

**Bush Boake Allen Essential Oils** 

Bush Boake Allen Essential Oils 
A review of the expertise gained during more than 100 years of distillation and processing of essential oils and the technological advantages to be gained from the usage of these products

Lecture by Henry Heath

#### INTRODUCTION

The industry, science and technology of usage of the essential oils have undergone very radical changes within the past twenty years. The pace of our increasing knowledge is rapid and more far-reaching than could ever have been anticipated by the early workers in this field. Illustrious names like Gildemeister, Wallach, Tiemann and Hoffman, to mention but a few, established by classical chemical analytical methods the firm basis from which such advances have been made. In introducing this fascinating topic I do not propose to take too long a backward look, but it is both interesting and useful to know something of the long history of these valuable flavouring and perfumery raw materials.

Before going further, however, it is necessary to establish just what we mean by the words "essential oils". Early references simply define these as the volatile oily substances obtained by steam distillation from plant material. a definition was obviously aimed at differentiating between them, and the fixed or fatty oils which were obtained by expression from certain plant seeds. The prime characteristics of the essential oils is that they are volatile and are of plant origin. For this reason it is now necessary to include in the definition those essential oils which are obtained by means other than distillation. For example, many of the citrus oils such as lemon and orange are obtained by mechanical expression. Certain oils do not exist per se in the living plant and are formed only as the result of an enzymic reaction after the plant material has been crushed and macerated with water. The oil which is then formed in the plant tissues can be recovered by distillation. Examples of these oils are mustard oil and onion oil. Most of the delicate floral oils are recovered by either cold or hot solvent extraction and it is only by such methods that one is able to capture the delicate natural perfume present in the flowers themselves.

To summarise, then, we can say that an essential oil, or a volatile oil, as the two expressions are used synonymously, is a volatile mixture of organic compounds derived by some physical process from odorous plant materials. A specific oil is derived from one botanical source with which it agrees in both name and odour.

Essential oils are found in special cells, glands or ducts that are located in several parts of the plant - i.e., in the leaves, barks, roots, flowers, fruits and seeds - sometimes confirmed to particular structures and in others throughout the whole of the plant's tissues. As a rule, the oils are present in only very small amounts and so constitute only a small fraction of the total plant weight. The quality and composition of the essential oil varies not only with the type of plant but also, in particular, with the conditions prevailing during its growth, i.e. light, climate, soil and altitude.

The function of the essential oils in the living plant is far from completely understood. Odours of flowers may be directly concerned with insect attraction or repulsion, so affecting the pollination and - to some extent - the natural selection. Many may act as a form of protection against parasites and others make the plant objectionable to animal depredation. Whatever their botanical value, they are a wonderful source of raw materials for the flavourist and the perfumer.

## LOOKING BACK

The history of progress in the techniques of distilling in the pharmaceutical context is, in fact, not easy to trace. Reference to one or two oils, other than turpentine, can be found as early as the beginning of the 14th century, but it was not until the early 16th century that the first substantial evidence of oils being collected by a distillation process

was recorded. From that time onwards progress was steady and fruitful. The most active workers in this field were French and German, and there were the usual band of chemists, pharmacists and physicians - all obviously very convinced that the herbaceous plants provided by providence concealed something of great medicinal benefit to the human race. The process of distillation is only one of the ultimate accomplishments of this cooperative effort and a great variety of other products, known as liquors, infusions, extracts. tinctures, decoctions, etc., were produced as part of a rapidly increasing knowledge of the curative properties of many plants. Any material put into a vessel for pharmaceutical processing was known as a drug and, strangely, the description is still widely used today. Pharmacies appear to have provided the laboratory facilities as well as a great deal of the know-how in the earliest distilling experiments. It is not surprising, therefore, that the connection with pharmaceutical practice has been preserved right up to the present time, as indeed it is in our own company.

By the beginning of the 17th century at least sixty essential oils were known, but much had still to be done to establish the facts about their chemical constitution and also their medicinal value. It was not until much later - in fact, well into the 19th century - that reliable analytical ability began to reveal something of the nature of these volatile oils. This was the beginning of a flood of knowledge regarding their properties and, particularly, about the chemical constitution of the compounds which we now know as the terpenes. This sort of progress soon gave rise to new ideas about the likely uses of these fascinating natural products and, particularly so, when spices proved to be so amenable to the distillation process. The cause of medicine, in consequence, slumped a little but fortunately the old-established collaboration between the professions was maintained. Now, of course, there prevailed

a keen commercial atmosphere which is the best possible stimulus to further progress. It was at about this stage of history that our own roots became well established and our ideas, it seems, firmly fixed on processing natural materials. Then, as now, our motto seems to have been - "only the best is good enough" - and, in the prevailing chaotic conditions, that was indeed a very commendable aspiration.

In the light of over a century of distilling essential oils, this ambition and optimism of our Bush Boake Allen forbears has proved to be fully justified and rewarded by the success and lasting reputation of the proudly proclaimed English distilled essential oils. Our legacy, therefore, is a great name and a great responsibility.

In due course, knowledge of what was going on in Europe and what was being done with the materials, now so much in demand, began to stimulate the idea of on-the-spot distilling in the faraway places in which the plants were native. Out of this eventually grew the family-type business of growing and distilling a suitable crop and disposing of the resulting oil to a merchant. The custom spread to many countries and oils like aniseed, cassia, citronella and lemongrass were produced in this way. As was to be expected, the native pioneers usually had only makeshift equipment and their technology was crude and mostly rule of thumb. However, what they lacked in this respect they certainly made up for with a considerable degree of ingenuity. Paraffin oil and rosin, and even water were soon proved to be useful extenders of the oils which they obtained and stones and scrap metal were good for making up the weight of the raw plant materials when these were offered for sale.

This, then, was the background against which we were determined to establish some sort of order and stability into the new products of natural origin.

### THE PRESENT

Essential oil production, even today, ranges from extremes of primitive cultivation and inefficient field stills to intensive farming and ultra-modern processing and distillation methods. Unfortunately, most aromatic plants still grow wild or, at best, are cultivated by primitive methods in small quantities as garden or patch crops by native smallholders. We realise only too well that skill and long experience are important in cultivation, as the crop must be collected at the right time to ensure the best yield of the correct oil. Any error or slackness at this stage can result in an oil having an unacceptable odour or flavour profile. Only a very few aromatic plants distilled for their essential oils are cultivated by methods based on modern agronomic practice, involving selected plant breeding, mechanised agricultural methods and a skilled labour force. The reason for this is the high capital investment involved so that such an outlay can only be justified when the demand for the oil warrants it. The floral oil industry based in Grasse in southern France, the citrus oil industry in both Florida and California and the immense peppermint and spearmint growing operation in the western states of the U.S.A., are all good examples of such concentrated expertise.

The processing of natural vegetable materials which, of course, includes a very large proportion of our food, has undergone countless changes in the basic techniques during the past century. Nowadays, simple processes such as cooking and baking on a factory scale are a completely new dimension but, in the pharmaceutical field, the basic processes like distillation and extraction have changed hardly at all either in technique or in dimension during the past hundred years. Refinements in equipment have, of course, resulted from the better understanding of the chemistry of the aromatic material but, basically, the process of prime distillation is much the same today as it always has been.

As a company, Bush Boake Allen is directly concerned with the specialists approach to the production of essential oils. In our factory at Long Melford, one finds the most advanced distillation plant designed to achieve the optimum conditions for the production of essential oils. The superior quality of these oils is far removed from those produced under primitive and largely uncontrolled field conditions.

# THE RAW MATERIALS

One of the first things we learned about distilling was that results depended very much on the quality of the material used. As we extended our range of imported materials, so our difficulties in maintaining uniformity and the desired high standards became greater.

## The sources of variation

It may sound very elementary to say that any given raw material must always conform to a specified botanical type. There is, unfortunately, a great deal of overlapping in the natural orders and this, added to the ever-present vagaries of climate, habitat and cultural conditions, makes selection of the material for distillation an exacting responsibility.

(For instance, thyme - which grows in most European countries and, particularly round the Mediterranean shores, - is a very good example of the many different flavours to be obtained from the same botanical entity - Thymus vulgaris.

The same difficulties apply to the origanums and marjorams, both individually and collectively with the thymes because, within this whole family there is very considerable overlapping and cross-fertilization resulting in hybrids.

There is also another very confusing complication in the fact that many plants produce oils in different parts of their

anatomy. The oils may be similar in character and constitution, as in the case of cassia bark, and cassia leaves or angelica root and angelica seeds but, in most cases, they are quite different.

This is best demonstrated in the case of cinnamon bark and cinnamon leaves, or pimento berries and pimento leaves and, perhaps the best example of all - clove buds, clove stems and clove leaves.

I would like, at this stage, for you to examine the samples in front of you which illustrate these differences very well.

In the case of our English distilled oils we are, however, on very safe ground because these are all derived from a very carefully defined part of a plant which, in turn, has both a specific botanical entity and a clearly defined proscribed geographical source.

In addition to these general requirements, there are further detailed considerations such as age, grade, oil content and oil constitution of the raw material - all of which have to be taken very carefully into account in our purchasing specifications. Finally, to narrow down as far as possible the risk of variation in the product, it is customary to use material from a specified location as, for example, Spanish thyme to the exclusion of French or Moraccan, or any other source. In many instances, indeed, the crop is grown specifically for our use. No material is accepted into our factory until a representative sample has been through the tight mesh of our analytical and quality control laboratories.

Unfortunately, a detailed study of the source of all the English distilled oils would take far longer than we have time available, but we can look at the basic principles which govern our standards for the whole range and perhaps take one or two examples for closer examination.

Caraway seeds from Holland are a perfectly good example of material from a specified source. These are the best seeds available because they have been cleaned and freed from dirt and dust, immature seeds, and the many foreign seeds, such as the grasses, which are present in the less carefully prepared samples. They are, of course, the most expensive caraway seeds which we can buy but we know that they will give the highest yield of oil - somewhere about 4.5%, containing the maximum amount of carvone, i.e. about 60%. There is, also, a distilling quality of caraway seeds available on the market. These are merely the siftings and sortings of the best quality export grade which I have just described, but the distillation results obtained, related to the cost of the raw material, do not in any way compare with those of the best seeds and, furthermore, the flavour of the oil is considerably inferior and may, indeed, be abnormal due to the presence of an excessive amount of foreign matter.

The same considerations apply to other seeds in the umbelliferae and these include, celery, coriander and fennel.

Cloves is perhaps the best material of all to illustrate our ability for obtaining the best results. The finest oil can only come from the best quality clove buds and I do want to emphasize the words "clove buds". As I said previously, both clove stem and clove leaves also yield oils and, although both have analytical pretensions in terms of the prime constituent engenol, they are, in odour and flavour, bad by comparison. is because they contain little or no eugenyl acetate whereas the bud oil has up to 15% of this sweetening ester which makes the characteristic difference. The harsh terpene, caryophyllene, and traces of naphthalene are also present in the stem and leaf oils whereas the bud oil is virtually free from these compounds. In selecting clove buds for distillation the greatest care has to be taken to make sure that they are free from stems and leaf fragments because, even minute amounts of these will contribute off-notes to the aroma which will seriously impair

the quality of the bud oil, particularly from the perfumery point of view. Here again, the exporting of the best quality clove buds poses the problem of the disposal of the sortings, and this is usually done by a local distillation - such oils being used in the preparation of cheap blends. Incidentally, when it comes to the question of purchasing blended oils, if one is not very alert it is very easy to be led astray.

Cardamom is one further example of our specifications drawn up to control this elegant and most English of all the essential oils. We clearly specify that the material to be purchased shall be Alleppi green cardamoms, signifying - of course - fresh material with a tightly closed unbroken capsule with no splits and a promise of a 6% yield of essential oil. In this instance, the capsule should not have been bleached. In fact, this variety is only a fancy grade for the spice trade.

The same meticulous consideration and stringent limits apply to all the materials which yield essential oils for inclusion in our English distilled category. The best materials, however, will only produce best oils if the equipment and the technical skill is of the same calibre.

#### THE DISTILLATION PROCESS

To define the best equipment for handling a very wide range of aromatic raw materials is obviously not possible. As a result of long experience we have established that each still has to be of a size, shape and general design which best suits the material that will go into it. As a result, an efficient distillation department displays a very varied assembly of distilling apparatus. Our own facilities, in fact, conform very closely to this pattern and we operate what is perhaps the most modern distilling plant of its kind.

As with equipment, standardisation of processing methods is also impossible. Every material requires individual consideration

and treatment. Distilling times for each charge vary enormously, ranging from as little as an hour or two for some of the seeds or fresh herbs, to as much as 100 hours or more for a hard material such as sandalwood. Some distillations are carried out by direct steam injection, others by what is known as water distillation. There is no rule of thumb which gives one an indication as to the best method to use. This is purely a question of long experience.

In the case of steam distillation, the comminuted plant material is packed into the still, frequently on spaced supporting grids, and live steam - often at pressures higher than atmospheric - is introduced through a perforated steam coil below the charge. The oil-laden steam passes into a suitable condenser and the oil separated from the condensate in a specially designed vessel. In some stills a re-boiler unit can be incorporated so that the separated water can be re-used, not only as a further source of steam, but also to ensure an efficient stripping of any residual aromatic fractions which may have been carried over from the separating vessel.

In the case of water distillation a mixture of the comminuted material and water is heated while being mechanically stirred. Condensation conditions are similar to those used in steam distillation and after separation of the oil from the condensate the water is returned to the still by way of a special water trap.

Some materials can be distilled by either method but, usually, there is an advantage in one method over the other. Clove buds, for instance, respond better to water distillation. The yield of oil is a little less than that achieved by using steam distillation and the distilling time is much longer - almost double in fact - but the quality of the oil is incomparably better. We, of course, adopt the method which yields the highest quality oil.

Distillation, like another traditional process - brewing, is a complex of art and science and it could be said that the equipment is only as good as the people who use it. There are very many temperamental oils, like cinnamon and onion to quote but two, which will only respond to the master's touch and are never, never for beginners - even with the very best equipment. The niceties of distillation techniques are not amenable to description in text books. They hover, it seems, rather like haloes around the stills to illuminate the need as it arises.

In the case of celery seed oil, for example, sedanolide is one of the most important constituents and this compound comes over late in the distillation process, i.e., in the high-boiling fractions. If the distillation is pressed beyond the point necessary to capture all the sedanolide, an undesirable note will begin to show in the odour of the oil. An expert will immediately settle for a yield of perhaps 2.5% at the optimum point, whereas the tyro - looking for maximum return - will proudly collect 2.7% with, of course, tragic results in the quality of the resulting oil.

Throughout the range of English oils there is a built-in history of experience and skill in each individual oil which only time can produce and which only the oils themselves can exhibit.

#### THE ENGLISH DISTILLED ESSENTIAL OILS

Before leaving the specific subject of the English distilled oils there are one or two points to which I would like to refer relating to their general status in the highly competitive and exacting conditions of modern business. First of all, they are what they are labelled and there is no equivocation about what that label means. They also represent the best that can be achieved with the finest materials of universally accepted standards. In true flavour and odour potential they are the most

concentrated products of their kind because they are produced from material as near 100% pure and genuine as we can locate and specify. They are also all prime distillations carried out under the strictest technological control in our own factory and they are not subjected to any subsequent treatment such as rectification or distillation which could in any way impair the constitution of the truly natural character of these prime essential oils.

## ESSENTIAL OIL BLENDS

In all discussions about essential oils one must be absolutely certain of what one means. When we say cinnamon oil we open up an enormous gap between the two extremes of cinnamon bark oil and cinnamon leaf oil. You have already examined the samples in front of you of these oils so you will appreciate the vast difference that exists between them. Such gaps are filled by what we call blends. Once we enter this field it becomes a matter of taste or, perhaps it would be more correct to say - what do we expect to get for our money. Whatever the blend may be, it is pretty certain that any pretensions to quality or even to authenticity will depend entirely on the amount of the genuine oil which is in it.

Most of us like good wines and we choose these with very great assurance. We also like hard liquors, where the choice might be between the very finest cognac on the one hand and methylated spirit on the other. We know that, indeed, there are tastes for both, but the extremes are very wide and very closely defined and there are all stages inbetween. We all have our likes and dislikes. I can best illustrate this by telling you of our experience in Malaya. The Malayan native thoroughly enjoys a hand-made cigarette containing shredded cloves in the tobacco - the whole being rolled into a conical shape inside a rice leaf blade. To inhale from this local delicacy is, to the uninitiated, a once in a lifetime experience. It can only be

likened to sword swallowing and, when you recover from the effects, you are really not worrying too much about the quality of the clove which was used in its preparation. The Malayan, on the other hand, can readily distinguish even very small differences in his normal 'smoke'. This we soon discovered when we once tried to put clove stem or clove leaf oil into the tobacco instead of the normal shredded clove buds.

## CONSTITUTION OF ESSENTIAL OILS

I want to consider other processing techniques applicable to the essential oils but, before doing so, I think it would be as well to establish something of the constitution of these natural products. Early investigation showed that the oils are very complex mixtures of several substances although, in certain instances, one main constituent may predominate to the extent of 85% or more. The constituents, for the sake of convenience, may be classified as follows: -

- (a) the hydrocarbons of the general formula  $(C_5H_8)_n$  and
- (b) oxygenated derivatives of these hydrocarbons

The fundamental building blocks of the hydrocarbon group of compounds is the isoprene unit  $(C_5H_8)$ . Two such units joined together, i.e. when n=2, are known as terpenes  $(C_{10}H_{16})$ . Where n=3, they are called sesquiterpenes and when n=4 the compound is called a diterpene. A further, more sophisticated classification is possible, depending upon the chemical structure of the hydrocarbon molecule. At this stage, however, I do not think that we need to go any further into this very complex field of chemistry.

The second group of oxygenated derivatives may, however, be classified with great advantage as follows:-

(a) <u>alcohols</u> - these include several important members such as menthol, which is found both free and in a

combined state in peppermint oil, geraniol in palmarosa and citronella oils, and linaloöl in oil of bois-derose.

- (b) aldehydes
- this class includes such well-known compounds as citral, the principal compound in lemon oils, citronellal in citronella oils, cinnamic aldehyde in cinnamon bark oil and benzaldehyde in bitter almond oil.
- (c) esters
- the principal esters include linalyl acetate, present in both bergamot and lavender oils, menthyl acetate, present in peppermint oils, and geranyl acetate present in geranium oil.
- (d) ethers
- these include anethole which is present in aniseed oil and safrole, present in sassafras oil.
- (e) ketones
- among this group of oxygenated constituents are the ketones thujone, which occurs in Dalmatian sage oil and carvone, in caraway and dill oils
- (f) phenols
- probably the best known phenols are eugenol and thymol occurring respectively in cinnamon leaf and clove oils and in thyme and certain origanum oils.

There are several other groups of oxygenated bodies such as the oxides and lactones and also certain compounds containing nitrogen

and sulphur which are of great significance in specific oils.

An understanding of the chemical nature of these compounds helps us to explain some of the well-recognised characteristics of the essential oils.

The terpenes and sesquiterpenes possess as their outstanding characteristic that of comparative insolubility in weak spirit and it is due to their presence in the essential oils that the oils themselves are relatively insoluble in this solvent. The second characteristic of the terpenes and sesquiterpenes is their tendency to oxidise readily. This is due to the fact that they are unsaturated hydrocarbons and tend to resinify with the consequent deterioration of taste and odour. This property of resinification can be seen in the thickening up of certain essential oils on long standing and, of course, is made use of in the case of oil of turpentine in the paint and varnish industry. Thirdly, the terpenes and sesquiterpenes possess little flavour value. It is probably not quite true to say that they have no flavour value as they may contribute a little to the overall flavour effect of the oil but, in comparison with the flavour value of the oxygenated constituents, their contribution to the total effect is relatively small.

Although, as I have already mentioned, one main constituent sometimes predominates in an essential oil, the presence of the other substances, even though they may be present in only a fraction of 1%, may modify the odour and flavour and, in some instances, such minute trace materials can be entirely responsible for the characteristic odour of a given oil. Thus, in lemon oil, in addition to the citral present we have other substances such as geraniol, geranyl acetate, linaloöl, linalyl acetate, terpineol, etc., all of which have their part to play in modifying the citral note. Again, in clove oil, where the prime constituent is eugenol, the presence of eugenyl acetate, methyl amyl carbinol and methyl amyl ketone all play a very significant part in rounding off the clove oil flavour when this is compared with pure eugenol.

These differences can be appreciated by examing the samples in front of you.

# FURTHER PROCESSING TECHNIQUES

Having considered the chemical nature of the essential oils, I would like to go a little further into our other essential oil activities and to tell you something about our reprocessing of imported oils, particularly the mint oils and citrus oils. You will probably already be familiar with the terms "fractionation", rectification" and "deterpination". All of these techniques we do with the same skill and devotion based on our long experience, as with the prime English distilled oils and, again, the quality of our products is closely dependent upon the nature of the oils used in their preparation.

# Peppermint

I would like now to look at a very important, and I may say at a very interesting, side of our business - the handling of peppermint oils - not to be confused incidentally with the arvensis mint oils.

Before we can have good rectified peppermint oil we must first find a really good natural oil and we can look in several different directions for this. Peppermint (Mentha piperita) grows in North America, Italy, Russia. Morocco, France and several other European countries including England. Although the production in England is very very small indeed, all the peppermint grown today is said to be derived from the famous Black English or Mitcham Mint. Naturally, we are experts in the rather primitive hand-operated methods of English oil production, but we are also very well versed in the highly mechanised American system. The English peppermint oil is good by any standard, but the economics of the operation are now hopelessly out when compared with that in the United States.

A yield of 4 kilos per hectare or 22 lbs. per acre in England is considered to be quite good whereas, in American, it would spell complete failure. There the figures vary between 12 and 20 kilos per hectare or 65 - 110 lbs. per acre, according to the season.

Needless to say, we take a very close interest in anything that is destined for our warehouse and our distillery and we maintain, therefore, a continuous liaison on the entire cycle of operation from the selection of the stock, through planting the herb, harvesting and finally to the distillation of the oil. We regularly visit the scene of this operation and receive regular reports of growth, weather, weed control and irrigation right throughout the season. By mid-August the fields tell their own story about the care and attention they have had or indeed, perhaps, the lack of it. they look the more acute will be the concern of the good farmer. The time of decision about cutting is rapidly approaching and he knows that to be too early is just as bad as being too late. As with most things, there is an optimum point in the life of the herb which satisfies both the yield and the quality requirements of the oil, and the beginning and the end of the cutting and distillation operation must be as close as possible to this optimum. When there are several hundred acres to consider, this will obviously be a matter of fine judgment. A bad decision almost certainly will be reflected in the physical figures of the oil as well as in its odour and flavour. It is essential, of course, that distillation capacity should be adequate in terms of both quantity and time. This is a very concentrated operation requiring a considerable expertise.

Apart from the U.S.A., we are equally at home in the Italian fields where there is not quite the same sense of urgency and the scale of the operation is nothing like so vast. We have been directly involved with the establishment of the peppermint crop in large areas of the upper Po valley, near Turin. In these

areas the movement skills do not really compare with their American counterpart. The Italian herb is much less leafy than the American. It is hand-loaded unchopped into fixed stills of about 250-kilo capacity, whereas the American charge is about 4000 kilos of chopped material or "hay" as the Americans call it. The American still is, in fact, the truck complete with steam coil which collects its own charge from the chopping machine in the field. Back at base the canopy or distilling head is lowered onto the truck, bolted down and the steam pipe connected so that the distillation operation can proceed, as it were, in situ. In just under two hours the truck can be disconnected and the spent material taken out and tipped for spreading later on to the fields where it is then ploughed in as manure.

Having assured ourselves of supplies of high quality oils by prime distillation, we can approach with complete confidence any further processing such as rectification. This process is sometimes referred to as if it were an expedient for turning a bad oil into a good one, but this is just not the case. Rectifying, in effect, concentrates an oil and, in so doing, often concentrates any abnormalities - it does not remove them. Rectification can be performed by varying degrees according to the purpose for which the oil is intended. We normally have two standards for peppermint oil and these can be described as single rectified or double rectified. The single rectified oil is virtually a redistillation which rejects the last 2 - 4% of the oil according to its nature. residues in the still are usually resinous and there is always a danger of taking out, unwittingly, the natural antioxidants of the oil. Double rectification rejects the first fractions of the redistillation up to whatever percentage is deemed necessary - perhaps 5% or even up to 10% - according to the oil and the purpose for which it is being prepared. This is in addition to the rejected residues at which we call the bottom end. Single rectification , therefore, remove this bottom end. Double rectification cuts something off both ends.

The rectified oils have a variety of uses in flavourings such as chocolate centres and toothpastes. The double rectified oil is much more satisfactory where solubility is a prime factor as, for example, in the manufacture of liqueurs. Whatever the purpose, the operation requires the right equipment and the right skill and experience to preserve the natural balance of the oils with the minimum of damage to their thermo-labile components.

The <u>Arvensis</u> mint oils referred to earlier are also always improved by rectification. The bite and characteristic harsh flavour appear to reside in the terpene fraction and also, to some extent, in the resinous residue and the double rectification of this oil smooths its aromatic character very considerably. This double rectified oil blends reasonably well with peppermint oil but percentages as low as 10% can readily be detected by sensory means.

Prices should, of course, be consistent with the composition of the blends, but they seldom are. Of the Chinese, Brazilian, Formosan and Japanese oils the last named responds best to rectification.

The spearmint oils are also much improved by this redistillation to the double stage. The terpenes have a strong muddy character which seems to imprison the true flavour of the oil. It may well be that this explains the strange paradox of spearmint as a general flavour. As I am sure you are all very well aware, Spearmint is a very popular flavour right across the world, but only in two main products - chewing gum and toothpaste. It could well be that this rather unpleasant feature of the natural oil is absorbed or perhaps neutralised by the character of the chicle gum, thus effectively filtering off the flavour and, similarly, this could occur in the toothpaste base. Rectified spearmint oil certainly has a much more attractive flavour and it is better still when it is rendered terpeneless.

## Citrus Oils

This important and well-known group of oils includes orange, lemon, lime, mandarin (usually called tangerine if from the U.S.A.) and grapefruit, as well as several other oils used primarily in perfumery. Only the first three of these are really of significant commercial importance. As time is limited, therefore, let us confine our attention to lemon oil as representative of the group.

Varieties of the lemon, <u>Citrus limonum</u>, like the orange are very widely distributed but only Sicily, Italy and California are important producers. In Europe, lemon cultivation is concentrated in Sicily and Calabria - the former being by far the most important area. Much of the industry is of a primitive nature with small fruit growers selling the fruit to local packers and oil producers. In the past, much oil was expressed by hand or by simple machines but this practice no longer operates to any extent. Generally, the first grade fruit is sold as such and the smaller and imperfect fruit used for oil production. Today well over 90% of lemon oil is machine-pressed in what is called a "sfumatrici" machine which handles both the pulp and the expressed oil.

As one would expect, the quality of this oil varies widely with climate, the season of the year at which the fruit is gathered, the region in which the lemons are grown and, not least of all, the method and care with which they are processed.

American lemon oil production is confined almost exclusively to California but there is some small production carried out in Arizona. This is because the Florida climate, although good for oranges, is too humid and is, therefore, unsuitable for lemon cultivation. The method of processing is somewhat different and, as one would expect, is on a rather larger scale than that in Italy. After pressing, the partially exhausted skins are

often subjected to steam distillation in order to achieve the maximum yield of oil - howbeit of a quite different quality. Although the analytical figures for Californian lemon oil may only differ a little from those of the Sicilian oil, they are usually somewhat lower, the aromatic quality is very much inferior to that of Italian lemon oil and the oil so far produced in Arizona is even worse.

Our prime concern is, therefore, in the superior quality Sicilian oil. It has long been our practice to maintain a very close personal relationship with the main producers and visits are made each year, usually in January and February, in order to assess the crop and the available oil on the spot. It is at this time of the year that one gets the highest grade of oil, usually called "winter oil". As with all our other raw materials only those oils which comply with our tight purchasing specifications are accepted.

The principal flavouring component of lemon oil is 4 to 5% of citral, the main bulk of the oil consisting of terpenes, primarily d-limonene.

#### THE TERPENELESS OILS

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This matter of terpenes and deterpination deserves our close attention and now is the time, I think, to consider some of the aspects of this much debated subject. On the whole, there is a tendency to generalise and regard terpenes as troublesome and things better to be got rid of. This is not altogether true. As we have already seen, without them chemistry would not be quite the same subject and essential oils would be very scarce indeed. To take all the terpenes out of pepper oil, for instance, would leave almost an empty bottle and only a vague sort of odour and yet it is a very fine and expensive oil. On the other hand, some oils have a relatively low percentage of terpenes with a correspondingly high percentage of oxygenated derivatives.

Good examples of this are citronella, lemongrass, caraway and eucalyptus oils. The terpenes are really the corner posts of essential oil chemistry and their versatility is really amazing and unlimited. I have already indicated that, with oxygen, they give rise to a tremendous variety of alcohols, aldehydes, ketones, esters, ethers and phenols which, with their isomers, present an infinite range of odours and flavours. Chemically, the terpenes themselves are very complex. In the living plant, and it is necessary to stress the word 'living', they have a particular affinity for ogygen which is both remarkable and far-reaching, giving rise to this wide range of oxygenated compounds to which we have already referred. These are the components which are primarily responsible for the characteristic odour and flavour of any essential oil. Once the plant is cut and so killed, the whole biochemical balance is upset and this applies equally to any essential oils which may be present in the plant cells. Under these changed conditions oxygen becomes an enemy, not a friend, and its reaction with terpenoid bodies gives rise to unwanted offodours. The removal of the terpenes, therefore, from an essential oil is one way in which we can protect the valuable flavouring components.

The terms 'terpeneless' and 'sesquiterpeneless' need some explanation. If in an essential oil the terpenes (and usually the residue) are removed then the oil is designated as 'terpene free' or as a 'terpeneless oil'. It still, however, contains the sesquiterpenes and the flavour constituents. If now by further processing the sesquiterpenes are removed as well as the terpenes, leaving only the oxygenated flavouring materials, the oil is said to be 'sesquiterpenes'. In some cases where the sesquiterpene content of the oil is negligible the two terms have come to be used synonymously.

The process of deterpination is very necessary in order to get the benefit of the oxygenated aromatic bodies, particularly

when use is being made of these oils in the flavouring of beverages and jellies and other products requiring soluble essences. It must, however, be done by skilled people using proved equipment. This is an important part of the door closing mechanism. Many of the virtues of terpeneless oils are lost and perhaps forgotten because of indifferent technical manipulation. Oils such as ginger, nutmeg, coriander and juniper have a character, when deterpinated, that cannot be simulated by synthetics. Coriander, we can say, is linaloöl but linaloöl is most certainly not coriander. Likewise, lemon is primarily citral but, again, citral is far removed from lemon. I would rather like to illustrate the difference between these various oils and you have in front of you certain bottles which I would now like you to examine.

#### CONCENTRATIONS

I have said that the removal of terpenes results in a concentration of the flavouring components in the oil. subject of the level of concentration of processed oils is one which can lead to a great deal of controversy. Technical facts and commercial figures appear so often to be greatly at variance. Sometimes the concentration is calculated from the ratio of the principle constituent in the concentrated oil to that in the whole oil. For instance, with terpeneless lemon oil, the citral content of the concentrated oil may be quoted as 40 - 45% while that of the natural oil varies between 4.0 and 4.5% - thus indicating a concentration of tenfold. Similarly, with terpeneless bergamot oil the ratio of linalyl acetate in the concentrated oil can be compared with that in the original oil. It is very obvious that this method of selecting just one component completely fails to take into consideration the several other soluble components of the oil - components which, in fact, may have far more importance in the overall flavour effect than the compound selected. The concentration can really effectively be calculated from the actual yield of terpeneless or sesquiterpeneless oil obtained from a known weight of the whole oil. Lemon oil, for

example, will deterpinate to a 16 X concentration and the cost will, therefore, be 16 times the cost of the original oil plus, of course, the cost of the operation. Terpeneless orange and lime oils also conform to much the same basic calculation. The best way of evaluating the concentration is for the user of the oil to make comparative trials using both the whole oil and the concentrated oil side by side. In the case of the terpeneless lemon oil, for example, if an essence is made and some mineral water syrup flavoured with it, the quantity necessary to give the same flavour strength as the ordinary whole oil of lemon can readily be determined by a series of trials.

This highlights once more the absolute necessity of a clear understanding between buyer and seller of the precise product under discussion and the full range that that product may cover. Terpeneless lemon oil, for example, is at one end of a range - synthetic citral is at the other end. We produce both and from them we can make anything inbetween. However, whatever the needs may be, deterpination is one of our prime disciplines and, when it is related to our English distilled oils, we like to think we can talk with real authority on this subject.

#### CLASSIFICATION OF ESSENTIAL OILS

Generally, when considering a group of products such as the essential oils, it is a considerable help to our understanding to be able to present them in some form of classified system. There are very many ways of grouping together the mass of essential oils but few have any valid reality unless based on a purely subjective assessment of their aroma and flavour effect. Botanically, one might assume some relationship of aromatic effect and, if we compare the oils obtained from ginger and turmeric, or again, between rosemary, lavender and sage, one can appreciate some similarities. On the other hand, such closely related botanical materials as parseley coriander and

aniseed clearly indicate the impracticability of such a botanical basis of classification. Such a grouping would also fail to recognise certain other well-known 'pairs' such as anise oil from true aniseed and that from the quite unrelated plant China Star aniseed, or again, the oils obtained from clove leaves and cinnamon leaves.

It is, of course, appreciated that essential oils are primarily characterised by the balance of their chemical constituents and one can utilize this to give a fair comparison of their effects. In many cases this is so but, in others, the differences are too marked to be of any real value. For instance, there is very little similarity between clove bud oil containing, as it does, 95% of the phenol, eugenol, and clove leaf oil containing 82 - 88% and, again clove stem oil containing up to 96% of this same phenol. Here the other related chemicals play a much more significant part in the total odour balance and this gives to each oil its quite characteristic note.

Unfortunately, therefore, I cannot really present to you a simple classification and one must, of necessity, fall back upon personal experience as a guide to the nature of the oils one is handling.

# THE USES OF ESSENTIAL OILS

Essential oils have an amazingly wide and varied application in many industries for the perfuming and flavouring of a multitude of many finished products as well as covering objectionably odours encountered in certain processes. Apart from the acknowledged value of some of them as powerful external and internal antiseptics, others have marked physiological activities of approved worth in medicinal practice. The spices, the value of which is largely due to their essential oil content, have been used in the seasoning of food since time immemorial and their wide use in perfumes, cosmetics and other toiletry items

hardly needs mentioning. There is barely an industry which does not make some use of essentials in some aspect of its production activities.

The advantages which the essential oils display in any particular industry are best discussed within a framework of the knowledge of what they are required to do and therefore any generalisation on this subject is likely to be of very little value.

The production of essential oils is a highly specialised field and, in order to ensure a consistently high quality, the chemists involved in their production and control require a sound analytical understanding, a great deal of ingenuity and a carefully trained sense of taste and smell. They must always be on the alert, not only for known adulterants and impurities, but also for 'the latest wangle'. The whole of this must be closely allied to an understanding of your problem, how best the many essential oil products can be applied and the effects that any variation may have in your end-products. For this reason, the greatest advantage to any user can only be achieved by individual technical discussion during which the parameters of the problem can be quite clearly defined and the best solution evolved as the result of our long experience of these very effective materials.

To summarize then, perhaps we can take a look at the general picture of essential oil operations. Our literature gives you a clear indication of the extent of our products and I hope that I have been able to review the processes we employ and some of the pitfalls which we have to face. In many ways it is something of a paradox, with a soundly based scientific knowledge allied to a high degree of skill, often trying to support low merit or even bad value demanded by the market place. All the latest instrumental techniques with which we are completely familiar, it seems, cannot deny the merits of a cheaper price.

On the other hand, the human ability to discern and appreciate the satisfying features of any particular taste and smell still, fortunately, sets the standards for success in the flavouring of our foods and beverages. This, obviously, has something to do with that mystic word 'quality' which, to define, I regret to say not only defeats me but also the pundits - to jedge by much of the literature on this subject.

In conclusion, I would merely like to draw your attention to the range of English distilled oils, terpeneless oils and concentrated oils which we have laid on for your examination. If, as a result of having a look at any of these, you have any specific problems involving their usage then, of course, we will be only too happy to help you resolve them. As you will realise I have concentrated on our production facilities and problems; the application of these versatile flavouring materials requires separate treatment.

