Indiarubber.*—1. *Use of alcohol as a coagulant for the latex.* According to Morisse, "One volume of 90 per cent. alcohol coagulates 6 volumes of latex, yielding a fine superb rubber of brilliant whiteness, and yellowing but slightly on ageing, but the dearness 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 aid of a still, much of it might be recovered and used over again. It will be seen 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 drown the smell of coal-tar naptha, 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 of a sort of 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.

12. *Laboratory.*—The uses of alcohol in the laboratory are numerous. 1. As a solvent, 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 takes the place of 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 alcohol of concentrated 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 are 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 must prove invaluable in such situations. There is also a great future for alcohol engines 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 5 per cent. of petroleum. *Lincrusta Walton.*—In the case of pegamoid the function of the 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 rosinates, 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.

13. *Mahogany stain.*—Enormous quantities of alcohol, whether as 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 same, but also in preserving natural history specimens, *e.g.* serpents. Besides, it is used in the varnishes employed to coat 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.

14. *Oil refining.*—Alcohol is used as a solvent of free fatty acids and other impurities. In 1901, 1150 gallons of unmineralised methylated spirit were used for *paint cleaning*, according to the Excise. Most likely the alcohol in question was used as a remover ingredient 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 the vehicle for the perfume. It also acts as a diluent. 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.

15. The *perfumery* industry consumes enormous quantities of alcohol. 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 a sort of converse of the principle of the purification of animal fats, so as to render them edible, to which reference has already been made. In the former case the alcohol is used to remove the bad-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 started in the competition for this trade without a single advantage in the race, 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 lemons, 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 built up synthetically, all by German chemists. Both natural and synthetic perfumes come upon the market as alcoholic solutions.

16. *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 24 hours, but there is no disadvantage in adding at the same time 10 per cent. of the 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; as it is, absolute alcohol costs 4s. 4d. per lb. 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, because they must get the negatives dried quickly. Methylated spirit is not suitable. 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 of the mixtures used to dilute it. Throughout the trade a great deal of collodion is used that is made with methylated spirit. A proportion is made with methylated spirit, but few commercial collodions are made entirely with it because they give too much trouble. They foul the silver-bath, and you cannot get the same class of negative. The dot in the half-tone process has ragged edges instead of cleanly and sharply defined edges. *Colour photography.*—Alcohol figures largely in photography in colours, *e.g.* Lippman uses, *inter alia*, in the making of his gelatine plates, an alcoholic solution of cyanine. He 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 0·5.

17. *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 aided by crape and embroidery manufacture consumed 834 gallons of alcohol, mainly for stiffening in 1901. *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 further on. No. 2 Solution: Dissolve 25 grammes of common 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 mirror to be silvered (1) with nitric acid, (2) with a mixture of equal parts of solution 3 *and alcohol*, then the mirror is washed, standing it in the distilled water. Then 100 c.c. of No. 1 solution are run into a test-tube, then poured into a saucer or plate according to 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 mirror which has remained under water is brought rapidly into the liquid and kept at ½ centimetre from the bottom of the dish, taking care to agitate it gently. If the solutions have been well made, the transparency of solution No. 1 is not altered when the mixtures of solutions Nos. 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. But 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.

18. *Spirit stains.*—Alcohol is a solvent and vehicle for 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 come away at normal temperatures. As the soap is kept, it gets 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, which can only be used in 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 nature of the soap which tend to make it a superior article. The real object of using alcohol is to eliminate the excess of the alkali and the other uncombined materials from the true soap-forming substance. The principal object in using alcohol in the finishing of soap, is to bring about a closer chemical combination between the alkali and the grease. *Sugar—Use of alcohol in extraction of sugar from molasses.*—Again, alcohol has long been used

to extract the impurities from lime sucrate, so as to recover sugar from molasses. The alcoholic processes of extracting sugar from molasses have, however, nearly all been abandoned. There, however, remain a few applications of Manoury's system in Germany and Russia.

19. *Tannin.*—Alcohol is largely used abroad in the extraction of tannic acid from tanniferous substances. *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 not only is alcohol used to extract the tan from gall nuts and other tanniferous products, but here, and this is a somewhat novel if recent use of alcohol, the alcoholic solution of tannin is used to tan leather. We have only to go one step further, that is, to cure, tan, and dye the skin with one application of one solution. That will no doubt be done in the not far-distant future, and here again alcohol will come once more successfully into play.

20. *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 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, and there is now no tax on malt. France converted in 1901, 50,576 hectolitres of alcohol directly into vinegar, say, 1,112,672 bulk gallons; Germany in 1903, 3,624,588 gallons; Switzerland in 1903, 60,980 gallons; Belgium in 1903, 240,548 gallons.

21. *Waste products from distilleries, Utilisation of.*—Amongst products obtained from distillery residuals may be mentioned *acetal and furfurol*, both of which find a use in coal-tar colour manufacture. *Butyric ether* is also another product, with the odour of pine-apples, obtained from distillery residuals. Methyl chloride is obtained from beet distillery spent wash. Amylic acetate is got 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 XI

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 products—*i.e.* coal tar, petroleum, and shale naphthas,—capable of imparting that luminosity to the alcohol which it lacks. (2) Rendering certain earths incandescent by the ignition of the vapour of alcohol previously mixed with air.

1. The enrichment of alcohol with liquids capable of burning with highly luminous flames was the object of numerous researches during the latter part of the first half of the nineteenth century. Spirits 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 spirits 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 spirits of turpentine, than with the view of invoking the aid of spirits 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 a "burning oil" from a mixture of alcohol and hydrocarbons were made about 1832. About that time 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 only a more or less well-rectified spirits of turpentine pure and simple. The French gasogene was a mixture in predetermined proportions of ethylic alcohol with wood spirits, spirits of turpentine, naphtha, shale oil, etc. Both French and British patents insisted, as was natural, on the alcohol being free from water, as otherwise the mixtures would separate into as many layers as there were ingredients in the mixture. Besides, it was insisted upon that the mixture should be distilled prior to use. The light afforded by the combustion of this fluid in suitable lamps—camphine and gasogene (vapour) lamps—was white and pleasant. The inventors

all claimed this method of illumination as generally suitable, but particularly so for distilleries. Amongst the numerous patents for this style of illumination, that of Ludersdorf of Berlin, of date 1834, is the most simple. Ludersdorf used a mixture of four volumes of 95 per cent. alcohol and one volume of rectified spirits 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, and it is the vapour thus produced which burns with a fine white flame. Moreover, this mixed fluid, if it yielded a less intense light than that got by the use of turpentine alone, was more easily managed and less liable to smoke owing to its lower carbon content. Further, the inherent difficulty of the diminution of capillarity in the wick incidental to the combustion of resinous products and due to the deposition of carbon in the pores of the wick was overcome, and the lamp was lit at the outset by burning a little alcohol in a cup fixed round the tube containing the wick, so as to vaporise the alcohol 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, so as to prevent the spirit in the reservoir getting overheated.

2. *Rendering a mantle of the rare earths incandescent by the ignition of the vapour of alcohol.*—The alcohol is first of all vaporised, and the vapour is burned in a Bunsen burner capped by an incandescent mantle on the Auer principle, or by some similar mantle. This latter method is simply a variation of the principle on which the limelight is based. The intense luminosity of the limelight is due to the heat of an oxyhydrogen flame impinging on quicklime, the intense heat rendering the lime incandescent. The ordinary incandescent gas mantle is rendered intensely luminous by the coal-gas being burnt in admixture with excess of air in a Bunsen burner. This mixture of coal-gas and air burns in air with a non-luminous flame, but the heat generated is so intense as to render the mantle incandescent. If the mantle were not made of intractable material it would fuse it, and the heat would be used up in fusing it and keeping it in a state of fusion. But as the heat cannot be used up 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 did not occupy much time. Incandescent alcohol lamps were in fact the natural sequence of incandescent gas burners. But as a matter of history that is not quite correct. It would be more correct to say that incandescent gas 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 appropriate tissue, steeped in a paste of lime, magnesia, water, and gum arabic. The preparation of the paste, the method of shaping the tissue cone, steeped in this paste and dried, 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 only differs 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 imitators, as the numerous English and German patents for this style of lighting testify. But it was not until 1895 that a really practical solution to the point raised by Frankenstein was found in Auer's mantle, and in improved lamps which yield a fine light with a moderate consumption of methylated spirit. But the most remarkable point of all is that, however imperfect Frankenstein's system of alcoholic incandescent lighting may have been, it probably afforded a clue or a starting-point to Auer. In all probability it served as the basis of his system of incandescent gas lighting, which in its turn has served as the basis of a new and practical system of incandescent lighting by alcohol, a system of illumination now in its infancy, but of which much is to be expected, much to be gained. 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 and 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 have table lamps with the incandescent light. The incandescent light was formerly only possible practically with gas, although paraffin lamps have been made for an incandescent mantle. It is stated that one firm sold 53,000 alcohol 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 got 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 up by 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 great force through the small cone of the burner and to suck in a draught of air, and the alcohol-charged air, on reaching the upper part of the jet, burns with an extremely hot but non-luminous flame, which immediately renders the Auer mantle of incandescent tissues of the rare earths incandescent. It has been urged against this method that it 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; it, however, burns regularly and very cheaply, but, as will be seen, it is somewhat dangerous, and does not admit 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 gasifier, in which the alcohol is vaporised, is fed with alcohol by means of wicks. The alcohol vapour issues from the injector, sucks in an exactly regulated draught 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 by raising or lowering at will a hooked tube to a greater or less extent. To light the lamp, the hooked tube is lowered to the bottom, the small jet lit, and in a minute or so the lamp is lighted by holding a light of some kind at the top end of the glass chimney. The mantles hitherto used for alcoholic lamps are those made for use with coal-gas, without regard being had 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 determined on by the result of the experi-

ments of Landolt and Hintz, who found that mantles of the same dimensions, steeped in solution of the same concentration, gave, according to the relative proportion of oxide of cerium, the following luminous intensity estimated in carcels of 9·62 bougies :—

TABLE XXII. LUMINOUS INTENSITY OF INCANDESCENT MANTLES.

Thorium.	Cerium.	Carcels.	Thorium.	Cerium.	Carcels.	Thorium.	Cerium.	Carcels.
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

3. The effect of the Auer mantle on the luminosity of an alcohol flame is very wonderful. 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 excess 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 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, and the latter immediately becomes 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, and acted only fairly well. But other makers succeeded in doing better, so that 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.

4. Jean Delamotte's alcohol (Figs. 67, 68) vapour lamp has neither heating jet nor recuperator. The alcohol is drawn into the heater A (Fig. 68) 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 simply 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 orifices *o* first becomes alight itself, warms the heater A, then lights the mixed gases above the orifice of the Bunsen D, and the mantle Z becomes 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" is 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 it being necessary to bring a 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 quite vertical stride-legs across 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 button is turned to the right. In two or three seconds the button is given two turns to the left, so as to open it, and then it is left so. This manœuvre is necessary. In a word, the tap of the lamp should always be open, it is only closed for a moment to extinguish the light. *The mantle.*—Take the mantle in its box,

FIG. 67.

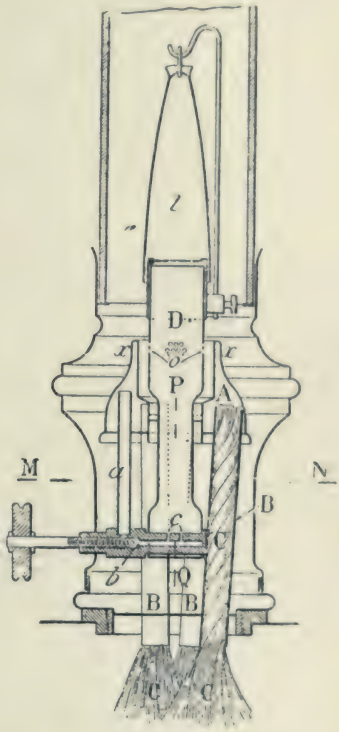


FIG. 68.

Alcohol vapour lamp on incandescent principle for domestic illumination (essential parts) (DELAMOTTE, Paris).

being careful not to squeeze it in the hand. It is far better not to touch it, and to seize it, 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 it on the socket, pressing the screw seen in. Fig. 68. The mantle fixed, the socket is placed on the Bunsen. The first time a 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 small layer of collodion with which it is covered so as to protect it before being brought into use, is instantaneously burned. *Wicks.*—These last very long 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.

5. The working arrangements of the "Monople" lamp for indoor and outdoor illumination are shown in Fig. 69. The alcohol in the circular reservoir *a* descends through *c* into another small, lower circular receptacle into which the extremities of tubes lined with asbestos dip, which suck up the alcohol, which is then vaporised by the heat of the mantle, and the vapour passes 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 *a a* is to ensure the regularity of the flame, by automatically regulating the feeding of the alcohol into the vaporisers by hydrostatic pressure. But it is urged against this arrangement 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.

6. The use of petroleum lamps in projection of light appliances presents many inconveniences of which all public lecturers are aware. So as to avoid the bad smell and the smoke, alcohol lamps have been invented for use in halls not fitted up either with gas or electricity. These alcohol lamps are fitted with an Auer mantle, which the ignited mixture of vapour of alcohol and air renders incandescent. The chief defect of these mantles is their great fragility. The conical form of the mantle causes, moreover, 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 has another advantage, the price of 6 sheets is almost 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. 71) has been calculated so as to feed the lamp in a continuous manner and with maximum intensity for two hours at least. Ordinary methylated spirit is run in through the screw stopper *b*, 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, at 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, when that is fed by the vapour produced in A, led by the tube *t*, and mixed with the air

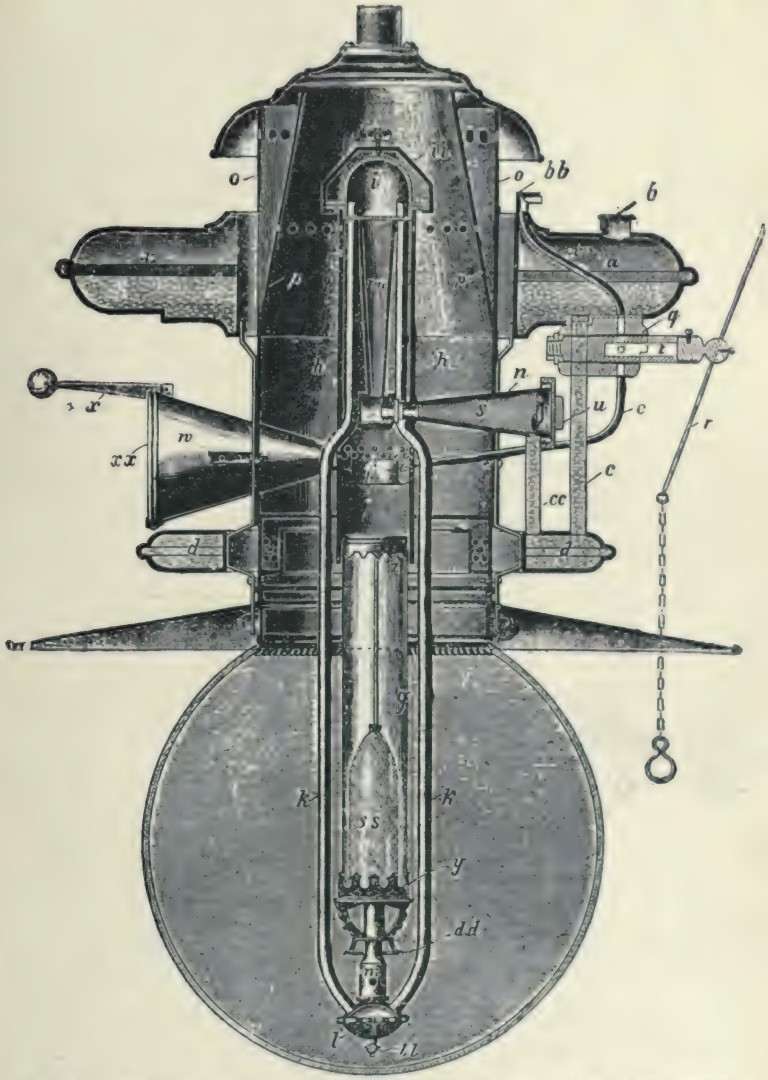


FIG. 69.—Monopole lamp for indoor and outdoor illumination (DELAMOTTE, Paris). *a*, reservoir ; *b*, aperture for filling ; *c*, feed : *d*, regulator ; *e*, pipe leading alcohol to *f* ; *f*, receptacle for lighting ; *g*, lamp glass ; *h*, cylinder prolonging lamp glass ; *i*, vaporiser ; *k*, alcoholic 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.)

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 alcoholic vapour contained in R 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* and produce a cracking sound in O, where they might become inflamed. They may even be projected by the jet B on the

FIG. 70.

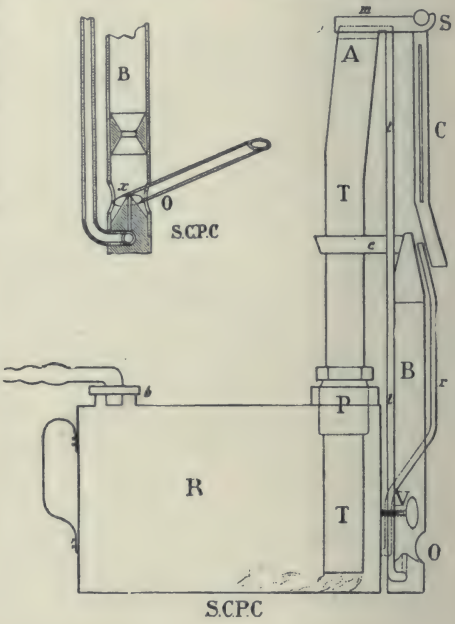


FIG. 71.

Alcohol vapour incandescent lamps, for magic lantern demonstrations, with flat incandescent film instead of conical mantle. Fig. 70, general view of lamp. Fig. 71, sections. The top section to left shows method of extinguishing.

tissue of the frame C and damage it. All 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 happens in two or three minutes. If the profile of this flame be examined, it seems 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 ought to 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 to and from the flame. The frame C is easily removed, opened, shut, replaced 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. By 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 condensator. 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* right to the bottom. On the first occasion, this should be done half an hour beforehand; it is necessary for the wick in the tube T to get 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 cue frame.

7. *Alcohol heating apparatus.*—Polo's alcohol vapour furnace (Figs. 72, 73) is a type of this class of stove. Other forms only differ in detail. It consists of a cylindrical reservoir, on the periphery 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 feeding a perforated ring which touches the flat brass socket *s*, already referred to, on both its flat sides. The furnace is lit 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 tap *f*, the vaporisation of the alcohol depends on the orifice

of this tap and the height of the flame coming from it. According to Lindet, all these heaters, whether with or without wicks with simple

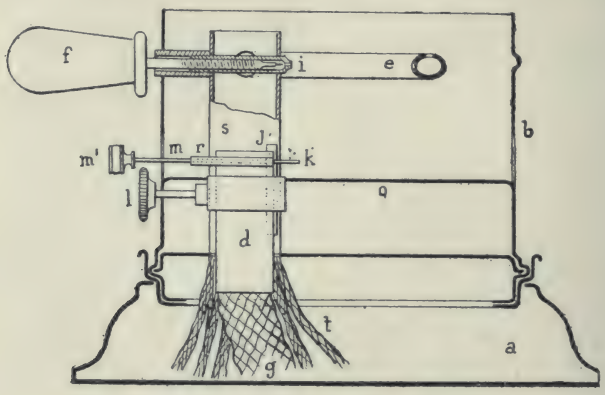


FIG. 72.—Polo's alcohol vapour heater (section).

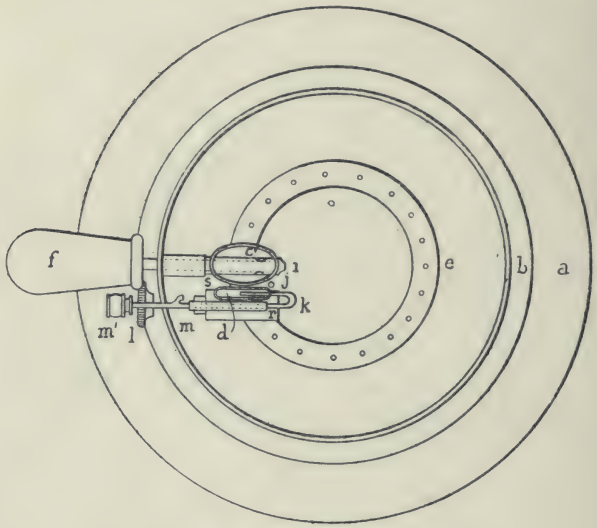


FIG. 73.—Ground plan.

vaporisation and mixing with air, consume in bringing a litre of water to boiling-point almost the same amount of methylated spirit, say, 30–35 grammes of 90 per cent. methylated spirit. However, the time

required to do this varies in different heaters, and depends on the power of the furnace. In any case, vaporisation heaters, especially those where the alcohol gas is mixed with air, are better to regulate and produce a flame easily directed under the vessel which it is desired to heat.

FIG. 74.—Twin alcohol vapour heater, for cooking, etc., purposes.

8. Amongst sundry appliances heated by alcohol, there may be enumerated—soldering and brazing blowpipes, iron heaters, plate-heaters, frizzing and curling tongs, stoves for sitting-rooms, bedrooms, greenhouses, camp stoves, etc. Fouillard's brazing and soldering blowpipe (Fig. 75) is especially useful when it is desired to get 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 blowpipes or brazing lamps is the same, they consist essentially of a Bunsen burner fed by 50 per cent. carburetted alcohol from a reservoir under a pressure from a hand-pump of 1–3 kilogrammes. The vaporisation of the alcohol is produced by a preliminary heating, then by a special piece of metal to recuperate the waste heat a nozzled tap regulates the intensities 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.

9. *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 only driven intermittently. The 4-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 effort; (4) expulsion of the burnt gases. 1. In the first forward course 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 mixture. At the end of that course then the mixture is ignited by a special arrangement so as to explode 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

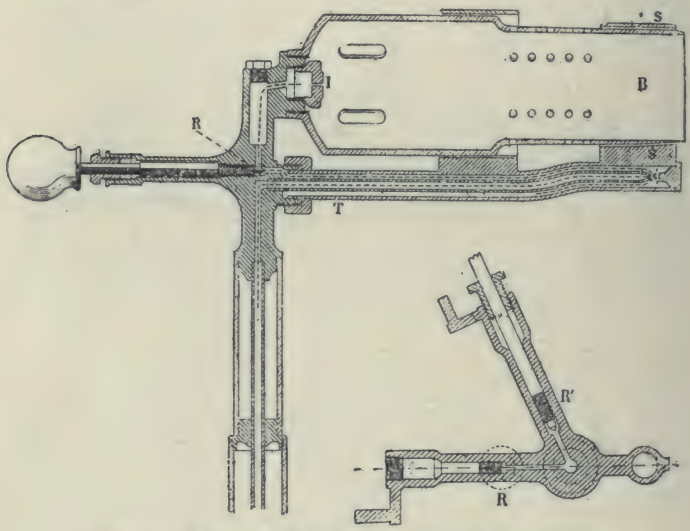


FIG. 75.—Fouillard's alcohol vapour soldering blowpipe.

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 wrought 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 4-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 carburetted, has a calorific intensity of only 6000 calories; hence it was deduced theoretically that petrol was perforce more

economical than alcohol. (But see sec. 10.) 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 were made in a petrol engine constructed by Grob of Leipsic, capable of working also with alcohol. Hartmann reported on the characteristic advantages of the alcohol: combustion was better and inodorous, but it consumed 839 grammes of alcohol against 426 grammes of petroleum per horse-hour. Later on, trials were made at

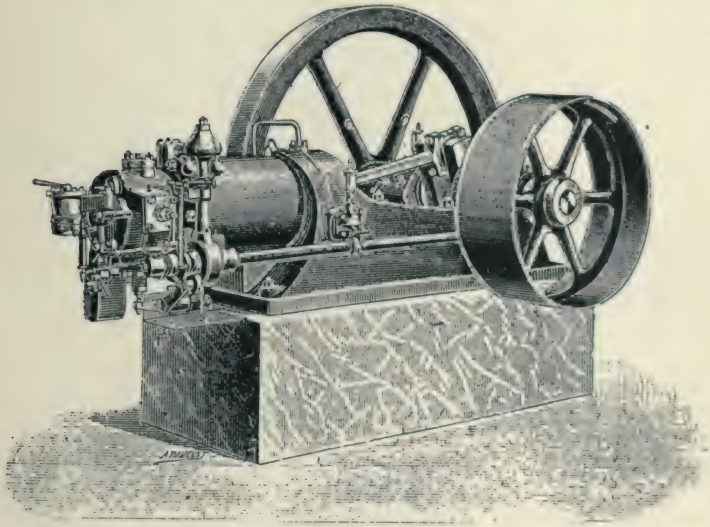


FIG. 76.—Alcohol vapour motor engine, Charon type. (There are also vertical engines of same type.)

the Berlin Fermentation Institute under encouragement and auspices of German Government. The alcohol used was 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 5 horse-power motor fitted with a Petreano evaporator, and obtained per horse-hour a consumption of 550 grammes of alcohol of 86·2 per cent. (90·4 G.L.), say, a yield of 24·6 per cent. In March 1897, Haak tested a 6 horse-power Koerting system petroleum spirit motor internally fitted up for the use of alcohol (sec. 11), and obtained a force of 9·93 horse-power with a consumption per horse-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 pulveriser 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

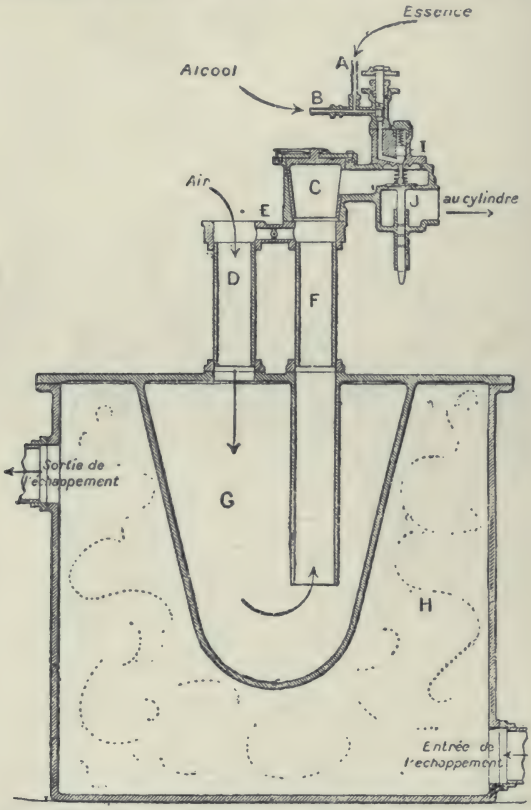


FIG. 77.—Section of feeding and escape arrangements of type of alcohol engine shown in Fig. 76. A, entrance of petrol for starting; B, entrance of alcohol; C, hot air valve; D, cold air entrance; E, temperature regulator; F, hot air entrance pipe; G, air heating chamber; H, escape gas chamber; I, liquid feed valve; J, feed and pulverisation valve.

tests. Carburation by benzol and the effect on the horse-power was first examined. The results got 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, say, 9d. the gallon, is less than that of the general German denaturant 2 per cent. of wood spirit and ½ per cent. of pyridine bases.

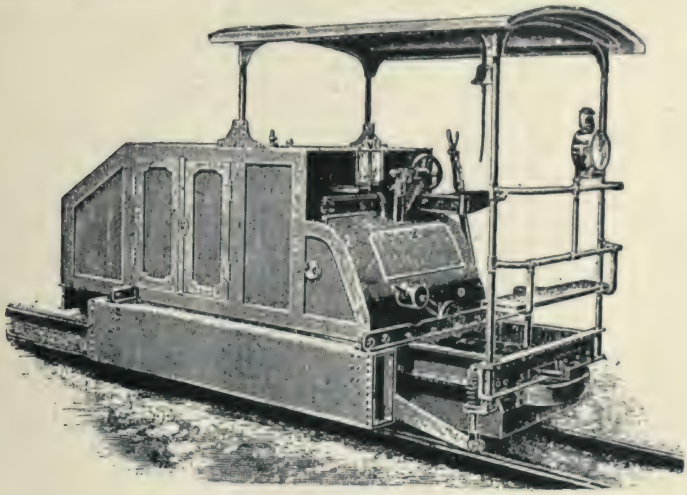


FIG. 78.—Alcohol engine in use on light railways.

TABLE XXIII. COMPARATIVE EFFICIENCY OF CARBURETTED AND PURE ALCOHOL AS MOTOR POWER GENERATORS (GOOLICH).

Benzol Per Cent. by Weight.	H.P.	Consumption Per Horse-hour.	Economy in Fuel compared with Pure Alcohol.	Benzol Per Cent. by Weight.	H.P.	Consumption Per Horse-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 above is that 20 per cent. carburation is the maximum. Meyer (Charlottenburg Polytechnic) made analogous tests on an alcohol-driven Otto locomobile 14 horse-power. His results given in Table XXIV. confirm those of Goolich.

TABLE XXIV. 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-hour.	H.P.	Consumption of Pure Alcohol Per Horse-hour.	H.P.	Consumption of Pure Alcohol Per Horse-hour.	H.P.	Consumption of Pure Alcohol Per Horse-hour.
Full .	16·14	445	19·00	422 gr.	15·90	388 gr.	16·09	375 gr.
Normal .	13·78	463	14·07	433 ,,	14·09	412 ,,	14·18	385 ,,

10. The first alcohol-driven locomobile (of Oberursel construction) of 15 horse-power was tested in February 1900, and 21·8 horse-power obtained with a consumption of 410 grammes of 88 per cent. alcohol carburetted with benzol in the proportion of 20 per cent. The above and similar tests prove that alcohol, whether pure or carburetted, can be advantageously and economically employed as a motor 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-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 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 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, say, 5·598 calories per litre, or the mechanical equivalent of heat being 427 kilogrammes, the complete combustion of 1 kilog. of that alcohol producing 6·633 calories will give $\frac{6{\cdot}633 \times 427}{3{\cdot}600 \times 75} =$ 10·49 horse-power. But in actual practice the horse-power takes 400

grammes of alcohol, say, 2·5 horse-power for 1 kilog. of alcohol, hence $\frac{25 \times 100}{10{\cdot}49} = 23{\cdot}8$ per cent. efficiency. This result is certainly very high, since the best steam engines only give an efficiency of 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 over not only a steam-driven engine, but over that driven by any other liquid fuel. It is, of course, in the comparison with steam-driven engines, a question of intermittent engines that require heating to get up steam before they can be used. There is no question of comparison with a stationary engine working continuously, against which the alcohol-driven engine does not compete. But we shall soon see that better results still have been obtained with alcohol-driven engines. But, first of all, before we go further let us glance at 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 was ascertained by the formula—

$$\frac{P = Pn\ L}{60 \times 75} = 0{\cdot}001396\ Pn\ L$$

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 Economic No. 3002 of 15–18 horse-power specially designed for alcohol had the following characteristics:—

Course, 360 m.m.
Diameter, 230 m.m.
Ratio of course to diameter, 1·56.
Speed, 200 revolutions.
Capacity of compression chamber, 2·65 litres.
Height of reservoir, 1·95 metre.
Diameter of fly-wheel, 1·9 metre.
Width of fly-wheel, 130 m.m.
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 was found. The general results are embodied in the following table:—

TABLE XXV. ACTUAL TESTS MADE BY PERISSE TO DETERMINE THE EFFICIENCY AND CAPACITY OF AN ALCOHOL-DRIVEN ENGINE, AND 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-hour.
J, consumption in grammes per horse-hour.
K, consumption per hour in kilogrammes.

11. There are two points in changing from a petrol-driven motor to be considered. There is first the carburettor and the increased compression. The full advantage is not got from the alcohol without an increased compression, but that is an exceedingly simple alteration, it merely means placing on the end of the piston or cylinder head a blank to fill up so much space. So there would be no serious difficulty in adapting the present petrol engine. Petrol cars in France 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 commonly running now. The Cremorne carburettor runs quite well with petroleum. Such efficiency is being got with agricultural motors that the actual 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 to run as many miles on a gallon of alcohol as it will on a gallon of petrol. That result has been obtained with perhaps four or five firms working on it who have not bothered probably to tackle the matter in that way which a motor-car builder would where efficiency counts for much more in his sales. A few years' practice might possibly lead to a larger alcohol mileage than petrol mileage. A larger number of calories can be got from a given cylinder capacity with alcohol than with petrol. It would be more safe 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. That is important, as every motor-car builder desires to find an engine which for a given 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 of the volatility and explosive risk of petrol, and there are such tremendous objections to the use of paraffin oil. There is not, of course, any reason why petrol should not be used in hot countries as long as care is taken. The Indian Government at the present time 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 of petrol, taken from one week's end to another in India, is apt to come out very low, because of the evaporation, that would make it distinctly a pastime for rich people in India. Therefore if from that point of view, if this country has got 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 so 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, etc., owners of agricultural alcohol engines are interested in having 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. But the conditions are somewhat different in this country to the conditions in Germany, in so far as coal-gas is so widely used here. There is no place of any reasonable size here but what has a coal-gas supply, or where coal is not easily available.

If we analyse the results of alcohol-driven engines 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, takes place. Where 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 in motors in Germany, in spite of the advantages which are given, is not very extensive yet; but the reason lies very much on the surface. The agencies for the distribution of motor alcohol are not so complete as are the distributing facilities for petrol, since petrol is distributed through all the agencies instrumental in distributing ordinary paraffin oil, or can be, since they are largely controlled by the same people. Again, it would not pay the big motor manufacturing firms to start making engines and put down plant and make templates for machines specially designed to run with alcohol, which would only be sold in Germany, say, where the alcohol is comparatively easily obtainable, and which would be useless outside the boundary of their own country. The German manufacturers probably send more cars to this country than they sell in their own. 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 a high compression that they could not be used with petrol at all. The use of alcohol must become pretty equal 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. Besides 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.

FINIS.

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Printed by MORRISON & GIBB LIMITED, *Edinburgh.*

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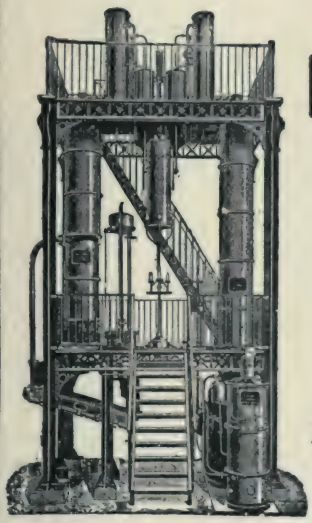

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