

MEETING OF THE LONDON SECTION, HELD AT THE CHARING CROSS HOTEL,  
STRAND, ON MONDAY, APRIL 16TH, 1928.

MR. JAMES STEWART in the Chair.

The following Paper was read and discussed :

"CHARACTER IN POT STILL WHISKY."

PART II.

By S. H. HASTIE, O.B.E., B.Sc., F.I.C., and W. D. DICK, F.I.C.

RECENTLY one of us had the privilege of reading a paper before this Section of the Institute of Brewing (this *Journ.*, 1926, 32, 209) on the subject of "Character in Pot Still Whisky," and we now propose to carry forward the discussion of this exceedingly wide subject from the point reached in that paper. Character is the term by which the palate flavour of whisky is designated, and the character of a whisky has such commercial value that it is the basis upon which whisky is appraised. In the same way whiskies are distinguished one from another by their character, and the distinctions thus arrived at are not matters of chemical composition, but are determined by taste. In the previous communication it was shown that the substances determining character were derived from the barley employed, the peat used as fuel for drying, the malt on the kiln, and the peat present in the highland water with which mashing is carried out. It was also shown that while the nature of these raw materials of the process could affect the product very considerably, at the same time the twofold operation of distillation could also exercise a very important influence upon the character of the whisky produced.

The chief substances affecting character in pot still whisky are classified as acids, esters, aldehydes, furfural, alcohols, and probably very many other classes of substances of which little is known at present. The production of these classes of substances present in all whiskies is dependent upon the nature of the raw materials used (and in this connection peat must be considered as a raw material), upon the mashing and fermentation processes, and upon the distillation of the fully fermented liquids in the wash still.

The re-distillation of the distillate from the wash still is the process during which

the potable whisky is fractionated from the total distillate, and the non-potable fractions are collected in separate receivers. During this second distillation interaction between the substances composing the liquid in the still proceeds continuously and these interactions largely determine the character of the potable fraction.

In the wash still, the distillation of the fermented wash is carried on until the distillate no longer contains alcohol, but in the spirit still, although the distillation is again carried to the same point, three fractions are separately collected, the *foreshot*, containing undesirable secondary constituents, the *potable whisky*, and the *feints* also containing undesirable secondary constituents.

Now the decision as to when these fractions should be collected depends largely, in fact almost entirely, upon the skill and experience of the operator, who determines the point of change by two methods. He decides to discontinue the collection of "foreshot" and to commence collecting potable whisky when the distillate no longer turns blue and clouded when water is added in time-honoured fashion. He later decides to cease collecting potable spirit and to proceed to collect the third and final fraction by observing the alcoholic strength of the distillate, experience telling him at which particular strength to affect the change over for the particular plant with which he is concerned. It will be obvious that the decision as to the duration of these three fractions is vital to the whole process, and it is surprising that so little is known as to what changes take place in the still, or as to why certain strengths of distillate are selected, as points at which to cease collecting the potable fraction in different distilleries. It was with the ultimate object of trying to understand something of the mechanics, physics and chemistry of the two distillations that the work of which

this papers forms a small part, was originally undertaken. As the investigation proceeds the difficulties appear to grow and to multiply. The pot still is simple—from a copper-smith's point of view; it is crude—from an engineer's point of view; and from a chemist's or a physicist's point of view it is as involved and complicated and irregular in operation as it very well can be.

In previous communications, efforts have been made to deal with the origin and distillation of acids in the two stills, the production of esters and their interaction with alcohol and acids, and about a year ago one of us attempted to discuss the presence of furfural in the process. In this paper we have made an attempt to carry forward this investigation of furfural in pot still whisky, because furfural must be regarded as one of the important substances in the pot still product. Fortunately, furfural is one of the character-giving substances which can be dealt with as an individual substance. It is not necessary to go into the question of the chemical nature of furfural, or of the methods by which it can be produced, as these are common knowledge, and the following notes are concerned with the appearance of furfural in the pot still process, the factors governing its production and the behaviour of the substance during the two distillations.

As a starting-off point we will take the conclusions of a previous communication on the subject, by one of us, that when the fully fermented wash is transferred to the wash still for the first distillation, it contains no furfural, and that furfural is produced during this distillation. The distillate from this first distillation is an alcoholic liquid containing about 20 per cent. of alcohol by volume and 2.0 parts of furfural per 100,000 parts of the distillate.

This amount of furfural has been produced in the wash still during the first distillation and the manner of its production is the first question to be studied, and if this investigation is to have practical value it is necessary to find answers to the following two questions:—

(1) Is the furfural content of the distillate from the wash still influenced by the actual operation of the still during distillation, or by the composition of the wash, or by both of these factors?

(2) What are the factors controlling the production of furfural in the wash still?

To decide whether the operation of the wash still influences the production of furfural and controls the amount passing to the distillate, it is necessary to determine the manner in which furfural distills from fermented wash. This is an important point because the wash still in practice is operated on a basis of the alcoholic strength of the distillate, the operation being stopped when the distillate no longer contains alcohol. A quantity of fully fermented wash prepared from a wort of sp. gr. 1025°, and to which, after fermentation was complete, 0.003 gm. of furfural had been added, was distilled in an electrically-heated still, the heating of which could be readily controlled, and the furfural estimated in successive fractions of distillate. Each fraction was made to an alcoholic strength of 50 per cent. by volume before testing, as the furfural estimation cannot be satisfactorily carried out at any other strength.

The results obtained are given below.

*Distillation of fermented wash containing added furfural.*

200 cc. wash containing 5 per cent. of alcohol by volume and with 0.003 gm. furfural added before distillation. The liquid was distilled to the last drop. Still A.

Fraction.	Temp. of Distillate. °F.	Alcoholic Strength of Fraction % by Volume.	Mgm.: Furfural in Fraction.
1	199	40.9	0.88
2	208	57.4	0.76
3	210	1.6	0.72
4	210	0.0	0.42
5	211	0.7	0.35
6	212	0.2	0.14
7	212	0.2	0.13
8	212	0.2	0.08
9	212	0.0	0.07
10	212	0.0	0.05
11	212	0.0	0.04
12	212	0.0	0.02
13	212	0.0	0.02
14	212	0.0	0.01
15	212	0.0	0.03
16	212	0.0	0.03
17	212	0.0	Nil.
18	212	0.0	"
19	212	0.0	0.08
20	212	0.0	0.30

Total furfural distilled=0.004 gm.

From these results it may be inferred that:

(1) Furfural is transferred to the distillate at the commencement of distillation when the distillate has an alcoholic strength of 57.4 per cent = 0.7 o.p.

(2) Furfural is transferred to the distillate for the most part at a temperature of about 204° F.

The wash employed in the above test contained 5 per cent. of alcohol by volume, and the furfural practically all distilled at the beginning of the operation when the alcoholic strength of the distillate was at the maximum. It will be shown later that, during the distillation of spirit the furfural distills when the alcoholic strength of the distillate is about 16 o.p., i.e., 66 per cent. of alcohol by volume. Possibly, therefore, the furfural in the case of wash would distill at the same strength if the wash were sufficiently concentrated to give such a strength of distillate.

As a matter of practical interest there is no point in preparing a wash of greater concentration than 12 per cent. of alcohol by volume, as washes of greater strength do not occur in the distillery, and therefore if a distillation is carried out at this strength, the resulting graph will represent what occurs in practice. Accordingly a distillation of wash with a concentration of alcohol of 12 per cent. was carried out with added furfural. It must also be remembered that the temperature of maximum transfer of furfural may be significant.

*Distillation of fermented wash containing added furfural.*

200 cc. of wash containing 12 per cent. of alcohol by volume. Furfural added—0.003 grm. The liquid was distilled to the last drop. Distilled in still Type A.

Fraction.	Temp. of Distillate, °F.	Alcoholic Strength of Fraction % by Volume.	Mgm. Furfural in Fraction.
1	192.0	65.5	0.71
2	201.0	52.8	0.94
3	204.0	43.1	0.94
4	207.0	32.9	0.84
5	209.0	20.1	0.65
6	210.0	10.7	0.37
7	211.0	9.0	0.20
8	211.0	6.0	0.13
9	212.0	10.2	0.07
10	212.0	3.5	0.04
11	212.0	5.4	0.04
12	212.0	5.5	Nil.
13	212.0	7.9	"
14	212.0	6.8	"
15	212.0	3.1	"
16	212.0	7.9	"
17	212.0	8.6	"
18	212.0	8.6	"
19	212.0	8.6	"
20	212.0	8.6	"

From these results it would appear that:—

(1) The maximum furfural transfer to distillate occurs at strengths of between 7.5 to 24.2 u.p.—52.8 to 43.1 per cent. by vol.

(2) The maximum furfural transfer to the distillate occurs at a temperature of from 201° F. to 204° F.

These two distillations indicate that whatever furfural is added to wash, distills over chiefly around proof strength and at a temperature of 204° F., that is, long before the still ceases to deliver alcohol. In practice however, the furfural in the wash is not present when the wash is transferred to the still, but is produced in the still during distillation, and the graph of distillation of the furfural thus produced will most probably differ from the graph obtained from the wash to which furfural is artificially added. The velocity of transfer of the furfural to the distillate under conditions similar to those of practice will most probably be dependent upon the velocity of furfural production, which in turn will probably be controlled or at least influenced by the amount of acid and by the amount of available pentoses present in the wash, when heating commences.

To digress for a moment, it is interesting to note that in the first distillation of wash carried out above, there is marked production of furfural in the last two fractions after all the added furfural had apparently passed to the distillate.

This additional furfural is explained by the fact that at the end of the distillation slight charring of the residue in the still accidentally occurred. That such charring will produce furfural is to be expected, furfural being known to be present in wort prepared from caramelised malt and from highly dried malts (Brand, this *Journ.* 1898, 4, 562). Furfural was also recognised by Lintner (*ibid.*, 1910, 16, 688), as a normal constituent of roasted malt. A practical point in connection with furfural seems to arise from these facts, and it is that insufficient use of the device fitted for continuously scraping the bottom of the wash still inside will most certainly be reflected in a higher percentage of furfural in the still and in the distillate.

To return to the main discussion, it appeared desirable to investigate the velocity of the production of furfural in the wash still. We have shown how furfural artificially added to wash, distills with the spirit from the wash, and it is not without interest to ascertain how the curve of furfural transfer to the

distillate is affected by the velocity of production of furfural in the wash still. To obtain this information a distillation of wash was carried out to which acid had been added, but to which no artificial addition of furfural had been made, and the following results were obtained:—

*Distillation of fermented wash with added acid.*

Concentration of alcohol in wash = 18.5 per cent. by volume. Acid added = 0.5 cc. concentrated hydrochloric acid. Distilled in Still Type A.

Fraction.	Temp. of Distillation °F	Alcoholic Strength of Fraction % by Volume.	Mgms. Furfural in Fraction.
1	190	78.8	Nil.
2	199	60.2	"
3	203	43.0	"
4	206	30.0	"
5	208	19.0	"
6	209	13.2	"
7	209	8.8	"
8	209.5	10.4	"
9	210	7.6	"
10	210	10.1	"
11	210	6.6	"
12	210	7.3	"
13	210	4.5	"
14	210	8.5	"
15	210	4.5	"
16	210	10.8	"
17	210	10.3	0.03
18	210	10.8	0.08
19	210	8.8	0.09
20	210	11.3	0.23

As the production of furfural was exceedingly small in the above experiment, the test was repeated, with the addition to the wash of a quantity of arabinose and xylose, in the hope that sufficient furfural would be produced to give a definite curve of furfural distillation showing the effect of the delay, if any, resulting from the fact that the furfural must be produced before it can be distilled. As arabinose and xylose are pentoses present in wash at the commencement of distillation the greater quantity now present in the wash should not, we believe, greatly accelerate the production of furfural and so give a misleading curve of furfural transfer. The results obtained in this way are given below.

*Distillation of fermented wash with acid and arabinose and xylose added before distillation.*

Concentration of alcohol in wash = 17.3 per cent. by volume. Acid added = 0.5 concentrated hydrochloric acid. Arabinose

added = 0.3 gram. Xylose added = 0.3 gram. Distilled in still Type A.

Fraction.	Temp. of Distillation °F	Alcoholic Strength of Fraction % by Volume.	Mgms. Furfural in Fraction.
1	192.5	80.0	Nil.
2	202.0	55.2	"
3	205.0	36.8	"
4	209.0	25.5	"
5	210.0	15.5	"
6	211.0	11.7	"
7	211.5	8.7	0.04
8	212.0	21.1	0.04
9	212.0	3.4	0.05
10	212.0	10.4	0.05
11	212.0	6.3	0.06
12	212.0	6.0	0.03
13	212.0	9.0	0.07
14	212.0	8.0	0.08
15	212.0	4.8	0.10
16	212.0	11.5	0.12
17	212.0	6.2	0.22
18	212.0	12.0	0.27
19	212.0	4.1	0.36
20	212.0	10.3	0.54

From these results it may be accepted that the velocity of production of furfural in the wash still is a most important factor, influencing the content of furfural in the distillate, no actual furfural appearing in the distillate until the operation was well advanced, and a considerable portion of the still contents had been distilled. Furfural is produced after a definite time of boiling in the still, and this time factor is of first importance.

Before proceeding to ascertain the effect of control of still, and shape of still head and lyne arm on the distillation curve of furfural, we must deal with a difficulty of technique. It would be a matter of extreme difficulty to duplicate a given fermented wash for the series of comparative experiments required to investigate the effect of still head, etc., and to overcome this the use of a "synthetic" fermented wash was decided upon. An alcoholic solution, containing 12.0 per cent. of alcohol by volume, with the pentoses, xylose and arabinose in reasonable amount, was considered a satisfactory standard "wash" to employ, as the other substances present in the distillery wash, that is to say exhausted yeast, acids, higher alcohols, aldehydes, esters, etc., do not so far as we can see influence the production of furfural in the wash still. Our standard "fermented wash" employed in the experiments on the wash still which follow, has the following composition, which was arrived at on the basis of providing a

sufficiency of pentoses for the reaction:—Alcohol, 12.0 per cent. by vol.; arabinose, 0.3 grm. per 100 cc.; xylose, 0.3 grm. per 100 cc.

A distillation of this artificial wash was carried out to ascertain if it behaved, so far as furfural is concerned, in a manner comparable to the wash of the distillery.

*Distillation of standard artificial fermented wash plus added acid.*

Concentration of alcohol in wash = 19.2 per cent. by volume; 200 cc. of artificial wash plus 0.5 c.c. concentrated hydrochloric acid. Distilled in still Type A.

Fraction.	Temp. of Distillation. °F.	Alcoholic Strength of Fraction % by Volume.	Mgms. Furfural in Fraction.
1	195.0	71.3	Nil.
2	198.0	69.8	"
3	202.0	54.5	"
4	206.0	37.5	"
5	208.0	25.5	"
6	211.0	17.9	"
7	212.0	10.9	"
8	212.0	7.9	"
9	212.0	2.5	"
10	212.0	7.6	0.03
11	212.0	7.1	0.04
12	212.0	9.7	0.05
13	212.0	9.0	0.07
14	212.0	9.3	0.08
15	212.0	6.9	0.10
16	212.0	9.0	0.10
17	212.0	5.3	0.18
18	212.0	7.7	0.36
19	212.0	8.8	5.63
20	212.0	6.4	3.75

From these results it is evident that such an "artificial" fermented wash as we have utilised behaves sufficiently like actual fermented wort so far as furfural production is concerned for our purpose, and it fulfils the necessary conditions of yielding comparable results, as it is of constant known composition. If we summarise the position at this point we have prepared an artificial wash capable of giving comparable results from different distillation experiments, and we have answered part of our first question in that we have decided that the operation of the wash still influences the furfural content of the distillate, because time appears to be a factor during distillation, as furfural appears in the distillate at the end of the distillation of each charge of the still when a wash containing no furfural (as in practice) but containing pentoses is distilled with acid. Using the artificial fermented wash employed by us for these tests and hydrochloric acid in the still, we obtain furfural at the end of the distillation in very

small amount when compared with theory.

The acids present in a distillery fermented wash are lactic and acetic, chiefly, and therefore we distilled further quantities of our artificial wash with these acids to find out whether furfural was actually produced during distillation under conditions similar to those obtained in the distillery.

The results obtained were as follows:—

*Distillation of artificial fermented wash plus added lactic acid.*

Concentration of alcohol in the wash equals 18.3 per cent. by volume; 200 cc. of wash plus 0.5 cc. of lactic acid.

Fraction.	Temp. of Distillation. °F.	Alcoholic Strength of Fraction % by Volume.	Mgms. Furfural in Fraction.
1	194.5	61.0	Nil.
2	197.0	61.8	"
3	201.0	50.1	"
4	204.0	41.1	"
5	207.0	26.0	"
6	210.0	17.6	"
7	210.5	10.7	"
8	210.5	5.8	"
9	211.0	5.5	"
10	211.0	6.0	"
11	211.0	7.3	"
12	211.5	5.8	"
13	212.0	6.8	"
14	212.0	3.2	"
15	212.0	10.8	"
16	212.0	10.0	"
17	212.0	9.0	"
18	212.0	12.3	0.04
19	212.0	8.8	0.07
20	212.0	7.7	0.05

*Distillation of artificial fermented wash plus added acetic acid.*

Concentration of alcohol in the wash equals 18.3 per cent. by volume; 200 cc. of wash plus 0.5 cc. of concentrated acetic acid.

Fraction.	Temp. of Distillation. °F.	Alcoholic Strength of Fraction % by Volume.	Mgms. Furfural in Fraction.
1	193.0	65.9	Nil.
2	196.0	62.4	"
3	199.0	54.5	"
4	202.0	40.9	"
5	206.0	27.1	"
6	207.5	15.5	"
7	208.0	11.8	"
8	209.0	6.8	"
9	209.0	8.7	"
10	209.0	0.0	"
11	209.0	7.9	"
12	209.0	7.3	"
13	209.0	7.2	"
14	209.5	9.8	"
15	210.0	10.5	"
16	211.0	10.0	"
17	212.0	6.7	"
18	212.0	8.8	"
19	212.0	11.1	0.09
20	212.0	8.2	0.04

The inferences to be drawn from these distillations are, that if acids actually met with in practice are employed, there is a very limited production of furfural in the wash still and such furfural as is produced appears in the last few fractions only.

Time would appear to be a factor. The total amount of furfural produced is, however, so small, and falls so far short of the theoretical amount to be expected from the amounts of acid and pentoses present that limiting factors other than time must be sought for. The probability is that the amount and concentration of the acids in the still is an important factor, together with the available pentose present as the raw material of the furfural production. To examine the effect of the total amount and concentration of the acid present in the still, 100 cc. portions of artificial wash were heated with varying amounts of hydrochloric acid under reflux condensers for two hours followed by distillation in the same still as before, Still, Type A.

The results were as follows:—

*Distillation of artificial wash with varying amounts of hydrochloric acid.*

100 cc. of wash used in each case. Two hours under reflux condenser.

HCl. added.	Mgms.: Furfural per 100 cc. wash.
0.5 cc. N/10	0.12
1.0 cc. "	0.16
10.0 cc. "	0.80
5.0 cc. Concentrated	50.00

The important point in this test is that furfural is produced in large quantities when the acid employed is concentrated, but that dilute acid produces little or no furfural. Further the furfural produced in presence of dilute acid is all produced towards the end of distillation. If time is the governing factor then the fact that the furfural is produced at the end of the distillation would be attributed merely to the time necessary for the reaction, but as will be shown below time is only a factor when the artificial wash is distilled with acid and not when it is merely heated under a reflux condenser. This is significant and indicates that it is a question not of length of time of boiling nor of amount of acid, but of actual concentration of acid.

The results concerned are given below:—

*Effect of reflux compared with distilling.*  
100 cc. artificial wash plus 10 cc. N/10 HCl.

Time boiled under reflux.	Furfural after boiling under reflux.	Furfural after distilling subsequent to boiling under reflux.
2 Hours	Faint trace	0.5 mgrm.
4 "	" "	0.4 "
6 "	" "	0.75 "
8 "	Trace	0.6 "

Evidently mere boiling of the wash with acid does not produce furfural if the acid concentration be low, but on distillation of the liquid which has been boiled for several hours under a reflux condenser furfural was obtained towards the end of distillation. It appears to be clear that using hydrochloric the furfural does not appear until the acid is highly concentrated as a result of the distillation of the bulk of the still charge, which of course is the case when an acid such as HCl is employed. With acetic, however, this concentration does not occur, as much of the acid passes to the distillate. In practice lactic and acetic acids are the acids we are concerned with, and the amounts of such acids found in wash are very small, so much so, that it is doubtful whether the maximum concentration possible in the case of complete distillation of the charge of the wash still, would concentrate the acid sufficiently to produce furfural. But distillation is never carried beyond the point at which all the alcohol is distilled, the residue at this point being of such bulk that the dilution of the acids present is very considerable, and certainly much greater than that with which furfural is produced. If then furfural is only produced from the pentoses present in wash when the acid is at a relatively high concentration and is not produced by boiling with dilute acids, then we must conclude that furfural is in no case produced in the pot still by the action of acids on pentoses, as the conditions necessary for its production never obtain in the wash still.

How then is the small amount of furfural produced which is undoubtedly found in all fire heated wash stills in pot still distilleries? We are forced to a conclusion which is very much in the nature of an anti-climax. We are driven to one explanation of the presence of furfural in pot still whisky, and it is, that the heating of the wash still by fire in direct contact with the still itself, and in spite of

the action of the stirring gear, causes a certain amount of local superheating of the wash, a partial charring in fact, and that this charring produces furfural. Furfural is certainly very readily produced as we have shown early in this paper by charring of fermented wash containing pentoses, or in fact by the charring of pentoses alone. It is also clear from a series of tests which we carried through that the acid is present after heating under a reflux condenser and subsequently distilling, in quantity almost equal to that added, and as excess of pentoses has been arranged in all our tests, it follows that the acid is not rendered inactive by combination with any of the other substances present in the fermented wash of practice. We were however reluctant to accept the charring explanation of the origin of furfural without exhaustive elimination of all possible factors, and we decided to deal with the following possibilities before finally accepting what appeared to be the facts. In the first place we decided that in addition to the evidence of other workers as to the production of furfural from roasted malt we would attempt to obtain further evidence of this indication of the effect of charring of pentoses. We prepared malt extract by mashing 50 grms. of pale malt with water, heating under a reflux for 4 hours, and then distilling, alcohol being added before heating under the reflux condenser. We obtained no furfural. When, however, we repeated this experiment after adding 5 cc. N/10 HCl, we obtained furfural in the distillate. When amber malt was used in the same type of test, furfural was obtained in the distillate whether acid was added or not. Roasted malt gave similar results. These results indicate that charring produces furfural independent of the presence of acid, but that pale dried malt yields furfural only in presence of strong acid—N/10 HCl concentrated by distillation. Xylose similarly yielded furfural in the distillate when acid was added, concentration again occurring towards the end of distillation, the point at which the furfural appeared in the distillate. The liquid was heated under a reflux and subjected generally to the same conditions as in the case of the malt extract solutions. When xylose was treated with acid as above, heated under a reflux and distilled in two portions, one over a naked flame, the other over glycerol, the former gave much furfural, the latter a mere trace.

This trace was due apparently to concentration of the acid on distillation, the large quantity of furfural in the former being the result we believe of charring.

Roasted and crystal malts gave furfural, whether distilled over a naked flame or over glycerol, but more so over the naked flame, indicating that roasted and crystal malts (like amber malt) contain furfural, while the distillation over a naked flame increases the yield considerably.

Those results confirm our conclusion that the furfural of practice is the result of partial charring of the pentoses in the wash during the first distillation.

Having treated, we fear, somewhat superficially, the questions of the production of furfural in the wash still, and the subsequent distillation of the furfural from the still, the next step is to follow the practical process and attempt to show what happens to the furfural which is at this stage contained in the distillate from the wash still. No further production of furfural takes place in the pot still process, but the question of the distribution of the furfural already produced is of first importance. At the commencement of the second distillation in the distillery, that is, distillation of the distillate from the wash, there are three liquids to be dealt with.

(1) The low wines, the actual distillate from the wash still, is an alcoholic liquid containing about 20 per cent. of alcohol by volume, together with the usual secondary products of the pot still, and is already more or less stamped with the distinguishing characteristics of the distillery in which it has been produced. With special reference to our subject, the low wines contain all but a small proportion of the total furfural which has been produced, a very small amount being present in the pot ale, that is, in the residue remaining in the wash still after distillation is complete, and this furfural is rejected from the process with the pot ale. The low wines are collected in the low wines receiver.

(2) The foreshot and feints mixed in the feints receiver are the two fractions from the second distillation representing the first and the last runs of the second or spirit still. These are regarded as non-potable and are returned to the process for re-distillation.

The charge of the spirit still consists of the low wines from the wash still mixed with



the feints and foreshot from the previous operations of the spirit still.

To give a general idea of the nature of these liquids partial analyses giving the alcohol and furfural contained in them are given below.

and runs the remainder of the alcoholic distillate into the feints receiver. It becomes therefore, a matter of great interest and considerable importance to know what the effect of this judgment of strength of distillate is upon the distribution of the second-

	Low Wines.	Foreshot.	Feints.	Spirit Still Charge Low W + Foresh. + Feint.
Alcohol per cent. by volume ... ..	20.0	70.0	33.0	26.0
Furfural (parts per 100,000 of the liquid) ...	1.0—2.0	2.0	3.0	2.1

The objects of the second distillation are two. In the first place the spirit is concentrated from a strength of about 26 per cent. of alcohol by volume to about 63 per cent., the strength at which whisky is normally filled into casks for maturing purposes, and secondly, the fractions considered non-potable, the foreshot and the feints are separated from the whisky and returned to the process. These fractions are not selected in relation to their furfural content, nor in fact in relation to any scientific principle, so far as we know at present, but their selection is the result of a system of trial and error which has been going on for generations.

In the absence of any scientific system, such a method is inevitable, and it has produced in the past and is now producing the required results, and we do not wish to appear cynical concerning such methods, but we do wish to understand better what we are doing and so place ourselves in such a position that we may be able to make alterations with some knowledge to guide them, and to be better able to deal with the situation when things go wrong and "trial and error" fails. At the moment then the decision as to when to collect the three fractions depends upon the experience of the operative distiller, and the proportion of furfural which passes to any particular fraction is at present governed by fortuitous circumstances.

The distiller ceases to collect foreshot and commences to collect potable whisky at a definite strength of distillate which he believes from previous experience will give him the result he desires, and similarly by observing the alcoholic strength of the distillate, he ceases to collect potable spirits,

any products especially furfural over the fractions.

The questions of importance to the practical distiller in so far as they will, if answered adequately give him some idea of what he is doing in controlling the spirit still, are the following:—

(1) Does furfural distill during the second distillation in a definite relationship to the alcoholic content of the distillate, to the temperature of distillation, to both, or to neither?

(2) In general, is furfural collected chiefly in foreshot, potable whisky, or feints?

(3) Is the distribution of furfural over the fractions influenced by the shape of the still head and lyne arm?

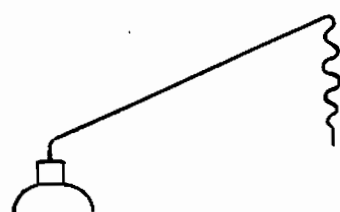
Before dealing with those three questions, it is necessary to understand how furfural behaves when distilled from alcoholic solutions under conditions similar to those in the distillery. A series of experiments were carried out in which whisky and pure alcohol containing furfural were distilled and successive fractions examined for furfural content, three widely differing lyne arm and still head combinations being employed. The factor of irregular heating of the still was eliminated in these tests by the use of the same electrically heated still, the heating throughout being controlled by a rheostat.

*Distillation of pure alcohol of 50 per cent. strength to which furfural has been added in known amount.*

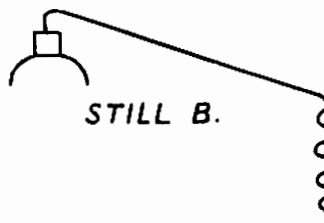
Three types of still were used, designated A, B and C, of which sketches are given, from which it will be noted that type B is the least rectifying type, C highly rectifying, and type A intermediate in form between the other two.



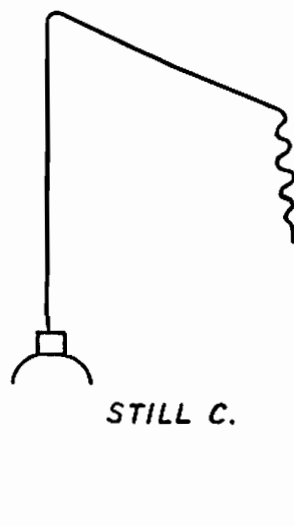
## STILLS EMPLOYED.



STILL A.



STILL B.



STILL C.

The results obtained by distilling 50 per cent. pure alcohol with furfural are given below.

Strength of alcohol = 52.8 per cent. by vol.

200 cc. pure alcohol with 0.007 gm. furfural added and using Still, Type A.

Fraction.	Temp. of Distillation, °F.	Alcoholic Strength by Volume.	Mgms. Furfural in Fraction.
1	179.0	85.6	0.10
2	180.0	88.1	0.12
3	180.5	88.6	0.16
4	181.0	87.3	0.17
5	181.5	86.0	0.21
6	182.0	86.1	0.21
7	183.0	84.3	0.22
8	183.5	79.8	0.29
9	184.5	77.5	0.33
10	186.5	78.1	0.50
11	189.5	74.1	0.75
12	194.0	66.3	0.95
13	201.0	51.9	1.15
14	207.5	16.7	0.95
15	212.0	9.5	0.55
16	212.0	2.3	0.25
17	212.0	1.8	Nil
18	212.0	2.6	"
19	212.0	4.3	"
20	212.0	2.0	"

Strength of alcohol = 54.1 per cent. by volume; 200 cc. pure alcohol with 0.007 gm. furfural added and using Still B.

Fraction.	Temperature °F.	Strength of Fraction % Alcohol by Volume.	Total Mgms. Furfural per Fraction.
1	178.0	81.8	0.14
2	179.5	85.5	0.15
3	180.0	87.6	0.17
4	180.5	87.6	0.20
5	180.5	85.8	0.23
6	181.5	85.6	0.25

Fraction.	Temperature °F.	Strength of Fraction % Alcohol by Volume.	Total Mgms. Furfural per Fraction.
7	182.0	83.3	0.29
8	183.0	79.3	0.33
9	184.5	80.0	0.40
10	186.5	73.3	0.47
11	190.0	68.8	0.71
12	194.5	68.6	0.94
13	199.5	48.4	0.94
14	205.0	32.9	1.00
15	209.0	16.6	0.60
16	211.0	5.8	0.25
17	212.0	5.6	0.07
18	212.0	0.0	Nil.
19	212.0	2.3	"
20	212.0	3.1	"

200 cc. alcohol 61.5 per cent. by volume with 0.007 gm., furfural added, and using Still C.

Fraction.	Temperature °F.	Strength of Fraction % Alcohol by Volume.	Total Mgms. Furfural per Fraction.
1	180.0	93.3	Nil.
2	180.0	96.2	0.08
3	180.0	97.6	Nil.
4	180.0	96.2	0.07
5	180.5	96.4	0.14
6	181.5	95.9	0.17
7	182.0	96.4	0.17
8	183.5	91.9	0.29
9	185.0	95.0	0.42
10	188.0	88.1	0.58
11	192.0	83.3	1.15
12	198.0	67.9	1.50
13	205.0	46.3	1.67
14	210.0	17.0	0.60
15	212.0	13.6	0.27
16	212.0	6.6	0.05
17	212.0	12.4	Nil.
18	212.0	10.5	"
19	212.0	13.8	"
20	212.0	11.4	"

These results show that when furfural is distilled with 50 per cent. alcohol, the furfural distillation has the following characteristics.

(1) Maximum furfural transfer to distillate takes place around 24 u.p. = 43.0 per cent. of alcohol by volume, and the larger proportion of the furfural is transferred to the distillate between the strengths of 18 o.p. and 44 u.p. = 67 per cent. and 32 per cent. by volume of alcohol.

(2) The transfer of furfural is confined to a smaller number of fractions, and consequently takes place over a shorter range of temperature rise when the head is a highly rectifying one.

(3) The maximum transfer of furfural under the conditions of the experiment takes place at a temperature of 204° F. and the larger proportion transfers between 195° F. and 205° F.

In the following experiments whisky was used with added furfural in place of pure alcohol.

*Distillation of whisky of 50 per cent. alcoholic strength by volume to which furfural has been added in known amount.*

200 cc. of grain spirit at a strength of 54.0 per cent. by volume with .003 gm. furfural added, using Still A.

Fraction.	Temperature °F	Strength of Fraction % Alcohol by Volume.	Total Mgm.; Furfural per Fraction.
1	178.0	82.4	0.07
2	178.5	85.5	0.08
3	179.0	89.1	0.05
4	180.0	87.8	0.12
5	180.5	87.6	0.14
6	181.0	86.8	0.21
7	181.5	84.3	0.22
8	183.0	83.1	0.24
9	184.5	79.5	0.32
10	187.0	77.1	0.40
11	191.5	69.5	0.52
12	196.0	63.3	0.58
13	201.0	47.6	0.63
14	207.5	27.8	0.60
15	211.5	10.3	0.33
16	212.0	1.0	0.12
17	212.0	3.6	0.05
18	212.0	2.3	Nil.
19	212.0	5.3	"
20	212.0	1.0	"

200 cc. of grain spirit at a strength of 57.8 per cent. by volume with .003 gm. furfural added, using Still B.

Fraction.	Temperature °F	Strength of Fraction % Alcohol by Volume.	Total Mgm.; Furfural per Fraction.
1	179.0	84.8	0.05
2	180.0	93.6	Nil.
3	180.5	92.4	"
4	181.0	91.8	0.09
5	181.5	86.3	0.12
6	182.0	90.3	0.12
7	182.5	89.6	0.14
8	184.0	86.3	0.17
9	185.0	83.6	0.22
10	188.0	78.6	0.24
11	192.0	72.6	0.49
12	197.0	64.6	0.63
13	202.0	49.8	0.60
14	207.0	27.5	0.44
15	210.0	11.8	0.30
16	212.0	10.3	0.05
17	212.0	11.4	Nil.
18	212.0	10.5	"
19	212.0	9.6	"
20	212.0	10.8	"

200 cc. of grain spirit at a strength of 54.8 per cent. by volume with .003 gm. furfural added, using Still C.

Fraction.	Temperature °F	Strength of Fraction % Alcohol by Volume.	Total Mgm.; Furfural per Fraction.
1	180.0	89.3	Nil.
2	181.5	91.3	0.04
3	182.5	92.9	0.07
4	183.0	90.8	0.10
5	184.0	91.3	0.12
6	185.5	90.9	0.19
7	186.0	96.2	0.27
8	187.5	89.6	0.34
9	190.0	86.6	0.50
10	194.0	85.3	0.68
11	198.0	74.3	0.88
12	204.5	53.7	1.00
13	210.0	19.8	0.75
14	212.0	10.6	0.30
15	212.0	6.2	0.08
16	212.0	9.1	Nil.
17	212.0	4.8	"
18	212.0	Nil.	"
19	212.0	6.8	"
20	212.0	7.9	"

These results show that when furfural is distilled with whisky of about 50 per cent. strength by volume the furfural distillation has the following characteristics:—

(a) Maximum furfural transfer to distillate takes place around 12 u.p. and between the strengths of 18 o.p. and 43 u.p. = 67 per cent. by volume and 32 per cent. by volume.

(b) The transfer of furfural is confined to a smaller number of fractions, and consequently takes place over a shorter range of temperature rise when the head is a highly rectifying one.

(c) The maximum transfer of furfural takes place at a temperature of 204° F. and the larger proportion transfers between 197° F. and 206° F.

Obviously pure alcohol with added furfural behaves similarly to whisky with added furfural when the same alcoholic strength is employed and furfural is added.

The following experiment shows that if whisky be distilled without added furfural, the furfural already in the whisky distills in the same general way as in the distillation carried out above.

200 cc. of whisky at a strength of 49.0 per cent. by volume, using Still A.

Fraction.	Temperature °F.	Strength of Fraction % Alcohol by Volume.	Total Mgrm.; Furfural per Fraction.
1	180.0	76.5	Nil.
2	180.5	80.0	"
3	181.5	79.0	"
4	182.0	78.8	"
5	182.5	78.0	"
6	183.0	75.0	Traces.
7	183.5	79.0	0.0345
8	184.5	78.0	0.0415
9	186.0	78.0	0.0505
10	189.0	68.3	0.0625
11	192.5	66.4	0.0895
12	198.5	59.0	0.1100
13	204.5	38.3	0.1070
14	210.5	16.8	0.0860
15	212.5	4.8	0.0550
16	212.0	4.1	Nil.
17	212.0	2.3	"
18	212.0	3.3	"
19	212.0	1.0	"
20	212.0	7.4	"

(a) Maximum furfural transfer to distillate takes place around 33 u.p. and the greater proportion of the furfural is transferred between the strengths of 16.5 o.p. and 70.7 u.p.

(b) The maximum transfer of furfural under the conditions of the experiment takes place at a temperature of 204° F. and the larger proportion between 193° F. and 210° F.

The general impressions from these experiments are:—

(1) That furfural distills from 50 per cent. alcoholic solution whether it is pure alcohol or whisky in such a manner as to yield a curve with a very pronounced peak; in other words, the transfer of furfural to the distillate takes place over a very limited

range of temperatures of distillation and alcoholic strengths of distillate.

With a 50 per cent. alcoholic solution this temperature is 204° F. and the strength from 16 o.p. to 50 u.p.

(2) The shape of the still head and lyne arm affects the transfer of furfural to a limited extent only, the more rectifying the head and lyne arm combination the more restricted is the range of temperatures and strengths of distillate over which the major portion of the furfural is transferred.

Of the above conclusions the most important is the fact that the temperature of maximum transfer is common to all distillations of 50 per cent. alcoholic strength, but there is more variation when the strength of distillate is considered. If we remember that the still is controlled in practice on a basis of alcoholic strengths then we see that so far as furfural is concerned the strength of the distillate is a somewhat loose control, whereas the temperature of distillation might be a very definite control. As, however, the alcoholic content of the charge of the spirit still is in practice 20 per cent. by volume, and not 50 per cent. an experiment on exactly similar lines to the foregoing, but employing spirit of 20 per cent. by volume, was carried out, as was also an experiment utilising low wines from the distillery.

The results obtained are given below:—

*Distillation of alcohol of strength 20 per cent. by volume, containing furfural, using Still A.*

Fraction.	Temperature °F.	Strength of Fraction % Alcohol by Volume.	Total Mgrm.; Furfural per Fraction.
1	188.5	75.0	0.42
2	191.0	72.5	0.56
3	194.0	62.5	0.75
4	197.0	58.5	0.83
5	201.0	50.0	1.15
6	204.0	33.5	1.07
7	208.0	18.0	1.00
8	210.0	10.5	0.72
9	210.5	5.0	0.44
10	211.0	5.0	0.15
11	212.0	2.0	0.11
12	212.0	4.0	0.07
13	212.0	2.5	Nil.
14	212.0	1.0	"
15	212.0	1.0	"
16	212.0	4.0	"
17	212.0	—	"
18	212.0	Nil.	"
19	212.0	"	"
20	212.0	"	"

*Distillation of low wines, feints, and foreshot mixture from distillery containing furfural, using Still A.*

Fraction.	Temperature °F.	Strength of Fraction % Alcohol by Volume.	Total Mgm.; Furfural per Fraction.
1	189.0	73.5	0.09
2	191.5	77.5	0.11
3	193.5	76.5	0.12
4	199.5	71.5	0.15
5	200.0	62.5	0.17
6	203.0	47.0	0.17
7	206.0	32.5	0.15
8	209.0	20.5	0.12
9	210.0	10.5	0.06
10	211.0	9.0	0.05
11	212.0	8.5	Nil.
12	212.0	4.0	"
13	212.0	8.5	"
14	212.0	2.5	"
15	212.0	5.5	"
16	212.0	8.0	"
17	212.0	0.00	"

18—20 nil.

The results of these distillations show that at a concentration of alcohol similar to that employed in the distillery, the furfural distills as in the other distillations carried out, that is the maximum transfer of furfural takes place at a fairly sharply defined point in the graph when the strength of the distillate is around 12 u.p. = a concentration of alcohol of 50 per cent. by volume and at a temperature around 201° F. It would appear then that the transfer of furfural from still to distillate when the low wines, feints and foreshot are distilled together takes place essentially at a definite temperature of distillation, which will correspond to a more or less definite strength. The temperature of transfer may be deduced as 204° F. As low wines, feints and foreshot (the actual charge of the still used in practice), give similar results for the furfural transfer to those obtained, using pure alcohol and furfural alone, it is evident that the other secondary products present in the still in practice do not substantially affect this temperature and strength of transfer. This is a point of considerable practical importance, for when we consider that the control of this distillation is carried out in the distillery by observation of the strength of the distillate throughout the operation and when we note that the other substances in the spirit do not appreciably affect the distillation of the furfural, it becomes obvious that the periods at which the collection of the different fractions are commenced and discontinued are directly related to the temperature of distillation.

The graphs of the distillation of furfural also show that by varying the strength at which the potable fraction is discontinued so is the content of furfural of the potable fraction altered. In practice the point at which the potable fraction is discontinued varies at different distilleries and may be anything from 10 o.p. = 63 per cent. of alcohol by volume and 5 o.p. = 60 per cent. of alcohol by volume. Now this particular range of alcoholic strength of distillate and corresponding temperature of distillation covers in each of the experiments detailed above that portion of the distillation from where the furfural transfer increases suddenly and rapidly in velocity, to the point where it reaches the maximum for the distillation. If then we can decide how much furfural is desired in the ideal whisky we can to some extent arrange to include this proportion by altering the cutting off point of the potable fraction, *other things being equal*. The qualification is very necessary and in the present state of our knowledge, all important. An obvious conclusion from the distillation curves is that furfural, although distributed more or less over the entire distillation, passes chiefly to the potable fraction just before the collection of this fraction is discontinued, and similarly passes to the feints at the beginning of that fraction.

It must be remembered, however, that furfural is only one of the secondary constituents affecting the character of the spirit, and general practical conclusions must not be arrived at until all the others have been similarly investigated, but we have at least one important link in the chain which should eventually lead us to an understanding of the mechanics and chemistry involved in making a desirable whisky from a given fermented wash. Is there any source of the substances from which furfural can be produced in the wash still in the practical pot still distilling process other than the possible sources already discussed, namely, barley and malt, with acids? The possibilities are few; there is the mashing water in most cases heavily charged with peat, there is also the peat used to dry the malt on the kiln, and finally there is the brewer's yeast, which is added to the wort in the tun room.

It is of interest to know if substances which might yield furfural under the conditions obtaining in the wash still are derived from either peat or yeast.

H. V. Feilctzen and B. Tollens (this *Journ.* 1898, 4, 44) showed that peat contained pentoses varying with the depth at which the peat was found from 6 per cent. to 12 per cent., the smallest percentages being obtained at the greatest depths.

It has also been found that of the amounts of alcohol which it was anticipated would be obtained on fermenting peat, based on the sugar solvent, 28 per cent. was obtained and the discrepancy was attributed to the fact that a portion of the sugars being pentose are unfermentable. One possible source, therefore, of pentoses which will yield furfural in the pot still is the peat which is used so extensively in the practical process. Whether the peat burned in the kiln fire, or the unburned peat carried in the mashing water has the greater influence on the pentose content of the wash would be well worth studying, but this point must be merely noted at the moment for future investigation. The remaining uninvestigated factor in the process is the yeast. Can yeast either actively fermenting or in the form of the exhausted residue in the fully fermented wash yield pentoses or other substances capable of yielding furfurol in the wash still? When yeast as received from the brewers is distilled with or without acid in alcoholic solution of strength 12 per cent. by volume, no furfural is obtained. Yeast therefore would not appear to be a potential source of furfural under the conditions obtaining in the wash still.

We have, we believe, demonstrated the manner in which furfural is produced in the process of manufacture of pot still whisky, and we have described how this furfural is distributed over the potable and non-potable fractions, and how the practical control of the still as now carried out may influence decisively this distribution. We are of opinion that it may be possible to control not only the amount of furfural produced, but the amount passing to the potable spirit. The practical questions arise: how is furfural desirable in whisky? and how much is desirable? There are two methods by which these questions can be answered. We may take a given whisky with a low furfural content, add furfural in varying amounts thereto, and submit the samples to expert blenders and take an average of their opinions. In this way we would find out whether the additions improve the whisky,

and what the optimum furfural content of a whisky is. We would also in this way obtain the practical blenders' opinion of the effect of furfural in whisky as to whether it gives body, character or flavour. The second method is to analyse all the whiskies available, place them in order of furfural content and examine them to see if furfural content and commercial appreciation are related. The second when utilised by us gave the results below:—

Pot Still Whisky.	Class.	Order in Class.	Furfural parts per 100,000 of Absolute Alcohol.
A	1	1	3.86
B	1	2	5.36
C	1	3	3.69
D	1	4	2.65
E	1	5	6.33
F	1	5	6.07
G	1	5	6.17
H	1	5	2.36
I	1	5	2.27
J	1	5	3.89
K	1	5	2.84
L	1	5	2.83
M	1	5	2.81
N	2	1	5.25
O	2	1	4.82
P	2	1	4.43
Q	2	1	2.31

The conclusions to be drawn from this tabulation is that furfural content alone is no criterion of quality amongst the better whiskies, and each may contain from 2.20 up to as much as 6.30 parts of furfural per 100,000 parts of absolute alcohol and still be first-class whiskies. The other method of determining the desirability of furfural, that of adding furfural to a known whisky and submitting the samples to experts is rather difficult of practical application, as necessarily a pot still whisky of low furfural content must be utilised for the test. Fortunately a well known pot still whisky containing very little furfural exists, and when this whisky was treated as suggested above, the opinion of the practical experts was that furfural up to a point appears to improve the whisky, but beyond this point the addition is very objectionable. Furfural then, originally present as a result of the methods employed in manufacture has now become a desirable constituent if present in well defined amounts. The object then of the distiller should be to secure this optimum content of furfural in the whisky by controlling the distribution of furfural in the

spirit still, and this transfer of the proportion of the total furfural produced which passes to the potable fraction may be controlled by the suitable adjustment of the fractions of the second distillation, but this again is subject to the qualification that the other equally important and possibly more important secondary products are found to be transferred to the distillate in a manner which can be controlled in accordance with the furfural transfer.

#### *Furfural in casks.*

The changes occurring in furfural content during the years of maturing in cask are obscure and difficult to definitely describe. The maturing changes are exceedingly small in amount, and we imagine of little practical importance so far as furfural is concerned, but concrete evidence is difficult to obtain. In the one outstanding series of observations carried out on the same whisky over a period of years on this point on American whisky, results, unfortunately, with little practical significance to-day, for purely legal reasons, the furfural content increased very slightly or remained constant, but the changes were very slight. Similar results have been observed with Irish whiskies, while with Scotch whiskies speaking generally, there is distinctly less furfural in the mature than in the new whisky, again, however, with some exceptions. These observations on Scotch and Irish whiskies, however, do not refer to the same whisky observed over a period of years, although they refer to the product of the same distillery, and, therefore, in the light of our observations on furfural production and distribution are of little value. This is a subject which is in great need of investigation.

As a matter of interest, we conclude with some figures collected from various sources

showing the proportions in which furfural is found in spirituous liquors other than whisky.

We trust we have advanced our knowledge a little in the direction of a better understanding and control of the pot still, but we specifically limit the deductions which can be drawn from these results until the other secondary products have been similarly investigated.

#### DISCUSSION.

Professor J. T. HEWITT said that for years it had been known, or rather suspected, that furfural must be derived from the pentoses, and there was an idea that the conversion of pentose into furfural was due to the action of acid. The authors had confirmed that acid converted pentoses into furfural, but that in the case of pot still whisky the low concentration of comparatively weak acids, such as acetic acid and lactic acid, could not be held to be responsible, and that the charring must be the real cause. That, undoubtedly, was a very great advance. There was one thing Mr. Hastie had remarked upon early in the paper which provoked a smile, and that was with regard to the cleaning of the stills. He himself could give a case in point in which the absence of cleaning of the stills might have a very considerable influence on the character of the whisky produced. He was in a certain distillery for about a week some years ago and a very conscientious still-man was in charge. At the end of the period the whisky was not what was desired, and one of the proprietors told him that the whisky tasted in a manner which they had been trying to prevent for the previous five years. When the still-man was cross-examined it turned out that he had been rather too energetic in his cleaning, and had cleaned beyond the point to which he should have gone in order to

Spirituous Liquor.	Furfural. Parts per 100,000 of absolute alcohol.	Authority.
American Rum	10.5	Goot.
Rum (one sample 14.61)	1.0—6.0	Heron.
Rum from West Africa	14.0	Goot.
Brandy	—	Fernbac.
Eau de vie des Midi	Traces to 1.26	..
Eau de vie des Chartres	1.1 to 3.00	..
Public-house Brandy	2.4	Schidrowitz.
Gin from West Africa	24.5	Goot.
Irish Whisky	.5 to 8.9	Yeed.
Pot Ale	2.3	Schidrowitz.
Spent Lees	0.3	..

maintain the characteristic flavour of the particular whisky.

His first interest in furfural in whisky arose some 30 years ago when people attributed rather pronounced poisonous properties to furfural, and it was thought that if furfural could be removed from whisky, a comparatively harmless drink would be produced. Now, however, it was admitted that furfural had very little to do with possible ill-effects from drinking whisky, and that these ill-effects were much more likely to be due to the quantity a person drank than to the quality.

With regard to the non-occurrence of furfural in grain-spirit, he would like to ask the author whether this was not due to the action of the rectifying column of the patent-still which caused the furfural to separate with the fusel oil rather than with the spirit fraction. Most specimens of fusel oil contain furfural, and he believed that before the reactions of furfural had been properly studied, the coloration with aniline acetate was looked on as a test for fusel oil or amyl alcohol itself.

He looked forward with great interest to the authors' future work, as matters of considerable interest would arise when the cause of the production of other by-products was closely examined. It was a pity that samples could not be taken during the actual distillation, but he was afraid that the easy-going ways of the South of France would not be allowed in this country.

Mr. J. L. BAKER expressed his thanks to the authors for a most interesting paper, and for the logical and scientific way one of the most difficult problems facing the distiller had been tackled. The authors, with the instinct of the chemist, had fixed on furfural, a body which was capable of being determined with a very considerable degree of accuracy, and had confined themselves to an estimation of that particular substance in whisky. Similar investigations would require to be made of the other secondary constituents, and those who knew of the difficulties of such work would realise that the authors had a Herculean task before them. He was interested in the remarks about the presence of pentoses in the wort, because it had some bearing on work which Mr. Hulton and himself carried out ten years ago, which was concerned with the presence of an enzyme

in green malt which was capable of splitting up some of the pentosans or, as he preferred to call them, furfuroids, into simple pentose sugars. The malts which the distillers used were dried at lower temperatures than brewers' malts, and would, therefore, contain more pentosase, as that enzyme was susceptible to high temperatures. This was the reason, he expected, why a distiller's wash would contain more pentose than a brewer's wort. He would ask the authors if they had made any attempt to estimate the amount of pentose sugars in distillers' worts?

Mr. A. C. REAVENALL said that at one time he had some connection with distilling, and he began to wonder whether he was quite wise to leave it for brewing. It looked as if matters biological were of very small concern to the whisky branch of the fermentation industries. Possibly the authors would try to associate the types of yeast used with some of the characters recognised in the finished product, whisky. Apparently the kind of yeast used and the type of fermentation employed had very little to do with the final character of whisky.

This must be disappointing to many manufacturers of the sort of plant in which brewers are keenly interested. What interest had a distiller in the respective merits of, say, copper or aluminium for fermenting vessels? All he asked for was "a vessel" in which to ferment his wash. Acidity was a matter of indifference to him unless of course the acid were acetic, which would mean loss of alcohol. No problem of refrigeration troubled the distiller. Truly a brewer might wonder why he had ever left the seeming Elysium of the distilling industry.

But there was answer enough when one remembered the irksomeness of the Excise regulations governing distillery practice, and it was in itself a high tribute to the determination and pertinacity of the authors that in face of these restrictions and limitations they had been able to carry on useful research work.

It had been suggested that the production of furfural from the pentose sugars was due to the caramelisation resulting from the super-heating of the inner face of the copper bottom. This brought to mind the old controversy in brewing of fire *versus* steam boiling of coppers. It was a fact that a great many of the more important brewers now boiled entirely by steam and not by direct



fire, and there was a great deal of evidence that the change over had not in any way affected the flavour of the beer. That was some evidence that such caramelisation, if it took place, was so small as to be quite immaterial, and in view of the considerable body of opinion on that point in the brewing trade, he would like to suggest that the explanation of the formation of furfural that had been put forward in the paper needed the closest scrutiny. It did not look as if any other explanation could be found, and the presence of furfural in caramelised malt products was a very great point in support of the authors' supposition. The point he wished to make was that what was long regarded as a factor in flavouring beers was now largely held to be no factor at all.

Mr. C. A. WARREN remarked that the authors had stated that brewers' yeast as received and distilled with acid yielded no furfural, but it was possible that such yeast had exhausted its reserve of carbohydrate which might conceivably give furfural. Yeast built up a reserve of carbohydrate during fermentation, and got rid of it during the resting stage, and the yeast the authors obtained in the pot still would be yeast presumably which, having finished fermentation, had its full quota of reserve carbohydrate. If the authors would test the yeast separated from a wash, that might possibly yield pentoses where the brewer's yeast did not.

Dr. J. H. OLIVER remarked that it was recognised that the carbon-dioxide obtained from brewing was different in character from that produced by other methods, and the difference was generally attributed to traces of the higher alcohols and esters. The work of which this paper formed a part of a series, was likely to be of considerable importance when brewers came to assess the value of carbon-dioxide collected from their own fermentations. He was under the impression that a very small amount of furfural could be produced by fermentation and that traces were to be found in wines and champagnes, although, in the case of the latter, it might be introduced by the liqueur added, which was a product of distillation.

Mr. R. J. B. STOREY (President) said that in the brewing trade, the quality of the beer produced seemed to leave the purchaser of the brewery perfectly cold. It seemed, however, that whisky was produced more

by tradition than by science, and a man who had a distillery which could produce a full whisky had a valuable asset. Mr. Hastie had suggested that furfural was largely produced by caramelisation and he wondered if it were possible to remove the rousers from the stills. Years ago all the coppers at his Chiswick brewery were fitted with the rather alarming chain rousers, which were practically impossible to keep clean and needed constant renewal. Contrary to all the traditions of the place at that time, he had insisted on removing the rousers, and the coppers were much easier to clean in that they were unhampered by the rousers, and except that the bottoms of the coppers showed a certain amount of caramelisation, the rousers were never missed. Possibly the little extra caramelisation which was obtained in this way would produce more furfural.

Mr. HASTIE, replying to Professor Hewitt on the question of pentoses and acid, said that when they started on this question of furfural they took it as the basis of the whole matter that furfural arose from the action of acids on pentoses in the still, and for a long time they worked from that point of view, and did not consider any other possibility until they got to the point where furfural was not obtained with the amounts of acids and pentoses which they had to deal with. In fact, even when pentoses were in the wash in large excess, they still got no furfural worth speaking of, but they also found that when the acid was concentrated by distillation or by direct addition of strong acid, furfural could be obtained. So long, however, as the acid was in the concentration in which it appeared in the wash still—because the concentration of that was never very high, as practically 50 per cent. of the still charge was sent to the drains—the acid never reached the concentration at which furfural was produced. That seemed to call for some other explanation, and they felt that the acid and pentoses as found in the wash were not sufficient to explain the presence of furfural in whiskies. He seemed rather to have given the impression that distillers never cleaned their plant. He should, perhaps, not have said that the stills were never cleaned, but that it was not the custom to remove the deposit which was automatically left in the worm and in the lyne arm by the action of distillation. It was that, rather

than that the distilleries were not cleaned, because the fermenting and other vessels were scrupulously clean. With regard to furfural being a poison, he had heard of this before, but had been unable to trace the statement in the literature. A curious thing was that certain countries which import whiskies in large quantities now insist upon a signed analysis by an independent analyst showing that the particular whisky imported contains a certain percentage of furfural, and if this whisky does not show, on analysis, the prescribed proportion of furfural, it is rejected. As to the effect of furfural upon health, the amount in present-day whisky was very small. When one considered the strength of alcohol in whisky and the proportion of the secondary constituents which could be in a bottle of whisky, it would be appreciated that it must be very small indeed and, as had been suggested, the danger came rather from the quantity of liquor consumed than from the furfural that was in it. With regard to furfural appearing in grain spirit, they had examined many examples of grain spirit and had never found furfural, but it was interesting to hear that it was found in fusel oil from grain distilleries. The paper did not deal with patent still whisky, but with pot still whisky. He had always been under the impression that there was no furfural in patent still whisky, and the fact that it occurred in the fusel oil was an interesting point, and one which he would like to follow up. As to Mr. Baker's hope that they would live long enough to carry on the work dealing with the other constituents of whisky, he was afraid that he was up against difficulties, for, whilst it was possible to determine acids and esters and furfural in whisky, the methods for examination of the higher alcohols which affected the character of whiskies, were extremely difficult to work with, very vague in their results, and altogether undependable, and it would be a great step forward if somebody could devise really reliable methods for estimating these substances. The next step was not to estimate what happens with the higher alcohols and aldehydes, but to find a reliable method of dealing with the higher alcohols in such a manner that it was possible to estimate their effect upon whisky. The yeast used by distillers was mentioned by Mr. Reavenall who stated that distillers

did not seem to care what sort of yeast was used. The fact was, however, that Mr. Reavenall probably knew more about distillers' yeast than the distillers themselves, because they bought their supplies from brewers and had to take what they could get. So far as his own particular process was concerned it did not matter where the yeast came from so long as it was good, and sound and clean. There was no question of a pot distillery requiring anything in the way of a pure culture. As a matter of fact, better results seemed to be obtained by taking a mixture of yeasts obtained from different sources rather than from one source only, although why that was so he did not know. He also agreed with Mr. Reavenall as to the annoying Excise restrictions. The fact was that the distillery was hardly controlled by the distilling firm. Everything must be done in accordance with the Excise regulations, and as an example of the difficulties, he mentioned that he had once desired to take samples every quarter of an hour from a still during a four hours' operation, in order to examine the samples, but although he tried every possible way to get permission they would not allow him to take 20 cc. samples at quarter of an hour intervals throughout the run of the still. The question of fire heating *versus* steam heating was of importance in connection with pot still distilling, and it had been found that the character of the whisky was frequently detrimentally affected by reverting to heating the wash still with a steam coil, where it had previously been heated by an open fire. They were not prepared to explain why that was so. They would not say that it made the whiskey worse, but the conservatism of the distillers was such that if there was an alteration of any sort in character they would want to go back to where they were originally. As far as they could find out from testing a large number of samples, there was no furfural in a distillers' wash when it went to the still. They had tried a large number of fermented wash samples in various distilleries, and had found no furfural in the wash as it went to the still. He was interested in the remarks of Mr. Warren regarding the reserve of carbohydrates in the yeast and the difference in the yeast as it was received from the brewer and as it appeared in the wash at the end of fermentation. It was quite possible that furfural was not produced

from the yeast when distilled with alcohol as it was received from the brewer, whilst there might be furfural in the yeast in its exhausted condition in the wash back at the end of the fermentation. Dr. Oliver had referred to the higher alcohols, and these were the most interesting and probably the most decisive factors in the making of pot still whisky. The higher alcohols had more influence on whisky production than anything else, but they had been quite unable to tackle this side of the branch. Until there was a really reliable method for investigating these higher alcohols, individually, if possible, it would not be possible to find out very much about them. He and Mr. Dick hoped, however, some day to be able to tackle that very interesting question.

The different values of the products of different distilleries had been a matter of surprise to Mr. Storey, but it was a peculiar thing that in Campbelltown, for example, distilling was very unpopular, and at the moment there were stills and plant there which were practically without value, but a similar plant might be worth thousands of pounds on the Spey side in the North.

The character of whisky depended not only on the plant but also on the location of the distillery. Whether that was due to the particular peat which existed in the area where the whisky was produced, he was not prepared to say, but there was no doubt that the location of the distillery made a great difference to the value of the distillery. As to the removal of rousers from a still, if that were done, it would be impossible to carry on at all because the burning and charring that would go on in the still would be so excessive that the whisky would actually taste not only of furfural but of burning. In the case of the brewer the wort was put into the copper without any yeast, etc., but the distiller was putting into the still a liquid which, by comparison, was almost a semi-solid. It was carrying suspended yeast and other matters which came from the mash tun. Mashers were not run with so much care as in a brewery. The worts were not run brilliant and some suspended matter went through. This was more difficult to handle than the liquid which went into the brewer's copper, and therefore it would not be feasible to remove the rousers.