COMMUNICATIONS

THE MANUFACTURE OF SCOTCH GRAIN WHISKY

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Received 10th October, 1964

The special character of grain whisky distilled in Scotland depends on a number of factors. The conversion of the starch from cooked maize during the mashing process by the diastatic activity of barley malt is done in such a way that, besides a spectrum of soluble sugars, oligosaccharides and dextrins, some insoluble solids are also present. A significant proportion of initially unfermentable material becomes fermentable by a process of secondary conversion during the fermentation stage in the distillery. The fully fermented worts contain, besides ethanol, the higher alcohols, *N*-propanol, *N*-butanol, *iso*-butanol and *iso*-amyl alcohol, certain esters and aldehydes, and glycerol. After distillation in a Coffey still, the whisky contains higher alcohols, esters and aldehydes derived from the fermented worts. Lactic and acetic acids are also present as a result of fermentation by microbiological flora other than yeast. Among the changes in composition that take place during the maturing process are increases in the concentrations of esters, volatile acids and aldehydes.

Introduction

Scotch whiskies are of two main types, malt whiskies which are made by batch distillation of fermented worts prepared solely from barley malt, and grain whiskies which are made by the continuous distillation of fermented worts produced by malt enzymes acting on a substrate of cereal grains. As a general rule, grain whisky is distilled in the type of still designed by Aeneas Coffey in 1830[2] and is made by a process, the principles of which remain unchanged after 135 years. Both types are covered by the accepted definition of Scotch Whisky,[3] as having been "obtained by distillation in Scotland from a mash of cereal grain saccharified by the diastase of malt, and . . . matured in warehouse in cask for a period of at least three years." Both grain and malt whiskies from individual distilleries are drunk as singles, or, more commonly, combined to form blends.

The present review includes a description of the current methods used in the production of grain whisky in Scotland, together with observations on the composition of worts, the changes occurring, from both fermentation and secondary conversion, during the fermentation stage and some recent analyses of newly distilled and matured spirit.

Malt

The malt used in grain whisky production contributes to the character of the spirit obtained, but at the present state of knowledge, it is not possible to specify the components that are of significance for flavour and aroma. The second function of the malt is to serve as a source of enzymes to convert the starch derived from the cooked maize into fermentable sugars. The measurement of enzyme concentration can be done in a number of ways. Diastatic activity measured by the Lintner method[1] is made up of a combination of the effects of α-amylase and β-amylase. Preece[12,13] and also Preece & Shadaksharaswamy[14,15,16] have pointed out that although high β-amylase activity, as measured by Lintner determination, is usually accompanied by high α-amylase, this is not always so. The ratio of α- to β-amylase depends partly on the character of the barley and partly on the conditions of malting and kilning. As is shown by the results of the experiments set out graphically in Fig. 1, the measurement of diastatic activity by the Lintner method undoubtedly provides a useful method for assessing the effectiveness of malt in converting the starch of maize grist to fermentable sugars for grain whisky; nevertheless Thorne, Emerson, Olsen & Paterson[19] have expressed doubts as to whether Lintner values, even combined with α-amylase determinations, give a complete evaluation of malt for use in malt-grain grists.

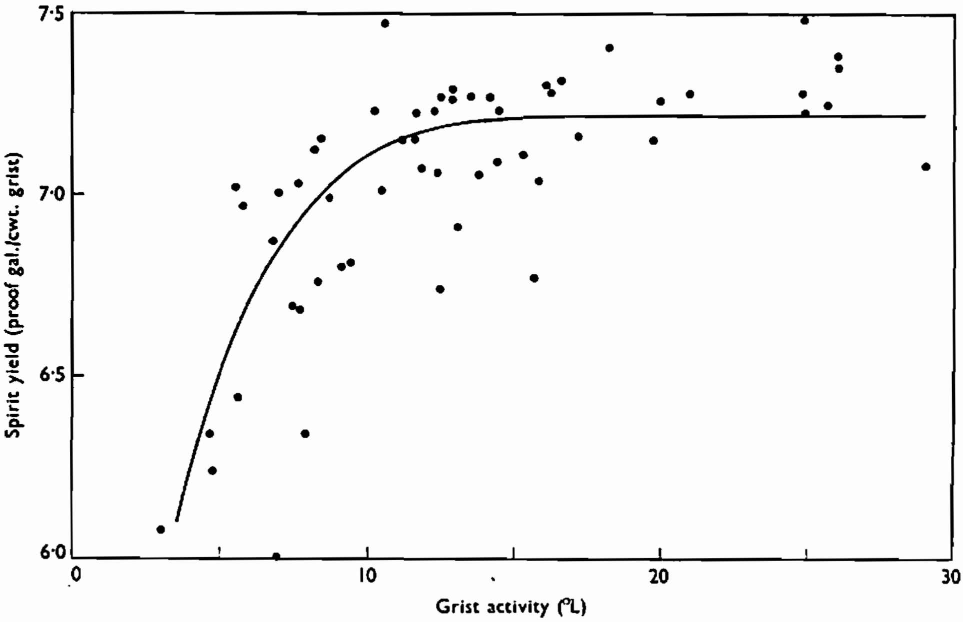

Fig. 1.—Diastatic activity of grist (°L.) and spirit yield (proof gallons per cwt. of grist).

Fig. 1 shows the spirit yields obtained after laboratory grinding, mashing and fermentation when varying proportions of different malts were used to convert a maize grist. The Lintner values of the malts varied from 47° to 113° and their α-amylase values from 7·3 to 10·4 units.[3] It can be seen that when the proportion of malt in the grist provided insufficient diastase to allow all the starch to be converted to fermentable sugar and a full spirit yield to be obtained, the yield could be correlated with the Lintner value of the grist. The use of determinations of α-amylase gave a less satisfactory correlation than the Lintner value. It can be seen, however, that even using °L. as a criterion of the value of malt in grain-whisky grist, a considerable scatter occurs. This underlines the point made by Thorne *et al.* that in the present state of knowledge the only sure test of malt is to examine its performance under the conditions existing in the distillery.

Milling and Cooking

Maize is the cereal which, during the last century and a quarter, has most commonly been employed as the main raw material for Scotch grain whisky. In order to bring the maize kernels into the most favourable physical state to allow the diastatic enzymes of the malt to act upon them and break down the starch into sugars fermentable by yeast, it is necessary, first, to grind them and then, by the action of heat, to burst the starch granules and the hemicellulose framework in which the granules are enclosed.

Maize is transferred from storage hoppers by a conveyor, freed from extraneous matter by being passed through a screen, weighed automatically, and then ground in an attrition mill. The fragmented maize grains, now termed "grist," are next treated by heat in a cooking process. A conventional cooker is a horizontal mild-steel cylindrical vessel capable of withstanding a working pressure up to 90 p.s.i.g. and fitted with stirring gear. The grist is mixed with "fourth water" (the origin of this is described later) and steam is injected. The pressure is maintained until the maize starch can be shown to be completely gelatinized. To achieve the desired results from cooking, adequate grinding and sufficient heat are required. Insufficient treatment leaves a proportion of the starch granules intact; excessive heating, however, appears to cause a degree of caramelization of the starch together with a production of pentose sugars from the maize husk, which is reflected in a reduction in the amount of spirit produced by yeast fermentation after the mashing process.

The Mashing Process

During the course of mashing, four operations occur: (*a*) diastatic enzymes are released from the ground malt; (*b*) the malt enzymes operate on the starch derived from the cooked grain; (*c*) the soluble sugars produced by the malt enzymes acting on the maize starch are washed out of the solid residues, and (*d*) the coarse solids, washed free from soluble material, are separated as draff. These overlapping operations are usually carried out as a batch process in a mash-tun. In mashing, ground malt is mixed with warm water and the suspension is run into the mash-tun. When the appropriate cooking time for the ground maize has elapsed, the steam pressure is released through an exhaust and the hot cooked maize is blown directly into the mash-tun. Sufficient cold water is added as it flows in order to achieve the desired temperature in the full mash-tun. In this operation it is essential for the cooked maize to be brought immediately into contact with the malt enzymes, because, should the now gelatinized starch be allowed to cool, the entire mash will solidify.

The mash of cooked maize and malt is well stirred and then allowed to settle. After some minutes, the valves controlling discharge ports under the perforated bottom of the mash-tun are opened and the worts draining through are pumped from the under-back, through a cooler into the fermenting vessel, which in Scotland is called a washback. A suspension of yeast is run simultaneously into the washback with the first of the worts. At the same time as the worts are allowed to drain through the mash-tun bottom, supernatant liquor, which, though cloudy, is substantially free from any of the larger particles of grain husks, is permitted to overflow by the removal of slides fitting into a vertical slit in the side of the mash-tun.

As soon as the bulk of the liquid has been removed from the mash-tun, the grain residues are stirred up again with sufficient "second water" to half-fill the vessel. When good mixing has been achieved, the liquid is drained off as before, passed through the cooler, and combined with the "first water" in the washback. The mixed first and second "waters" constitute the worts from which, when fermentation is terminated, the grain whisky is distilled.

The enzymic reactions that take place during mashing are complex. It is generally accepted that long-chain portions of the starch molecule are split by the action of α-amylase from the malt and that maltose units are released by the action of β-amylase. Considerable study has been given to the activity of limit-dextrinase,[5] by which residual portions of branched-chain molecules are thought to be broken down and rendered fermentable, but the evidence on this point is by no means clear. The general relationship between the enzyme activity of the malt and the degradation of the maize starch to a fermentable form has already been discussed (see Fig. 1) but in practice, Scottish grain distillers use a surplus of malt. This was observed and commented on by Valaer[20] as follows, "In order to enhance the flavour, barley malt is used in excess of the quantities required to completely convert all of the starch to sugar." Earlier (in 1908) Schidrowitz[17] had also stated, "In the manufacture of Scotch Grain whisky, a very much larger quantity of malt is used than is necessary for starch conversion, in order to enhance the flavour." The analysis of a typical grain wort at this stage of the process is shown in Table I.

It can be seen that the breakdown of cereal grains by the enzymes of malt during mashing produces a significant proportion of sugars other than maltose, together with soluble dextrins and nitrogenous compounds. The mixture of constituents in the grain worts thus produced explains in part the diversity of components present in the fermented wort and in the whisky which is

TABLE I

COMPOSITION OF GRAIN WORTS

Total soluble carbohydrate (as glucose)	9·00%
Insoluble solids	2·20
Fructose	0·13%
Glucose	0·29
Sucrose	0·28
Maltose	4·65
Maltotriose	0·96
Maltotetraose	0·15
Dextrin	2·54
Amino nitrogen (as leucine) ..	0·09%
Ash	0·27%
containing P_2O_5	0·09
K_2O	0·09
MgO	0·02
Thiamin	0·46 μg./ml.
Pyridoxin	0·61
Biotin	0·01
Inositol	236
Niacin	11·1
Pantothenate	0·71

distilled from it. But the mashing process for the production of grain whisky, unlike that in beer brewing, does not stop when the worts leave the mash-tun. Whereas the brewer boils his worts and thereby stops all further enzyme activity, the whisky distiller allows the residual malt enzymes to continue their activity through a proportion of the time when fermentation is simultaneously occurring. During this period a significant amount of the soluble dextrins is broken down; the quantity of additional fermentable material produced during this stage of the process contributes approximately 30% of the total spirit produced.[6]

After the "second water" has been run off the grain residues, a "third water" is run into the mash tun. When this is drained off, it is stored hot in a holding vessel, termed a brewing tank, and used as "second water" in the following mash. The grain husks, now termed draff, are again extracted, this time with fresh boiling water, which is also held as "fourth water" and used in the cooker with the maize grist for the following mash.

FERMENTATION

As soon as the first of the worts from the mash-tun passes through the cooler and reaches the washback, fermentation begins. Consequently, when the entire first and second worts comprising a complete mash have reached the washback, fermentation by the yeast is becoming fully active.

The processes involved in the fermentation of grain-whisky worts are complex. Whereas the main reaction will be the Embden-Meyerhof-Parnas scheme[8] by which, through a series of some twelve stages, a molecule of glucose or fructose is converted into two molecules of ethyl alcohol and two of carbon dioxide, several other reactions are known to be taking place simultaneously. More, or less, glycerol may be produced by one or other of the mechanisms described by Neuberg.[9] More significant than this, however, is the effect of yeast growth on the progress of the reaction, bringing into play further biochemical chains of reactions including those of the tricarboxylic-acid cycle of Krebs.[10]

During the fermentation of worts, the nitrogen metabolism of the actively growing yeast produces as a by-product of protein synthesis a mixture of higher alcohols, which constitute what is commonly called fusel oil. The breakdown of amino acids to alcohols by yeast in this way was first described by Ehrlich,[4] and the validity of the mechanism he suggested has recently been demonstrated by Sentheshenmuganathan & Elsden.[18] An alternative pathway for the production of higher alcohols is that described by Ingraham & Guymon[7] who suggested that yeast could convert carbohydrate into higher alcohols while simultaneously producing amino acids for growth.

The complexity of the fermentation of grain wort possesses further facets. First, the sugars to be fermented are not restricted to glucose and fructose but include the disaccharides, maltose and sucrose, the latter of which during the course of its breakdown by invertase may give rise to trisaccharides. In addition, there are less readily fermentable trisaccharides and oligosaccharides. Secondly, as has already been mentioned, the malt enzymes are releasing further fermentable material by attacking soluble dextrins during the course of the fermentation of sugars initially present. A third factor, complicating still further the progress of these interlocking aspects of yeast fermentation, is the intervention of lactobacilli and *Leuconostoc*, which form part of the natural environment of a distillery and of streptococci, which are often associated with the yeast; these organisms convert a small part of the wort sugars into lactic acid and other minor constituents, characteristic of fermented grain-distillery wash. Table II shows some of the components of wash at the completion of fermentation.

TABLE II

GRAIN WORTS AFTER FERMENTATION

Ethanol..	4·8%
Volatile acids (as acetic acid)..	25·9 g./100 litres
Lactic acid	129·0
n-Propanol	3·4
n-Butanol	1·6
*iso*Butanol	5·0
iso-Amyl alcohol	13·5
Glycerol	250·0
Volatile esters (as ethyl acetate)	5·28
Aldehydes (as acetaldehyde)..	0·78
Total soluble solids	1·00%
—residual carbohydrate ..	0·27
Total insoluble solids	1·60
Yeast cells	150 × 10^6/ml.
pH	3·7

Yeasts vary in their ability to ferment different sugars. Different strains perform differently under varying conditions of temperature, pH, sugar concentration and the presence or absence of components other than sugars in the medium. The strain of yeast employed in grain whisky distilleries is selected for its capacity to induce a vigorous fermentation and to deal with trisaccharides and more complex oligosaccharides,[18] and it may either be propagated in the distillery or obtained from a yeast factory.

During the course of the fermentation, the sugars in the wort are utilized not simultaneously but in sequence. Glucose, fructose and sucrose are very quickly fermented. The main sugar, maltose, is fermented next while the latter half of the fermentation time is taken up by the very much slower fermentation of maltotriose, maltotetraose and the breakdown products of soluble dextrins (Fig. 2). It is during this last part of the fermentation process that the bacterial flora mainly develops. These micro-organisms may produce lactic and other acids and other substances in very low concentrations, which can influence the character of the whisky which is subsequently distilled.

DISTILLATION

A characteristic feature in the manufacture of grain whisky in Scotland has been the use of the "patent" still developed by Aeneas Coffey[2] in 1830. This apparatus is still in use in Scotland. Although a good deal of further information is now available about the details of its operation and, as is described below, significant advances have been made in its operation and control, the following account of its operation given at the Royal Commission in 1908 is still substantially valid.

"Now coming to the patent still distillation it was pointed out that that differed in no essential respects from the pot still—in fact it is only a small series of pot stills. . . . There were two columns in the patent still, one the analyser, and the other the rectifier. If I may trace it out from the wort tank, the wort containing the solid matter, the water, the ethyl alcohol, and all the bye-products, was passed, first of all, through the rectifier in a pipe. It entered cold. It passed backwards and forwards through the rectifier, being heated as I shall afterwards point out, by that which was coming from the analyser, so that the wort entered at the top cold and passed out at the bottom nearly boiling, and came up by a pipe and entered at the top of the analyser. Inside the analyser were a series of trays down which the contents of the wash passed. Instead of heating this whole

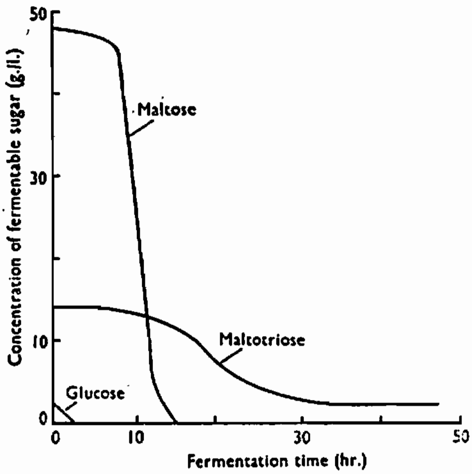

Fig. 2.—Utilization of sugars during the course of grain whisky wort fermentation.

structure by a fire or by jacketing it, you passed live steam into it. The live steam entering at the bottom gradually heated up—it affected the top first where it met the incoming wash, which was nearly boiling at the time, and the steam carried off with it all the volatile matters; it carried off the ethyl alcohol, it carried off all the bye-products, the higher alcohols, the ethers, and all the others, and then passed off into the bottom of the rectifier. Now, in the rectifier, products of different boiling points were separated; there was again a series of trays. Now, as I pointed out, the wash entering at the top was cold, and at the bottom was hot. There was a graduated temperature from the top to the bottom of the rectifier, and at the various stages the different products separated out, and at a point where the majority of the product was ethyl alcohol, at that point the spirit pipe was put and drew off the spirit, but as it was pointed out in evidence, in that form of still, as in every form of still, you cannot alter the laws of nature. You cannot separate by fractional distillation these various boiling point bodies one from the other. It is a more perfect still as was given in evidence than the old pot still, but a still can only partially fractionate, and the evidence . . . was that the spirit drawn off from the rectifier at the point where its chief product was ethyl alcohol, contained every body which was present in pot still whiskey, but the proportion, owing to the more perfect apparatus, was less than the proportion of the impurities presented in the product of the pot still."

No fundamental change has been made in the construction and operation of the still during the last half century. The principal improvements introduced in grain whisky distilleries have been instruments to control automatically the flow of fermented wash into the still and an exact automatic control of the strength of the whisky drawn off at the spirit plate. The way in which the precise control of still operation can affect the quality of the whisky produced is shown in Fig. 3. It can clearly be seen that if the control of the distillation, *i.e.*, the flow of wash and steam, is changed in one direction, the concentration of higher alcohols and other congenerics which accompany them will change although the spirit strength may remain the same. If the balance of the still is changed in the other direction, the proportion of congenerics will fall. Both changes will obviously affect the character of the whisky.

Fig. 4 is a gas chromatogram showing the presence of a number of the volatile components in newly made grain whisky. For comparison, a chromatogram of "neutral spirit," produced by a distillation process designed to purify the alcohol to the maximum degree, is also shown. The composition of new-make grain whisky as determined by analysis is shown in Table III.

In order to obtain whisky of good quality it is important to operate the still in such a

TABLE III

Typical Analysis of Scotch Grain Whisky

Constituent	New make	Matured
	% (by vol.) at 20° C.	
Ethyl alcohol	94·17 (65·1° Over Proof)	40·18 (70·2 Proof)
	mg./dl. Ap*	
Higher alcohols	118†	115
Esters (as ethyl acetate) ..	12·3	21·7
Volatile acid (as acetic acid) ..	1·1	23·3
Aldehydes (as acetaldehyde) ..	2·2‡	7·6

* Mg. per decilitre alcohol pure. This is equivalent to g. per 100 litre absolute alcohol.

	p.p.m.
† Includes *n*-propanol	500
and *iso*-butanol	760
‡ Includes *iso*butyraldehyde ..	0·5
and acetal	7·3

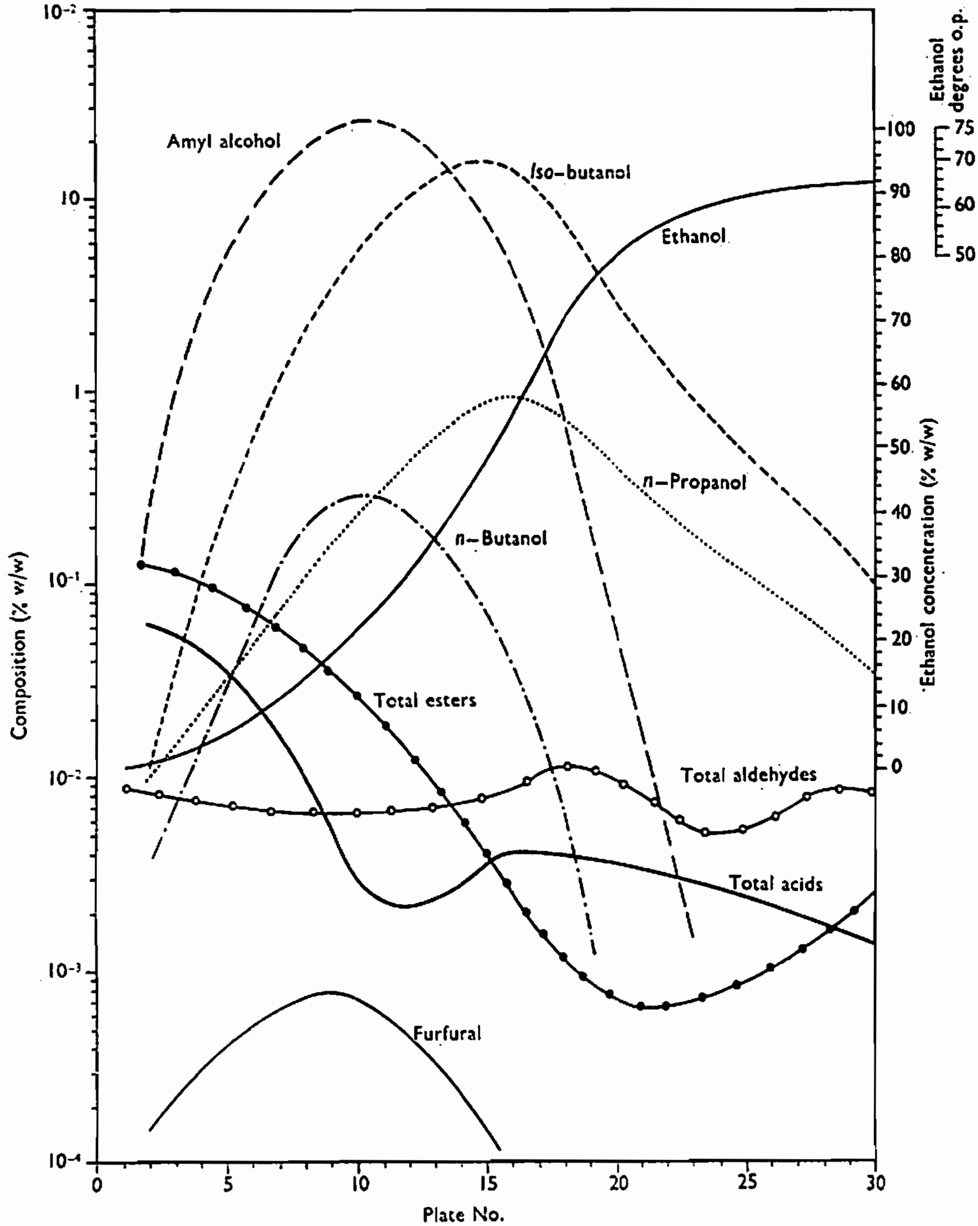

Fig. 3.—Changes in vapour composition at different plates in the Coffey still rectifier.

manner as to ensure that the alcohol concentration of the spirit drawn off at the spirit plate shall not exceed 94·17% by volume at 20° C. This is usually achieved by the ingenious method of maintaining the "bend temperature" at the spirit plate at 117–118° F. This temperature depends on (*a*) the rate at which the wash, which as it travels through the bends (*i.e.*, the curved tubes between one rectifier plate and the next) is acting as a condenser for the rectifier column, is fed to the still and (*b*) on the amount of steam being introduced into the analyser column. It also indicates the

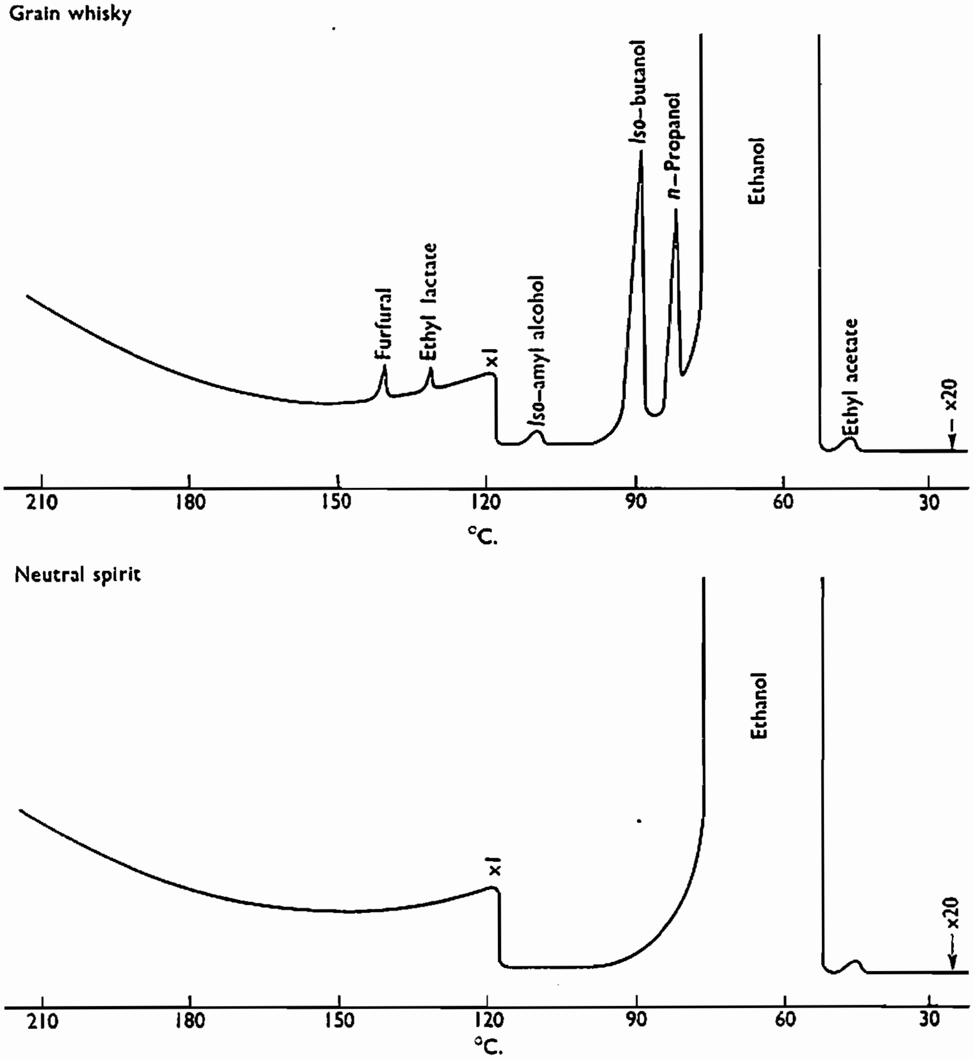

Fig. 4.—Gas chromatograms of grain whisky and of neutral spirit.

boiling point, and hence the spirit concentration, in the plate at which the bend temperature is recorded. If the temperature is allowed to rise and the concentration of ethyl alcohol to increase, the proportion of congenerics will drop and good whisky will not be obtained. On the other hand, if the bend temperature becomes too low, not only will the strength of the spirit drawn off fall but its quality will be adversely affected.

The aqueous part of the wash, which contains yeast residues and a proportion of grain solids carried over from the mash-tun as well as non-volatile dissolved material, finds its way as spent wash through a pipe at the base of the analyser column. In a similar way, volatile impurities which, although they have been carried over by the steam into the rectifier column, only boil at high temperature, tend to condense as hot-feints at the base of the rectifier. While the still is running, the condensed hot feints are pumped back into the top of the analyser to be redistilled. At the end of the distillation, however, the feints which have been collected from the rectifier column are allowed to cool. Oily impurities, the main constituent of which is amyl alcohol, separate on the surface and are removed as fusel oil. The appropriate balance of all these operations is essential for the production of whisky of the desired character.

Disposal of Draff and Dreg

The grain husks remaining in the mash-tun at the end of the mashing process and from which all the material solubilized by the malt enzymes has been removed by successive washing by the four "waters," are recovered and may be sold undried to farmers as draff, or wet grain. Alternatively, the draff may be dried for sale to compounders as dried grains.

The spent wash emerging from the base of the analyser column is made up of insoluble solids, including grain fragments and yeast, together with soluble but non-volatile substances. The more important of these are oligosaccharides and dextrins unfermentable by yeast, and glycerol produced during the course of fermentation. A proportion of fat, partly derived from the grain but possibly, partly produced during the growth of the yeast, is also present. In some distilleries the spent wash is disposed of by a simple settling process to separate the solids, termed dreg, which are subsequently roughly filtered and sold un-dried, following which the aqueous fraction is discarded. Under modern conditions, however, the solids are first separated from the spent wash by means of a centrifuge or vibrating or wedge-wire screen and the liquid is then concentrated to a syrup by multiple-effect evaporation. At this point, the solids removed from the spent wash may, if desired, be combined with the syrup which is then dried to a powder. In some distilleries the separated solids are dried by themselves and marketed as light grains and the dried syrup is marketed as solubles. In one modern Scottish plant, quadruple-effect evaporation is followed by spray drying to produce a concentrate from the spent wash.

The dried distillers' solubles form a useful feed for poultry and animal nutrition. Recent trials reported by Boruff, Luthy & Van Lanen[20] showed that when used as a supplementary poultry feed it gave equal or better growth as replacement for brewer's yeast, dried skim milk and fish meal in a practical broiler diet. Besides containing protein and B-vitamins, as shown in Table IV, evidence of the presence of one or more unidentified growth factors was obtained when the dried concentrate was added to a semi-purified broiler ration.

TABLE IV

Chemical and Vitamin Analysis of Distillers' Feeds from a Scottish Grain Distillery

	Dried grains (light grains)	Dried solubles	Dried dreg (grains with solubles)
Moisture, %	5–7	5–7	5–7
Protein, %	16–20	28–30	35–45
Fat, %	6–9	8–10	12–16
Fibre, %	17	3	9
Ash, %	3	5	4–5
Riboflavin, mg./kg.	1·2	19·5	10·0
Niacin, mg./kg.	70	164	112
Pantothenic acid, mg./kg.	5	19	12

The Maturing Process

Freshly-distilled grain whisky possesses a flavour and aroma that can be variously described as "rough" or "fiery." In order to bring about the mellowing process which occurs when it is mature or aged, the whisky is diluted with sufficient water to reduce its alcohol concentration from 94·17% by volume at 20° C., *i.e.*, 65·1° Over Proof, to 20 Over Proof or to 11 Over Proof and it is filled into casks. The casks used in Scotland are of three main types. These are, first, butts which have been used previously to transport sherry from Spain to Great Britain. These casks have a capacity of 110 Imperial gallons. The second type of cask also has a capacity of 110 gallons, but it is newly made in Scotland, invariably from American oak. The interior is lightly charred over a fire of oak shavings and before the cask is used it receives a treatment with sherry. These casks are called puncheons. The third main type of cask in which it has been found that maturing takes place satisfactorily has a capacity of 56 Imperial gallons. This is called a hogshead. Before they are used for the first time, some hogsheads are also treated with sherry.

Although, as can be seen from Table III, the changes that are known to occur in the composition of grain whisky during the maturing process include increases in the concentrations of esters, volatile acids and aldehydes, and tannins derived from the wood of the cask, it is clear that there is still much more to be discovered about the process. Gas chromatography and other newer methods of analytical chemistry are being applied to investigate the trace substances which it is now clear exert an important influence on whisky character. At present, however, these methods have not yet achieved a sensitivity equal to that of the human senses.

Acknowledgements.—In writing this paper, I have drawn on the work of a number of people among whom I should like to thank especially my own colleagues, Dr A. R. Chambers, Dr R. E. B. Duncan, Mr D. H. Kenny and Dr K. G. MacKenzie; I should also like to thank Mr W. Dunnett of the General Laboratory. I am also grateful to the Directors of the Distillers Company Ltd. for permission to publish this paper.

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