MEETING OF THE· SCOTTISH SECTION HELD AT THE CALEDONIAN STATION HOTEL, EDINBURGH, ON THURSDAY, 22ND JANUARY, 1925.

Mr. DAN ROBERTSON in the Chair.

The following paper was read and discussed.

THE APPLICATION OF CHEMISTRY· TO POT STILL DISTILLATION.

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THE application of science in the forms of chemistry, physics, and biology, to the control of the pot still distillation processes is still in its infancy after a long series of years of intermittent attempts to make practical use of laboratory work in this direction. Thirty years ago the brewer and maltster regarded the chemist usually as an unnecessary encumbrance in practical work and a drain on profits. To-day we find the laboratory an established fact in all important breweries and maltings, and the services of the chemist utilised, and essentially required, by all progressive concerns.

It is unfortunate, but it is a fact, that pot still distilleries with a few exceptions occupy the same relative position to chemists and laboratories to-day that breweries did thirty years ago. Subsequent to the war, however, science has forced itself upon the attention of the conservative distiller, and efforts are now being made to apply laboratory control to pot still distillation.

The following notes have been collected with the intention of indicating some of the problems most urgently requiring attention, and secondly, to offer for criticism a small amount of preliminary work which appears to be suggestive of future lines of investigation.

It would be well, perhaps, at this point to say that the application of science to pot still work must be carried out under a carefully balanced scheme embodying control of existing practical processes and research into present conditions and methods with a view to putting them upon a scientific practical basis. Perhaps at this time of day it is not necessary to point out that a scientist must not be expected to enter a distillery and after a few months effect fundamental changes, for nothing short of a magic wand will operate in this way, and fundamental results arrived at thus rapidly, are, in the light of experience in other directions, very likely to prove misleading.

Economically sound results are only arrived at after long and patient scientific investigations combined with practical knowledge.

Immediate valuable benefits will, however, certainly follow upon a system of scientific control of processes carried out in close contact with practice, and from such continuous control ; questions, soluble only after painstaking research, will arise and in their turn may be dealt with. Academic research alone, apart from this control is, in the present writer's humble opinion, of little value, and an ever present danger to the general progress of the practical scientific combination which has been referred to. In a word, if we are to advance at all, the pioneer work must be carried out by technical men with scientific training in biology, chemistry and physics, with, and this is of the first importance, practical connection with and knowledge of the daily operations of the distillery.

It is proposed then to deal in the first case with control of practical processes.

The Objects of Practical Control.

At the present moment and hitherto, when the barley employed by pot still distillers did not for some reason " produce " the expected amount of spirit, the explanations given by the practical man were varied and generally (and one might say even naturally) directed to those materials employed, whose origin was extra mural ; thus, poor yeast, bad barley, and such like general and sweeping explanations were given. Sometimes they were accepted, sometimes they were not, but in either case, generally speaking, the definite specific cause of reduced yields was not forthcoming. This state of affairs resulted from a very superficial knowledge of a very complex and conservative rule-of-thumb series of processes, of the precise workings of which the general information was, and still is, very meagre.

The objects of control are to enable the distiller in the event of reduced yield to place his finger upon a detail of process and say the loss was *there*. In this way future losses may be avoided and a little more light may be shed on a complex and difficult manufacturing process. Considered as a rule-of-thumb process without guarantees of yield, the process is very simple, but considered as a problem in the production of full yields of a definitely uniform product the subject bristles with difficulties of the most complex kind.

Before touching upon systems of control, I would like to emphasise the fact that, although much will be said about yields in what follows, in no case are quality and character subordinated to mere total output. Quality and character are the prime essentials of the business, and all that I may say as to the value of control is to be taken subject to the proviso that this control is of economic value, and where it has affected existing character it has improved it, and has certainly not proved detrimental. In the few cases of application of science in which character has been likely to be adversely affected, the suggestions have either been ruthlessly scrapped or referred back for further investigation.

System of Control.

The most satisfactory system of control so far as economical working is concerned is based on a subdivision of the manufacture into malting, mashing and fermentation processes, the efficiency of each subdivision of process being finally arrived at as a percentage of the maximum theoretical yield with a standard set which the distiller is expected to attain. In this way when produce falls, the reason can usually be allocated to a particular subdivision, and the field of search for the exact practical cause of loss is at once localised. We thus ascend from art to science.

The method of working such a system of control generally is as follows :—The barley in the first case is examined, and starch percentage and germinative capacity determined. The nitrogen percentage we will leave out of account meantime, as this is regarded as a refinement of the valuation when it is possible to apply it, that is, when time permits. Under normal market conditions it is rarely feasible to delay purchase until such a complete analysis is carried out.

The question of moisture need not be discussed, as obviously the amount of water bought as barley is a simple proposition. When the malt made from this barley is examined on leaving the kiln, the amount of extract obtained in the malt of that available in the barley is estimated, using a moisture standard of 3 per cent. for malt, and the result taken as the efficiency value of the malting process for that particular kilning and barley. This ends the first stage of control, and an unusually low yield of the available extract is discussed with the practical maltster and the cause located. In this way it is found that most of the causes of loss can be systematically located and dealt with, and although it is not forgotten that these determinations are not absolutely accurate, yet the small differences between laboratory and distillery results are ignored. For practical purposes the approximation suffices. It is not suggested that this valuation is in any way novel, but my point is that it may be applied, as it has been applied, in any given set of circumstances after a certain amount of necessary preliminary work has been carried out. It is not the type of determination which one finds in text books, but requires the exercise of discretion in its application, and is largely a careful blending of laboratory and practical work with common sense.

As to the *quality* of the malt ; in distilling, the conversion power and fermentability are estimated in the laboratory by the usual estimations, all of which jointly and separately give clear evidence as to kilning treatment and probable available yield of spirit.

The second stage of control commences with the grist. The grist is examined and the yield available calculated in Brewers' lb. of extract. It is necessary to do this, as the malt is subject to change during storage, (and to very disadvantageous change in some distilleries where storage in open loft is still employed) and it is also impossible under practical conditions to trace individual kilnings to the mash tun process, owing to conditions of storage in bulk which obtain in most distilleries.

We commence therefore *de novo* with the grist, and compare the yield of extract with the yield in practice and again calculate the efficiency coefficient of the process as a percentage of the available. Hence the loss, if any, in the mashing process can be allocated. This is, I believe, common practice in up-to-date breweries, and

has been applied equally satisfactorily in distillery work.

The third stage of control commences with the fermentation process, that is, with a given amount of wort of a certain gravity. The fermentability test in the laboratory gives figures for complete fermentation which are compared with the actual results in the distillery and the difference is calculated out as a percentage of the available. The coefficient of the efficiency of the process for fermentation is thus arrived at.

In all of the above it must be remembered that losses due to practical conditions of working must be considered in setting the various standards of efficiency expected. This is a matter for experience in working the method of control, in relation to particular distillery plant. The results obtained are satisfactorily accurate, and, as indications of loss, and as means of locating definite losses, they are very valuable in practice.

Control of Yeast Supplies.

Hitherto the scientific care of yeast both as to storage and control in pot still distillery work simply has not existed. Consider the supply and transit of yeast. As soon as the brewer has finished skimming and pressing, the yeast surplus is dumped into the general sump which is the distiller's source of supply, from there it is forwarded (in casks or pressed in sacks) by rail and to the Western Isles by cargo steamer. It is frequently on deck in the heat of the sun, uncovered, it is collected at leisure and stored in the distillery yard regardless of temperature or anything else, and is finally drained off in buckets through a cock in the cask, the mouth of which is filthy as a rule with dried yeast, etc., from previous buckets. It is then tipped into the wash back to ferment the wort and is by this time a mixture of doubtful cleanliness, of very uncertain vitality, and certainly always a seething mass of organisms of every description. Truly a picture black enough to make Pasteur, the scientific father of our allied industries turn in his grave. Control has changed much of this, but much remains to be done. Arrangements for storing, suitably treating and revitalising the yeast subsequent to transit and prior to pitching are now being effectually employed. Control of yeast therefore up to the point of use is invaluable with determination of fermentative power, the general cleanliness, and the freedom from abnormal contamination by bacteria. Such control eliminates the most frequent excuse, if I may use the word, for poor yields, that is, alleged poor yeast.

Frequently the wort in which the yeast is expected to work is the cause of poor yields in fermentation process and not the yeast itself in distillery work, as it will be obvious that if the yeast is treated in the haphazard way indicated above, then it is almost impossible to prepare a wort regularly adapted to such a yeast in such a condition. There is no doubt that the yeast used is frequently blamed for defective fermentation, when the wort is the real determining factor, and so long as worts are prepared, may I say scientifically, blindfold, the distiller cannot definitely say whether the yeast or the wort is the cause of unsatisfactory results, but by controlling each of these factors he may strengthen his position enormously when complaints have to be made to the provider of his yeast. I do not for a moment suggest that the distillers wort is always or even most frequently to blame, but on the other hand, although the output of brewers' yeast is remarkably consistent and bacteriologically reasonably clean, yet there are, it seems, still a few brewers who regard pot still distillers rather as public destructors of their surplus yeast who pay for the privilege of removing it, and who are a valuable alternative to the drains.

Here then we have, briefly outlined, a process of control which can be elaborated in individual cases to enable the laboratory to precisely state the losses in any given period in proof spirit allocated to each part of the process with an accuracy quite sufficient for practical control and carrying with it information as to definite mistakes and misfortunes, thus we tend to decrease losses and increase knowledge of the processes concerned, and become acquainted with and may anticipate the most probable definite sources of loss.

In the application of the system, graphs are plotted of efficiency coefficients which are kept throughout the season, and afford immediate comparisons of various distillery productions in all departments of process on a basis strictly comparable, and independent of the nature or quality of the barley employed. Distillers are prone to compare the produce of different distilleries, although the materials may vary so much that such a comparison is unfair to the operative distiller and quite worthless.

In the control system described above, definite comparisons can be obtained although materials used may be altogether different. Instead of the usual comparisons of what two different distilleries are getting from any one barley, we are arriving at what any distillery is getting out of the available in any barley, and further we control this yield from beginning to end of the process at definite stages.

In my opinion much harm has been done to the application of science to pot still distillery work by the desire to go too fast, the desire to discover things of fundamental importance. This is a mistake, things are scarcely ever discovered in this way and control is the desirable jumping off point and must precede research. Control must come first and be complete and research must follow as the outcome of control.

Having superficially dealt with this question of control, it might be of value to indicate possible lines of research in other departments of pot still work. There are, of course, the questions of mashing and setting heats, yeast pitching rates, etc., conditions already more or less effectively standardised by long experience, but the questions which most urgently call for research are :—

1. *The mashing process. The question of extra yield.*

Apart from the obvious points to be attended to in order to increase the yield of extract in the distillery, such as the finest grinding compatible with good drainage and the lowest moisture compatible with low kiln heats, there is the question of treatment of mash and mashing water with inorganic salts. Nettleton in his book on distilling states that 20–40 grains per gallon of salts such as NaCl or $CaCl_2$ will enhance the diastatic power and increase the amount of protein matter dissolved in mashing.

He further states that using 35 grains per gallon of NaCl or $CaCl_2$ or those salts mixed in equal proportions an increased spirit yield of from 3 to 15 per cent. is obtained. If these salts are added to the collected wort at pitching then it is further maintained that a more complete and more rapid fermentation is obtained. The following experiments were carried out to ascertain the amount of the increased yield obtainable in the above manner.

In the first experiment, inorganic salts were added in varying proportions from 7–70 grains per gallon to the water used for mashing. The distilling water met with is an alkaline water containing 6–7 grains of salts per gallon. The inorganic salts were added to the mash tun at mashing in experiment 1 and in experiment 2 they were added to the wort after collection and before pitching.

In all cases the mashes consisted of 85 grms. of malt per 500 c.c. of water under the usual conditions of mashing, collection and pitching practised in the distillery.

The figures for a mash made without inorganic addition are given for purposes of comparison. The figures given are typical of a long series of tests.

TABLE I.

Mash No.	Substance Added.	Grains per gallon.	Original Gravity of Wort.	Increase.	Fermented.	Increase.
				Per cent.	Per cent.	Per cent.
1	Nil	Nil	45·66	—	81·6	—
2	Sodium Chloride	35	46·58	1·1	81·9	0·3
3	„	42	45·78	0·2	82·1	0·5
4	Calcium Chloride....	35	46·00	0·7	82·5	0·9
5	„	52	46·26	1·3	82·9	1·3
6	Sodium and Calcium Chloride	17·5 + 17·5	45·78	0·3	87·8	6·2
7	Potassium Chloride	35	47·18	3·3	81·6	—

TABLE II.

Mash No.	Substance Added.	Grains per gallon.	Original Gravity of Wort.	Increase.	Fermented.	Increase.
2	Sodium Chloride	35	—	—	82·0	—
3	Calcium Chloride	14	—	—	81·9	—
4	Sodium and Calcium Chloride	17·5 + 17·5	—	—	81·8	—
5	Potassium Chloride	15	—	—	definitely detrimental	—
1	Nil	—	—	—	81·6	—

The addition of the inorganic salts experimented with gave increases of extract yield and of fermentation yields when added at mashing. When added to the collected wort from a normal mash, the fermentation yield was increased in every case, except that in the case of Potassium Chloride. The large increases obtained by Nettleton were, however, not realised.

From further work carried out on this question and not recorded here, as being still in progress, the opinion has been formed that the hydrogen ion concentration of the mash water and mash materials was the real factor operating in these experiments and in those of Nettleton, as adjustment of the Hydrogen ion concentration of the water taking into account the mashing materials has a greater and more definite effect upon the yields than the addition of salts alone, and further the effect of these additions is found to be largely due to their influence upon the p_H of the mash, and is, in fact, an inefficient " trial and error " method of adjusting the p_H. The adjustment of the p_H of pot still distillery mashes and worts is of the utmost importance to distillers and is at present the subject of investigation, but it is regretted that further details cannot be set down here meantime.

It must be remembered, however, that the success of the treatment of the mash is determined not by the water composition alone, although this is of importance, but to a much greater extent by the p_H of the mash as influenced by the malt used.

2. *The Fermentation System.*

A great number of problems connected with fermentation are met with in massed formation in the distilling process. The conditions of yeast storage and treatment before its use in the wash back, call for improvement and change, without touching upon such subjects as the effect of the p_H on the fermentation process. As, however, there is something to follow about distillation itself, which will take up some time, it is not proposed to deal with the fermentation process in this paper.

3. *Distillation.*

The mechanics and chemistry of distillation in a Pot Still are, in spite of much work by Schidrowitz and others very obscure and until a great deal more is known definitely and precisely of the distillation process, cotrol on scientific lines is impossible.

In the present notes it is intended to deal only with the actual distillation process which is chiefly, although not altogether, responsible for the stamping of character on whisky. In making this statement, I am not forgetting that the substances responsible for the various definite characters in whiskies are produced throughout the process from barley field to cask, but one great difficulty is to so manipulate the primitive pot still, that the desirable is separated from the undesirable and to this difficulty, attention must be directed primarily. When a definite scientific method of control has been arrived at, then attention may be turned to the origin of the substances stamping the character on spirit, but so long as the distiller is in the dark as to the precise effect of varying the distillation process with a given fermented wash, it is premature to enquire into the origin of the character producing impurities in the wash.

It is a problem which must be dealt with step by step and the nature of the case forces us to deal with it backwards. That is to say, we must deal with the second and final distillation first, wherein separation and interaction of substances takes place, before we are in a position to deal with the primary distillation where separation and interaction of substances also takes place, but where in addition new substances are actually produced not previously present in the wash. In this way the control of the selection of the different desirable materials from the wash, and the exclusion of the undesirable, must be definitely settled, and then the origin and nature of the substances can be tackled. Briefly, we must understand the mechanics of the still first.

Without occupying time by going into the routine details of mashing and fermentation, with which most are familiar, and which Schidrowitz and others have dealt clearly with in various publications, suffice it to say that the wort is fermented in partially closed vessels under rather primitive conditions. Fermentation having ceased and the wort having attenuated from 1,050° or thereby to 997–998°, the resulting wash or fermented liquid is distilled in the " Wash " still, the distillate being collected in a receiver termed the low wines receiver. The distillate is a weak alcoholic solution containing in addition to ethyl alcohol in a concentration of about 66·2 u.p. (19·3 per cent. by volume of absolute alcohol) various amounts of impurities such as furfural and other

aldehydes, esters and higher alcohols, and a number of other substances in very small amount, concerning which the sum total of our precise knowledge at present is negligible.

In the first place the composition of a typical low wines may be considered. Schidrowitz and Kaye give the following figures (*Table III*) (this Journ. 1906, **12**, 506).

	Parts per 100,000 of the liquid.	
	Low Wines.	Mixed Feints and Foreshot
Alcohol per cent. by volume	20·19	41·5
Total Acid	41·7	20·9
Volatile Acid	36·5	19·4
Non-Volatile Acid	5·2	1·5
Esters	70·8	151·2
Higher Alcohols	35·2	44·2
Aldehydes	2·5	12·5
Furfural....	1·9	2·8

To digress for a moment in explanation of what follows, it appears to be the case that after the Royal Commission on whisky had given its findings the tendency was to attempt to eliminate partially or wholly many of those impurities from pot still whisky which constitute its commercial asset and as the amounts and nature of these impurities are of first importance, the endeavour of the distiller is now to attempt to retain definite proportions of them in his whisky where such is already in active demand, or to attempt to incorporate the desirable and eliminate the undesirable, where the existing demand for his particular product is small.

In other words, if a particular whisky which has been less valuable for blending purposes for some time on account of the undesirable impurities present, and also of the desirable impurities absent, could be altered in composition to adjust these impurities in a desirable direction then that whisky in spite of the possible prejudice accompanying its geographical location would eventually become more valuable to the blender and this is a far more important matter than the production of undesirable, or at least *less* desirable whisky at a low cost per gallon. The unhappy term impurity is used as descriptive of all substances present other than ethyl alcohol.

It is not easy, in fact it is not possible, in the present state of our knowledge to state definitely in chemical terms what are desirable and what are undesirable constituents amongst the impurities in whisky, which stamp the product with its distinctive character. Certain we are, however, that these substances are almost as small in amount as they are powerful in effect. They are doubtless largely derived from the peats used in drying the malt and also found in the water which in the large majority of cases (in Highland whiskies) drains through peaty soil. In addition there are the by-products of the fermentation process.

Without more than merely indicating our comparative ignorance of these matters (the nature and origin of the impurities) I will proceed to bring forward now some facts concerning the content of these impurities found in in the low wines and feints according to conventional analyses and to indicate the effect which the distillation process has upon their modification and selection. We will therefore start with the product of the first distillation that is, the low wines which in practice is normally collected and mixed with the foreshot and feints, that is, the first and last runnings from the second and final distillation of the previous period.

This liquid in any distillery will be of fairly constant content and composition. It will be already stamped with most of the characteristics of that distillery, but can still be greatly modified so as to make or mar good whisky in the second and final distillation.

Following the conventional whisky analysis, a typical charge for the Spirit Still consisting of low wines and feints and foreshot together has the following composition :—

TABLE 4.

Composition of Low Wines + Feints + Foreshot, mixture, before distillation.

	Mgrms. per 100 c.c. of liquid examined.			
	Low Wines.	Fore-shot.	Feints.	Low W. +F.+ F. Mix.
Acids, volatile	36·5	11·5	21·6	32·0
,, Total	41·7	11·5	23·5	36·1
Esters	70·8	396·8	81·9	89·3
Higher Alcohols	35·2	121·0	22·5	46·7
Aldehydes	2·5	80·4	Nil	6·8
Furfural	1·9	2·0	3·0	2·1
Per cent. abs. alcohol by volume	20·19	71·12	33·15	26·0

in addition to which a small proportion of other very important character giving bodies are present of which practically nothing definite is known beyond their undoubted existence, and which are therefore not given in the conventional analysis, individually.

If we consider now typical analyses of Scotch Highland pot still whiskies we find the following :—

The function of the second distillation therefore is to select primarily, in the whisky or second fraction an alcoholic distillate containing the above, and to exclude from this fraction by collection in the foreshot and feints and rejection *via* the spent lees, the undesirable balance of impurities. It is at this point that we come up against the primitive character of the pot still, and an examination

TABLE 5.

New Pot Still Whisky. Origin.	Mgrms. per 100 c.c. abs. Alcohol.					
	Acid.	Aldehydes.	Esters.	Higher Alcohols.	Furfural.	Total.
Speyside	16·9	9·2	77·4	407·2	2·9	513·6
Islay	15·0	21·8	63·8	451·6	4·2	556·4
Campbeltown	17·0	33·6	72·8	410·9	3·5	537·9

TABLE 6.

Laboratory distillation of Low Wines + Feints + Foreshot mixture. Analyses of Fractions.

No. of fraction.	Strength.		Acids.	Furfural.	Esters.	Aldehydes.
	Terms of Proof Spirit.	Abs. Alc. c.c. per 100 c.c.	Mgrms. per 100 c.c. Distillate.	Mgrms. per 100 c.c. Distillate.	Mgrms. per 100 c.c. Distillate.	Mgrms. per 100 c.c. Distillate.
1. Foreshot	5·75 o.p.	60·3	18·0	6·7	72·6	110·0
2. Spirit	4·5 ,,	59·7	20·0	7·7	58·6	95·0
3. ,, ...	3·4 ,,	59·0	12·3	9·7	75·6	66·7
4. ,, ...	3·8 ,,	59·3	11·6	1·0	41·0	60·0
5. ,, ...	1·5 ,,	58·0	15·7	1·6	41·0	63·3
6. Feints	0·3 ,,	57·3	15·7	1·6	35·6	20·0
7. ,, ...	7·5 u.p.	52·7	24·0	1·9	8·6	11·7
8. ,, ...	—	—	26·0	1·0	15·0	5·0
9. ,, ...	27·6 ,,	41·3	29·3	2·3	23·0	3·3
10. ,, ...	38·2 ,,	35·3	43·6	2·3	3·6	2·7
11. ,, ...	44·2 ,,	31·7	37·3	2·3	7·6	1·0
12. ,, ...	65·3 ,,	19·7	36·3	2·3	21·6	0·3
13. ,, ...	78·9 ,,	12·0	36·0	2·5	—	0·3
14. ,, ...	85·6 ,,	8·0	34·6	1·7	00·0	0·3
15. Tailings	89·2 ,,	6·0	32·3	1·4	6·0	0·3
16. ,, ...	93·2 ,,	3·0	51·3	0·7	43·0	0·3
17. ,, ...	96·3 ,,	2·0	36·0	0·5	5·3	0·2
18. ,, ...	97·7 ,,	1·3	34·0	0·2	5·3	0·2
19. ,, ...	98·3 ,,	1·0	38·3	0·0	0·0	0·2

of the products passing to the distillate during distillation is given below to indicate the general uncertainty of the distillation as a process of separation.

As will be observed from the figures, Laboratory analyses of fractions demonstrate how crude the distillation process is and how irregularly the impurities come over at different periods of the distillation. In order to ascertain whether in practice a like irregularity existed (as the laboratory duplication of practical processes is often deceptive), a complete distillation of the spirit still in the distillery was examined by collecting samples at 15 minutes intervals throughout the run.

TABLE 7.

Analyses of fractions from Distillery Spirit Still distillation.

No. of fraction.	Strength.		Acids.	Furfural.	Esters.
	Abs. alc. by vol. in 100 c.c. distillate.	Strength of distillate in terms of proof spirit.	Mgrms. per 100 c.c. distillate.	Mgrms. per 100 c.c. distillate.	Mgrms. Ethyl acetate per 100 c.c. distillate.
1. Foreshot	3·3	94·1 u.p.	23·3	0·00	62·0
2. ,, ...	45·2	20·8 ,,	32·0	0·28	104·0
3. ,, ...	74·2	30·0 o.p.	16·1	0·67	214·0
4. ,, ...	74·2	30·0 ,,	12·7	1·33	152·0
5. ,, ...	75·8	32·9 ,,	12·7	1·33	106·0
6. ,, ...	72·6	27·1 ,,	9·1	1·60	102·0
7. ,, ...	71·8	25·8 ,,	9·9	1·23	96·0
8. Spirits	72·6	27·1 ,,	12·5	2·00	86·0
9. ,, ...	77·4	35·8 ,,	10·8	1·00	34·0
10. ,, ...	75·4	31·5 ,,	9·1	1·14	52·0
11. ,, ...	73·4	28·5 ,,	9·9	1·78	42·0
12. ,, ...	75·8	32·8 ,,	10·8	1·34	22·0
13. ,, ...	73·4	28·5 ,,	10·3	1·14	68·0
14. ,, ...	74·2	30·0 ,,	10·9	1·34	86·0
15. ,, ...	70·2	22·9 ,,	11·4	0·89	70·0
16. ,, ...	71·8	25·8 ,,	10·3	1·00	64·0
17. ,, ...	71·8	25·8 ,,	13·0	1·34	80·0
18. ,, ...	71·0	24·4 ,,	12·7	0·94	46·0
19. ,, ...	72·6	27·2 ,,	13·8	1·06	—
20. ,, ...	71·8	25·8 ,,	10·9	1·22	14·0
21. ,, ...	71·8	25·8 ,,	9·5	1·34	56·0
22. ,, ...	74·2	30·0 ,,	7·8	1·22	—
23. ,, ...	71·8	25·8 ,,	9·5	1·45	50·0
24. ,, ...	71·8	25·8 ,,	9·9	1·45	22·0
25. ,, ...	65·6	15·0 ,,	9·9	1·22	54·0
26. ,, ...	60·1	5·3 ,,	9·9	1·60	96·0
27. ,, ...	67·9	19·1 ,,	10·8	1·23	156·0
28. ,, ...	66·3	16·2 ,,	11·0	1·45	—
29. ,, ...	64·8	13·6 ,,	11·3	1·60	—
30. ,, ...	64·8	13·6 ,,	11·3	1·60	—
31. ,, ...	64·0	12·0 ,,	11·9	1·45	24·0
32. ,, ...	66·3	16·1 ,,	11·4	1·78	38·0
33. ,, ...	62·4	9·2 ,,	11·4	1·34	38·0
34. ,, ...	60·1	5·3 ,,	11·8	1·34	68·0
35. ,, ...	64·8	13·6 ,,	13·0	2·00	72·0
36. ,, ...	62·4	9·2 ,,	13·2	1·22	68·0
37. ,, ...	61·7	8·0 ,,	12·9	2·67	76·0
38. ,, ...	61·7	8·0 ,,	13·8	2·67	96·0
39. ,, ...	61·7	8·0 ,,	14·5	1·78	84·0
40. ,, ...	60·1	5·3 ,,	15·0	1·45	80·0
41. ,, ...	58·6	2·7 ,,	14·5	1·60	82·0
42. ,, ...	61·7	8·0 ,,	15·6	1·60	66·0
43. ,, ...	60·9	6·8 ,,	16·9	2·00	40·0

TABLE 7—*continued.*

No. of fraction.	Strength.		Acids.	Furfural.	Esters.
	Abs. alc. by vol. in 100 c.c. distillate.	Strength of distillate in terms of proof spirit.	Mgrms. per 100 c.c. distillate.	Mgrms. per 100 c.c. distillate.	Mgrms. Ethyl acetate per 100 c.c. distillate.
44. Spirits	61·7	8·0 o.p.	15·6	2·00	64·0
45. ,, ...	60·1	5·3 ,,	15·4	1·78	60·0
46. ,, ...	58·6	2·7 ,,	17·8	2·28	54·0
47. ,, ...	57·8	1·5 ,,	14·8	2·28	54·0
48. ,, ...	57·8	1·5 ,,	18·5	2·28	46·0
49. ,, ...	56·3	1·3 u.p.	18·5	2·66	—
50 Feints	56·3	1·3 ,,	19·2	2·66	20·0
51. ,, ...	54·0	5·5 ,,	20·4	2·28	56·0
52. ,, ...	50·3	11·8 ,,	22·6	2·80	42·0
53. ,, ...	47·3	16·9 ,,	24·5	2·80	54·0
54. ,, ...	47·3	16·9 ,,	27·5	2·80	68·0
55. ,, ...	43·6	23·7 ,,	28·1	2·80	86·0
56. ,, ...	39·2	31·5 ,,	29·7	2·80	66·0
57. ,, ...	37·1	35·1 ,,	30·4	1·60	108·0
58. ,, ...	36·4	36·2 ,,	31·6	2·66	84·0
59. ,, ...	35·6	37·6 ,,	31·3	2·66	102·0
60. ,, ...	—	—	31·8	—	—
61. ,, ...	29·2	48·9 ,,	32·9	2·66	100·0
62. ,, ...	25·7	55·0 ,,	32·9	1·78	110·0
63. ,, ...	22·2	61·3 ,,	33·2	1·45	132·0
64. ,, ...	19·95	66·0 ,,	32·2	1·00	108·0
65. ,, ...	18·2	60·0 ,,	32·1	—	126·0
66. ,, ...	15·4	73·2 ,,	31·0	1·06	134·0
67. ,, ...	12·7	77·8 ,,	32·6	0·94	126·0
68. ,, ...	10·7	81·2 ,,	33·5	0·28	66·0
69. ,, ...	8·0	86·1 ,,	32·6	0·20	74·0
70. ,, ...	6·6	88·4 ,,	32·3	0·20	72·0
71. ,, ...	5·3	90·6 ,,	31·8	0·32	74·0
72. Tailings	4·6	91·9 ,,	32·3	0·28	62·0
73. ,, ...	3·3	94·5 ,,	33·0	0·28	72·0
74. ,, ...	3·3	94·5 ,,	32·4	0·24	46·0
75. ,, ...	1·3	97·7 ,,	30·6	0·20	52·0
76. ,, ...	2·6	95·6 ,,	24·3	0·16	—
77. ,, ...	0·7	98·8 ,,	30·4	0·08	60·0
78. ,, ...	2·6	95·6 ,,	30·2	0·08	84·0
79. ,, ...	2·0	96·3 ,,	31·0	0·08	82·0
80. ,, ...	2·0	96·3 ,,	30·2	0·05	30·0

TABLE 8.

Distribution of the impurities in the various fractions.

	Distillery.					Laboratory.				
	Mgrms. per 100 c.c.									
	Fore-shots.	Spirit.	Feints + tailings.	Feints alone.	Tailings alone.	Fore-shots.	Spirit.	Feints + tailings.	Feints alone.	Tailings alone.
Acid	18·5	14·00	60·3	25·5	34·8	17·5	14·6	67·9	30·5	37·5
Higher alcohols	—	—	—	—	—	35·3	26·2	38·5	19·2	19·3
Aldehydes	—	—	—	—	—	61·4	35·7	2·9	2·8	0·1
Furfural	20·7	35·1	44·2	40·2	4·0	16·5	20·0	62·6	48·7	13·9
Esters	36·4	18·7	44·9	26·3	18·6	47·5	35·3	17·2	9·4	7·8

These figures show primarily that the secondary products as distilled from the spirit still are distributed over the fractions very differently with dissimilar distilling, e.g., in the laboratory still and the distillery still, and we cannot say that any particular fraction will have a particular composition as apparatus, method of firing, etc., etc., used, appear to decide this distribution of secondary products over the several fractions.

One of the obvious methods in practice by which a given character may be altered always presupposing a definite low wines and feints and foreshot mixture is by collecting less foreshot or more foreshot, less feints or more feints, etc.

For any given type of distilling apparatus and given type of charge of liquid the total effect upon the analysis of the spirit of altering the collection of fractions is, from the above figures, strictly limited, but generally speaking the effect can ·be deduced from the analyses of a complete " run " of the spirit still given above.

The effect of collecting different fractions generally, will be as follows, using a still of the distillery type investigated, in the above, so far as it goes and referring to the laboratory still for data concerning higher alcohols and aldehydes.

This is, of course, not strictly permissible, as the stills behaved differently but the samples are only obtained with great difficulty from the distillery still and would not permit of all the data being obtained.

The effect of early turning from foreshot to spirits gives more higher alcohols, more acids, more esters, more aldehydes, and less furfural in the spirit.

The effect of late turning to feints from spirit (weak feints) gives less aldehydes, more esters, and increases the proportions of acids, furfural and gives less higher alcohols in the spirit.

The effect of early turning to feints (strong feints) again gives obviously reverse effects in the spirit.

The effect of late turning to spent lees (stopping still) gives feints containing less aldehydes, less esters, more acids and less furfural, and higher alcohols are practically unaffected in the spirit.

The effect of early turning to spent lees (stopping still) gives feints containing more esters and more furfural, higher alcohols practically unaffected, but less acids, and more aldehydes are obtained in the spirit.

It will at once be apparent, however, as already pointed out, that the amount of alteration of character possible by this means alone is very slight although it is of importance, and other methods must be sought to modify the substances characterising the whisky, or to effect their elimination.

The figures given above also indicate clearly the reason for the deep rooted antipathy of distillers to change of the slightest detail of the process or plant lest the product should alter. One can readily understand that any factor tending to greater or less rectifying effect of the apparatus will at once alter the character. Such factors are increasing the height or diameter of the still neck, and inclining the lyne arm ; and the use of plant designed to partially condense the spirit and so return a proportion to the still will have similar effect.

The speed at which the distillate is driven off from the still will also have its mechanical effect on the product. As an instance of the effect of one of these factors a few tests of runs of laboratory stills using the types of head and lyne arm detailed are given to show clearly how one of the variable impurities of the character producing substances, namely acid, actually reaching the whisky is varied with different apparatus.

In this simple case, wash has been distilled and the acids actually carried over when the alcohol has been practically all driven off have been estimated to show one of the effects on the distillate of the type of plant employed. In the case of the wash, of course, most of the acid remains in the pot ale.

TABLE 9.

Amount of Acid distilled when all Alcohol has been distilled.

Types of Lyne Arm and head.	Percentage of total alcohol collected in distillate.	Percentage of total acid collected.
Type as per sketch A	99·9	0·52
,, ,, B	99·6	1·58
,, ,, C	99·1	0·41
,, ,, D	99·9	0·32
,, ,, E	99·9	1·40

These figures show that the total acid reaching the low wines receiver from the wash still varies with the type of apparatus employed.
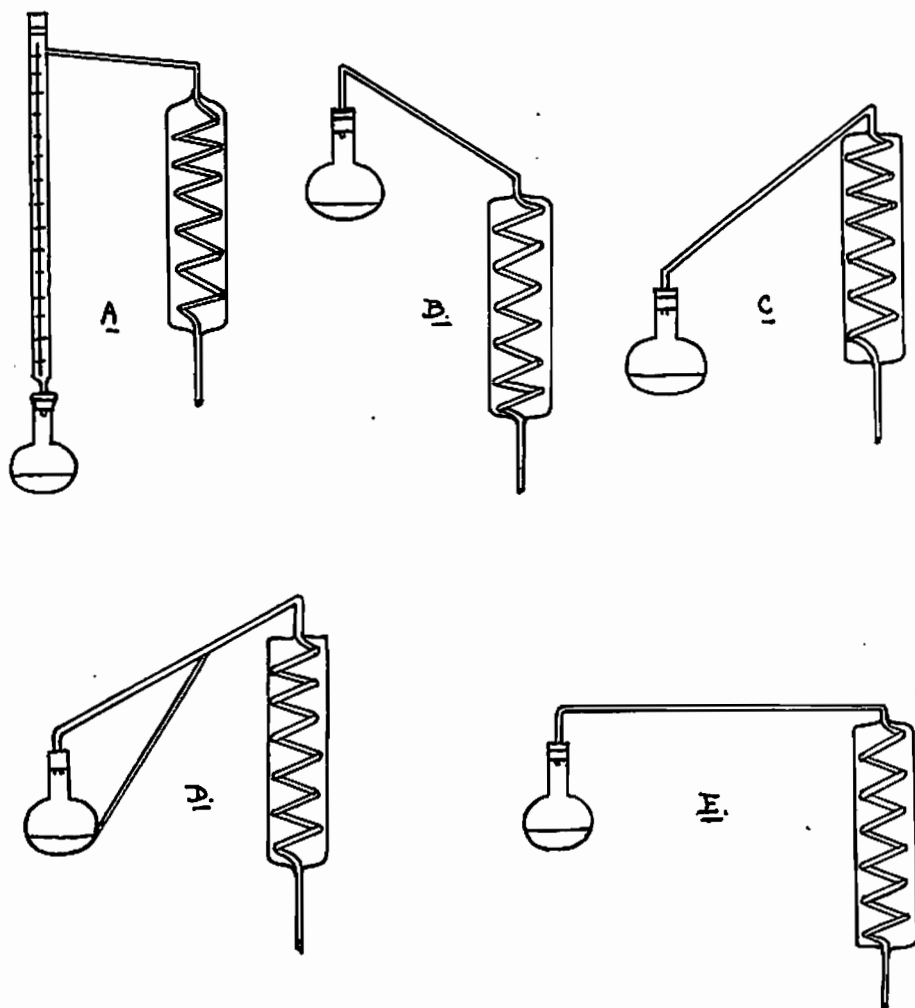
Examples of each of these forms of plant exist in practice, with the exception of type A in pot still distilleries.

The effect of this variable rectification also influences the amount and nature of the impurities obtained in spirit during distillation in the spirit still, as the impurities tending to come over into the distillate vary with the alcoholic strength of the vapour some tending to remain in the still at the higher strengths, and others tending to pass over, consequently more or less rectification will mean corresponding differences in the amounts of the different impurities actually passing into the distillate.

Schridrowitz (*loc. cit.*) pointed out that the simple distillation of low wines, etc., gave rise to changes in the amounts of the constituents in spirit with elimination of impurities via the spent lees.

An attempt has been made below to enquire into the precise effect of distillation, etc., on the contents of the low wines plus feints plus foreshot mixture, both as to amount and modification of impurities.

The process of distillation of the low wines, and feints in the spirit still is essentially a process of hydrolysis of the esters giving acids and water of oxidation of the alcohol to aldehyde, of production of esters from alcohol in presence of acid, oxidation of alcohol to acetic acid, and many other subsidiary actions no doubt. The esters and acids will be dealt with in the first place.

The effect of simple distillation of the low wines and feints on the acid and ester con-stituents is shown by the following tests in which the esters and acids have been estimated in an attempt to find out exactly what happens in this mixture.

Alcohol content of liquid employed before distillation. Strength, 15·18 per cent. by volume of absolute alcohol (73·2 u.p.) con-taining a total of 16·08 mgrms. acids as acetic acid and 17·52 mgrms. esters as ethyl acetate in the 200 c.c. of liquid employed.

The distillation was conducted in a still of

TABLE 10.

Effect of Distillation of Low Wines and Foreshot and Feints Mixture, upon the Concentration and Distribution of Acids and Esters in the Products of Distillation and in the Spent Lees (Residue in the Still).

	Liquid Distilled.	Distillation Products.	Acids as Acetic Acid.			Esters as Ethyl Acetate.		
			Total mgrms. in Liquid.	Per cent. of Total.	Increase or Decrease per cent.	Total mgrms. in Liquid.	Per cent. of Total.	Increase or Decrease per cent.
1	Low Wines and Fore-shot and Feints Mixture employed	—	16·08	—	—	17·52	—	—
	After distillation	Foreshot	0·57	3·0	—	0·92	10·4	—
		Spirit	3·69	19·2	—	1·73	19·5	—
		Feints	4·46	23·2	—	3·34	37·6	—
		Tailings....	1·31	6·8	—	0·43	4·9	—
		Spent Lees	9·20	47·8	—	2·45	27·6	—
	Total in products	—	19·25	—	—	8·87	—	—
	Total increase or de-crease on original contents	—	—	—	+10·7	—	—	−49·3
2	Low Wines and Fore-shot and Feints Mixture after hydro-lysis by boiling under reflux conden-ser for one hour	—	18·9	—	—	8·80	—	—
	Total increase or de-crease on original content	—	—	—	+17·5	—	—	−49·7
	After distillation following boiling under reflux con-denser for one hour.	Foreshot	0·31	1·7	—	0·96	8·5	—
		Spirit	2·27	12·5	—	2·30	20·3	—
		Feints	3·71	20·4	—	3·61	31·9	—
		Tailings	0·73	4·0	—	0·69	6·1	—
		Spent Lees	11·15	61·4	—	3·75	33·2	—
	Total in products	—	18·17	—	—	11·32	—	—
	Total increase or decrease on original content	—	—	—	+13·0	—	—	−35·3

the Thorpe type and fractions corresponding to distillery practice collected, *i.e.*, foreshot, whisky, feints, tailings and the residue of the spent lees.

The results are given in Table 10, Exp. No. 1 and 2.

Apparently the distillation in the normal courses in practice increases the acids at the expense of the esters and decreases the esters, due to the interaction taking place between an alcohol and acid mixture in presence of esters and water. When the low wines + feints + foreshot are subjected to hydrolysis under a reflux condenser a similar effect is observed on the total acids and esters. Further when hydrolysis for one hour is followed by distillation the cumulative effect is a smaller increase in the total acids, and a smaller decrease in the total esters in the fractions.

In these tests and in those which follow, the total amount of esters destroyed does not agree with the total amount of additional acid found, except in those cases to be given in detail below where alkali was added to the liquid before hydrolysis and distillation, the use of alkali limiting the number of possible reactions and allowing of more definite conclusions. In these cases the esters destroyed and the acid increase agree reasonably well as will be shown later.

In the case of the experiments wherein no alkali has been employed, the lack of agreement between esters destroyed and acids produced is, in my opinion more reasonable than a complete agreement would have been considering the conditions obtaining during the free hydrolysis and distillation of such a liquid as this low wines + feints + foreshot mixture. This is probably a case of the reversible reaction known to occur between acids + alcohol and esters + water. The acid and alcohol heated together, yield esters up to the equilibrium point dependent upon the conditions of temperature and relative quantities of alcohol and acid present.

The velocity of the action decreases as the quantity of acid to act upon alcohol diminishes, until there is no further change. This is the equilibrium point.

In the same way the esters and water mixture will under similar treatment yield free acid and alcohol, in decreasing velocity until the equilibrium point is reached.

In low wines and feints we have both actions possible with water, esters, alcohol and acids

(acetic, etc.), present and although an equilibrium point should be reached theoretically after a definite amount of hydrolysis, this equilibrium point will never be actually attained in distillation as the total esters and alcohol vary in amount as distillation proceeds, and products pass to the distillate.

Before proceeding to investigate the conditions determining the equilibrium point in acids and ester production during the hydrolysis of low wines and feints, an experiment with another sample of low wines and feints + foreshot hydrolysed in presence of sodium hydroxide was undertaken to find out whether the neutralising of the acid would limit the further production of esters by removing the acid present to interact with the alcohol, and would remove the total acid produced by ester hydrolysis ; by neutralisation. If these hypotheses were correct the effect would be to allow the ester to alcohol reaction to proceed ; to eliminate the total acid ; and to check the ester production by alcohol + acid and consequently to reduce the total esters.

200 c.c. low wines + feints + foreshot containing acid = 22·4 mgrms. in all were distilled. 225 c.c. NaOH = N/500 were first added, of strength such that 1 c.c. of this NaOH = 0·131 mgrm. of acetic acid, and capable of neutralising 29·4 mgrms. of acid. Therefore a surplus of N/500 NaOH remained, capable of neutralising a further 7·0 mgrms. of acid if formed.

The results obtained are given in Table 10, Exp. No. 3.

In the various fractions a total of 0·49 mgrm. acid were found.

On titrating the spent lees which were still alkaline a surplus of NaOH was found = 4·30 mgrms. acid.

Now the original surplus of NaOH was = 7·0 mgrms. of acid.

Therefore the acid formed and found in the spent lees = 7·0 − 4·3 = 2·7 mgrms. in all.

The total acid therefore formed is 2·7 + 0·49 = 3·19 mgrms. during distillation ; 2·7 mgrms. in the spent lees, and 0·49 mgrms. in the fractions.

This 3·19 mgrms. acid found, if all from hydrolysed esters, would equal 4·67 mgrms. destroyed esters.

Therefore the total esters left should be 20·1 (at start) − 4·67 = 15·43 mgrms. *if no more are formed.*

TABLE 10—EXPERIMENT 3.

Effect of Distillation of Low Wines and Foreshot and Feints Mixture upon the Concentration and Distribution of Acids and Esters in the Products of Distillation and in the Spent Lees (Residue in the Still).

Liquid Distilled.	Distillation Products.	Acids as Acetic Acid.			Esters as Ethyl Acetate.		
		Total mgrms. in liquid.	Per cent. of Total.	Increase or Decrease per cent.	Total mgrms. in liquid.	Per cent. of Total.	Increase or Decrease per cent.
Repeated No. 1 using NaOH in Still 225 c.c. N/500 Soda of strength = 29·4 mgrms. total acid in mixture of 200 c.c.	—	22·4 (all neutralised by NaOH added leaving a surplus of NaOH N/500 of c.c. 53·6).	—	—	20·10 (Free for Hydrolysis.)	—	—
After Distillation (No free acid being present at beginning of Distillation.)	Foreshot .... Spirits .... Feints .... Tailings .... Spent Lees	0·01 0·24 0·19 0·05 2·70	0·2 5·0 4·0 1·0 89·8	Total acid increased from 22·4 to 25·59* =14·3 per cent. increase. This must be due to Hydrolysis of Esters entirely.	1·90 3·91 1·64 0·19 7·84	12·3 25·2 10·6 1·2 50·7	Total Esters decreased from 20·10 to 15·48 = 23 per cent. decrease, due to Hydrolysis of Esters.
	Total in products	3·19	—	—	Total in products=15·48	—	—

NOTE.—(1) In No. 3 a different sample of Low Wines and Feints and Foreshot Mixture was used.
*(2) Consisting of :—

22·40 neutralised by NaOH added.
0·49 found in fractions.
2·70 neutralised by excess NaOH in Spent Lees.
25·59 mgrms. in the Products of Distillation.

——·——

The actual esters found were 15·48 mgrms. which agrees fairly well considering the nature of the liquid investigated.

Apparently then, as suggested, hydrolysis of esters to acids and alcohol and formation of esters from alcohol in presence of acid are taking place simultaneously each time the low wines + feints + foreshot mixture is boiled as in distillation in the spirit still, and by modification and control of this process it may be possible to vary the relative contents of these impurities in the whisky which are all important to the character.

For convenience of reference the results of Table 10, Exp. 1, 2, and 3, are summarised below.

Effect of Distillation and Hydrolysis on content of acids and esters in Low Wines and Feints.

200 c.c. low wines + feints + foreshot mixture employed.

SUMMARY OF RESULTS GIVEN IN TABLE 10.
EXPERIMENTS 1, 2 AND 3.

—	Total mgrms. in 200 c.c.	
	Esters.	Acids.
From experiments 1 and 2— Low wines and feints + foreshot as distilled in practice in spirit still (before distillation)	17·52	16·08
After simple distillation, by addition of esters and acids found in the various fractions and in the spent lees	8·87	19·25
After hydrolysis for 1 hour under reflux condenser the undistilled liquid contained....	8·80	18·90
After hydrolysis for 1 hour under reflux condenser and subsequent distillation and by addition of esters and acids found in the various fractions and in the spent lees	11·32	18·17
From experiment 3— Content of charge of still after adding excess NaOH	20·10	Nil.
After hydrolysis in presence of excess of NaOH and subsequent distillation and by addition of esters and acids in the various products	15·48	3·19

The next step is to investigate such a distillation to ascertain if possible whether an equilibrium point is reached in the hydrolysis occurring in the still. For this purpose a quantity of low wines + feints + foreshot mixture containing 13·8 mgrms. acids and 4·1 mgrms. esters per 100 c.c. was boiled under a reflux condenser and the acids and ester content of the liquid determined at intervals, during this hydrolysis.

PERCENTAGE INCREASE OR DECREASE IN TOTAL ESTERS AND ACIDS IN LIQUID.

—	Esters.		Acids.	
	Increase per cent.	Decrease per cent.	Increase per cent.	Decrease per cent.
Effect on low wines and feints and foreshot of simple distillation as in practice in spirit still....	—	49·3	19·7	—
Effect on low wines and feints and foreshot of hydrolysis under reflux 1 hour	—	49·7	17·5	—
Effect on low wines + feints + foreshot of hydrolysis 1 hour followed by distillation	—	35·3	13·0	—
Effect on low wines + feints + foreshot of hydrolysis 1 hour under reflux in presence of excess NaOH and followed by distillation	—	23·0	14·3	—

The results are given in Table 11.

In a liquid in which the acids, esters, water and alcohol mixture is free to hydrolyse, irregular proportions of ester and acids are found but the total result appears to be to increase the acids and reduce the esters.

An important point is that after 2¼ hours hydrolysis had brought the acid and ester content to certain values, a further hydrolysis took place during the distillation of the hydrolysed liquid a very definite increase of acids being shown distributed over the fractions and in the residue, with a similar decrease in esters.

It will be noticed that the hydrolysed liquid used for the distillation was not the identical liquid used for the investigation of the process of hydrolysis. The use of a different liquid was rendered necessary by the consumption of the first liquid during the test, but the liquid used for the second test, the distillation test, was hydrolysed under exactly the same conditions as in the hydrolysis test.

TABLE 11.

To determine Equilibrium Point (if any) during Hydrolysis of Low Wines and Feints and Foreshot without Alkali.

Liquid.	Distillation Products.	Acids as mgrms. Acetic per 100 c.c.					Esters as mgrms. Ethyl Acetate per 100 c.c.				
		Mgrms. per 100 c.c. of Liquid.	Increase.	Decrease.	Per cent. Increase.	Per cent. Decrease.	Mgrms. per 100 c.c. of Liquid.	Increase.	Decrease.	Per cent. Increase.	Per cent. Decrease.
(A) Normal Low Wines and Feints and Foreshot	—	13·8	—	—	—	—	4·1	—	—	—	—
During Hydrolysis	After 15 mins.	13·0	—	0·8	After 2½ hours' Hydrolysis, Acids were same as at start, having increased and then decreased.		6·7	2·6	—	After 2½ hours' Hydrolysis the Esters increased and finally decreased slightly.	
	,, 30 ,,	12·5	—	1·3			4·6	0·5	—		
	,, 45 ,,	13·0	—	0·8			7·2	3·1	—		
	,, 1 hour	—	—	—			—	—	—		
	,, 1¼ hours	11·0	—	2·8			6·7	2·6	—		
	,, 1½ ,,	16·3	2·5	—			6·2	2·1	—		
	,, 1¾ ,,	14·9	1·1	—			7·0	2·0	—		
	,, 2 ,,	13·8	—	—			6·5	2·4	—		
	,, 2¼ ,,	13·4	—	0·4			0·34	2·24	—		
	,, 2½ ,,	13·8	—	—			3·88	—	0·22		
Hydrolysed Liquid.	At Start.	Total mgrms. in 200 c.c. = 21·1.			Per cent. of Total.		Total mgrms. in 200 c.c. = 21·8				
(B) Distillation after Hydrolysis 2½ hours	Foreshot ...	0·31	—	1·4	Increase of 3·8 per cent. of Acids due to Hydrolysis during Distillation.		1·75	9·8	—	Decrease of 17·8 per cent. due to Hydrolysis during Distillation.	
	Spirit ...	4·77	—	20·8			2·13	11·0	—		
	Feints ...	6·52	—	28·4			3·26	18·2	—		
	Tailings ...	1·02	—	4·5			1·40	7·7	—		
	Spent Lees ...	10·30	—	45·0			0·40	52·3	—		
		22·92	—	—			17·94	—	—		

NOTE.—(A) Was a sample of Low Wines, Feints and Foreshot.   (B) A dissimilar sample originally, but hydrolysed under identical conditions as for (A).

It is almost impossible to investigate one comparatively large quantity of such a liquid throughout a series of tests as the samples of liquid available for these tests are necessarily small, as they are obtained from practice with difficulty.

To make more clear the actual course of the hydrolysis, the experiment was repeated *in presence of NaOH in excess* to limit esterification but permit unlimited hydrolysis, the process of hydrolysis being examined at intervals.

The results are given below :—

TABLE 12.

Hydrolysis of Low Wines + Feints + Foreshot Mixture in Presence of Excess NaOH

Low wines + feints + foreshot mixture used contained :—

Acid as Acetic acid = 11·52 mgrms. per 100 c.c.
Esters as ethyl acetate = 4·04 mgrms. per 100 c.c.

Time.	Mgrms. per 100 c.c.			
	Total Acid.	Acid Found Increase	Total Esters per 100 c.c.	Esters Lost.
1. At start	11·52	—	4·04	—
2. At boiling point	13·71	2·19	1·05	2·99
3. After 15 min. boiling	13·71	2·19	1·05	2·99
4. ,, 30 ,, ,,	13·71	2·19	1·05	2·99
5. ,, 45 ,, ,,	13·71	2·19	1·05	2·99
6. ,, 1 hr. ,,	14·80	3·28	Nil.	4·04
7. ,, 1¼ ,, ,,	14·80	3·28	,,	4·04
8. ,, 1½ ,, ,,	—	—	—	—
9. ,, 1¾ ,, ,,	14·80	3·28	,,	4·04
10. ,, 2 ,, ,,	14·80	3·28	,,	4·04
11. ,, 2¼ ,, ,,	14·80	3·28	,,	4·04
12. ,, 2½ ,, ,,	14·80	3·28	,,	4·04

The esters hydrolysed in the above test amounted to 4·04 mgrms. per 100 c.c., while the acids formed amounted to 3·28 mgrms. per 100 c.c.

This amount of acid 3·28 mgrms. per 100 c.c. is equivalent to 4·01 mgrms. esters per 100 c.c. as compared with 4·04 mgrms. esters per 100 c.c. actually found in the experiment.

As in the case of the previous test, the liquid after hydrolysis was distilled.

The contents of esters and acids in the fractions of distillate were as under :—

TABLE 13.

Fraction.	Acid as Acetic.	Esters as Ethylacetate.
At start	14·80	Nil.
Foreshot	0·38	,,
Spirit	1·94	,,
Feints	2·94	,,
Tailings	0·33	,,
Spent Lees	2·18	,,
	7·77	Nil.

During hydrolysis the esters entirely disappeared and no more were formed during subsequent distillation as shown by the absence of esters in the distillate fractions, and this is to be expected from the fact that the acid has been neutralised by the added excess of caustic soda.

The distillate fractions, however, show small amounts of free acid as also does the " spent lees " residue.

The origin of this acid is not clear, but a possible explanation of its appearance is that as the alcohol distills over and the boiling point of the residue in the still rises, there is a liberation of acid which in the case of the volatile acid passes to the distillate fractions, while the non-volatile remains in the still.

It is probable that no further acid would be liberated during hydrolysis of the alcoholic liquid subsequent to the disappearance of all the esters, at the lower boiling point of the liquid during hydrolysis, but that when distillation begins there is a liberation of acid due to the rise in boiling point of the liquid in the still, this acid being carried over into the distillate.

Although this suggested explanation is mere surmise, at all events clearly esters are hydrolysed in the spirit still, and acids liberated which in their turn interact with the alcohol to produce further esters. So long as the conditions are those of hydrolysis under a reflux condenser, then hydrolysis will proceed until the equilibrium point is reached in the interaction of acids, alcohol, esters and water. When the conditions are, as in practice, those of distillation, the hydrolysis of esters will occur, and the production of esters will also take place, but an

equilibrium point will not in fact be reached, owing to the removal of varying amounts of the interacting substances to the distillate. The proportions of these substances removed will vary with the original content, the alcoholic strength, and the mechanical effect of distillation, the latter dependent upon the form of the head and lyne arm and the rate of distillation.

The above are not put forward as conclusive results, but more as general indications that in the hydrolysis of esters and liberation of acids in the low wines and feints and foreshot mixture in practice we have the basis of possible methods of alteration of the character of a pot still whisky.

By continuing the investigation of this acid-ester problem it may be possible to arrive at a more precise understanding of the process, and we may gain more knowledge of how it can be more definitely controlled ; and even if we can vary character at will, the ultimate goal is only thereby brought nearer by a small fraction of the total distance we have to advance, nevertheless the investigation will be worth while.

If I should have succeeded in arousing interest in this complicated problem sufficiently to attract any additional workers to this field of investigation, then I will be more than satisfied.

DISCUSSION.

A number of members joined in the discussion which followed and many questions were asked.

In reply to these questions Mr. Hastie stated that he appreciated the helpful attitude of the majority of brewers in the question of supplying their surplus yeast to the distiller, and expressed the hope that, if any brewers remained who still regarded the handling of their surplus yeast as not worth their careful consideration, that such would soon take the same view as the helpful majority.

In reply to questions regarding malt, Mr. Hastie stated that he regarded malt of high moisture content as detrimental to the process of fermentation, and he considered that prolonged storage up to 9–12 months would not prove harmful to fermentation, provided that the malt was not allowed to become slack.

He also agreed with a member who suggested that the handling and control of the fire heating of the still, and the shape of the still were of importance in determining the nature of the product.

He did not agree with the suggestion that character might be determined by local bacterial conditions, so far as to account for differing whisky characteristics in the distilleries in a district.

He agreed that the conventional analysis of whisky *alone* was of little value as a means of deciding its commercial value.

ABSTRACTS.

1. MATERIALS AND ANALYSIS.

Infection of Barley by " Ustilago Nuda " through Seed Inoculation. W. H. TISDALE and V. F. TAPKE (*Journ. Agric. Res.*, 1924, **29**, 263-284).—Floral infection has hitherto been recognised as the only means of infection of barley by the loose smut fungus *Ustilago nuda*. The investigations recorded in the present paper show that, in addition, seedling infection may also take place abundantly in certain varieties of barley when the dehusked seed is inoculated. The seedlings of both susceptible and resistant barleys are injured by the fungus and the surviving plants from inoculated seeds produce a high percentage of heads smutted with loose-smut. The work shows conclusively that seedling infection does occur in a number of varieties and the long-accepted theory that only floral infection occurs must therefore be abandoned. Many years after the discovery of the hot-water seed treatment by Jensen (this Journ., 1924, **30**, 1050), this method was recommended as being the only treatment that would control loose-smut of wheat and barley. Later, however, it was shown that formalin was of use as a counter measure, and, in 1923, Tisdale, Taylor and Griffiths obtained satisfactory control in the case of barley by treating the seed with formaldehyde and with chlorophol, an organic mercury