# Making Gin & Vodka \*\*\*

A Professional Guide for Amateur Distillers

> loy John Stone



# **FOREWORD**

Making pure ethyl alcohol at home is 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, 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 then, instead of drinking the brew, it is subjected 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 sparkling, crystal clear alcohol of almost pharmaceutical quality.

The pure alcohol is then diluted with water to 40% and used as such (vodka), or flavored 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 pleasant after-dinner liqueur. The freedom to make spirits extends considerably the range of beverages available to the amateur and he/she is no longer restricted to just beer and wine.

Although it is illegal in most countries for amateurs to distil alcohol, and even illegal to own the equipment amazingly enough, fortunately it is not illegal to write about it or read about it. The purpose of this book therefore, like that of its predecessors, is to open up the subject to intelligent discussion. This it will do by describing in detail how to construct the equipment, followed by a description of how to use it to make vodka. The reader will then know, from a complete understanding of the subject, how the present attitudes of officialdom are based on a completely false premise.

It might well be asked why anyone should bother to read about a procedure which is illegal, or learn how to build equipment which it's illegal to own. The answer is that this is the first step, the necessary step, in changing the law so that such an innocent hobby becomes as legal as making beer and wine.

New Zealand has recently (1996) legalized amateur distillation, probably as a result of its isolated location in the south Pacific and freedom to think for itself. It does not have to march in lockstep with the hidebound democracies of Europe and N. America. Surely the rest of the world must follow New Zealand's lead soon if it is not to look ridiculous. However, governments are notoriously slow to change and it will take persuasive arguments to overturn entrenched opinions. For those crusaders who wish to embark on such a noble task it is imperative that they know the facts thoroughly and can dispose intelligently of the myths which surround the subject of distillation. This book will provide such persons with the ammunition they need.

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# **INTRODUCTION**

Many books are available to amateurs on the methods and equipment involved in making beer and wine, and such books can be found in abundance in most bookstores and in beer- and wine-making supply stores. However, when it comes to the use of a small still to produce distilled spirits it is no use looking in bookstores. To find books on this subject it is necessary to search the Internet for independent publishers, but then we run into another problem. The books which are found on the Internet invariably deal with the production of whiskies, a spirit which may be quite enjoyable when well prepared but which also can be harsh to the point of being undrinkable.

What has been missing is a literature dealing with the production of the very pure ethyl alcohol used for making vodka and gin. The same pure alcohol is used in chemical laboratories, the pharmaceutical industry, and in the production of perfumes and colognes, etc. This book has been written in an attempt to rectify such an anomalous situation because the starting point for many drinks — vodka, gin, liqueurs, punches — is an alcohol which can provide the "high" without contributing any flavour of its own. Moonshine cannot do this because its own flavour is far too harsh, and the strange little moonshine stills which are offered for sale on the Internet will certainly lead to disappointment if pure alcohol is what you are looking for.

The two previous books in this series have been well received, but the advantage of short printing runs is that it is possible to make improvements with each edition. In line with this thinking the present volume will provide some additional information on both the theoretical and practical aspects of distillation, and will describe a simplified 2-stage procedure using less equipment which will save both money and space.

The production of extremely pure alcohol is rather simple as it happens, far easier in fact than making a spirit of lesser purity such as whisky, rum or brandy. It is even simpler than making beer or wine. This should be encouraging for those who have never embarked upon distillation and are worried that it might be a bit too technical and equipment-oriented. The explanation as to why it is easier to make a pure alcohol than an impure one will become apparent in the next chapter.

The book should appeal to two groups of readers: 1) those who live in countries where it is currently legal to distil alcohol for one's own use, New Zealand being the best example although there are some others in eastern Europe. And 2) the rest of the world, particularly western Europe, N. America and Australia, where the laws respecting distillation by amateurs need to be challenged since they are based upon a false premise. This premise is that distillation produces a highly intoxicating alcohol, whereas the truth of the matter is that distillation doesn't produce any alcohol at all. This statement is not made merely to be controversial and argumentative, it is a simple fact. Distillation does not make alcohol. It never has, never will, and is incapable of doing so.

The first group will find complete details of the equipment and procedures required to a) ferment ordinary table sugar (sucrose) to a crude "beer" using bakers' yeast and b) the steps involved in fractionally distilling this beer to remove all the impurities. The alcohol so produced is a sparkling, crystal clear vodka. Instructions follow for flavouring the vodka with juniper berries and other herbs and botanicals to produce the well-known bouquet of London Dry Gin. There are also suggestions for making a wide variety of alcoholic drinks by the simple expedient of adding the appropriate flavouring agent.

The second group can use the same detailed information in its campaign to get the law changed. Such campaigns will only succeed if they are 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 thoroughly muddled about distillation — with what it is and what it isn't. They are certain, for example that distillation makes alcohol. It doesn't. They are equally certain that distillation is a dangerous practice which is liable to lead to blindness. It won't. When faced with such charges it is necessary to have all the facts at your fingertips, to be an authority on the subject, because then you will be in a position to counter such silly arguments in a convincing manner.

This book must not be seen in N. America and elsewhere as any sort of incitement to break the law. Far from it. The law has to be changed, not broken, and to change the law it is necessary 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 brainwashing combined with simple ignorance.

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.

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 procedures. We make no apologies for such overlap since it helps to make the various chapters self-sufficient. Also, repetition of the fact that distillation is simply a purification process and doesn't make alcohol can be excused on the grounds that repetition is not a bad thing if we wish to clear away the misinformation hammered into people's minds over the centuries 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, letting the reader's ingenuity fill in the gaps. It was decided, however, that a knowledge of why something works or doesn't work 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, or if you have modified the equipment and procedures described in the book (which many people do). It then becomes possible to solve the problem through knowledge rather than by trial and error.

The units of measurement to use present a problem. It will be much easier when the whole world uses the metric system, but many countries in the English-speaking world, particularly the United States, is largely non-metric. In this book, therefore, we have adopted an awkward hybrid system in which most volumes, weights, temperatures and pressures are in metric units while some dimensions, e.g. pipe diameters, are in inches. For convenience a table of conversion factors from one system to the other is provided in Appendix I.

Before getting down to the 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, as we all know, they cover an extremely wide range of products from wines and beers to whiskies, rum, brandy, gin, liqueurs, etc., and a very wide range of starting materials, from grapes to potatoes to milk. The common denominator which ties them all together is the alcohol itself, a pure chemical with the empirical formula  $C_2H_5OH$ .

# **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.

$$C_6H_{12}O_6 + yeast \rightarrow CO_2 + C_2H_5OH$$

Usually one does not start with a pure sugar but with fruit juices for wine, the starch in grains for beer and whisky, the starch in grain or potatoes for vodka, 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 even though the flavour and colour will be different.

In addition to the variations imposed by the source of sugar, the particular strain of yeast and the conditions under which it is used (temperature, nutrients, etc.) 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 drink.

No alcoholic beverage (with the exception of certain vodkas made in n. America) consists simply of alcohol and water with no other constituent present. If it did it would be colourless, odourless and tasteless. And rather boring to many palates unless you mixed it with something which had a flavour, e.g. vermouth for a martini, tomato juice for a Bloody Mary, orange juice for a Screwdriver and so on. Liqueurs too, normally use vodka as the alcoholic base.

The colour, aroma, and flavour of beers, wines and spirits are due entirely to the other constituents present, the alcohol having nothing to do

with it. These other constituents are known collectively as "congeners". Many of these congeners are relatively harmless but there are always a few produced during fermentation, even during the fermentation of a fine wine, which are actually poisonous. Methanol (rubbing alcohol) is one of them. Fusel oils are another. 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. You will never get a hangover from drinking vodka, but you will from beer, wine or whisky. More will be said about this interesting and little known fact in the next chapter dealing with health and safety.

### 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 permits the removal of some of the more noxious congeners by discarding some of the first liquid to distil over (the "heads") and the last (the "tails"). The middle fraction of congener-laden alcohol remains and is collected.

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. It would be nice if, after a fermentation had gone slightly wrong and the beer or wine were found to have an unpleasant taste, the offending congeners could be removed. Alas, science has not yet come up with a method for doing this. Which means in practice that beer- and winemaking must be carried out extremely carefully because you are stuck with whatever you've produced. Beer- and winemaking are highly skilled operations, 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?

To distil a liquid one simply brings it to the boil and condenses the vapour on a cold surface. To remove the hardness from water it is 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 calci-

um and magnesium salts which constitute the hardness are non-volatile and 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. Nor is it something to be furtive about, something to discuss with your friends in hushed tones. 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 although the principle is exactly the same — boil the liquid and condense the vapour — and later chapters will provide an exact description of the equipment required and the procedures involved. Emphasis will be placed on the production of high purity alcohol such as used in vodka and gin, but alcohol containing congeners for providing flavour, both good and bad, can be produced if that is what you want.

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. Furthermore, if all the "impurities" were removed there would be no taste and you would have produced vodka and not whisky. The other type of still is more elaborate in design and used for making pure alcohol. 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 right at the outset.

# 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. Analytical methods such as chromatography reveal that there are literally hundreds of compounds present after a fermentation. 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. The type of still used for making whiskies, brandies, rums and so on, all of which require that a percentage of taste-giving congeners remain, are called pot stills.

To make brandy (as an example of a distilled spirit) the fermented liquor (wine in this case) is brought to the boil and the vapours led over into the condensing section. This section contains a cooling coil with water running through it where the vapours are condensed to liquid. The first vapours to come over will be rich in the more volatile components such as acetone and methanol. 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 is 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 and include propyl, butyl and amyl alcohol. These three alcohols are known 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 ethanol contaminated with a little heads and tails, the amount of each depending on just where the cut-off is made.

## Whisky, brandy, rum, etc.

The distiller of these products uses a simple pot still or a pot still slightly modified to give a small amount of reflux (see next section). Such stills effect only a crude separation of the fermented liquor into heads, tails and a middle fraction. The skill in making a palatable whisky consists of a) fermenting the mash under a carefully controlled set of conditions to generate a particular mixture of organic compounds, followed by b) distilling the mixture and discarding a portion of the heads and a portion of the tails. For example, you wouldn't wish to drink the acetone and methanol which arrive first but you might wish to retain some of the congeners which arrive immediately afterwards. 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 of each batch, and since the amount retained is controllable, the flavour of the final whisky is affected accordingly. At this point there is no colour and the fiery liquid will look like water. Colour is imparted by storing the spirit 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 which has taken years of trial-and-error, taste panels, and feedback from consumers to reach the point where it is today. It has involved the production of a complex but controlled mixture of compounds 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 undoubtedly have left behind a number of exceedingly unpleasant constituents. Additionally, in order to increase the quantity of saleable product the moonshiners would have been strongly tempted to retain an excessive amount of the more noxious heads and tails.

Similar problems would face the amateur whisky-maker today without proper guidance, but for amateurs who wish to try their hands at making a corn whiskey there is an excellent book available on the subject written by Ian Smiley (see www..)

#### 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 a boiler. The column is packed with small pieces of an inert substance, e.g. short lengths of glass or ceramic tubing (known as Raschig rings), ceramic saddles, wire gauze, or in fact any non-reactive material with a large surface area and a large number of small pockets where liquid can accumulate.

The vapours from the boiling liquid rise up the column, are condensed to liquid in the stillhead at the top, and run back down through the packing in the column to the boiler. 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, ceramic 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 and the least volatile components emerge last.

### Vodka

To make vodka, fractional distillation equipment along the lines of that discussed in a later chapter must be used. The strong (190 proof), pure alcohol so produced is diluted with water to 40% to give vodka.

In sharp contrast to all other spirits, most vodka, particularly the vodka made in N. America, is made from pure alcohol, i.e. alcohol from which all the heads and tails have been removed. The US Bureau of Alcohol, Tobacco & Firearms (BATF) defines vodka as "A neutral spirit so distilled as to be without distinctive character, aroma, taste or color".

If the BATF definition is taken literally, it would mean that there should be no difference between vodkas made from potatoes, grains, wine, milk or any other fermentable sugar. Why then is there so much advertising hype about the unique qualities of a vodka from, say, Sweden, or Poland, or Russia, etc., etc.? If there's no difference, why then all the talk about triple distilling, carbon filtering, and so on? Or the difference between vodkas made from potatoes and grain? The following quotation from the London Daily Telegraph of June 14, 1997 is interesting in this connection, "Aleksander Orekhov, the Russian-born owner of Red, a Soho bar that offers some 40 different vodkas, makes no apology for saying that the best vodka is one that has no real flavour at all". In line with this thinking it may be noted that some manufacturers choose to use the lactose in milk to make vodka, not just because it is available locally but also because it gives no flavour to the vodka.

The fact seems to be that most vodkas, at least outside N. America, do have a slight flavour. They are lightly flavoured by the manufacturer using certain grasses or herbs, so delicately that it can barely be detected, in which case the source of the flavouring is not mentioned. Or glycerine is added to give the vodka smoothness and body. The use of such additives is allowed to remain a subtle mystery in order to tempt the palates of vodka aficionados around the world. Recently, however, more strongly flavoured vodkas have been introduced into the market, with flavours which include raspberry, strawberry, peach, vanilla, lemon, vanilla, coffee, cinnamon, pepper,

and so on. No mystery here — they are advertised as lemon vodka, etc. Such practice makes eminent sense — use pure alcohol, add a natural flavouring (of which there are hundreds, if not thousands) and you have a unique and pleasant drink with no congeners, no methanol, no fusel oils, nor (as will be discussed in the next chapter) any headaches or hangovers.

Another, more traditional way to make a delicately flavoured vodka, is to carry out a slightly "imperfect" fractional distillation so that very small amounts of the natural flavours in the original source of carbohydrate — potatoes, grain, etc. — are retained. This is much more tricky than making a pure, unflavoured alcohol because it involves a subjective judgement on the part of the distiller on what constitutes a pleasant taste when traces of the heads and tails are retained. The acquisition of such judgement requires many years of experience combined with constant feedback from satisfied or dissatisfied customers.

#### Gin

Gin is really nothing more than a special case of a flavoured vodka, the flavouring agent in this case being mainly juniper berries but also small amounts of other botanicals such as orris root, cardamom, coriander. Different distillers use different recipes, which accounts for their slightly different tastes. In a later section of the book a description will be given of the equipment and procedure involved in steam-distilling juniper berries and other herbs to produce a flavouring essence which can then be added to vodka to produce gin.

### **Summary**

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 the correct procedures followed. There are no subtleties involved such as quality of grapes or the type of yeast used. The starting material can be corn, potatoes, grapes, wheat, rice, milk, molasses — in fact anything which contains a fermentable sugar. One hardly even needs to worry about hygiene; just add large amounts of bakers' yeast to a solution of sugar and stand back. The sugar will be rapidly fermented to a crude alcohol known as "beer" in the trade, and then this "beer" is fractionally distilled to

remove all the extraneous, noxious substances to leave a clear, sparkling, 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, and shall no doubt say again, the quality of these beverages depends upon the presence of compounds other 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. The only difference between a cheap bottle of "plonk" and a vintage chateau-bottled wine costing an arm and a leg is a very slight difference in the congener make-up, and the only difference between a rot-gut whiskey and a single malt, lovingly produced in the Highlands of Scotland and aged for donkey's years, is the difference in the congeners. No such considerations apply in the case of gin and vodka. The "beer" produced by adding bakers' yeast to a 20% solution of cane sugar would be completely undrinkable by all but the most dedicated tipplers, but fractional distillation will rid the mixture of all the congeners, all the undesirable compounds, and produce 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. No aging is required, — gin and vodka are ready to drink the day you make them.

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 for gin, and from various fruits and herbs for liqueurs and punches. And this is simply a matter of personal taste and preference.

It is also worth mentioning here that, in addition to using one's own natural ingredients to flavour alcohol, ready-made flavouring essences can be purchased from beer- and wine-making supply stores. These essences cover a very wide range, from fruity liqueurs to whisky, rum, brandy, etc.

As a final word of encouragement, a litre of vodka can easily be made from 1 kg of sugar. So, depending on the price of sugar where you live, the cost of all the ingredients to make a litre of 40% vodka will be about \$1 (U.S.).

# **Health and Safety**

The three major concerns of people who might be interested in setting up a still at home are 1) the question of legality, 2) the possibility of getting poisoned, specifically of going blind, and 3) the danger of blowing one-self up. These are serious concerns, and people take them very seriously. In the next chapter the legality question will be dealt with at length, but for the moment the emphasis will be on health and safety.

### **Poisoning oneself**

The belief that there is some inherent danger in distilling one's own spirits is widespread and is reinforced whenever the news media report that a number of people have been taken ill, or even died, as a result of drinking homemade spirits. People associate "homemade spirits" with distillation, with moon-shining, but in fact there is no danger whatsoever in drinking home distilled spirits, or even moonshine properly made. The danger lies in buying liquor from a bootlegger because in order to increase his profits he may top up his moonshine with rubbing alcohol (methanol), or stove oil, or antifreeze or paint remover or any other pungent liquid he can lay his hands on. Naturally such a cocktail is poisonous, but don't be mislead into thinking that the toxicity is due to simple ignorance or lack of care on the part of the backwoods distiller. It's not. It's due to these gentlemen adulterating their booze and fobbing it off on an unsuspecting public.

Another possibility is that the moonshiner will use automobile radiators for cooling the vapours rising from his boiler, and radiators frequently contain lead soldering, so lead may get into the alcohol. Obviously there is no government supervision of a moonshiner's operation, so caveat emptor—let the buyer beware!

Our recommendation is that you never buy moonshine made in an illegal and unsupervised still, possibly adulterated with unknown chemicals. Make your own if it's legal to do so, in which case there will be no danger whatsoever to your health. This is particularly true of fractional distillation,

where you have removed ALL the impurities, but also for simple distillation where you have removed at least some of them. Your equipment will be made of glass, stainless steel or copper, and if made from copper the various parts will be joined with lead-free solder. It would be similar to a Scotch whisky distillery where copper stills have been used for centuries. As for dangers in the distilling operation itself, let us follow this through. Sugar is fermented to alcohol using bakers' yeast to make a crude "beer". No danger so far, right? The beer is boiled and the vapours collected. The first liquid to come over will contain some methanol (poisonous), acetone and small amounts of other substances which were in the original beer, the so-called congeners. They smell like paint remover and will be discarded. Then comes the potable alcohol which has no smell and is collected for use. Finally there arrive the fusel oils with a somewhat unpleasant odour so they, too, are discarded. Remember, the distillation has not created anything, it has simply separated out the noxious substances from the beer — the heads and tails.

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 to put it mildly.

## Headaches and hangovers

Headaches and hangovers are well-known consequences of overindulgence in alcohol, but what is far 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 the 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 were 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 above 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 — and not methanol as might have been expected.

These results are not really definitive — for one thing the size of the sample was rather 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 alcohol, or even in water, gave you an upset tummy. 3% is not a trivial amount when one considers that nowadays the authorities are concerned about parts per billion of contaminants in foodstuffs.

One of the conclusions to be drawn from such studies is that whisky production should be handled carefully by amateurs. As mentioned in earlier sections, pot-distilled spirits involve the retention of some of the congeners in order to give taste to the whisky, but some of these taste-providing congeners are poisonous so don't overdo it. It would be wiser, perhaps, and certainly easier, to remove all the impurities by fractional distillation to give a pure alcohol and then add a flavouring agent. The physiological effect of an alcoholic drink, the 'buzz', is due solely to the alcohol, and everything else is merely moonlight and roses!

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 determining whether or not someone becomes inebriated. Drinking a bottle of beer is not less harmful than a  $1^{1}/_{2}$ -oz. drink of 40% scotch just because it is weaker. They both contain identical amount 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 <u>amount</u> 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 to be 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 they see it as an opportunity to increase tax revenue. If a government wished to base their tax grab on a rational argument they should start by basing it on alcohol amount (so much per unit of alcohol) instead of on alcohol concentration. And then, if health were the primary consideration as they claim, an additional tax would be levied based on the amount of poison (congener) present. Vodka would then attract the lowest tax of all and we would all live happily ever after!

A final note for environmentalists and watchdog groups on health matters: Is it not time to demand that governments require all manufacturers of alcoholic beverages to list the composition on the label? This would enable us to choose the ones with the lowest levels of toxic ingredients. They do it for food so why not for drink, particularly for drink which is known to contain several poisons.

# Fire and explosions

This may sound a bit melodramatic but when you are dealing with a procedure for the first time, and know that alcohol is inflammable, you may wonder. Let's take the explosion issue first. At no time, from beginning to end, is there any pressure in the equipment used for distillation. It is always open to the atmosphere. Fully open. Completely open. You will see that this is so when you look at the equipment diagrams later on and read the description of the procedures involved. So don't worry about it — an explosion is virtually impossible.

As far as fire is concerned you are dealing with an aqueous solution of alcohol which is non-inflammable right up to the time you collect the pure alcohol dripping from the draw-off valve. This is inflammable, but most people will be using an electrically heated boiler so there is no open flame. Secondly, in the remote possibility that a fire occurred, e.g. if you were smoking and dropped some burning ash into the collection bottle, alcohol fires can instantly be doused with water because alcohol and water are miscible. For this reason it is an infinitely safer inflammable liquid than gasoline, and in the fuel alcohol industry this fact is always quoted as one of the benefits associated with ethanol when it is used alone as a fuel — in Brazil for example.

# The Question of Legality

This chapter is written specifically for those readers who live in countries where it is currently illegal for amateurs to make their own homemade spirits. This means almost all of us. It is also written for government officials, politicians, law enforcement agencies, the news media and any advocacy groups with an influence on public policy.

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 cost to the consumer after tax is so great that the incentive to circumvent the law is considerable. This incentive grows greater and greater with each tax hike until a point is reached where people are driven by taxation policy to smuggle liquor or make their own, the net result being that tax revenues actually decrease while crime is encouraged.

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 (U.S.) worth of alcohol. The retail price of a litre of vodka will lie somewhere between \$10 and \$20 depending on the country and level of taxation. The mark-up is enormous. To be fair, 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. In an attempt to justify their actions and to persuade consumers to accept them, governments promote the idea that drinking is not only sinful but harmful to your health, so (they say) the tax is made deliberately high in order to protect you! As Scrooge would say, "Bah, humbug"

In light of the above, is it any wonder that an unscrupulous operator will attempt to sell his alcohol direct to the consumer, perhaps at half the nor-

mal 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 the law enforcement agencies (the good guys) and the smugglers and bootleggers (the bad guys) has been a perfect subject for stories and movies, and one which turned into real life drama during Prohibition in the United States in the 1920's. Police and gangsters fought it out with bullets, bombs and bloody mayhem, one gang slaughtering another to gain control of the market, and while all this was going on the law-abiding citizens of the world sat on the sidelines, took it all to heart and shivered in their shoes. The average person is now convinced that the production of spirits is inherently evil, something to be tightly controlled by the authorities or blood will run in the streets.

Beer and wine do not suffer from such a bad press. Being of a philosophical turn of mind the author has speculated on the underlying reasons for this. One reason may be that beer and wine-making are traditional activities and therefore hallowed by tradition. It is an activity which poets and shepherds and decent country folk might engage in as they play their flutes and dance around the Maypole. Distilling, by contrast, invokes an image of unholy forces at work — alchemists and necromancers. Or the satanic mills of industry and the callous face of science.

A more prosaic reason based on dollars and cents is that it would be uneconomical for smugglers and bootleggers to transport a lot of water. So they concentrate the alcohol by distilling it and thereby reduce the weight and volume 8-fold. In this way much more can be loaded into a ship or truck.

Unfortunately, the "wickedness" of home distilling is now so ingrained in the social psyche that this alone is enough deterrent to make many law-abiding citizens not only refuse to engage in it but even to discuss it. Thus, it has become self-policing.

#### **Amateur distillation**

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 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? And why, commercially, should a distilled spirit attract a higher tax per

unit of alcohol? At the risk of being tediously repetitious it is worth reminding ourselves again that distillation is one of the most innocuous activities imaginable. Unlike beer- and wine-making 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. Strange really that the purification of a legal drug by removing the poisons is illegal. 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 that justified in demanding the right to do so.

Governments surely wouldn't do something without reason would they!! There must be a reason for the ban on amateur distillation. Surely! In attempting to find this reason the first thing which comes to mind is the potential loss of tax revenue. After all, if everyone started making their own spirits at home the loss of revenue might be considerable. However, 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 distilling is based on the following reasoning. Home-made beer and wine are often a bit inferior to a good commercial product, and their preparation takes quite a bit of time, so only the most enthusiastic amateurs will go to all that trouble. 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, and could make it in quantity in a short time. 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 (several hundred dollars) 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 either gin or vodka and it is only these two which can be made by amateurs with a quality

approaching that of commercial brands. 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 such 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 spirits is much higher than it is on beer and wine. A third restriction on spirits can be seen in the alcoholic beverage section in the supermarkets of some countries — beer and wine may be sold, and possibly fortified wines such as vermouth, but raise the alcohol concentration to 40% and the ancient shibboleth of 'hard spirits' comes into play. This is grossly unfair discrimination and naturally of great concern to distillers. As they point out over and over again, in advertisements and representations to governments, a glass of gin & 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 why is there this blatant discrimination on the part of governments which pride themselves on being non-discriminatory when it comes to race, religion, colour, gender, age and so on and so forth? 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,  $C_2H_5OH$ , an acknowledged mood-modifier, and it is this drug which governments seek to control, but the alcohol in beer, wine and gin are identical and imbibed in similar quantities will have identical effects in terms of mood modification. So why are they taxed differently?

The only explanation which seems to fit the facts is that governments and their officials cannot understand the difference between <u>concentration</u> and <u>amount</u>. As a matter of fact quite a lot of people have this difficulty. Just because beer contains 5% alcohol whereas spirits contain 40% does not mean that the gin-drinker is 8 times more likely to over-indulge than the beer-drinker. To believe this is to be naïve. The fact of the matter is that antisocial behaviour such as hooliganism at sporting events is almost 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. Usually they are young men who happen to prefer beer to a vodka martini with a twist of lemon. And after the first beer they'll have another, and another, always drinking 5% alcohol but increasing the amount with each can. The 5% alcohol content is comparatively low but this is irrelevant when you drink one can after another. It is not the alcohol concentration which is the issue here, it is the amount of alcohol.

An attempt has been made by the author to bring this rather simple point to the attention of officials in the Customs & Excise Branch but the argument falls on deaf ears. We pointed out that alcohol is made by fermentation and that amateurs are allowed to make as much as they like within reason for their own use. So why not allow them to distil it? We pointed out that distillation doesn't make alcohol, it merely purifies it. Ah, is the reply, but it makes it stronger. So we're back into the confusion surrounding concentration and amount. When all else fails, the hoary old argument about amateurs poisoning themselves and going blind is trotted out. Really!

The above discussion has been argued at some length because it is important for the reader to feel comfortable with the "moral" aspects of distillation and with the supposed dangers to health. There is no need for him to be furtive about it or feel like some sort of back alley abortionist. The socalled "offence" has no moral dimension to it. It is not sinful. But it is necessary 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 may be 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 (vodka).

# Equipment

The production of pure alcohol by distillation is not particularly difficult in principle — you simply have to make a batch of beer and then purify it. One cannot use a pot still however, or a moonshine still, or any of the strange little stills being written about and offered for sale on the Internet. This is because they do not incorporate the two essential requirements for high-purity fractional distillation. These two requirements are a) a packed column, and b) a split-stream stillhead.

In the earlier chapter where we discussed alcoholic beverages it was mentioned that simple distillation, using a pot still, divides the crude alcohol solution (or "beer") into three fractions — the heads, the tails and the middle fraction. The heads are the very volatile constituents of the beer such as acetone and methanol, the tails are the least volatile components such as fusel oils, while the middle fraction consists of mostly ethanol contaminated with both heads and tails. In other words the separation is far from perfect.

Theoretically, it would be possible to take this middle fraction and redistil it, thereby getting rid of a few more heads and tails. Then this process of re-distilling the middle fraction would be repeated over and over again until all the heads and tails were gone and we were left with nothing but ethanol. In practice, however, this is virtually impossible because we would be dealing with smaller and smaller volumes of middle fraction at each stage of purification until a negligible amount of ethanol remained. What good is one drop of pure alcohol!

Commercial producers of vodka, and other forms of pure alcohol such as that used in colognes, cope with this problem by adopting on a large scale the scientific process of fractional distillation. They use a counter-current flow of vapour up a tower (perhaps 100 ft high and 12 feet in diameter) against condensed liquid flowing down, the two meeting in a series of trays at many different levels within the tower. In these trays the rising vapour bubbles through the liquid and there is an exchange between liquid and vapour. For small-scale operations such as ours we use a packed column about 3 ft high and  $1\frac{1}{4}$ " in diameter which serves exactly the same purpose

as the commercial distilling towers. The construction of such a still will be discussed in detail later in this chapter.

# Scale of operation

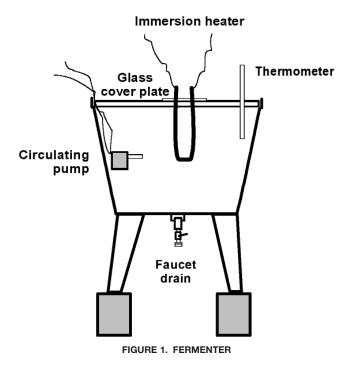
The first thing to think about is the scale at which you wish to operate. In pondering this weighty matter, take into consideration the following points. The cost of materials for building a still is almost independent of size. For example, in N. America at least, the most expensive item, the boiler, will be virtually the same price within the range 9 litres to 100 litres. Secondly, the smaller the equipment you use the more often you will have to use it in order to produce a given volume of alcohol. But on the other hand, going up in size, you don't want to build a piece of equipment which would take up a lot of space, is taller than the height of an average ceiling, or uses large amounts of electric current. In order to have something definite to work with, the discussion of equipment and procedures which follow are based on the fermentation of 10 kg of sugar to yield about 12 litres of vodka per batch, a batch taking about 7 days from start to finish. This is more than the average person would need to make assuming a second batch were started as soon as the first one were completed, but you don't want to be on a treadmill. One batch every couple of months might be about right, providing 1½ litres of vodka per week, and would be much more efficient in terms of time and effort than constantly producing small batches. Remember, alcohol keeps indefinitely.

# The fermenter

Before discussing distillation we need to make the alcohol. Many of you who read this book will have been making beer or wine for years and will have all the know-how and equipment you need for fermenting sugar to a potable alcohol. There may be others who aren't quite as familiar with the process, but even for the beer and wine makers — perhaps especially for the beer and wine makers — it is necessary to explain that fermenting for alcohol production is a very different type of operation to fermenting for wine and beer. This will be explained later on in the chapter dealing with procedures, but for now just accept that fermenting for pure alcohol production is a very crude and very simple operation compared with the great care required for making a fine wine or a palatable beer. All you will be concerned with is speed and simplicity and not at all with taste because we're not going to drink the stuff.

For those who do not already have fermenting equipment, 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 50-65 litres. A suggested arrangement is shown in Figure 1. The legs of the laundry tub are placed on four cement blocks so that the beer can be drained completely into the stripper by gravity flow following fermentation.

One can make this fermenter as simple or as elaborate as one wishes. In its simplest form one would simply 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, for speed, 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 due to evaporation and oxidation. An air-lock is not necessary. Another very useful gadget is an electrically driven stirrer. A third is a heater to maintain the optimum 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 still (see below) and wash water to be directed to the house drain when the fermenter is being cleaned out and rinsed.



### Drain

The drain outlet of a laundry tub is designed to take a tailpipe for connection to the house drain and does not match the sizes used for normal plumbing. But if you use a brass tailpipe you can, with a little ingenuity, connect to it a <sup>3</sup>/<sub>4</sub>inch ball valve and a hose connector. You will then be able to transfer the "beer" to the still using a length of hose with a female

connection at both ends (such as used with washing machines). Also, you can connect a length of garden hose for washing and draining the fermenter.

#### Cover

A transparent cover for the laundry tub can be made out of 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 <sup>1</sup>/<sub>4</sub>" thick is what you need, even though it is a bit difficult for an amateur to work with. So have your glass supplier cut it for you. A laundry tub usually has a shoulder a few centimetres below the top so get a piece of glass which will rest comfortably on this shoulder.

Two holes should be drilled in the cover, a largish one in the centre about 40 mm in diameter to take an immersion heater and another about 8 mm in diameter for a thermometer. A small notch on one edge will be useful for accommodating the power supply line if you intend to use a submersible circulating pump. Another refinement for a few extra dollars would be two holes for attaching a handle to lift the glass cover.

### Stirrer

We have tried everything from an impeller mounted through the bottom of the laundry tub to a vertical shaft through the glass cover driven by a small motor, and there is no doubt that by far the best method uses a submersible circulating pump such as used in an aquarium. Submerge the pump below the surface so that no air can enter it. Aerating the water is important for the well-being of fish but in fermenting it would simply make the yeast grow at an alarming rate. Fermentation is an anaerobic reaction which requires the absence of air if it is to produce alcohol.

### **Immersion heater**

The optimum temperature for fermentation if one is thinking of speed rather than flavour is about 33°C. Fermentation itself generates some heat but probably insufficient to maintain this temperature, particularly if the room is cool. An external heat source, therefore, should be provided and since only 100 watts or so are required an immersion heater such as used in an aquarium is ideal. If it does not contain its own thermostat, or if you use a different type of heater, an ordinary light dimmer switch works very well. They are inexpensive and can take up to 600 watts.

# The Fractionating Still

The purification of the crude beer produced from sugar and yeast is a 2-stage process, or even three in certain cases. The first stage is known as beer stripping and, as the name implies, is just a rapid and fairly rough method for separating most of the alcohol from the beer and leaving behind most of the water and the yeast. The volume of liquid after this first stage, a liquid known as "high wine", is less than one-quarter of that with which we started. So if we started with 50 litres of beer we would end up with around 10 litres of high wine, and if the strength of the "beer" had been 10% the strength of the high wine would be closer to 50%.

In the first edition of this book, published in 1997, two separate stills were used for the two stages, a large pot still for the rather rough beer-stripping stage and a smaller one for the more exacting purification stage. The sequence of events is illustrated in Figure 2. The reasoning behind the use of

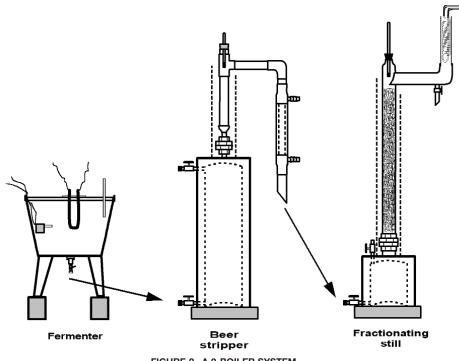


FIGURE 2. A 2-BOILER SYSTEM

two boilers was the large difference in liquid volume in the two stages. In this original system the beer stripping boiler had a volume of 100 litres and consequently was able to accommodate all the 50-60 litres of beer from the fermenter. The 10-15 litres of high wine so produced were then purified in a much smaller boiler of 25 litre capacity. It seemed to make sense at the time and was used for at least 10 years with excellent results.

However, with the sacrifice of a little convenience it is possible to make do with just a single boiler, thereby saving considerably on cost and the amount of space required for the equipment, and since it is apparent that most readers prefer this arrangement it has been decided that for this edition of the book we shall drop the two boiler system and concentrate solely on the single boiler.

### **Material of construction**

Glass is really the best material to use for making small-scale stills, being inert, clean and transparent. One can see exactly what is going on inside and it is aesthetically pleasing. For those fortunate enough to have access to a glassblower, either at a university or research institute, and are willing to pay the fairly high cost, the construction of a glass still will be described later.

For the majority of people the choice will have to be metal and the only decision left to make is whether the metal should be copper or stainless steel. Either will do an excellent job. In using metal the reader should appreciate that its only shortcomings are: a) that it lacks the aesthetic appeal of glass and b) you can't see through it. Large commercial stills are made of metal so it is obviously satisfactory.

The advantages of using copper are that it is relatively inexpensive, it can be purchased from any hardware store and, most importantly, it can be worked and soldered easily by amateurs. Naturally, doing the work yourself will reduce costs enormously. Copper also has a high thermal conductivity, a useful attribute for cooling coils. If there is any concern about copper being attacked by the vapours involved in distillation it is worth remembering that commercial whisky distilleries in Scotland have used copper stills for centuries.

#### Construction

In Figure 3. a still is shown which, because of its offset design, we refer to as the "Mexican cactus". It consists of a 25 litre boiler surmounted by a  $2^{1}/_{2}$  to 3 ft. length of  $1^{1}/_{4}$ " copper tubing. This is the column. It will be packed with a suitable material (to be discussed later). At the top of the column is the stillhead where the vapours rising from the boiler are condensed to liquid and the liquid then split into two streams. The major stream, consisting of 90% of the condensed liquid, flows back down the column to the boiler while the other 10% is directed to the outside world via a small valve. Let us look at each part of the still in more detail.

### **Boiler**

When it comes to amateur distilling there seems to be a burning desire on the part of the handyman to improvise a boiler out of some odd vessel which happens to be available, and no-one should be surprised to learn that everything from pressure cookers to beer kegs to milk churns to vacuum

cleaner tanks have been adapted by ingenious do-it-yourself types for this purpose. However, we strongly recommend that you save yourselves a lot of time, trouble and expense by using an ordinary domestic hot water heater. In N. America these are available in all sizes from 9 litres up to several hundred litres, and are ideally suited for acting as the boiler in all amateur distillation systems. They are rugged, glass lined, already have an immersion installed, they are insulated, they have pipe fittings in all the right places, and are housed in attractive white-enamel steel housings. What more could you wish for? If you had drawn up the specifications yourself for the ideal boiler

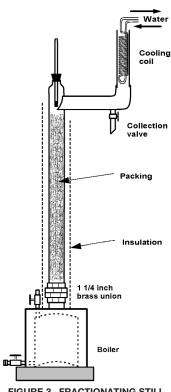


FIGURE 3. FRACTIONATING STILL

required for a still it would not be very different from a hot water heater. In N. America they cost around \$140 in all sizes up to 100 litres.

A few simple modifications to the hot water heater are required. Firstly, remove or by-pass the thermostat. We need the contents of the boiler to boil, so a thermostat which switched off at a temperature of, say, 75°C. would obviously defeat our purpose. Removing the thermostat may seem dangerous, and it would be if we had a closed system, but the system is open to the atmosphere at all times (see Figure 3) so there can be no pressure build-up. It is just like a tea-kettle. For this reason you also can dispose of a pressure-relief valve if one is installed because the pressure inside the boiler is never above atmospheric.

The pipe fittings on water heaters vary from manufacturer to manufacturer, but whichever one you choose you'll find a fitting at the bottom (the cold water inlet) and several at the top. If you need another  $^{3}/_{4}$ " pipe fitting at the top you may find one by removing the sheet metal cover and fiberglass insulation from the top of the housing. This is where in some models the magnesium rod used as an anti-corrosion device is installed. It can be removed because it is not essential in our application and the  $^{3}/_{4}$ -inch female pipe fitting may be useful to you for mounting the column.

The lower connection, the cold water inlet when the tank is used for domestic hot water production, will become the inlet for beer from the fermenter and also the drain for the exhausted beer (the stillage) after stripping. Fit this connection with a <sup>3</sup>/<sub>4</sub>" ball valve and screw into it an adapter for connecting a rubber hose. Use a ball valve at the drain, and not an ordinary faucet, because the yeast in beer forms sticky lumps when boiled and there should be a wide opening for the yeast clumps to exit to drain.

# **Power supply**

The packed column which will be mounted above the boiler (see later) 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 (flood). The 1500 or 3,000 watt heater supplied with these boilers is, in fact, unnecessarily large and we need to reduce this wattage to about 750 in some way. Several methods for doing this suggest themselves. One would be to buy a 750 watt immersion heater from a manufacturer of heater elements but this would be costly and time-consuming. We are not even certain if a 750 watt immersion heater

exists. Another would be to buy a step-down transformer, either fixed or variable, but this would be even more expensive. A very simple and inexpensive solution to the problem for residents of N. America is to buy a water heater with a 3000 watt, 240 volt element already installed and use it on 120 v. Or, if the boiler is fitted with a 120 volt element remove it and substitute a 3000 watt, 240 v. element. The voltage has been cut in half, which will cut the current in half, so the wattage will be reduced by a factor of 4, i.e.  $\frac{1}{2}$  x  $\frac{1}{2} = \frac{1}{4}$  and 3000 x  $\frac{1}{4} = 750$  watts.

For the electricians among you another solution would be to carry out half-wave rectification of the electricity supply using a diode. This will cut the wattage in half. If you want continuous, variable control you could use a triac, but unfortunately the inexpensive household variety (a light dimmer switch) has a capacity of only 500/600 watts. A 1000 watt dimmer can be purchased for about \$40 (US) and a 2000 watt model for perhaps \$150.

You do not need to continuously vary the wattage input to the boiler and we recommend that you avoid this unnecessary complication and expense. Rather, arrange by one means or another to use the appropriate wattage for the column you are using (e.g. 750 watts for a 1 ½ column) and stick to it. Incidentally, you do not need to measure either the temperature or the pressure in the boiler — the pressure is atmospheric and the temperature is the boiling point of beer, e.g about 100°C.

# **Soldering**

Before discussing the construction of the column and stillhead a word should be said about soldering. There are two solders in common usage — the low temperature lead-free solder which melts at around 350°C. and silver solder which melts at about 1300°C. It is possible to manage with just the low temperature solder, but there are situations where a small joint needs to be made close to an adjacent one, or close to a larger one which needs a large flame, and in such situations there is a danger of one joint melting while the other is being made. By using silver solder for the small pieces this can be avoided.

### The column

The fractionating column consists of a  $2^{1}/_{2}$  to 3 ft. length of  $1^{1}/_{4}$ " tubing. The rule of thumb is that the height of a column should be at least 15x

its diameter, which would mean a column height of 19" minimum, but why not be generous and add a few more inches. The higher the column the better (within reason), because it provides a larger number of solid/vapour interfaces up the length of the column and therefore more re-distillations. Two and a half to three feet is convenient but you won't wish to go much over 3 ft. or you will hit the ceiling!

At the top of the column (see Figure 3) an elbow is provided for the passage of vapour across to the stillhead condenser and for a thermometer to measure the vapour temperature. At the base of the column there is a series of adapters, including a  $1^{1}/4$ " union, to go from the  $1^{1}/4$ " diameter of the column to the 3/4" pipe fitting on the top of the boiler.

The column must be well insulated to ensure a stable temperature regime up the full length of the column while it is refluxing.

## Thermometer adapter

In both stages of distillation it is necessary to know the temperature of the vapour stream inside the system in order to know what's going on, since temperature and composition are closely related, and the simplest method for introducing a thermometer would be to use a cork. This is the method we used for many years but it leaves much to be desired and there is a better method. Hot alcohol/water vapour is very aggressive and corks rather quickly turn into a gnarled object closely resembling the withered core of a rotten apple. Rubber is unacceptable because it gives a flavour to the alcohol. Nowadays we use a brass compression fitting and teflon seal for all thermometer inserts into metal columns.

The construction of the thermometer adapter is shown in Figure 4. Use a  $^{3}/_{8}$ " x  $^{1}/_{4}$ " compression fitting. There is a shoulder inside these fittings at the mid-point and you will need to drill away this shoulder to let the glass thermometer pass right through. Use a  $^{17}/_{64}$ " bit and drill from the large end, trying to avoid going right through and damaging the seat for the ferule at the  $^{1}/_{4}$ " end. If you use a digital thermometer, which usually has a  $^{1}/_{8}$ -inch compression fitting is large enough as is without removing the internal shoulder.

Solder a short length of 3/8" copper tubing vertically to the elbow at the top of the column and attach the compression fitting. Use teflon plumber's tape to make the seal, winding several turns around the ther-

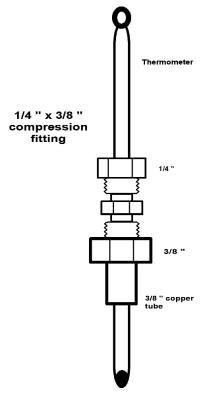


FIGURE 4. THERMOMETER ADAPTER

mometer so that when the nut is tightened the teflon is compressed between the thermometer stem and the brass fitting. There is no pressure in the apparatus and no leakage. The bulb of the thermometer should be at the midpoint of the elbow so that it is in the main stream of vapour flow.

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

Note 2. A glass thermometer in such a rigid set-up is very vulnerable to breakage. The slightest touch and ......! It is prudent, therefore, to remove it while working round the still.

Note 3. Some of you may wish to use an electronic digital thermometer. They usually have  $\frac{1}{8}$ " diameter probes. They can be sealed into the system in the same way as a glass thermometer, using either a small teflon plug with a  $\frac{1}{8}$ " hole drilled through it or simply by winding more teflon tape around the stem.

# The packing

The packing inside a fractionating column is very important and many articles in the scientific literature are devoted exclusively to this topic. Everyone has his own ideas on what constitutes the ideal packing and the writer is no exception. Unlike scientific texts, however, cost is a consideration here. 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 and should be of such a shape that they leave plenty of free space for vapour to rise up against a descending flow of liquid;

- b) they should pack uniformly in order to avoid channeling, and
- c) they should have a large surface area and crevices where liquid can be trapped.

Scientific glass columns frequently use short, e.g. 6 mm lengths of 6 mm glass or ceramic tubing called Raschig rings. Ceramic saddles are another popular shape. Glass marbles might be used in large diameter columns but do not have sufficient surface area for a small diameter column such as ours. Also, unlike Raschig rings, they do not have any pockets where liquid can be trapped, so are rather inefficient.

The packing which we recommend has a very domestic origin but is cheap and highly effective. It consists of the scrubbers or scourers used for cleaning pots and pans and found in any supermarket. These are not the fine steel wool pads impregnated with soap but the much coarser scrubbers made from lathe turnings which usually come in a ball. They are available in copper, brass and stainless steel, and the ones to choose are the stainless steel. Several will be required for the column. Commercial packings using the same principle are available (at a price), and are very neat and uniform in surface distribution because the stainless steel filaments are woven into a blanket and the blanket is then rolled into a cylinder to exactly fit the inside of the column.

Packing the column is relatively simple if you have a 1½" union joining the base of the column to the boiler because then there is no bottleneck and you have the full width of the column to work with. Pull out the balls of tangled filaments into a sausage-shape, dip them in soapy water to reduce friction, and carefully shove them up the column with a minimum of compaction. This type of packing only occupies about 4% of the column, leaving 96% open space, appreciably better than Raschig rings. It also has a much larger surface area, so you will find it very effective.

### The stillhead

The purpose of the stillhead is to divide the vapour emerging from the top of 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 through the column to the boiler, 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.

Two different designs for a stillhead made out of copper are shown. The first, an offset design which was shown in Figure 3 and which for obvious reasons we whimsically refer to as the "Mexican cactus", has the stillhead shown in more detail in Figure 5. The second, again because of its shape, we refer to as the "Hatstand", and is shown in Figure 6. They both work very well — it's simply a matter of appearance and ease of construction — so the choice is up to you.

# The Mexican cactus stillhead. (Fig. 5).

The diagram is more-or-less self-explanatory. The alcohol vapours rising up the column are directed horizontally along a 1<sup>1</sup>/<sub>4</sub>" tube (length not critical) and then vertically into the condenser housing.

### The condenser

The alcoholic vapours are condensed by means of cold water running through a coil of copper tubing inserted in the stillhead. To make this coil use

16 feet or so of  $\frac{3}{16}$ " flexible copper tubing.\*\* Such tubing is not usually stocked in the plumbing section of a hardware store but can be found in the automotive supply section since it is used for fuel lines. Even 1/8" tubing can be found there if required, so don't be fobbed off by a salesman saying that <sup>1</sup>/<sub>4</sub>" tubing is the smallest made. Make a hairpin from the  $\frac{3}{16}$ " tubing about 14" from one end with the two arms close enough to one another to fit inside the  $1^{1}/_{4}$ " condenser jacket. Gently grip the hairpin vertically in a vise so as not to flatten it, put a short

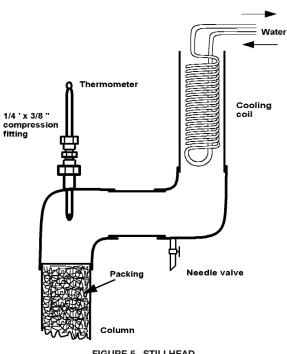


FIGURE 5. STILLHEAD

length of 3/4" pipe over the short side of the hairpin to act as a mandrel, jam a piece of wood down inside to stop rotation and now wind the remainder of the 3/16" tubing around the outside. The whole operation will take about 5 minutes.

Note that the cooling water enters at the <u>top</u> of the coil in order to provide countercurrent flow of water and vapour. All heat exchangers work in this fashion and are much more efficient than when used with concurrent flow.

Several readers have asked about the top of the stillhead being open to the atmosphere. Shouldn't it be closed, they ask, to prevent vapour escaping? The answer is "no". The vapour rising up the column should be totally condensed to liquid, leaving nothing to escape through the top. If any vapour does manage to by-pass the cooling coil (detectable by putting your nose over the top and sniffing) then the coil hasn't done its job properly and you need more cooling surface or a lower water temperature.

The draw-off needle valve is attached to the underside of the horizontal portion of the stillhead by means of a short length of ½" tubing soldered to the stillhead, while the valve is attached to this tube by means of a compression fitting. This will avoid the necessity of having to heat the valve itself during soldering. In order to ensure a clear passage for the exiting liquid, and also to strengthen the joint, a useful tip is to attach it where the elbow overlaps the 1½" tube (see Fig. 5). Before soldering the elbow in place drill a ¼" hole in it where it will overlap the inside tube. Then solder the elbow in place. Position the short length of ¼" tubing in this hole in the elbow, butting it up against the tube inside. Solder in place. Then drill right through the short length of tubing, penetrating the inner tube. This ensures that the draw-off tube is flush with the inside surface. If it stood proud the condensed liquid might flow around it instead of going down the hole.

The exit tube of the draw-off valve is shown in the diagram as being very short. The condensed alcohol emerging from the valve is quite hot, hot enough, in fact, for some people to put a small heat-exchanger on it to cool the alcohol before it falls into the collection bottle. A simpler method is to add a copper extension tube below the needle valve so that the alcohol is air-cooled before it enters the bottle. A long extension tube also allows you to place the collection bottle on a table. DO NOT use a length of plastic tubing for this purpose. Hot alcohol is a very aggressive solvent and will attack the plastic and make your alcohol cloudy.

\*\* Note. If you cannot find any  $\frac{3}{16}$ " copper tubing for the cooling coil you could use  $\frac{1}{4}$ " tubing, a more common size. But if you do you will need to use 2" diameter copper tubing for the condenser housing since  $\frac{1}{4}$ " tubing would flatten if you tried to wind it more tightly.

### The "Hatstand" model

When faced with the problem of how to condense a stream of vapour to liquid and then split the liquid stream into two parts, everyone has his own idea of how to do it better, easier, more cheaply and more beautifully than the "Mexican cactus" design shown above. We, too, have played around with dozens of different ideas, and the conclusion reached is that there isn't anything too much wrong with the Mexican cactus. It has a lot going for it. But, to demonstrate that there <u>are</u> different designs, a linear rather than an offset model is shown in Figure 6. We call it the "Hatstand" model.

The same boiler and packed column are used, but as will be seen

from Figure 6, the column and stillhead are in line with one another and there is no jog as there is with the Mexican cactus. Its features are:

- 1). The thermometer is set in at a 45° angle to the column using a compression fitting and with the thermometer bulb just above the packing.
- 2). A collection "cup" is made from a \_" copper pipe cap. In the centre of the cap a short length of <sup>1</sup>/<sub>4</sub>" copper tubing is silver-soldered in place with about <sup>3</sup>/<sub>8</sub>" standing proud above the bottom of the cap to act as overflow, this tubing extending far enough below the cup to convey the overflow of liquid to the top of the packing while by-passing the thermometer

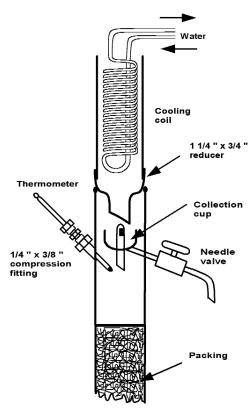


FIGURE 6. THE "HATSTAND" STILLHEAD

bulb. The overflow tube should be closed at the top and a hole drilled in the side of the tube just below the top. This is to prevent condensed liquid falling straight down through the overflow to the packing.

- 3). A second short length of copper tubing connects the bottom of the cup to the outside world where the flow is controlled by a needle valve. In use it will be seen that, when the valve is completely closed, all the liquid falling into the cup will overflow on to the packing. When the valve is completely open all the liquid will exit the column and none fall onto the packing.
- 4). To ensure that all the vapour condensed by the cooling coil runs down into the collection cup, a  $1^{1}/_{4}$ " x  $^{3}/_{4}$ " adapter is soldered, upside down, to the top of the column. The  $^{3}/_{4}$ " end points downwards and is cut at a  $45^{\circ}$  angle to give a drip-tip so that all (or at least most of) the condensed liquid falls into the cup. Without this arrangement, for example if the  $1^{1}/_{4}$ " x  $^{3}/_{4}$ " adapter were omitted, some liquid would miss the cup altogether.

# Water supply

It is worth mentioning that there is considerable resistance to the flow of water through a 16 ft length of  ${}^{3}/_{16}$ " tubing and you may find that friction alone will be insufficient to hold in place the plastic tubing leading from the water supply in the house. There is nothing worse than having the water line blow off in your absence and finding your workshop flooded when you return. So play it safe. One neat solution to this problem is to use the metal fitting and  ${}^{1}/_{4}$ " o.d. high-density polyethylene tubing used for connecting a humidifier to the house water supply line.. These little kits are inexpensive (\$12-ish) and come with 25 ft of high-pressure polyethylene tubing, a length which is convenient when the water supply is not adjacent to the still. The drain from the cooling coil involves no pressure so any type of tubing will do.

If you smell alcohol fumes as soon as distillation starts it means that there is insufficient cooling. Test the cooling water leaving the condenser and, if it is warm, you should increase the flow-rate. If it is cool then there is no point in increasing the flow of cooling water because the problem is insufficient cooling <u>surface</u>. This is unlikely to be the case if you are using a 750 watt heater in the boiler because the cooling coil as described can easily handle this amount of heat input, but with a higher wattage heater you should not take it for granted. If you find you need more cooling surface then you'll be

forced to use a longer length of  ${}^{3}I_{16}$ " copper tubing. 16 ft is only just sufficient for a 750 watt heater so you might wish to consider going to 18 ft at the outset.

The volume of water you are likely to be using during the course of a distillation is discussed in Appendix IV. If water is a problem you could experiment with air cooling, circulating the cooling water through an automobile radiator with a fan blowing air through it.

### The glass still

The glass still shown in Figure 7 and described in the following paragraphs is essentially the same as the hatstand model discussed above, but there are one or two innovations which make it particularly pleasing. All dimensions are given so you can take it along to a glassblower and he will know exactly how to make it for you.

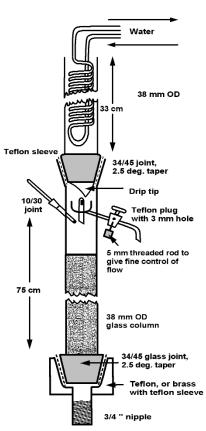


FIGURE 7. THE GLASS STILL

The column and stillhead are made from 38 mm O.D. glass tubing, joined by means of a 34/45 drip-tip standard taper joint. A teflon sleeve is placed between the male and female halves of this joint in order to avoid "freezing". Normally the joint could be greased but hot alcohol would soon flush this out leaving a dry glassioint which to-glass would "freeze" and be very difficult to separate if you ever needed to. Hence the teflon sleeve which the glassblower will supply.

The cooling coil is made of copper rather than glass because a glass coil would have insufficient cooling capacity to condense the alcohol vapours effectively. As with the copper hatstand model, all the condensed vapour, from both

the cooling coil and the interior walls of the stillhead, falls into the collection cup, the drip-tip being located inside it. This collection cup has two outlets, i) a tube at the bottom-side leading to the draw-off valve on the outside of the column, and ii) a central tube which acts as overflow when the draw-off valve is closed sufficiently.

To use, turn stopcock to OFF position for total reflux. Then turn 90 deg. for pre-set 10:1 reflux ratio.

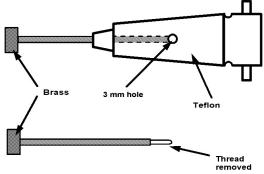


FIGURE 7a. FINE CONTROL

The draw-off valve, therefore, has the ability to change from zero to 100% the ratio of condensate drawn off to the outside world to that which is returned back down through the column to the boiler.

Normal teflon stopcocks are not easy to adjust for fine control of liquid flow, so a fine control has been added and is shown in Figure 7a. To make this modification a hole is drilled and tapped from one end of the teflon plug and a small brass bolt introduced which can close completely the hole supplied by the manufacturer. With a fine thread on this bolt and a knurled knob to turn it, very precise control is possible. A particularly nice feature of this design is that the teflon stopcock can be turned to the closed position for total reflux while equilibrating the column, and then turned 90° to the open position where the pre-set fine control will immediately provide the 10:1 reflux ratio for product withdrawal. Note. This will become understandable after you have read the operating procedures and the principles of fractional distillation a bit later on.

The thermometer is introduced on the opposite side of the column to the draw-off valve, and slightly offset so that it avoids the down-comer from the collection cup. If temperature is to be measured with a digital thermometer then a small teflon plug can be inserted in the  $^{10}/_{30}$  joint.

### Attachment to boiler

At the base of the column there is the problem of joining a glass column to the <sup>3</sup>/<sub>4</sub>" pipe nipple on the boiler and you will require the services of a machinist to solve this problem for you. Glass-to-metal joints are always tricky, so four different arrangements are shown in Figures 8 (a), (b), (c) and

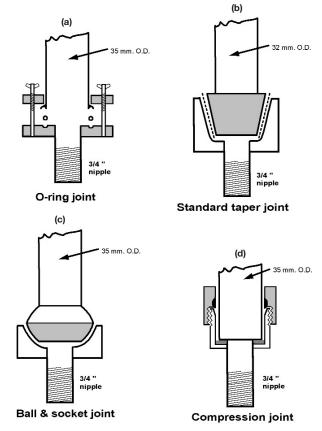


FIGURE 8. ALTERNATIVE ATTACHMENTS - GLASS COLUMN TO BOILER

(d). In the first case (a) a teflon O-ring makes the seal between the base of the column and a brass adapter specially made with a groove on the top plate to match the groove on the end of the glass column. A clamp is necessary to hold the two halves together, the top one being a ring which the glassblower must include before he fuses on the O-ring joint.

The next (b) requires the glassblower to fuse a  $^{34}/_{45}$  standard taper joint to the bottom of the column and the machinist to make a corresponding female half of the joint from brass. The angle of the taper is  $2^{1}/_{2}$  degrees. A thin teflon sleeve must be used between the metal and glass in this joint, identical to the one between the top of the column and the stillhead.

The third (c) glass-to-metal joint uses a glass ball at the base of the column (size  $^{50}/_{30}$ ) which nests in a brass socket, made by your obliging machinist. A thin gasket of teflon film produces the seal between the glass ball and the brass socket, or you can use a teflon-based plumber's grease which comes in a tube like toothpaste.

The fourth and last adapter (d) uses a specially made compression fitting. It has the advantage of being able to use a glass column cut off square at the bottom, which is much cheaper than using a glass standard taper or ball joint. The compression fitting, which is similar in size and principle to the sort used to attach the tailpipe under a sink, must be made by a machinist. This joint, using a compression fitting, is our personal preference.

The chances are, after reading the above, and after hearing that the cost might be around \$1,000 for a glass column, you will opt for one of the metal designs. For this reason we have not gone into more detail.

# Support table

A fractionating still is rather tall and needs some support. Some people build their still close to a wall so that they can use brackets to support the column and the collection bottle. Another method is to make a table 30" high, put the boiler underneath and bring the column up through a hole in the tabletop. The hole should be large enough to accommodate both the column and the insulating sleeve around it. Use a spirit level to ensure that the column is upright. Not only does the tabletop support the column very firmly but it can also support the stand on which you place the collection bottle. Additionally, a table is useful for holding a digital thermometer and for writing up your notes.

There are many refinements you can make to this set-up. For example, a set of built-in drawers is very useful. Then, if you put the whole thing on castors, with the boiler resting on a shelf close to the ground, you can wheel the still from one part of the room to another or even into a closet or another room.

# **Fermentation**

# **Principles**

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

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

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. The yeast cells die, however, and in the absence of oxygen will not replenish themselves, so eventually the yeast becomes inactive.

The atomic weights of carbon, hydrogen and oxygen are 12, 1 and 16 respectively, and when these weights are applied to each of the atoms in 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 about 500 grams of alcohol. Because the specific gravity of ethyl alcohol is 0.8 the 500 grams represent 625 ml of absolute alcohol or 1½ litres of 40 per cent alcohol, the normal strength of vodka and other spirits.

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 to 80 per cent of theory. There are two main reasons for this, one being 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. The second and major reason is a failure to recover all the alcohol from the fermentation broth during beer stripping. Losses such as these would not be tolerated in a commercial operation but are acceptable for the amateur. After all, even with

a recovery as low as 70% of theory a kilogram of sugar valued at a dollar or so 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 and nutrients 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 generate high yields of alcohol and large quantities of carbon dioxide. The  $\mathrm{CO}_2$  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 unacceptable 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 of gin- and vodka-making 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. This is to stop sugar falling down the hole. Add 10 kg of sugar, place your hydrometer on the pile of sugar, add about 50 litres of cold or lukewarm water and start the circulating pump. The pump should be positioned just below the surface of the water and well above the bottom so that it does not suck in grains of sugar and damage the rotor. Then add the yeast, cover with the glass plate, install the immersion heater and thermometer in their respective holes in the cover, and switch on the heater. The reason for adding the yeast before the sugar has dissolved and the water warmed up is to avoid too vigorous a reaction at the start. If the yeast is added to a strong sugar solution at fermentation temperatures the reaction can be vigorous enough to raise the temperature and harm, or even kill, the yeast. There is also excessive foaming which touches the underside of the glass cover and obscures the view.

### **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. Dry yeast which has been in storage for several months without refrigeration and without being vacuum-packed could be useless. Of all the enquiries received from readers the most prevalent concern a failed fermentation, or one which refuses to go to completion. In most cases the cause of the problem has been traced to the yeast having lost its activity due to poor storage, and this is really self-evident because we only have yeast and sugar and there can be nothing wrong with the sugar.

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 where it will disperse and sink.

With this amount of yeast and the time being allowed for fermentation (5+ days) there is no need to add nutrients. Also, do not be seduced by claims that special yeasts will produce 15% or more alcohol solutions because this does <u>not</u> mean that you get more alcohol, only that you can use less water and spend more money. It's the same old confusion about concentration and amount. The <u>amount</u> of alcohol you get is determined by the amount of <u>sugar</u> you have used and all the yeast does is convert this sugar to alcohol. It might reduce the fermentation time from 5 days to 3 days but it is scientifically impossible for a yeast, any yeast, to produce more alcohol than allowed by the equation at the start of this chapter.

When the temperature in the fermenter has reached 30 deg. to 35 deg. 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 may be quite vigorous at first but diminishes steadily with time until eventually the fermentation ceases and the beer looks dark and still. To confirm that it is complete, switch off the pump and look at the hydrometer. The original sugar solution will have had a specific gravity of about 1.06 and the hydrometer will be bumping up against the underside of the glass cover, but as the sugar is converted to alcohol the hydrometer will sink and the S.G. fall to about 0.99, below 1.00 because of the presence of alcohol with a S.G. of 0.8. With a little experience you will know exactly when to expect the fermentation to be complete (e.g. 5 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 (but not all) of the yeast when you transfer the beer to the still.

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. With a 25 litre boiler for the still the beer must be stripped in three batches of about 16 litres each, so make a dipstick marked at three equal heights and use it to gauge when each 16 litre batch of beer has flowed into the still.

**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 boiler 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**

Some of what needs to be said about the principles of distillation was covered in the chapter on beverages, and there was also some mention in the chapter dealing with the construction of a still. In both these places, 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 and. if something goes wrong, what you can do about it. 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.

At the outset it will be useful to dispose of a myth concerning distillation which is quite prevalent, so prevalent in fact that it is the basis of several small-scale stills being offered for sale. The myth goes as follows: If you have a mixture of three liquids with different boiling points, e.g. methanol (64.7° C.), ethanol (78.4° C.) and water (100° C.) it is believed that, if you raise the temperature to 64.7° C. and hold it there the methanol will boil off. Then, if you raise the temperature to 78.4° C. the ethanol will boil off. This is completely untrue. It might be approximately true for liquids which do not mix with one another, such as gasoline and water, but is totally untrue for the lower alcohols which are completely miscible with water. Being miscible they associate with one another at the molecular level and no longer act independently as individuals.

Having expunged this fallacy from our minds let's take a look at what really happens. Some of the more important chemicals we are dealing with, together with their boiling points, are shown in the table below.

Compound	<b>Boiling Point, °C</b>
Acetone	56.5
Methanol	64.7
Ethyl acetate	77.1
Ethyl alcohol (100%)	78.4
Ethyl alcohol (95%), the azeotrope	78.1
Propyl alcohol	97.2
Water	100.0
Butyl alcohol	117.5
Amyl alcohol	137.8
Furfural	161.0

Chemicals of different volatility such as those in the table above have different vapour pressures, the most volatile with the lowest boiling point having the highest vapour pressure at any particular temperature. A liquid boils when its temperature is raised to the point where its vapour pressure equals atmospheric pressure. When a mixture of liquids of different boiling points is heated the vapour contains all the compounds which are in the liquid but is slightly richer in the more volatile components. This will be found by condensing the vapour to liquid and analysing it. It is the basis of all distillations — the vapour is richer than the liquid in volatile constituents.

# Simple distillation. The pot still

First let's take a look at the simplest situation — the events taking place in a pot still when beer is distilled. The vapour is richer than the liquid in the most volatile constituents, i.e. the ones with the lowest boiling points such as acetone and methanol in the above table. When they distil over they are referred to as the "heads". There is no clear-cut separation of the various compounds so the heads will still be coming over when the ethanol starts to appear. Similarly, before all the ethanol has distilled over, the "tails" will begin to appear in the distilate. 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 may be 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 re-distil the middle fraction, repeating this process over and over again until the last of the impurities had been wrung out of the ethanol. Unfortunately, as mentioned before, apart from being very time consuming, the loss of ethanol on repeated re-distillation would be such that the final yield of pure alcohol would be virtually zero.

The retention of some of the "impurities" in the original beer when carrying out a distillation with a pot still does not bother many people because they have grown (or have been taught) to like the taste of these impurities. They add character to the alcohol. They add flavours (some of them pretty vile but some quite pleasant). By playing around with the distilling conditions it is possible to retain more or less of these impurities, or "congeners" as they are called, the manufacturers then referring to their product as whisky, brandy, etc., etc.

For those who wish to drink vodka or gin, however, or to obtain pure alcohol in order to make liqueurs, it is necessary to get rid of the congeners and the multiple counter-current distillation procedure described below must then be resorted to.

### **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 type of whiskey or moonshine. It may be palatable if carefully prepared but it certainly will 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!

In fractional distillation the vapours emerging from the boiling mixture are passed 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 interface on the packing material there is an exchange between liquid and vapour, the vapour giving up its latent heat to the descending liquid. Thus, the liquid is turned into vapour while the vapour is condensed back to liquid. The newly-formed vapour rises and the same exchange takes place on the next surface it meets up the column. Similarly, the descending liquid meets rising vapour at an interface in the packing further down the column.

The more volatile components of the mixture which have entered the vapour phase rise to the top of the column while the less volatile components which have gone into the liquid phase flow 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. It is not too dissimilar to what is happening in a heat exchanger where we also have counter-current flow — the cold water is being warmed by the hot vapour while the hot vapour is being progressively cooled to lower temperatures by the flow of water in the opposite direction.

In a commercial operation, which runs continuously, the different components of the mixture are concentrated at various heights within the column, and can be drawn off, and this continues indefinitely. Methanol, for example, would be continuously withdrawn from the top of the column while ethanol would be continuously removed from a point a little further down. The chemicals in each draw are not completely pure, but are much purer than they were before.

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 condensed vapour from the top of the column. First come the acetone and then the methanol and any other low boiling point compounds. They are discarded. Then the ethanol starts to appear, and when it does a por-

tion 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.

# The alcohol-water azeotrope

Water is an important constituent of the fermentation broth and with a boiling point of 100 deg. 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 96\*\* percent ethanol (v/v) and 4 percent water. The boiling point of this azeotrope is 78.1 °C. whereas the B.P. of 100% ethanol is 78.4 °C. As far as the system is concerned it "thinks" that this mixture of ethanol and water is a single liquid with the lower boiling point of 78.1 °C. and proceeds to separate it on that basis. The ethanol which is purified by a fractionating column is not, therefore, pure 100 percent ethanol but pure 96 percent, 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 special methods are available to accomplish this. For our purposes, however, where we are going to dilute the alcohol with water to 40 percent anyway, the presence of 4 percent water is of no consequence.

\*\*Footnote. In the literature you will find slightly different values for the azeotrope composition, all hovering around 96%. One reason for this is that the percentage can be expressed either volumetrically (v/v) or by weight (w/w). There is a difference because ethyl alcohol has a specific gravity of 0.8 compared to 1.0 for water. For example, 96% ethanol v/v works out to 95% w/w. If so inclined you may worry about this, but a more important question is — should a good martini be shaken or stirred!

# **Procedures**

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

## First stage — beer-stripping

Beer-stripping is not absolutely essential and theoretically 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 is concentrated into a relatively small volume in a relatively short time and quite a few of the impurities eliminated. It would be extremely tedious to fractionally distil all that beer at 4 ml per minute (it would take about 8 days). Also, the yeast is left behind and does not interfere with the more exacting fractionation process. A further, and very practical consideration, is that the purer and simpler the mixture of chemicals to be separated the purer the final product, so the more rubbish one can get rid of during beer-stripping the better.

We have 50-60 litres of beer which need purifying but the boiler of the still has a maximum volume of just 25 litres. It is important not to put too much beer in the boiler because it foams quite a lot and liquid would foam up into the bottom of the column and be swept over into the collection bottle by the rush of vapour. About 60% full is suitable which, with a 25 litre boiler, means about 16 litres. So we have to strip the beer in three separate 16-17 litre batches.

Proceed as follows: Run 16 to 17 litres of beer into the boiler, start the flow of cooling water, switch on the boiler and open the collection valve WIDE. Under these conditions there is very little reflux so, to some extent, the still is operating like a pot still. This is what we want for beer stripping. If you are using 750 watts it will take about 2 hours to come to the boil., and when it does liquid starts to drip quite rapidly into the collection bottle.

The temperature of the vapour coming over from the boiler at the start will be about 80 deg. C. and will rise to 96+ deg. C. or so as the ethanol in the boiler becomes exhausted. This will take about  $2\frac{1}{2}$  to  $3\frac{1}{2}$  hours at 750 watts. You will have collected about 3 litres of high wine. Although there will be some ethanol remaining in the boiler at this point, the amount will barely be sufficient to warrant the cost of the electricity to drive it over, but you can continue to  $98^{\circ}$ C. or so if you wish.

There are two possible ways to judge when beer stripping should be terminated — the volume of distilate collected and the temperature of the vapour. Always use the latter, the vapour temperature, regardless of the volume of distilate collected.

Allow the boiler to cool somewhat before opening the bottom valve and sending the contents to drain. Then add the second and third batches of beer and strip them just as you did the first. You will have collected about 9 litres of high wine with a strength of around 50% alcohol.

Drain and flush the boiler and then add the high wine plus several litres of water to raise the volume to 15 litres or so. There are two reasons for this: one is to ensure that the heating element is still covered with liquid at the end of distillation, while the second is because a purer alcohol is obtained when distilling from a dilute alcohol solution than from a concentrated one. We are now ready to carry out the important second stage of fractional distillation.

### **Fractional distillation**

In the equipment section we have illustrated and discussed three different types of stillhead, two in copper and one in glass. For simplicity, the following discussion will be restricted to the model shown in Figures 3 and 5, the one we call the "Mexican cactus".

In contrast to the situation during beer-stripping, in the case of fractional distillation the small draw-off valve in the horizontal part of the still-head is completely closed initially so that all the vapour condensed at the top will run back down the column to the boiler. Under these conditions the column is said to be operating under "total reflux".

While the boiler is heating up, 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. It is prudent, for the reason discussed in the next paragraph, not to walk away from the still and let this event take place in your absence.

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 is unlikely to be a problem with the 1½-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 the immersion heater in the boiler as recommended. With a glass column choking is easily detected because liquid can be seen bubbling away in the packing, but with a metal column this is not possible. So listen. Choking or flooding may be detectable by a slight rumbling noise. The other method of detection is to look at the thermometer. Liquid rising from the boiler is much hotter than the vapour so, instead of registering 70+°C. the thermometer may register 90+°C. If this happens, switch off and try again. It is somewhat like a smoking chimney — once the chimney is warmed up the smoke stops billowing into the room.

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. To understand why this takes time consider the following homely analogy. A long corridor is packed with people of different heights waiting to get through a door at the end in order to enter the store. The store manager announces that before he lets anyone in he wants everyone to sort themselves out by height, the short people at the front and the tall people at the rear, with a uniform height gradient between. There is a lot of shuffling about and it takes quite a while for a perfectly even gradient from shortest to tallest to be established. The same is true of a mixture of liquids of different B.P. in a packed column.

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 deg. C., the exact figure depending on the atmospheric pressure (see Appendix V), while the heads such as acetone and methanol have a lower B.P. The thermometer will register this and, although a temperature of 78°C. might be registered at first it will slowly fall a few degrees as the acetone and methanol find their way to the head of the queue.

Periodically crack open the valve in the stillhead a fraction to bleed off these heads into a spoon, leaving room for the ethanol to rise a bit higher in the column. Referring back to the analogy of people of different height shuffling about, if you let some of the shorter people through the door, even if the sorting out isn't quite complete, you will make it easier for the remainder to get organized. A suitable withdrawal rate would be 2 or 3 drops per second.

These heads not only have a strong smell (test them with a spoon) but also a terrible taste so you can congratulate yourself that you're getting rid of them and not 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 78+ deg. C. indicating that most of the heads have now been drawn off and ethyl alcohol is beginning to appear.

### **Temperature measurement**

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 deg. 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 deg. 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. And don't forget that atmospheric pressure affects the boiling point of water. Digital thermometers are extremely useful in that they are much easier to read than the glass type, sit right in front of you on the bench and are accurate enough for our purposes, more accurate in many cases than the other sort.

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. For one thing the temperature is influenced markedly by atmospheric pressure (see Appendix V). Constancy of temperature is sufficient and is what we are looking for. Thus, if the temperature has risen to just over 78 deg. C. and has stayed there for 15 minutes or so you can be fairly sure that all the heads are gone.

Briefly then, proceed as follows: Operate under total reflux for a couple of hours to equilibrate the column, bleeding off the heads periodically

into a spoon and sniffing them until there is very little smell and until the temperature remains constant at just over 78°C. Then start to collect the distillate by opening the valve in the stillhead.

### **Collection rate**

In simple distillation or in beer-stripping you collect everything which vaporizes from the boiler, but in fractional distillation you collect only about 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 back 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 very little 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 distilate 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 on the surfaces within the packing 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 with this type of packing.

So the first step involved in determining just how much alcohol can be drawn off per minute or per hour is to find out the rate at which vapour is arriving in the stillhead, i.e. the boil-up rate. When we have this figure we divide by ten and this is the volume of 96% alcohol which can be drawn off through the collection valve. There are two ways of doing this, one by calculation from the wattage input to the boiler and the other by direct measurement. First by direct measurement using the Mexican cactus stillhead.

With a known wattage input establish steady refluxing conditions and then open the collection valve WIDE for perhaps 30 seconds. It helps to tilt the column slightly to prevent condensate from running back down into the boiler. Measure the output per minute, either in terms of volume using a graduated cylinder or, more accurately, by weight using a sensitive scale. Take as small a sample as you can reasonably measure so as not to unbalance the system. You may wish to repeat with other wattage inputs. The same procedure can be used with either the metal or the glass "hatstand" design of still.

You can also calculate the rate at which a 750 watt (or any other wattage) heater will boil an alcohol/water mixture. The method of making this calculation is thoroughly explored in Appendix II, which also explains a little more about the mechanism of fractional distillation. Or, you might wish to accept the figures we provide below.

We found that with 750 watts input to the boiler the rate of reflux was about 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  $4^{1}/_{2}$  ml of 96% ethanol per minute. In practice we draw off 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, the amount of insulation you have used, the true wattage of your heating element, etc. 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 distilate and put to one side for future processing and then start to collect the purest alcohol in a clean receiv-

er. Throughout this early phase test the distilate with your nose to see if you can detect any trace of heads.

The 250 ml or so of early distilate which have been put aside may be perfectly pure but the nose and the palate are extremely sensitive organs, particularly the palate, (and particularly your wife's palate!), and you would quickly detect an off-flavour if it got through into your final drink. Even commercial producers, with a laboratory full of sophisticated analytical equipment such as gas chromatographs, rely on taste panels to judge the quality of their product. It is called "organoleptic" testing and is the ultimate in testing for palatability. Play it safe, therefore, and put aside a generous portion of the initial distilate, 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 distilate accumulated, it can all be redistilled and really pure alcohol recovered from it. It will amount to a triple distillation and be exceptionally pure.

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. There is no point in collecting extra tails for redistillation because they contain negligible amounts of ethanol.

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 240 ml. per hour, about 3 litres of pure, 96% alcohol will be obtained in a 12 hour day. It can even be left running overnight. This, when diluted to 40% with water will provide over  $7\frac{1}{2}$  litres of yodka

#### Yield of alcohol

In the chapter on fermentation it was explained that the theoretical yield of pure, 100 percent alcohol from 10 kg of cane sugar is 6.25 litres. This is equivalent to 6.58 litres of 96 percent alcohol or 15.63 litres of 40 percent 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. Or the yeast may have lost some of its activity. And then there are all those unwanted side reactions which produce the congeners such as methanol, fusel oils, etc., instead of ethanol. However, the major 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 96 percent 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  $11\frac{1}{2}$  litres of vodka or 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.

# Water quality

A word must be said about the quality of water used to dilute pure 96 percent alcohol to the 40 percent 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.

**Storage:** Store your pure 96% alcohol in glass, not in plastic. A few 1½ 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**

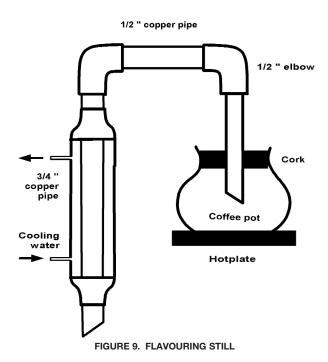
The flavours used for converting vodka to gin are contained in a number of herbs, berries and fruits, collectively known as "botanicals". The preferred method for extracting these flavours is to use steam distillation, a method which is commonly used for extracting the essential oils from many plant materials involved in the production of liqueurs. A brief discussion of the principles of steam distillation will be found in Appendix VI.

#### Steam distillation

Steam distillation requires the use of a simple pot still, and an example of such a still, improvised from a coffee pot, is shown in Figure 9 to illustrate the principle. Depending on how much steam distilling you are thinking of carrying out you may wish to devise and make a much larger and bet-

ter one. One requirement is that it must have a large opening for introducing and removing the botanicals, these botanicals either being used loose or contained in a muslin bag for easy removal. A pressure cooker with a steam condensing system silver-soldered to the lid would work very well. the only disadvantage being that they are somewhat expensive.

The condenser is made from a short length of <sup>3</sup>/<sub>4</sub>-inch copper tubing



acting as a cold water jacket around an internal \_-inch copper tube. Adapters for connecting \(^{1}\structriangle^{2}\)-inch to \(^{3}\structriangle^{4}\)-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. A large cork, obtainable from any winemaker's supply store, is used as lid and has a hole drilled in the centre to take the \(^{1}\structriangle^{2}\)-inch copper tubing from the condenser. It looks crude, and it is crude, but its saving grace is that it works and is very cheap. In operation there is very little pressure in the apparatus and no problems are encountered with steam leakage.

The botanicals and water are placed in the flask and the water brought to the boil. The steam generated releases the flavouring constituents from the herbs and carries them over into the condenser in the form of oily drops suspended in water.

### Gin

In order to illustrate the use of steam distillation for extracting essential oils from botanicals we'll take a look at gin.

As is rather well known the major flavouring ingredient in gin is 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	Cardamom	Anise
Orris root	Ginger	Lemon peel	Cinnamon
Angelica	Nutmeg	Bitter almonds	

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 wine-

making. 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 following recipe has been found to give a pleasant flavour:

Juniper berries	35 g	rams
cardamom	1	"
orris root	1	"
coriander	1	**

Place the above ingredients in the flask (the coffee pot), add about 350 ml of water and install the cork and condenser. Start the cooling water and bring to the boil. The steam generated will carry over 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 distilate add an equal volume of 96 percent 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 percent 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.

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 essences from a winemaker's supply store. These little bottles of essence come in a wide variety of flavours (one manufacturer provides a list of 200) including rum, scotch, brandy, gin, etc. and most liqueurs such as the various fruit brandies, crèmede-menthe, etc. The fruit essences produce very pleasant liqueurs, and the rum is very good, but the whiskies, brandies and other spirits have a somewhat artificial flavour and are a bit too sweet. You would never mistake them for the real thing.

# **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.

### Materials

Sugar and yeast. Flavouring herbs.

# **Equipment**

Fermenter,

Fractional distillation apparatus (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 and the hydrometer.
- 3. Run in tap water. 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 sufficient beer into the boiler to fill it no more than two-thirds full

# **Beer-stripping**

9. Switch on the boiler and run cooling water through the condenser. It will take a couple of hours to come to the boil. Open draw-off valve WIDE. Collect distilate in bottles for later transfer back into the boiler. Temperature of vapour coming from stripper will have risen to about 96-98°C. before switching off.

### Fractional distillation

- 10. Transfer the high wine back into the boiler and add sufficient water to give a total volume of about 12 litres. Close the draw-off 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 distilate and sniff it to detect presence of "heads". Put aside for future use as fondue fuel or discard.
- 12. When no more heads can be detected and temperature is staying completely constant in 78-79°C. range, collect 300 ml. or so of distilate (at the pre-determined rate of \(^{1}\seta\_{10}\text{th}\) 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 stop. This collection will probably last 15 to 20 hours so time it so that you'll be present. 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 hot water.

#### Redistillation

15. When sufficient discard ethanol has been accumulated, about 5 litres or so, pour it into the boiler of the fractionating still and add an equal volume of water, sufficient to cover the immersion heater at the end of the run. (Remember, your discard alcohol has only 4% water in it). Then proceed exactly as in steps 10 to 14 above.

### **Flavouring**

16. Put the selected botanicals into a flask with about 350 ml of water, bring to the boil and collect the condensed steam. Add an equal volume of 96% ethanol to the distilate to dissolve the flavoring 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, approaching \$1000 in fact if you start from scratch. 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, and also some of the minor ones. Prices vary from country to country of course, and it's always possible to make shortcuts, but we feel it's best to be realistic and not pretend that these things can be done for nothing.

The costs provided below refer to the United States, 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. 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. Other variables are: i) the number of items you already have such as fermentation equipment, thermometers, hydrometer, plastic tubing, solder, nuts and bolts. ii) whether you choose to make the two boiler system, the single boiler system, or fabricate something from scrap. And so on and so forth.

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 about \$4 whereas a 1,000 watt dimmer is likely to cost \$40 and a 2,000 watt dimmer \$140. Quite a difference! A sensitive domestic kitchen scale, graduated in 5 gram divisions, can be found if you shop around a bit and at \$10 to \$15 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
LABOR

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 costs of major items are 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 or less so hardly warrants being considered a major item.

#### **Fermenter**

Laundry tub	\$20.00
Glass cover	\$30.00
Circulating pump	\$35.00
Electric heater	\$15.00
Light dimmer	\$4.00
Thermometer	\$10.00
Copper pipe, elbows, etc.	\$10.00
Miscellaneous	\$20.00
Total:	\$144.00

### **Fractional Distilling System**

#### **Boiler**

Water heater, 25 litres. 1650 watts, 115 volts	\$140.00
Replacement heater for 750 watts	\$15.00
Miscellaneous	\$30.00
Total for boiler	\$185.00

Column & Still	-head:
Сор	per column with joints top and bottom,
still	- head, cooling coil, needle valve, etc
Mis	cellaneous
	Total:
Total for still:	\$185 + \$180\$365
<b>Instruments:</b>	
Volt-amr	meter
Sensitive	kitchen scales\$15.00
Measurin	ng cylinders (0 - 10 ml),(0 - 100 ml)\$20.00
Hydrome	eter
Tota	al:\$86.00
Total for all Eq	<b>uipment</b>
Materials & Su	pplies
	owing figures are based on the production of 11 one-litre f gin or vodka from 10 kg of sugar.
Sugar. 10	) kg @ \$1.15/kg\$11.50
Yeast. 15	50 g. @ \$8.25/kg\$ 1.24
Flav	ouring ingredients — negligible cost
	Total:\$12.74
Electricit	cy .
Ferr	mentation negligible
	r-stripping 8 kWh
Frac	etional dist'n 10 kWh
Total: 18	kWh @ 7 cents/kWh\$1.26
Total for	Material and Supplies

### 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 vodka from \$12.74 worth of sugar and yeast and \$1.26 worth of electricity, so that works out at \$1.27 per litre. Not bad.

But how about all that equipment? Let's assume a figure of \$600 for its cost and see how long it would take to pay this off from the savings we realize on making our own vodka instead of buying it. If we produce and consume 1 litre of vodka per week it has cost us \$1.27 against maybe \$20 if we'd bought it at a liquor store. So we save about \$18.75 per week. At that rate it will take us 32 weeks to break even. After that the equipment is free and the cost of the gin would simply be the cost of the ingredients, \$1.27/litre, in perpetuity. A payback period of 8 months would be considered extremely good in industry where 5 to 10 years is much more normal.

Another way of looking at the economics of investing in the equipment is to compare it with the investment required to purchase the vodka 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 vodka with what is left or would one rather lay out \$600 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 already have various instruments and measuring devices. Under these conditions you should be able to bring the costs down below \$400.

The figures used above are simply an example of how to look at the costs and benefits of making your own spirits. In the United States, for example, where vodka is relatively cheap, the savings would be less and the payback period that much longer. Using figures appropriate for where you live — i.e. the cost of making the equipment and the local price of vodka, sugar, etc. — you can work out the savings for yourself.

To allay the concern of 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 N. 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

1 Imperial gallon	=	4.55 litres
1 fluid ounce	=	28.4 millilitres
40 fluid ounces	=	1.14 litres
1 U.S. gallon	=	3.78 "
1 U.S. quart	=	0.946 "
1 litre	=	35 fluid ounces
	=	0.22 Imp. gallons
	=	0.26 U.S. gallons
	=	1.04 U.S. quarts

### Weight

1 pound (lb)	=	= 454 grams		
1 ounce (oz)	=	28.4 "		
1 kilogram (kg)	=	2.2 pounds		
1 gram (g)	=	0.035 ounces		

### Length

1 inch = 2.54 centimeters (cm)

1 foot = 30.48 "

1 centimeter = 0.39 inches

1 meter = 39.37,

### **Temperature**

32 deg. Fahrenheit (F) = 0 deg. Celsius (C.)

212 deg. " = 100 deg. "

General:

 $[\deg. F. - 32] \times 5/9 = \deg. C.$ 

#### **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**

## Latent heat of vaporization

In order to know how much pure alcohol can be produced per minute or per hour by a 750 watt immersion heater we first need to know the rate at which the alcohol in the boiler is being vaporized and condensed in the still-head, i.e. the boil-up rate. When we know this volume we take 10 percent of it. That is the amount we can draw off and put into our martinis.

As discussed in the text, there are two methods of determining the rate of vaporization from the boiler — by direct measurement and by calculation. The calculation method is outlined below.

The rate at which liquid is vaporized is dependent upon two quantities; a) the energy input to the boiler, and b) the latent heat of vaporization of the liquid in the boiler (LHV). The LHV is the amount of energy required to convert a boiling liquid into vapour at the same temperature, and it is a surprisingly large quantity. The reason why energy is required to convert a boiling liquid into vapour without any rise in temperature is that molecules in a liquid are much more closely packed than in a vapour, and to convert one into the other the molecules must be wrenched away from the clutches of their fellows and push against the atmosphere. It takes energy to do this.

The energy required to vaporize water, i.e. the latent heat of vaporization (LHV), is 540 calories per gram. For ethyl alcohol the energy required is 220 calories per gram, the lower value being a reflection of its greater volatility. The composition we are involved with is 95% alcohol w/w. Simple arithmetic gives 236 calories per gram for the LHV of the 95% w/w alcohol azeotrope.

Why, you might ask, are we concerned with the energy required to vaporize 95% alcohol when we know very well that the contents of the boiler are mostly water and this water is being vaporized along with the alcohol? The explanation is this: 95% of the water vapour going up the column, car-

rying with it its latent heat of vaporization, is condensed in the column by the descending flow of liquid from the stillhead. The 5% water which does get through only does so because it is associated with ethyl alcohol in the azeotrope. When the 95% water condenses in the column it gives up its energy, this energy being known as the latent heat of condensation (LHC). It has the same value as the latent heat of vaporization. Therefore, the only energy escaping into the stillhead is the latent heat contained in the 95% alcohol and the 5% water. That's all there is in the stillhead and all that is being condensed by the cooling coil. Most of the water never gets there.

It is known that 860,000 calories/hour = 1 kilowatt. Therefore 860 calories/hour = one watt and 236 calories/hour = 0.27 watt

What this means is that 0.27 watts of electric power are required to vaporize 1 gram of a 95% alcohol/water mixture in one hour, so 750 watts would vaporize 2778 g/hr. or 46 g/minute. Ethanol having a S.G. of 0.8 the volumetric figure for the total reflux rate is 58 ml/minute.

When we measured the rate of reflux at total reflux with 750 watts input to the boiler we found a value of 45 ml per minute. This is less than the calculated value of 58 ml per minute because of heat loss due to imperfect insulation. This loss is equivalent to 168 watts.

If you cannot or do not wish to measure the rate of reflux yourself, you could use our figure of 45 ml. The insulation used for your boiler and column may be better or worse than ours, but is unlikely to differ very much, so you'd be pretty safe to use this figure of 45 ml.. This would mean that you could draw off 10% of this, or 4.5 ml per minute, as usable alcohol. This is particularly true since the reflux ratio of 10:1 is not critical anyway.

A footnote to this discussion is that the rate of reflux does not change during the course of a distillation, even though alcohol is steadily leaving the boiler and changing the composition and the boiling point of the liquid in the boiler. The composition of alcohol vapour in the <u>stillhead</u> remains constant from the time the heads are finished until the arrival of the tails, and that's all that matters; the composition of the liquid in the boiler is irrelevant.

# **Appendix III**

## **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, produces moonshine, and this contains some pretty unpleasant things, so activated charcoal remains the only hope of removing some of the worst of them and producing a palatable beverage. By contrast, the alcohol produced by the equipment described in this book should not require "cleaning up" because all the unpleasant things have been removed in the distillation process. Mistakes can happen, however, particularly in the early days before experience has been gained, and when it does one may be faced with a batch of alcohol which is a bit "off". In such cases a polishing with activated charcoal may be beneficial.

Activated charcoal is used in gas masks, in water purification and in many other areas where small quantities of an adulterant need removal. Its effect is a physical one, not chemical. The adulterant is adsorbed on the enormous internal surface area available. This surface can amount to  $1000 \, \text{m}^2/\text{gram}$  and is produced in a number of ways but often through the use of superheated steam on ordinary charcoal. The cheapest source is a water treatment company.

To use it, dilute the alcohol from 96 to 40% (vodka strength) and use about 150 grams of charcoal per 6 litres of ethanol. Put into a container, stir occasionally over 5 days, allow to settle and then filter. It is a messy and time-consuming business and you may find it more convenient to use a continuous charcoal treatment. Clamp filter paper over the end of a 1½-inch pipe, add charcoal to a depth of 12 inches or so, and then pour the alcohol through. It should be completely pure when it emerges.

The best method of obtaining pure alcohol is to distil it so well that no charcoal treatment is necessary. It is cheaper and saves a lot of time and trouble. We have not used charcoal for the last 15 years and you will find that, with experience, you too can dispense with it.

# **Appendix IV**

# **Cooling water requirements**

A number of people have expressed concern about the volume of cooling water required to condense the vapour from a 750 watt heater operating over many hours. It is not all that great, but if water is scarce or expensive where you live you will be interested in the following calculations.

The calculations cannot be exact because there are many imponderables. For example, the temperature of the cooling water, the permitted rise of cooling water temperature, the desired drop in the temperature of condensed alcohol, the rate of heat transfer between the cooling water and the alcohol (affected by thermal conductivity of coil material, e.g. copper, stainless steel, glass, and the thickness of the coil walls), so please read the following with these things in mind.

We are going to assume the following: The cooling water enters the coil at 10°C. and leaves it at 30°C., a 20° rise in temperature. By increasing the flow of cooling water you could decrease this rise in temperature, and by accepting a greater temperature rise you could reduce the flow of water. We also assume that the alcohol vapour is condensed in the stillhead and, following condensation, is cooled from 78.1°C. to 68.1°C., a drop of 10°C., before withdrawal.

The cooling water in the stillhead is condensing 45 g/min of a 95% w/w alcohol-water mixture (see Appendix II). The latent heat of this mixture is such that 10,856 calories per minute of energy must be drained off by the cooling water. The latent heat of vaporization of the cooling water is not involved, only its sensible heat, and this is 1 calorie per gram per degree C., the specific heat of water. So, just to condense the vapour without changing its temperature we require 10860 grams of water per degree C. per minute. Let's call it 10 litres. The collection of alcohol from a particular run will occupy (let's say) 20 hours. So the number of litres of cooling water would

be 20 x 60 x 10 litre = 12,000 litres. This is just to condense the alcohol, not cool it. If we decrease cooling water flow so that its temperature rises, not by 10 C. but by 200 C. then the volume of water would be reduced to  $12,000 \div 20 = 600$  litres.

You might wonder why the stillhead doesn't cool the alcohol to room temperature. It is a matter of experience that, using the type of stillhead with cooling coil described in this book the alcohol vapour condenses on the lower turns of the coil, turns into liquid, and immediately drops off, avoiding further cooling. It is so hot, in fact, that some people suggest cooling it further by having the condensed liquid flow through a secondary heat exchanger before dropping into the collection bottle. Otherwise, they say, a lot of alcohol will be lost by evaporation. There is some truth in this but we have found it sufficient to draw off the hot alcohol and let it fall through a copper tube before entering the collection bottle. In effect, this is an aircooled condenser.

We have calculated that 600 litres of cooling water are required just to condense the vapour. Now let us assume that the condensed liquid, before dropping off the bottom turns of the cooling coil, is further reduced in temperature by 10°C., i.e. from 78.1°C. to 68.1°C. This will require additional cooling water as follows:

We are concerned here with, not latent heat of condensation but the specific heat of alcohol. This varies a bit with temperature but is about 0.6 calories per gram per degree C. So the number of calories to be withdrawn for a  $10^{\circ}$  C. drop in temperature is:  $10 \times 0.6 \times 46$  grams per minute = 276 g/min or 330 litres of cooling water over a 20 hour distillation period.

Therefore, 600 + 330 = 930 litres of cooling water are required in *toto*. To this, of course, must be added the water consumed while the column is being equilibrated. And then there's the water consumed during beer stripping. Whether or not you consider this a lot of water depends on your particular circumstances. If you feel it <u>is</u> a lot then you might wish to try air cooling by circulating the cooling water through a car radiator and blowing air through it. This would also avoid the need for a drain. And if you wished to get really fancy you could experiment with circulating freon through the cooling coil and refrigerating it.

# **Appendix V**

# Effect of pressure on boiling points

The boiling points of liquids quoted in reference books refer to the values measured at a standard atmospheric pressure of 760 mm mercury. As we all know, atmospheric pressure changes, varying considerably from day-to-day as weather patterns change and cold or warm fronts cross the region. Atmospheric pressure also changes with elevation. Not everyone lives at sea level under a stable air pressure of 760 mm Hg so the following table will allow you to interpret any temperature readings you might get in terms of ambient atmospheric conditions.

					Boiling	point
Pressure			Elevation	Ethanol	Water	
mm Hg	inches Hg	kPa	millibars	Feet	°C.	°C
853	33.6	113.7	1137	-3280	81.5	103.3
806	31.8	107.5	1075	- 1640	79.9	101.7
760	29.9	101.3	1013	Sea level	78.4	100.0
716	28.2	95.4	954	1640	77.0	98.3
674	26.5	89.8	898	3281	75.6	96.7
634	25.0	84.5	845	4921	74.2	95.0
	853 806 760 716 674	mm Hg inches Hg  853 33.6  806 31.8  760 29.9  716 28.2  674 26.5	mm Hg         inches Hg         kPa           853         33.6         113.7           806         31.8         107.5           760         29.9         101.3           716         28.2         95.4           674         26.5         89.8	mm Hg         inches Hg         kPa         millibars           853         33.6         113.7         1137           806         31.8         107.5         1075           760         29.9         101.3         1013           716         28.2         95.4         954           674         26.5         89.8         898	mm Hg         inches Hg         kPa         millibars         Feet           853         33.6         113.7         1137         - 3280           806         31.8         107.5         1075         - 1640           760         29.9         101.3         1013         Sea level           716         28.2         95.4         954         1640           674         26.5         89.8         898         3281	Pressure         Elevation         Ethanol           mm Hg         inches Hg         kPa         millibars         Feet         °C.           853         33.6         113.7         1137         - 3280         81.5           806         31.8         107.5         1075         - 1640         79.9           760         29.9         101.3         1013         Sea level         78.4           716         28.2         95.4         954         1640         77.0           674         26.5         89.8         898         3281         75.6

Not too many of us live below sea level but quite a few must live at elevations of several thousand feet, and it will be seen from the above table that the effect on the boiling point of ethanol is far from trivial. The same holds true of changes in atmospheric pressure at a fixed elevation, due in this case to the movement of air masses.

You will recall from the discussion of temperature changes during

fractional distillation that, after the column has reached equilibrium, the heads are bled off until the temperature remains constant, indicating that pure ethanol is now distilling over. Clearly, to avoid being misled, it is useful to have some idea of what the boiling point of pure ethanol is on that particular day. The table will help in this regard.

# **Appendix VI**

## Steam distillation

A brief description of steam distillation was given in the chapter dealing with flavoring, where we showed how to extract the essential oils (chiefly —pinene) from juniper berries and other botanicals. But steam distillation is not, of course, restricted to juniper berries and gin flavoring — there is a whole world of plant materials out there containing aromatic and flavorsome oils, and many readers have expressed a wish to know more about the extraction process. At some later date we may write a "how to" book on the subject, but for the time being a few words attached to the present book could be of interest.

### Principles of steam distillation

Whereas ordinary distillation deals with the separation of miscible liquids, e.g. water, ethanol, methanol, etc., steam distillation deals with the separation of immiscible or partially miscible liquids, e.g. oil and water. When two immiscible liquids are heated, each exerts its own vapour pressure independently of the other. When the <u>sum</u> of the vapour pressures of the two liquids becomes equal to the atmospheric pressure, the two distil over together, and the temperature of distillation and the composition of the distilate remain constant until one of the liquids is entirely evaporated.

An example of how steam distillation works will be given, drawn from the literature, using water and chlorobenzene as the two liquids. A mixture of these two liquids was distilled when the atmospheric pressure was 740.2 mm of mercury. The mixture boiled at 90.3° C. At this temperature the vapour pressure of water is 530.1 mm Hg while that of chlorobenzene is 210.1 mm, making a total of 740.2 mm. Chlorobenzene has a boiling point of 132° C., yet when distilled with steam at a temperature 42° C. lower, the distillate contained over 70% of the organic compound.

Another example is aniline and water. Under the standard atmospheric pressure of 760 mm Hg a mixture of these two liquids boiled and distilled over at 98.5° C., at this temperature the vapour pressures of aniline and water being 43 mm and 717 mm respectively, for a total of 760 mm.

### **Steam distillation** — practice

Most people who read this book will be interested in the steam distillation of plant material in order to isolate the essential oils contained in the leaves, needles, berries, etc. One could build a steam generator and conduct the steam through a bed of plant material contained in a kettle, which is the method used commercially, but a simpler system consists of a kettle containing water at the bottom and a grid just above the water holding the plant material. When the water is boiled the steam carries over the essential oils into a cooling condenser where the two liquids collect and separate out into two layers.

Unlike the boiler described in this book, where a mixture of miscible liquids is being distilled and where liquids can be introduced and removed through <sup>3</sup>/<sub>4</sub>" piping, for steam distilling plant materials it is necessary to have a large opening in the boiler (kettle) to add and remove solids.

## The Author

The author has his Ph.D. in physical chemistry from the University of London, England, and has published over seventy scientific papers concerned with the chemistry of plant materials and the production of fuel alcohol from agricultural residues. He eventually became the Director of the Forest Products Laboratory in Ottawa. He is now spending his retirement years in a small village in eastern Canada on the shores of The Lake of Two Mountains.

His interest in the theory and practice of small-scale distillation stems from a botched attempt at making wine many years ago. It was so awful that it should have been poured down the drain. However, he decided to try and recover the alcohol by distillation and found to his chagrin that it was not as simple as it seemed. This "how-to" book, like its predecessors, is the result.

