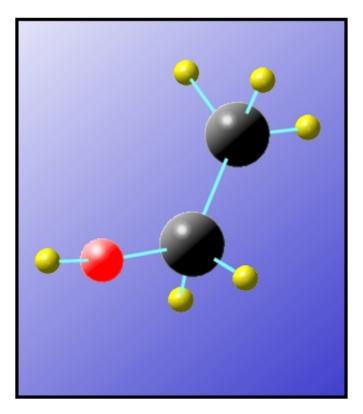
THE DISTILLATION OF ALCOHOL A Professional Guide for Amateur Distillers



by John Stone & Michael Nixon

Foreword

Making pure ethyl alcohol at home could be a satisfying and profitable hobby for those who live in countries where it is legal to do so. Do-it-yourself types who currently enjoy making beer or wine would find it particularly interesting because it is a logical extension of both these activities. There is the same fermentation stage where sugar is turned into alcohol, but instead of drinking the brew we subject it to a very rigorous purification process. This process is fractional distillation, a scientific procedure which can be guaranteed to produce a perfect product every time --- a crystal clear alcohol of almost pharmaceutical quality.

The pure alcohol is then diluted with water to 40% and used as such (vodka), or flavoured with exotic herbs such as juniper berries, cardamom, orris root, coriander and other botanicals to give London Dry Gin. Or fruit is steeped in the alcohol to make a delicious after-dinner liqueur.

This is not a hobby for everyone, but what hobby is? In the first place you would only wish to become involved if you particularly liked the beverages which are made from gin and vodka, e.g. a martini, a gin-andtonic, a Bloody Mary, or a liqueur. Secondly, you should enjoy the challenge of constructing a scientific apparatus which involves a little plumbing and a little electrical work.

The satisfactions you receive will include the knowledge that you have made something which is exceptionally pure, so pure in fact that no headaches or hangovers will ever result from drinking it. And finally there will be the pleasure derived from making a beverage which is less than onetenth the cost of the commercial product. Copies of the previous book in this series* were sent for comment to the Customs & Excise Branch of Revenue Canada in Ottawa and to the Bureau of Alcohol, Tobacco and Firearms (BATF) in the United States. Both authorities agreed that it is not illegal to sell or purchase a book which deals with amateur distillation but that it <u>is</u> illegal to actually engage in it without a license. No doubt many other countries around the world would react similarly.

The reasoning behind this law remains obscure. Distillation is simply a purification process which not only doesn't make alcohol but is incapable of making it. Alcohol is made by fermentation, not by distillation, so it might be expected that fermentation would be the process subject to control. This is not so however ---- amateur beer- and wine-makers are free to make as much alcohol as they wish for their own use. It is abundantly clear, therefore, that the law is based upon a completely false premise.

Individuals in New Zealand, Italy and several other countries already enjoy the freedom to distil alcohol at home for their own use. It is hoped that the publication of this book will eventually make it possible for amateurs in all countries to make their own vodka, gin and other spirits in the same manner that they now make beer and wine.

* Footnote: "Making Gin & Vodka" by John Stone. Published in 1997 by Saguenay International.

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Table of Contents

Page No.

1.	Introduction	6
2.	Alcoholic Beverages Beer and wine Distillation what is it? Simple distillation pot stills Whisky, brandy, rum, etc. Fractional distillation Gin & vodka Health & Safety Headaches & hangovers	9
3.	The Question Of Legality	17
4.	Equipment Fermenter Beer-stripper Fractional distillation apparatus The boiler The column The still-head The flavouring still	21
5.	Fermentation Principles Procedure	37
6.	Distillation Principles Procedures Beer-stripping Fractional distillation Collection rate Yield of pure alcohol	41
7.	Flavouring Procedure	53
8.	Summary of procedures	57
9.	Costs & Economics	60
10.	Appendices I. Conversion factors II. Activated charcoal III. Distillation - How it Works IV. Diode heater control	65 66 67 72

Introduction

Innumerable books are available on the home production of beer and wine but very few on the production of distilled spirits at the small scale required by hobbyists. This book has been written in an attempt to rectify such an anomalous situation. The emphasis is on the production of vodka and gin, and there is a reason for this. It is actually simpler to produce the very pure alcohol required by these two beverages than it is to make a spirit of lesser purity such as whisky. The explanation as to why it is simpler will become apparent in the next chapter. This emphasis on complete purity should not be taken to mean that whisky, rum, brandy, etc. are excluded from the list of alcoholic drinks which could be produced — after all, every bottle in the liquor cabinet contains alcohol, the only differences between them being flavour and alcohol concentration. The emphasis on vodka and gin simply means that the primary consideration in this publication is the production of pure ethyl alcohol — C_2H_5OH .

The book should appeal to two groups of readers: 1) those who live in countries where it is legal to distil alcohol for one's own use, e.g. New Zealand and Italy, and 2) the rest of the world, including North America and most of Europe, where the irrational and arbitrary law respecting distillation by amateurs needs to be challenged.

The first group will find complete details of the equipment and procedures required to ferment cane sugar to a crude 'beer' and then fractionally distil it to remove all the impurities, thereby producing a pharmaceutically pure alcohol. Instructions follow for flavouring the alcohol with juniper berries and other botanicals to give the well-known bouquet of London Dry Gin.

The second group can use the same detailed information in its campaign to have the law changed. Such a campaign will only succeed if it is based upon a thorough knowledge of the subject matter, because those who embark upon it will soon realize that legislators and officials in government are completely muddled about distillation --- with what it is and what it isn't.

This book, therefore, must not be seen in North America and elsewhere as any sort of incitement to break the law. Not at all. It is an attempt to clarify in the minds of the general public, and in governments, the misconceptions about a simple purification process which have become rooted in society as a result of centuries of mischievous brain-washing. Armed with the facts, the public can then embark upon the formidable task of bringing common sense to bear upon the problem.

A whole chapter will be devoted to this question of legality since it is highly important for everyone to know exactly where they stand and to be comfortable with what they are doing. It is hoped that legislators and law enforcement agencies themselves will read this chapter and possibly one or two others, think about it, and be prepared to be receptive when law reformers come knocking at their doors.

The units of measurement to use present a problem. Most of Europe uses the metric system whereas North America, particularly the U.S., is largely non-metric. In this book, therefore, we have adopted a hybrid system in which most volumes, weights, temperatures and pressures are in metric units while most dimensions, e.g. pipe diameters, are given in inches. For convenience, a table of conversion factors from one system to the other is given in Appendix I.

There is quite a bit of repetition in several of the chapters. Thus, when describing the equipment it has been necessary to describe to some extent just how it is used, even though this is dealt with at length in the chapters which deal with the procedures involved in fermentation and distillation. We make no apologies for such overlap since it helps to make the various chapters self-sufficient.

Repetition of the point that distillation is simply a purification process can be excused on the grounds that repetition is not a bad thing if we wish to clear away the misinformation planted in people's minds over the years by zealots of one sort or another.

In writing this description of small-scale distillation for amateurs it was difficult to decide on an appropriate amount of detail to provide. Distillation, even fractional distillation, is really a very simple process and it might have been sufficient simply to provide a bare outline of how to proceed. It was decided, however, that a knowledge of *why* something works is as interesting to the enquiring mind as knowing *how*. Furthermore, it can be very useful to know the underlying principles involved in a process if something doesn't work out exactly as expected the first time you try it. It then becomes possible to solve the problem through knowledge rather than by trial-and-error.

Before getting down to these details of fermentation and distillation a few general observations will be made in the next chapter on the subject of alcoholic beverages *per se* because they cover a very wide range of products from wines and beers to whiskies, rum, brandy, gin, etc. Comparisons will be drawn between these various products, mentioning in particular that highly purified alcohol in the form of gin and vodka is considerably less harmful to health than beer or wine, notwithstanding widely held beliefs to the contrary.

Alcoholic Beverages

All alcoholic beverages are made by fermenting a sugar solution with yeast, a process which converts the sugar to carbon dioxide and ethyl alcohol. Usually, one does not start with a pure sugar but with fruit juices for wine, the starch in grains for beer and whisky, molasses for rum, etc. Over the centuries trial and error have shown that a bewildering variety of sugar sources can be exploited in this manner, even such an unlikely substance as milk being usable because of the sugar lactose it contains. Regardless of the sugar source the alcohol is the same.

In addition to the variations imposed by the source of sugar, the yeasts themselves and the conditions under which they are used also make their contribution to the character of the final product. This is because yeasts produce small quantities of other substances in addition to the main product --- ethyl alcohol. It is no wonder, therefore, that the flavour, colour, aroma and general quality of fermented beverages vary so widely and that a great deal of skill and experience is required in order to produce an acceptable beverage.

No alcoholic beverage (with the possible exception of certain vodkas) consists simply of alcohol and water with no other constituent present. If it did it would be colourless, odourless and tasteless. And rather boring unless you mixed it with something which had a flavour, e.g. vermouth, tomato juice, orange juice, etc.

The colour, aroma, and flavour of beers, wines and spirits are due to the other components present, components which collectively are known as "congeners". Many of these congeners are relatively harmless but there are always a few produced during fermentation, any fermentation, which are actually poisonous. Methanol (rubbing alcohol) is one of them. Surprisingly enough to those of us who have been brought up to believe the opposite, it is the congeners and not the alcohol which are responsible for headaches and hangovers following over-indulgence. More will be said about this interesting and little-known fact towards the end of the chapter.

Beer and wine

Alcoholic beverages can be divided into two broad categories according to whether or not there is a distillation stage following fermentation. Beer and wine fall into the non-distilled category whereas whisky, rum, brandy, gin, etc. have all been distilled. The latter are often referred to as "spirits" or "hard liquor".

Simple distillation removes some of the more noxious congeners produced by fermentation. Because beer and wine do not receive any such purification treatment it is necessary to live with whatever mixture of chemicals the fermentation has produced. This means in practice that beerand wine-making must be carried out extremely carefully for, if they are not, the resulting brew could be very unpalatable. Beer- and wine-making are highly skilled occupations, more akin to gourmet cooking than to science, and involve many subtleties and many opportunities for error. Which explains why there is such a wide range of qualities and prices of wines and why amateurs have such difficulty in producing a really first-class product.

Distillation --- what is it?

Distillation is simply the heating of a liquid to the boiling point followed by condensing the vapours on a cold surface. To remove the hardness from water it can be boiled in a kettle and the steam which is produced condensed against a cold surface to give a pure water free of minerals and all other types of impurity. The calcium and magnesium salts which constitute the hardness remain behind in the kettle. Nature carries out her own distillation in the form of rain --- the sun evaporates water from the surface of lakes and oceans leaving salt and impurities behind. Clouds form, condense, and a close approximation to distilled water falls to earth.

So distillation is not a mysterious subject, nor is it threatening. It is as commonplace as a rain-shower or a tea-kettle boiling and causing condensation on a nearby window. And as innocuous.

As you can imagine, the actual practice of distillation is a little more complicated than this and later chapters will provide an exact description of the equipment required and the procedures involved in making one particular type of high-purity spirits, i.e. gin and vodka. There are actually two different types of still, the choice of which to use depending on the level of purity required in the product. Whisky uses one type, rather simple in design since only a modest level of purity is required. Gin and vodka production on the other hand requires a more sophisticated type of still because a very high level of purity is desired. A brief description of the two types will be provided in this chapter dealing with beverages because it is quite important for the reader to appreciate the differences.

Simple distillation

As mentioned before, the fermentation of sugars derived from grapes, barley, corn, potatoes, molasses, milk or any other source produces a wide variety of chemicals, the major one being ethyl alcohol (ethanol). Minor constituents will be methyl, propyl, butyl and amyl alcohols, aldehydes, ketones, esters and a host of other organic compounds in small amounts. These minor constituents are the congeners and the amount of each will determine the flavour, bouquet and colour of a particular beverage. They are also responsible for unpleasant side-effects such as headaches and hangovers since many of them are very poisonous.

When such a mixture is distilled, the first vapours to come over will be rich in the more volatile components such as methanol and acetone. This first fraction is referred to as the "heads". There is no sharp separation so, long before the heads are completely exhausted, the ethanol begins to appear and could be collected, even though it would be somewhat contaminated with heads. Later, when ethanol production is tapering off, the "tails" begin to emerge. These are the least volatile components of the mixture, the propyl, butyl and amyl alcohols known collectively as "fusel" oils. Thus, in a simple distillation using a pot still there are three main fractions --- the heads, the tails, and the middle fraction of mainly ethanol contaminated with a little heads and tails, the amount of each depending on where the cut-off is made.

Whisky, brandy, rum, etc.

The distiller of these products uses a simple pot still for batch distillation and this, as mentioned above, effects only a crude separation of the fermentation broth into heads, tails, and middle fraction. The skill in making a palatable whisky consists of: a) fermenting the mash under conditions which give rise to a certain mixture of chemicals followed by b) distilling the mixture and discarding a portion of the heads and a portion of the tails. The middle fraction, consisting chiefly of ethanol, will also contain the retained portion of heads and tails. It is these heads and tails which impart the characteristic flavour and aroma. At this point there is no colour. Colour is imparted by storing the spirits in oak barrels for a number of years, a process which also modifies the chemical make-up of the whisky to give the unique characteristics of a particular brand.

Clearly, the manufacture of a palatable whisky is a highly skilled operation since it involves the production of a complex but controlled mixture of chemicals followed by the selective removal of a certain proportion of them. This makes it easy to understand why the moonshine produced in the hills of Kentucky during prohibition days was such a rough and even dangerous product. The fermentation carried out under less than ideal conditions would have produced a witches brew of chemicals while the crude pot stills used without proper controls would have undoubtedly left behind a number of exceedingly unpleasant constituents. The same problems and dangers would face the amateur whisky-maker today without proper guidance.

Fractional distillation

As mentioned above, simple distillation of a mixture of liquids does not produce a clear-cut separation of the various components. If such a separation is required it is necessary to resort to the use of a fractionating column. The theory and practice of this will be described in detail in a later chapter but a few words will be said about it here. The procedure involves the use of a vertical column attached to the top of the boiler which is packed with inert particles such as short lengths of glass tubing known as Raschig rings, ceramic 'saddles', wire gauze, or in fact any non-reactive material with a large surface area.

The vapours from the boiling liquid pass up the column, are condensed to a liquid at the top, and run back down through the packing in the column. This counter-current flow of vapour up and liquid down has the effect of producing a series of mini distillations at the surface of each piece of glass or metal in the column. It is equivalent to carrying out a simple distillation in a pot still and then re-distilling the product over and over again. The final result is an almost perfect separation of the mixture into its various components, allowing each one to be drawn off in sequence from the top of the column in the order of its boiling point. Thus, the most highly volatile components emerge first while the least volatile components emerge last.

Gin and vodka

In sharp contrast to all other alcoholic beverages, gin and vodka are made from almost pure alcohol, i.e. alcohol from which all the heads and tails have been removed. This, when diluted with water to 40%, is vodka. To make gin, a flavouring essence based on juniper berries is added.

Using a pure alcohol as the basis for a beverage has many advantages in terms of the ease of manufacture, the raw materials which can be used, and the quality of the product.

In terms of ease of manufacture, the production of pure alcohol is a science, not an art, and results therefore can be guaranteed if the proper equipment is used and procedures followed. There are no subtleties involved such as quality of grapes or the type of yeast used. One hardly even needs to worry about hygiene; just add baker's yeast to any solution of sugar to produce a "beer" and then remove all the extraneous, noxious materials by fractional distillation to leave a pure alcohol. What could be simpler?

By comparison, the production of a fine wine, beer or whisky is much more difficult. As we have said before, the quality of these beverages depends upon the presence of compounds <u>other</u> than ethyl alcohol (the congeners) and it is very difficult to ensure that these are present in exactly the right amounts and the right proportions. No such considerations apply in the case of gin and vodka. The "beer" produced by adding baker's yeast to cane sugar would be completely undrinkable by all but the most hardy, but fractional distillation will rid the mixture of all the undesirable compounds to leave a crystal-clear, unadulterated ethyl alcohol. Even the dregs from glasses after a party could be thrown into the pot and out will come the purest alcohol.

The result will be the same every time, with no variations and no failures. The only art involved will be in the preparation of the flavouring

essence from juniper berries and other botanicals, and this is simply a matter of personal taste and preference.

It is also worth mentioning here that liqueurs can be made by steeping fruit in alcohol, or by using ready-made flavouring essences available from stores selling wine- and beer-makers' equipment and supplies. Flavoring essences for the preparation of light and dark rum, brandy, whisky, etc. are also available from the same source.

As a final word of encouragement, depending on the price of sugar, the cost of all the ingredients required to make a litre of 40% vodka or gin will be about one dollar (US).

Health and Safety

One of the claims made by certain people when the subject of amateur distillation of alcohol is raised is that, if permitted, people would be liable to poison themselves. Specifically, there would be the danger of going blind. Examples of this having happened to individuals or even whole communities in various countries around the world are cited. But when specifics are asked for it is all very reminiscent of the Indian Rope Trick ---everyone has heard about it but no-one has actually seen it.

The fact of the matter is that it would be virtually impossible to poison oneself by drinking home-distilled spirits. As mentioned before, distillation does not produce anything so there can be nothing in a distilled spirit which was not already in the original beer. How can one convert a harmless beverage into a lethal one simply by boiling it? Of course, beer does contain poisons --- methanol and fusel oils for example --- but their only harmful effect is to produce the headaches and hangovers which people experience when they over-indulge.

Distillation separates these congeners, permitting them to be discarded. They smell like paint remover. So, to poison oneself, it would be necessary to remove the congeners from the beer by distillation, pour the purified alcohol down the drain and then, ignoring the pungent smell and sickening taste, drink the paint remover. This is about as likely as plucking a chicken, throwing away the meat and eating the feathers. It strains credulity.

Headaches and hangovers

Headaches and hangovers are well known consequences of overindulgence in alcohol, but what is less well known is that these unpleasant side-effects are largely due to the impurities, the congeners, and not to the alcohol *per se*.

This interesting fact will be confirmed by many people who habitually drink gin or vodka rather than pot-distilled spirits such as rye, bourbon, scotch, rum or even wine and beer. More objective proof that the congeners and not the alcohol are the bad actors can be found in scientific literature. Numerous studies have been made and all investigators find the same thing, i.e. that the symptoms of hangover --- headache, halitosis, gastric irritation, fatigue and dizziness --- were far more severe when the same amount of alcohol was consumed in the form of whisky than in the form of vodka. When you think about it, this is hardly surprising considering the poisonous nature of some congeners.

As an example of such studies, in one clinical investigation 33 men and 35 women were each given 2 ounces of either whisky or vodka on separate occasions. The incidence of after-effects in the group following a single drink of 2 ounces of whisky was halitosis 27%, gastric irritation 25%, headache 9%, dizziness 7% and fatigue 6%. These symptoms persisted during the following day. After the same amount of vodka, temporary headache and gastric irritation were observed in only 2% of the subjects while there were no complaints of halitosis, dizziness or fatigue in any of the cases. It should be noted that all the subjects in this trial were light social drinkers.

The effects described were produced by a commercial whisky in which the congeners occurred to the extent of about 3%. As part of the study the congeners were separated from the whisky and given to the subjects in the absence of alcohol. The effect was the same as when the whisky itself was imbibed, proving that the congeners and not the alcohol were responsible for the adverse reactions. The chief culprit among the congeners was considered to be one of the fusel oils --- amyl alcohol.

These results are not really definitive -- for one thing the size of the sample was too small -- but even without such a trial it is not difficult to believe that drinking such things as methanol and fusel oils, even in small

amounts, will be bad for you. If it were a different poison, e.g. arsenic, it would not be surprising if a 3% solution in water gave you an upset tummy.

One of the conclusions to be drawn from such studies is that whisky production should be avoided by amateurs. Not only is it difficult to produce a blend of alcohol and congeners to give a palatable beverage but, additionally, the consequences of error could be unpleasant. Far more sensibly, remove all the impurities by fractional distillation to give a pure alcohol and then add a flavouring agent. Such a beverage may not be identical to commercial gin (actually all brands of gin have slightly different flavours) but it will be absolutely safe.

A final comment concerns the question of alcohol concentration in beverages. In beer the concentration is about 5%, in wine it is 8 to 13%, while in distilled spirits it is usually 40%. Only a moment's thought is required to appreciate that the <u>concentration</u> of alcohol in a drink is irrelevant; it is the <u>amount</u> consumed which is the determining factor in whether or not someone becomes inebriated. Drinking a bottle of 5% beer is not less harmful than a 1½-oz. drink of 40% scotch just because it is weaker. They both contain identical amounts of the same alcohol, i.e. 17 ml. Adding tonic water to a shot of gin dilutes it from 40% to maybe 6% but this has not rendered the gin less intoxicating --- the amount of alcohol has remained unchanged.

This is all so obvious that it may seem a little absurd to even mention it but, in most countries, the concept appears somewhat too difficult for the official mind to grasp. This is shown by the fact that governments put a much higher tax per unit of alcohol on distilled spirits than on beer and wine. The reason for doing this, it is claimed (somewhat piously), is to discourage people from drinking something which could be harmful to their health. A more likely reason is that it is seen as an opportunity to increase revenues.

The Question of Legality

This chapter is written specifically for readers who live in countries where it is currently illegal for amateurs to distil their own home-made beer and convert it into gin or vodka. The rest of us can happily jump ahead to the chapters dealing with equipment and procedures.

The conflict between governments and moonshiners has been going on for centuries and the reasons are not hard to find. From the government point of view alcohol in one form or another is in such demand that it can be heavily taxed without fear of killing the goose that lays the golden egg. From the moonshiner's or smuggler's point of view the spread between the cost of manufacture of alcohol and the cost to the consumer after tax is so great that the incentive to circumvent the law is considerable.

The dollar figures involved are informative. When alcohol is made on a large scale, as it is for the fuel-alcohol industry (gasohol) its cost of manufacture is about 25 cents per litre. This is for 100% alcohol. If diluted to the 40% commonly used for vodka, gin and other distilled spirits a litre would contain about 10 cents worth of alcohol. The retail price of a litre of vodka will lie somewhere between \$10 and \$20 depending on the country and the level of taxation. Some of the difference is due to the scale of manufacture, the purity of the product, transportation, the profit margin, etc. but even allowing for these factors the tax burden on the consumer is extremely high. Is it any wonder that an unscrupulous operator will attempt to sell his alcohol direct to the consumer, perhaps at half the normal retail price which would still give him a very handsome profit? Or is it any wonder that the authorities crack down hard on anyone attempting to interfere with their huge source of revenue, their milch cow?

This battle between illicit alcohol producers (moon-shiners) or importers (smugglers) and the authorities has now become the stuff of legend. Consider the number of stories written or movies made about desperate men rolling barrels of rum up a beach at midnight! Or about the battles between gangsters and police during prohibition days in the United States! Unfortunately, such stories have been taken too much to heart by the general public so that the whole idea of distillation, and the spirits made by this process, is now perceived as being inherently more wicked than the gentle art of beer- or wine-making. And the "wickedness" is a strong deterrent to most people. It is understandable why a government would wish to put a stop to smuggling and moonshining for commercial purposes, that is to say in order to re-sell the product and avoid the payment of taxes. But why would there be a complete ban on distillation by amateurs, on a small scale and for their own use? At the risk of being tediously repetitious it is worth reminding ourselves again (and again) that distillation is one of the most innocuous activities imaginable. It doesn't produce a drop of alcohol. Not a drop. What it does is take the beer which you have quite legally made by fermentation and remove all the noxious, poisonous substances which appear inevitably as by-products in all fermentations. Far from making alcohol, a little will actually be lost during this purification process. Instead of prohibiting it, the authorities should really be encouraging distillation by amateurs. And the general public, which is so rightly health-conscious these days, would be more than justified in demanding the right to do so.

In attempting to find the reason for governments to ban the purification of beer or wine by distillation the first thing which comes to mind is the potential loss of revenue. After all, if everyone started making their own spirits at home the loss of revenue could be considerable. But this cannot be the real reason because the home production of beer and wine for one's own use is legal, and both are taxable when sold commercially, so the authorities must not be all that concerned about the loss of revenue when people make their own alcoholic beverages.

A possible, and somewhat cynical, explanation for the prohibition of home distillation is based on the following reasoning: Home-made beer and wine are usually so inferior to a good commercial product that only the most dedicated amateurs will go to the trouble of first making and then drinking such doubtful concoctions. Consequently, there is no real threat to the sale of commercial products nor to the revenues generated by taxation. If, however, home distillation were permitted, every Tom, Dick and Harriette would be in a position to make a gin or vodka which was every bit as good as the finest commercial product on the market. This could, it might be argued, make serious inroads into commercial sales and into government revenues.

Further thought, however, makes it very unlikely that amateur production of spirits would have any appreciable effect on commercial sales. For one thing the equipment is moderately expensive and it is necessary to follow directions rather carefully when using it so it is unlikely that the practice would ever become really widespread. Moreover, many people prefer scotch, rye, rum, etc. to gin and vodka and it is only the latter which can be made safely and effectively by the amateur. So, if distillation were legalized for amateurs, it would probably become nothing more than an interesting hobby, just like making wine, and offer little competition to commercial producers.

No, we have to look deeper than this in our search for a reason why governments have a hang-up about distillation. You see, it is not just amateurs who are penalized. Commercial producers also feel the heavy hand of government prejudice and disapproval. This is illustrated by several restrictions which apply in many countries. One is the fact that the advertising of beer and wine on television is permitted whereas the advertising of distilled spirits is prohibited. Another concerns the tax imposed on distilled alcoholic products --- per unit of alcohol the tax on the distilled product is much higher than it is on beer and wine. A third restriction on spirits can be seen in the alcoholic beverage section of supermarkets ---- beer and wine are sold, and possibly fortified wines such as vermouth, but raise the alcohol concentration to 40% and the ancient shibboleth of 'hard spirits' reigns supreme. This is grossly unfair discrimination and naturally of great concern to distillers. As they point out, a glass of gin and tonic, a glass of wine, and a bottle of beer all contain similar amounts of alcohol, so it is inequitable to tax their product at a higher level.

So just why is there this official discrimination against distilled alcoholic beverages? Irrational attitudes are always difficult to deal with, but in order to reform the law we have to deal with it, and this requires that we try to understand the thinking behind it. The drug involved is ethyl alcohol, an acknowledged mood-modifier, but ethyl alcohol itself is not singled out by governments as being the bad actor. The alcohol in beer, wine and gin are identical and imbibed in similar quantities will have identical effects in terms of mood modification. No, apparently distillation *per se* is perceived as evil, to the point where even owning the equipment is illegal.

There is only one explanation which seems to fit all the facts and this is that governments and their officials fail to make a distinction between <u>concentration</u> and <u>amount</u>. Actually, quite a lot of people have this problem. Just because beer has 5% alcohol and gin has 40% does not mean that the

gin-drinker is eight times more likely to over-indulge than the beer drinker. The fact of the matter is that anti-social behaviour such as hooliganism at sporting events is invariably caused by beer drinkers. And many studies of drinking and driving have shown that the vast majority of those pulled over have been drinking beer, not spirits. People drink until they've had enough, or feel in a certain mood, and if this takes five, ten, or even more beers then that is the number which will be drunk. It is the testosterone concentration which causes the problem, not the alcohol concentration.

A few attempts have been made to dig deeper into the reasons behind the official attitude to distillation but it is a frustrating experience. Invariably the person spoken to seems bewildered by the question, almost as though one had asked why it was illegal to murder someone. One individual explained patiently and kindly that it was because the law is the law. Another made the extraordinary statement that distillation was prohibited because it makes alcohol and this is illegal. (Of course distillation does <u>not</u> make alcohol. Alcohol is made by fermentation, not by distillation, and in any case fermentation to make beer and wine for one's own consumption is completely legal).

The above discussion has been argued at some length because a) it is important for the reader to feel comfortable with the "moral" aspects of distillation, and not feel obliged to be furtive about it, and b) in order to illustrate the difficulties which would be encountered in any attempt to change the law. There would be no point in approaching government officials who in many cases are sympathetic to the arguments but are powerless to do anything about it. No, it would be necessary to first air the subject in the news media to get the public (the voters) up to speed and then work through politicians. The approach could be based upon two issues, both of which are important to many people nowadays. One is the question of health --- governments should respond favorably to any suggestion which will lead to more healthy drinking habits (and make no mistake about it, gin and vodka are much less harmful to health than beer and wine). The other concerns our basic rights and freedoms --- it should be an absolute right for anyone to remove the poisonous substances from a legally produced beverage (beer) in order to produce another legal beverage (gin and vodka).

Equipment

The home production of pure alcohol for use in gin, vodka or any other beverage is a rather technical and equipment-oriented activity. In this respect it differs quite a bit from wine- and beer-making which involve the use of very little specialized equipment but a lot of skill, careful selection of the ingredients used, and rigorous attention to matters of hygiene. Wine and beer making are equivalent to the activities of a gourmet cook. The production of pure alcohol on the other hand is a scientific operation, with no requirement for any special talents or flair but every requirement for using the correct equipment according to established scientific principles and set procedures. Not many people can make a first-class wine but anyone, using the right equipment and following recommended procedures, can easily make alcohol of the highest purity.

Ideally, one would use scientific glass equipment for distillation. Flasks with heating mantles, columns, column packings, still-heads, condensers, thermometers, etc., all made of glass and nicely fitting together with ground-glass joints, are available from scientific supply houses. They come in all sizes from tiny bench-top models to the large equipment used in pilot plants. And the whole thing would be elaborately instrumented. Nice to look at and fun to use. Unfortunately, such equipment is horrendously expensive. Furthermore, even if the prices were reasonable or you were an eccentric millionaire, you would find it difficult to locate and do business with the suppliers. They cater to universities and research institutes and are not geared to supplying the needs of individuals and enthusiastic amateurs.

A relatively inexpensive and convenient solution to this problem is to use domestic appliances wherever possible. They need some modification and adaptation to be sure, and certain items will need to be fabricated, but the task is well within the capabilities of the average handyman. Also, everything you need will be available close to where you live ---- at a hardware store, a supplier of plumbing equipment, or a machine shop. The final cost will be a fraction of what it would have been if scientific equipment had been purchased. Also, in addition to saving a great deal of money, you undoubtedly will be a lot more knowledgeable as a result of putting together something with your own hands. Metal is also a lot more rugged than glass. A consequence of deciding to use domestic appliances is that one is obliged to operate at a certain level of production. Fortunately, this level, although perhaps a little larger than one might wish for, is not unreasonable and indeed could be just about right for many people.

Specifically, the equipment and procedures to be described in this book are based on the fermentation of 10 kg of sugar to yield 10 to 11 litres of 40% alcohol, either in the form of gin or vodka.

There are four major equipment items. They are the fermenter, the beer stripper, the high-purity alcohol still with fractionating column, and the little pot still for producing the flavouring ingredient for gin. This last item would be unnecessary if a) only vodka were required, b) if you intended to use unflavoured alcohol for making liqueurs, or c) if you made your flavouring essence by steeping the botanicals rather than by distillation.

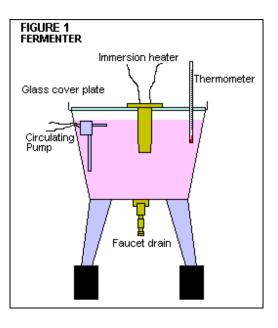
The Fermenter

A polypropylene laundry tub makes an ideal fermenter. A common size is 45 x 50 cm by 30 cm deep, standing on four legs to give a total height of 85 cm above the ground. The working volume is about 65 litres or 17 US gallons.

One can make this fermenter as simple or as elaborate as one wishes. In its simplest form one would merely close the drain-hole with a rubber stopper, add the sugar and dissolve it in warm water, add the yeast and stir periodically. This presumably is how they made "bathtub" gin in the old days, using a bathtub instead of a laundry tub. But for convenience and to get the best yield of alcohol a few refinements should be added. One is a cover to keep out dust, any insects flying around, and to reduce losses by evaporation and oxidation. Another is an electrically driven stirrer. A third is a heater to maintain the right temperature over the several days of fermentation. A fourth is a faucet attached to the drain to permit the beer to be run directly into the stripper (see below) and wash water to be directed to the house drain when the fermenter is being rinsed out.

A suitable arrangement is shown in Figure 1. The fermenter stands on four legs which in turn stand on four cement blocks. The purpose of these blocks is to raise the bottom of the laundry tub to a point where all the beer can be transferred to the beer-stripper by gravity flow following fermentation.

Cover: A cover for the laundry tub can be made out of either thick sheet plastic or plate glass. The plastic is easy to work with but suffers from the disadvantage that it bends up at the edges as the high humidity in the fermenter expands the underside of the sheet. For clarity in viewing and stability in operation plate glass about ¹/₄ inch thick is ideal, albeit difficult for an amateur to work with. A laundry tub usually has a convenient shoulder a few centimetres below the top so have your glass supplier cut a piece to a size which will rest comfortably on this shoulder.



Two holes should be drilled in the cover, one in the centre about $1\frac{1}{2}$ inches in diameter to take an immersion heater and the other about 5/16 of an inch for a thermometer. A small notch along one edge will be useful for accommodating the power supply line if you intend to use a submersible circulating pump (see below).

Stirrer: There are at least three methods of stirring the fermentation brew. They are: a) with a motor mounted above the fermenter driving a shaft which goes through a hole in the glass cover-plate; b) with an impeller mounted through the bottom of the laundry tub. The impeller in the base of a food blender can be adapted to this purpose; c) with a submersible pump such as used for circulating the water in an aquarium or for driving the fountain in a small ornamental pool. Our strong recommendation is to use a submersible pump, the reason being that the shaft of a stirrer mounted as in a) above tends to whip while a stirrer mounted in the bottom of the tub as in b) above tends to leak. A submersible pump on the other hand suffers from neither of these two disadvantages. If you use an aquarium pump, be sure to close off the air inlet provided for the aeration of the aquarium since

aeration during fermentation will simply lead to the growth of yeast rather than to the production of alcohol. Alternatively, submerse the pump sufficiently deeply in the beer that no air can reach it.

Immersion heater: The optimum temperature for fermentation is between 30 °C and 35 °C. Fermentation itself generates some heat but probably insufficient to maintain this temperature, particularly if the room is cool. An external heat source should be provided, therefore, and since only 100 watts or so are required an immersion heater such as used for an aquarium is ideal. If it does not contain its own thermostat an ordinary light dimmer switch works very well. The immersion heater can be attached to a small piece of sheet plastic or metal and suspended through the large hole in the plate-glass cover.

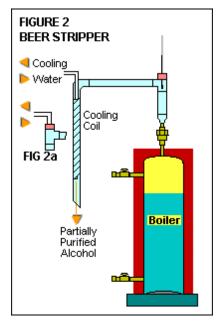
Drain: The drain outlet of a laundry tub is designed to take a tailpipe for connection to the house drain. This should be modified to take a 3/4 inch ball-valve and hose adapter.

Use a brass tailpipe and some ingenuity(!) to connect it to the ballvalve. A length of hose with a female connection at both ends, as used for the hose connection to a washing machine, will enable you to couple the fermenter to the beer-stripper (see later) when you need to transfer the beer.

Beer Stripper

Beer stripping is simply a fast, crude distillation of the beer in a pot still in order to obtain most of the alcohol in a smaller volume of water. This smaller volume of distillate, about a quarter of the original volume of beer, is easier and cleaner to handle in the small precision equipment used for the final stage of fractional distillation.

An effective and fairly inexpensive beer-stripper can be fabricated from a 30 US gallon (113 litre) domestic hot water heater. A sketch of the water heater and the modifications required are shown in Figure 2. A 3/4 inch inlet for cold water is provided by the manufacturer on the side at the bottom and another 3/4 inch hot water outlet near the top. A third 3/4 inch pipe connection will be found by removing the sheet metal cover and fibreglass insulation from the top of the tank. This is where the magnesium rod used as an anti-corrosion device is installed. Remove it since it is not needed in our application and we may need the 3/4inch connection for the installation of the steam-condensing system.



The steam-condensing system, as shown in the diagram, is made from $1\frac{1}{2}$ inch copper pipe. An adapter, or series of adapters, will be needed to go from the 3/4 inch female pipe thread in the top of the boiler to the $1\frac{1}{2}$ inch copper pipe used for the rest of the system. We suggest that a union be provided to permit easy disassembly if required.

A $1\frac{1}{2}$ inch copper tee as shown permits the fitting of a cork and thermometer to read the temperature of the vapours distilling over. These vapours are condensed by means of cold water running through a coil of copper tubing inserted in the down-stream vertical section of the $1\frac{1}{2}$ inch pipe. To make this coil use 12 feet or so of 3/16 inch flexible copper tubing (obtainable from automotive supply stores), push one end into a short length of 3/4 inch pipe and wind the remainder tightly around the outside. The two ends of the coil are either brought out through the top elbow where they are soldered into place or, more simply, brought out through a large cork inserted in a copper tee. The second version is shown in Figure 2a. Be careful to ensure that the direction of cold water flow is counter-current to vapour flow as it is more effective this way. The lower side connection to the boiler, normally the cold water inlet when the apparatus is used for domestic hot water production, will become the inlet for beer from the fermenter and also the drain for the exhausted beer after stripping. Fit this connection with a 3/4 inch ball valve and screw into it an adapter for connecting a rubber hose. Do use a ball valve at this location, and not an ordinary faucet, because the yeast in beer forms sticky clumps when boiled and there should be a wide opening for the yeast clumps to exit to drain.

The upper side connection (the hot water outlet) is seldom used and could be plugged, but it is just as easy to close it with a faucet. It could then be opened if required and used, for example, as an overflow indicator when washing up.

The thermostat which controls the temperature of the water must be removed or by-passed. Since we wish to boil the beer and collect the vapours, a thermostat which switches off the current at a temperature well below the boiling point of water would obviously defeat our purpose. Disconnecting the thermostat may seem dangerous, and it would be if we had a closed system, but as will be seen from the diagram the top of the boiler is constantly open to the atmosphere via the 1½ inch inverted-U vapour line and condenser so there can be no pressure build-up. It is no more dangerous therefore than a boiling kettle of water.

Small domestic hot water immersion heaters of this size will probably have a single 3000 watt, 240 volt heating element at the bottom. If there is a top element (as there is in larger water heaters) it must be disconnected permanently because the boiler as used in the present application is never full and a top element would burn out. A 3000 watt element should provide about 6 litres of distillate per hour.

After beer stripping, allow a little time for the exhausted beer to cool down and then dispose of it through the ball valve to drain. Back wash with fresh water and drain a couple of times after each run to reduce the possibility of yeast build-up.

Fractional Distillation Apparatus

The crude alcohol produced by the beer-stripper is transferred to the fractional distillation apparatus shown in Figures 3, 4, 5, 6 and 7. Whereas

the beer stripper is an elementary piece of equipment, easy to understand and easy to use, the fractional distillation apparatus is rather more complicated. Few non-scientists have ever heard of such an animal, and have never seen or read about one. Yet a fractionating still is the one essential piece of equipment required to produce pure alcohol and is the key to the success of this whole project.

Material of construction: Glass would really be the ideal material for making small-scale stills, being inert, clean, and transparent. One can see exactly what is going on and it is aesthetically pleasing. For those fortunate few who live close to a university or research institute therefore, and have access to a glassblower, a glass apparatus is described later with a glass stillhead being shown in Figure 6.

For the majority of people however, the choice will have to be metal and the only decision left then is whether it should be made of copper or stainless steel.

The advantages of using copper are that it is relatively inexpensive, it is readily available from any plumbing supply store and, most importantly, it can be worked and soldered together easily by amateurs. Furthermore, the high thermal conductivity of copper makes the cooling coil extremely effective. Commercial whisky distilleries have used copper stills for centuries so it is clearly a very acceptable metal to use.

But then there's the solder. There is no reason to believe that ordinary lead solder is not completely safe — it has been used routinely for many years in domestic plumbing. However, lead-free solder is readily available nowadays and you may wish to use it.

An alternative would be to use silver solder, frequently employed professionally for the fabrication of equipment where the joint may come into contact with chemical solutions. Silver-soldering does, however, require the use of high temperatures, so if you decide to go this route, it probably would be a good idea to assemble the parts yourself and then take the apparatus along to a professional for brazing or silver-soldering. It would only be a few minutes work and should not be expensive.

Stainless steel, of course, is a perfect material of construction for an apparatus such as a still, but it is not one which an amateur will find it easy

to work with. We have taken the design to a stainless steel fabricator, however, and obtained a price, and this information will be listed later for those who prefer to use nothing but the best.

Construction: As will be seen from the sketch in Figure 3., the apparatus consists of a boiler surmounted by a 3 to 4 ft. length of 1¹/₄ inch copper tubing. At the top of the tube is the still-head where the vapours rising from the boiler are condensed and split into two streams. The major stream, consisting of 90% of the condensed liquid, flows back down the column while the remaining 10% is directed to the outside world via a small valve. Let us look at each part of the still in more detail.

The boiler: Just as we did for the beer-stripper we use a domestic hot-water heater for the boiler but in this case it can be quite a bit smaller in size. The kind used for apartments is ideal. They vary in size but are usually in the 5 to 8 US gallon range (20 to 30 litres) and are normally heated by a single element of about 1500 watts at 120 volt or 240 volt, depending on which country you live in. We want the contents of the boiler to boil so, after removing the insulation, remove or by-pass the thermostat just as you did in the case of the beer-stripper.

To the cold water inlet at the bottom of the boiler fit a ball-valve with a hose-bib attachment. To the hot water outlet at the top fit a short 3/4 inch brass nipple and the adapter necessary to install a $1\frac{1}{4}$ inch union. If there is a magnesium corrosion-prevention rod in the boiler, remove it and close the opening with a 3/4 inch ball-valve. This valve is not really necessary but access to the boiler is useful for cleaning.

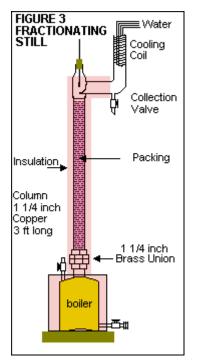
The packed column which will be mounted above the boiler has only a limited capacity to allow vapours to rise up through the packing against the downward flow of condensed liquid so the boil-up rate must not be too great or the column will choke. The 1500 watt heater supplied is, in fact, unnecessarily large so we need to reduce the wattage in some way. A simple and cheap solution is to substitute a 750 watt heater for the 1500 watt one supplied with the water heater. It can then be plugged straight into a socket with no need for a voltage controller.

An alternative is to introduce half-wave rectification of the electricity supply. This has the advantage that you can then heat quickly at 1500 watt, reducing to 750 watt when up to temperature. A diode is an electrical component which accomplishes this by allowing current to pass through it in one direction only. Power supplies in most countries deliver alternating power at around 50 to 60 cycles per second. The diode therefore acts like a switch that opens and closes 100 to 120 times every second. With a diode in the circuit, the heater is therefore switched on and off at this rate and, being powered for only 50% of the time, delivers only half the energy it would if left switched on all the time. A 1500 watt heater would therefore deliver only 750 watts if controlled with a diode. A discussion of diodes and how to construct a controller using one is included in Appendix IV.

The column:

The fractionating column consists of a 3 ft. length of either $1^{1}/_{4}$ or $1^{1}/_{2}$ - inch I.D. copper tubing, whichever you prefer.

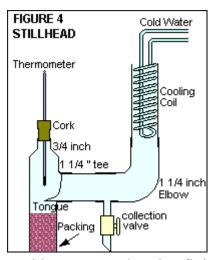
The bottom end of the column is joined to the top of the boiler by means of a union to permit disassembly when required. You could use either a $1^{1}/_{4}$ inch or a $^{3}/_{4}$ inch union, and of course the smaller one is cheaper, but use the large one so that you will have free access to the column for introducing the packing (see later). At the top of the column a tee is provided for the passage of vapour across to the still-head condenser and for a thermometer to measure the vapour temperature.



The column must be well insulated to ensure a stable temperature regime within the column while it is refluxing. Use an insulating sleeve of foam rubber obtainable from your plumbing supply store.

The still-head:

The purpose of the still-head is to divide the vapour emerging from the column into two streams. This it does by first condensing the vapour to liquid in a heat-exchanger and then, as the liquid runs back down towards the column, diverting a portion of it to the outside world via a small valve. This valve has only a small volume of liquid to handle so for fine control choose a needle valve. Solder a short length of 5/16 inch tubing to the still-head as



shown in the diagram and attach the valve with a compression ring fitting. This will avoid the necessity of having to heat the valve itself during soldering.

To make a strong joint, and to ensure a clear path for liquid flow, the following procedure is recommended: Before soldering, drill a 5/16 inch hole in the $1^{1}/_{4}$ inch elbow where it will overlap the inner tube. Then slip it over the $1^{1}/_{4}$ inch tubing and solder in place. Position the 5/16 inch tubing through the hole in the elbow and butting up against the inner tube. Solder in place. Drill right through the short length of 5/16 inch tubing, penetrating the inner tube.

When the valve is closed, all the liquid returns to the column and back down into the boiler. If the valve is wide open all the condensed liquid will run out through it and none return to the boiler. In practice, the valve is adjusted to a setting at which about 10% of the liquid is drawn off into a receiving bottle while 90% returns to the boiler. A valuable refinement is to have a tongue protruding about 3/4 inch into the column from the horizontal portion of the still-head so that the returning liquid cascades down the centre of the column. Without the tongue the liquid is liable to channel down the wall of the column and thereby fail to baste the packing uniformly. The tongue is shown in Figure 4 (but see alternative still head in Figure 7).

The condenser for cooling the vapour and returning it to the column is made from about 10 feet of 3/16 inch copper tubing.

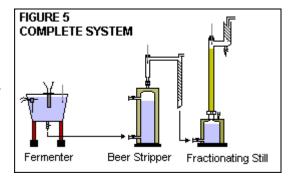
A thermometer in the still-head measures the temperature of the vapour at the top of the column and is an excellent indicator of just when reflux has started. It also lets you know when the "heads" are coming over, when it is pure ethyl alcohol, and when the "tails" are starting to appear.

Packing: The packing inside a fractionating column is very important, and many articles in the scientific literature have been devoted to the subject. What is needed are pieces of glass, ceramic or metal which are inert to the liquid being refluxed and which have the following characteristics:

- a) they should not pack tightly, but should be of such a shape that they leave plenty of free space for vapour to rise up against a descending flow of liquid; and
- b) they should have a large surface area and crevices where liquid can be trapped.

Scientific glass columns frequently use short (6 mm) lengths of 6 mm glass tubing called Raschig rings. If you decide to use a glass column the glassblower you employ will be able to supply you with them. For a metal column such as ours, an excellent and cheap packing is provided by ordinary scouring pads such as used in the kitchen for cleaning pots and pans. They are available in copper, brass, and stainless steel. The stainless steel ones are ideal but are not always stocked so if you have difficulty locating a supplier just use copper or brass. If they are held together by a rubber band, remove it and stretch out the balls of metal turnings into cylindrical shapes. Gently push the packing up the column, doing your best to avoid compaction. For a 3 foot column you will need about 8 scouring pads.

Another possibility for an effective column packing would be the spiral turnings from a lathe. See if you can find a local machine-shop which works in stainless steel and have them put some turnings aside for you. Since they normally go to the scrap-bin you can probably get them for nothing. The fermenter, beer-stripper and fractional distillation apparatus are shown together in abbreviated form to illustrate the sequence of operations in going from sugar to pure alcohol.

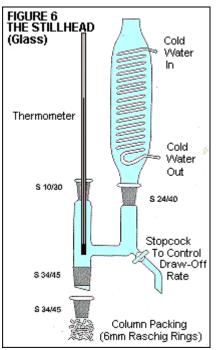


Stainless steel: The design will be just the same as in copper but you will find that the steel fabricator who makes it for you will probably use butt welding rather than fittings to join the pieces together. This adds to the labour costs so that the cost of the column, still-head and condenser will likely prove to be two or three times greater than the same equipment in copper where you have done most of the work yourself.

Glass apparatus:

For those people who have access to a glassblower and, through him, to a scientific supply company, an all-glass still may be appealing. The boiler will be exactly the same as in the copper system shown in Figures 3 and 4, but the column, still-head and condenser will be put together with standard-taper ground-glass joints. The details of a glass still-head are shown in Figure 6.

At the base of the column use a spherical glass ball-joint which will rest on the female half of the brass union fitted to the top of the boiler. As a seal, either use a ring of cork or teflon plumber's tape. The weight of the

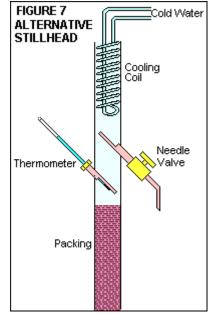


column should be sufficient to prevent leakage since there is virtually no pressure in the apparatus, but if not your glassblower will be able to supply you with a clamp. For the column packing use either Raschig rings or stainless steel scouring pads.

An Alternative Still-head.

The offset type of still-head shown in the preceding diagrams works very well, but you may prefer to use the linear design shown here:

It has several advantages over the previous model. They are: 1) It is cheaper to make since it involves no T's or elbows; 2) No corks are involved for the thermometer; 3) It is more streamlined and elegant in appearance.



Construction. The diagram illustrates the design concept but a few construction details are necessary.

a) The collection "spoon" is cut from 3/8 inch copper tubing. A metalcutting blade on a table-saw is useful for this purpose. Use a 3 inch length of tubing and remove one-half of the diameter for a distance of 1 inch from one end. Cut "nicks" at the root of the trough so that the trough can be slightly flattened to give a shallow spoon 1/2 inch wide.

b) Drill a 25/64 inch hole in the column 13 inches from the top and tilt the drill bit to elongate the hole to an oval — 1/2 inch along its major axis. The spoon will now enter the hole if tilted sideways. Turn it 90° and spring-load to hold it in position at a 45° angle while soldering in place. Attach a 1/4 inch needle valve using a 3/8 x 1/4 inch compression coupling.

c) For the thermometer, drill a 25/64 inch hole $14^{1}/_{2}$ inches from the top of the column on the opposite side to the collection spoon. Elongate the hole into an oval by tilting the drill bit in exactly the same manner as in b) above.

Take a 3 inch length of 3/8 inch tubing and remove one half of the diameter for a distance of 1 inch from one end, just as in a) above. Insert it in the hole so that the open side of the trough is facing down and the closed side facing up. Solder it in place at an angle of 45° . The purpose of this design is to shield the thermometer bulb from dripping condensate while leaving it exposed to rising vapours.

d) Using a 3/8 x 1/4 inch compression coupling, drill through with a 17/64 inch bit (or slightly larger) from the 3/8 inch end to remove the internal shoulder. Be careful not to go all the way through as this would remove a little of the seat for the 1/4 inch ferule at the other end. A mercury/glass thermometer should now slip through nicely. For sealing, a brass ferule is not possible, but a very effective seal is obtained by wrapping a few turns of teflon plumber's tape around the stem and compressing with the nut on the coupling.

<u>Note 1</u>. Some thermometers may have stems which are slightly too large in diameter to go through a 17/64 inch hole. Be careful, therefore, to choose a thermometer which <u>will</u> go through. Or, drill a larger hole.

<u>Note 2</u>. A glass thermometer in such a rigid set-up is very vulnerable to breakage. It is prudent, therefore, to remove it while working around the still.

<u>Note 3</u>. By using a 3/8 inch needle valve one can eliminate one of the $3/8 \times 1/4$ inch compression couplings.

A Single-boiler Distillation System

The purification of a crude "beer" by distillation is a 2-stage process. In the preceding pages we have described a system which uses two boilers — a large one with a high-wattage heating element for the first stage of beer-stripping and a smaller one for the smaller volume of liquid involved in the second stage of fractional distillation. At the sacrifice of a little time and convenience it is possible to carry out both stages with just one boiler, thereby saving the cost of a second boiler and the space which it occupies.

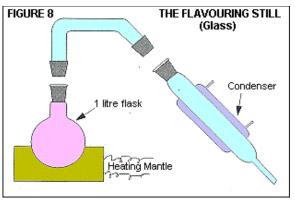
We recommend the following: a boiler of about 40 litres (10 US gallons) and a 750 to 1,000 watt heating element. The first stage of beer stripping will be slow, but many readers have found that the slower, less

vigorous boiling is quite convenient. If they wished, North American readers would be able to employ a 240 volt 3,000 watt element, using the full wattage on 240 v. for the first stage of beer-stripping and then switching to 120 v. for the second stage in order to reduce the power to 750 watts for fractional distillation.

The procedures involved in using this single boiler system are described in the chapter entitled **DISTILLATION**.

The Flavouring Still

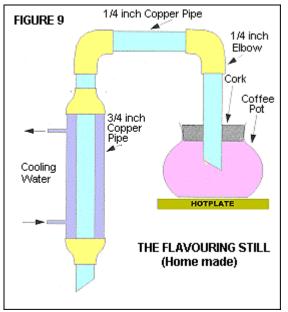
The flavours used for gin-making are contained in a number of herbs and berries, collectively known as "botanicals". One simple method of extracting the flavours without the use of any special equipment is to boil the botanicals in 50% alcohol for several minutes,



cool, and let stand for 24 hours. Then filter the extract through a coffee filter-paper folded into a cone.

The method we shall describe here involves the use of steam distillation. In this method the flavours are extracted from the botanicals with steam and added to the alcohol afterwards. One advantage of this is that no colour is extracted from the plant material.

Steam distillation requires the use of a simple pot still such as that shown in Figures 8 and 9. The botanicals and water are placed in the flask and the water brought to the boil. The steam which is generated releases the flavouring constituents from the herbs and carries into them over the condenser in the form of oily drops suspended in water. In Figure 8 an allglass apparatus is shown,



but this is expensive and only obtainable from a scientific supply house or through your glassblower.

Because steam distillation is such a simple process it is possible to make do with a less elegant but still effective apparatus as follows and as sketched in Figure 9. The condenser is made from a short length of 3/4 inch copper tubing acting as a cold water jacket around an internal 1/2 inch copper tube. Adapters for connecting1/2 inch to 3/4 inch tubing are standard items and are used for sealing the jacket to the inside tube. Cold water inlet and outlet tubes are soldered to the jacket as shown.

The boiler is a glass coffee pot. A large cork, obtainable from any winemakers' supply store, has a hole drilled in the centre to take the 1/2 inch copper tubing. In operation there is very little pressure in the apparatus and no problems with steam leakage.

Fermentation

Principles

The biochemical reaction which converts sugar to ethanol is depicted below:

yeast + $C_6H_{12}O_6$ = $2C_2H_5OH + 2CO_2$ glucose ethanol

This equation tells us that one molecule of sugar (glucose) in the presence of yeast produces two molecules of ethyl alcohol and two molecules of carbon dioxide. The yeast itself, which is a living organism, is not consumed in the reaction but merely acts as a catalyst and remains available for repeated transformations. The atomic weights of carbon, hydrogen and oxygen are 12, 1 and 16 respectively, and when these weights are applied to the above equation we find that 180 parts of glucose will lead to the production of 92 parts of ethyl alcohol and 88 parts of carbon dioxide. As a close approximation, therefore, a given weight of sugar will produce about one-half its weight of alcohol, i.e. 1 kg of sugar should give 500 grams of alcohol. Because the specific gravity of ethyl alcohol is 0.8 the 500 grams represent 625 ml of absolute alcohol or $1^{1}/_{2}$ litres of 40% alcohol, the normal strength of gin and vodka.

It should be understood that the above figures represent the ideal situation, the theoretical yield. Such yields are approached very closely in commercial practice and in well-equipped laboratories, but in the hands of amateurs the yield is unlikely to reach more than about 70% of theory. There are two main reasons for this: one is the occurrence of side reactions which convert the sugar into a whole range of unwanted organic compounds such as methanol, acetic acid, fusel oils, etc., while the second is a failure to recover all the alcohol from the fermentation broth. Losses such as these are not serious for the amateur: after all, at 70% of theoretical recovery a kilogram of sugar valued at a dollar or less would produce over a litre of gin or vodka.

The conversion of sugar to alcohol by means of yeast is an anaerobic reaction; that is to say it occurs in the absence of air. If air is present the yeast, instead of producing alcohol, will multiply and grow. Wine-makers habitually buy a small quantity of an expensive, specialty yeast and let it grow in the presence of a little air until they have the quantity they require. Then they cut off the air supply and the yeast starts making alcohol instead. In our situation such refinements are unnecessary because we use massive quantities of cheap baker's yeast which generates high yields of alcohol and large quantities of carbon dioxide. The CO_2 is quite effective in excluding air without the use of air-locks.

Under such crude conditions the yeast and sugar will produce a wide range of organic compounds in addition to ethanol, a situation which would be disastrous if we were making wine or beer and had to drink these unpleasant and even harmful substances. However, the presence of such impurities is of small concern to us because they will all be removed during distillation.

The production of extraneous compounds will be aggravated by sloppy practices so, although it is not as necessary to be as careful as it would be during wine-making, reasonably hygienic conditions should be maintained at all times. Otherwise one is simply wasting sugar.

Procedure

Those of you who are familiar with the making of beer and wine will find the fermentation of supermarket sugar with baker's yeast in a laundry tub a rather simple and crude procedure. Don't be disconcerted by this. All we are doing at this stage is producing the alcohol we need. Not being the final product, and not being intended for drinking, our concern is simply to make the alcohol as rapidly and as cheaply as possible. Taste is of no importance. The sophistication comes later on when we take this noxious beer and purify it by distillation.

The laundry tub fermenter described in the equipment section is washed with soapy water and then rinsed. Also wash the accessories such as circulating pump, immersion heater, thermometer and glass cover. Avoid the use of scouring powders as they tend to mar the polished surface of the polypropylene tub.

After rinsing, close the drain valve and insert a rubber stopper in the drain hole of the laundry tub. Add 10 kg of sugar, about 50 litres of warm water and stir with a wooden spoon until most of the sugar has dissolved. Then start the circulating pump, making sure that the inlet to the pump does

not suck in grains of sugar as this could lead to damage. Cover with the glass plate, install the immersion heater and thermometer in their respective holes in the cover, and switch on the heater.

Yeast: There are two forms of active yeast the instant, dry, powdered type and the active, moist variety which comes in blocks. Either one sort or the other will be obtainable from the baking section of your local supermarket or perhaps from a delicatessen and it makes little difference which you use. The powdered yeast is about three times as active, pound for pound, as the moist yeast in block form, so work out which of the two sorts is the best buy. If there isn't a great deal of difference in price choose the dry type because of its much longer shelf life but do check the "use-by" date to ensure that it is fresh. The yeast should be vacuum packed and have a "use-by" date at least several months ahead. Yeast obtained from a bakery is almost certainly fresh and active because of the rapid turnover.

To ferment 10 kg of sugar use 450 grams (1 lb) of the moist yeast in block form or 150 grams of the dry, powdered variety. In the first case, to prepare it for use you will need to make it into a cream. Use a stainless steel bowl and two wooden spoons. Break the block into walnut size pieces and let them stand for about 15 minutes in a small amount of water before attempting to cream them. The chunks of yeast will swell in the water and be far less sticky as a result. Work at it gently until a lump-free cream is produced and then pour the cream into the sugar solution. The dry powdered yeast can simply be sprinkled slowly on to the top of the sugar solution.

With this amount of yeast and the time being allowed for fermentation (5+ days) there is no need to add nutrients. Do not be seduced by claims that special yeasts will allow you to produce a 15% alcoholic solution instead of the more normal 12% because this does <u>not</u> mean that you get more alcohol, only that you can use less water. The <u>amount of</u> alcohol you get will be determined solely by the amount of sugar you have used and the yeast has nothing to do with it.

When the temperature in the fermenter has reached 30 to 35 $^{\circ}$ C. adjust the thermostat or light dimmer control to hold it in this range. For the next five days or so the only attention required is a periodic check of temperature.

The completion of fermentation can be judged in several ways. One is the absence of foam on the surface of the solution; this foaming is quite vigorous at first but diminishes steadily with time until eventually the fermentation ceases and the beer looks dark and still. The best way to know when fermentation is complete, however, is to float a hydrometer in the sugar solution. At first, the specific gravity of the solution will be so high — about 1.06 — that the top of the hydrometer may be pressed against the underside of the glass cover. As the sugar is converted to alcohol with a specific gravity of 0.8, the hydrometer will slowly sink until it shows a specific gravity below 1.00. With a little experience you will know exactly when to expect fermentation to be complete (e.g. 5 to 6 days) and can make a closer examination at that time.

When fermentation is complete, switch off the pump and heater and remove them for washing. Reach down into the beer and remove the rubber stopper, substituting a short (perhaps 1/2 inch) length of $1^{1}/_{2}$ inch copper tubing in the drain-hole. This will act as a dam and help to hold back some of the yeast when you drain the beer into the beer-stripper.

Allow the beer to stand for several hours or preferably overnight in order to give the yeast a chance to settle to the bottom of the fermenter. At the end of this settling period, connect a hose between the drain valve under the fermenter and the inlet at the base of the beer-stripper. Open the valve and allow the beer to flow by gravity into the stripper. Chase it with a little water to clear out the beer in the hose. Close the valve at the base of the beer-stripper, remove the hose and wash the spent yeast from the fermenter to drain.

Note: Some yeast will inevitably get into the beer-stripper. It will do no harm, but be alert to the possibility that it may accumulate in the bottom of the stripper over a period of months and start to clog the drain valve. Back washing with water after each run is therefore quite important.

Distillation

Principles*

Much of what needs to be said about the principles of distillation was covered in the chapter on beverages. There, the distinction was made between the comparatively simple pot stills used in the manufacture of whisky and the more elaborate still with fractionating column used to remove all the impurities and leave a pure alcohol, as in the manufacture of gin and vodka. The present chapter will explain just what is involved in carrying out a fractional distillation and how you go about it, but first a few words about principles. These will let you know just why a certain procedure is being followed. There is nothing more irritating in an instruction manual than to be told arbitrarily to do something without an explanation as to why it is necessary.

Firstly, as mentioned earlier, distillation does not make anything. It is a purification process in the same sense that a water-softener removes hardness from water. In the case of alcohol, distillation does not and cannot produce a drop of alcohol, and there will be the same amount of it at the end of the purification (or in practice a little less) as there was at the beginning.

Distillation separates the various chemical compounds produced during fermentation, using the difference in boiling points to effect the separation. The boiling points at standard atmospheric pressure of some of the more important chemicals found in the beer produced by fermenting sugar with yeast are shown in the table below. The same chemicals are found to a greater or lesser extent in commercial wines, beers and whiskies, but in these beverages they are, of course, quite happily consumed. Sometimes with unfortunate consequences the next morning!

^{*} Footnote. In Appendix III will be found a detailed description of the mechanisms involved in distillation, a subject which should be of interest to all those who wish to know exactly *why* something happens in addition to knowing *how*.

Compound	Boiling Point, °C.
Acetone	56.5
Methanol	64.7
Ethyl acetate	77.1
Ethyl alcohol	78.4
Propyl alcohol	97.2
Water	100.0
Butyl alcohol	117.5
Amyl alcohol	137.8
Furfural	161.0

When a mixture of these compounds is boiled the most volatile, i.e. the ones with the lowest boiling points such as acetone and methanol in the above table, will vaporize first. When they distil over they are referred to as the "heads". There is no clear-cut separation of the various compounds so the heads are still coming over when the ethanol starts to appear. Similarly, before all the ethanol has distilled over, the "tails" will begin to appear in the distillate. These tails are the compounds at the lower end of the above table, i.e. those with the highest boiling points such as propyl, butyl and amyl alcohols. These alcohols are known collectively as "fusel oils" and, like methanol and some of the other compounds, are quite poisonous.

In such a system there is probably a small fraction in the middle which is pure ethyl alcohol but most of it will be contaminated with either heads or tails. One could discard the first heads and the last tails and redistil the middle fraction, repeating this process over and over again until the last of the impurities had been wrung out of the ethanol. Unfortunately, apart from being very time consuming, the loss of ethanol on repeated redistillation would be such that the final yield of pure alcohol would be virtually zero.

Fortunately, it is possible to overcome this problem by a very elegant procedure called fractional distillation, a process which has already been described to some extent in the equipment section. It will be useful to refer back to the diagrams in that section as you read on.

In fractional distillation the vapours emerging from the boiling mixture pass up a column packed with small pieces of glass, ceramic, stainless steel, or other inert material. Each of these pieces can hold a small amount of liquid, either internally (if they have internal crevices) or in the interstices between adjacent particles. At the top of the column the emerging vapour is condensed into a liquid by means of cold water running through a heat exchanger. The condensed liquid runs back down the column until it reaches the boiler where it is reheated, converted into vapour once more, and once again moves up the column.

At equilibrium, which may take several hours to achieve, the system consists of vapour rising up the column meeting a flow of liquid running down the column. At each vapour-liquid interface on the packing material within the column a partial separation occurs wherein the more volatile components of the mixture go into the vapour phase and rise to the top while the less volatile components go into the liquid phase and are carried down into the boiler. At equilibrium, the many components in the mixture become stacked up in the column in the order of their boiling points, the most volatile at the top and the least volatile at the bottom.

In a commercial operation, which runs continuously, the different components of the mixture are drawn off at various heights within the column, and this continues indefinitely. Methanol, for example, would be continuously drawn off from the top of the column while ethanol would be continuously removed from a point a little further down.

Very small operations such as we are concerned with here do not employ a continuous system. Rather, fractional distillation is carried out batch-wise. After column equilibrium is established, with acetone and methanol at the top and fusel oils at the bottom, we start to progressively draw off liquid from the top of the column. First come the acetone and then the methanol and any other low boiling point compounds. Then the ethanol starts to appear, and when it does a portion of it is drawn off and bottled for use. The remainder is allowed to run back down the column to continue the counter-current flow and the purification process. Eventually the ethanol will be exhausted and the higher alcohols, the so-called fusel oils, will start to emerge. At this point (or in practice somewhat before) the boiler is switched off.

Water is an important constituent of the fermentation broth and with a boiling point of 100 °C. lies intermediate between the least and the most volatile components of the mixture. It has one important difference from the other components, however, in that it forms an azeotrope with ethanol. An azeotrope is a mixture of two liquids with a boiling point lower than either constituent. In the case of ethanol and water the azeotrope occurs at a mixture of about 95% ethanol and 5% water. As far as the system is concerned it "thinks" that this mixture is a single liquid with the lower boiling point and proceeds to separate it on that basis. The ethanol which is purified by a fractionating column is not, therefore, pure 100% ethanol but pure 95%, the "impurity" being pure water. No amount of re-distillation under the conditions we are using will influence this percentage.

If it is absolutely essential to remove all the water, for example if it is to be mixed with gasoline to produce gasohol, then it is necessary to break the azeotrope by adding a third component such as benzene. For our purposes, however, where we are going to dilute the alcohol with water to 40% anyway, the presence of 5% water is of no consequence.

Procedures

As a practical matter the purification of beer by distillation is carried out in two stages. The first stage is known as beer-stripping and consists of a crude, rapid distillation in a pot still to contain the alcohol and impurities in a smaller volume. This smaller volume is then purified much more slowly and carefully in the second stage of fractional distillation.

Beer-stripping: Beer-stripping is not essential and if one wished to avoid the expense of the equipment required it would be quite possible to fractionally distil the beer itself. However, beer-stripping has a number of advantages. The chief is that the alcohol and impurities are concentrated into a relatively small volume in a short time. Also, the yeast is left behind and does not enter the more delicate fractionating still. A further advantage is that the crude alcohol solution will be sterile after beer-stripping and therefore can be safely stored for short periods pending fractional distillation if this is required for some reason.

As discussed in the equipment section, an inexpensive beer-stripper can be made from a 30 US gallon (113 litre) domestic hot water heater (see Figure 2). If the fermenter is mounted on blocks the beer can be transferred to the stripper by gravity flow via a short length of rubber hose. For complete drainage it is only necessary to ensure that the bottom of the fermenter is higher than the final liquid level in the stripper. Any beer in the hose can be driven over with a little water poured in from the fermenter end. With the beer in the stripper, close the bottom valve, start running cold water through the condenser, and turn on the electric current. You will be collecting about 16 litres of distillate so have several large plastic bottles ready to receive it. An even better arrangement is to run the output from the beer-stripper directly into the fractional distillation boiler, using a funnel and a length of rubber hose. With a 3000 watt heater the 50 to 60 litres of beer will come to the boil in about 2 hours and liquid will then start to drip from the condenser.

The temperature of the vapour coming over from the boiler at the start will be about 80 °C. and will rise to 98 to 100 °C as the ethanol in the boiler becomes exhausted. This will take about $2^{1}/_{2}$ hours. Although there is still a little ethanol remaining in the boiler at this point, the amount will be too small to warrant the cost of the electricity to drive it over.

Allow the boiler to cool for a few hours before opening the bottom valve and sending the contents to drain. Run a little water through the boiler to flush out as much of the dregs (probably containing a little yeast) as possible.

Fractional distillation: This is the most important step in the whole process of producing pure alcohol from sugar. And an essential step. Any description of alcoholic beverage production which does not include it is describing the production of an impure product, a moonshine. It may be palatable but it will certainly not be pure alcohol.

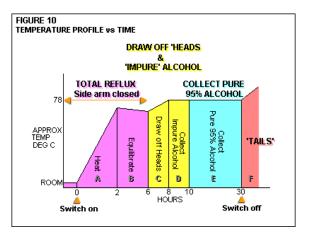
Because of its importance it will be described in some detail, a detail which unfortunately may be intimidating to some and boring to others. To those in the first category we say this: Once you have assembled the equipment and made a few runs it will all become incredibly routine. It's like riding a bicycle a lengthy description of how to do it would probably decide you to take up walking instead, but once you've set off down the road there's no looking back. It's easy!

The 14 to 16 litres of impure alcohol produced by the beer-stripper is now in the boiler of the fractional distillation apparatus illustrated in Figure 3. The boiler, which is an apartment-size hot water heater with the thermostat removed, can be obtained with a capacity in the 7 to 10 US gallon range (25 to 38 litres) and consequently can easily accommodate the entire output of the beer-stripper. The column is insulated in order to maintain a steady temperature regime within the full length of the column during the many hours of operation.

Note that the top of the still-head is completely open to the atmosphere and not sealed. This means that there is no pressure in the still and no danger of an explosion. No vapour can escape through the open top of the column and still-head because the cooling coil is very effective in converting the vapour to liquid, which then runs back down into the boiler.

Cold water is run through the condenser in the still-head and power supplied to the boiler. At the start the small valve in the horizontal part of the still-head is closed so that all the vapour condensed at the top will run back down the column. Under these conditions the column is said to be operating under "total reflux".

Keep an eye on the operation until the thermometer in the stillhead suddenly rises and you know that the hot vapours from the boiler have heated the column and its contents and have risen into the condenser where they are being cooled and converted back to liquid.



The boil-up rate must not be greater than the column can handle. A packed column provides only a limited path for liquid to flow down against a rising stream of vapour so, if the boil-up rate is excessive, the column will choke with liquid and become ineffective. This should not be a problem with the $1^{1}/_{4}$ inch diameter column and the type of packing described in the equipment section, especially if the heat input is reduced to 750 watts by changing or controlling the immersion heater in the boiler as recommended.

The next several hours are spent equilibrating the column. This is the period during which the various components of the mixture sort themselves out with the more volatile components moving to the top of the column and the least volatile moving to the bottom. Don't omit this step because it is quite important. The progress of equilibration can be followed by watching the temperature of the vapour at the top of the column. Ethyl alcohol has a boiling point between 78 and 79 °C., the exact figure depending on the atmospheric pressure, while the heads have a lower boiling point. The thermometer will register this and a temperature as low as 70 °C. may be observed. Periodically open the valve in the still-head a fraction to bleed off these heads into a receiver, leaving room for the ethanol to rise a bit higher in the column. A suitable withdrawal rate would be 2 or 3 drops per second.

You will notice that these heads have a terribly pungent smell and you can congratulate yourself that you won't be drinking them. They are highly inflammable and make an excellent fondue fuel or starter fluid for the barbecue. As the heads are bled off the temperature will slowly rise to a value just above $78^{\circ}C^{*}$, indicating that most of the heads have now been drawn off and ethyl alcohol is beginning to appear. A word must be said here about the accuracy of thermometers. A thermometer purchased from a scientific supply house should be accurate to 0.1 °C. but don't count on it. Thermometers purchased at a drugstore or a winemaker's supply store can be off by as much as 2 degrees. We recommend that you always check the accuracy of a thermometer by placing it in boiling water and recording the temperature. You may be lucky and find you have purchased one which reads 100 °C. but if it doesn't, simply make a note of the deviation and apply the appropriate correction whenever you use it to read a temperature.

Fortunately for us it is not necessary to rely on the exact temperature during a fractional distillation in order to indicate when the heads have finished coming over and it is safe to start collecting ethanol. <u>Constancy</u> of temperature is sufficient. Thus, if the temperature has risen to just over 78 °C. and has stayed there for 15 minutes or so you can be fairly sure that the heads have pretty well finished.

^{*}Footnote: The ethanol/water azeotrope has a boiling point of 78.14 °C. at the standard atmospheric pressure of 760 mm Hg. This changes with a change in atmospheric pressure. The B.P. of pure 100% ethanol is 78.4 °C at standard atmospheric pressure.

Briefly then, proceed as follows: Operate under total reflux for several hours to equilibrate the column, bleeding off the heads periodically until the temperature remains constant between 78 and 79 °C. Then start to collect the distillate by opening the valve in the still-head. A diagram (not to scale) is provided in Figure 10. to illustrate the sort of changes in temperature to expect from the moment the apparatus is switched on to the moment you switch off and start to clean up.

Collection rate: In simple distillation you collect everything which vaporizes from the boiler, but in fractional distillation you collect only 10% of it. The reason for this is as follows:

The efficiency of a fractionating column in separating liquids of different boiling points is dependent upon two factors. One is the length of column and the type of column packing, i.e. its physical characteristics. The second is the reflux ratio, i.e. the way in which the column is used.

The principle of fractional distillation requires that the vapours rising up the column encounter the condensed liquid running down the column. If, in the extreme case, all the vapour rising up the column were drawn off at the top via the collection valve there would be no liquid left for flowing back down the column. So there would be no counter-current flow and no separation. At the other extreme, if the collection valve were closed and all the condensed liquid flowed back down the column (total reflux) the separation would be excellent but no product would be obtained. Obviously there has to be a compromise and this is achieved at a reflux ratio of about 10:1.

This ratio refers to the volume of liquid flowing down the column at total reflux compared to the volume drawn off through the collection valve. Thus, if the heat input to the boiler were causing the liquid to reflux at a rate of 1000 ml per hour, 100 ml per hour of distillate could be drawn off as usable product. The balance of 900 ml per hour would be flowing back down the column to provide the multiple mini-distillations required for the separation. It will be appreciated that the 10:1 ratio is not critical ... 8:1 would be acceptable and 12:1 even more so. The 10:1 figure is simply a reasonable value which is known to give good results.

So the first step involved in determining just how much alcohol can be produced per minute or per hour is to find out the rate of reflux in the fractionating column, i.e. the boil-up rate. When we have this figure we divide by ten and this is the volume of 95% alcohol which can be drawn off through the collection valve in the still-head.

The way to proceed is as follows: With a known wattage input establish steady refluxing conditions and then open the collection valve WIDE. Measure the output per minute, either in terms of volume using a graduated cylinder or by weight using a sensitive scale. You may wish to repeat with other wattage inputs.

We found that with 750 watts input to the boiler the rate of reflux was 45 ml per minute. Other wattage inputs gave proportional volumes. This means that with 750 watts input and a reflux ratio of 10:1 we can draw off 4.5 ml of 95% ethanol per minute. In practice we use about 4 ml to be on the safe side.

With slight variations in the construction of your column, in the way you have packed it, and the amount of insulation you have used, you'll probably get slightly different results from the above, so do measure the rate of reflux for yourself. It's simple and informative.

It is not very convenient to set the collection valve each time you carry out a distillation by using the volume which flows out in one minute. It is too cumbersome. A better method is to laboriously find a valve-setting which does deliver 4 ml per minute and then count drops using a stopwatch. Thus, 4 ml per minute might represent, say, 30 drops in 10 seconds. Knowing this you can quickly adjust the collection valve to the right setting by counting drops with a stopwatch.

Collect at least 250 ml of this first distillate and put to one side for future processing and then start to collect the pure alcohol in a clean receiver. Throughout this early phase test the distillate with your nose to see if you can detect any trace of heads.

The 250 ml or so of early distillate which have been put aside may be perfectly pure but the nose and the palate are extremely sensitive organs, particularly the palate, and you would quickly detect an off-flavour if it got through into your final drink. Play it safe, therefore, and put aside a generous portion of the initial distillate, even as much as 500 ml. It will not be wasted because, in a few weeks time, when a number of distillations have been completed and several litres of doubtful distillate accumulated, it can all be redistilled and really pure alcohol recovered from it.

When all the ethyl alcohol has distilled over, which may take as long as 20 hours, the temperature will start to rise as the higher boiling point "tails" appear. Experience will tell you when to expect this to happen and you should start switching receivers well ahead of this point so that only a small volume of alcohol will be contaminated. The last receiver containing a trace of tails can be added to the discard bottle for later purification.

When the fractional distillation is complete the packing in the column will be flooded with tails. These should be thoroughly washed from the column by pouring generous quantities of hot water down from the top.

When carrying out a fractional distillation for the first time the rate of production of pure alcohol will seem to be extremely slow. At a few drops per second one can believe that it will take forever to produce a reasonable amount and there will be a tendency to open the collection valve a little wider to increase the flow. Resist this temptation and be patient. The apparatus requires no attention and it is surprising how much alcohol is produced at a flow rate of 2 or 3 drops per second for several hours. Thus, at 750 watts input to the boiler and a draw-off rate of 270 ml. per hour, over 3 litres of pure, 95% alcohol will be obtained in a 12 hour day. This, when diluted with water and flavoured, will provide over 7½ litres of gin.

Single boiler system.

The preceding discussion has been based on the use of two boilers: a large one for beer stripping and a smaller one for fractional distillation. As discussed in an earlier chapter dealing with equipment, it is possible to make do with a single boiler of intermediate size. In this case proceed as follows:

Make 50 to 60 litres of "beer" and place half of it (say 30 litres) in the boiler. With the packed column in place and the collection valve **WIDE OPEN**, bring to the boil and start collecting distillate. Because the valve is wide open the rate of recovery of distillate will be much faster than it will be later during the second stage of fractional distillation. Also, because the packed column is in place, there will be some reflux and the concentration of alcohol in the distillate will be higher than after a normal beer stripping. The volume collected will therefore be somewhat smaller when the temperature reaches 100°C. and you switch off.

Drain the stillage from the boiler, flush out with a little water and add the remaining 30 litres of beer. Strip it just as you did with the first 30 litres. You will now have 8 to 10 litres of impure alcohol ready for purification. Drain the boiler and again flush with water. Add the partially purified alcohol to the boiler, plus a few additional litres of water to make sure that there is sufficient volume of liquid at the end of fractional distillation to cover the heating element.

Close the collection valve and reflux the high wine for several hours to equilibrate the column. Now proceed in the usual way, bleeding off the heads until the temperature stabilizes at just over 78°C. and there is no discernible odour. Then start collecting 95% alcohol at a reflux ratio of 10:1 and put aside the first several hundred ml for later re-distillation. From then on, completely pure ethanol will be dripping from the needle valve and, after dilution to 40%, will be ready for use.

Yield of alcohol: In the chapter on fermentation it was explained that the theoretical yield of pure, 100% alcohol from 10 kg of cane sugar is 6.25 litres. This is equivalent to 6.58 litres of 95% alcohol or 15.63 litres of 40% alcohol. While it is possible to approach such a yield you will find in practice that you only reach 70-80% of this value due to various losses along the way.

One place where you can expect losses to occur is in the fermentation process ----for example, you may not have left the brew long enough for all the sugar to have been completely used up. And then there are all those unwanted side reactions which produce the congeners such as methanol, fusel oils, etc., instead of ethanol. Another place where losses occur is in the last stages of beer-stripping where time and energy consumption require that the stripping cease long before the last drop of alcohol has been extracted. As a result, the practical yield of 95% alcohol is likely to be no better than about 5 litres which is a yield of 73% of the theoretical value. This is equivalent to 11½ litres of gin, which is not too bad.

In commercial practice such a low yield would not be tolerated, but for us it should be quite acceptable, particularly on economic grounds. Higher yields, which are certainly possible, offer an interesting challenge to the dedicated amateur.

Storage: Store your pure 95% alcohol in glass, not in plastic. A few $1^{1}/_{2}$ litre wine bottles with screw caps are ideal. There is, of course, no need to "mature" gin and vodka; it is ready for drinking the day you make it.

Flavouring

Before discussing flavouring a word must be said about the quality of water used to dilute pure 95% alcohol to the 40% which is characteristic of most spirits. Unless the water is very soft, hardness will precipitate out when alcohol is added because the calcium and magnesium salts which constitute the hardness are less soluble in an alcohol-water mixture than they are in water alone. Depending upon the degree of hardness the effect will vary from a cloudiness to a white precipitate which falls to the bottom of the bottle.

The effect described above is perfectly harmless, the white precipitate being nothing more than the hardness present in the original water before the alcohol had been added. It is actually quite good for you. However, it is aesthetically unpleasing and should be avoided by using distilled or demineralized water obtainable very cheaply from supermarkets and from certain stores which make distilled water on the premises. A further advantage of using it is that city water frequently contains chlorine which would interfere with the delicate flavour of a good gin or vodka.

Once pure alcohol is available there are many things you can do with it to prepare a pleasant drink. One is to mix it with fruit juices and make a tropical punch. Another is to prepare a liqueur by steeping fruit in an alcohol-sugar solution, a procedure which is fully explained in a number of books on the subject.

A third option is to purchase flavouring essence from a winemaker's supply store; these little bottles of essence come in a wide variety of flavours including rum, scotch, brandy, gin, etc. and most liqueurs such as the various fruit brandies, crème-de-menthe, etc. The fruity essences are particularly good, and the rum flavorings are quite acceptable, but the whiskies frequently leave something to be desired, having a somewhat artificial flavour. The quality may, of course, vary from manufacturer to manufacturer.

In this chapter we deal specifically with gin and vodka. The latter can be disposed of very quickly --- just add distilled water to the 95% alcohol coming from the still and hey presto, vodka! It will actually be a little purer than commercial brands and will have virtually no taste, so it can be used with confidence in any of those cocktails which call for vodka, e.g. a Bloody Mary, a screwdriver (vodka & orange) or a vodka martini.

And now let's talk about gin. As is rather well known the major flavouring ingredient of gin consists of juniper berries. There are other ingredients, however, and lists of such ingredients can be found in encyclopaedias and sometimes on the labels of commercial gins. Among the more important listed will be found:

Coriander	Cassis bark
Orris root	Ginger
Angelica	Nutmeg
Anise	Cinnamon
Cardamom	Bitter almonds
Lemon peel	

What is never mentioned is the quantity of each ingredient used in a particular brand, nor the exact method by which the flavour is extracted from the herb. These are closely guarded secrets of the manufacturer and the reason why amateurs have difficulty in duplicating a commercial gin.

Articles on gin-making stress the point that the country of origin of the juniper berries is important in determining flavour, as is the time of harvest and the weather prevailing during the growing season. The juniper berries are supposed to mature for 18 months or so after harvest and then used within a critical period of one week! It is all very reminiscent of winemaking. The amateur cannot possibly cope with such stringent requirements, but one is led to wonder just how much of these stated conditions is fact and how much merely folklore and a deliberate attempt to introduce a mystique into the operation. And if so, who can blame a manufacturer for so doing?

The amateur gin-maker is obviously on his own when it comes to flavouring, and it has to be admitted that we have never duplicated exactly the flavour and bouquet of a commercial gin. However, what we produce is very pleasing and there is the satisfaction of knowing that we have made it ourselves from authentic ingredients, so why worry? And then there is the continuing challenge of modifying the flavour by ringing the changes on the quantities of the various botanicals used. The flavouring step is the only one in gin-making which involves art rather than science and where there is scope for imagination, so the absence of a commercial recipe may not be such a bad thing after all.

Procedure

The flavouring in juniper berries and other botanicals is contained in oils which can be extracted either by alcohol or by steam. We have tried both methods, and both give very pleasant results, but it would be incorrect to say that the flavour is exactly like commercial gin. It is not. But many appreciative guests claim that it is actually superior to the commercial product. And who are we to contradict them!

The extraction method we use ourselves and recommend is the one involving steam distillation. It is very simple and consists of nothing more than boiling the botanicals in water and collecting the condensed steam. The equipment required is shown in Figures 8 and 9, one version being constructed from scientific glassware while the other is a cheap homemade version put together from copper tubing and a glass coffeepot.

The following recipe has been found to give a pleasant flavour:

juniper berries	35 grams	The juniper berries may be broken up in a
cardamom	1 "	blender before use in order to hasten the
orris root	1 "	extraction of the oils.
coriander	1 "	

Place the above ingredients in the flask, add about 350 ml of water, and bring to the boil. The steam generated will carry over into the condenser the oils contained in the botanicals. These oils can be seen as little droplets or globules in the collection bottle. Collect about 75 ml of condensate in one bottle and a second 75 ml in another. The flavour is slightly better in the first bottle. Switch off and discard the contents of the flask.

To each bottle containing 75 ml or so of distillate add an equal volume of 95% alcohol. This will dissolve the globules of oil and will also act as a preservative.

To use this flavouring essence, add about 10 ml to each litre of 40% alcohol. There is unlimited scope for trying to improve on this procedure

and on the recipe given above. Using other botanicals in quite different amounts is one obvious way to get a different flavour. The other is to extract the flavours from the herbs with a hot alcohol-water mixture, and use the essence directly without resorting to distillation. This has a major advantage in needing no special equipment but the gin would be slightly coloured. However, if you are of an experimental turn of mind, this approach would be well worth exploring.

Summary of Procedures

The detailed explanations provided in the previous pages are likely to give the impression that making alcohol is a pretty complicated business. But all it really consists of is adding yeast to sugar and distilling the resulting brew. Nothing to it. So let's just run over the procedures again, but as briefly as possible. The summary refers to the 2-stage process using two boilers.

Materials

Sugar and yeast. Flavouring herbs.

Equipment

Fermenter, beer-stripper, fractional distillation apparatus involving boiler, column and still-head. Simple pot still for extracting flavour from botanicals.

Fermentation

- 1. Clean the fermenter and accessories with soapy water and rinse.
- 2. Close valve under fermenter and place a rubber stopper in drain hole. Install circulating pump and add 10 kg of sugar.
- 3. Run in tap water and stir with wooden spoon to dissolve sugar. When water level is above the circulating pump start the pump, being careful to avoid any undissolved sugar crystals getting into the pump inlet.
- 4. Make up a yeast cream using 1 lb. of active baker's yeast in block form. Break into pieces and soak in a small volume of water for 15 minutes. Use mixing bowl, two wooden spoons and minimum amount of water to make the cream.
- 5. Pour in the yeast cream or sprinkle 150 g. of dry, powdered active yeast onto the sugar solution, close fermenter with glass cover-plate and install immersion heater and thermometer.

- 6. Switch on heater and raise temperature of sugar solution to 30-35 °C. Maintain this temperature for 5 days or until fermentation is complete.
- 7. When fermentation is complete, switch off pump and heater, reach down into the beer and replace the rubber stopper with the copper dam. Allow to stand for several hours (overnight?) to let yeast settle to bottom.
- 8. Run the "beer" into the beer-stripper via rubber hose.

Beer-stripping

9. Switch on the beer-stripper (boiler) and run cooling water through the condenser. It will take a couple of hours to come to the boil. Collect 14 to 16 litres of distillate, either in bottles for manual transfer or directly via a hose into the fractional distillation boiler. Temperature of vapour coming from stripper will have risen from about 80 °C to 98-100 °C. Drain and flush the stripper.

Fractional distillation

- 10. Close the collection valve in the still-head, run cooling water through the condenser and switch on the boiler. Be present when it comes to the boil to reduce heat input if necessary.
- 11. Reflux for several hours to equilibrate column. Check temperature. Periodically draw off a few ml. of distillate and sniff it to detect presence of "heads". Put aside for future use as fondue fuel.
- 12. When no more heads can be detected and temperature is staying constant in 78 79 °C. range, collect 300 ml. or so of distillate (at the predetermined rate of 1/10th total reflux) and put to one side for future redistillation.
- 13. Start collecting product until you know from previous experience that ethanol production will soon begin to cease. This collection will probably last 15 to 20 hours. Switch receivers towards the end and put aside for redistillation any receivers contaminated with tails.
- 14. Switch off, drain boiler, and flush out column from the top down with boiling water.

Redistillation

15. When sufficient discard ethanol has been accumulated, perhaps about 5 litres, pour it into the boiler of the fractional distillation apparatus and add an equal volume of water. Proceed exactly as in steps 10 to 14 above. The purpose of adding water is to prevent the boiler running dry since the discard alcohol contains very little water of its own.

Flavouring

16. Put the selected botanicals into a flask with ca. 350 ml of water, bring to boil and collect the condensed steam. Add an equal volume of 95% alcohol to the distillate in order to dissolve the flavouring oils and to preserve them from mold growth. Use about 10 ml of this essence per litre of 40% alcohol.

Costs & Economics

What does it all cost you ask? All that equipment and those elaborate procedures! The answer is --- quite a lot, perhaps as much as US\$1,000 or the equivalent if you buy everything new. Is it worth it? Well, that is a very individual decision and to help you decide, an estimate has been made of all the major costs involved.

The costs provided below refer to the United States in 1997, even though none of the experimental work and none of the purchases were made there. It is simply a shopping list of the things you will need with a rough idea of what you may have to pay. No sales tax is included. Undoubtedly in your own country you will find that some things are cheaper and some more expensive than they are in the United States. Even within a country prices can vary widely so it is up to you to shop around for the best deals.

Costs can be reduced by using, as far as possible, common domestic articles made for the mass market. For example, an ordinary light dimmer switch good for 600 watts is readily available and quite cheap, but similar controllers for high wattages are less in demand and are therefore much more expensive.. A sensitive domestic kitchen scale, graduated in 5 gram divisions, can be found if you shop around a bit and will be a tiny fraction of the cost of a scientific balance.

As in any manufacturing operation, even if it is only a hobby, the costs involved can be broken down into three main categories. They are:

CAPITAL MATERIALS & SUPPLIES LABOUR

Such a listing seems a little formal for a simple hobby so the same items can be re-worded as:

Equipment required Cost of sugar, yeast, etc. Time occupied by the hobbyist

Equipment

Only the cost of major items is listed below. Minor things like nuts and bolts, electric wiring, corks and stoppers, bottles for containers, plastic tubing, etc. are listed as miscellaneous and an estimated lump sum provided.

The three major equipment items are the fermenter, the beer-stripper, and the fractional distillation system. The little pot still for producing flavouring essence can be homemade for \$50 so hardly warrants being considered a major item. (Note: all costs have been given in US\$)

Fermenter

Laundry tub	\$20.00
Glass cover	\$30.00
Circulating pump	\$35.00
Electric heater	\$15.00
Light dimmer	\$4.00
Thermometer	\$10.00
Plumbing	\$10.00
Miscellaneous	\$20.00
Total	\$144.00

Beer Stripper

Water heater, 30 USG (113 litres), 3000 watts, 240 volts	\$110.00
¹ / ₂ inch copper condenser including T's, elbows and	
cooling coil, 3/4 inch union, adapters	
$(3/4 \text{ to } 1^{1}/_{2} \text{ inch})$, ball-valve	\$85.00
Thermometer	\$10.00
Miscellaneous	\$20.00
Total	\$225.00

Fractional Distilling System

Boiler:	
Water heater, 10 USG (40 litres) 1650 watts, 115 volts	\$139.00
Replacement heater for 750 watts	\$10.00
Voltage regulator (1000 watts)	\$45.00
Ball valve	\$6.00
Miscellaneous	\$20.00
Total for boiler	\$220.00

Column & Still-head: Glass column with joints top and bottom, Raschig ring	
packing, still- head, thermometer, condenser	\$250.00
Miscellaneous	\$20.00
Total for glass column	\$270.00
Total for copper column	\$155.00
Total for stainless steel column	\$450.00
Pot still for flavouring:	
Homemade model	\$50.00
Instruments:	
Volt-ammeter	\$45.00
Sensitive kitchen scales	\$15.00
Measuring cylinders (0 - 10 ml, 0 - 100 ml)	\$20.00
Hydrometer	\$6.00
Total	\$86.00
	·
Total for all Equipment	
Based on glass fractionating column	\$995.00
" copper "	\$880.00
" stainless "	\$1,175.00

Materials & Supplies

The following figures are based on the production of 11 one-litre bottles ($9^{1}/_{2} \ge 40$ oz.) of gin from 10 kg of sugar.

Sugar. 10 kg @ \$0.80/kg	\$8.00
Yeast. 150 g. @ \$5.55/kg	\$0.83
Flavouring ingredients - negligible cost	
Total	\$8.83
Electricity	
Fermentation negligible	
Beer-stripping8 kWh	
Fractional dist'n10 kWh	
Total: 18 kWh @ 7 cents/kWh	\$1.26
Total for material and supplies	\$10.09

Labour

It takes about 7 days from the time the fermentation starts to the time the collection of the pure alcohol is complete. During this period the amount of time involved in actually doing something with one's hands is probably no more than 3 or 4 hours. Periodically it is necessary to check a temperature or change a collection bottle but, to a large extent, the operation carries on quite happily by itself. It is not possible, therefore, to assign a cost to labour and we shall not attempt to do so here. In any case, being a hobby, it should be a labour of love!

Economics

So now we know what it all costs. The next question is ---- is it worth it? Well, we have made 11 litres of gin from \$8.83 worth of sugar and yeast and \$1.26 worth of electricity, so that works out at 92 cents per litre or \$1.05 for a 40 oz. bottle (1.14 litres). Not bad.

But how about all that equipment? Let us use the round figure of \$1,000 for its cost and see how long it would take to pay this off from the savings we realize on making our own gin instead of buying it. If we produce and consume 1 litre of gin per week it has cost us 92 cents against maybe \$20 if we'd bought it at a liquor store. So we save about \$19 per week. At that rate it will take us 53 weeks (a year) to break even. After that the equipment is free and the cost of the gin would be 92 cents/litre in perpetuity. A payback period of one year would be considered extremely good in industry where 5 to 10 years is much more normal. Note that if one were consuming 2 litres of gin per week the payback period would be only 6 months.

Another way of looking at the economics of investing in the equipment is to compare it with the investment required to purchase the gin commercially instead of making it. At a commercial price of \$20 per litre and a consumption of one litre per week the annual expenditure will be \$1040. It would require a bank deposit of \$30,000 to generate this \$1040 assuming a 5% interest rate and taxation on the interest of 30%. So what it would boil down to is the question ---- would one rather put aside \$30,000 in a savings account, earn \$1500 in interest, pay \$450 in tax and buy commercial gin with what is left or would one rather lay out \$1,000 on equipment and use the \$30,000 in some other way?

A considerable reduction in equipment costs will be possible if you already have facilities for carrying out a fermentation and if you adopt the single boiler option. Under these conditions you should be able to bring the costs down below \$500.

To allay the concerns of the tax authorities who may fear that the equipment and process under discussion might be used for illicit commercial production of distilled spirits, consider the following: A full-time operation with this equipment could only produce 500 litres per year and would generate only \$10,000 if each bottle were sold for \$20. Being illicit, the selling price would likely be no more than \$10, leading to total sales of \$5,000. From that must be subtracted the cost of materials and the labour involved, suggesting that anyone considering going into the moonshining business would be well advised to take up some other line of work.

APPENDIX I

Conversion Factors

Throughout the text you will find an awkward mixture of metric units and the foot/pound/gallon system still used extensively in North America. Different individuals, depending on age, occupation and whether they live in a British Commonwealth country or the United States, will use a different mixture of the two systems. So, for everyone's convenience, a list of conversion factors is provided below.

Volume	 Imperial gallon Imp fluid ounce Imp fluid ounces U.S. gallon US fluid ounce US fluid ounces litre 	= = 1 Imp pint = = = 1 US pint = = =	 4.55 litres 28.4 millilitres 568.1 millilitres 3.78 litres 29.6 millilitres 473.6 millilitres 35 fluid ounces Imp 0.22 Imp. gallons
		=	0.26 U.S. gallons 1.04 U.S. quarts
	(TIP Instead of converting to or from Imperial to US units for volume, just count all measures in fluid ounces - they are practically equivalent, e.g. 1 US quart = 32 US or 32 Imp fluid ounces (near enough)		
Weight	0.8.1 0.8 quart 0.2 0.8 0		
	1 pound (lb) 1 ounce (oz) 1 kilogram (kg) 1 gram (g)	= = =	454 grams 28.4 grams 2.2 pounds 0.035 ounces
Length	1 inch 1 foot 1 centimetre 1 metre	= = =	2.54 cm 30.48 cm 0.39 inches 39.37 inches
Temperature	32 °Fahrenheit (F) 212 ° "	= =	0 °Celsius (C.) 100 ° "
	General:	$[^{\circ}F 32] \times 5/9 =$	°C.
Pressure	1 atmosphere	= = =	14.7 lbs/sq.in. (psi) 29.9 inches of mercury 760 mm " " 101.3 kilopascals (kPa)
	1 psi	=	6.9 kPa

Appendix II Activated Charcoal

Most amateur distillers are familiar with activated charcoal, using it to remove some of the more noxious substances present in their crude spirit. An ordinary pot still - the standard type of equipment used by amateurs - is incapable of producing pure alcohol so activated charcoal remains the only hope of cleaning it up and producing a palatable beverage.

By contrast, the alcohol produced with the equipment and procedures described in this book should conform to the definition of vodka given by the Bureau of Alcohol, Tobacco & Firearms (the BATF) in the United States, i.e. "a neutral spirit so distilled as to be without distinctive character, aroma, taste or color"*. If properly made, therefore, it should not require a charcoal treatment.

Mistakes can happen, however, particularly in the early days before one has gained experience, and when it does one may be faced with a batch of alcohol which is slightly "off". In such cases a polishing with activated charcoal can be beneficial.

Activated charcoals are 'custom built' for their end purpose, generally involving careful selection of ingredients and very high temperature and gas treatment. They work by physical adsorption (not absorption) on the enormous internal surface area of the carbon, typically 1,000 square metres per gram (hard to believe, but true!). Note that it is a physical and not a chemical effect that makes them work. It pays to be very careful about choosing the source and type of activated carbon you use to clean a spirit. Aquarium carbon will **not** do! It is an impure substance not designed to be used with products intended for human consumption, and it may well introduce rather nasty trace elements and flavours to your hard won product. Properly sourced activated charcoal is now readily available from winemakers' suppliers, specifically designed for the purpose of cleaning and 'polishing' spirits.

To use it, dilute the alcohol from 96 to 40% (vodka strength) and use about 150 grams of charcoal per 6 litres. Put into a container, stir occasionally over 5 days, allow to settle and then filter. Alternatively, make a continuous filter by clamping filter paper over the end of a length of $1^{1}/_{2}$ inch pipe (preferably not plastic), add charcoal to a depth of a foot or so, and then pour the alcohol through. It should be completely pure when it emerges.

Used charcoal can be regenerated by rinsing with water, spreading on a metal baking sheet and heating in an oven at 135 $^{\circ}$ C (275 $^{\circ}$ F) for several hours. The pungent smell of adsorbed congeners being driven off will be very apparent and will demonstrate that the charcoal has done its job.

* An interesting conclusion to be drawn from this definition is that either:

- a) all commercial vodkas are identical, or
- b) the various brands have been delicately (and differently) flavoured.

(It may be noted that in Russia, hundreds of differently flavoured vodkas are available!)

Appendix III

Distillation - How it Works

The mechanism by which alcohol can be purified by distillation is a subject shrouded in mystery for most amateurs. There are those who know that the process involves the boiling of a dilute, impure alcohol and separating the various constituents by means of the difference in their boiling points, but just how or why this separation takes place is known only vaguely.

Take a mixture of methanol, ethanol and water for example. The first has a boiling point of 64.7 $^{\circ}$ C while the second boils at 78.4 $^{\circ}$ C. So it is thought that by heating the mixture to 64.7 $^{\circ}$ C and holding it there the methanol will boil off. Raise the temperature to about 78 $^{\circ}$ C and the ethanol will boil off, leaving the water behind. This is completely wrong and has led to many disappointments.

In this book we have attempted to shed a little light on the subject, but it is apparent from readers' comments that there is an unsatisfied thirst for additional information. In this discussion, therefore, we shall go into the mechanism of distillation in somewhat more depth. Let's start by talking about vapour pressure.

Vapour pressure.

All liquids (and solids too for that matter) have a vapour pressure. That's why we can smell them — molecules escape from the surface and penetrate our nostrils. Every substance is a collection of molecules held together by mutual attraction and vibrating about their mean position. The higher the temperature the faster they vibrate.

At the surface of a liquid vibration enables some of the molecules to escape the clutches of their neighbours in the body of the liquid and enter the vapour phase, and the higher the temperature and the more the molecules vibrate the greater the number which are able to escape. The vapour pressure of a substance is the contribution these freed molecules make to the pressure of the surrounding atmosphere. This may be illustrated by a simple experiment. Take a glass tube about a metre long, closed at one end, and fill it with mercury. Upend it in a beaker of mercury and the mercury in the tube will fall and leave a vacuum above it. The column of mercury is being held up by the pressure of the surrounding atmosphere and the height of the column is a measure of the atmospheric pressure.

Now introduce a few drops of water into the bottom of the tube. The water floats to the top of the mercury and will be seen to boil rapidly. Continue adding water until there is some liquid water floating on the mercury and you will notice that the mercury column has been lowered by about an inch. This is the <u>vapour pressure of water at that temperature</u>. If the temperature is <u>raised</u> the V.P. will increase also, and when ca. 100 °C. is reached the mercury level in the column will be the same as in the beaker and the column will be full of water vapour. Repeat the experiment with methanol instead of water and you will find that

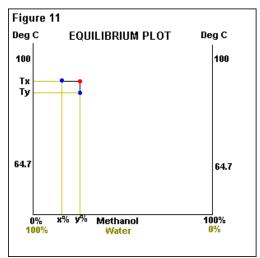
the tube will finally be empty of mercury at 64.7 °C, the boiling point of methanol. The vapour pressure of a liquid at its boiling point equals atmospheric pressure.

Latent heat of vaporization.

It takes a certain amount of energy to raise the temperature of a liquid from, say, room temperature to its boiling point, but it takes very much more energy to convert the boiling liquid into vapour, even though the temperature stays the same. This energy is called the latent heat of vaporization and is large because it has to work against the mutual attraction of the molecules in the liquid and provide them with enough kinetic energy to remain apart. So you cannot raise the temperature of boiling water by pouring more energy into it — it will stay at 100 °C.

Mixtures.

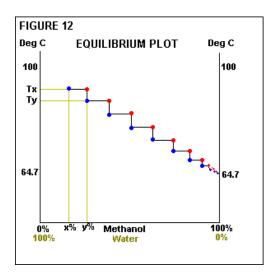
Take pure methanol, B.P. 64.7 °C. and start adding water. The boiling point of the mixture will rise the more water you add, indicating that the molecules — both water <u>and</u> methanol —are finding it more and more difficult to escape from the mixture to form vapour. The water molecules exert a higher attraction than the methanol molecules and this attraction extends to <u>all</u> the molecules in the mixture. This is the crux of the distillation process — that a mixture boils at some temperature depending on the relative concentrations of its constituents and produces a vapour which is a mixture of the two. The constituent with the higher vapour pressure will contribute more molecules to the vapour than will the constituent with the lower vapour pressure. In the case of a methanol/water mix, whatever mixture you started out with you end up with a vapour which is richer in methanol than water. Condense this vapour and then re-boil it and the result will be a vapour with yet more methanol than before. This process is illustrated in the diagrams below.

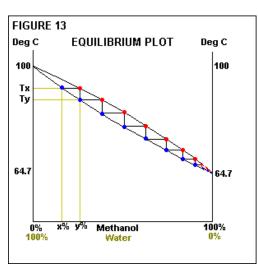


Take a mixture of methanol and water with X% methanol by volume. The top left dot charts the boiling point of the liquid <u>mixture</u> as being Tx C.

It must be emphasised again that the boiling point of a mixture is <u>not</u> the boiling point of either of the constituents, but lies somewhere in between (if in any doubt about this, please read again the paragraphs above.)

The vapour from this mixture contains more methanol than the mix it came from, let's say Y% methanol (shown by the second dot at Tx °C) and this new mixture condenses at Ty °C. Note that this new mixture with more methanol condenses at a <u>lower</u> temperature than it took to boil the mixture it came from.





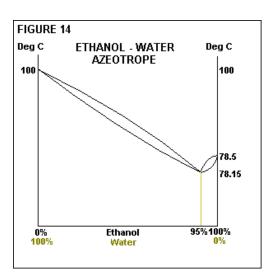
We now take this condensed liquid and heat it again until it boils. Once again, the vapour contains more methanol than it did before in mixture that's boiling, and that this vapour will therefore condense at an even lower temperature.

Subsequent vaporizations and condens-ations are plotted in this chart. As the concentration of the condensed liquid approaches 100% methanol the boiling point, as might be expected, decreases at each stage and eventually approaches the boiling point of pure methanol 64.7 °C.

Joining up these dots gives us two curves. The upper one may be called the vapour line. Anything above it is vapour above the boiling point for that mixture, and anything below the lower liquid line is liquid below the boiling point for that mixture.

It is sometimes said that any point lying in between the lines represents a transition phase between liquid and vapour, but a little reflection will show the fallacy of this view. We chose to start at a certain concentration of methanol, but another concentration would have resulted in a similar set of points offset either to the left or right of those shown.

An area where vapour condenses, hangs around and then vaporizes again is termed a 'plate'. It may be likened to an actual plate or tray fitted in a column.



The diagrams above relate to a methanol-water mixture, and are quite simple. In the case of an ethanol-water mixture we would find that there is a kink in the bottom of the curves. This results from the fact that ethanol and water form azeotropic mixture when an the concentration of ethanol is around 95%. Subsequent vaporization of liquid at this concentration will not yield vapour with a higher concentration of ethanol but one of the same concentration as the liquid. If we started with a mixture that had more than 95% ethanol, then the concentration of the vapour would be less and, once again, the system would tend to settle at the azeotropic point. Distillation alone cannot give

concentration of ethanol higher than 95%

So that's what happens when we heat a mixture of two volatile liquids. The constituent with the highest vapour pressure will appear in greater and greater quantity in the vapour as we boil the mixture, then condense it, then repeat the boil-condense cycle over and over again.

So the question is, how do you get enough cycles of boiling-condensing-boiling into a device which is suitable for use by amateurs. The answer lies in using a column packed with surfaces where the vapours rising from the boiler meet the liquid falling from the condenser in the still-head. At each surface the hot vapour gives up its latent heat to the descending liquid and re-vaporizes it. So one gets a whole series, probably many hundreds, of mini-distillations down (or up) the length of the column. As noted in the book, it is possible to provide the very large surface required by packing the column with stainless steel filaments.

Reflux and Balance.

A column packed as described will enable the boiling-condensing cycle to be repeated many times and the constituents of the original mix will start to separate out, the most volatile at the top. Condensed liquid that runs back down the column is termed the reflux. It is richer in the most volatile constituents than the vapour rising to meet it, and you will recall that its boiling point is lower than the vapour further down in the column. It therefore boils as it passes down the packing and the resulting vapour is even richer in the volatile constituents.

This process may be 'hurried along' by condensing out all the vapour that reaches the top of the column and returning it as reflux. By this means, the most volatile constituent of a mix is concentrated in the top section of the column, the less volatile constituents being confined to the lower section. A high degree of purity is achieved in this manner.

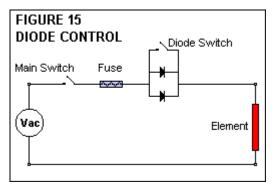
The process of separation takes time as many cycles of boiling-condensation have to occur before the lightest constituent is fully isolated in the top section of the column. When no further variation in concentration of the various constituents occurs along the length of the column, the column is said to be in balance. As the boiling point varies according to the relative mix of the constituents, it follows that the temperature of the column will be high at the bottom and will decrease the higher you go. When the column is balanced then the temperatures along the length of the column are stable and exhibit no variation with time. The top section of the column will be at the boiling point of the most volatile constituent.

With the column balanced, a start may be made on withdrawing the lightest constituent condensed at the top. However, only a small amount of the total condensed may be withdrawn if balance is to be maintained. The quantity withdrawn compared to that which is supplied is termed 'Reflux Ratio'. As noted in the book, experience has shown that a reflux ratio of 1:10 in a column about 1 metre long and between 25 and 35 mm diameter gives consistently good results.

Appendix IV

Heater Control Using Diodes

Care must be taken to choose a diode that will cope with both the voltage presented to it and the current it will pass. The calculations are quite straightforward and use only one simple equation: W=VI, where W=watts, V=voltage, and I=current. The current passing through a 240 volt 1500 watt heater is therefore 6.25 amp, or through a 120 volt 1500 watt heater 12.5 amp. However, this is just the average current ('Root Mean Square' or RMS value for a sinusoidal supply). The peak current is the square root of 2 times this value, or 8.84 amp with a 240 volt supply, or 17.68 amp with a 120 volt supply. Similarly, the peak voltage is 340 volt for a 240 V(RMS) supply, or 170 volt for a 120 V(RMS) supply.



We must choose our diode with these peak values in mind. A commonly available power diode is rated at 600 volt 10 amp. This would cope alone with a 240 volt supply, particularly with the voltage, but it is always a good principle to use a component at only around 50 to 60% of its rated value. Two diodes in parallel would have to deal with only half the current each, three diodes in parallel one third each, and so on. So a good choice for a 1500 watt heater on a 240 volt supply would be two diodes in parallel, and for a 120 volt supply three diodes in parallel. These diodes should be mounted on a heat sink - a small metal sheet would suffice as they are operating well within their rating and should not warm up very much.

The resulting circuit would look like the diagram above (for a 240 volt supply). Note that the switches must be rated to cope with the voltage and current as well, so should be at least rated at the mains voltage used and the peak current. A fuse is always a very good idea, and it is strongly recommended that a 10 or 20 amp one be used, depending on whether the supply is 240 or 120 volt. The importance of good insulation and safety cannot be stressed too highly. All electrical parts should be well insulated or shielded so that casual contact cannot be made when mains voltage is applied. Always triple check a mains circuit to satisfy yourself that there are no short circuits or stray leads before switching on power.

The Authors

John Stone

John Stone has his Ph.D. in physical chemistry from the University of London, England and has published over seventy scientific papers. Before retiring he was the Director of Research at the University of Ottawa and before that the Director of the Forest Products Laboratory, both in Canada.

His interest in the theory and practice of small-scale distillation stems from a botched attempt at making wine. It was so awful that it should have been poured down the drain. However, he decided to try and recover the alcohol by distillation, finding that there was a lot more to it than he'd imagined. This "how to" book is the result.

Michael Nixon.

Mike is a Chartered Engineer, a Member of the Institution of Electrical Engineers. His grounding was in physics and chemistry, leading to a career in electronics as an engineering officer in the Royal Air Force in England. Investigation into equipment reliability led him to question many assumptions made in design, and this in turn led to a certain degree of cynicism when faced with claims made for equipment that turned out to have no foundation in fact. His interest in home distillation was sparked for this reason. His collaboration with John Stone grew from a desire to participate in writing a book that provided reliable scientific information to those who were genuinely interested in the subject.

