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SECOND UNIDO WORKSHOP ON THE ESSENTIAL OIL INDUSTRY (WESOIN - 2)

PROCEEDINGS

Organized jointly by
the United Nations Industrial Development Organization (UNIDO)
and the Government of the Philippines with the collaboration
of the Philippine Council on Industry and Energy
Research and Development, Department of Science
and Technology (PCIERD/DOST)

Manila, Philippines
4-8 February 1991

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I. INTRODUCTION

1.1 The first UNIDO Workshop on the Essential Oils Industry was sponsored by the government of India (Central Institute of Medicinal and Aromatic Plants) and the Economic and Social Commission for Asia and the Pacific (ESCAP) in 1981 at Lucknow, India. Several objectives were set for the succeeding workshops on essential oils. The first one was to hold more workshops and seminars in order to discuss and exchange information on the essential oils industry. The second one was to publish a manual on the essential oils industry to assist entrepreneurs and technologists in developing countries. The second objective has been accomplished with the publication of a Manual* on the Essential Oils Industry which was a collective effort of scientists in developing countries who had acquired expertise in different aspects of the industry.

1.2 The first objective was fulfilled with the holding of the second Workshop in Manila, Philippines on February 4-8, 1991. Copies of the first Manual were made available to all participants of the Workshop. The second Workshop was held with the following objectives:

1) To review the progress made in essential oils technology, marketing and export within the developing countries.
2) To review the progress made in the implementation of the recommendations made during the first UNIDO workshop.
3) To serve as a forum for the exchange of experiences between researchers and technologists involved in the essential oils industry.
4) To serve as a means of planning and initiating programmes of technical assistance and cooperation among developing countries in the essential oils industry.

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* Practical Manual on the Essential Oils Industry edited by R.O.B Wijesekera and produced by the Thailand Institute of Scientific and Technological Research (TISTR) with collaboration from CIMAP, Lucknow, India for UNIDO, Vienna, Austria (19--)

The offer of the Government of the Philippines to host the Second Workshop was considered favourably upon the endorsement of a UNIDO Technical Expert, Dr. R.O.B. Wijesekera, who visited the Philippines in May, 1990. The contribution of the Government of the Philippines was made through the Department of Science and Technology and its two agencies:

- The Philippine Council on Industry and Energy Research and Development; and
- The Institution responsible for the organization of the Workshop sessions and formal activities: the Industrial Technology Development Institute (ITDI) at Metro Manila.

All the experts were invited by UNIDO while the participants were selected by the host country with the final approval of UNIDO. The Phillipines hosted all the participants on a tour of an aromatic plants culitvation and an extractor in Laguna with side trip to a historical place and soap-making cottage industry utilizing natural essential oils.

A list of participants, experts and invited countries is in Annex A.

KEYNOTE ADDRESS

Dr. EDUARDO R. MAGTOTO
Undersecretary
Department of Science and Technology
Manila, Philippines

On behalf of Secretary Ceferino L. Follosco of the Department of Science and Technology, I am extending our country's warm welcome to the participants of this 2nd UNIDO Workshop on the Essential Oil Industry. While this workshop was planned a long time ago, it is a tribute to your interest and dedication to Essential Oils to be able to come to this workshop despite the recent inconveniences of international travel.

My congratulations go to UNIDO, specially to Dr. Wijesekera and to ITDI, specially Dr. Lirag and Dr. Luis, for taking the lead in organizing this most important workshop which would enable scientists and technologists in the Essential Oil Industry to exchange technical information. As in all fields of technical endeavours, such exchange of views and technical information is very vital to the progress of the industry.

I have noted that this workshop covers various aspects of the industry that are most important to developing countries' needs, namely agriculture, processing, compounding and equipment fabrication.

In the field of agriculture, areas of concern normally include cultivation practices and disease and pest control. In this age of biotechnology, one cannot ignore the potentials of selection and development of high yielding plant varieties of top quality oil. Development of appropriate agriculture equipment and machinery, however, simple they may be, is also needed to increase productivity of agricultural labour.

While traditional processing methods may continue to produce satisfactory essential oil, new practices in processing may result in higher productivity or higher quality products. Obviously, while one should not arbitrarily change processing techniques, as it may affect product characteristics, steps must be taken to keep abreast of new developments.

The field of product formulation is an area where developing countries have been left behind. I must therefore congratulate UNIDO for taking the initiative in assisting developing countries in this area. Success in this endeavour will give the developing countries added value to the raw materials they produce. In addition, it is also expected to increase the market for essential oils since additional products could be more quickly developed to suit specific local market requirements.

To be competitive, Essential Oil processors need to have suppliers of equipment appropriate to local conditions. Normally, this is best done through local fabricators. The experiences that you would be sharing with each other will reduce the time and money otherwise spent in equipment development.

In summary, in the next few days, we look forward to a week of profitable exchange of ideas which would lead to the high yielding/high quality agricultural production, processed by appropriate locally fabricated equipment into high yielding/high quality oils, a substantial fraction of which is to be locally compounded with other materials.

During the week, you can rely on our ITDI and PCIERD staff to assist you to make this Workshop a pleasant success.

Agro-technology of Essential Oil-Bearing Plants
(An Indian experience)

Dr. Baldev Gulati

In the present context of the flavour and fragrance industry in the world, while the standards for natural essential oils are demanding, the prices are competitive. This necessitates optimum production of the bio-mass and the oils per unit area from cultivation which can only be achieved by adopting proper cultivation practices. The problem becomes all the more serious especially when suitable, if not ideal, agro-climatic conditions for growing aromatic plants are not available in the countries where these are introduced for large scale cultivation. However, by using proper technique of cultivation it becomes possible to grow successfully aromatic plants in various countries or even in different places within a country. Some useful information on the cultivation of aromatic plants is given in this presentation with particular reference to India where some aromatic plants have been introduced on large scale and where a great deal of work has been done in various Research Institutes located at different agro - climatic zones.

Essential oils - bearing plants, as a matter of rule, do not come up well in water - logged areas; exceptions are few, such as Acorous calamus which grows in marshy areas or water logged areas. In general soils suitable for maize and/or vegetables are considered good for cultivating aromatic plants. (In India, maize is raised during monsoon and any place where water stagnation occurs, does not permit growth of maize). In our work on Ocimum sanctum, it was observed that water - logged area gave only 4 harvests while other areas gave as many as 9 harvests over a period of 2 years; production of oil was much higher in the latter case.

Soil pH is another crucial factor for not only yield of oil but also its quality. In general, soil pH around 7 is suitable for most of the crops. In case of citronella (Java type) it was observed that, in soils having pH above 7 while oil content in the leaves was normal, content of citronellal (a vital component of the oil) was depressed and was always around 30 percent as against 35 percent for Standard quality oil. This oil when produced in Meghalaya and Assam (North Eastern India) and in Karnatka (South India) was always of standard quality. The soil pH in these areas was around 6 and occasionally lower. One of the crops, which can tolerate very high pH is

Matricaria chamomilla. It was found to be a successful crop in Usar (Saline alkali soils) in the Gangetic plane in Uttar Pradesh where hardly any other crop can be grown successfully.

Next to soil quality, altitude and latitude are other important factors for successful growing of aromatic crops. However, in certain cases, it is possible to grow successfully aromatic plants under different altitude by adopting suitable modifications in the cultivation practices. In this category Mentha group of plants and geranium were tried with promising results in India.

In Mentha group of essential oils, the following four species are important:

- Mentha arvensis subsp. haplocalyx var. piperescens. Holmes (Japanese mint)
- Mentha piperita Linn (Peppermint)
- Mentha spicata Linn (Spearmint)
- Mentha citrata (Bergamot mint)

Japanese mint cultivation has developed considerably in India. During large scale cultivation in 1963 in the Terai area of Uttar Pradesh (having fertile irrigated soils, large holdings of farmers), it was observed that growing stage of the crop passed through hot season while harvesting stage coincided with the monsoon. Both these factors were detrimental for growing this crop. For proper growth, growing season should have rains while sunshine during harvesting. Agro-technology for Japanese mint had to be worked out afresh. By adhering to correct planting time i.e. before 15 January, it was found possible to harvest the crop at least one month prior to the onset of monsoon. Due to great deal of work done on the agronomy of this crop, the area has now become the most important for cultivating Japanese mint, average annual production of oil is about 1000 tonnes involving an overall area of 10,000 to 15,000 ha.

As regards altitude example of Mentha piperita, M. spicata and M. citrata are more relevant. These species of mentha are long - day plants which condition prevails at higher altitude. Under these conditions the plants flower profusely indicating the proper stage of maturity for harvesting giving an oil with requisite odour and having standard physico - chemical characteristics. These three species when grown in the plains were observed not to acquire bushy growth and with very scanty flowering. It was, therefore, not possible to judge the correct stage of harvesting. However, as there is paucity of cultivable land at higher altitude in India there was no alternative but to grow these species at lower altitude. Work was done on the agronomy with special reference to correct stage of maturity in the absence of flowering. It was found that after about 120 days of planting, Mentha piperita and M. spicata gave oil in proper yield meeting the standard specifications. In case of M.piperita the oil was sometimes slightly below standard in respect of menthol content and a little higher in menthone content. The oils of both peppermint and spearmint are now produced regularly, up to 100 tonnes per annum, and used in the local industry.

Case of M.citrata was slightly different not only in its lack of flowering at maturity stage but its death during monsoon. In this case it was observed that if it is planted during January - February and harvested in April - May, cultivation was economical and quality of oil acceptable to the industry. The crop was not maintained for second harvest. This oil is now being produced up to 20 tonnes per year.

Geranium (Pelargonium graveolens) is yet another example. It is now possible to grow this crop in areas previously considered unsuitable. Geranium was previously cultivated in Shevroy Hills, Yercaud, (South India). Later on, some of the cultivators shifted to Octacamund (Altitude 2000 - 2500m above m.s.l.) an important area for cultivating Eucalyptus globulus eucalyptus oil (medicinal). The area has limited potential for growing geranium; the plants also suffered due to disease problems.

Two types of trials were undertaken to grow this crop in areas much different than the present agro - climatic zones.

i) It was observed that geranium (which is perennial and lasts for 5-6 years) can be grown successfully with economic yield of good quality oil at an altitude of 200 - 300m. above m.s.l. and having severe summers (temp. up to 40°C during May - June), provided it is grown for one harvest only. The plants die during hot summers and monsoon. This work, has not, however, resulted in commercial cultivation of the crop primarily due to the fact that planting material required (rooted plants from cuttings) could not be organised regularly. Geranium has now been introduced in

Hyderabad (Deccan plateau, Central India). The area does not have severe summers, there is no excess soil moisture which is detrimental to plant growth. So far, results have been encouraging; geranium can be grown as a perennial, planting material can be produced regularly, oil yield and quality is good.

Above examples clearly show that in certain cases, it is possible to grow successfully aromatic plants even though ideal agro-climatic conditions are not available.

Another set of plants grow in higher altitudes in cooler conditions (temp. not above 15 - 20°C during summer season) as summer crops. Such plants can also be grown at different and lower altitudes successfully as winter annuals. Examples quoted are: Tagetes glandulifera and Matricaria chamomilla.

We have observed that Tagetes glandulifera which occurs naturally in higher altitudes of Himachal Pradesh, Jammu and Kashmir (2500 m. and above) starts growing after April after the winter snow disappears, flowers in August and is ready for harvesting and distillation. This plant comes up very well as a cultivated crop during winter season in the lower altitude areas (free from snow fall; minimum temp. 2°C to 4°C during peak winter season). Large scale cultivation gave economic yields of the crop and an oil of acceptable quality. Oil was also accepted in markets in Europe and was exported in fairly large quantities.

Matricaria chamomilla which also grows best in areas as in the case of Tagetes as a summer crop in higher altitudes can be grown in the plains (lower altitude) as a winter annual. Growth of crop cultivated here was good with economic yield of the flowers and oil content and quality. Another big advantage of growing chamomilla at lower altitudes is that this plant comes up very well in Usar soils i.e. saline - alkali soils with pH of 8.0 and above.

Constraints of availability of land:

In most of the developing countries availability of fertile, irrigated land is a problem as most of it is used for the production of food crops. Aromatic plants, for optimum economic yields (returns) also need such land.

Growing of aromatic plants on marginal lands and rainfed areas is possible but the yields or economic returns are not the best. Some examples of use of these areas in India are the following:

A variety of Ocimum basilicum giving an oil rich in methyl chavicol plus a good percentage of linalool developed for large scale cultivation was found to fit well after harvesting potatoes (Sept. - October) or wheat (December). This pattern provided an additional crop and income to the farmers. About 50 tons of this methyl chavicol rich oil is being produced in India which is a useful starting material for producing anethole, anisic acid and aldehyde.

Similarly, Japanese mint can be cultivated as one harvest crop in between newly planted sugar cane. This aromatic plant can also be grown as:

Mentha (January planting) with stolons - Mentha (April May planting by cuttings/young seedlings) followed by wheat - Mentha (after wheat harvesting mentha seedlings come up throughout the field) and wheat.

Such combination of food crops with aromatic plants is a promising area but has to be worked out for each country having different agroclimatic zones, and cropping patterns.

For growing aromatic plants in hilly terrain, example of Meghalaya (North Eastern India) is worth mentioning. Citronella cultivated on hillocks was worked out as under.

- Top of the hill intact with natural forestry growth.
- Middle portion used for aromatic plants.
- Lower part used for planting citrus plants.

In this way there was no environmental degradation of forests, very little soil erosion and reasonably good crop of citronella with good harvest of citrus fruits.

Planting Material:

Among various factors, selection of correct and authentic planting material is one of the most important and crucial factors for successful cultivation and production of standard quality essential oils. While stem cuttings, rooted slips and stolens give plants true to the original, plants raised from seeds can give rise to plantations which are heterogenous. This problem is common in the case of Ocimum species, and palmarosa. In the case of later, as many as 20 different plants were observed. *Ocimum basilicum* also gives rise to plants which are morphologically different. However, it is necessary to use seeds from correct species.

It was also observed that deterioration in aromatic plant species occurs when cultivation is carried on for a long time. Japanese mint introduced in India in 1953 and brought under large scale cultivation in 1963, gave an oil rich in menthol. However, in late seventies and early eighties it was found to yield oil which was so low in menthol that its isolation by chilling was not possible. Content of methone increased to a very high level. Work on improvement of Japanese mint was undertaken. Improved material was supplied to growers of this crop which has again resulted in an oil high in menthol content. Continuous improvement in the cultivated species, therefore, becomes necessary to maintain level of standard quality oil production.

Preparation of Nursery:

Raising of planting material in the nursery needs proper planning; seedling at the time of transplanting should neither be too small and tender (to withstand transplanting) nor too big with over - developed root system. Nursery is raised either on flat, raised beds or in polythene bags where planting material is to be transported over long distance or in cases where exposure or damage to the root system (which sometimes goes deep into the soil) can occur. Particular cases are that of *Eucalyptus citriodora*, other eucalyptus species and geranium.

Depending on the species, it becomes necessary to raise nursery using different types of material such as:

Seeds:	for plants such as Ocimum Species, Martricaria, Salvia spp., Palmarosa, Citronella (Sri Lanka), Lemongrass (East Indian; *Cymbopogon flexuosus*), Eucalyptus spp. *Artemisea pallens* (Davana) *Melissa officinalis*, Lavender.
Cuttings:	Patchouli, Geranium, Japanese mint (for transplanting out of season if required) *Bursera delpechiana* (Linaloe), Jasmine.
Bulbs:	Tuberose

Stolons: Mentha group of plant species.

Rooted Slips: Citronella (Java type), Vetiver, Palmarosa (under certain circumstances), Lemongrass (Cymbopogon citratus - West Indian Lemongrass).

As mentioned earlier, planning of nursery needs careful attention as the seedlings in the nursery may have to remain there for periods ranging for a few weeks to few months, examples are Ocimum Species (few weeks) and lemongrass (East Indian tupe - few months).

Availability of seedlings have to coincide with the transplanting season which in most of the cases is more or less specific.

It is not possible to describe nursery practices for various species of aromatic plants. Nursery practice for Artemisea pallens (Davana) is described as a special and specific example:

Seeds of davana are very small; sowing in nursery poses problems with respect to depth of sowing and equal distribution. Small size beds are made evenly with fine pulverised soil. The beds are filled with water and seed sprinkled. The seeds settle down as the water seeps into the soil with the germinating tip facing upwards. Fine ash or soil is sprinkled over the bed thus covering the seeds evenly. Germination occurs uniformly over the entire bed. Seedlings are tended properly till ready for transplanting.

Transplanting and Planting

Preparation of field by removing all the dead and dried debris and material from the previous crop is important. Land preparation and lay out, especially with respect to irrigation need attention, consideration for easy inter-culture for cultivation, manual by or mechanical means, is to be given prior to the land preparation.

Proper spacing and application of basal dose of fertilizers contribute to the success of the crop. While spacing can be decided prior to planting with seedlings, in case of direct sowing by seeds, needs thinning of plants after germination and when plants attain sufficient height to avoid over crowding which would affect the eventual growth of the plants. In over crowded fields, the plants tend to grow straight without acquiring bushy and fertilizers encourage the growth of weeds. Weeding is done either manually or by using weedicides. Dose and efficacy of the weedicides, however, depends on the intensity and the nature of weeds.

However, once the main crop attains sufficient growth (especially Cymbopogon spp) the interspace is covered which does not allow growth of most of the weeds.

Harvesting and Post Harvest Treatment of the Crop:

For most of the crops, stage of harvesting is known such as in Mentha spp: flowering stage; for Ocimum spp: full flowering stage but prior to seed setting.

In many cases, the harvested crops such as Japanese mint, and other species of Mentha and Cymbopogon species need to be field-dried for short period to get rid of excess moisture. On the other hand, flowers are to be processed without delay. Therefore, harvesting schedule is also linked with the processing which varies from crop to crop.

In general aromatic plants can be broadly categorized into two groups:

a) Aromatic crops which need processing soon after harvesting ranging from a few hours to few days. Crops are also wilted to some extent prior to processing such as Mentha species, Cymbopogon spp: Ocimums; flowers are required to be processed without delay.

b) Crops that can be kept for sometime prior to processing:
Vetiver, Artemisea pallens (davana), Iris, patchouli, Umbellifera (seeds) except Anethum graveolens for herb oil, Acorous calamus most of the spices i.e. pepper, nutmeg, cardamom, clove (stem and bud). Vetiver cannot be kept for a very long time but can stay for one to two weeks.

In case of patchouli, oil quality is reported to improve with the age of the harvested leaves. Iris rhizomes are required to be stored for 3 years before odour of the material develops.

It may, however, be mentioned that the material which to be kept after harvesting have to be dried and stored properly otherwise deterioration of the material sets in.

Processing:

A great deal of work has been done on the proper processing of practically all the essential oil - bearing crops in the world. While general procedure of producing oil by steam or water distillation can be applied, some of the materials need specific procedure for producing good quality oil. Some typical examples are:

- Citronella (Java type) should not be distilled till the end to produce an oil having requisite percentage content of citronella.
- Japanese mint has to be distilled till the end for better menthol content and odour value of the oil.

It is always advisable to consult literature for processing information when work is started in a country as, improper processing can lead to production of sub-standard oil even if proper planting material is used and good cultural practices are adopted.

For the essential oils, lighter than water, distillation and resultant separation of oil does not pose much of a problem. However, in case where the oils have specific gravity nearly equal to water or more than that of water, distillation and complete recovery of the oils is not easy and complete. The problem is compounded further when the oil is rich in oxygenated compounds such as especially eugenol-rich oils, due to their solubility in water/distillate where temperature is also more. Oils having phenyl ethyl alcohol such as rose also pose a problem due to its solubility. In such case cohobation becomes essential which adds to the cost of the distillation unit.

In case of certain plant materials like costus root (Sassurea lappa), vetíver, cloves, more than one florentine receivers are used to effect better collection and separation of the oils. For effective separation of oil, distillate temperature in some cases is increased by increasing steam input and/or controlling the flow of condenser water.

Oil Content and Yield of Essential Oils:

Oil content data on aromatic plants is important as they give an idea not only on the yield potential of a crop but also economics of its production in respect of processing. The data are more useful if it is calculated on dry weight basis i.e. on moisture free basis, which permits comparison of oil content in the material harvested during different seasons and places, within a country and with other countries. Unfortunately, in many cases data on oil content published in literature are not indicative of the moisture content of the material distilled.

Yield of oil per unit area is not only linked with the oil content in the plants but also yield of the bio-mass. Both of these have a limitation, hence limit to the yield of oil which can be produced from a unit area. However, optimum yield of oil can be produced if the crop is grown keeping in view the part of the plant which can give maximum oil. As an example, palmarosa (Cymbopogon martinii var motia) is distilled after harvesting the entire over growing plant. Palmarosa plant comprises of: inflorescens, leaves and stalks. Oil content is maximum in inflorescens followed by leaves and stalks. As stalks contribute very little (negligible) amount of oil, the technique in raising palmarosa lies in the fact to get maximum quantity of leaves and inflorescens as the oil at that stage is maximum and of standard quality if harvested at the stage of flowering. Spacing and optimum use of fertilizers play an important role in growing a crop having less of stalks but more of leaves and flowers. In case of Mentha group of plants, leaves are the most important. If planted closely, the plant looses most of lower of leaves, due to overcrowding, resulting in a crop having more than desirable quantity of stalks (having negligible oil) thus giving not only less yield of oil per unit area but with higher cost of distillation.

Pest and Diseases:

Pest and diseases are a common occurrence in the essential oil bearing crops. Incidence of pests and diseases not only adversely affect the yield of crops but in some cases affect the quality of the oil also. In our work on Ocimum basilicum (French basil) we observed that due to incidence of Cercospora, increase of eugenol content to as much as 80 percent as against normal content of 5 to 10 percent in normal oil samples.

A great deal of published data is available on the control of pests and diseases in aromatic plants.

Crop Rotation:

In general, most of the essential oil bearing plants are soil-exhausting especially when grown over a period in the same fields. Depletion of both macro and micro nutrients can occur. Besides replenishing these during growth period, it is also necessary to adopt a suitable sequence of crop rotation. Crop rotation will vary from country to country and also from place to place within a country. Work done in India on this aspect may be relevant. As an example the following may be mentioned:

-Mentha - Mentha - Wheat - Paddy - Mentha

-Mentha - Paddy - Wheat - Mentha - Mentha

For crops of long duration like citronella and lemongrass, the fields are not planted again under these crops for 2 -3 years.

Proper crop rotation also helps in discouraging weed association. Paddy as a rotational crop suppresses weed growth to a great extent in the subsequent crop.

It may be seen from this write up that essential oil bearing crops have specific requirement of agro-climatic conditions for optimum yield. Such conditions are not always available. Nevertheless, most of these plants can come up well even under different agro-climatic conditions if Research & Development work is undertaken to work out parameters of cultivation. Development of large scale cultivation in India of some crops such as Mentha group, Geranium, Cymbopogon species, Ocimums, etc. is a clear instance. Adequate literature on cultivation practices of most of essential oil bearing plants is available which can give useful information to begin with.

Technological Variations in the Essential Oil Industry

Dr. R.O.B. Wijesekera

The essential oil industry as discussed here includes the production of essential oils, concretes, absolutes, resinoids and oleoresins, extracts produced by super-critical carbon dioxide, isolates, and aroma chemicals. All these products demand great variations in the technologies used. It is, therefore, the purpose here to discuss, albeit in very brief fashion, the main technologies that go to form this large and varied range of products. The discussions will bear special relevance to the requirements of developing countries.

The most important and widely used technique, and the most important component, therefore, of the technology, is the method of steam distillation. Steam distillation is essentially the release of the volatile constituents of a plant material through the agency of the passage of steam through it, and generating a mixture of essential oil and water vapour which can then be condensed, and the oil phase separated from the aqueous. The essential oil is therefore, obtained at relatively low temperatures, compared to the individual boiling point of the oil.

There are many variations of the general theme of steam distillation. A more detailed examination of the technique of the steam distillation would be in place particularly where it would be necessary to examine the factors that determine the construction of equipment used in the process of steam distillation. A general purpose distillation assembly (Fig. 1) consists of: a still body, a loading mechanism to load the vegetable material into the still, because one is dealing with about a thousand kilograms or more of plant material on an industrial scale; a steam source to deliver the steam at the required pressure and flow rate for its passage through the vegetable material in order to extract the essential oil; and a condenser or a heat exchanger in order to condense the mixture of oil and water vapours; and on oil separator to separate the oil from the water.

The steam can be generated within the still body itself or as in this instance, it can be provided by a satellite boiler. The advantage with an external steam source is that one could always have two or three still bodies attached to the steam source depending on the raw material availability.

The two main types of condensers or heat exchangers available are the shell-and-tube type, which is used in all modern distillation assemblies, or the coil-in-water type which was traditionally used and is still employed particularly in remote field stations. The oil separation is carried out using a florentine vessel or oil separator, and there are two types, one that could separate oils lighter than water and one that could separate oils heavier than water. But of greater utility is a combined type (fig. 2) which can be utilised both for oils heavier than water as well as oils lighter than water.

As stated before there have been a great many variations within this theme of steam distillation. Notable is an attempt to utilize an air-cooled condenser and the justification was that this was designed for areas in very remote villages, where during the heavy drought season water in large quantities as required by the usual type condenser was not available. (Fig.3). This is frequently the case in many south and southeastern countries in areas where such crops as citronella, lemongrass, palmarosa are cultivated. Whenever there are prolonged droughts, rivers and streams dry up and water is extremely limited. The principle is the use of a latent heat exchanger, a small vessel in which the water is allowed to boil and absorb the latent heat to partially cool and outcoming vapours. Then there is a pressure chamber which leads to the air condenser unit, which consists of a series of parallel tubes with metal fins or vanes attached to it in order to provide the cooling surface. Although this has been designed for a contingency situation, optimisation of this design can bring about the possibility that it could be used with comparable efficiency in conditions of water scarcity.

A further variant of this is a type of water condenser, a coil immersed in a large reservoir of water. The coil at the top is zig-zag and serves as a latent heat exchanger, and the expanded part of the coil at the bottom serves to complete the condensation (Fig.4). These have been tried out in various locations in Sri Lanka, and enjoyed encouraging degrees of success. These have mild steel bodies and aluminium is used to construct the condenser and is relatively very inexpensive.

Before one embarks on designing a distillery, it is important to work out certain parameters that are characteristic of the type of oil to be distilled. Firstly, one should work out an optimum way of packing the vegetable material into the still body. One must work out the optimum steam pressure, the best flow rate, and one must also determine the optimum rate of time for which the distillation should be carried out. In order to determine this, it is best to construct a simple experimental still and the easiest way of constructing this simple inexpensive still is to use clean oil drums (Figs. 5 and 6). The oil drums can be joined together by a flange, in order to get a still of twice the height and if one were to use a single oil drum to which is coupled two half drums which are joined by flanges, then one could vary the height of the vegetable material to 1.0 oil drum height, 1.5 x oil drum height or 2.0 x drum height. Accordingly one could make the still so that the height could be varied in several ways.

The compactness of packing is important because the rate at which the steam passes through the vegetable material could also determine the efficiency of the extraction process. Having constructed such a still body, certain tests have to be conducted in order to determine these parameters. For example, it is important to determine the optimum flow rate, or range of flow rates, within which one can expect the best yield of oil. Likewise the optimum load, and the tightness of packing of the vegetable material - to avoid "channelling of steam" - within the still body has a bearing on the yield of oil. It is important to determine when, that is after how long, the distillation process is over, in other words, when the distillate no longer contains any significantly measurable percentage of oil.

One must determine these parameters because they do vary from oil to oil. For example, if one takes an oil like eucalyptus, and one finds that the content of eucalyptol or 1,8 cineole is lower than one usually expects from this oil, that is compared to say, a predetermined composition from laboratory distillation, then one knows at once that the distillation has been too fast, or the condensation has not been good enough. Generally if the distillation rate is too fast, the condenser may not be able to handle it and provide adequate condensation. One can control the distillation rate so that the 1:8 cineole content would come within the expected rates. The cineole is a low boiling component of eucalyptus oil, and like the majority of such constituents distils specially fast, and there could be incomplete condensation. If on the other hand one distils an oil like peppermint oil and one finds that the monoterpene hydrocarbons which are the most volatile components and therefore then to come out first are low in content in comparison with an authentic sample, then one immediately knows as in the previous case that the distillation rate is too fast. In on the other hand one distils a sample of citronella oil, and finds that the higher boiling components that is the components that by virtue of their higher boiling should emerge later. are quantitatively lower, then one draws the conclusion that the distillation has not been carried out to full completion. So one has to go hand in hand with analytical control of the process of distillation. The composition of the distilled oil is not uniform throughout the distillation process. The first few millilitres of oil will contain a larger proportion of the lower boiling components, and the latter portions of the distillate will contain a larger proportion of the higher boiling components. It is the pooling of the entire distillate that gives the characteristic composition of any essential oil.

In order to determine the optimum size of a still and the best charge to load, one has to make certain tests and that is why the importance of the test stills cannot be over-emphasized. The following factors are known to effect the size of the charge.

1. the nature of the plant material.
2. the method of harvesting.
3. the particular chemical composition of the oil itself.
4. the method of distillation.
5. the problems, logistic problems, of handling and storage.

So the optimum size of any charge whether it be 1000 kg, or 5,000 kgs, has to be determined by examining all of these factors. In any distillation, continuity of operation is most important because it leads to the most economical methodology. The number of hours at which the still is used will determine the amount of oil that will be produced and therefore the income. It is logical to ensure that there is sufficient raw material to keep the still going for the maximum practical period of time.

Over the years, it has bee determined that the best options are the following:

A cylindrical still body with a grid at the bottom on which the plant material rests; a loading device which can be of several types. The " cartridge type" is the type of inner basket into which the material is preloaded. By means of a pulley and chain device or some other hoisting mechanism, the pre-loaded basket is inserted into the still body. The advantage of this type is that a series of them can be pre-prepared. They can be moved on rollers and the loading is facilitated. They can also facilitate the quick disposal of the spent plant marc after the distillation process is over. The basket type can also be replaced with a flange type, or a centrally secured multiple basket type (Fig. 7). The multiple type is where several flat flanged baskets are inserted along a central axial pole. The loading operation takes place sequentially; when a ceratin amount has been loaded, to the based flanged basket, the second flange is inserted and when this flange too is also loaded, another flange is inserted and so on. The flanged basket runs concentrically to the central pole which is attached to the bottom-most flange and then the whole can be lifted off with a crane and serves the same purpose as the basket type.

Some considerations, in the design of the commercial essential oils distillery may now be considered.

First of all, any distillery must have some kind of a steam source and this takes the form of a boiler which can deliver steam at about 1-3 bars pressure and the capacity of the boiler could be up to 500 kgs/hr steam. Then there must be a water-treatment capability. In some instances the water, and the continuous use can clog up the condenser tubes and the piping. It is more convenient and less expensive to have pretreatment of water of deionization rather than to continuously clean up the "caking" on the pipes due to inorganic salt formation.

The next consideration is the economical use of water. Water is used for the condensers, water is used for producing the steam and water is used for several other purposes. The economical use of water is most important because water has often to be transported from one site to another and this utilises energy. Then there must be safety requirements. Stills use steam and if there is any way in which steam pressure build up could occur, there is danger. Accordingly, the body of the still must be tested to counter such eventualities and not to cause catastrophic explosions. Steam can also cause severe burns. It is necessary to have a trained and certified boiler attendant to supervise the work in connection with the boiler.

Energy conservation is another important factor because expensive energy means higher pricing of a product. There is some merit in pre-heating of the boiler charge and this may be done by the use of solar heating, and by the use of water which is discharged from the heat-exchanger after the condensation; because the condenser water, once it is discharged has absorbed heat from the distillate and so it would considerably be warmer than ambient. It is also necessary to have the still body jacketed with some inert material to prevent heat losses. Another method of energy conservation is the use of inexpensive fuels, such as the exhausted plant marc, that is only if the exhausted plant marc does not find better use as a fertilizer. Or else the use of fuels such as are readily available in the vicinity of the distillery, like waste bagasse, discarded woods, coconut fibre waste, peat, and the like much be considered.

In the economic use of water, recycling cannot be overstressed and one of the ways in which not only the recycling of water, but the conservation of the traces of oil in the distillate, that is solubilized by the water can be conserved, is by a process called "cohobation". This means that the oil after separation from the water, leaves the water fully saturated with the oil. Although this is a very small percentage of the oil, in continued distillation it can be build up to a handy volume particularly when some major constituents are distinctly water soluble. eg. Eugenol in Clove oil and Cinnamon Leaf Oil. Therefore it is expedient to siphon this discharged water back into the distillation still be redistilled along with the new material. This process of cohobation must be used judiciously, because sometimes repetitive heating can cause chemical decomposition and may alter the quality of the oil so it has to be first tested as to whether it is practical and advantageous.

Considering the construction of a distillery, the flow diagram (Fig. 8) is a useful guide. It displays a system to have steam from a satellite boiler come into the still body, the exit from the still body into the condenser and then to the separator and the need for cooling water in a head tank that could be utilized for serving the condenser and the return of the water from the separator to a holding tank from whence it is pumped back to the head water tank.

The foregoing consideration dictate the basis of the construction of a distillery. In developing countries steam can be generated by a boiler that uses wood fuel or equivalent (Fig. 9). The steam produced in such a way is led into the distillation vessel to which is attached a multi-tubular condenser.

Modern field stills utilize the same principles and can be elegantly designed (Fig. 10,11). One (Fig. 10) is a field still, with an external boiler and the next (Fig. 11) is a more modern type of energy saving, electrically heated still. These basic stills are very costly items and therefore, in developing countries, there must be a very good reason for their acquisition and one of them could be that they could be locally copied and duplicated.

An interesting design which is quite common in many of the industrialised countries is what is called the box type of distillation unit (Figs. 12 and 13). Here the distillation unit and the still body is in the form of a box and it can be used with a mobile vehicle. It can be taken through the harvesting area where the harvesting is carried out by a mechanised harvester and this has been utilized in European countries in their expansive lavender fields. When the still body is full with the harvested lavender, it is brought alongside the distillation station which has a tube to which the still body is then attached. This connects it to the condenser system and the oil recovery vessels.

The field distillation technology has been given considerable attention as this is of the greatest importance to developing countries. The other variations and technologies in the essential oil industry also merit consideration at this stage.

First of all, essential oils have been produced as such, or can be utilised in the production of isolates that have concentrated fragrance or aroma chemicals from which, by synthetic manipulation, many aroma chemicals themselves could be prepared. For example, citronella oil can be separated into citronelol, or geraniol and these in turn could be converted by simple chemistry to the relatively expensive aroma constituents like hydroxy citronellal, or esters of cintronellol and geraniol.

This operation must be initiated by fractionating the oil; and for this purpose one has to use a fractionating column (Fig. 14). Fractionation is carried out under reduced pressure and vacuum. The column has a special packing to enable it to separate the constituents effectively. A column that has about 30-40 theoretical plates i.e about 5-7 theoretical plates to the meter, is an ideal column and can serve a 40-75 litre capacity distillation kettle. The distillates are collected in several vessels, and samples are monitored by means of GLC in order to identify the characteristics of the isolated fractions, and further purified if necessary by a second round of fractionation. Fractional distillation is therefore the next stage in the processing of Essential oils and the production of fragrance and for value added products for export. In developing countries therefore, it will be a profitable exercise to fractionate their essential oils for value-added products (Refer specialist contribution of Dr. W. S. Brud).

We may now very briefly consider the technology that is used for the extraction of resinoids or what are called in the perfumery trade as "fixatives". These are certain odour-preserving substances that tend to "fix" the fragrance when the perfume of fragrance material is formulated and compounded. They are generally relatively high boiling constituents and are known as those that retain the "bottom notes" of a fragrance. The resinoids can be resins from plant material like pine exudates or they can be extractives of mosses and lichens. The technology utilized for resinoid extraction generally (Fig. 15) consists of an extractor which is most effectively used with what is known as a "floating filter". Such a filter enables the filtration of the "micella" through it in continuous and convenient fashion. By the application of a vacuum the saturated micella is passed into a concentrator and the finished product led out of the bottom of the concentrator.

The next is the solvent extraction unit by which one could extract "concretes" from flowers such as roses, jasmine, ylang ylang and this can be a straightforward solvent extraction process. In a typical commercial scale assembly (Fig. 16) there are two extractors linked to two condensers. The heating is carried out by steam injection and the micella is led from both extractors through a control valve into the primary evaporator. Then from the primary evaporator, the major part of the solvent is recovered and passed into solvent storage tanks. The partially concentrated micella is removed from the bottom of the primary evaporator and pumped into the finishing evaporator from whence the final traces of solvents are removed and the concrete which is the finished product is led from the bottom. Such assemblies can very well be designed for the construction in developing countries where facilities for welding of stainless steel is available.

The production of concrete for local use is carried out in many institutions but often it becomes necessary to convert the concretes into "absolutes". The absolute is the alcohol soluble fraction of a concrete. It has to be soluble in cold alcohol because absolutes are generally the basis of the formulated colognes. The principal technology is that the concrete and the alcohol is led into a reactor vessel in which it has very briskly stirred. The waxes separate at the bottom of the reactor vessel on cooling. The alcohol is led through a rotary filter into the evaporator. The finishing evaporator and the primary evaporator affords a two-stage evaporation process as in the previous case where the major part of the solvent is removed in the primary evaporator and the rest of is in the finishing evaporator from which the absolute is led out on completion of the solvent removal (Fig. 17).

We may now consider the new technique of extraction with super-critical carbon dioxide.

These can then be known as extractives or fragrances and flavours produced by the extraction utilising the properties of super-critical carbon dioxide. The carbon dioxide under pressure is introduced into the extraction vessel and then by release of the pressure, it is led into an expansion chamber and then to a separation vessel which eventually leaves an extract of the flavour ingredients extractable in carbon dioxide, when the last traces of carbon dioxide is removed (Figs. 18 and 19). Then one has what is equivalent to a solvent extract conducted at very low temperatures. In the technology involved, the carbon dioxide is first compressed and then it is pumped into the extractor which has to be such that it can hold the raw material or semi-finished product with the carbon dioxide under pressure. It is constructed of heavy metal to withstand pressure. The extractor is connected to a separator vessel where by release of carbon dioxide, the extract collect in the separator. There are pros and cons in the utilization of the technique of extraction with super-critical fluids. Carbon dioxide - extracted essential oils were possibly conceived in a search for a complete replication of the natural flavour. CO_2 can be considered a totally natural solvent without toxic residuals. Accordingly a positive side is the reality that very low temperatures are used and the fragrance and flavours are conceivably more representative of the natural material. On the negative side, is the fact that the initial outlay is prohibitively expensive. It could be only utilized when large amounts of relatively inexpensive carbondioxide are available as a by product from another industry and the availability of the right type of raw material that lends itself to CO_2 extraction, and the need for products of the type which have sensitive components that constitute the extracted raw materials.

CO_2 extraction is used successfully in industry in the extraction of hops and ginger. Recent work by Moyler (1984) and also by the Chinese group of Chu Chin Chen indicate the potential of the technique. The liquid CO_2 selectively extracts the essential oil plus the lighter fractions of resins thus making the product something between an essential oil and a concrete.

The advantages of CO_2 extracts as against essential oils/solvent extracts include:

- Lack of solvent residues
- Lack of off-notes in the flower
- More top-notes due to extraction of highly volatile substances
- More back-notes - eg. gingerols and shogaols in ginger
- Better solubility characteristics.

GLC comparisons of oils produced by CO_2 extracts distillation are reputed to show all of these advantages. It is contended (Moyler 1988) that examples of ginger, clove bud, juniper berry, vanilla, hop, pepper, pimento and nutmeg have been advanced to establish the utility of this technique (Moyler and Health 1988). However, reservations till exist not only on grounds of cost of initial outlay, but in the fact that the post-harvest preparation of the raw material often involves conditions such as sun drying and curing where flavours are subject to relatively drastic conditions.

In a similar vein, one may consider the technique that is know as "hydro diffusion". This is a form of steam distillation where the steam is introduced from the top of the vessel. The distillate runs down through a "cooler" underneath the still body (Fig. 20). The technique lends itself to rapid distillation compared to the traditional methods and reportedly higher yields are claimed but the evidence is not yet convincing. The other advantage is ease of loading and unloading. The question of enhanced yields must be viewed in the light of the fact that these are often reported in the case of raw material that is high in fatty oils. The extent to which the fatty material comes down with the distillate could very well account for the marginally higher yields.

Other sophisticated extractors are available which have the same drawback of considerable expense in initial outlay. The Carousel Extractor (Fig. 21) is popular with industry where the cost can be afforded and is both elegant in principle and commendable in performance. But here we step outside the scope of this lecture. In any event, here too, the initial outlay is generally not within the consideration of developing countries.

In concluding, suffice it to add, that the technological diversity of the essential oil industry is considerable, and for developing countries there is a considerable scope and challenge for design innovation towards simplicity, economy and good performance, that is relevant to the case in point.

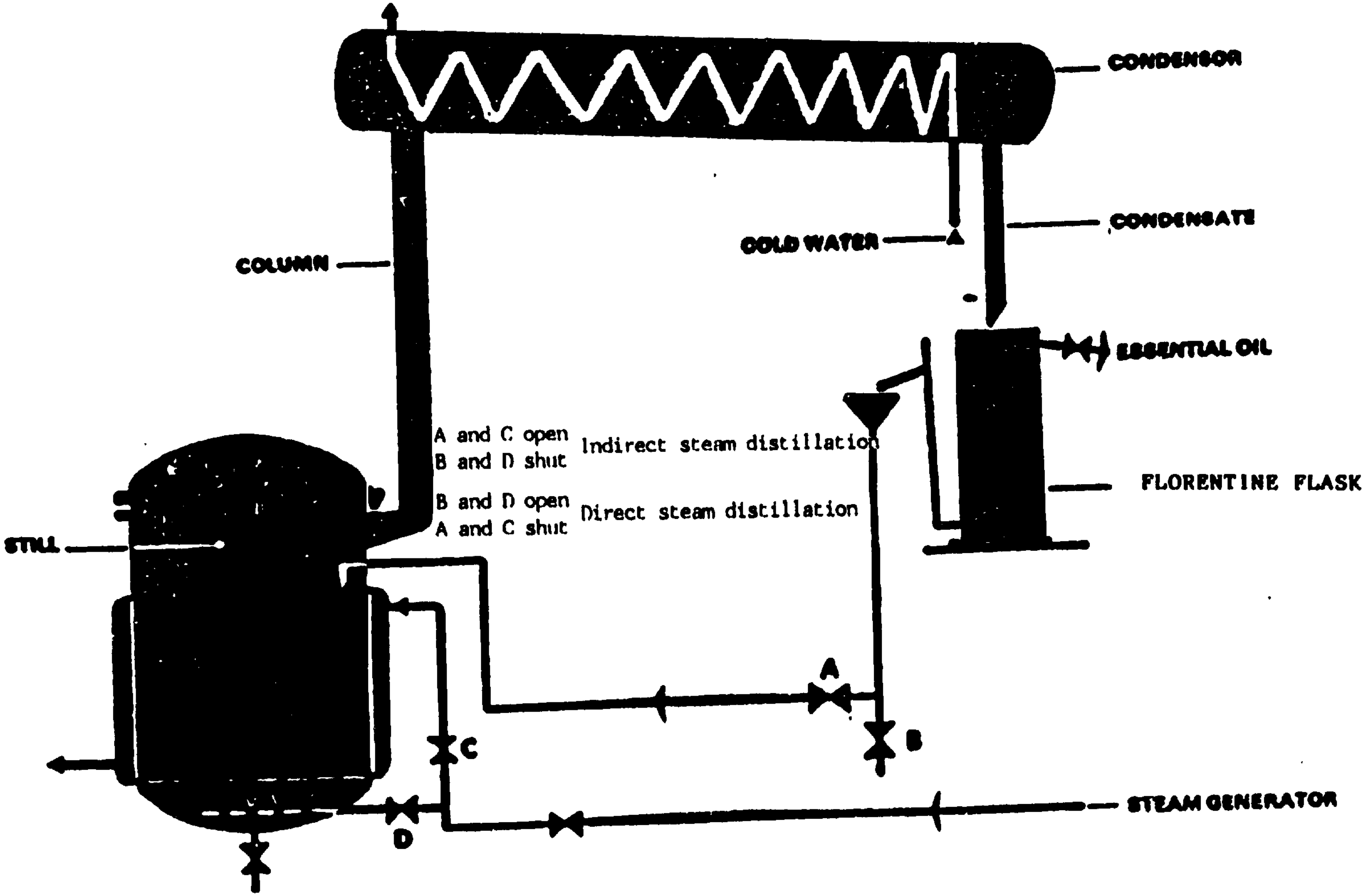
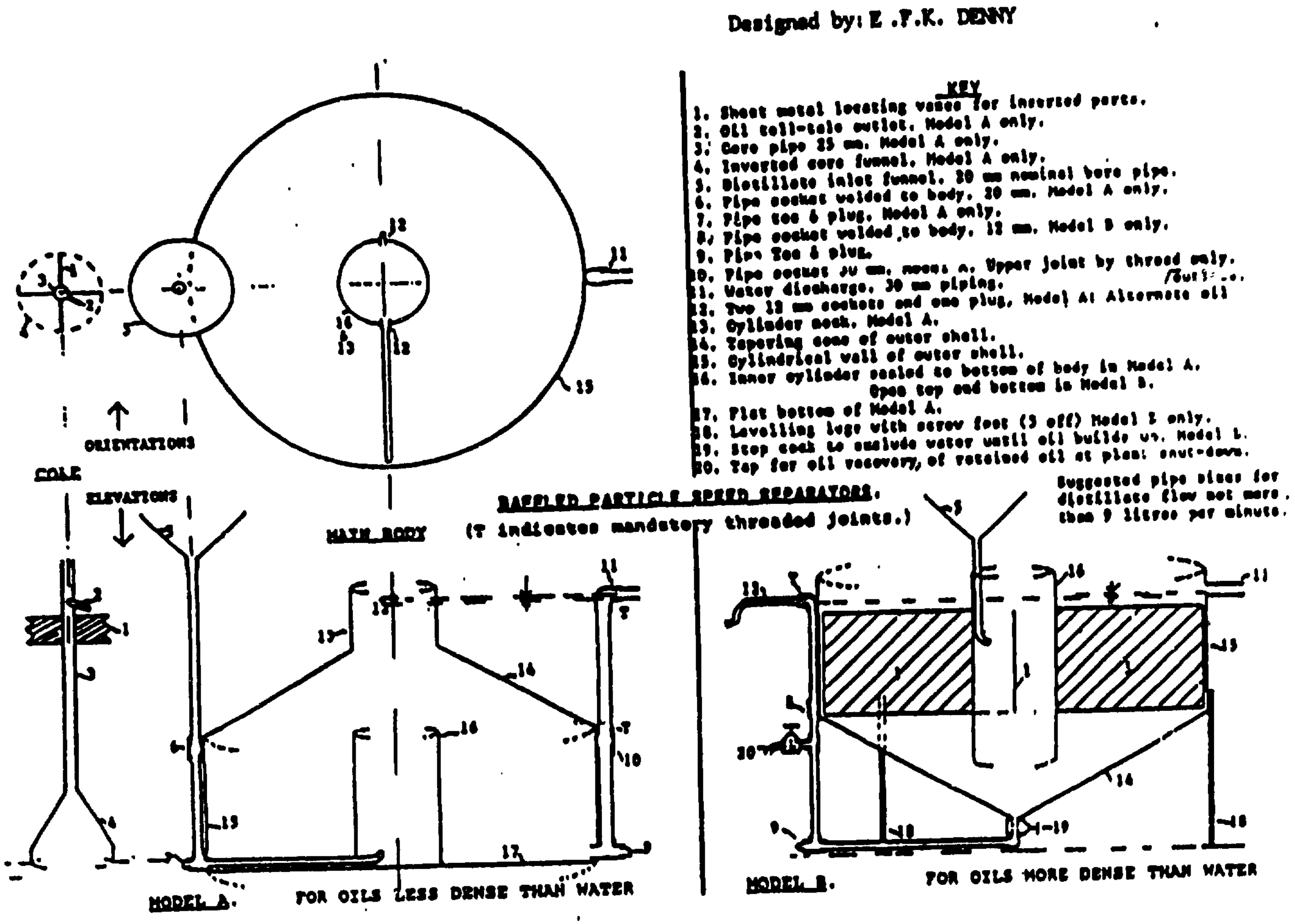


Fig. 1 Steam distillation diagram



Designed by: E.F.K. DENNY

KEY

1. Sheet metal locating vanes for inverted parts.
2. Oil tell-tale outlet. Model A only.
3. Core pipe 25 mm. Model A only.
4. Inverted core funnel. Model A only.
5. Distillate inlet funnel. 20 mm nominal bore pipe.
6. Pipe socket welded to body. 20 mm. Model A only.
7. Pipe tee & plug. Model A only.
8. Pipe socket welded to body. 12 mm. Model B only.
9. Pipe Tee & plug.
10. Pipe socket 20 mm. Model A. Upper joint by thread only.
11. Water discharge. 30 mm piping.
12. Two 12 mm sockets and one plug. Model A: Alternate oil /outlet.
13. Cylinder neck. Model A.
14. Tapering cone of outer shell.
15. Cylindrical wall of outer shell.
16. Inner cylinder sealed to bottom of body in Model A. Open top and bottom in Model B.
17. Flat bottom of Model A.
18. Levelling legs with screw feet (3 off) Model B only.
19. Stop cock to exclude water until oil builds up. Model B.
20. Tap for oil recovery, of retained oil at plant shut-down.

BAFFLED PARTICLE SPEED SEPARATORS.
(T indicates mandatory threaded joints.)

Suggested pipe sizes for distillate flow not more than 9 litres per minute.

Fig. 2 Florentine vessel or oil separator

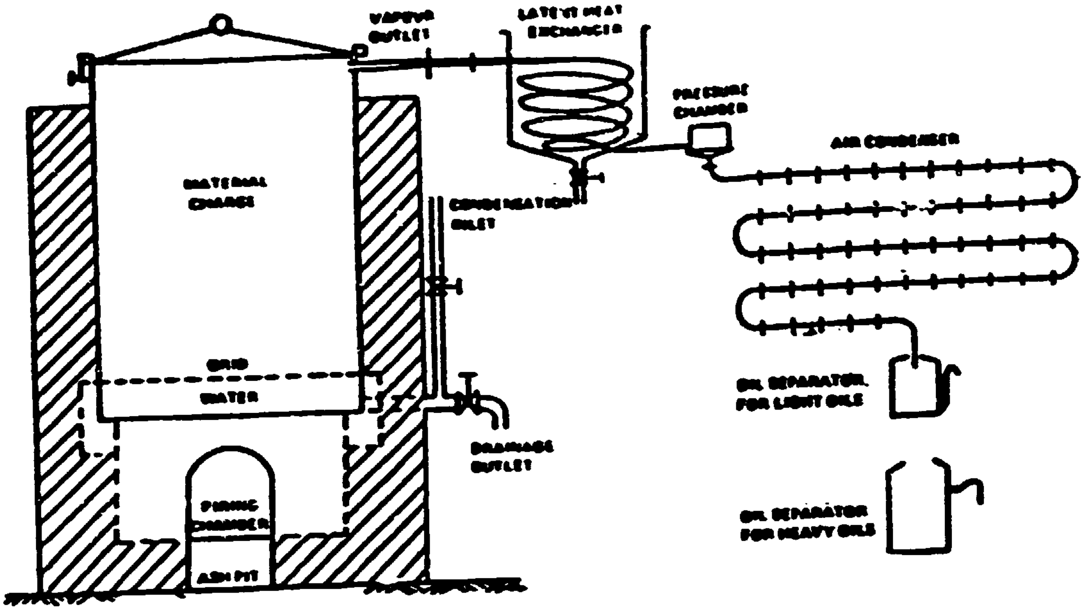
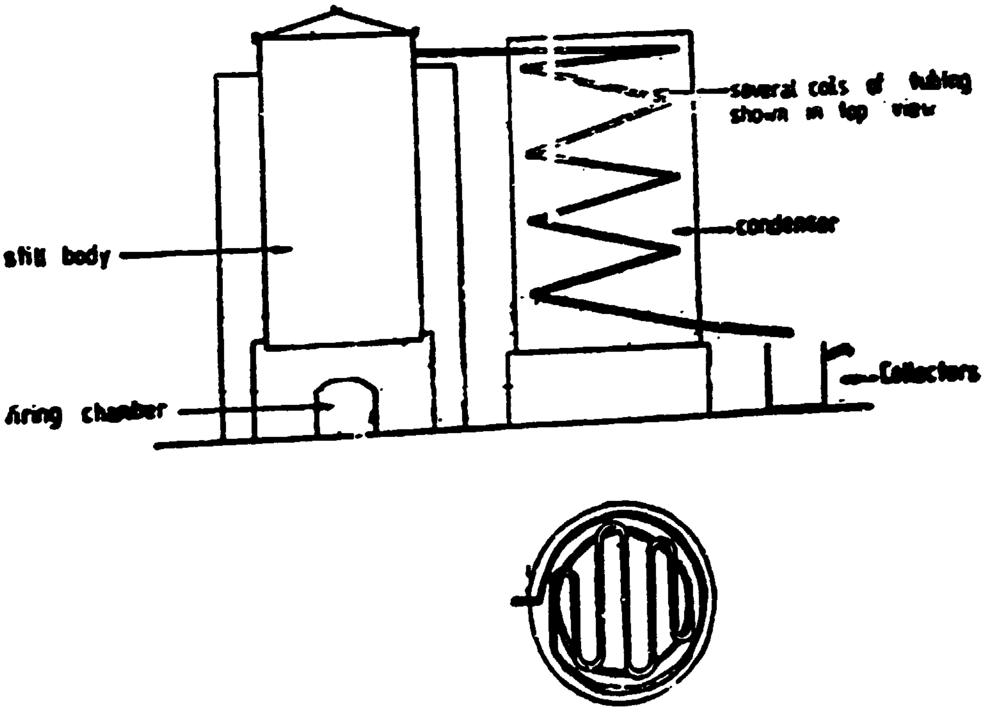


Fig. 3 **MANAKOKA assembly**



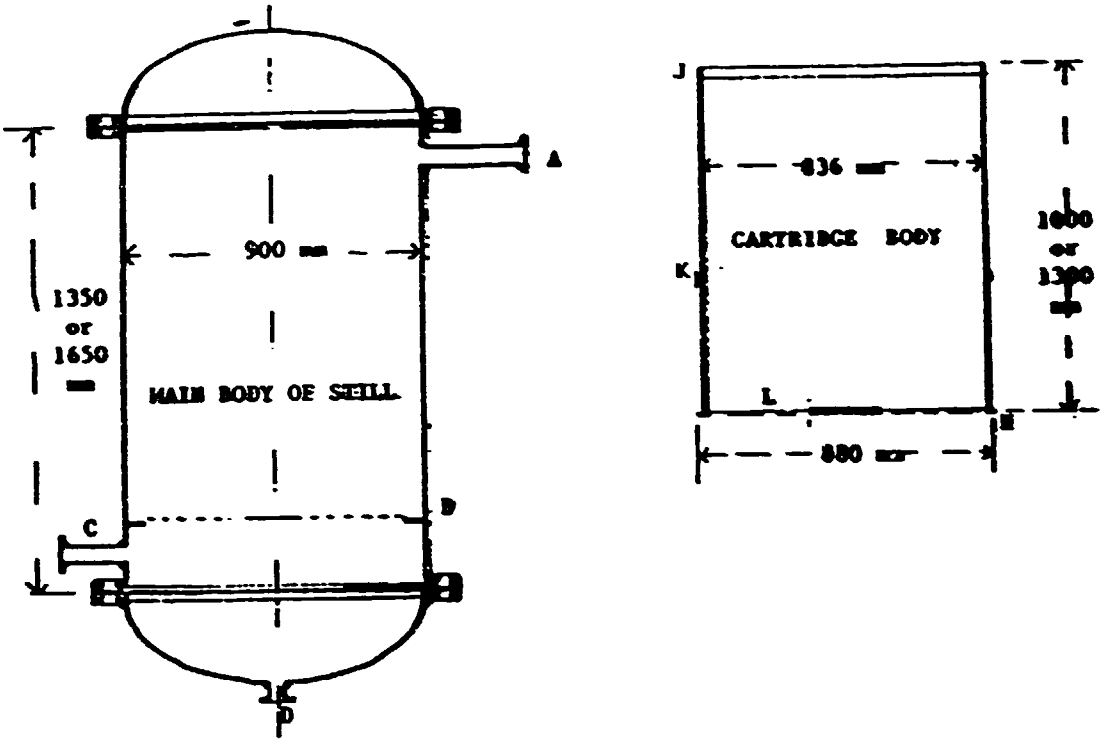
PLAN (top view of the condenser)

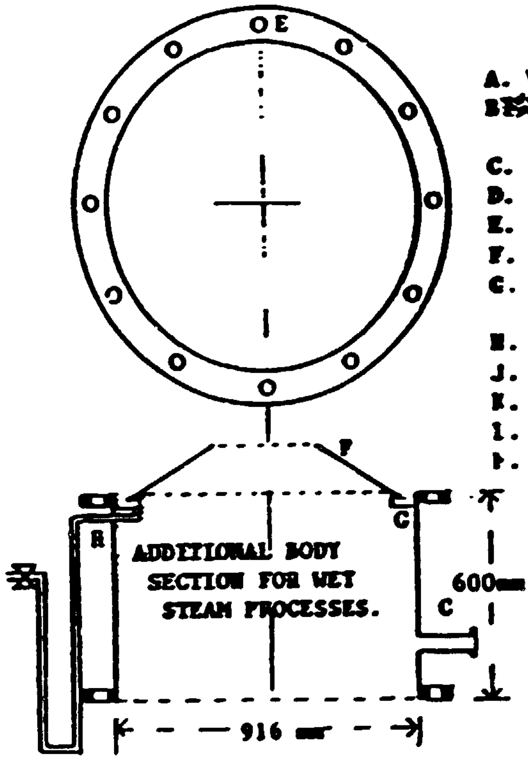
Fig. 4. CISIRILL BOITARE
Source: Wijesekera

A. The basic still with actual charge height 0.95 metres.

B. The same still with extension taking the potential actual charge height to 1.3 metres.

Fig. 5. Simple inexpensive stills
Source: E. F. K. Denny (Tasmania)





KEY

A. Vapour outlet 50mm s-steel flanged Table 'E'.
B. Ledge to hold cartridge & seal. 46 x 12 flat bar rolled into circle & welded to cylinder wall.
C. Steam inlet, alternates. 50 mm mild steel Table 'E'.
D. Drain 40 mm.
E. Bolts for holding sections together.
F. 1 mm thick s-steel cone open top and bottom.
G. Rolled angle 50 x 75 to make circular gutter for catching internal condensate.
H. Outlet from condensate gutter.
J. 50 x 6 reinforcing ring & two vertical side straps.
K. Swivel lifting points for wire suspending ropes.
L. Mesh bottom with centre solid over cone opening F.
M. Perimeter of 6 mm m.s. bottom making flange to rest on B.

NOTES.

1. Straight section of body and extension fabricated from 900 mm I.D. pipe, wall thickness 8 mm.
2. Top and bottom standard dished ends 8 mm thick.
3. Sections joined by flanges to B.S.T. Table D.
4. Top lid of still and top 200 mm of cylinder to be coated with pure tin after all welding etc completed. Inside surfaces only.
5. Vessel to be manufactured to code requirements and regulations for unfired pressure vessels for a working pressure of 105 kPa (15 p.s.i.g.)
6. All external surfaces of still to be insulated with 50 mm mineral wool or equivalent.
7. Cartridge body rolled from 3 mm mild steel plate.

APPLICATIONS.

According to type, this still will hold about 175 kg of field crop herb if built to take the 1300 mm tall cartridge. This height is advisable for carrying out parameter tests required for design data. The 1000 mm cartridge size (135 kg) is for testing the prolonged distillations of herbs bearing sesqui-terpenic oils. The still is also suitable for small production runs of such things as seeds of umbelliferae, or any other material where load sizes of 130 to 175 kg are appropriate.

Fig. 6. General purpose test still for wet or dry steam process
Source: E. F. K. Denny

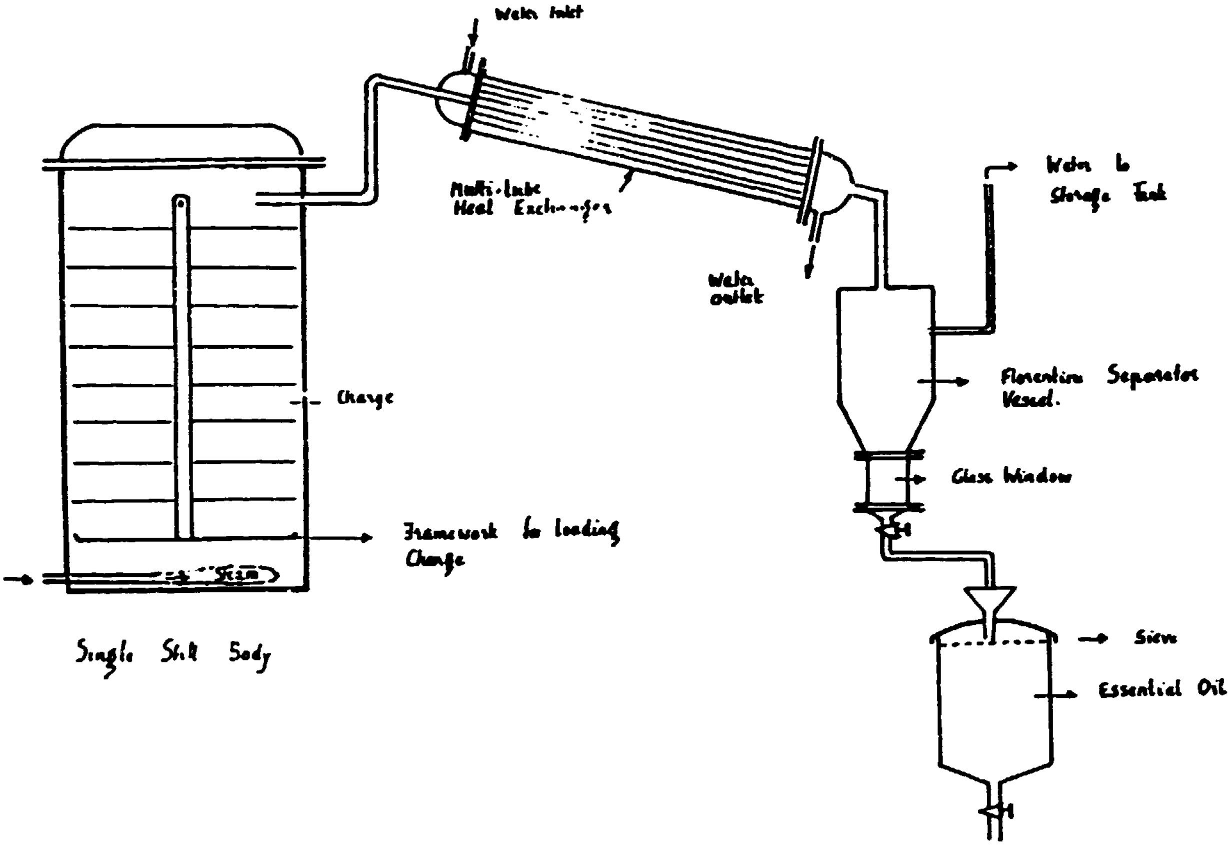


Fig. 7. Commercial still unit with multi-flanged loading gasket

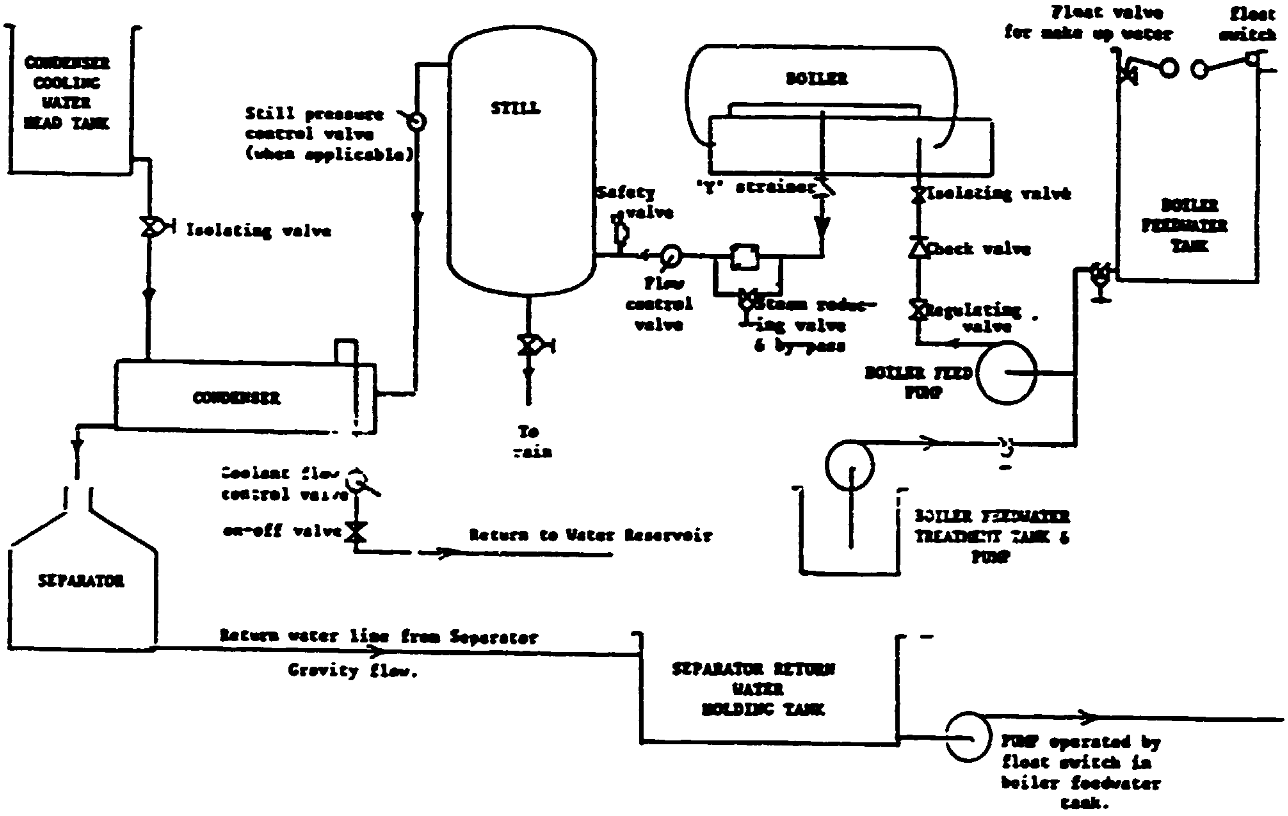


Fig. 8. Flow diagram and scheme of pipework for essential oil distillery equipped with satellite boiler for steam supply
Source: E. F. K. Denny

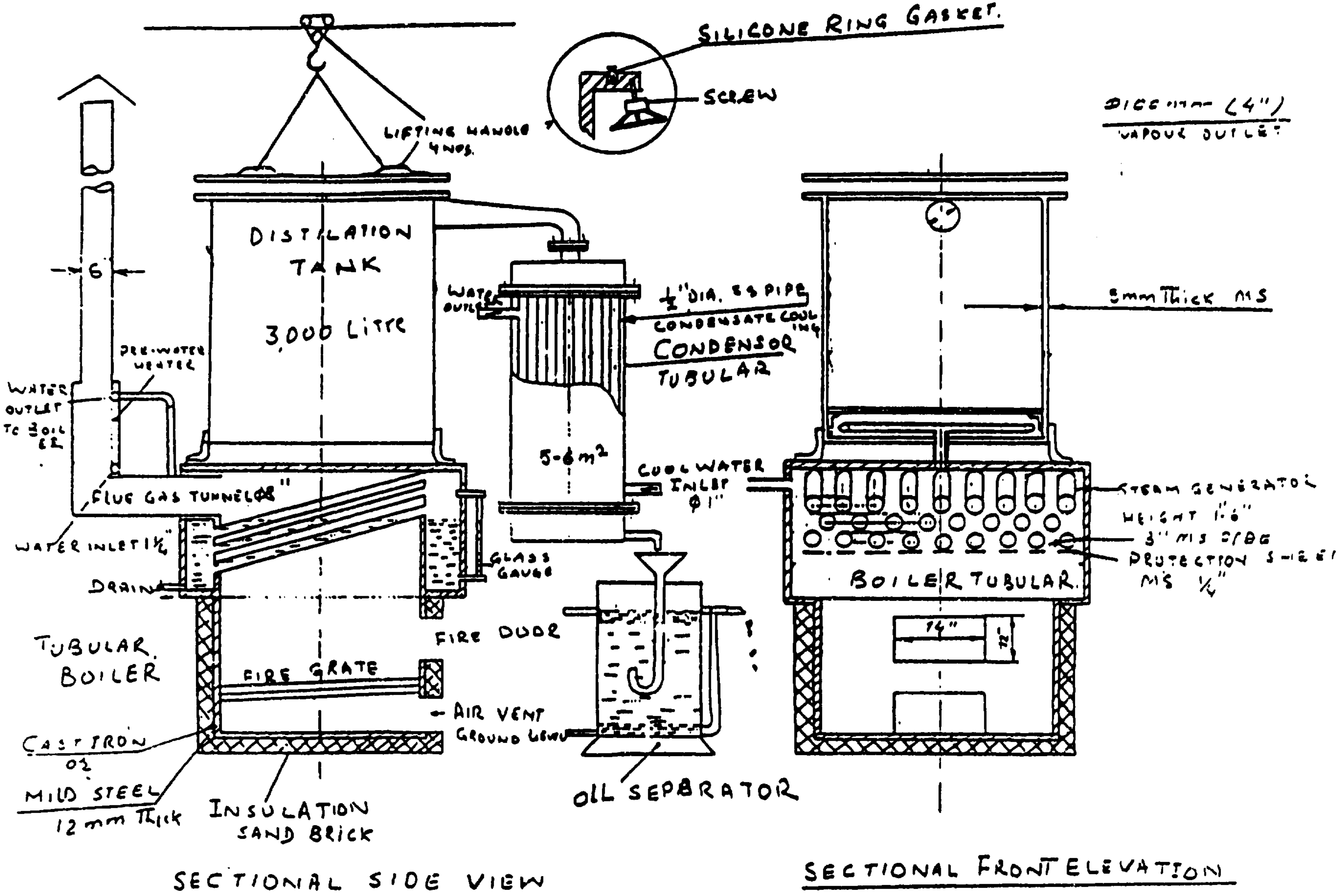


Fig. 9 Design Diagram of a steam distillation unit (Narasimha)

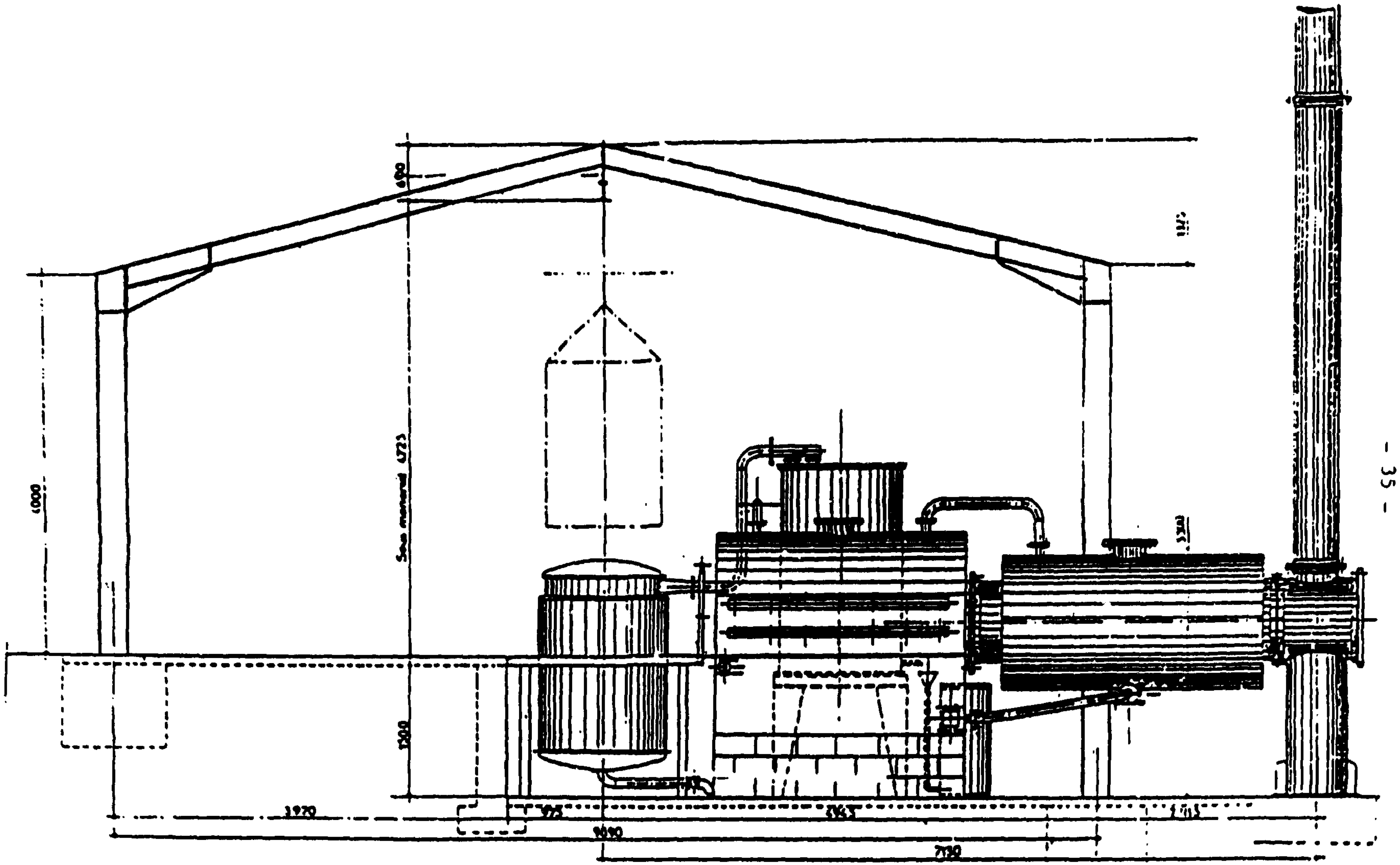
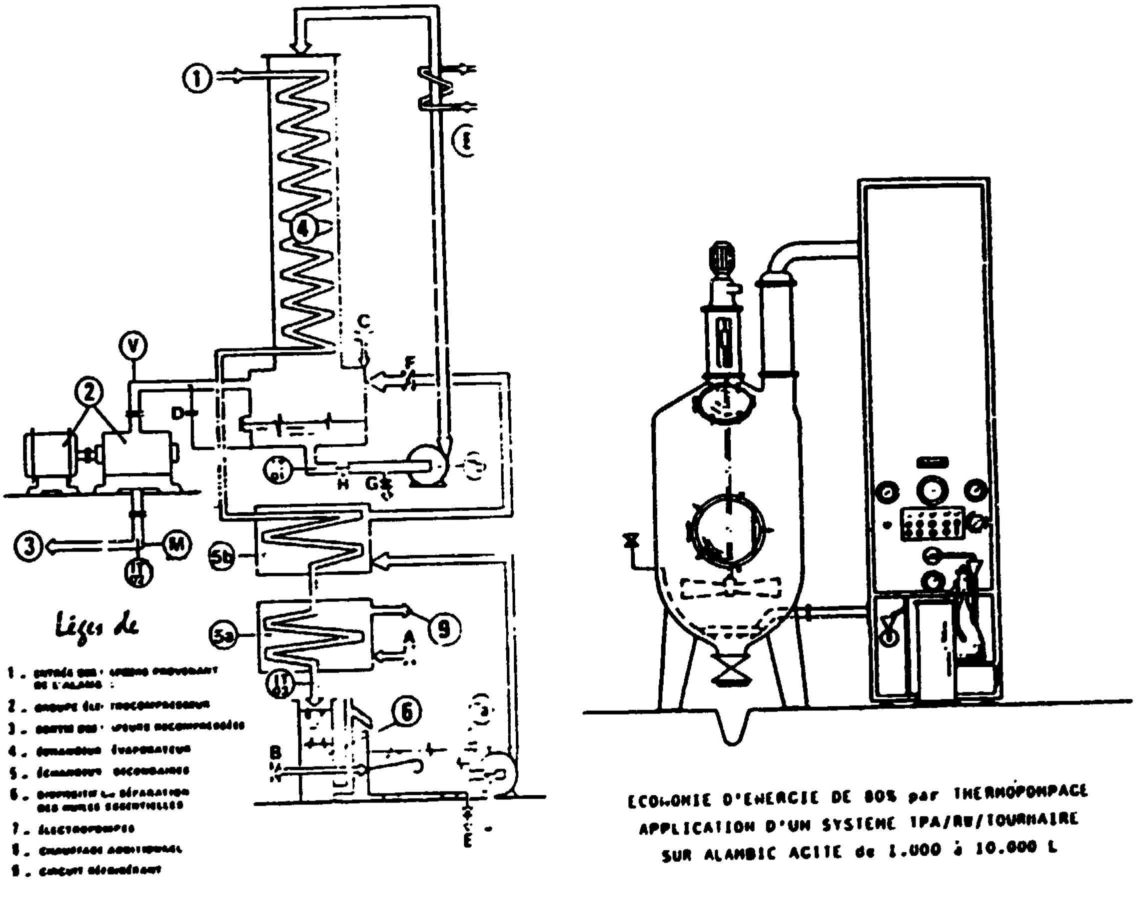


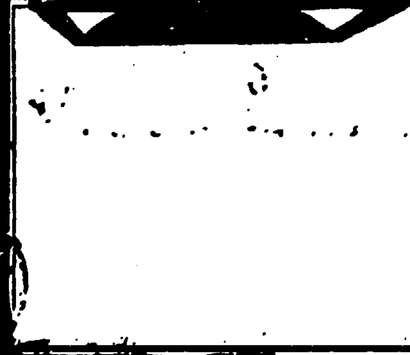
Fig. 10. Field still with an external boiler
Source: eyserric (France)



ECONOMIE D'ENERGIE DE 80% par THERMOPOMPAGE
APPLICATION D'UN SYSTEME TPA/RV/TOURNAIRE
SUR ALAMBIC ACIER de 1.000 à 10.000 L

Fig. 11. A modern type of energy saving, electrically heated still
Source: G. Tournaire (Grasse)

Fig.



Harvesting
Eucalyptus
polybractea

Suspended lid
awaiting new
trailer still

Stills
working

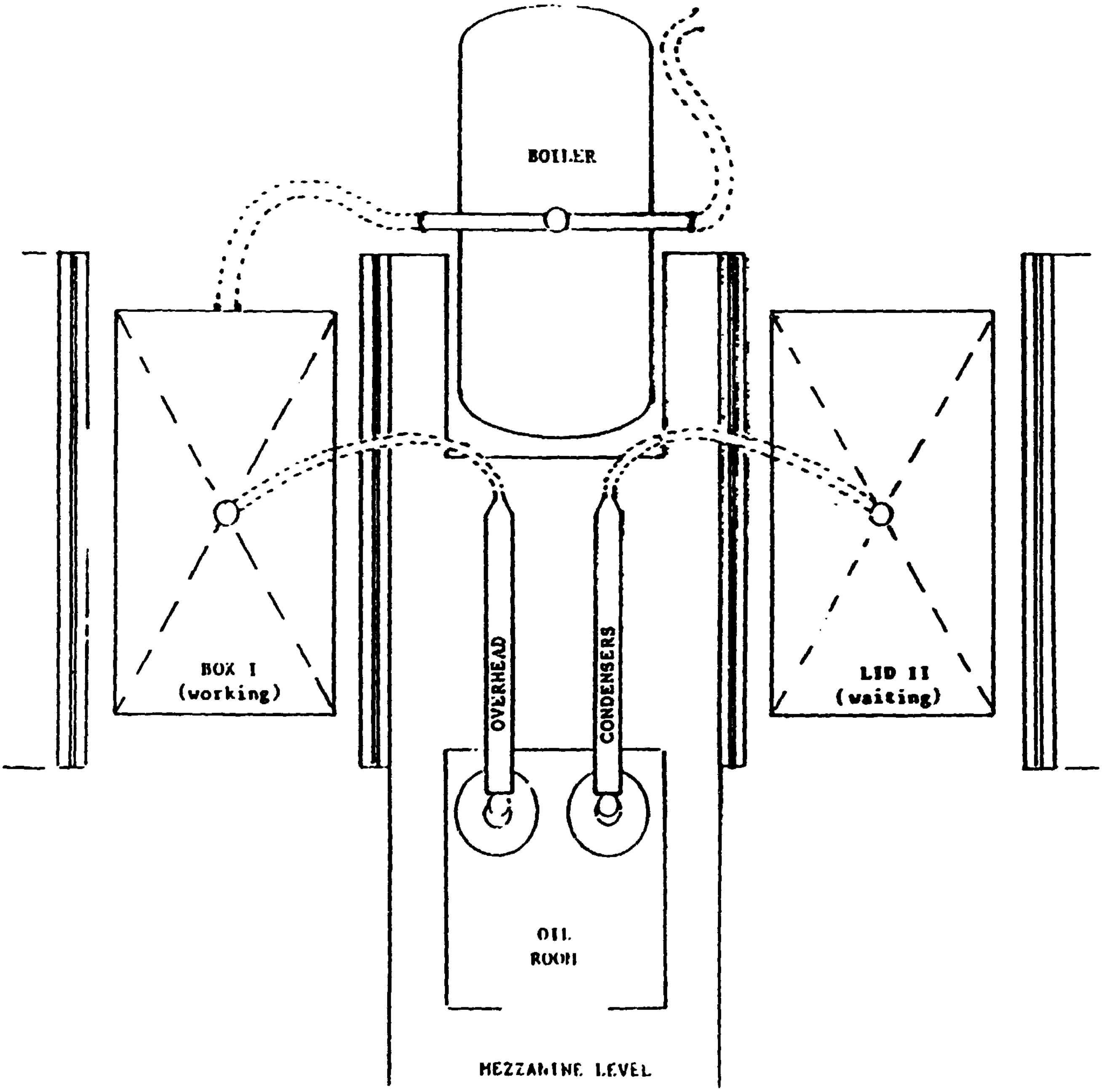
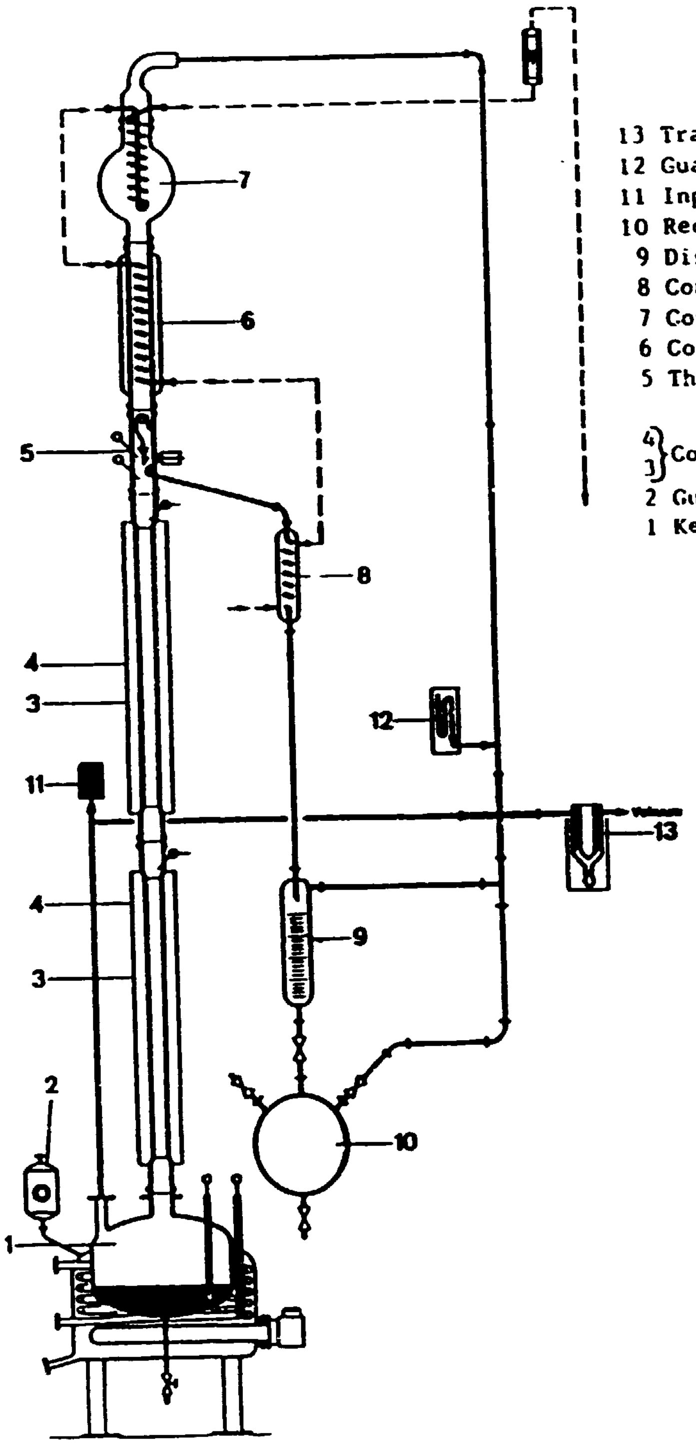


Fig. 13. Main items for box stills, plan arrangement



13 Trap
12 Guage
11 Input
10 Receiver/Collector
9 Distillate II
8 Condenser
7 Condenser
6 Condenser
5 Thermometer/Solenoid valves
4, 3 } Column lagging
2 Guage
1 Kettle

Fig. 14 Fractionating column

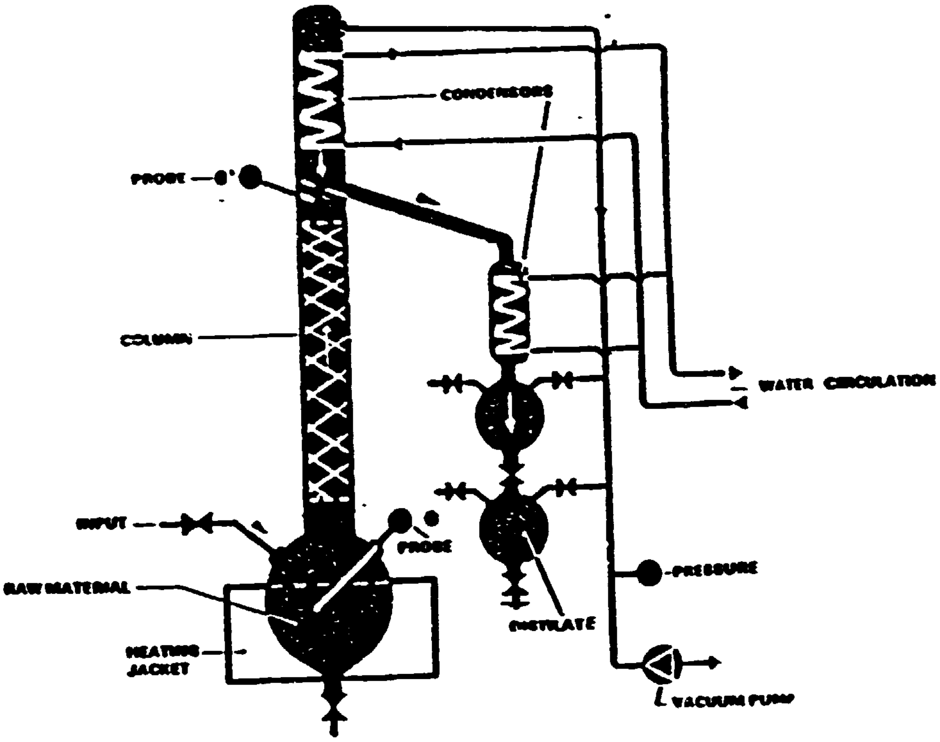


Fig. 14a. Vacuum distillation diagram

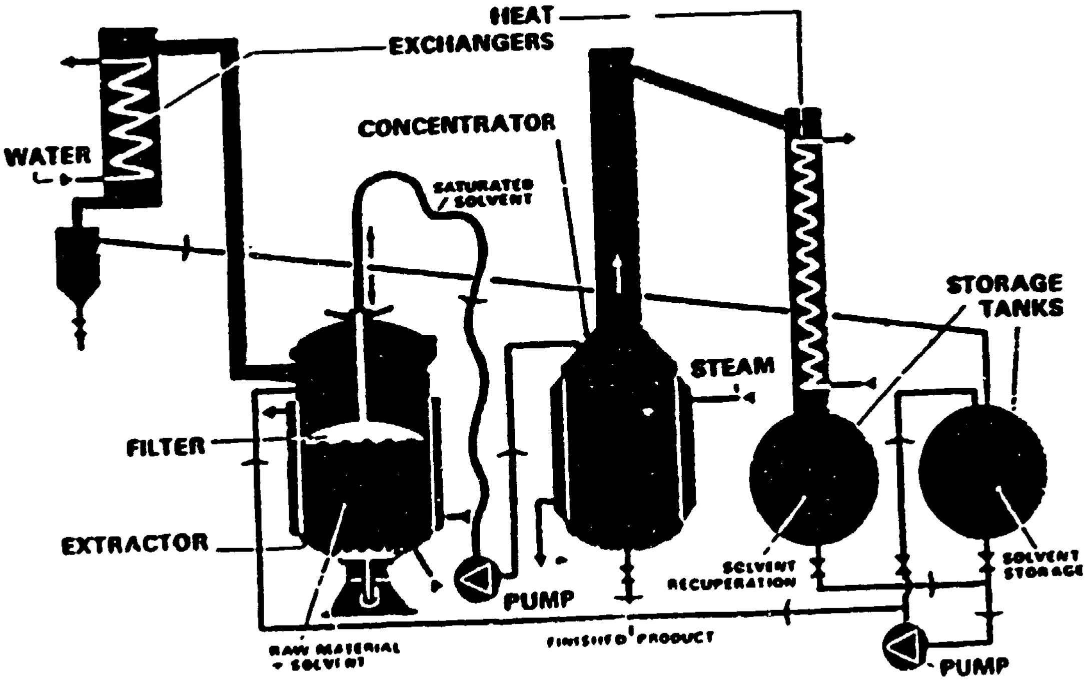


Fig. 15. Resinoid extraction unit

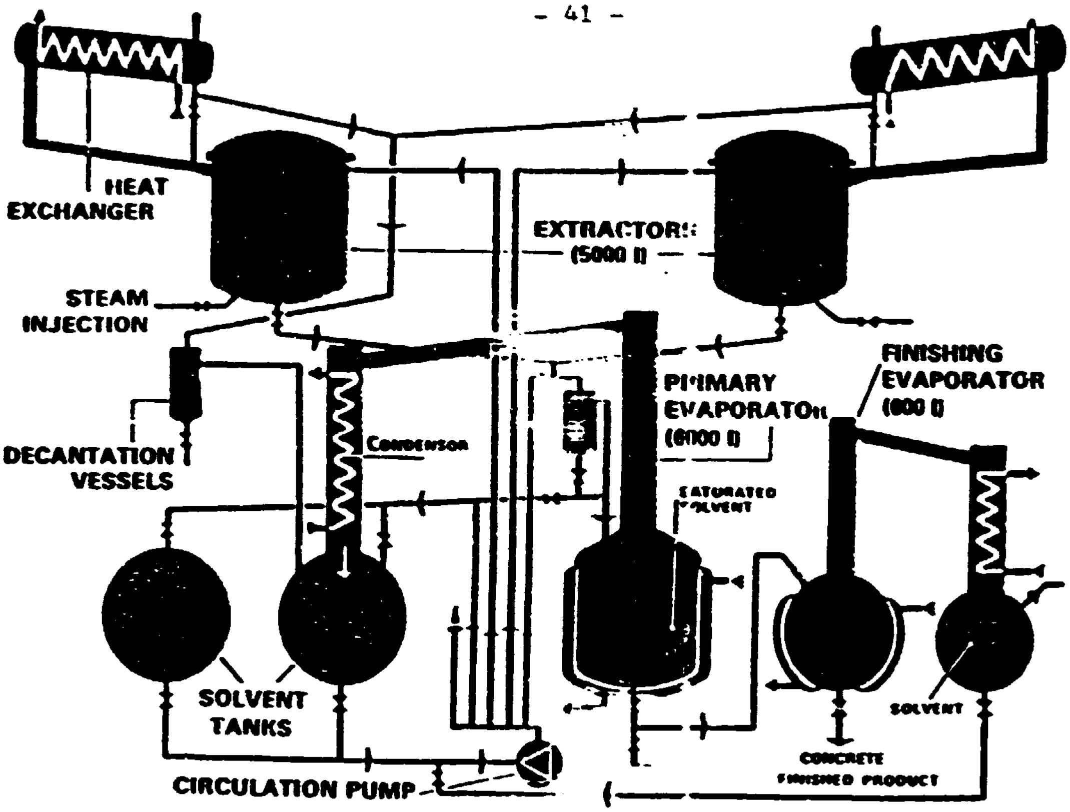


Fig. 16. Solvent extraction unit

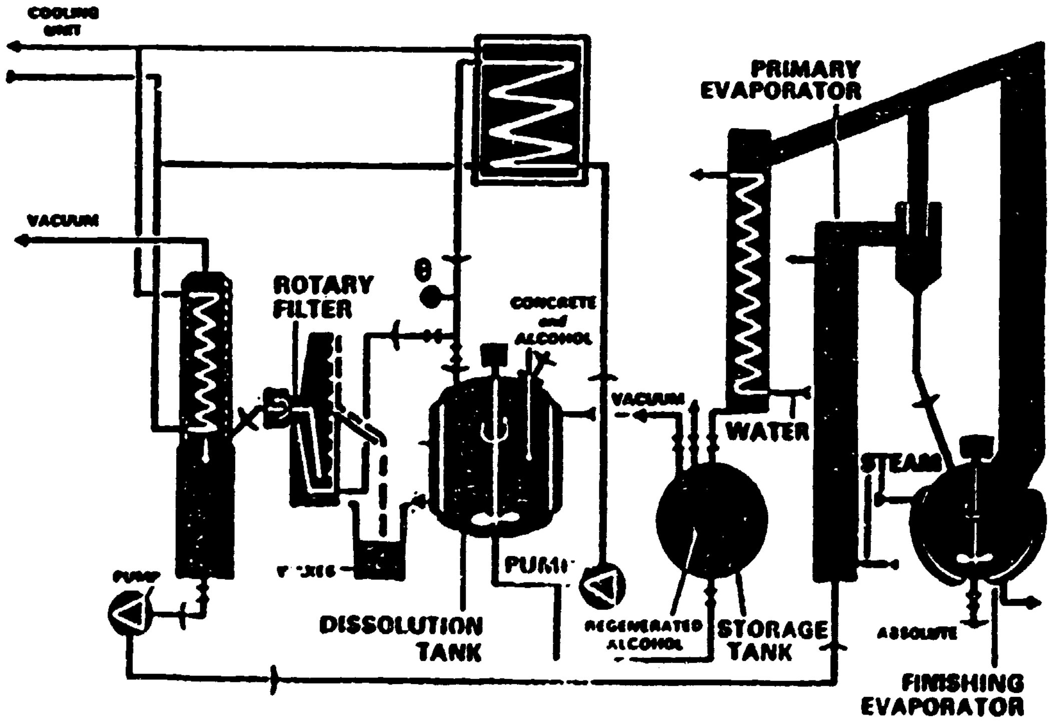
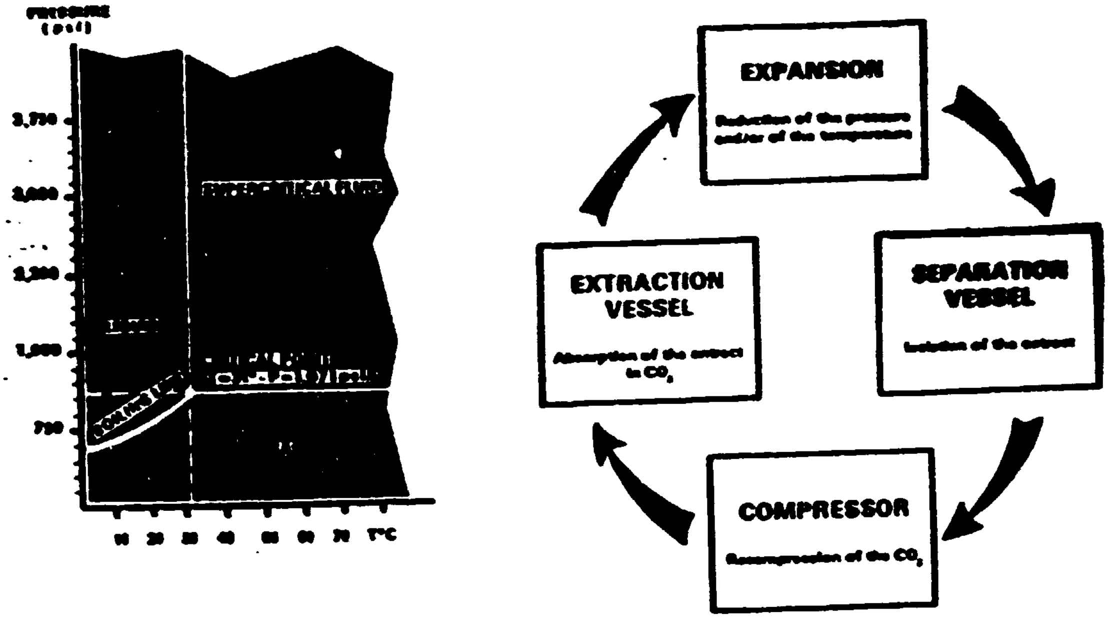


Fig. 17. Transformation of concretes into absolutes



PHYSICAL STATES OF CO_2

PROCESS DIAGRAM

Fig. 18. CO_2 extraction diagrams - physical states and process

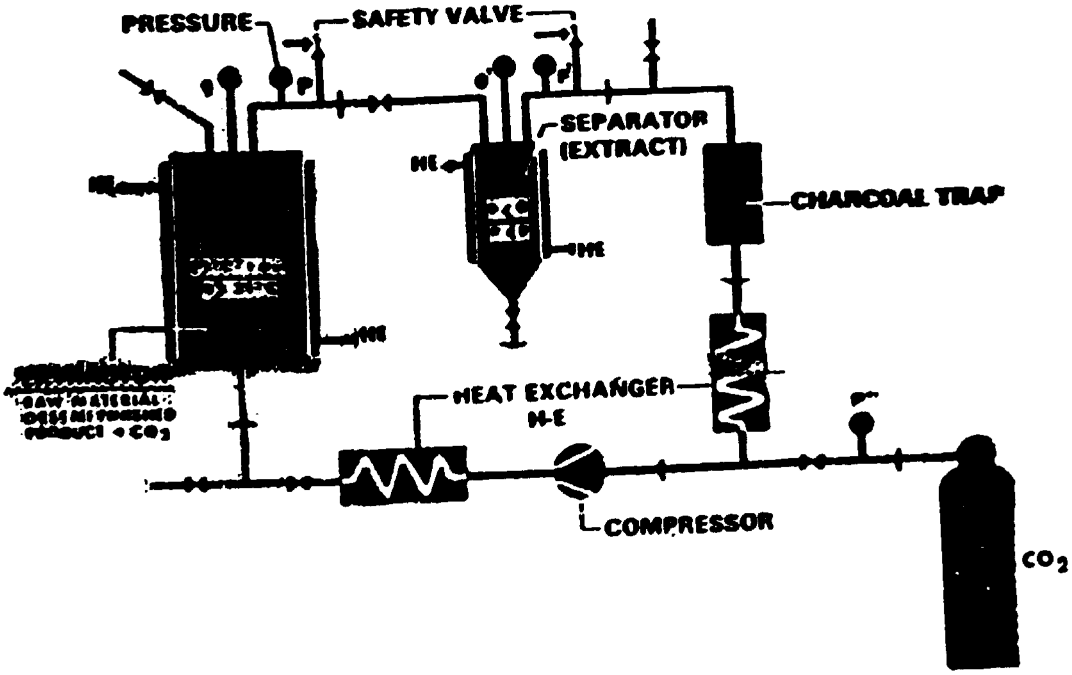
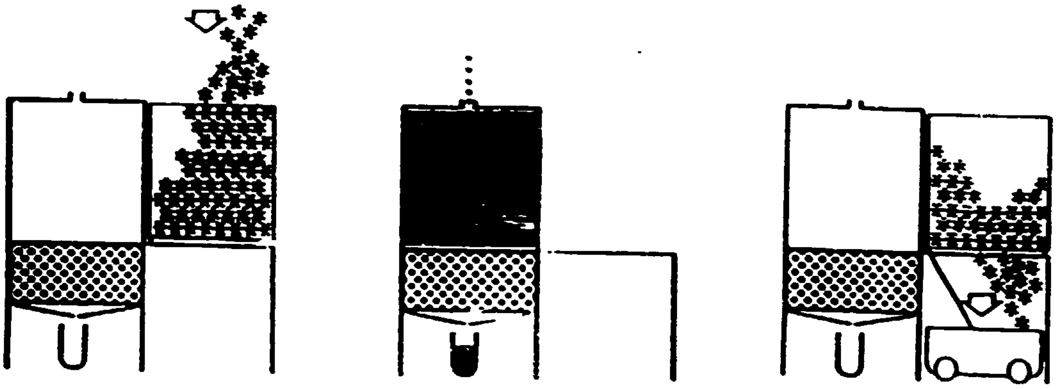


Fig. 19. CO_2 extraction diagram



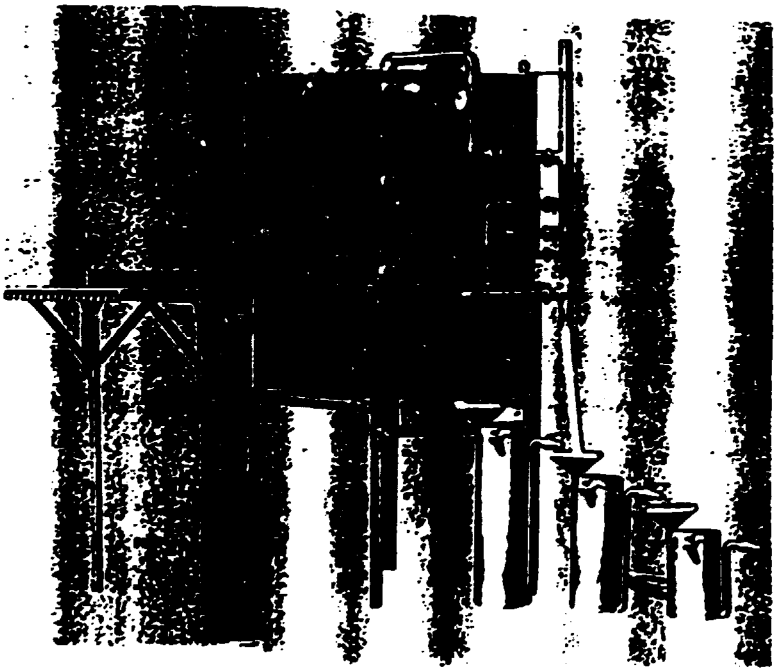


Fig. 20. "Cooler" underneath the still body (hydro diffusion technique)

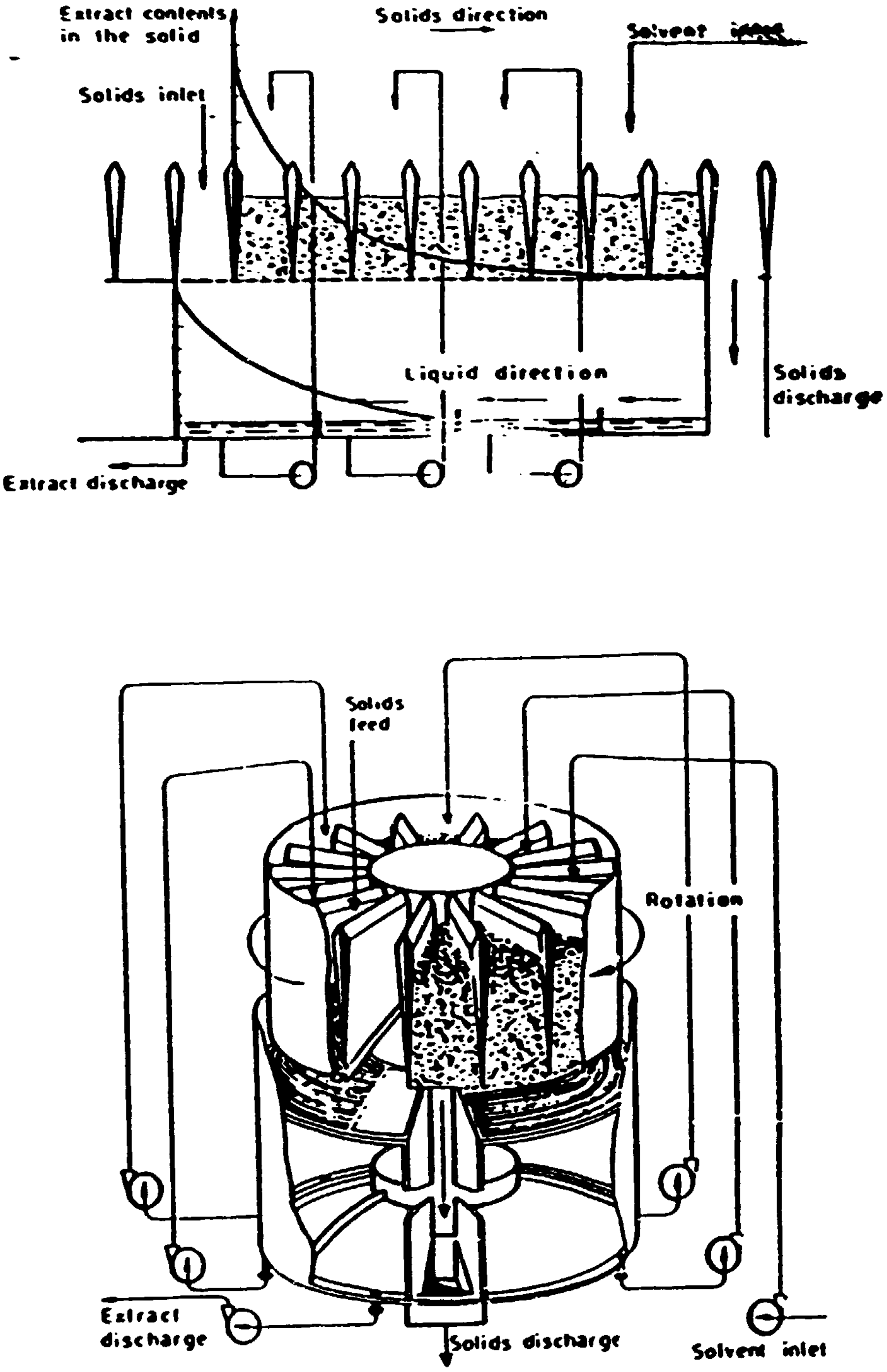


Fig. 21 - Diagram of carousel extractor

Fractionation and Production of Isolates

Dr. W.S. Brud

Introduction

Vacuum and normal pressure fractional distillation (fractionation, rectification) is the most commonly used process for purification, upgrading or isolation of components from essential oils prepared by steam distillation of plant materials or by other methods.

Basically fractionation of essential oils is used for:

1. Purification of crude oils by separation of impurities, residues, and/or preparation of the oils for blending. For this purpose very often fast simple distillation is enough (e.g. separation of insoluble resins from some pine oils). This can be also performed in a fractionation unit without reflux.

2. Upgrading (quality improvement) of the oils by separation of unwanted components, increase on contents of main (most important) component (s), and/or preparation of certain fractions of the oil for specific use (e.g. terpeneless citrus oils).

3. Isolation of important components either for direct use in perfumery, flavours, medicine or aromatherapy, or for further processing in aim to obtain more valuable aroma chemicals (e.g. citral ex Litsea Cubeba eugenol from clove oil). Pure components can be also used for blending of the oils in aim to increase content of the component in blended oil.

4. Research work (analysis) of new types of oils from new plants or new variety of known plants. Fine fractionation in analytical work is auxiliary method for GC-NMR-MS methods.

Before any attempt is made to set up a fractional plant or a laboratory fractionation unit and select proper equipment, the basic goals and scale of the process must be established. These can be one or more of the above listed applications of fractionation of the essential oils.

In all cases the fractionation plant for essential oils shall consist, apart from the basic unit (see selection of the equipment) of laboratory scale fractionation column (scale from 100 ml to 1000 ml), of standard analytical equipment (density, refraction index, optical rotation) and as an absolutely necessary instrument, a gas chromatograph (high speed) with integrator and computer for quantitative determination and compositional analyses of oils and fractions. Also most crucial is a good library of standard chromatograms of the oils to be fractionated. It is assumed that the Gas Chromatograph if outfitted with a variety of different columns for a range of analytical purposes.

Unless the basic unit is aimed for routine production scale fractionation of one oil e.g. for separation of one component, a laboratory with all analytical equipment is essential. In most cases for which this manual is prepared, especially when fractionation should be performed to find out the main contents of the oils, sequence of fractions, boiling points of fractions and components, before any larger scale processes could be started. This is to avoid experiments with valuable products which may result in decomposition of the oil, unexpected loss of the material and so on. For the same reason, the gas chromatograph and general analytical laboratory services should be at permanent disposal of the chemist operating the fractionation unit to enable immediate analysis of the fractions during the process (see next page on practical remarks). This is also an absolute necessity during all experimental batches as fractionation of an unknown oil (or oil of a new quality) can be properly performed only according to analytical results for each fraction. Otherwise either process shall be regularly interrupted for analysis (which will certainly affect the process and quality of the product) a large number of small fractions will have to be collected, which after analysis will be combined to give final fractions of specified composition.

Before some details on equipment and the practical aspects of the process will be presented basic information on fractional distillation itself shall be given for a proper understanding of the terms and phenomena described.

As stated before, both theoretical and engineering aspects of this booklet have had to be simplified. Also not all problems of fractional distillation are covered. Information given here are limited to fractionation of essential oils only and shall be good enough for a basic understanding of work performed and equipment used. All readers who wish to extend their knowledge of chemistry and technology of fractionation, must of course have further instruction and refer to other more detailed and specialized publications.

FRACTIONATION PROCESS AND ITS PARAMETERS

The process of fractional distillation of essential oils consists of the following steps:

- boiling of the oil in the evaporator (evaporation)
- separation of constituents of the oil (fractions) according to their boiling points in the column on surface of packing.
- condensation of vapours emerging from the top of the column with partial return of the condensate to the column.
- cooling of the condensate, collection of the fractions.

Basic information on fractional distillation of essential oils under vacuum will be given here. The principles of fractional distillation under normal pressure, rectification, deterpenation, desequiterpenation are in most cases the same as for vacuum fractional distillation (vacuum fractionation) thus only differences or specific data will be mentioned when necessary.

BOILING (EVAPORATION)

The basic process of any distillation is transformation of liquid into its vapours. A migration of molecules from liquid into gas over the liquid surface (i.e. evaporation) is a continuous process and its rate increases with temperature. Boiling is the process when the temperature of the liquid is so high that evaporation takes place within the liquid. The vapour pressure of the liquid is then equal to the external pressure above it. At this moment the heat supplied is consumed mainly by energy (heat) of evaporation, loss of energy to the surrounding air and temperature increase due to changing composition of the liquid.

It must be considered that except in flow-through multitubular external heat exchangers used in some production scale units (see Equipment), the heating takes place through walls of the evaporator or by a heating coil (or multitubular heat exchanger) inside the boiler. The heating medium (steam, water, oil, electric coil) temperature shall be usually much higher than the actual boiling temperature of the liquid due to the dissipation of the heat referred to earlier. Thus the temperature inside the liquid is different at different points and this could result in local super heating of the liquid (on the walls or coil) which in turn can decompose or chemically alter the constituents of the distilled oil. Various chemical reactions or polymerisations can occur which can alter the chemical and physical characteristics of the oil. Although inevitable in standard equipment, the super-heating can be reduced to minimum by stirring, which is a necessity in all units over 500 l. To decrease adverse results of local super heating, the author advises not to use heating coils or tubes inside the evaporator. They slow down natural migration of molecules inside the liquid and also are extremely difficult to clean from deposited polymers or waxes or resins contained in oils or formed during distillation.

Permanent, quiet, even boiling shall be maintained during the entire process by adequate heating. It is the general rule that temperature of the heating medium shall be around 10-20 °C higher than boiling point of the liquid (i.e. temperature) <u>in</u> (not over) the liquid. This shall be controlled by appropriate measuring devices (mercury thermometer, thermocouple) and preferably automatically regulated according to set parameters. The rate of heating will have an effect on the evaporation rate which shall be adequate to ensure the vapours flow through at an even rate in the column. Factors such as the optimum pressure drop, reflux rate condenser efficiency etc. shall be established in test runs as they will be different for different oils (see example instruction for unknown oil).

When the liquid is a mixture of different chemicals which is the case in all essential oils - there is certain equilibrium of contents in liquid and vapour phases with higher percentage of lower boiling products in the vapour phase. During the course of the fractionation process, as vapours after additional separation in the column (see below) are condensed and collected, the components will decrease with corresponding increase in the amount of higher boiling products. This separation takes place on the phase boundary i.e. on liquid-vapour surface which in the evaporator is limited to the surface of the liquid in a flask or boiler. Thus, to improve separation this area is enlarged by use of the fractionation columns.

As stated above many components of the oils are heat sensitive and with all precautions in heating systems they will decompose at or below their boiling point. This problem can be solved by use of vacuum i.e. distillation under reduced (lower than atmospheric) pressure. With decrease of pressure temperature of the boiling of the constituent mixtures of chemicals (boiling point) will also decrease. This can be presented on the graph showing the relation between boiling point and pressure for citral, geraniol, linalyl acetate and geranyl acetate.

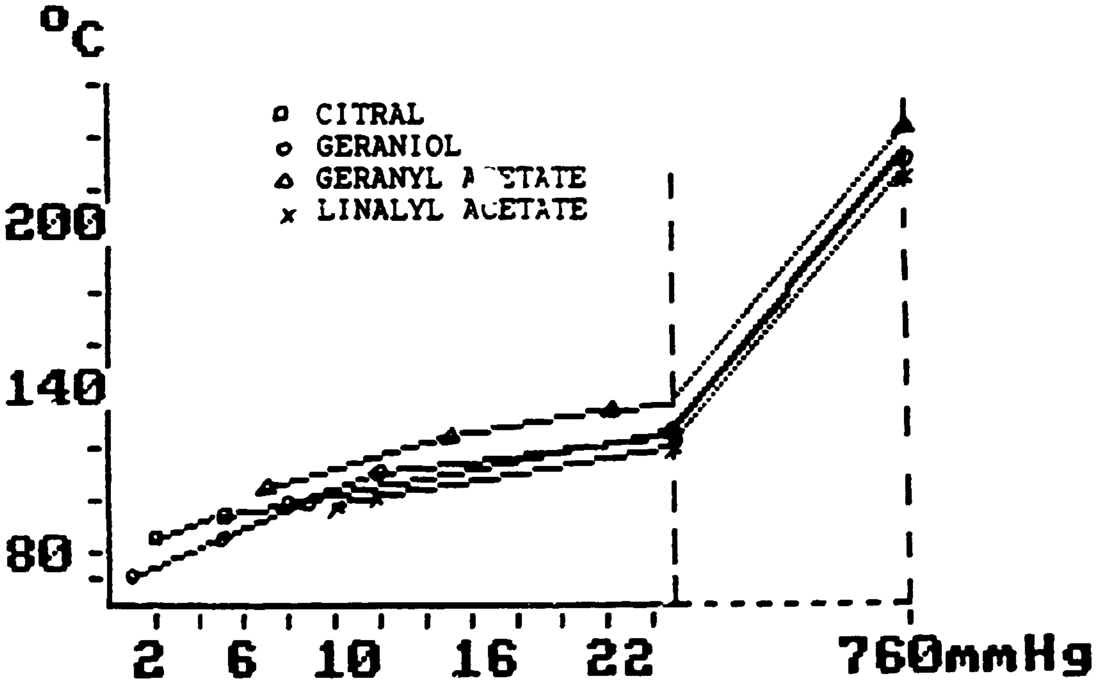


Fig. 22. Graph showing the relation between boiling point and pressure for citral, geraniol, linalyl acetate and geranyl acetate

Reduced pressure is traditionally expressed in conventional millimetres of mercury (1mm Hg - 1.333224 hectopascal) called sometimes Torr. Normal, atmospheric pressure is equivalent to around 760 mm Hg. Reduced pressure used in fractionation of essential oils is most common at the level of 5 - 20 mm Hg sometimes going down to 1 mm Hg or lower in research processes.

It must be considered that essential oils are complicated mixtures with some components boiling at very low temperatures (e.g. terpene hydrocarbons) and others with very high boiling points (sesquiterpenes, polymers). In practice it is advisable to keep the temperature in the evaporator under 150°C. This is safe for most of the components and the process could be controlled by stepwise reduction of the pressure. Thus the process shall be started at normal pressure and finished at around 5 mm Hg. Gradual reduction of the pressure (especially in fractional of an unknown oil) shall also prevent losses of low boiling components which at reduced pressure may boil at the temperature lower than the temperature of the cooling water used in the condenser or heat exchanger.

In such a case, vapours will pass through the condenser and/or cooler and will be exhausted via the pump and lost. This can also damage the pump (especially oil pumps). Although proper procedures can prevent the above-mentioned damage, it is necessary to install the vacuum line between the unit and pump, "cold traps" (see Equipment) which are at very low temperatures and will be able to condense all vapours.

It is the common practice that the technology parameters for vacuum fractionation of any specified essential oils shall be established experimentally by one or more test runs starting with a laboratory scale. Some theoretical calculations may be helpful but only for designing of industrial scale units for routine fractionation process instructions with just the use of theoretical data (e.g. boiling points, chromatography). Especially when crude oils, with variable composition, are distilled.

All evaporation units of all scales and materials should be well insulated to prevent heat loss and ensure good heat exchange. Upper parts of glass flasks should be covered with insulating material (e.g. mineral wool) especially in cool climates.

THE FRACTIONATION COLUMN

As already stated evaporation i.e. mass exchange between liquid and vapour takes place at the phase boundary. Thus better separation can be achieved with increasing interphase surface. For that purpose fractionation columns packed with materials that give a large surface area in the column, are used. Vapours from the evaporator enter the bottom of the column and flow upwards with partial condensation on the column and packing surface. The condensate flows down and meets the vapours. This way a continuous exchange of molecules between upgoing vapours and down flowing liquid creates the system of fractionation. The composition of the vapours over the liquid at the same point with vapours containing always high amounts of lower boiling products. As condensation and vaporisation occurs along the column (due to different temperatures at different levels) the composition of both phases is different right along the column. The highest content of lowest boiling product is at the top most point of the column. As fractionation proceeds lower boiling products, one after another, will appear at the top of the column and in the condenser wherefrom they will be collected. Their mixtures will appear in between as interfractions.

The effect of separation of the components in the state of equilibrium is expressed in the so called "theoretical plates". The number of theoretical plates is equal to the number of following equilibria which is necessary to obtain a specific content of lower boiling product in the condensed distillate. This can be schematically illustrated by the graph of fractionation of a mixture of transdichloroethylene (A) and 2-butanone (B) at 760 mm Hg.

On the graph x is A content in liquid and y is its content in vapours. In the state of equilibrium vapours over a liquid containing x 1 of A will contain y 1 of A. At the same level of the column the condensed liquid will have the same x 2 content of A which is in equilibrium with vapours containing y 2 of A. Each of these steps will represent one theoretical plate. According to this graph three theoretical plates are necessary to achieve Y 3 concentration of A in distillate from starting mixture containing x 1 of A.

Usually the fractionation process in the column is carried out in isothermal conditions i.e. there is no external heat exchange along the column. For that purpose all the columns shall be ideally insulated (e.g.

vacuum jacket on glass columns) or in cold climates heated to prevent heat loss during the process.

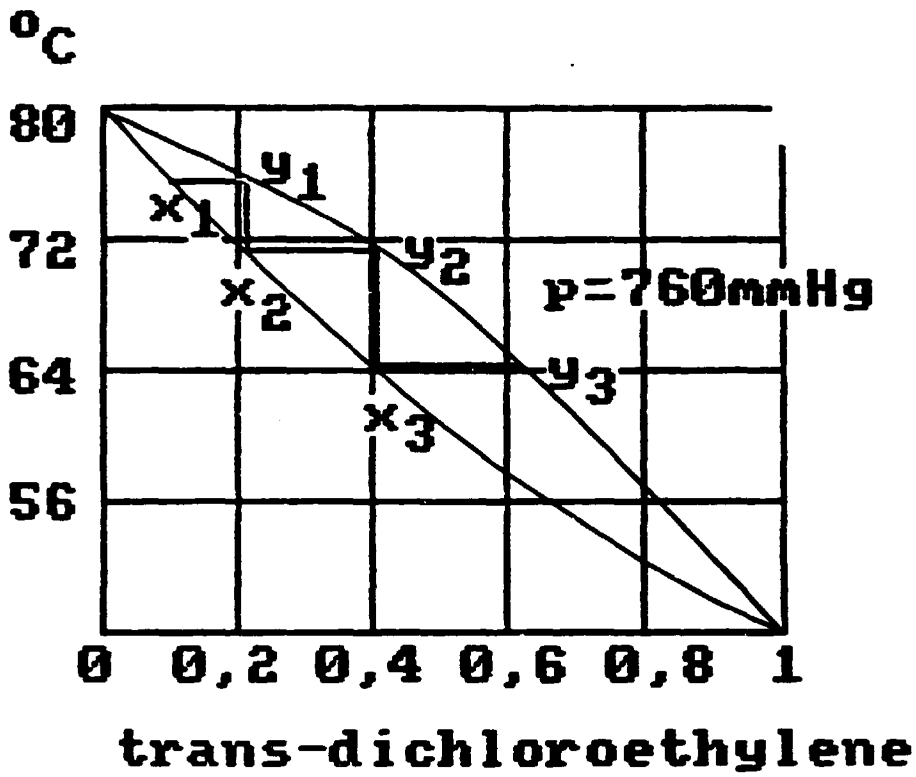


Fig. 23. Graph of fractionation of a mixture of transdichloroethylene (A) and 2-butanone (B) at 760 mm. Hg

The effect of separation of the components of the mixture in the fractionation column depends on the following factors:

- boiling point difference at selected vacuum
- packing surface in the column i.e. length of the column and packing type selected.
- rate of the process (vapours flow rate) i.e. heating rate and reflux ratio.

It is obvious that with a significant difference in the boiling points of two components their separation is easy unless they form so called azeotropic mixtures. Thus it is sometimes advisable to carry on fractionation at higher temperatures (and/or pressures) to use higher differences of boiling points in such circumstances.

Longer column and larger surface area of packing will certainly improve separation. It must be however considered that a longer column will mean higher pressure drop and (with the best insulation) higher temperature difference which will result in the need for a higher temperature in the evaporator and consequent possible damage to the oil. Heating of the column may help to some extent. Some practical compromise must be achieved between necessary parameters and expected results. A higher heating rate will increase vapour flow through the column but at the same time condensation and flooding in the column will adversely affect separation. A slow rate of heating will give better separation but a longer time taken by the process and longer contact of the oil with the heating surface again can damage the material.

Separation rate of the process is significantly affected by the reflux ratio.

COLUMN HEAD, REFLUX AND CONDENSER

The vapours emerging from the column enter the column head and condenser which should be large enough to condense all the vapours with 1/3 of its cooling surface. The remaining 2/3 of its cooling surface is a necessary reserve for emergency situations, overheating, lower water pressure etc. Design conditions should take into account such a necessary safety margin.

From the condenser the liquid condensate i.e. all condensed vapours is directed into a reflux system. This together with the column packing, is the most crucial factor influencing separation. The reflux system is a part of the column head which divides the condensate partially back to the column top and partially (through the cooler) to the receivers. The condensate flows to the column to enter the liquid/vapour mass exchange process. As the composition of the condensate is nearly the same as vapours on the top of the column its return to the column shifts the equilibrium and increases an amount of higher boiling component in the vapours. Thus a higher amount of the condensate coming back to the column improves separation it must be moreover considered that a high reflux ratio will slow down the process of fractionation. Similarly as for other parameters (heating rate, column height, packing type, vacuum) a compromise should be found to give the best result. Again it has to be established experimentally.

It is advisable to connect the reflux ratio controls with the column head temperature. This is important when a specific product is to be separated from the oil. At a specific point (e.g. 1° -C over b.p. of the product) the reflux system shall be closed to give "total reflux" i.e. all condensate back to column until b.p. of the products will be reached again in the column head. By this method the maximum possible amount of the product can be separated.

CONDENSATE COOLER AND DISCHARGE

Part of the condensate directed to receivers should be cooled down for easier collection and to condense any vapours which may flow through the condenser. Receivers must permit the collection of the fractions with or without minimum disturbance of the vacuum system and phase equilibrium in the column). This is described in more details under "Equipment".

It is important that an appropriate number of containers is available to collect fractions. During test runs, it is crucial to have the GLC available for immediate analysis of each fraction collected.

EQUIPMENT

GENERAL DESCRIPTION

In this chapter only a typical assembly of equipment used for fractional distillation of essential oils and related products will be described. Thus large scale continuous systems, plate columns, super high vacuum units, thin film evaporators etc. will be either briefly mentioned or neglected.

The fractionation unit itself is of course the most important part of the equipment. It must be however observed that quite a range of auxiliary equipment and accessories are an absolute necessity to run the unit. Also, an appropriate supply of water heating and cooling media and electricity shall be required. All these details will be briefly described below. Proper selection of the equipment for specific purposes will be discussed in the next chapter.

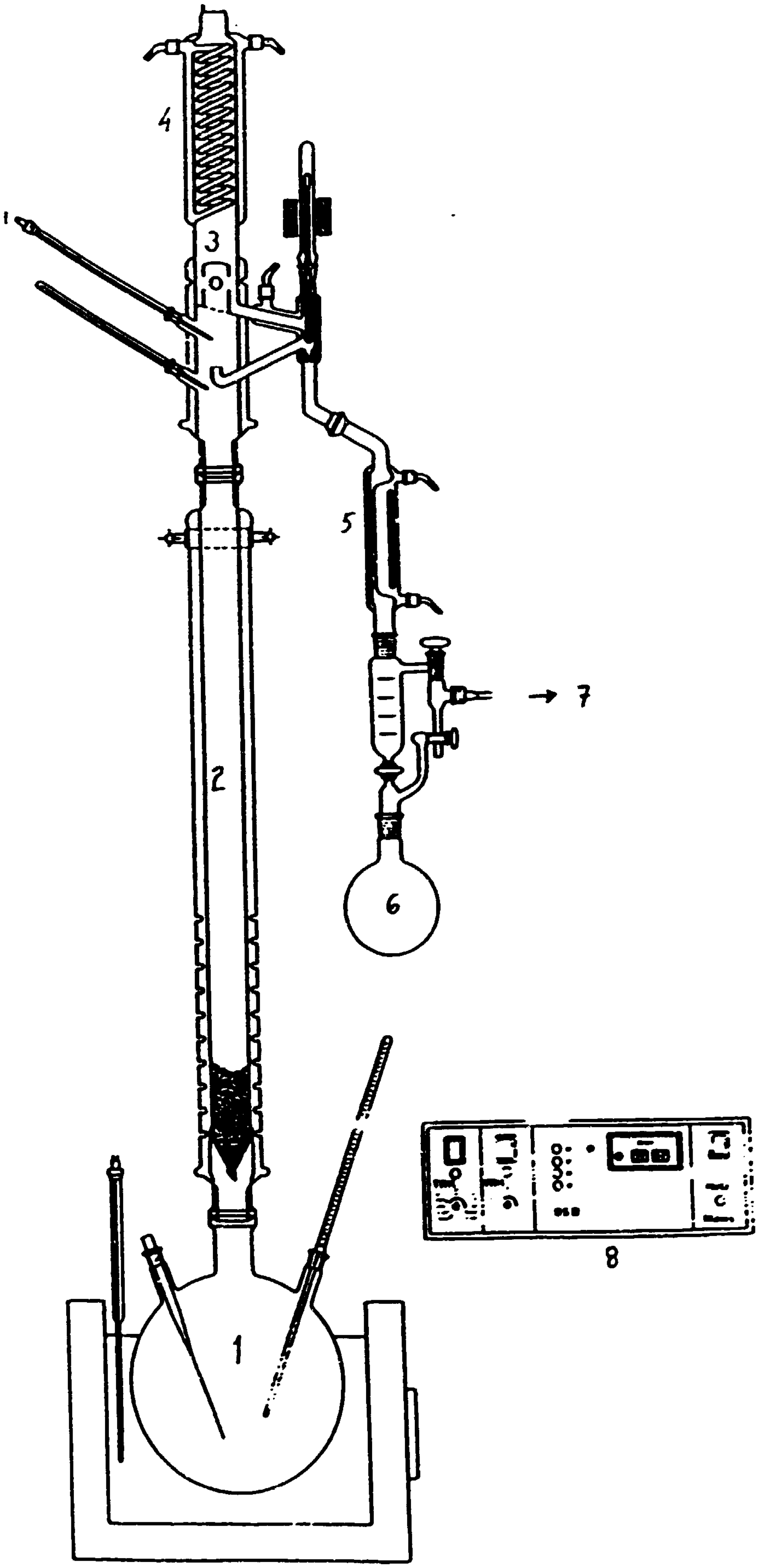


Fig. 24. A fractionation unit

A fractionation unit consists of the following main parts (see drawing-Fig. 24).

1. Evaporator with heating system.
2. Fractionation column with packing.
3. Column head with reflux system.
4. Condenser
5. Cooler
6. Receivers
7. Vacuum system
8. Controls

EVAPORATOR AND HEATING SYSTEM

In most cases fractionation of essential oils is performed in batches and there are very few continuous units used for this purpose (e.g. turpentine fractionation in production of aroma chemicals). Therefore, the evaporator (boiler, distilling tank, distiller, still, distilling apparatus, distilling vessel, distilling flask) is usually either a round-bottom multineck glass flask for laboratory of pilot plant scale (up to 100 L) or high class stainless steel pilot plant or production scale stills. Steel units are of different shapes and construction; usually round bottom cylinders with removable cover with all necessary connections for column, charging pipe, control equipment, inspection windows etc. A stirrer mechanism is also installed if necessary (preferable turbine or propeller type). Rounded ended cylinders can be also used. A bottom discharge valve is necessary. Sometimes glass line steel units are used combined with glass lined or glass column and other parts. In such cases very high quality glass lining must be used.

Numerous heating systems are used for fractional distillation of essential oils. Laboratory glass units are commonly heated with the use of electrically heated oil or water bath preferably with electric heating mantle with automatic control of heating rate.

No open fire (gas burners) shall be used for distillation of essential oils due to their inflammability.

Pilot plant and production scale units are constructed with a variety of heating systems with the use of water and stem of oil as heating media. Heating mantle or welded semicoils are the most commonly used constructions.

Immersed (internal) heating coils are very efficient but cause problems in cleaning due to the usually hard residues (polymers) left from essential oils. Sometimes heat exchangers are used in tubular horizontal evaporators where heating coil or a multitubular exchanger is constructed in a form with removable parts for easy cleaning.

There is no need to use stirrers in laboratory and even bench-scale or small pilot plant units, but it is advisable to have them in production stills especially with a capacity over 500 l.

The most efficient, safest, giving design which ensures a quality product, are systems with external, flow through a multitubular heat exchangers used as heaters, where contact of the oil with heated surface is reduced to a minimum. These systems (manufactured e.g. by Sulzer) as well as energy saving systems are however most expensive and are advisable for manufacturing purposes in industrial ventures.

FRACTIONATION COLUMN WITH PACKING

As a rule, the fractionation column is a tube, glass or stainless steel of one or more pieces, insulated and filled with packing material which is the most important element of fractionation system.

There are many different packing materials used in fractionation columns varying in their separation efficiency, pressure drop, process rate, material and of course cost. Best known are various rings with Raschig rings as they are easiest to obtain or manufacture. These are rings (cylinders) with height equal to diameter made of glass, ceramic material or metal. The easiest to produce are glass Raschig rings which can be made by simple cutting of glass tube with appropriate diameter. Paul and Bialecki rings are a variety of Raschig rings with protrusions inside the rings and holes in the walls.

These are made of metal or ceramic material. Dixon rings are similar to the above in shape but fabricated from wire mesh and have larger exchange surface and low pressure drop. Fenske helices are glass coils. Cut metal springs (curls) are also used for that purpose. All the above described packings are loose in columns. Therefore, a special support (in long columns) and also liquid distributors are installed in the columns to keep the packing in place and to distribute any returning liquid uniformly over the packing surface so as to prevent formation of separate channels for liquid and vapours. There are many other types of loose packings but they are very seldom used for fractionation of essential oils.

Other commonly used column packings for fractionation of essential oils are packet types usually manufactured from highly resistant stainless steel wire either in the form of wire cloth rolls or wire gauze formed in special shapes and packed into the column in a special way. They direct both vapours and liquids to achieve maximum exchange surface i.e. best possible separation. These systems of the highest class for all sizes and types of columns are manufactured by Sulzer in Switzerland.

The efficiency of packing within a column can be expressed in HETP (Height Equivalent of Theoretical Plate) in cm per theoretical plate. The column efficiency will be expressed in number of theoretical plates. It should be emphasized that packing or column efficiency is calculated for standard test mixture at specific rates of fractionation. Therefore the HETP value has only an indicative meaning for comparison e.g. 1 cm HETP packing will be twice more efficient than 2 cm HETP packing in the same column, for the same product, in the same conditions. In the same manner two columns ready made with packing can be compared by the number of theoretical plates given in the specification.

COLUMN HEAD WITH REFLUX SYSTEM

Another most important part of the fractionation unit is the reflux system i.e. the mechanism of partial or total return of condensed vapours back to the column. There are two basic methods of reflux:

- Continuous, where the condensed vapours are separated into two streams - to receiver and back to the column and the volume of these two streams is regulated.

- Periodical, where by a valve system condensed vapours are directed to the receivers or back to the column and the proportion of the volumes is regulated by a timing device in the system (Valve open or closed, funnel directed to appropriate outlet etc.)

The above two systems are used when vapours coming out of a column head are totally condensed. In some units reflux can be achieved by partial condensation of the vapours (size of condenser and cooling rate will regulate reflux). This system is however very difficult to control so in modern columns total condensation is used (see below).

CONDENSER

There are many designs of construction of the condensers. In glass units it is usually glass coil in the tube where cooling water is inside the coil and vapours outside. Stainless steel units may have a similar construction (very efficient) or be multitubular heat exchangers with cooling water inside the tubes and vapours outside or vice versa. According to the construction of the condenser vapours and cooling liquid can flow concurrently or countercurrently. The former is usually more efficient for total condensers.

DISTILLATE COOLER

Although the basic condenser shall be sufficient for total condensation of the vapours, the condensed liquid, especially for higher boiling fractions is warm (near to boiling point). Therefore the tubing between condenser outlet (after reflux system) and receiver is usually constructed in the form of tube in tube (liebig) cooler with condensed liquid inside and cooling water countercurrently outside (coldest point at outlet).

RECEIVERS

As fractionation of essential oils is usually carried out under vacuum two receivers are necessary to provide possibility of discharge of fractions without or with minimum disturbance of the vacuum in the system. Volume of the main receiver should be around 30-50 per cent of the working volume of the evaporator. The intermediate receiver (or second parallel receiver) shall be min. 5-10 per cent of the working volume of the evaporator (this is usually the volume of interfraction).

VACUUM SYSTEM

The most important part of the system is the vacuum pump. Other parts are tubing, connections, valves and controls (see below). There are three main types of vacuum pumps - piston, water or oil rotary, and steam or water jets. High vacuum (1 mm Hg or less) can be achieved with piston and rotary oil pumps or steam jet. Lower vacuum (20-50 mm Hg) can be achieved with the use of water ring pumps (production and pilot plant scale) or water jet in a laboratory. More details on parameters and applications of the pumps are available from the manufacturers.

As stable low vacuum is an important factor of the process, it is necessary to use proper system of connections, valves, sealing, etc. Tight ball valves are best in steel units and teflon sealing are recommended. High quality glass joints and valves should be used in glass equipment. When flexible tubing is necessary, oil, resistant vacuum, reinforced rubber or plastic (teflon) tubes should be used, tightly connected with the unit and the pump. High class silicone grease should be used for lubrication of the glass joints and valves

For the safety of the pump and total condensation of all the components, cold traps (cold finger coolers) should be installed between the unit and vacuum pump and cooled with solid carbon dioxide, liquid nitrogen or ice/salt mixture.

CONTROLS

The following parameters should be controlled:

a) The temperature of:
- Liquid in evaporator
- Column head at reflux level

OPTIONAL
- Heater (bath, mantle, jacket)
- The inside of the column at different levels
- Cooling liquid in condenser and cooler
- Condenser top (at vacuum connection)
- Cooler outlet.

b) The heating rate:

The steam flow and temperature or electrical heating system should be controlled manually or better even automatically by appropriate regulation systems according to the set temperature, the increased rate (in the evaporator and/or column head) or maximum temperature, or specified temperature curve (e.g. heating rate vs. pressure drop). Therefore, the heating system controls should be connected with temperature or pressure drop measuring instruments:

c) The vacuum in:

- The column head or condenser top.
- The evaporator (especially when pressure drop controls heating system)

OPTIONAL in:

- receiver
- pump inlet.

d) Cooling water flow (or temperature)

e) Reflux ratio

Measured and/or controlled according to system used (see above)

f) Emergency and safety controls and switches.

For safety purposes and in order to prevent damage to the unit and products, the following parameters should be controlled and connected with OFF switches.

- maximum temperature in evaporator with OFF switch of heater.
- Cooling water flow or maximum temperature with evaporator heater OFF switch.
- Vacuum set value (minimum or maximum) with vacuum OFF/ON valve.
- Head temperature set point with reflux CLOSE (full return) switch.

BASIC EQUIPMENT FOR FRACTIONATION OF ESSENTIAL OILS

1. Fractionation unit(s) selected according to purpose of the plant.
2. Vacuum pump(s).
3. Collection of containers for fractions - products.
4. Appliances - electricity (heating, pump power, controls and lighting). steam (if steam heating is used) cooling water.

5. Cold source (for cold traps - see above) and if necessary in hot climates water cooler for cooling system.
6. Balance for weighing of crude oil and products.
7. Discharge system (suction flask for units without bottom discharge valve).
8. Cleaning solvents.
9. Gas chromatograph with sampling equipment, integrator and computer.
10. Basic library with gas chromatograms of oils.
11. Report cabinet and report forms.
12. Safety equipment - gloves, glasses and face masks, fire extinguishers.
13. Basic spare parts for unit equipment and accessories (fuses, glass parts, valves, tubing, bulbs, basic tools).

EQUIPMENT SELECTION

Any selection of equipment for fractional distillation of essential oils must be based on the purpose for which it will be used. The scale (batch size), and the financial possibilities. Usually, when installation is considered, a multipurpose pilot plant unit should be selected, preferably all glass unit with 10-20 l evaporator (flask) capacity, an electric heating mantle, a column insulated with vacuum jacket around 1,5 - 3,0 high, a 20-40 theoretical plate efficiency, a timer controlled reflux, a coil total condenser, an oil rotary vacuum pump and a control system with heating rate including reflux vs temperature and a vacuum regulated automatically to preset values. An example of L&T Labortechnik unit is illustrated on sketch. Many other manufacturers of glass equipment can deliver this kind of apparatus according to the buyer's specification. This kind of unit can be used for research and testing as well as for scale production.

Purchase of ready made equipment manufactured by specialised company up to the specification or specified purpose is always the best solution for any kind or size of fractionation unit. However, this is the most expensive solution. In some cases, some manufacturers tend to offer more sophisticated units with numerous computerized control systems and gadgets which extend the usual needs. Considering the repair and maintenance services, especially for electronics that are usually not available, this type of equipment can be more of a nuisance than a useful tool. Therefore, it is wiser to resign to the use of available resources and purchase additional spare parts and accessories whenever required. This is especially important when such easily breakable glass equipment is used. It is also advisable to have the unit designed in such a way that parallel traditional control systems could be used if necessary (e.g. mercury thermometers, vacuometer, etc.)

With limited resources and objectives similar to the above manually controlled system, numerous simplifications can be completed from glass parts and standard control equipment. However, an efficient column, ready made by the manufacturer with specified separation capacity (Sulzer packing) should be purchased and timer controlled reflux system installed.

The above recommendations is valid for any size of multipurpose pilot plant or research unit.

With very modest resources, a self completed unit can be considered with locally available parts and equipment. There are some principles which should be observed based more on practice than on theory or engineering calculations. The remarks given below should be used only when no specialities are available to design the unit or specify the date for the workshop engineer. The data and factors given are based on the author's practice and work with many types and sizes of fractionation units from the most sophisticated, fully automatic to the most primitive self designed units used in many parts of the world.

The size of the evaporator and material of construction, heating system and cooling, are selected according to scale of the unit and materials available.

The column height (packing height) with standard Raschig rings shall be calculated at 20-100 times with a diameter of 1:50 i.e. 5 cm in diameter and 250 cm height.

Raschig (and other rings) size shall be around 0.1 of the column diameter. Columns with other types of packing may have different sizes which allows a higher fractionation rate and a better separation (e.g. Sulzer or similar packings).

Generally speaking, the column should be selected or constructed in such a way that its efficiency shall be from 5-10 theoretical plates for simple fractionation processes like deterpenation, desesquiterpenation and isolation of the main component to around 30 theoretical plates for testing units, pilot multipurpose plants, fine deterpenation, isolation of minor components, etc. A much higher efficiency is necessary for laboratory units and for analytical work i.e. for the separation of components of unknown oil for identification.

The following two examples will illustrate the efficiency of the column according to packing:

Column packing	Raschig rings	Sulzer
Column height	250 cm	150 cm
Column diameter	5 cm	7 cm
Packing size	5 mm	---
Pressure drop	5 mm Hg	2 mm Hg
Fractionation rate	2 1/hr	15 1/hr
Efficiency	20 TP	30 TP

As already discussed, the fractionation unit (and column) for specified routine work with one oil, can be designed very precisely according to theoretical data and test runs on laboratory scale. One should consider the total cost of the project. Generally speaking, the purchase of ready made unit designed and produced by specialised manufacturer is always cheaper than research engineering work and erection of the plant. The cost of possible mistakes in design or in transformation of experimental results into engineering data shall be considered. It should be emphasized once again that there is no way to design a multipurpose fractionation unit for essential oils on the basis of theoretical data by only providing such data (which is not true). Any attempt to do so is a waste of time and money. One can imagine the designing of a specific unit after a good training and experimental work has been performed with ready made pilot plant equipment. In this way good production unit for routine work with one or two oils can be designed and constructed with relatively low cost.

All other elements of the equipment (see list of "BASIC EQUIPMENT") should be selected similar to the column and packing i.e. as a compromise between needs, technical possibilities and cost.

It is however advisable for the first multipurpose unit to have:

- ready made, well insulated column with efficient packing.

- total condenser of coil (glass, metal) or multitubular (metal) type with cooling surface 3-4 times larger than calculated.

The length and diameter of the column and the length and type of packing are adjusted to capacity of the evaporator. The requested efficiency of the unit (fractionation rate, separation), cost limited and size (height) of the room where the unit will be placed as well as the general purpose of the unit (multipurpose fractionation, the testing and small scale production of selected isolates) should also be considered.

The final conclusion of the selection of various factors as well as auxiliary equipment should be always a reasonable compromise between expected results, technical possibilities and financial resources. The data and figures given below should be used as indications only for preliminary decisions. It is always preferable to take final decisions on the basis of experiments. Therefore it is most advisable to start with small size pilot plant units to test oils, technology, methods and train staff. Even ready made equipment for the purpose of production scale is a big risk without prior installation and testing of small scale equipment starting with laboratory scale.

The following remarks may be considered as basic guidance:

Full load of the evaporator in normal working conditions should be fractionated within one working day (shift) i.e. fractionation rate for the batch shall be calculated as

$$R = \frac{V}{6}$$

Where R is fractionation rate l/hr.
and V is evaporator working volume l

The above is not applicable for analytical and test units where one batch may be fractionated for a few days.

The column diameter should be adapted to the fractionation rate and packing flow-through factor. For simple mixtures, the column diameter and height can be calculated according to the requested separation level using engineering data. These are however useless when one has to work with multicomponent mixtures containing different chemical compounds (which essential oils are) and when in most cases, the same unit will be used for different oils.

For practical purposes, for columns packed with standard Raschig rings, column diameter can be calculated as:

$$D = \frac{V}{3 - 8}$$

Where D is column diameter in cm
and V is evaporator volume in litres.

This means that for e.g. a 20 l still shall be equipped with a column of 2.5 - 7.0 cm in diameter. For standard batch units, this can be applied up to 2000 L stills.

- open/close time controlled reflux system easy to regulate and measure.
- electric heating mantle for glass unit with PID system of heating rate controls and appropriate safety switches.
- steam heating for steel units with heating rate related to pressure drop in the column.
- rotary oil vacuum pump with capacity allowing to achieve 1 mm Hg in empty unit within 5 - 10 min.
- stirrer in units over 500 l.

The basic controls should allow:

- precise automatic regulation of heating rate according to the chosen method.
- set and maintain automatically vacuum value with possible manual change of set value during the process,
- set and maintain automatically reflux ratio with possible manual change of set value during the process.
- automatical switch off heating with temperature increase over limits (overheating), vacuum system malfunction, and cooling disturbances,
- measure all necessary parameters of the process especially temperature in evaporator and column head, vacuum, and reflux ratio.

SOME PRACTICAL REMARKS

1. In glass units, always use capillary tube down to the bottom of the flask and control its performance during the process. If not available, broken pieces of porous ceramic could be placed in evaporator.

2. Consider that residues after fractionation and cleaning (usually viscous or vaxy/tar type solidifying liquid) should be discharged from the unit bottom discharge valve is of course the best solution. If not available in the construction, which is usually the case in glass units, prepare appropriate set of tubings and suction bottle to withdraw the residue when warm and liquid.

3. The column and evaporator should be cleaned, especially when different oils are distilled in the same unit. Method of cleaning, solvents used, and conditions should consist the integral part of the operator's instruction.

4. When gas chromatography is used for analysis of the oil prior to fractionation, it should be borne in mind that the sequence of appearance of the oil components in the GLC detector (peak sequence) is not always the same as the sequence of the components in the fractions. Therefore, laboratory scale fractionation of the oil with GLC identification of components of the fractions can be only a source of information on identity of the components in the fractions.

5. If it is necessary to stop the process of fractionation or leave the unit after the process without cleaning, it should be considered that many components of essential oils are very active chemicals. Some of them can easily oxidize or polymerise in very exothermic reactions especially on large active surface of metallic packing. This can lead to the decomposition and carbonization of the packing which is difficult or impossible to clean and which affects the performance of the packing. In worse situations, the mentioned reactions can set fire or glow in the column which will certainly damage the packing and create danger. Never leave the column with residues of active products in it and allow air into the unit. Inert gas or steam should be used

to fill the unit. With some risk very well sealed unit (pressure loss max 1 mm Hg per hour) can be left overnight under vacuum.

6. Sealing of the unit of any size is tested with an empty unit. 0.1-1.0 mm Hg. vacuum should be easy to achieve in laboratory and pilot plant units and 3-5 mm Hg in production units. With the above values achieved, the unit should be cut off from the vacuum pump and left to stay under vacuum (preferably overnight). Vacuum loss (decrease) should not exceed 1mm Hg/hour in smaller units and 2-3 mm Hg/hour in production units.
7. Explosion proof electricity systems and very good ventilation should be installed in the fractionation room. Any spillage should be cleaned immediately. It should be known to all operators and visitors that any terpene vapours can blow in contact with electric sparks or cigarette glow. No open fire or smoking in the fractionation room should be allowed until all products are removed and equipment cleaned.

8. It is very convenient to take a GLC sample from the still during the process. According to the construction of the evaporator, an appropriate system should be installed.

9. The central electricity switch should be installed outside the fractionation room which will allow to switch the power off any time when the unit is left without supervision or in emergency situations.

10. Any training of operators and other staff should start with safety instructions. They should be familiar with all hazards of the process as well as with properties of the oils, components and auxiliary chemicals (solvents) used.

BASIC INSTRUCTIONS
FRACTIONATION UNIT CHECKOUT AND CLEANING

Checking

1. Check if any residue, water, organic liquids, etc. are in the distillation flask (still), receivers, cold finger trap or column. Remove if any - see cleaning.
2. Check all joints, connections (flanges) screws if tight, greasing of glass sealings, valves, if not blocked.

3. Check all water connections for leaks.
4. Check all electrical and control connections, no loose cable should remain.
5. Check vacuum:
   - connect vacuum pump to power
   - connect vacuum control system
   - switch on mains
   - switch on controls
   - switch off heating system
   - set automatic vacuum value at 1 mm Hg
   - switch on vacuum pump
   - slowly close the unit with appropriate valve.

Observe vacuum true value - with unit empty clean and dry, and all connections tight, after 10-15 min. according to pump capacity. Pressure should drop to a minimum of 3 mm Hg (properly 1 mm Hg). If higher, check all connections (under vacuum) for possible leaks and tighten if necessary.

If the unit is working properly, switch off the vacuum pump and slowly open aeration valve until the pressure stabilises to around 760 mm Hg. Thus the unit is ready for work.

Drying

In case there are water vapours or droplets in the unit, check by odour if no other vapours are present and proceed as follows:-

1. Follow instructions on checking unit the vacuum true value stabilises for a few minutes and a certain level (usually much higher than set 1 mm Hg) arises.

2. Switch on the heating system (low heating rate).

3. Keep on heating under the vacuum until all droplets and vapour disappear from the unit (check visually the glass unit, through the inspection window in the steel unit) and true value of the vacuum should achieve a minimum of 3 mm Hg. Unit will be ready for work once this is achieved.

Cleaning

In case there are vapour or condensed liquid in the unit which are identified by appearance or odour as organics, check in records the last batch and select a proper solvent to clean the unit (methanol, acetone - pure).

1. Check the unit as described above.
2. Place around 1/10 of the evaporator working volume of the selected solvent in the evaporator.
3. Connect all systems except the vacuum.
4. Set the heater at maximum temperature - around 20 grades higher than boiling point of the solvent used (i.e. 70°C for acetone and 80°C for methanol).
5. Set reflux at full return (close).
6. Observe boiling of the solvent and control the temperature. Keep heating until vapours reach condenser and condensate returns via reflux system. Keep boiling under total reflux for 15-30 min.
7. Open reflux and collect around 10 per cent of the solvent. Close the reflux and switch off the heating.
8. Wait around 30 minutes until boiling stops and all liquid flows down from the column.
9. Remove the solvent from the evaporator, add a small amount of pure solvent and remove again. Discharge the solvent distilled to the receiver.
10. Proceed as in "Check vacuum". With the residue of the solvent present in the unit "true value" of the vacuum will be much higher than the set point and will go down very slowly. At around 100 mm Hg set the reflux to "open" and heater "on" as for solvent boiling.
11. Continue heating under the vacuum until all the solvent evaporates i.e. vacuum will go down to 1-3 mm Hg.
12. Follow the procedures as after the vacuum check.
13. Open the inspection window (or flask neck) and smell the inside. There should be no traces of odour of solvent or oil found. If there is a distinct odour of strange organic compound repeat "cleaning" with different solvents.
14. Water can be used as a cleaning solvent. Heating should be set at around 120°C. After finishing of the cleaning procedure continue with "drying".

THE GENERAL PROCEDURE FOR FRACTIONATION CF ESSENTIAL OILS

1. Follow check and cleaning instructions until the unit is ready for work.
2. With all the elements of the unit switched off and the heating elements cold, charge the oil to be fractionated into the evaporator. Not more than 3/4 of its volume should be filled.
3. Set the unit for fractionation:-
    - close all inlets
    - turn on cooling water
    - fill cold traps with freezing agent
    - connect unit with vacuum pump
    - switch on controls
    - set initial vacuum value (around 100 mm Hg)
    - set reflux to full return (close)
4. Switch on the vacuum pump and observe the oil in the evaporator and the vacuum true value indicator.
5. If no boiling starts at 100 mm Hg set the vacuum at 50 mm Hg and continue with the observation. If boiling starts at 100 mm Hg decrease the vacuum stepwise to 50 mm Hg and stay at this point unit the boiling slows down or stops. Sometimes water appears at this moment or boiling continues at this pressure. In such cases open the reflux and distill off the water and lower the boiling fraction. Usually at conditions described above, very low boiling products go through the condenser and re trapped in cold trap only.
6. At 50 mm Hg start low rate heating stepwise up to 50 - 80°C in the evaporator. If the boiling starts and vapour enters the column up to the condenser and comes back via the reflux (closed) slow down the heating rate and after stabilisation of the temperature both in the evaporator and the column head, set the reflux ration at 1:10 and start collection of the distillate.
7. When boiling slows down and/or head temperature decreases, set the vacuum stepwise down to 40 then 20, 10, 5 (2, 1) mm Hg each time controlling the unit performance as in point 6 above.
8. At the lowest possible pressure, when boiling slows down, increase the heating rate and continue stepwise using the same manner as with the vacuum decrease above.
9. During the above operation and when fractionation is stable, the lowest possible vacuum fraction should be collected in the following manner:

- When the temperature in the column head is increasing (vacuum stable at set point) the reflux ratio should be set at 20:1 to 10:1 and fractions equal to around 1 per cent of the volume of the oil taken to fractionation should be collected (interfractions).
- When the vacuum value temperature in the column head stabilises at a certain value and boiling in the evaporator is smooth and quiet, the reflux can be changed to 5:1 and 1:1 ratio and fraction could be collected until increase in temperature to 1-2°C is observed. The collected fraction (isolated product) should be discharged and the reflux set back to 20:1 and the procedure continued.

The fraction collection is much easier if the test fractionation of the oil is done in the laboratory and the oil contents known. In such cases, preliminary fractionation instructions should be elaborated with relations to temperature, vacuum and the fraction volume.

With unknown contents of the oil, more fractions should be collected and if possible immediately analyzed by the GLC. With all the limitations described in Practical Remarks, the GLC graph can help in this procedure.

10. Discharge of the fractions should be performed according to the unit manual. It is important that disturbances in the vacuum should be minimal as any vacuum drop will affect the equilibrium in the column, thus separation.
11. Fractionation is continued as long as required according to the oil contents and aim of the process. It is not advisable to exceed 200°C in the evaporator as at this temperature some products may decompose or polymerise and create residues that are very difficult to clean. Also decomposition may form strong bad smelling products which can affect the quality of the product.
12. After the final fraction has been collected, the process shall be finished as follows:
    - Switch off the heating and wait until boiling stops and the temperature in the head and evaporator decreases significantly (around 50°C in the evaporator).
    - Switch off the vacuum pump and cut off the pump unit connection.

- Very slowly allow inert gas into the unit until vacuum true value stabilises at around 760 mm Hg.
- Discharge residue when warm.
- If possible clean column at once.

13. If the same oil is distilled in the next batch or the unit is left uncleaned overnight, fill the system with inert gas. All controls should be switched off and the unit cut off from the power. The cooling system should be also closed.

14. During the whole process, each action is noted in the records as well as each chance in parameters. If the process is running for a long time at the same parameter, notes should be taken every 30 minutes. Fractions should be numbered according to the records for easy identification and weighed. Records shall be clearly signed by the operator and the supervisor. The basic parameters to be noted in the records are:
    - time
    - temperature in evaporator
    - temperature in column head
    - true vacuum value
    - reflux ration
    - fraction collected number and weight

15. The status of each unit should be noted e.g.
    - clean
    - batch no...
    - full with ...
    - to be cleaned after batch no ....

SPECIFICATIONS FOR A BENCH-SCALE FRACTIONAL DISTILLATION UNIT CAPACITY 5 - 20 L PER BATCH

1. General description

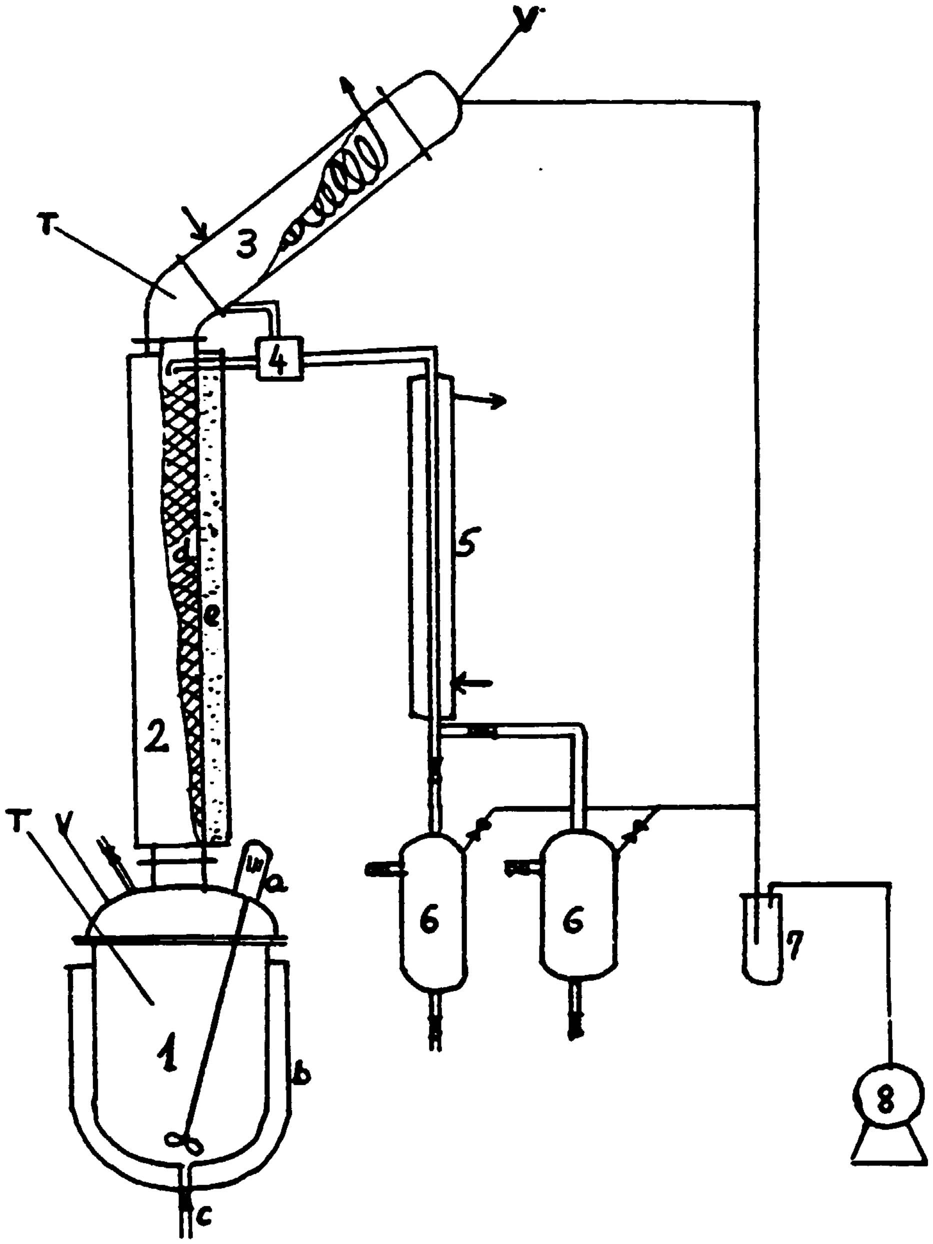
The unit is suitable for Bench-scale work, for processing 5-20 L per batch of essential oils. The reservoir (flask) is of glass and is heated by an electric mantle.

Electronic control units are used for, control of power, distillation control and vacuum control. The unit is mounted on a scaffolding.

2. Specific data

Theoretical plates:	30
Inner column diameter:	70mm
Capacity Range:	5-20L
Boil-up (maximum):	15 L/hr.
Hold-up (depending on boil up)	100 ml.
Operating temperature range:	50-275oC
Operating pressure range:	1013-1m.bar.
Power supply:	220/380V. 40-60c/s 20 amp.
Total Height of Unit:	3.5m

3. Estimated Cost Range: US$ 35,000 - 40,000



SCHEME OF VACUUM FRACTIONATION UNIT

1 - evaporator, 2 - fractionation column, 3 - condenser, 4 - reflux system, 5 - cooler, 6 - receiver, 7 - cold trap, 8 - vacuum pump

a - stirrer, b - heating jacket, c - discharge valve, d - column packing, e - column insulation

measuring points: T - temperature, V - vacuum

Fig. 25. Scheme of vacuum fractionation unit

Table 1. Boiling points of some components of essential oils

Name	b.p.°C	Pressure mm Hg
Anethol	232-234	760
	81-82	2
Anis aldehyde	258-259	760
	97-98	3
Borneol	212-213	760
Carvone	230-231	760
	104-104.5	13
	97-98	9
	88-90	4
Cinnamic aldehyde	252-253	760
	120-121	10
Citral	228-229	760
	110-111	12
	98-99	8
	92-93	5
	86-88	2
Citronellal	205-208	760
	89-91	14
Citronellol	225-227	760
	112-114	14
	108-109	12
	103-104	10
Citronellyl acetate	119-121	15
	97-98	11
Eugenol	253-254	760
	121-122	10
	110-111	5
Geraniol	229-230	760
	129-130	25
	72-73	1
Geranyl acetate	242-245	760
	130-132	22
	127-129	15
	114-115	7
Limonene	177-178	760

Linalool	199-200	760
	85-87	10
Linalyl acetate	220-221	760
	115-116	25
	105-106	11
	98-100	10
Menthol	216-217	760
	111-112	20
	105-106	15
	98-99	10
	86-87	5
Methylheptenone	173-174	760
	67-68	16
	56-57	9
Methyleugenol	248-249	760
	128-129	11
Nerol	224-225	760
	125-126	25
	103-105	9
Nerolidol	118-120	2
	96-98	0.2
Pinene alfa	157-158	760
Pinene beta	166-167	760
Safrol	234-235	760
	100-102	10
	91-92	4

Fractional Distillation of Essential Oils

Dr. C.K. Atal

Introduction

Essential oils belong to the most vital constituents of many cosmetic preparations, spices and medicinal plants. In cosmetics, essential oils are mainly responsible for fragrance, and in spices, essential oils are the principle constituents which constitute to smell taste and pungency. In medicinal plants, the essential oils are responsible for their antiseptic character. Some of the essential oils are digestive toners and curer to many ailments. Thus essential oils play a very vital role in day to day life of mankind.

Most essential oils occurring in nature consist of mixtures of hydrocarbons like monoterpenes, sesquiterpenes, oxygenated compounds like alcohols, esters, ethers, aldehydes, ketones, lactones, phenols and small percentage of non-volatile solid residue like paraffin waxes etc. Out of these the oxygenated compounds are the principal odour carriers. They are more stable against oxidizing and resinifying atmosphere as compared to other constituents. The other constituents like monterpenes and sesquiterpenes which are less stable due to their unsaturated character have tendency to oxidise or resinify easily under the influence of air and light or under improper storing conditions and spoil the odour and flavour of oil, thereby degrading the quality of oil. The constituents responsible for degrading the oil i.e. terpenes if removed from the oil give an oil called terpeneless oil which mainly consists of oxygenated compounds. They are much stronger in odour and retain most of the odorous and flavour characteristics of the original oil. The separation of terpenes from the oils or concentration of oils in terms of oxygenated compounds is accomplished by subjecting the oil to fractional distillation. Similarly if essential isolates which are also called aromatic isolates are to be isolated from [illegible] r their further use in making synthetic or natural perfumery materials, the oil is subjected to fractional distillation. For instance, isolates like citronellal, citronellol, geraniol, and their esters present in the oil of Java citronella can be separated. Citral can be isolated from Lemon grass oil and eugenol can be separated from clove oil. Thus wherever separation of oils into its constituents is required fractional distillation becomes a very important tool. In other words fractional distillation is a backbone of essential oil industry.

FRACTIONAL DISTILLATION

By expression fractional distillation is meant the process of separating so far as it may be possible, a mixture of volatile feed into its constituents by causing the mixture to vaporise by suitable application of heat, condensing the vapours in such a way that fractions of varying boiling points are obtained, re-vaporising these fractions and separating their vapours into similar fractions, combining fractions of similar boiling points and repeating until the degree of separation desired is finally obtained. Such a process is quite laborious and a time consuming operation but it is possible to carry on fractional distillation by means of certain mechanical devices which eliminate almost all this labour and time and which permits separation not only equal to those obtained by the more tedious process but for surpassing it in quality and purity of product. This mechanical device is called a fractionating column which consists of a reboiler for heating the charge, a column in which separation of more volatile and less volatile components take place, a condenser for condensing the vapours and a reflux dividing system for sending a portion of the condensate to product receivers and remaining condensate back to the column as reflux.

The operation of fractionating column can be made clear if we consider what happens after the distillation has started. For simplification no distillate is removed at the top of the column. The whole condensate formed in the condenser is fed back at boiling point to the column as reflux. It is further assumed that the column is under isothermal conditions. At first the vapour shall reach the condenser unchanged. The condensate formed in it by total condensation has the same composition as that of vapour rising form the still into the column. Since the reflux being at the initial boiling point it will have a lower temperature than the vapour, on account of this difference in temperature the less volatile component in the vapour stream shall get condensed and shall run along with down stream reflux. The latent heat of condensation of less volatile component in the vapour shall get utilised in vaporising more volatile component in the reflux which shall be taken up by the vapour stream. As a result of this change there will be at any crossection of the column a concentration difference between liquid and vapour. The vapour being richer in more volatile component and the down stream reflux being richer in less volatile component. However the total matter carried upward by the vapour stream per unit time and the amount of matter carried down by the liquid stream will not be appreciably affected by exchange. Therefore a net transport of the more volatile component will take place through a vapour stream to the top of the column and a net transport of heavy component through the reflux stream into the still. In order to achieve this heat and mass transfer within the column there should be enough contacting area for the vapour and liquid to come into contact. This is achieved by providing either some sort of trays in the column through which the vapour and liquid mix intensely and these are called tray columns. The turbulant mixing may also be achieved by providing some small irregular solids that when packed as a random bed leave irregular passage for counter current travel of vapour and liquid. These irregular solids are called packings and the column using these packing is termed as packed column.

The fractional distillations process can be divided into two categories:-

1. Continuous fractional distillation.
2. Discontinuous or batch fractional distillation.

The continuous process takes the material to be distilled continuously at a uniform rate and deliver the products of the distillation continuously. It has the advantage:

1. That the concentration of components remain the same at any point in the system.
2. It can handle unlimited amount of feed and deliver products of constant composition.

However this process needs:

1. Constant composition feed.
2. (n-1) Number of columns for a feed mixture of n number of components.
3. Heavy initial investment on column and column controls.
4. Uneconomical on limited feeds.
5. Any variation in feed composition can alter the design of the column to get desired product composition and output. This process however is best suited for petroleum industry.

6. Continuous columns when designed for a particular product and for a particular capacity frequently must undergo considerable rearrangement in order to use for other purposes.

The discontinuous or batch process takes the material to be distilled in batches, each batch being completed before the next is started. The disadvantage of this process is that:

1. It can take up limited feeds.
2. Since the concentration changes progressively with the distillation it needs change of reflux ratio.
3. It needs more energy per unit volume of distillate.

The discontinuous type is however much more popular from the continuous in great many industries especially in essential oil industry because:

1. It is frequently possible to adopt a discontinuous still designed for one purpose for use with other materials with a considerable degree of success.
2. The discontinuous stills are of simpler design and construction, considerably less expensive to fabricate and instal.
3. More suitable for small scale operations where quality and quantity of the materials to be distilled are likely to be subject to frequent change.
4. Sharp distillate cuts are possible in discontinuous still.

Keeping in view the advantages of discontinuous process the essential oil industry by and large is adopting this process. Fractional distillation process can further be divided depending on the mode of operation:

1. High pressure fractional distillation.
2. Atmospheric fractional distillation.
3. Sub-atmospheric fractional distillation.

Most of the essential oils are fractionated under sub-atmospheric pressures. This is done because most of the essential oil isolates are of the increasing molecular weight and complexity of molecular structures are being produced and these products become more difficult to purify because of degradation or loss of odour at elevated temperatures. The thermal hazards can be reduced by either reducing operating pressure and hence the temperature or by reducing the material residence time in the reboiler.

Although the reduction of pressure and reduction of residence time are of equal importance in reducing thermal degradation but there are many practical difficulties in reducing residence time and in order to retain the basic simplicity of reboiler design most developmental work has been carried on achieving a low pressure at the column head and low pressure drop along the column. The former can be calculated but the later is the function characteristic and loading of the column packing and operations conditions.

Fractionating column

Fractionating column can be divided into two main categories:

1. Tray column
2. Packed column

Tray Column

The tray column may use bubble caps, sieve plates, valve plates and are designated as per the tray shape like bubble cap column, sieve plate column, valve tray column.

The tray columns are very efficient because of highly intimate contact offered by trays to the upgoing vapour and down coming reflux.

Their main disadvantages are that:

1. They offer more pressure drop.
2. They cannot handle viscous and corrosive materials.
3. Some portion of the material is constantly held by the trays which is subjected to thermal degradation.
4. More expensive.
5. Not suitable for foaming materials.
6. More column hold up.

Therefore these columns are not suitable for heat sensitive materials.

Packed column

Packed column use packing material. These are more popular in laboratory pilot plants and essential oil industry because of the fact that these columns are used for distilling corrosive and viscous materials, close boiling materials which require very large number of equilibrium stages and viscous materials that cannot be handled in tray columns. They are also used in situations where low pressure drop and low column hold ups are important such as when handling heat sensitive materials.

The basic unit consists of:

1. Reboiler
2. Column
3. Column packings
4. Packing supports and distributors
5. Condensers
6. Reflux divider
7. Receivers
8. Vapour traps
9. Vacuum system

Reboiler

This is a cylindrical shell either vertical or horizontal in shape made out of mild steel, stainless steel, aluminium or any other alloy depending on the corrosive nature of feed. The function of the reboiler is to hold the charge to be distilled to bring it into contact with the source of heat, so that it may be vaporised to permit disengaging space for the separation of vapour from the liquid and to act as a receptacle for the less volatile residual product of the distillation. On the smaller reboilers the heating is frequently done by means of a jacket surrounding the lower portion of the kettle on larger vertical and on all of the horizontal types of reboilers the heating is done by means of internal coils.

The reboiler is fitted with connections for introducing the charge, for dumping the distilled residue, for the passage of the vapour from the reboiler to the fractionating device and for the return of the reflux from the fractionating device to the reboiler. The reboiler is also fitted with accessories such as thermometers, gauge glasses for determining the level, vacuum gauges, light and sight glasses, manholes and other necessary accessories.

The size of the reboiler is function of the amount of material to be handled per charge, the time in which each charge is to be distilled and the behaviour of the charge during distillation so far as foaming and excessive entrainment may occur.

Column

It is usually a tall cylindrical shell made out of mild steel, stainless steel, aluminium or any other alloy depending on the corrosive nature of the material to be handled. This is filled with disturbing material which breaks the descending current of liquid up into thin streams and brings the ascending current of vapour into intimate contact with these streams. The column may be an integral shell or in sections joined together with the help of flange joints. These columns may be as big as 180 cm or more in diameter and 30 meters or more in height soaring in the sky.

Their height and diameters are decided by the sharpness of separation required, amount of material to be handled and the nature of packing material used.

Column packings

The packing is the heart of the performance of a fractionating column. Its proper selection entails an understanding of packing operational characteristics and the effect on performance of the points of significant physical difference between the various types. Any of the available packings will usually perform the operation of another. The difference being in efficiency of intimate contact expressed as HTU, H.E.T.P. or Kga and pressure drop for the particular packing on fluid system. Therefore system data is very important and helpful in selecting a packing. Cost of the packing and its effect on the system cost must be considered as some packings are much more expensive than others, yet produce very little improved performance. Therefore while selecting the particular packing material, the following points are kept in mind:

1. It should have larger surface area per unit volume.
2. Larger percentage voidage to give greater throughouts.
3. Low pressure drop.
4. Easy to replace.
5. Low density but good crushing strength.
6. Easy to clean.
7. Available in varied material of construction.
8. Higher loading limits.
9. Cheaper in cost.

The most common packings and hence the ones with the most available data are:

1. Rashing rings
2. Pall rings
3. Partition rings
4. Lessing rings
5. Berl saddles
6. Intlox saddles

The other special type of packing are:

1. Panapak
2. Steadman
3. Hyflux (wiremesh packings)

Rings

Rings are hollow cylinders with height equal to diameter. When dumped into a bed they leave zig zag passages for vapour and liquid travel. Rings range in size from 0.63 to 15 cm, because both surface and open areas (which effect throughout) increase as the thickness is reduced. Walls are made as thin as possible consistent with crushing strenth requirements. Rashing rings are most popular type, usually cheaper per unit cost but less efficient than others. These are available in widest variety of materials to fit in service and are very sound structurally. They are usually packed by dumping wet or dry.

These produce considerable side thrust, on columns, have higher pressure drop per unit height of packing and poor contact efficiency which necessitates use of higher temperature in the reboiler. This restricts their use where heat sensitive materials are to be frationated.

Lessing rings slightly better than Rashing rings because of their better vapour liquid contact but offer slightly higher pressure drop. Cross partition rings are usually used stacked and as first layer on support grids for smaller packings above. Pall rings are of modified design among rings and differ in that their cylindrical walls are not solid. Openings are stamped into the wall of metal rings, the stamping being bend inward to provide additional surface. These have lower pressure drop. lower H.T.U., higher flooding limit, good liquid distribution and high capacity. These are available in metal, plastic and ceramics. Hydrodynamic studies of liquid flow show that at similar loadings the pressure drop offered by metal pall rings is about four times lower than that offered by smaller size Rasching rings. Also the efficiency of the packing as considered by the gas phase mass transfer coeffiecient obtained is 50-60 % higher than the Rasching rings.

Saddles

There are two types of saddles, Brel saddles and intlox saddles whose size range from 0.63 to 3.79 cms. Berl saddles are more efficient than Rasching rings in most applications but are more costly. They have lower H.T.U. values and pressure drop as compared to Rasching rings.

Intlox saddles are one of the most efficient packings but more costly. These have very little tendency or ability to nest or block areas of bed, gives fairly unifrom bed, higher flooding points, lower pressure drop and lower HTU than Rasching rings or Berl saddles.

Panapak and Spraypak

These differ from other packings in that they share some of the characteristics of sieve trays. These are made up of corrugated sheets of expanded metal so arranged that they appear as diamond shaped cells. Panapak and spray pack promote uniform distribution of liquid and vapour regardless of the diameter of the column because their open area for vapour flow are three to five times greater than that of bubble cap or sieve trays. Their better throughput, higher contacting efficiency and lower H.T.U. and lower pressure drop make them as efficient as bubble trays but they are used in towers 60 cm dia or more.

Steadman packing

This is available in metal, only used in batch and continuoos distillation in small diameter columns not exceeding 60 cm. This has best fractionating ability per unit height.

Goodloe packing

This is made up of pads which fit snugly in the column and each rests directly on the lower one. Thus Goodloe packing fills the packed section of the column completely. Metallic Goodloe packing is made by knitting 12 strands of 0.01143 cm diameter wires to form a tubular strand which is then flattened. The flattened tube forms a double thickness ribbon 10-15 cm wide which is crimped, the crease being 60° angle to the centre line. Two such ribbons so placed that their creases cross each other are then rolled to form the cylindrical pad.

Goodloe packing is close to bubble cap column in capacity and almost equal to steadman packing in contacting effectiveness. In India, it is manufactured under the name Hyflux.

The other packings which are of very high efficiency include gauge type (Macmohan, Dixon, Board) cannon protruded metal and helix packings. These are very expensive with very low throughputs and hence mostly used in small laboratory columns.

Packing support

Movement of bed could cause particles of packing to break. Packing supports are placed on the top and bottom of beds of random packings. Such a support should be heavy enough to prevent movement of bed but not so heavy to crush the packings. The opening in the plate should be sufficient to permit free passage of liquid and vapour while preventing packings to fall into the reboiler.

Liquid Distributor

Liquid distribution plays an important part in the efficient operation of packed column. A good packing from the process point of view can be reduced ineffective by poor liquid distribution across the top of its upper surface. Poor liquid distribution gives rise to liquid channelling and also produces less effective wetted packing area. This decreases the contact area between the vapour and the liquid thereby reducing the separation efficiency. Liquid distributors of proper design are provided in the column at a distance of three meters to achieve uniform flow of down coming liquid and proper wetting of the packing material. These are made out of stainless steel, mild steel, aluminium or any other alloy. The factors influencing condenser design are:-

1. The amount of heat which should be removed by the condenser in condensing the vapour.
2. The amount of vapour entering the condenser.
3. The amount of inert gas or other no-condensables present in the vapour.
4. The temperature of the vapour entering the condenser.
5. The temperature of the condensed vapour leaving the condenser.
6. The amount of cooling water available.
7. The temperature of cooling water available.
8. Temperature at which cooling water should leave condensing system.
9. The velocity of vapour.
10. The velocity of cooling water.
11. The physical characteristics of the condenser vapour.

The cooling surface area of the condenser is function of overall heat transfer coefficient, heat load and logrithmic mean temperature difference.

$$A = \frac{Q}{U \times Tm}$$

Where A is cooling surface area.

Q is total heat load

Tm is logrithmic mean temperature difference.

For a given heat load, the cooling area A is calculated by calculating U & Tm with the help of process conditions available.

Reflux Divider

This is a device by which reflux ratio of the column is set. This is done by providing three way solenoid valve at the outlet of the condenser. The outlets of the valve are connected to reflux inlet at the top of the column and to product cooler which in turn is connected to a programme timer which opens and shuts the reflux port and the product port depending on the time cycle adjusted on the timer. This way a very efficient and variable reflux control is achieved.

Receivers

The product receivers are two small vessels made out of metal and connected to the outlet of the condenser through product cooler. The two vessels are so connected that one can be isolated from the other without disturbing the vacuum in the system. The vessels are connected to the vacuum pump through vapour traps.

Vapour Traps

These are very efficient coil type condensers in which the non-condensable vapour and water vapour are trapped in order to prevent their entry into the vacuum pump thereby saving the pump from damage. In the traps, the vapour is in the coil and the coolant usually brine mixture is circulated in the jacket.

Column Design

To design a fractionating column, two things are of utmost importance:

(a) The packed height of the column.

(b) The diameter of the column.

In order to determine the packed height and the diameter of the column, the following factors are taken into consideration:-

1. Nature of feed to be fractionated.
2. Amount of feed to be fractionated.
3. Time for completion of fractionation.
4. Feed composition.
5. Number of fractions present in the feed.
6. Degree of separation.
7. Boiling point difference or the relative volatility of the components.
8. Vapour equilibrium data.
9. Permissible pressure drop in the column.
10. Minimum wetting rate.
11. Permissible column hold up.
12. Steam pressure in the reboiler.
13. Optimum reflux ratio.
14. Boil up rate
15. Vapour density
16. Vapour velocity in the column at flood point.

17. Physical properties of the feed like density, boiling point, specific heat, viscosity, thermal conductivity and latent heat etc.
18. Packing characteristics
19. Material of construction.

Packed height = Number of transfer units x height of a transfer unit.

Number of transfer units for a particular separation is determined graphically with the help of equilibrium data and the process conditions whereas height of a transfer unit is determined with the help of packing characterestics and the process parameters of the column.

To determine diameter of the column, area of cross section of the column is calculated as under:

$$\text{Area of cross section} = \frac{\text{Vapour load}}{\text{Velocity of vapour}} = \frac{D^2}{4}$$

Where D is diameter of the column. The vapour load and the velocity of the vapour are calculated from process conditions.

Column Efficiency

An efficient coumn is that which gives the best performance for which it is designed. An efficient column should have:

1. High separation efficiency.
2. Low pressure drop at maximum boil up rate.
3. Minimum hold up.
4. Proper instrumentation to control the operating parameters.
5. Easy to clean for using it on different systems.
6. Easy to fabricate and also cheap in cost.

Inspite of best design, the efficiency of the column also depends on the efficient operation of the column.

A typical fractionating column was designed and fabricated by Regional Research Laboratory, Jammu (India) for fractionating turpentine oil.

The capacity of the column is to process 5000 litres per batch of turpentine oil to give pure isolates of α - pinene, β - pinene and longifolene of more than 95% purity. The column has a diameter of 750 mm and packed height of 15 meters.

Figs. 26 and 27 show a fractionating column for separating Java citronella oil into monoterpenes, citronellal, citronellol, geraniol, their esters and sesqui terpenes of very high purity.

It is a 150 mm dia column with 8 meter packed height using hyflux as the packing material. The capacity of the unit is to fractionate 200 litres of oil per batch. This column has also been designed and fabricated by Regional Research Laboratory, Jammu (India).

References:

a. Ludwig, Earrest. E., Applied Process Design for Chemical and Petrochemical plants (Gulf Publishing Co., Houston, Texas) (1964).

b. Leva, M.Tower Packings and Packed Tower Design, 2nd Ed. (U.S. Stone Ware Co., Akron Ohio), (1953).

c. United States Stonware Co., The Bulletin PR-9R on the Pall rings.

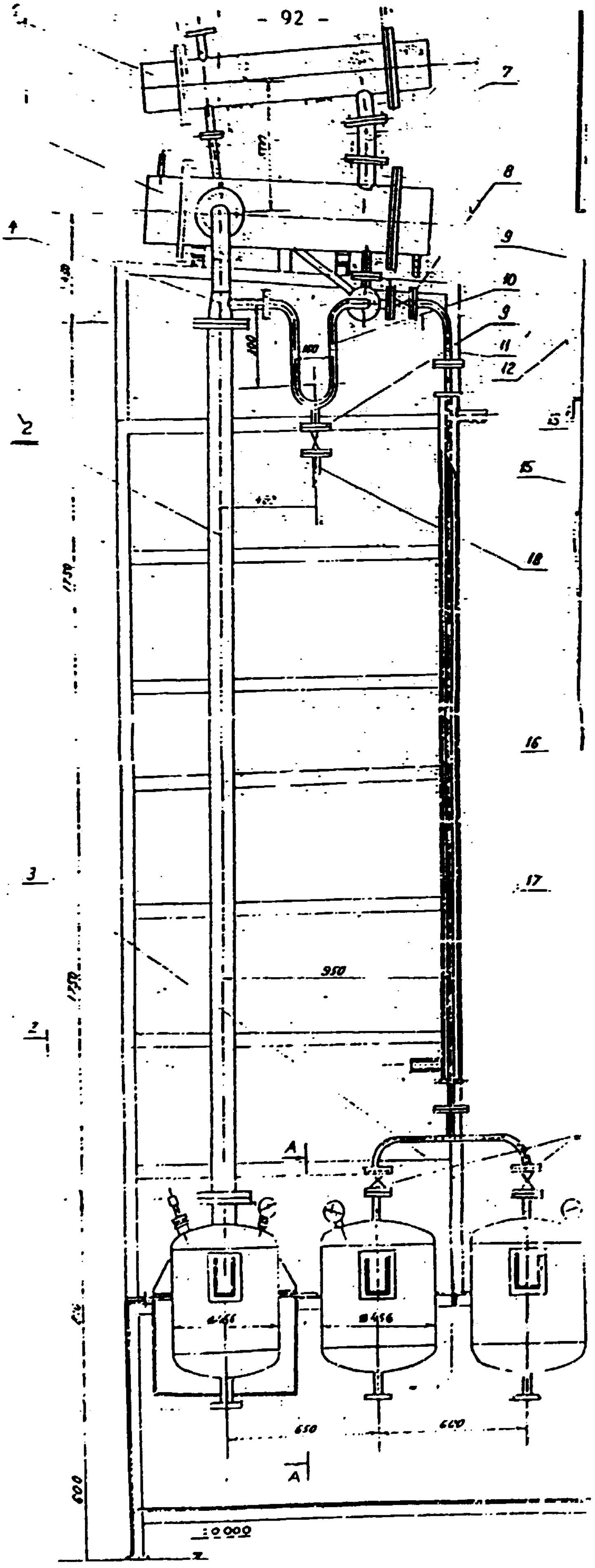
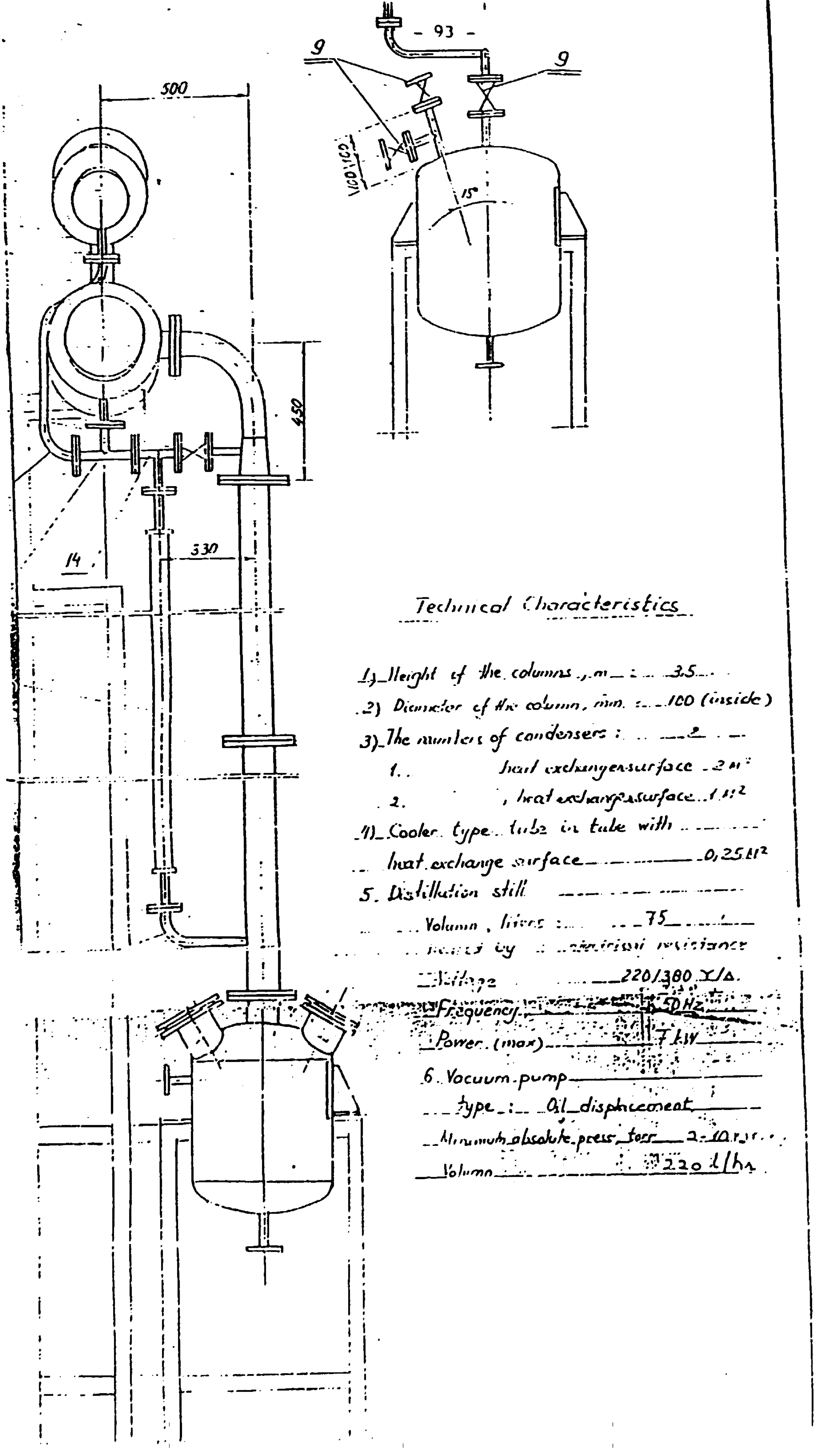


Fig. 26. A 40L cap. vacuum fractionating system (front view)



Technical Characteristics

1)_Height of the columns, m : 3.5

2) Diameter of the column, mm. : 100 (inside)

3)_The numbers of condensers : 2

1. heat exchange surface 2 m²
2. , heat exchange surface 1 m²

4)_Cooler type tube in tube with heat exchange surface 0,25 m²

5. Distillation still

Volumn, liters : 75

heated by electrical resistance

Voltage 220/380 Y/Δ.

Frequency 50 Hz

Power (max) 7 kW

6. Vacuum pump

type : Oil displacement

Minimum absolute press. torr 2-10

Volumn 220 l/hr

Turkish Rose Oil

Dr. K. H. C. Baser

Introduction

Strong and pleasant fragrance attributes of roses have been known to mankind since ancient times. Reference is made to rose fragrance in old Chinese and Sanskrit texts. It is widely believed that the so-called "Rose Oil" mentioned in ancient texts was prepared by macerating fresh rose flowers in a liquid fixed oil. This technique is known in India as "Attar". During the 4th century B.C. "Rosaceum Oleum" (Rose Oil) was prepared in Anatolia according to Hippocrate. It was merely a macerate of fresh roses in olive oil. A Similar Information was given by Dioscorides who lived in the 1st century A.D.. These are the earliest reports of rose cultivation and its use in Turkey (1,2).

Ibni Haldun mentioned the production and use of rose oil and rose water for the first time in his books. He reported that the best quality rose oil was obtained by distillation. The distillate waters were used as rose water which was traded as an important commodity in the 8th and 9th centuries. It was imported to India and China (3,4).

Although no definite information exists, the production of rose oil by distillation probably originated in Iran. About the 17th century, rose cultivation had spread from Iran to India, northern Africa and Turkey. Katip Çelebi mentioned in his work that, in the early 17th century, cultivation of roses existed around Edirne in Thrace, that is the European portion of Turkey and rose water in Istanbul in the mid 17th Century (5).

Towards the end of the 17th century, rose cultivation was introduced by a Turkish Merchant in Bulgaria which was, then, a province of the Ottoman Empire. Soon after, Kizanlik, Eski Zagra and Karhova regions became a World Centre for the cultivation of rose and the production of rose oil. By 1750, Bulgaria had become the principal source of supply for rose oi. Rose oil was used to be produced by water distillation of fresh roses and consequent cohobation of the distillates, in open-fire copper stills. The oils collected by merchants were exported from the ports of Gallipoli, Istanbul and Izmir (6). Annual production of rose oil in the region was recorded as 1500-1800 kg in 1850s.

Although various rose species were, previously, utilized for the production of rose oil, later Rosa damascena Miller (Damask Rose, Oil Rose, Isparta Rose) became established as the only source. It is widely accepted to have originated from the hybridization of Rosa gallica L. and Rosa phoenicia Boiss. Both of them grow wild in Turkey.

During the reign of the Ottoman Sultan Abdulhamid the second, systematic cultivation of roses for the production of rose oil and rose water was initiated in 1880s in several provinces of Turkey with royal incentives. This period coincides with the fleeing of Turkish immigrants from Bulgaria following the 1877-78 Turkish-Russian war. It is believed that these Turkish immigrants brought the rose stocks along. The cultivation must have started in the early 1880s, since the first production of rose oil by distillation was realized in Bursa in 1885, and in Istanbul, the following year (6).

Nowadays, the production of rose oil and rose water is confined almost solely to the Isparta and Burdur provinces of Turkey. Hence, rose oil has been produced in Turkey for over a century. Initial production by cottage industry was largely replaced by modern Industry with the establishment of the first large distillery in Isparta in 1934.

Cultivation practices

In December and January, rows of ditches with 50 cm in depth and 50 cm in width are prepared and manured. Rose twigs cut at the soil level are placed in this ditches which are then covered with soil. It takes at least 3 years for a rose plant to attain maturity. A mature rose field normally yields 5 tons of fresh roses per hectare. However, in a well looked after field, yield may go up to 7-8 tons per hectare. A field is productive for up to 20-30 years. Once in every six years plants are cut at the soil level to rejuvenate the field. Blossomed flowers are hand picked at the early hours of the day. A skilled worker may pick about 40 kg of roses in 8 hours (7).

Objectives of the present study

Turkish rose oils have been analysed by several workers (4,8-10). For the last four years, we have been engaged in a comprehensive study of the Turkish rose oils. Some of the results were presented elsewhere (11). Here, I shall report on the results of gas chromatographic (GC) and gas chromatographic/mass spectrometric (GC/MS) analyses of the Turkish rose oils produced by peasants and the Industry, processing conditions at commercial and bench scales and an exhaustive olfactory evaluation of the entire range of Turkish rose oils. The objective of the last exercise was to find correlation between the odour profile and the GC results of the oils.

The work materials were as follows:

1. 14 village produced rose oils (12 samples of 1989 produce + one each of 1987 and 1990 blends) from 11 villages in Isparta region.
2. 20 factory produced rose oils from several factories in Isparta region (9 samples of 1990 produce; 5 samples of 1989 produce; 2 samples of 1988 produce; 3 samples of 1987 produce; 1 sample of 1986 produce).
3. 1st, 2nd and total oils of fresh flowers produced by bench scale and commercial scale water distillation.
4. 1st, 2nd and total oils of fermented flowers produced by bench scale and commercial scale water distillation.

Production

Rose oil is produced by water distillation of fresh Rosa damascena flowers.

In the village-type distillation, freshly picked flowers are loaded into 150 to 1000 liter copper or galvanized steel open fire stills, most stills are of 300 liter capacity and consist of a retort and a head. The removable spherical head is connected to a pipe which leads through a pool filled with lukewarm water to cool the condensate. At the outlet, there is a 9 liter glass-collecting flask. 10 kg of flowers and 60 liters of water are loaded into 300 liter stills and are distilled for 1-2 hours to collect 2 flasks full of the distillate (18 liters). Due to low concentration of oil in the distillate, there is no separation ca. 60 liters of the distillate obtained is redistilled to get 18 liters of distillate. The oil that floats on top is decanted. The aqueous phase is diluted with distilled water and marketed as rose water.

For the Industrial production, generally 3000 liter copper or stainless steel stills are employed. Each still can take about 400 to 500 kg flowers and 1500 to 2000 liters of warm water. The stills are steam jacketed. There may also be provision for the injection of live steam to speed up distillation. The distillation is carried out for 1.5 hours. The condenser temperature is kept at 35°C to avoid the solidification of waxes. The distillate is collected in 200 liter stainless steel florentine flasks. The oil that separates out is called "Crude oil, first oil or direct oil". Distillation is terminated when the distillate is no more bitter to taste. The overflow of the florentine flasks is collected in 500 liter tanks. These "Bottom waters" or "First water" are then pumped into 5000 liter stainless steel tanks. These are cohobated in 3000 liter stills for 1-1.5 hours to get the "Second oil", "cooked oil" or "Indirect oil". The distillate after removal of the oil is sold as rose water.

The first and second oils are filtered and kept in glass flasks in the dark. When the production season is over, all the first and second oils are mixed to yield the Turkish rose oil and packed in special 2-5 kg tinned steel containers called "Kumkuma".

Generally, 3.5-4 tons of flowers yield 1 kg rose oil. So the yield is about 0.02%.

Chemical and olfactory evaluation of the Turkish Rose oil

GC/MS analysis of the Turkish rose oil revealed the presence of 85 components of which 68 have been identified making up of 96% of the total components detected.

For comparison purposes, only percentage of certain components shall be considered. Criteria in selecting these components were based on their contribution to the overall odour quality of the oils examined and on the fact that any change in percentage of these components resulted in some kind of change in the typical olfactory character of the oil. The selection of these criteria was decided jointly with a perfumer.

Results can be summarized as follows:

Basic rosaceous character of rose oils is determined by the content of citronellol present. Citronellol content in the village oils (ranging between 25-39%) was lower than in factory oils (31-43%). Higher citronellol content leads to increasing sweetness which when combined with almost equivalent quantities of geraniol leads to strength and fortification of the body note. Conversely, when geraniol is comparatively low, the sweetness of the body note is maintained but strength diminishes. Geraniol content in village oils ranged between 20-32% against 8.5-16.65% in factory oils. In some village oils, geraniol is observed to be higher in content than citronellol. In such cases while the rosaceous character is maintained, it acquires an undesirable green grassy aspect.

In order to simplify the comparison of gas chromatographic results, citronellol/geraniol ratios of each oil were taken. This ratio was previously used in 1934 by Parry and Seager for the evaluation of the quality of Bulgarian rose oil (3). Village oils gave a ratio of 0.85-1.92 while the factory oils gave 2.30-4.84.

The basic character of rose oil, established by citronellol and geraniol is further modified by nerol and farnesol. Nerol in village oils was in the range of 7.5%-13.4% compared to 4.52%-8.89% in factory oils. Farnesol content, on the other hand, ranged between 0.43-1.32% in village oils and 0.24-0.64% in factory oils. Higher contents of farnesol lead to the establishment of a strong floral character and overall improvement of volume in the body note. Nerol adds not only to the rosaceous character but also to freshness. However, in those cases where geraniol is low, the freshness of nerol manifests itself as a slightly citrusy aspect which means that when geraniol content is high, then the combination of citronellol, geraniol, farnesol and nerol has a strong, sweet, floral, fresh, rosaceous character. This odour picture changes when geraniol is low and the citrusy aspect of nerol becomes apparent in the overall odour profile of the rose oil.

Similarly, the geranyl acetate present in the rose oil adds to the freshness and the top note in those oils where geraniol content is high. However, when geraniol is low, geranyl acetate tends to boost citrusy aspect in combination with nerol and, thus, this aspect becomes typical of those varieties of rose oils where geraniol is proportionately less than citronellol. Geranyl acetate content in factory oils (0.55-1.67%) is relatively lower than in vilalge oils (0.74-2.54%). The overall strength, sweetness and typical rosaceous character of the rose oil is determined by the content of terpenes, nonanal, linalol, citronellyl formate and citronellyl acetate. Geranyl acetate content in factory oils (0.55-1.67%) is relatively lower than in village oils (0.74-2.54%).

Total terpenes content in village oils (0.15-1.31%) was relatively lower than in factory oils (0.80-2.71%). Nonanal content was similarly lower (0.00-0.04%) in village oils compared to factory oils (0.03-0.09%). The combined content of citronellyl formate and citronellyl acetate was also slightly lower (0.19-0.34%) in village oils than in factory oils (0.24-0.54%). However. linalol content in village oils was relatively higher (0.85-2.01%) as compared with factory oils (0.53-1.27%).

Oils with high citronellol and geraniol content, but with low terpenes, nonanal, linalol, citronellyl formate and citronellyl acetate content, tend to have weak top notes relative to those oils where these components are present in higher quantities.

Citronellyl formatte and citronellyl acetate create the typical fresh rosaceous character in the top note which is boosted, in turn, by the terpenes and nonanal. The volume of the top note as well as its floral character is affected by the percentage of linalol. Higher linalol adds to the volume and floral character of the top note.

Ethanol has been accepted as a natural ingredient of the rose oil. Its content in Turkish rose oils varies within a wide range. Village oils, in general, have a low content of ethanol (0.00-1.67%), while it ranges between 0.5-5.2% in factory oils. Even though it is considered as a genuine constituent, high ethanol content should be suggestive of the use of an appreciable amount of fermented roses during the process. This is especially the case with factory oils since the influx of roses into factories is unmanageable during the peak season and the roses have to stand in bags for more than 24 hours before distillation. In village type distillation, on the other hand, roses are immediately processed. Therefore, the ethanol content is low. The presence of ethanol would enhance the top note. Higher ethanol contents would lead to stronger top notes. High ethanol content in rose oil is not desirable.

General remarks on some other constituents

Eugenol and methyleugenol would have no effect on the rosaceous character of the oils. They would have an effect only on the overall general odour picture in terms of volume strength and lift. They would impart a green spicy note. Higher quantities of these constituents would lead to some improvement in the body notes. Eugenol contents in village oils are higher (1.17-2.25%) than in factory oils (0.35-1.27%). Methyleugenol content in factory oil, however, is higher (2.27-3.10%) in proportion than village oils (1.40-2.65%). Higher percentages of cis-rose oxide lead to stronger top notes. The cis-rose oxide content in factory oils was higher (0.47-0.72%) as compared to the village oils (0.29-0/48%). This is reflected in the difference between the strength of the top notes of the two types.

α-terpineol content was almost the same both in village (0.20-0.53%) and factory (0.13-0.46%) oils. In higher percentages it increases the floralcy of the body note.

Phenyl ethyl alcohol content varied between 1.35 to 3.55% in village oils, while it was relatively lower in factory oils (1.13-199%). This component also adds to the floral character of the oil.

Higher stearopten contents would lead to dilution of the body note. According to a gravimetric method mentioned in the rose oil monograph of Turkish Standards Institute, stearopten content in village oils are generally found lower than in factory oils (12).

Very minor constituents such as damascones and some sulphur compounds detected in Bulgarian rose oil, would tend to enhance the power and volume of the top note.

Odour description of typical Turkish oils

a) Village type

i) Village type oils with citronellol/geraniol ratio more than 1:

Subdued top note. No by odours. body note sweet, rosaceous and strong. Ideally, best odour is produced when ratio is between 1.25-1.30.

ii) Village type oils with citronellol/geraniol ration less than 1: Subdued top note. Strong, sweet, rosaceous body note, but with a slightly grassy aspect.

b) Factory type

Strong. fresh top note. Overall more terpenic with rosaceous and sweet characters being slightly subdued. Body note, sweet and rosaceous but somewhat thin. This is due to high citronellol to geraniol where geraniol content is low.

The above mentioned descriptions of typical village and factory type oils have been correlated to their GC analysis picture. It is evident that the difference in the odour picture of village oils as compared to factory oils and vice versa can be explained quite clearly in terms of the difference in their analysis picture vis-a-vis the constituents that have been described before. To verify the validity of our assumption and to establish the fact that the odour is significantly influenced by these aforementioned constituents, samples of Turkish oils were selected on the basis of their analysis in such a way that they were complementary to each other in terms of these particular constituents. Upon blending these oils together in proportions which maximized the percentages of the significant constituents identified by us, we were able to create a blend of natural oils which, upon independent odour evaluation, was found to be significantly better in all its odour attributes when compared with any other sample of rose oil of any Turkish origin.

Again, after odour evaluation, the oil was analysed and the odour evaluation done prior to the analysis was found to have direct correlation with the critical components identified as having a significant effect on the odour quality of rose oils.

The case of fermented flowers

As per tradition and ideal practice, only fresh flowers should be used for the distillation of rose oil. However, in actual practice, due to large quantities of flowers that have to be handled during the season, it is not possible to limit the distillation being done only from fresh flowers. Flowers that have been picked some time ago have to be accepted by the distillation companies. These flowers are processed as and when distillation units become available during the course of the day. During this time of storage, flowers undergo varying degrees of fermentation which has a direct bearing upon the constituent of the volatile oil of such flowers. In order to find out how processing conditions affect the composition of the oils, two sets of experiments were carried out with fresh and fermented roses at bench scale and commercial scale.

At bench scale experiments, the actual practice at commercial scale was simulated with a Clevenger apparatus. Ist and 2nd oils were collected and analysed by GC. These experiments with fresh flowers were repeated for fermented flowers in the same manner.

In general, these changes can be summarized as follows:

The most significant change that occurs during fermentation is that citronellol increases and geraniol decreases. Therefore, oils distilled from fermented flowers have a sweet rosaceous note but relatively weak body strength. Also, the geranial increases while nerol and farnesol decreases. Since neral and geranial have a clear citrus odour, oil from fermented flowers tends to have a slightly citrusy aspect which depresses the rosy character and enhances the terpenic aspect. In those oils where the geraniol content is high, this citrusy terpenic aspect is manifested as freshness. The decrease in nerol and farnesol is compensated to some extent by the increase in phenyl ethyl alcohol due to fermentation. The nonanal and linalol contents increase as a result of fermentation and this adds to the strong top notes that such oils have.

Citronellol contents in the 1st oils were lower than in the 2nd oils. The increase in citronellol content was more prominent in fermented roses as indicated above. Higher percentages of citronellol in the 2nd oils may be explained in part, by the presence of lower stearopten content in the 2nd oil.

Conclusion

1. There are two types of Turkish Rose oil available, namely village type and factory type. Both have quite distinctive characteristics. But the commercially recognized Turkish Rose oil is the factory produced oil.

2. Production techniques are standardized. Variations in chemical composition is due to extrinsic factors such as altitude, climate, picking time and the degree of fermentation before distillation.

3. Odour quality of rose oils can be predicted by the examination of gas chromatogrammes. Careful blending of different varieties of rose oils can produce a rose oil which may more readily satisfy the quality requirements of a perfumer.

4. This should be the practice to be followed when evaluating any essential oil with a market potential. An essential oil manufacturer should work with a person who can do odour evaluation, as closely as possible. This way production of quality oils can be guaranteed.

Acknowledgements

My cordial thanks are due to Mr. Sudhir Jain, UNIDO expert in Perfumery for the olfactory evaluation of rose oil samples. I would like to extend my gratitute to Mrs. Mine Kürkçüoglu for conducting most of the practical work and to Mr. Zeki Konur for the generous supply of village oils.

References

1. T. Baytop, Türkiye'nin Tibbive Zehinli Bitkileri, Istanbul Univ. Publ., Istanbul (1963)
2. T. Baytop, Türkiye'de Bitkilerle Tedavi, Istanbul Univ. Publ., Istanbul (1984)
3. E. Guenther, The Essential Oils, R.E. Krieger Publ. Co., Vol.5, pp.3-33, (1952)
4. A. Gürgen, Ankara Y Ziraat Enst. Der., 6(2), 201, 304-307, (1956)
5. T. Baytop, Türk Eczacilik Tarihi, Istanbul (1985)

6. T. Baytop, The Cultivation of Oil-bearing Rose (Rosa damascena) and the Production of Rose Oil (Oleum Rosae) in Anatolia during the Ottoman Era, International Conference on Essencial oils for Perfumery & Flavours, 26-30 May 1990, Antalya, Turkey

7. O.Z. Konur, Genral Review of Turkish Essential oils Industry, International Conference on Essential oils for Perfumery & Flavours, 26-30 May 1990, Antalya, Turkey

8. M. Kürkçüoglu, MSc Dissertion, Anadolu University, Eskisehir (1988)

9. J. Garnero, G. Guichard, P. Buil, Parf. Cosm. Aromes (8) 33-46 (1976)

10. O. Anaç, Perfumer and Flavorist, 5 (2)29-32 (1980)

11. K.H.C. Baser, M. Kürkçüoglu, O.Z. Konur, The Production and Properties of Turkish Rose oil, International Conference on Essential oils for Perfumery & Flavours, 26-30 May 1990, Antalya, Turkey.

12. Turkish Standards Institute, Rose oil monograph TS 1040, September 1971

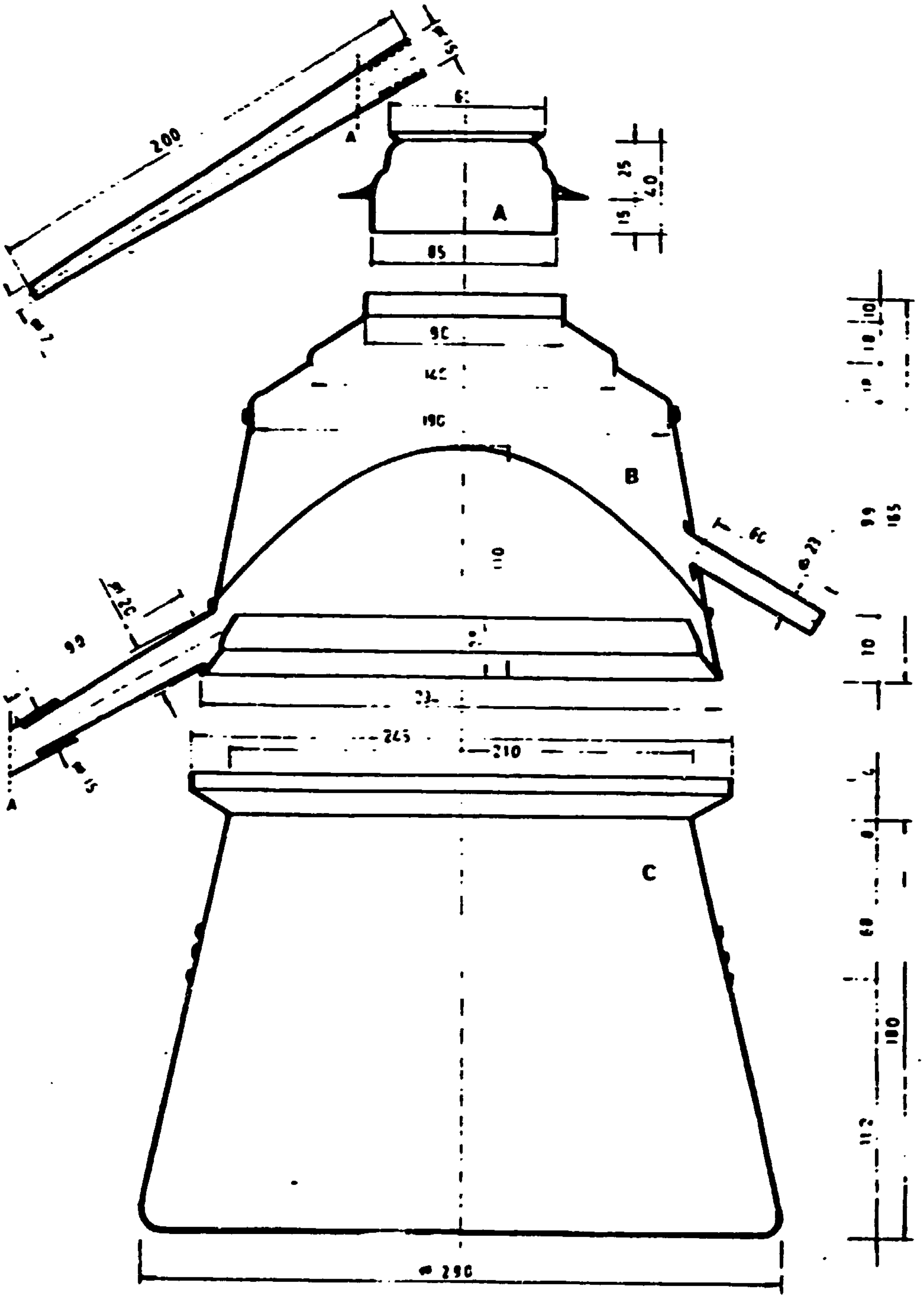


Fig. 28.Cross-section of a traditional still used in Turkey (Courtesy T.Baytop).

Table 2. Gas chromatographic (GC) / gas chromatographic-mass spectrometric(GC-MS) analyses of the Turkish rose oils

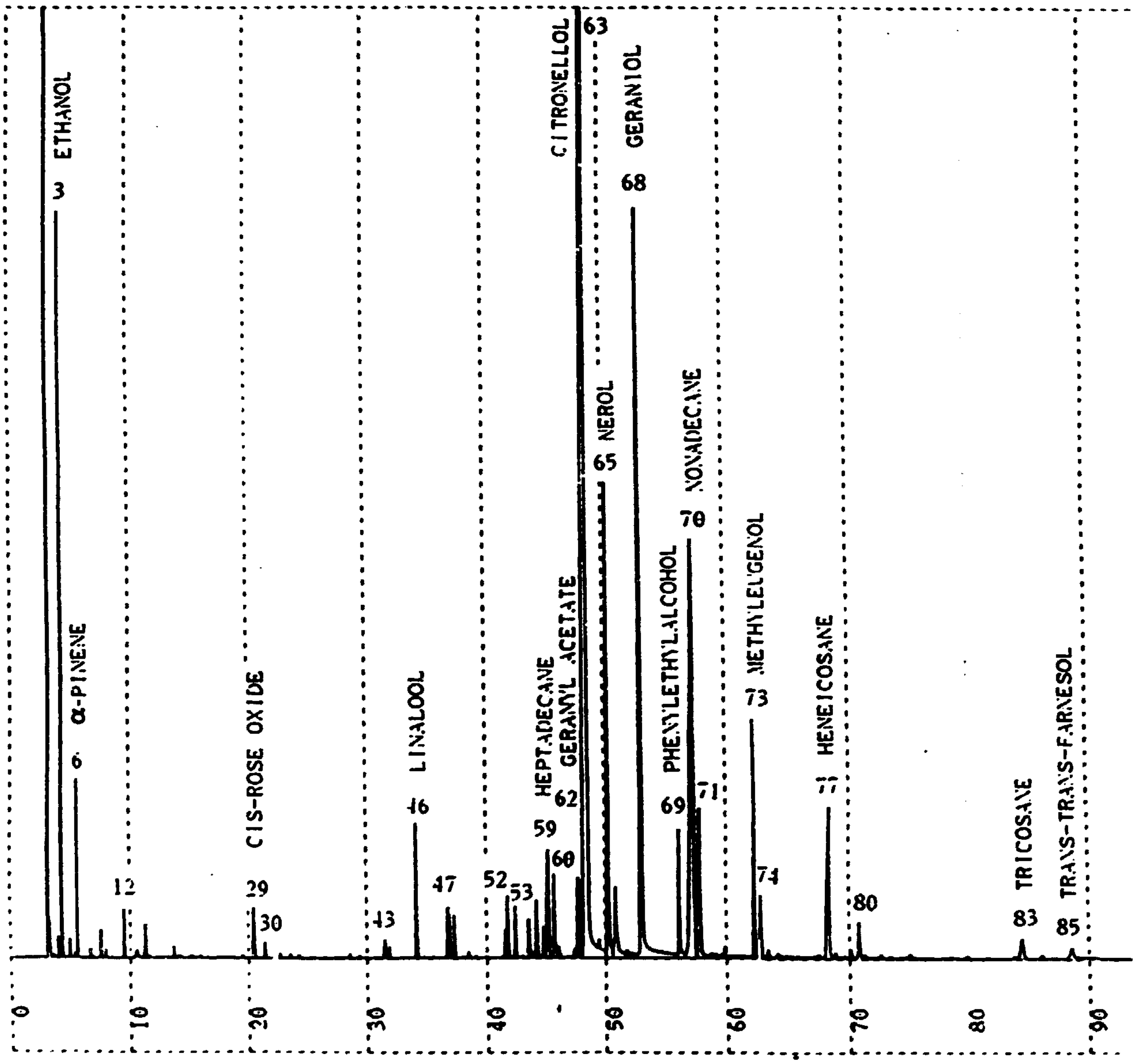
Peak no	Compounds	Z.Konur blend of village oils		Z.Konur 1989	Gülbirlik 1990	E.Erçetin 1990	M.Gürkan 1990
		1987	1990				
1	acetone	tr	0.011	0.022	0.016	0.041	0.025
2	ethylacetate	0.028	0.040	0.356	0.096	0.125	0.146
3	ethanol	0.419	0.772	5.182	2.310	2.532	4.337
4	pentanal	-	0.017	0.084	0.143	0.057	0.088
5	2-butanol	-	0.006	0.013	0.019	0.025	0.019
6	alpha-pinene	0.086	0.084	1.071	0.546	0.628	0.416
7	isobutanol	-	0.016	0.024	0.079	0.062	0.075
8	beta-pinene	0.018	0.024	0.217	0.142	0.155	0.104
9	sabinene	-	0.009	0.085	0.051	0.047	0.037
10	1-butanol	-	0.006	0.012	0.009	0.018	0.010
11	2-propen-1-ol	-	-	-	0.008	0.007	0.009
12	myrcene	0.065	0.063	0.543	0.283	0.285	0.183
13	alpha-terpinene	-	0.006	0.026	0.019	0.006	0.006
14	heptanal	-	0.035	0.146	0.203	0.075	0.125
15	2-methyl-1-butanol	0.061	0.120	0.166	0.347	0.317	0.393
16	1,8-cineole	-	0.018	0.013	0.014	0.013	0.009
17	2-hekzanal	-	-	0.011	-	-	-
18	2-amylfuran	-	-	-	-	-	0.007
19	cis-ocimene	-	0.005	0.012	-	-	0.006
20	1-pentanol	-	0.064	0.103	0.111	0.089	0.107
21	gamma-terpinene	-					
22	*t*-ocimene	-	0.008	0.036	0.025	0.012	0.018
23	p-cymene	-	0.012	0.028	0.025	0.068	0.015
24	alpha-terpinolene	-	0.006	0.029	0.019	-	0.013
25	t-3-hexen-1-ol	-	-	0.009	-	-	-
28	6-methyl-5-hepten-2-one	-	0.035	0.019	0.047	0.040	0.030
29	cis-rose oxide	0.404	0.431	0.570	0.747	0.621	0.706
30	*t*-rose oxide	0.078	0.111	0.178	0.194	0.158	0.199
31	cis-3-hexen-1-ol	0.040	0.061	0.036	0.049	0.050	0.041
32	benzyl methyl ether	-	-	tr	-	-	0.008
33	nonanal	-	0.026	0.076	0.089	0.039	0.081
38	6-methyl-5-hepten-2-ol	-	0.019	0.020	0.022	0.023	0.021
42	citronellal	-	-	-	-	-	-
43	pentadecane	0.184	0.225	0.270	0.349	0.425	0.377
45	benzaldehyde	0.034	0.053	0.048	0.108	-	-
46	linalool	1.138	1.422	0.530	1.249	1.148	0.812
47	alpha-guaiene	0.381	0.543	0.930	1.179	0.945	0.833
48	terpinene-4-ol	0.308	0.358	0.299	0.369	0.451	0.271
49	hexadecane	0.060	0.041	0.076	0.061	0.072	0.069
50	citronellyl formate	0.036	0.044	0.029	0.036	0.223	0.027
51	citronelly acetate	0.151	0.189	0.322	0.360	0.317	0.292
52	humulene	0.479	0.520	0.677	0.729	0.718	0.628
53	neral	0.367	0.561	0.423	0.536	0.457	0.723
54	alpha-terpineol	0.284	0.327	0.153	0.299	0.320	0.184
57	gamma-muurolene	0.364	0.511	0.635	0.902	0.476	0.708
58	delta-guaiene	1.647	0.214	0.361	0.525	0.387	0.349
59	heptadecane		0.822	1.642	1.079	1.449	1.179
60	geranial	1.020	0.952	0.633	0.930	1.009	0.926
61	heptadecene		0.102	0.256	0.129	0.186	0.140
62	geranyl acetate	1.519	1.598	0.923	1.215	1.194	0.668
63	citronellol	26.740	32.863	36.439	42.436	35.832	43.545
65	nerol	9.722	10.586	5.362	6.249	6.195	5.266
66	octadecane	0.813	0.231	0.158	1.255	1.027	1.210
67	2-phenylethylacetate		0.847	0.951	0.567		
68	geraniol	24.105	25.834	11.090	12.360	13.447	10.182
69	phenylethylalcohol	3.450	2.918	1.877	1.432	1.989	1.078
70	nonadecane	7.720	5.287	9.834	7.692	8.379	8.217
71	cis-9-nonadecene	2.296	1.670	2.729	2.189	2.994	2.231
73	methyleugenol	1.861	2.436	3.342	2.485	2.607	3.099
74	eicosane	2.373	0.430		0.765	0.869	0.786
75	(E)-9-eicosene	0.111	-	0.126	0.127	0.445	0.122
76	d-nerolidol	0.060	-	0.082	0.058	0.132	0.054
77	heneicosane	1.920	1.554	3.278	2.745	2.603	3.079
79	(E)-5-eicosene	0.100	0.016	0.108	0.083	0.147	0.102
80	eugenol	1.517	1.232	1.266	0.509	0.354	0.563
82	docosane	-	0.082	0.064	0.070	0.055	0.104
83	tricosane	0.329	0.206	0.688	0.500	0.408	0.623
85	*t-t*-farnesol	0.753	0.693	0.427	0.332	0.426	0.381

*Tentatively identified   tr<0.003%

Total components detected : 85

Components identified : 68

96 % of the total components detected.



Column : Thermon 600T FSC column.
(50 m.L., 0.25 mm.I.D.)
Detector : FID (GC), Mass (GC/MS)
Carrier gas : Nitrogen (GC), Helium (GC/MS)
Injetor temp. : 250°C
Column temp. : 10 min. at 70°C to 180°C
2°C/min. at 180°C for 30 min.
Detector temp. : 250°C
Split ratio : 60:1

Fig. 29. Gas chromatogram of factory produced Turkish rose oil (1989 .

Table 3. GC/GC-MS analyses of the Turkish rose oils produced by peasants and the industry

Compounds	Village oils	Factory oils
	14 samples (1987-1990)	20 samples (1986-1990)
ethanol	0.00-1.67	0.50-5.18
total terpenes	0.15-1.31	0.80-2.71
nonanal	0.00-0.04	0.03-0.09
linalool	0.85-2.01	0.53-1.27
citronellyl formate	0.19-0.34	0.24-0.54
citronellyl acetate		
alpha-terpineol	0.20-0.53	0.13-0.46
geranyl acetate	0.74-2.54	0.55-1.67
citronellol	25.04-38.97	30.67-45.83
nerol	7.50-13.36	4.52-8.89
geraniol	20.05-31.80	8.50-16.65
phenylethylalcohol	1.85-3.55	1.16-2.60
methyleugenol	1.40-2.67	1.93-3.37
t-t-farnesol	0.43-1.32	0.24-0.64
stearoptenes (Acc.to TSE)	12.20-19.83	14.00-25.96
citronellol/geraniol ratio	0.83-1.92	2.30-4.84

Tables 4. & 5. Analyses of factory produced Turkish rose oils from several factories in Isparta region.

Factory Produced Turkish Rose Oils

Compounds	Gülbirlik					Z.Kocur			Rosette 1990 blends		
	1986	1987	1988	1989	1990	1987	1988	1989	1	2	3
total terpenes	1.31	1.09	1.57	1.42	1.23	1.37	2.14	2.07	0.93	1.46	1.23
nonanal	0.05	0.05	0.06	0.06	0.09	0.03	0.04	0.08	0.07	0.08	0.05
linalool	1.20	1.01	1.21	1.21	1.25	0.53	0.78	0.53	0.96	0.86	0.61
citronellyl formate citronellyl acetate	0.24	0.37	0.38	0.37	0.40	0.27	0.34	0.35	0.18	0.33	0.37
alpha-terpineol	0.29	0.29	0.31	0.38	0.30	0.13	0.22	0.15	0.23	0.19	0.17
geranyl acetate	1.67	0.91	1.04	1.03	1.21	1.18	1.23	0.92	0.97	1.09	0.89
citronellol	35.33	42.98	41.28	38.46	42.44	30.67	38.30	36.44	45.83	40.87	40.66
nerol	7.37	5.79	5.77	5.53	6.25	5.33	8.59	5.36	7.42	7.11	6.33
geraniol	14.41	10.44	10.72	11.96	12.36	10.88	16.61	11.09	14.59	14.31	13.82
t-t-farnesol	0.60	0.50	0.37	0.38	0.33	0.58	0.40	0.43	0.35	0.39	0.43
citronellol/geraniol	2.45	4.12	3.85	3.22	3.43	2.82	2.30	3.29	3.14	2.86	2.94

Factory Produced Turkish Rose Oils

Compounds	Erçetin 1987	Erçetin 1990	Gürkan 198[illegible]	Gürkan 1990	Kınacı 1989	Tanus 1990	Şahver 1989	Başmakçı 1990	Aytekin 1990
total terpenes	1.61	1.30	1.[illegible]	0.80	1.65	1.87	1.39	2.71	1.24
nonanal	0.05	0.04	0.08	0.08	0.08	0.07	0.09	0.06	0.05
linalool	1.27	1.15	0.95	0.81	0.83	1.11	0.94	0.83	0.77
citronellyl formate citronellyl acetate	0.47	0.54	0.[illegible]	0.32	0.35	0.35	0.45	0.34	0.27
alpha-terpineol	0.46	0.32	0.[illegible]	0.18	0.21	0.27	0.25	0.19	0.19
geranyl acetate	0.8[illegible]	1.19	1.[illegible]	0.67	1.59	1.18	0.55	1.21	1.40
citronellol	41.13	35.83	33.8[illegible]	43.55	37.49	40.18	41.31	39.34	38.82
nerol	4.68	6.20	7.06	5.27	7.81	4.78	4.52	7.97	8.89
geraniol	8.50	13.45	14.71	10.18	15.49	9.43	9.61	15.16	16.65
t-t-farnesol	0.24	0.43	0.56	0.38	0.64	0.42	0.36	0.37	0.39
citronellol/geraniol	4.84	2.66	2.30	4.28	2.42	4.26	4.30	2.59	2.33

Table 6. Analyses of village produced Turkish rose oils from several villages in Isparta region.

Village Type Turkish Rose Oils

Compounds	[illegible] 1989	Ilyas 1989	[illegible] 1989	Senirce ([illegible]) 1989	Tepecik [illegible] 1989	[illegible] 1989	Ardıçlı 1989	[illegible] 1989	[illegible] 1989	Senirce 1989	Çimbilli 1989	Aşağı Çimbilli 1989	[illegible] Village blends	
													1987	1990
total terpenes	0.27	0.23	0.26	0.40	0.23	1.31	0.27	0.27	0.27	0.37	0.48	0.15	0.17	0.30
nonanal	0.33	-	0.03	-	0.03	0.02	0.03	0.03	0.03	0.04	0.04	0.02	-	0.03
linalool	0.96	0.97	0.82	1.27	2.01	0.85	1.65	1.27	0.96	1.29	0.99	1.10	1.14	1.42
citronellyl formate														
citronellyl acetate	0.24	0.23	0.26	0.23	0.19	0.29	0.28	0.29	0.22	0.29	0.25	0.21	0.19	0.23
alpha-terpineol	0.20	0.24	0.22	0.28	0.53	0.21	0.52	0.24	0.24	0.31	0.23	0.24	0.28	0.33
geranyl acetate	1.57	1.71	1.73	1.97	1.22	1.96	0.74	1.60	1.77	1.74	2.55	1.34	1.52	1.60
citronellol	26.38	33.24	29.11	29.44	27.95	25.04	25.07	32.33	38.50	28.52	26.35	29.83	26.74	32.86
nerol	8.99	10.73	11.06	13.36	11.96	10.04	9.77	7.50	8.39	9.83	11.70	8.53	9.72	10.59
geraniol	31.51	25.42	29.14	30.15	30.14	27.34	24.52	25.24	20.06	25.67	31.80	30.16	24.1.	25.84
t-t-farnesol	1.32	0.65	0.82	0.43	0.94	1.32	1.13	1.01	0.66	0.87	0.83	1.08	0.75	0.69
citronellol/geraniol	0.84	1.31	1.00	0.98	0.93	0.90	1.02	1.28	1.92	1.11	0.83	0.96	1.11	1.27

Table 7. Effect of processing conditions (fresh and fermented) on the composition of Turkish rose oils at bench scale and commercial scale.

Compounds	Clevenger				Factory				Fresh→Ferm.
	Fresh flowers		Fermented flowers		Fresh flowers		Fermented flowers		
	1st oil	2nd oil	1st oil	2nd oil	1st oil	2nd oil	1st oil	2nd oil	Trend
total terpenes	0.63	0.85	0.29	0.69	3.86	0.27	1.78	0.33	↓
nonanal	0.04	-	-	0.03	-	0.04	0.07	0.05	↑
linalool	0.24	0.55	0.56	1.06	0.20	1.23	0.40	1.62	↑
citronellyl formate									
citronellyl acetate	0.31	0.06	0.31	0.08	0.80	0.19	0.80	0.19	—
alpha-terpineol	0.67	0.41	0.16	0.28	0.03	0.03	0.05	0.35	✓
geranyl acetate	4.31	0.36	0.25	0.11	2.67	0.23	0.35	0.16	↓
citronellol	21.79	47.11	34.87	65.49	29.60	50.90	37.26	58.85	↑
nerol	8.15	7.53	6.77	4.57	2.58	3.90	1.55	5.36	↓
geraniol	16.00	16.55	11.01	7.63	4.22	7.10	2.49	8.58	↓
phenylethylalcohol	1.48	4.40	2.04	5.13	0.67	1.15	0.82	1.77	↓
t-t-farnesol	1.44	0.26	0.53	0.08	0.44	0.19	0.30	0.14	↑
citronellol/geraniol	1.36	2.85	3.17	8.58	7.01	7.17	14.96	6.86	↑

↓ Decreasing ↑ Increasing — Stable ✓ Variable

Table 8. Composition of blended village and factory rose oils.

Compounds	Blend of village +factory oils
total terpenes	0.578
nonanal	0.054
linalool	0.802
citronellyl formate	
citronellyl acetate	0.2772
alpha-terpineol	0.212
geranyl acetate	1.164
citronellol	35.645
nerol	7.785
geraniol	19.089
t-t-farnesol	0.580
citronellol/geraniol	1.870

COUNTRY STATUS REPORTS

SRI LANKA

ESSENTIAL OILS INDUSTRY IN SRI LANKA

DR. U.M. SENANAYAKE

AROMATIC PLANT RESOURCES OF SRI LANKA

Most abundant species:

(i) Cinnamon (*Cinnamomum zeylanicum* Lin)
(ii) Pepper (*Pipper nigrum* Lin)
(iii) Citronella (*Cymbopogon nardus* Lin)
(iv) Cardamom (*Elettaria cardamom*)
(v) Nutmeg (*Myristica fragrance* Lin)
(vi) Cloves (*Syzygium aromaticum* Lin)
(vii) Ginger (*Zingiber officinale* R)

Less abundant species:

(i) Lemon grass (*Cymbopogon citratus* Lin)
(ii) Eucalyptus (*Eucalyptus globulus & citriodora*)
(iii) Curry leaf (*Murraya koenigii* L)
(iv) Pinus (*Pinus carrebaeae*)
(v) *Pipper longum* L
(vi) Vetiver (*Vetiveria zizanoides*)
(vii) Vanilla (*Vanilla planifolia*)
(viii)Lime (*Citrus sinensis*)

TRADE IN ESSENTIL OILS/FRAGRANCES IMPORTS

(a) Fragrances, flavour and related products

Product (1987)*	(Quantity tonnes)	Value (Million of Rs)
Essential oils	38.6	9.8
Aromatic mixtures	268.2	74.3
Perfumes/cosmetics	3.0	8.5
Eau de cologne	18.2 (litres)	2.4
Tooth paste/shampoo	59.4	6.1
Other toiletries	23.0	9.4
Soap	13.8	2.3
Plant part for perfume	712.8	12.5
Beverage concentrate	16.1	33.6
Food flavours/gums	23.2	29.1
Turpentine	10.8	0.3
Pine oil	15.6	0.4
Rosin	281.0	4.2
Total	1497.2	196.9

Source: Sri Lanka Customs: Further break down of products not given.

* Latest figures available.

Perfumery, flavour and beverage materials imported during the year (1985)
(Source custom returns 1985)

Beverages, waters (ice and snow waters)	15,389 Lts	Rs. 643695.00
	5,591	64195.00
Lemon non alcoholic	196,099	456229.00
Essential oils, resinoids, perfumery cosmetics and toilet preparation	1,237 kg	280969.00
Essential oil resinoids	26,147 kg	3729360.00
Mixtures of two or more odoriferous substances and other mixtures	193,184 kg	44087672.00
Perfumery cosmetics and toilet preparation perfumed spirits etc.	640 Lts	6364373.00
Perfumery cosmetics and toilet preparations		
Eau de cologne	1,850 Lts	3045251.00
Toilet powder	10,867 kg	445219.00
Hair dyes	6,547 kg	4639274.00
Joss sticks	1,795 kg	132609.00
Toothpaste, shampoos	8,630 kg	2427548.00
Others	18,669 kg	5957500.00
Soap toilet, perfumed or medicated	2,856 kg	1038386.00
Turpentine (spirit of)	1,436 kg	81414.00
Pine oil	5,900 kf	198104.00

Total 219569 Lts. 277268 kg Rs. 776,97861.00

IMPORTS OF SPICES AND HERBS OF VALUE TO THE ESSENTIAL OIL INDUSTRY

Product (1988)	Quantity (Tonnes)	Value (Rs.Million)
Chillies	689.3	12.6
Coriander	8721.4	126.9
Cumin Cumin	1321.8	51.8
Fennel	542.5	5.6
Ginger	59.9	16.7
Turmeric	1002.0	14.5
Fenugreek	995.7	11.0
Mixed seasoning	30.9	5.7
Total	13363.5	244.8

EXPORT OF ESSENTIAL OILS FROM SRI LANKA

Oil	1985	1986	1987	1988
Cinnamon (leaf)	18.6*	14.5	26.2	36.7
Citronella	10.3	8.2	16.5	29.1
Cinnamon bark	6.2	6.7	11.8	18.9
Nutmeg	4.2	8.0	11.7	na
Cardamom	2.1	1.3	0.8	2.3
Other	1.8	4.0	14.2	9.2

* Millions of rupees

VOLUME AND VALUE OF EXPORTS OF ESSENTIAL OILS

Year	Volume (MT)	Value (Rs. Millions)
1984	346.4	49.1
1985	291.3	45.8
1986	3295.4	76.8
1987	1246.7	95.4
1988	902.5	135.6

ESTIMATED LOCAL PRODUCTION OF ESSENTIAL OILS IN SRI LANKA (1988)

Citronella oil	400 MT
Cinnamon leaf oil	300 MT
Cinnamon bark oil	0.5 MT
Lemon grass oil	5 MT
Nutmeg	18 MT
Cardamom oil	0.6 MT

Source - Export Development Board

Names and Addresses of leading Houses/Corporations

1. Volanka Limited,
74, De Waas Lane,
Colombo 14

2. Oleo Essences and Perfumes Ltd., (Hayley's Group),
50, Pedlar Street,
Galle

3. A S Chatoor & Co. Ltd.,
372, Cyril C Perera Mawatha,
Colombo 13

4. Chemanex Limited
52, Galle Face Court 2,
Colombo 3

5. G P de Silva & Sons
24, Front Street,
3rd Floor,
Colombo 1

6. Link Natural Products Limited,
83, Kinsey Road,
Colombo 8

Local demand of some of the perfumery and flavour compounds

Vanilla	15 Mtons/year
Strawberry	02 " "
Orange Flavour	02 " "
Mango and banana flavour	500 kg/y
Ginger oleoresin	03 Mtons/year
Lemon oil	300 kg/y
Citral	1 Mtons/y
Aldehyde C20	500 kg
Ethyl vanillin	1 Mtons/y
Ethyl butarate	300 kg/y
Ethyl iso-amyl iso-valerate	200 kg/y
Lime oil	300 kg/y
Pine oil	15 Mtons/y
Rose oil	200 kg/y

INDUSTRIAL PRODUCTION

(a) Types of essential oils/fragrances produced locally

The major essential oils produced locally are:

(a)	Cinnamon leaf oil	300 MT/yr
(b)	Cinnamon bark oil	0.5 MT/yr
(c)	Citronella oil	400 MT/yr
(d)	Nutmeg oil	18 MT/yr
(e)	Lemongrass oil	5 MT/yr
(f)	Cardamom oil	0.6 MT/yr

To a lesser extent essential oils are distilled from Eucalyptus globulus leaves and pine oleoresin. These oils are locally used by medicated balm manufacturers.

Sri Lanka does not produce any fragrances by the way of fractional distillation of essential oils or by chemical synthesis. Plans are under way to fractionally distill local essential oils under UNIDO project.

Equipment used

(a) Locally fabricated equipment

For many decades Sri Lankan essential oils were distilled in traditional stills, what is called "Boitare" in the native language. A larger type of stills are used for leafy essential oils such as citronella, lemon grass, cinnamon leaf. Small type pot still or goose neck type are used for distilling cinnamon bark and fine spices. A general survey showed that over hundred such units are scattered in southern Sri Lanka.

Diagramatic representation of a typical local still is given in the Fig. 30. This unit has four parts:

(a) A steam generating boiler
(b) A still body, normally made of air tight timber flanks, capacity about 50 kg.
(c) An elaborate condenser system, immersed in a pond or static tank.
(d) A condenser collecting system.

The steam generated by the boiler is fed into the still body holding the leafy material. The liberated volatiles together with steam is pushed into the condenser system. The condensed essential oils is collected at the end of the condenser.

In the case of cinnamon bark or fine spices distillation, bark is immersed in water and boiled directly by a firing chamber.

Recently Ceylon Institute of Scientific and Industrial Research (CISIR) introduced a new type of still called Manakoka and later and improved version called CISIRIL BOITARE for the distillation of leaf oil. A diagram of Boitare is given in the Fig. 31. Instead of a separate boiler, the steam generation is done at the false bottom of the main still body. The holding capacity of the raw material is about 400 kg. The main still body is constructed with mild steel sheets, while condenser coil is made up of 2 inch. diameter aluminium tubing CISIRIL BOITARE has a static water tank in which the condenser coil is immersed. Because of this arrangement, boitare can be operated in dry areas where running water is not available.

For the distillation of cinnamon bark and fine spices, earlier a unit called CISIRILL SPICAA was introduced. Later, this was replaced by a more efficient unit designed at CISIR. A diagramatic representation is given in Fig. 32. An efficient multibtubular condenser, cooled by running water is a main feature in this unit. This unit could distill about 50 kg batch load of spice at a time.

Most of the industrialists use locally fabricated distillation units for the production of essential oils. A number of small units (about 50 kg capacity) is operated by a central boiler of sufficient steam capacity. Each unit has an efficient water cooled multitubular condenser system. An advantage of such multidistillation units is that different spices can be distilled simultaneously. This is particularly useful when different varieties of spices are available simultaneously.

A general survey indicated that no one industrialists in Sri Lanka use imported distillation units for the production of essential oils. Eventhough the design may be of foreign origin, the fabrication is done locally. The two reputed companies that fabricate distillation unit are:

(i) Rowlands Ltd.
Colombo 7
(ii) Colombo Commercial Co. Ltd.,
Colombo 2

Research and Development Institutions

The major Institutions are:

(i) Ceylon Institute of Scientific & Industrial Research,
363, Baudhaloka Mawatha,
Colombo 7,
SRI LANKA

Contact Person

The Director of CISIR or Dr. U.M. Senanayake

Main activities

(i) R & D in the area of distillation of essential oils from local aromatic plants

(ii) Studies on chemical composition

(iii) Fractionation of essential oils

Special reference is given to

(a) Cinnamon leaf and bark oil

(b) Citronella oil

(c) Eucalyptus leaf oil

(d) Lemon grass oil

(e) Pine oleoresin

(f) Local spices as pepper, nutmeg, clove, ginger and cardamom

(ii) Industrial Development Board (IDB)
615, Galle Road,
Moratuwa,
SRI LANKA

Contact Person: c/o Director, IDB

Main Activities:

R & D work in the area of field distillation of essential oils.

(iii) Dept. of Export Agriculture,
Research Station,
Matale,
SRI LANKA

Contact Person: c/o Deputy Director, Research

Main R & D Activities

Breeding of high yielding varieties of cinnamon and other spices.

Institutions/Firms Utilizing Fragrance Material

Fragrance material are used mainly by Private Firms for the manufacture of colognes, perfumes, soft drinks, laundry and toilet soaps, confectionery and other products needing fragrance material.

	Firm	Industry
1.	Lever Bost. (Lanka) Ltd., Colombo 14	Laundry & toilet soap, shampoos, colognes, tooth paste
2.	B C C Lanka Ltd. Colombo 14	Laundry and toilet soaps
3.	Swadeshi Industries Wattala	- do -
4.	Harischandra Industries Matara	- do -
5.	Reckitt & Colman Ltd., Ratmalana	House hold cleaners, Hair creasm, colognes, polish & waxes
6.	Naharajah Organisation Ratmalana	Colognes & Perfumes, House hold cleaners, hair cream
7.	Lankem Lanka Ltd., Colombo 9	Disinfectants & Deodourants
8.	Ceylon Cold Stores Ltd., Colombo 2	Soft Drinks, Cordials
9.	Pure Beverages Ltd., Colombo 2	- do -
10.	Maliban Industries Ratmalana	Biscuits, Cakes
11.	Ceylon Biscuits Ltd., Homagama	- do -

Quantitative estimates of fragrance material annually used

Essential oils	45 MT
Aromatic mixtures	300 MT
Perfumes/cosmetics	4 MT
Eau de colognes conc.	25 (Lit)
Toothpaste/shampoo	65 "
Other toiletries	25 "
Soap	15 MT
Plants parts for perfumes	720 MT
Beverage concentrate	20 MT
Food flavours	25 MT
Turpentine	12 MT
Pine oil	20 MT
Rosin	300 "
Vanilla	15 Mtons/year

Strawberry	02 " "
Orange flavour	02 " "
Mango and banana flavour	500 kg/y
Ginger oleoresin	03 Mtons/year
Lemon oil	300 kg/y
Citral	1 Mtons/year
Aldehyde C20	500 kg/y
Ethyl vanillin	1 Mtons/y
Ethyl butarate	300 kg/y
Ethyl iso amyl iso valarate	200 kg/y
Lime oil	300 kg/y
Pine oil	15 Mt/y
Rose oil	200 kg/y

All these materials are imported. Out of the locally available materials manufactures use about 20 MT of citronella oil 1 MT of cinnamon leaf oil (for medicated balms) in their products.

Any other relevant information

Earlier all the essential oils distilled locally were exported. At the moment, under a special UNIDO programme a scheme is under way to fractionate the local essential oils to prepare value added products and formulate acceptable perfumery material, at least incorporating local essential oils.

At the moment varieties of citronella and lemon grass grown in Sri Lanka do not yield high quality oil. The root stock of citronella grown in Sri Lanka is over 60 yrs old. The yield and quality of oil is poor. The oil yield is around 1% and the actual geraniol content is around 15-20%. Plans are under way to replace the old root stock with high yielding and higher geraniol content varieties. The type called Jammurosa, obtained from India is under consideration.

Few decades ago lemon grass was a major essential oil crop in Sri Lanka. At the moment the area under lemon grass is around 2000 hectares. During the last few years a few private firms have taken the initiative to cultivate lemon grass on a large scale in the newly developed Mahaweli B and C scheme.

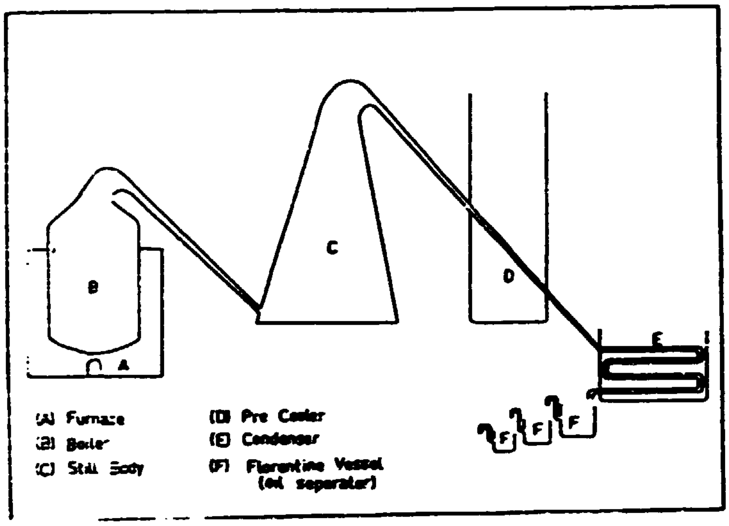


Fig. 30 Traditional cinnamon leaf oil still

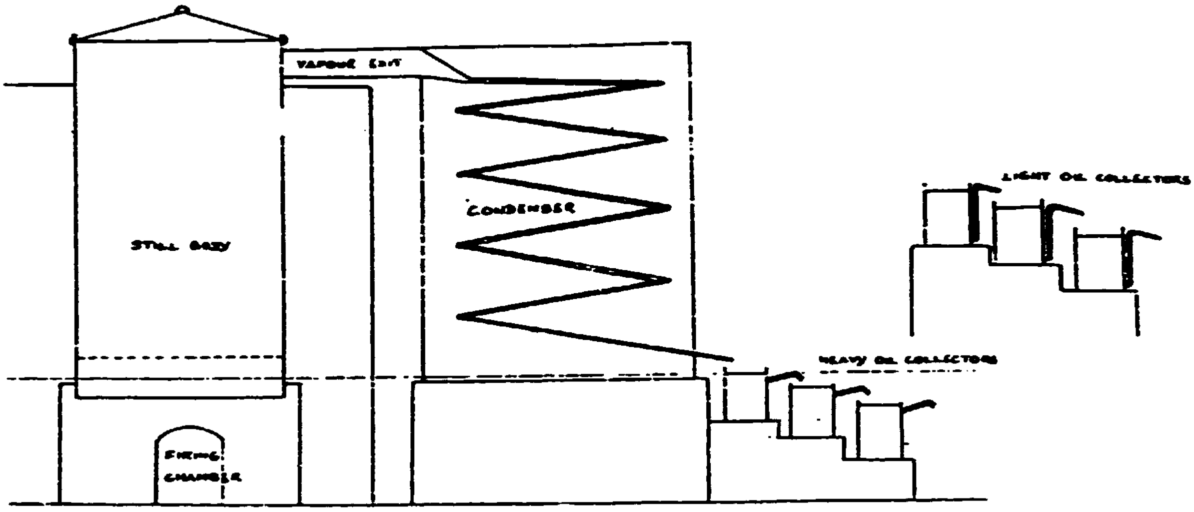


Fig. 31 CISIRILL BOITARE'

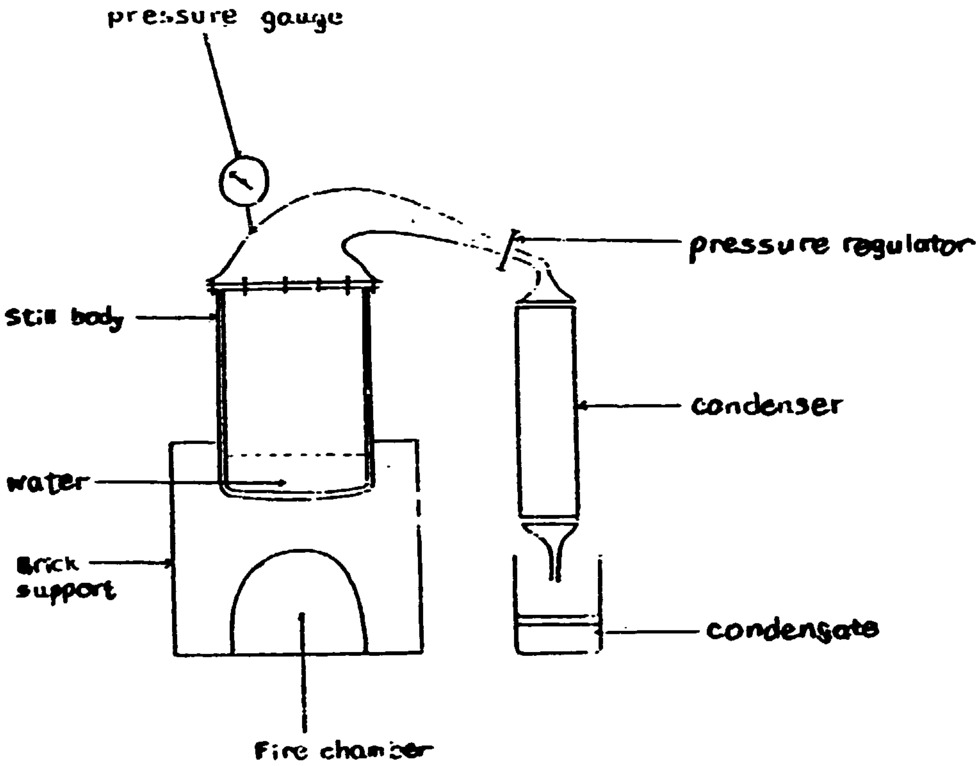


Fig. 32 CISIR field distillation unit

VIETNAM

TRADE IN ESSENTIAL OILS/FRAGRANCES WITHIN THE ESSENTIAL OILS ENTERPRISE IN VIETNAM

MRS. DOAN THI HOA BINH

I. INTRODUCTION

The Essential oils Enterprise (for short called ENTEROIL) has been established in 1985 within the National Centre for Scientific Research of Vietnam (CNSR) which is prominently the biggest centre for scientific research in Vietnam. ENTEROIL includes 6 divisions/ sections with a number of good and qualified experts and engineers. They are Marketing division, Technical division, Production division, Workshop, Administration and Liason office in Haiphong port.

The Essential Oils Enterprise is now handling the research and development of the production of essential oils with internationally accepted quality and the direct import-export activities through joint-venture, barter trade or any kind of contracts signed with domestic and foreign clients. Since early 1988, ENTEROIL has been funded by the United Nations Industrial Development Organization (UNIDO) with a Project entitled " Production and processing of Vietnamese essential oils and natural related products". During this short time of the Project Implementation, the Essential Oils Enterprise has already obtained complete up to date laboratory equipment and other modern facilities such as computerised Gas Liquid Chromatography (GLC), High Performance Liquid Chromatography (HPLC), IR, UV, hydrodistillation and steam distillation stills etc... imported from United Kingdom, France , West Germany, Japan and India. These advanced and sophisticated instruments are now fully utilized and help ENTEROIL to issue Certificates of Quality on essential oils produced in Vietnam for export.

II. DISCUSSION

I. EXPORT

Since 1988, with new economic reforms in Vietnam, The Government of Vietnam has given ENTEROIL the authority to deal with the direct import-export activities which were formerly passed through traditional official channels of other Agencies like Vietnam National Forest and Native Products Import-Export Corporation (GENERALEXIM).

ENTEROIL is now an independent Organization which could establish links and sign contracts with customers. This ability is, on one hand, due to its efforts and achievements, but to a large extent, due to considerable and efficient directives and support given by CNSR and UNDP/UNIDO. The export quantity of different kinds of essential oils is around 150-200 tons a year. However, the export amount and its value have been increasing annually as shown in Table 11.

Table 11: Vietnam exports of essential oils

Commodity	QTY (kg)	VALUE (UD$)	Year
O. gratissimum	8,200	49,200	1988
	32,000	192,000	1989
	60,000	360,000	1990
	75,000	tentative	1991
Mantha arvensis and menthol crystals	4,155	49,860	1988
	4,000	46,000	1989
	7,000	80,500	1990
	25,000	tentative	1991
Citronella oil	358	2,506	1988
	40,000	160,000	1989
	150,000	525,000	1990
	150,000	tentative	1991
O. basilicum	800	40,000	1988

(Methyl chavicol	3,000	75,000	1989
	12,000	276,000	1990
	10,000	tentative	1991
Cassia oil	7,000	189,000	1990
	10,000	tentative	1991
<u>Litsea cubeba</u>	136	1,360	1988
	10,000	tentative	1991
Pemou oil	201	2,010	1988
	110	1,254	1990
	5,000	tentative	1991
Melaleuca oil	10,000	50,000	1990
	15,000	tentative	1991
Star anise seed oil	10,000	85,000	1990
	20,000	tentative	1991
<u>O.Basilicum</u>	23	1,185	1990
(Linalool type)	2,000	tentative	1991

The contents of major components in essential oils for export depend on the need of the clients. However, the essential oils exported by ENTEROIL are always of good quality. Nowadays, a lot of businessmen are increasingly coming to ENTEROIL for trading due to the good reputation of the company. ENTEROIL has also started upgrading and extracting essential oils to make isolates/fragrances such as Citronellal, Citronellol from oil of Citronella Crystal Menthol from <u>Mentha arvensis</u> etc... for export.

Moreover, ENTEROIL always guarantees time of delivery of goods exported according to terms set out in the contracts.

ENTEROIL also offers joint venture, barter trade or any kind of contracts. The international markets of ENTEROIL includes U.K., France, Soviet Union, and other Eastern European countries.

Terms of payment: by irrevocable letter of credit or payment made in different times depending upon the agreement of the two parties.

II. IMPORT

In order to strengthen the trading activities, ENTEROIL imports materials for fabrication of distillation stills (such as stainless steel, iron, valves...) and new galvanized drums for packing essential oils to be exported. It also imports agricultural materials and consumption goods i.e. urea, pesticides, tyre, etc... that depend on the requirements from growers/farmers. It provides growers/farmers with new distillation techniques, harvesting, etc... ENTEROIL is ready to procure their products with good price. The growers are therefore willing to grow and enlarge the area of essential oil cultivation.

III. LOCAL FABRICATION OF DISTILLATION STILLS AND FRACTIONATING COLUMNS

Through the UNIDO Project, the hydrodistillation and steam distillation stills (300 kg/batch cap.) S.S. French type are available at ENTEROIL. These modern and advanced equipment served for demonstration purposes and optimisation of essential oils. ENTEROIL has also obtained an all glass fractionating column (15 L/batch cap.) for upgrading and extracting essential oils to make isolates/fragrances.

Based on the drawings of the above equipment and with materials imported from Hongkong, Thailand and India, ENTEROIL staff in co-operation with CNSR workshop have designed and fabricated one S.S. and mild steel fractionating column of 100 L/batch capacity and two S.S. and mild steel distillation stills of 300 kg/batch capacity. After trial commissioning and test of all parameters of the equipment, they will be transfered to the production centres. This is the best approach because it is at least 2 or 3 times cheaper than imported ones and it can exploit all technical expertise of its experts/engineers.

The engineers and technicians are now very experienced in designing and fabricating fractioning columns and distillation stills of any capacity for any kind of essential oils.

III. CONCLUSION

With a very short time, the Essential Oils Enterprise has already achieved very good and considerable results in research and development and trading of essential oils. The prestige of the Essential Oils Enterprise among its clients has been increasing. More than that, it is very important for trading that the capital of ENTEROIL has also increased.

Due to its efforts and progress, the Government of Vietnam approves that the Essential Oils Enterprise can set as a national model in terms of research/development and marketing of essential oils industry in Vietnam.

PHILIPPINES

ESSENTIAL OIL INDUSTRY IN THE PHILIPPINES

Dr. FELICIDAD E. ANZALDO

The Philippines is one of the tropical countries endowed with rich flora which are known to be potential sources of some natural products like the essential (volatile) oils with industrial and medicinal values.

The essential oil industry in the Philippines had its commercial beginning around the year 1864 when small quantities of ylang-ylang were exported to Europe. Exportation continued with the late 19th and early 20th centuries, when ylang-ylang oil won recognition abroad and became one of the important ingredients of European and American perfumes.

Unfortunately, the advent of World War I resulted in the closure of firms engaged in the essential oil business which were mostly owned by the German nationals. Also, ylang-ylang trees were introduced into French territories, where cheaper oils were produced. These events consequently led to the death of the industry.

A. <u>Local Production</u>

To date, the Philippine essential oil industry has not yet recovered. There are no sizeable producers of essential oil. Local production is limited to very small operations or pilot plant facilities. As a result, virtually all of the local requirements for essential oils are met by imports. The local industry is basically limited to compounders/blenders of these imported essential oils.

At present, the most impressive pilot plant is that of Pilipinas Kao in Jasaan, Misamis Oriental which was provided technical support by Kao International of Japan.

Alicia Cooperative Milling and Marketing, organized by Filipinas Foundation and Mr. Juan Duñgog has a 23 ha. (projected expansion to 100 ha.) experimental farm planted with citronella (intercropped with mango) in Alicia, Bohol. Estimated production capacity is about 2.5 tons of oil/year and at P200.00 /kg. This is about P500,000.00

Mr. Cyrus Aparri has organized a small-scale contract growing of citronella in backyards. Citronella grass grown for supplemental income by farmers is bartered with herbal soap produced by Mr. Aparri. This counter trade operation ensures markets for both citronella and herbal soap. However, it is doubtful whether one could competitively expand this type of operation to a much larger scale. Volume of the small-scale operation is estimated at 100 kgs. of oil/month.

Himmel Philippines, a local compounder of flavours and fragrances conducted a joint field study with the Bureau of Plant Industry on growing of citronella grass and extraction of citronella oil. Although data were gathered and citronella oil was actually produced, the farmers and buyers of the oil failed to come to a mutually satisfactory pricing of the oil so that production did not proceed further.

The National Institute of Science and Technology (now Industrial Technology Development Institute) operated a small essential oil extraction facility. It is principally a steam distillation unit used for trial tests on local plant sources of essential oil.

The Department of Science and Technology Region IV has a pilot plant in Nagcarlan, Laguna. The essential oils being extracted for further studies are citronella and patchouli.

B. Local Usages

Value and volume statistics taken from Central Bank Data for Philippine imports of Essential Oils, perfume and Flavour Materials are presented in Tables 12 and 13 for 1983-1987. From a CIF value of $27.1 million, in 1983, these importations went down to $20.6 million in 1985 and up to $25.4 million in 1987. These data cover essential oils, synthetic materials and their mixtures. Only a few essential oils had specific data and these were anise, lime and lemon, citrus excluding lime and lemon, peppermint, spearmint, eucalyptus and ylang-ylang. The rest of the essential oils are covered under "Others". From the statistics given, the CIF value of essential oils and synthetics/mixtures of essential oils and synthetics were estimated as follows:

CIF VALUE OF IMPORTS (U.S. DOLLARS)

Year	Essential Oils	Synthetics, Mixtures of Synthetics and Essential Oils
1983	10,765,245	16,336,040
1984	9,312,775	15,730,546
1985	8,425,811	12,211,950
1986	8,114,794	14,640,814
1987	9,304,703	16,092,590

Unfortunately, there is no way of deriving the exact quantity/volume of essential oils covered in the mixtures of essential oils and synthetics. One can, however, assume that the value of the essential oils in the mixtures is significantly less than the synthetics since synthetics are usually the less costly components.

Among the essential oils specified, the largest value of imports fall under peppermint and spearmint, followed by lemon and lime, other citrus and eucalyptus.

The principal countries shipping essential oils to the Philippines are listed in Table 14. United States is the top supplier; the second largest shipper is Hongkong which is not a producer but a transhipment point. Other principal suppliers are European countries except for Japan and Indonesia. Table 15 shows the Philippine companies importing essential oils

PRODUCTION AND TECHNICAL ASPECTS

A. Agriculture

Table 16 shows the list of aromatic plant species found in the Philippines.

B. Processing

Essential oils are usually a mixture of many substances. Chemical constituents of essential oils are chiefly hydrocarbons, ketones, aldehydes, alcohol and esters. The hydrocarbons are in the form of cyclic, bicyclic, and open-chain terpenes.

Essential oils do not occur uniformly in all plant parts. The essential oil-rich parts of a plant may be its flowers, leaves, roots, fruits or bark or any combination thereof.

Steam distillation is the most commonly used process for essential oil extraction in the country which is the use of hot water and/or steam to volatilize the oil and its subsequent separation as condensate. Other methods employed in essential oil extraction are:

a) expression b) solvent extraction
c) enfleurage d) adsorption and carbon dioxide extraction

Among the considerations for the choice of a particular process for the extraction of essential oil are: sensitivity of the essential oil to heat and solubility of the essential oil in water or solvent.

Bernard Meyer Warnod "Natural Essential Oils" Perfumery and Flavorist volume 9 April -May 1984 have observed that the trend of development in natural essential oils will be to:

1. Obtain products free from organic solvents.
2. Improve the preparation of the plant matter in order to get better yields.
3. Develop aromatic molecules through fermentation.

R & D Activities:

With the intention of initially reducing the importation of essential oils and also possibly producing products acceptable to the domestic markets, the Industrial Technology Development Institute (ITDI) of the Department of Science and Technology (DOST) is conducting researches on the following five prospective essential oils:

A. Citronella

Citronella is one of the few essential oils that has been studied locally with pilot plant studies. As earlier mentioned, previous studies were undertaken by Himmel Industries and the Bureau of Plant Industry. The Livelihood Corporation has taken over the extraction of citronella oil from plants grown by farms in Bilira, Leyte.

Locally produced citronella can provide the fragrance for low-price basic products like soap especially in the rural areas. But, as the rural economy develops and becomes sophisticated, citronella may lose its market to a number of synthetics.

Another possibility for citronella would be to provide one of the fragrances for those who desire "natural" essential oils.

B. Ylang-Ylang

Ylang-Ylang oil is extracted from the blossoms of ilang-ilang tree (Cananga odorata Linn). It is used to imporve the floral fragrance of high class perfumes. While the Philippines used to be the primary exporter of ilang-ilang oil, world supply currently come from Comaros and Madagascar.

Ilang-ilang is currently sold in the domestic market as fresh flowers for use in the preparation of garlands. There is no known significant local extraction of the essential oil. However, it is indicated in the trade that there is a potential domestic market for ylang-ylang oil as a component for local manufacture of perfumes.

Ylang-ylang thrives well in the Philippines especially in well-drained soil with a lot of decomposed organic matter. It is a hardy tree that is not easily susceptible to diseases.

About 50 hectares in the southern part of Luzon (San Pedro, Laguna and Nasugbu, Batangas) are planted with ylang-ylang trees and 100 hectares in the northern part of Luzon (Pampanga, Tarlac and Isabela). About 80 hectares in the eastern part of the country and about 20 hectares in Mindanao.

Ylang-ylang oil is extracted by distillation through directly fired or steam heated stills. The latter is more efficient and produces high quality oils. Total yield is about two percent (2%) of the weight of the flowers of which (35 %) can be of the extra grade.

C. Patchouli

The oil of patchouli is derived from distillation of dry leaves of tropical plant patchouli, Pogostemon cablin Benth which has been reported to be a native of the Philippines. The oil has a warm, powerful, sweet, herbaceous, aromatic, spicy fragrance which stays long and blends well with other perfumes, making it a valuable perfumery fixative. The oil is also almost a perfume by itself. Patchouli oil has no comparable competition from synthetics.

Patchouli is an aromatic undershrub which grows well in partially-shaded, well-drained fertile soil with evenly distributed rainfall. As a shade loving plant, it can be inter-cropped with coconut. The yield of patchouli oil is about 2-3%.

Patchouli is propagated vegetatively. About 2 hectares in Nagcarlan, Laguna are planted with Patchouli and around 10 hectares (scattered) in Palawan.

D. Vetiver

Vetiver oil is obtained from the distillation of the grass Vetiveria Zizanoides. The viscous vetiver oil has a sweet, persistent, heavy, woody-earthy fragrance. It also has fixative properties so that it is used in perfumery together with more volatile constituents.

The local market of vetiver oil is unknown and probably currently minimal. However, together with the local production of vetive oil, a programme for the compounding of perfume materials and manufacture of perfume itself could probably be developed.

Vetiver is a grass. The commercial oil is obtained from the roots. The most suitable soil for vetiver consists of loose sandy ground or if available, loose volcanic ashes.

The roots are usually harvested 16-24 months after planting. Yields of 1.0-1.3 tons of air-dried roots per hectare have been reported. The yield of oil is around 1.5-2.0%. Well managed operations will probably give a net yield of around 20kgs. of oil per hectare.

On a per kg. of oil basis, vetiver offers a very attractive price which is not as volatile as other oils. Thus far, there are no indications of competition from synthetics.

E. Mint

Mint represents the country's largest single essential oil import. These were declared as peppermint and spearmint oils although it is believed that a substantial fraction of these represents Mentha arvensis oil.

Mentha arvensis L. is grown in a one-hectare farm in Leyte and Mindoro Occidental. Examination showed that the oil contains (80%) L-menthol. Mentha arvensis is a source of natural menthol which is used extensively in cigarettes, toothpaste, mouthwashes, medical preparations, candies, deodorants, etc. While menthol used to be the only saleable product from Mentha arvensis, a recent development is the growth in the use of dementholized oil for toothpaste and chewing gum which has led to the increase in value of crude Mentha arvensis oil. With these two products, Mentha arvensis oil has once again become competitive and strong to meet the challenge of synthetic menthol.

The next Three Essential Oils

In addition to the five aforementioned priority, the following essential oils also are being investigated:

1. Calamansi - Calamansi is one local citrus fruit which has attracted attention as a source of juice drinks. The local beverage industry and possibly some markets abroad could make use of the "natural" essential oil extracted from calamansi. Recently, the laundry soap industry was looking for suppliers of calamansi oil. Considering that calamansi juice is now extracted by juice processors, extraction of calamansi oil from calamansi peel should increase the proceeds from calamansi processing.

2. Sampaguita- This very popular flower excites local essentil oil enthusiasts. For one thing, it is related to Jasmine which has significant world trade. Considering the popularity of sampaguita, it could be developed at least for the local market.

3. Eucalyptus - Eucalyptus is a tree planted not only for essential oil production and for utilization of its wood but also for reforestration. A feasibility study for using eucalyptus oil is being prepared.

There is a bright market prospect for the establishment of essential oil industry in the country considering its import substitutability and export potential.

The demand for essential oil is constantly increasing in our country and the market for locally produced essential oils is very promising:

Table 12 **Philippine imports of essential oils and related materials**

CODE	COMMODITY	CIF VALUE (U.S. DOLLARS)				
		1983	1984	1985	1986	1987
551.00	Essential oils, perfume and flavour materials	27,101,285	25,043,321	20,637,761	22,755,608	25,397,293
551.30-01	Anise oil for the manufacture of medicine	2,081	1,082	4,717	1,992	703
551.30-02	Anise oils other than for the manufacture of medicine	8,000	203	-	232	-
551.30-03	Lime and lemon oils for the manufacture of medicine	267,444	42,878	12,420	16,230	27,995
551.30-04	Lime and lemon oils other than for manufacture of medicine	2,832,075	2,427,840	1,052,930	1,147,604	986,570
551.30-05	Citrus oil excluding lime and lemon oils for the manufacture of medicine	11,304	19,850	5,356	3,781	9,021
551.30-06	Citrus oil excluding lime and lemon oils other than for the manufacture of medicine	91,727	177,332	119,318	163,789	166,299
551.30-07	Peppermint and spearmint oils for the manufacture of medicine	30,515	132,444	51,323	58,400	56,755
551.30-08	Peppermint and spearmint oils other than for the manufacture of medicine	6,475,233	4,147,468	5,239,413	5,160,432	5,074,823
551.30-09	Oil of Eucalyptus	44,066	61,597	45,682	98,312	54,348
551.30-12	Ylang-ylang oil	7,964	5,832	-	4,322	-

CODE	COMMODITY	CIF VALUE (U.S. DOLLARS)				
		1983	1984	1985	1986	1987
551.30-19	Other essential oils for the manufacture of medicine	192,193	183,578	140,354	158,245	257,677
551.30-29	Other essential oils (excluding oils used for the manufacture of medicine)	786,840	2,102,690	1,300,130	1,296,800	2,660,913
551.30-40	Terpenic by-products of the deterpenation of essential oils	12,560	9,881	4,168	4,725	9,519
551.30-60	Concentrates of essential oils in fats, fixed oils or in waxes or the like obtained by cold absorption or by maceration	3,243	-	-	-	-
551.41-00	Mixtures of essential oils, resinoids or synthetic aromatics	2,440,860	1,519,152	2,433,423	3,091,268	1,904,055
551.42-00	Synthetic Perfume and Flavor Materials and Concentrates, Enfleurage Greases and Mixture of alcohol and essential oils, for use in Perfumery	469,038	143,137	195,504	318,497	628,488
551.49-00	Synthetic Flavor Materials and Concentrates, Enfleurage Greases and Mixtures of Alcohol and essential oils, used in the Pharmaceuticals, food, etc. Industries.	13,426,142	14,068,257	9,583,023	11,231,049	13,560,047

SOURCE: Foreign Trade Statistics, Central Bank

Table 13 **Philippine imports of essential oils and related materials**

CODE	**COMMODITY**	**QUANTITY (KGS.)**				
		1983	**1984**	**1985**	**1986**	**1987**
551.00	Essential oils, perfume and flavour materials	3,294,839	3,950,075	2,350,325	3,171,424	3,573,088
551.30-01	Anise oil for the manufacture of medicine	174	92	357	136	70
551.30-02	Anise oils other than for the manufacture of medicine	500	5	-	12	-
551.30-03	Lime and lemon oils for the manufacture of medicine	5,778	4,602	750	561	2,358
551.30-04	Lime and lemon oils other than for manufacture of medicine					
551.30-05	Citrus oil excluding lime and lemon oils for the manufacture of medicine	227	460	89	374	863
551.30-06	Citrus oil excluding lime and lemon oils other than for the manufacture of medicine	15,887	21,995	13,395	25,395	26,651
551.30-07	Peppermint and spearmint oils for the manufacture of medicine					
551.30-08	Peppermint and spearmint oils other than for the manufacture of medicine	343,438	257,896	274,363	220,356	237,076
551.30-09	Oil of Eucalyptus	10,027	10,683	8,694	19,023	19,998
551.30-12	Ylang-ylang oil	308	211	-	164	-

CODE	COMMODITY	QUANTITY (KGS.) 1983	1984	1985	1986	1987
551.30-19	Other essential oils for the manufacture of medicine	10,963	32,875	7,623	5,938	12,036
551.30-29	Other essential oils (excluding oils used for the manufacture of medicine)	118,650	306,207	131,357	133,620	342,076
551.30-40	Terpenic by-products of the deterpenation of essential oils	4,876	3,732	1,430	1,560	5,926
551.30-60	Concentrates of essential oils in fats fixed oils or in waxes or the like obtained by cold absorption or by maceration	127	-	-	-	-
551.41-00	Mixtures of essential oils, resinoids or synthetic aromatics	390,035	119,283	173,625	213,567	154,042
551.42-00	Synthetic Perfume and Flavor Materials and Concentrates, Enfleurage Greases and Mixture of alcohol and essential oils, for use in Perfumery	17,406	4,333	5,105	6,763	21,510
551.49-00	Synthetic Flavor Materials and Concentrates, Enfleurage Greases and Mixtures of Alcohol and essential oils, used in the Pharmaceuticals, food,etc. Industries.	2,324,955	3,128,051	1,703,837	2,516,118	2,733,425

SOURCE: Foreign Trade Statistics, Central Bank

Table 14 Principal sources of Philippine oil imports (1987)

	COUNTRY	CIF VALUE (U.S. DOLLARS)
1.	United States	$3,984,843
2.	Hongkong	3,079,492
3.	Netherlands	542,734
4.	Ireland (Eire)	443,277
5.	United Kingdom and North Ireland	350,063
6.	France	189,463
7.	Japan	180,625
8.	Spain	155,132
9.	Germany, Federal Republic of	148,318
10.	Indonesia	93,260

Table 15 Principal Philippine companies importing essential oils

COMPANY	COMMODITY	LANDED COST (PESOS)
1. Fortune Tobacco Co.	Peppermint Oil Eucalyptus Oil	P 58,490,469
2. La Suerte Cigar & Cigarette Factory	Blue Grass "B"	33,692,991
3. Procter and Gamble PMC	Industrial Essential Oil Essential Oil Orange Terpenes Eucalyptus Oil Nutmeg Oil Lemon Oil (Synthetic) Cedar Leaf Oil Ginger Soluble Concentrate Lemon Oil	16,794,277
4. Coca-Cola Export Corp.	Tangerine Oil Natural Lime & Lime Oil Orange Oil Lemon Oil Lime Oil	14,043,271
5. Wrigley Phils. Inc.	Spearmint Oil Peppermint Oil	11,900,117
6. Seven-up Phils. Inc.	Lemon Oil Lemon Oil (Terpeneless) Lime Oil (Concentrate)	10,698,525
7. Dragoco, Phil. Inc.	Industrial Essential Oil Lemon Oil Orange	5,111,146
8. International Flavors and Fragrances	Rum Ether Concentrate Essential Oils Flavorina Materials Black Pepper Oleorisin Deertounge S.E. Foenugreek S. E. Vanilla Concentrate Peppermint Oil	4,986,012
9. Philippine Refining Corp.	Industrial Essential Oil ABF 1014	4,944,448
10. Cosmos Bottling Corp	Essential Oil	4,710,705
11. Actron Industrial Inc.	Industrial Essential Oil	4,542,307
12. Johnson & Johnson	Essential Oil	4,462,832
13. Berli Jucker Industry Inc.	Essential Oil	4,329,771

14. Pepsi-Cola Far East Trade Development Co.	Lemon Lime Concentrate Mixed Oil Concentrate Essence	4,186,289
15. Avon Products Manufacturing Inc.	Essential Oil Trazarra Fragrance Sunny Orange Bouquet Compound B	2,633,064
16. A.F. Merchants ABF 1014	ABF 1014 Mixtures of Odoriferous substances	2,607,683
17. Peerless Products Manufacturing Corp.	Industrial Essential	2,435,247
18. J.M. Manufacturing Enterprises	Essential Oil Compound	1,890,231
19. Pilipinas Kao	Essential Oil	1,147,341
20. Warner Lambert	Eucalyptol Peppermint Oil Oil of Eucalyptus Thymol Spearmint Oil	991,839

Data gathered from private monitoring agency which provided landed costs and/or FOB. HCV costs. Where only FOB or HCV costs were provided, landed costs were estimated.

Table 16 Sources of essential oils in the Philippines

SCIENTIFIC NAME AND FAMILY NAME	COMMON NAME/LOCAL NAME	PARTS UTILIZED
1. Acasia farnesiana L. Leguminosae	Aroma/Cassia Flower	Flowers
2. Acorus calamus L. Araceae	Sweet flat/Lubigan	Rhizomes
3. Aegle Marmelos Correa Pinaceae	Bael	Rind, Flowers
4. Agathis alba Pinaceae	Manila copal/Almaciga	Bark
5. Aglaia odorata Lour Meliaceae	Sinamomong-sungsong	Flowers
6. Allium cepa L. Liliaceae	Onion/Sibuyas	Bulb
7. Allium odorum Liliaceae	Kuchai	Leaves
8. Allium sativum L. Liliaceae	Garlic/Bawang	Bulb
9. Andropogon citratus DC. Graminae	Lemongrass/Tanglad	Leaves
10. Andropogon nardus var. hamatulus Graminae	Citronella	Leaves
11. Andropogon zizanoides (L.) Graminae	Vetiver/Moras	Roots
12. Gananga odorata Baill. Anonaceae	Ylang-ylang	Flowers
13. Canarium luzonicum (B) A.Gray Burseraceae	Manila elemi/Pili	Bark
14. Capsicum frutescens Solanaceae	Cayenne pepper/ Siling labuyo	Ripe fruits
15. Cestrum nocturnum Linn. Solanaceae	Dama de Noche	Flowers
16. Cinnamomum mercador Vidal Lauraceae	Kaliñgag	Bark
17. Cinnamomum mindanenae Flm. Lauraceae	Mindanao cinnamon	Bark

SCIENTIFIC NAME AND FAMILY NAME	COMMON NAME/LOCAL NAME	PARTS UTILIZED
18. Citrus aurantifolia Surincle Rutaceae	Dayap	Rind
19. Citrus aurantium Linn. Ritaceae	Kahel	Rind
20. Citrus grandis Osb. Rutaceae	Lukban	Rind
21. Citrus hystrix Rutaceae	Kabuyaw	Rind
22. Citrus macrantha Rutaceae	Samuy	Rind
23. Citrus microcarpa Bunge Rutaceae	Kalamansi	Rind
24. Citrus nobilis Lour. Rutaceae	Darangita	Rind
25. Citrus reticulata Blanco Rutaceae	Darangita	Rind
26. Clausena anisum (B) Merr. Rutaceae	Kayumanis	Leaves
27. Coleus amboinicus Lour. Labiatae	Oregano	Leaves
28. Curcuma Longa Linn Zingiberaceae	Barak	Rhizome
29. Curcuma zedoaria (Berg.) Rose Zingiberaceae	Barak	Rhizome
30. Coriandrum sativum Umbelliferae	Coriander/Kulantro	Seeds
31. Eucalyptus tereticornis Sm. Myrtaceae	Eucalyptus	Leaves
32. Foeniculum vulgare Gaertn. Umbelliferae	Anise/Anis	Seeds
33. Gardenia augusta (L.)Merr. Rubiaceae	Rosal	Flowers
34. Hedychium coronarium Koenig Zingiberaceae	Kamia	Flowers
35. Illicium anisatum Linn. Magnoliaceae	Sanke	Fruits

SCIENTIFIC NAME AND FAMILY NAME	COMMON NAME/LOCAL NAME	PARTS UTILIZED
36. Jasminum grandiflorum L. Oleaceae	Jasmine/Jasmin	Flowers
37. Jasminum sambac (L. Art) Oleaceae	Sampaguita	Flowers
38. Lininophila rugosa (Roth)Merr. Schropulariaceae	Tala	Leaves
39. Mangifera indica Linn. Anacardiaceae	Mango	Peels
40. Melaleuca leucadendron Myrtaceae	Cajeput	Leaves
41. Mentha cordifolia Labiatae	Philippine mint/Yerba buena	Leaves
42. Michelia champaca Linn. Magnoliaceae	Champaka	Flowers
43. Murraya paniculata (L.) Jack Rutaceae	Kamunina	Flowers
44. Ocicum basilicum L. Labiatae	Sweet basil/balanoi	Leaves
45. Ocicum sanctum L. Labiatae	Sulasi	Leaves
46. Pandanus odoratissimus Pandanceae	Pandan-mabango	Leaves
47. Pinus insularis Pinaceae	Benquet pine	-
48. Piper nigrum L. Piperaceae	Pepper/Paminta	Seeds
49. Plumbago indica (L.) Plumbaginaceae	Bay leaf/Laurel	Leaves
50. Plumiera acuminata Ait. Apocynaceae	Kalachuche	Flowers
51. Pogostemon cablin Renth. Labiatae	Patchouli/Kablin	Leaves
52. Posmarinus officinalis Linn. Labiatae	Rosemary/Romero	Leaves
53. Sesamum orientale Pedaliaceae	Linga	Seeds

SCIENTIFIC NAME AND FAMILY NAME		COMMON NAME/LOCAL NAME	PARTS UTILIZED
54.	Sandoricum koetjape Merr. Meliaceae	Santol	Wood
55.	Toona calantas Merr. & Rolfe Meliaceae	Kalantas	Wood
56.	Toddalia asiatica (L.) Lam Rutaceae	Dauag	Leaves
57.	Triphasia trifolia P. Wills. Rutaceae	Limonsito	Fruits, seeds
58.	Zingiber officinale Linn. Zingiberaceae	Ginger/Luya	Rhizome

THAILAND

ESSENTIAL OILS INDUSTRY IN THAILAND

Mrs. A. PUNRUCKVONG

Although Thailand is rich in aromatic plants but the essential oils industry has not yet been well established. Table 17 shows aromatic plant resources in Thailand. In 1974 mint oil production was successfully started by the cooperation of Thailand Institute of Scientific and Technological Research (TISTR) and Menthol Thai Import Export Co., Ltd. The mint oil industry was not smoothly operated, the company has overcome enormous problems before it could reach to this point. These problems ranging from quality standard of the oil produced by villagers and price collapsed due to smuggling in of menthol and mint oil from China. The company had to close down a few years later and reopened again in 1978. Menthol Thai Import Export Co., Ltd. is now producing mint oil, menthol (100 tons/year). Other essential oils were imported and purified before marketing such as eucalyptus oil (40 tons/year) and peppermint oil (155 tons/year). The distillation unit was locally made.

Table 18 shows the import and export of essential oils during 1985 to 1988. Table 19 shows names and address of leading of trading companies in Thailand.

The Pharmaceutical and Natural Product Department, of TISTR has a research project on the development of essential oils from potential aromatic plants in Thailand. These potential essential oils are basil oil, with a methyl chavicol content 90+-2% and lemongrass oil with a citral content 79%. The price of these essential oils could not compete internationally due to high cost of production. High labour charges and price of fuel are the main reasons. The extended use of essential oil in pharmaceutical products was found in the essential oil from Zingiber cassumunar as the main ingredient in anti-inflammatory cream.

Table 17 Aromatic Plant Resources in Thailand

Species most abundant:

Ocicum basilicum
Ocicum canum
Ocicum gratissimum
Ocicum sanctum
Zingiber officinale Rosc.
Zingiber cassumunar
Cymbopogon citratus
Mentha arvensis

Species less abundant:

Vetiveria zizanioides
Mentha piperita
Pogostemon cablin Benth
Carum carvi
Cinnamomum camphora
Cuminum cyminum L.

Table 18 Essential oils Export during 1985 - 1988

	Type of Essential Oil	**Value (US$)**
1.	Peppermint oil	10,800,000
2.	Other Mints oil	2,000
3.	Wintergreen oil	8,900
4.	Citrus oil	400
5.	Jasmin oil	6,000

Essential oils Import during 1985-1988

	Type of Essential Oil	**Value (US$)**
1.	Eucalyptus oil	120,000 - 461,499
2.	Orange oil	784
3.	Lemon oil	10,000
4.	Geranium oil	250
5.	Peppermint oil	200,000
6.	Cajeput oil	10,400 - 27,000
7.	Citronella oil	5,000 - 10,000
8.	Anise oil	1,000 - 52,000

Table 19 Names and Addresses of leading trading companies

1. Givaudan (Thailand) Co., Ltd.
38 Sukymvit 63 Bangkok, Thailand
Tel: 391 -0488

2. Bush Boake Allen (Thailand) Co., Ltd.
27/550 - 553 Lardprao Rd.
Bangkok 10240
Tel: 378 1449

3. Hong Huat Co., Ltd. (Authorized agent of HARABOT Grasse, France)
Tel: 223-3234-9

NEPAL

ESSENTIAL OILS INDUSTRY IN NEPAL

Dr. S.R. ADHIKARY

Aromatic Plant Resources of the Country

Nature has bestowed Nepal with a wide range of climatic conditions which is favourable for many essential oil bearing plants to grow. A large number of these economically important plants are found in spontaneous flora. Also many essential oil bearing crops can be raised at different agroclimatic zones of the country.

In Nepal, research and development work for the production of essential oil was started in the seventies by the Department of Forestry and Plant Research (formerly known as Department of Medicinal Plants) and as a result of these efforts some progress towards developing essential oil industry in the country was made. A few achievements made in this endeavour are given below.

(a) A list of essential oil bearing plants of Nepal was enumerated.

(b) Screening of wild flora was initiated and this programme resulted in identifying a number of essential oils which already have established use and a few new oils of economic value.

(c) Introduction of exotic aromatic plants for large scale plantation.

The above mentioned programme of scientific investigation such as agricultural practices and extension, phytochemical investigation, quality evaluation and extraction technology coupled with market development have been important factors in establishing an essential oil industry in Nepal.

A number of essential oils distilled from aromatic plants originating either from forests or agriculture land have been test marketed. Amongst these the aromatic plants of Nepal presently harvested from the forest area for the production of essential oils include the following:

1. Acorus calamus L.
2. Gaultheria fragrantissima Wall.
3. Cinnamomum glaucescens (Nees) Drury
4. Zanthoxylum Alatum Roxb
5. Nardostachys grandiflora DC
6. Pinus roxburghii Sargent
7. Lichens species for resinoids.

Similarly the aromatic plants presently cultivated in Nepal for the production of essential oils are as follows:

1. Mentha arvensis Linn.
2. M. Piperita L. Var. officinalis
3. Cymbopogon flexuosus (Steud.) Wats. (Lemongrass)
4. Cymbopogon martini var. motia (Palmarosa)
5. Cymbopogon winterianus Jowitt (Citronella Java)
6. Eucalyptus sps.

In addition to the above mentioned aromatic plants, there are a large number of other plants with potential for the production of essential oils. These plants collectively constitute the aromatic plants resource of the country and most important of them are given in Table 20.

Table 20 Some aromatic palnts growing spontaneously in Nepal

Botanical Name	Common Name/Vernacular Name
Species most abundant:	
Abies pindrow Spach	- /Talispatra
Abies spectabilis (D. Don) Spach (Syn: A. webbiana Lindl.)	Himalayan Silver Fir/ -
Acorus Calamus L.	- /Bojho
Allium sativum L.	Garlic /Lasun
Amomum subulatum (Cultivated)	Cardamom /Alaichi
Artemisia vulgaris L.	Armoise /Titepati
Boenningausenia albiflora (Hook.)Meisn.	- /-
Cinnamomum camphora (L.) Sieb.	Camphor /Kapoor
Cinnamomum glaucescens (Nees) Drury	- /Sugandhakokila
Cinnamomum tamala (Buch.-Ham.) Nees et Eberm.	- /Tejpat
Coriandrum sativum L. (Cultivated)	Coriander /Dhaniya
Cyperus rotundus L.	- /Mothe
Eucalyptus camaldulensis	- /Mashala
Roeniculum vulgave (Mill.)(Cultivated)	Fennel /Saunp
Gaultheria fragrantissima Wall.	Wintergreen /Dhasingare
Lichens sps	Moss /Jhyau
Pinus roxburghii Sargent (syn: P. Longifolia Roxb.)	Chirpine /Ranisalla
Pinus wallichiana A.B. Jacks. (Syn: P. excelsa Wall. ex D. Don)	Blue pine /Salla
Selinum tenuifolium Wall. ex C.B. Clarke	- /Bhoot kesh
Tsuga dumosa (D. Don) Eichler	Hemlock / -
Zanthoxylum armatum DC.	- /Timur
Zingiber officinale Roscoe (Cultivated)	Ginger /Aduwa
Species less abundant:	
Carum carvi L.	Caraway /-
Cedrus deodar (Roxb.ex D. Don)G.Don	Himalayan cedrus/Deodar
Chenopodium ambrosides L.	-
Citrus aurantifolia Swingle	Lime /Kagati

Citrus limonum (L.) Burm. f.	Lemon	/Nibuwa
Cuminum cyminum L.(Cultivated)	Cumin	/Jeera
Cymbopogon winterianus Java de Tong (Cultivated)	Citronella Java	/-
Cymbopogon martini Stapf.var. motia (Cultivated)	Palmarosa	/-
Cymbopogon flexuosus (Nees.) Stapf. (Cultivated)	Lemongrass	/-
Elsholtzia flava Benth	-	
Elsholtzia fruiticosa (D.Con) Rehder	-	
Hedychium spicatum Buch.-Ham.ex.J.E. Smith	-	
Iris nepalensis (non Wall., 1824) D. Don	-	
Jasminum officinale L.	Jasmine	/-
Junifperus communis L.Var.Saxatilis Pall.	Juniper	/Dhoopi
Juniperus recorva Buch.-Ham.ex D.Don	Juniper	/Dhoopi
Litsea cubebe (Lour.) Pers.	-	
Mangolia grandiflora L.	-	/Rukh Kamal
Mentha arvensis L. (Cultivated)	Japanese Mint	/-
Mentha piperita L. (Cultivated)	Peppermint	/-
Mentha spicata L. (Cultivated)	-	
Nardostochys grandiflora DC (Syn: N. jatamansi DC)	Nard spike	/Jatamansi
Ocimum basilicum L. (Cultivated)	-	
Origanum vulgare L.	-	
Origanum majorana L.	Marjoram	/-
Osmanthus fragrans Lour.	-	
Piper longum L.	Long Pepper	/Pipla
Rhododendron anthopogon D. Don	-	/Sunpati
Skimmia laureola Hook f.	-	
Saussurea lappa clarke	-	/Kut
Tagetes minota L. (Cultivated)	-	/Sayapatri
Thymus serpyllum L.	-	
Valeriana hardwickii Wall.	-	/Nakali Jatamansi
Valeriana jatamansi Jones (Syn: V. Wallichii DC)	-	/Sugandhawal
Vetiver zizanioides Stapf.	-	/Khas khas
Viscum album L.	-	

Trade in Essential Oils/Fragrances

The export and import statistics of essential oils, perfumes and flavour materials for the period 1985-1989 are recorded in Table 21.

Table 21 Nepal export and import statistics of essential oils and related materials (1985-1989)

Year	**Export** **Value in N Rs.x1000**	**Import** **Value in N Rs.x1000**
1985	224	9,104
1986	-	8,968
1987	5	11,595
1988	332	11,724
1989	1,839	47,634

Source: Foreign Trade Statistics, Department of Customs
His Majesty's Government of Nepal.

Current Exchange rate: 1 US$ = N Rs. 30 (Approximately)

The trade statistics unfortunately does not specify the name of essential oils and thus makes it difficult to ascertain the type of oils Nepal imports. It is evident from the data given in the above table that the import of essential oils fragrance materials is increasing over the years. Compared to the import figure, the export is not much. A major portion of the above import is made from Benelux and India. Other countries from where the essential oils, perfumery and flavouring materials are imported include U.K., Singapore, West Germany, France, U.S.A., China, Hongkong and Japan.

Names and addresses of leading trading houses/corporations:

1. Herbs Production and Processing Co. Ltd.
   Koteswore, Post Box No.2679
   Kathmandu, Nepal
   Phone: 472232

2. Nepal Rosin and Turpentine Factory
Attariya, Dhangadhi
Kailali, Nepal

Industrial Production

Types of essential oils/fragrances produced locally.

Thirteen essential oils are produced in Nepal.

Viz. Calamus oil
Wintergreen oil
Sugandhakokila oil
Timoor oil
Nardspike oil
Turpentine oil
Mentha oil
Lemongrass oil
Citronella oil
Palmarosa oil
Eucalyptus oil
Lichens resinoids.

The essential oil industry in Nepal is at its infancy. Two major industry in the public sector and a few local distillation units in private sectors are in operation. One of the major industry i.e. Rosin and Turpentine factory - established with the assistance provided by USSR - processes only the pine resins and produces about 750 tons of turpentine oil per annum. The other industry, Herbs Production and Processing Co. Tld (HPPCL) - an offspring of the Department of Forestry and Plant Research (formerly known as Department of Medicinal Plants) and later assisted by UNDP/UNIDO - concentrates more on other essential oils. Production data of HPPCL has been summarized in Table 22. HPPCL has started to build a network for processing aromatic plants at different sites of the country. It is important particularly when one considers the difficult mountainous topography of the country where a single factor, transportation cost quite often becomes limiting factor to distill a number of otherwise potential aromatic plants. However, such difficulty is much lessened in the case of cultivated aromatic plants in which case a better organisation in production is possible. Amongst the products of Nepal at present Palmarosa oil (6 tons) tops the list (excluding turpentine oil). Annual production of other oils are: Sugandhakokila oil (3 tons), Citronella

Java oil (3-4 tons), Lemongrass (2 tons), Mentha arvensis oil (1 ton), Wintergreen oil (1 ton).

Considering the fact that the essential oils as a commodity was unknown in Nepal till few years ago, the introduction, collection/cultivation, production and sale of this item though in a small scale points out a positive sign for the future expansion of essential oil industry in the country.

Table 22 Sales of essential oils produced by HPPCL

Year	Quantity (Kg)	Value in N Rs x1000
1982	805	10
1983	1,432	32
1984	3,748	155
1985	8,274	615
1986	25,448	1,626
1987	17,407	2,403
1988	32,994	3,370

Source: Marketing Division, Herbs Production and Processing Co., Ltd., Koteswar, Kathmandu.

Exchange rate: 1 US$ = N Rs 30 (Approximately)

The distillation units which are used in the processing of aromatic plants are mostly imported from India, France and U.K. Attempts have been made in the past to fabricate distillation units locally. A number of workshops in the country are capable of constructing and maintaining the distillation units. In fact a couple of such units made of stainless steel were constructed. But the demands of such units does not attract the attention of these workshops at present.

R & D Institutions involved in the Industry

Institution	Address	Contact Person	Main Activities
1) Royal Drug Research Labo-	P.O.Box No.2270 Thapathali	Dr.S.R. Adhikary	1.Screening of wild flora

ratory	Kathmandu, Nepal		2. Phytochemical reserach. 3. Improvement of processing Technology (Pilot plant). 4. Quality assessment of essential oils.
ii) National Herbarium and Plant Laboratories	Godavari Lalitpur, Kathmandu, Nepal	Dr. S.B.Rajbhandary	1.Plant identification. 2. Resource assessment. 3. Tissue culture 4. Pathology
iii) Herbal Farms located at different sites of the country	Herbal Farms Centre Thapathali Kathmandu Nepal	Mr. M.S. Bista	1.Introduction and experimental farming of aromatic plants. 2. Germ plasm maintenance 3. Extension.

Institutions/Firms Utilizing Fragrance Materials

Name of Institution/Firm	Industry	Quantitative Estimate	Sources of Fragrance Materials	
			Imported	Locally Formulated
i. Royal Drugs Ltd.	Pharmaceutical	760 kg	410	350 kg
ii. Jenson & Nicholson (Nepal) Pvt.Ltd.,	Paints	-	-	-
iii. Asian Paints	Paints	-	-	-
iv. Surya Tobacco Co. Pvt. Ltd.	Cigarettes	-	-	-
v. Nepal Beverages and Food Products Ltd.	Fruit, Jam Squash etc.	-	-	-

vi. Rijal Tashi Industries Pvt. Ltd.	"	-	-	-
vii. Nepal Liquor Pvt. Ltd.	Distillery	-	-	-
viii. Nepal Thai Foods Pvt. Ltd.	Noodles	-	-	-
ix. CTL Pharmaceutical (Pvt.) Ltd.	Pharmaceuticals	-	-	-
x. Deurali Janata Pharmaceuticals	"	-	-	-
xi. Hoechst (Nepal) Pvt. Ltd.	"	-	-	-
xii. NEBICO	Biscuits, contectionery	-	-	-
xiii. Gorkha Biscuits Co.	"	-	-	-
xiv. Trisakti Soap & Chemical Ind. Pvt. Ltd	Soap	-	-	-
xv. Mahasakti Soap Industries	Soap	–	-	-
xvi. Gorkha Brewery Pvt. Ltd.	Beer	-	-	-
xvii. Mount Everest Brewery Pvt. Ltd	"	-	-	-
xviii. Everest Tooth Paste Co.	Tooth paste	-	-	-

<u>Any other relevant Information</u>

As mentioned earlier, essential oil development in Nepal is relatively a new venture and within a short span of time it has shown promising results. The developmental work initiated by the Department of Forestry and Plant Research is accelerated by the technical and material assistance provided by UNDP/UNIDO under the project NEP 80/003 which is now at its terminating stage. Research and development capability is considerably increased and now the country is in a position to initiate works in the area of resource assessment, cultivation, screening of wild flora, phytochemical investigations of aromatic plants and quality assessment, production and marketing of essential oils. While it is important to continue and further develop this already acquired capability, there still remains a lot more to be done. In this respect we welcome assistance and co-operation in the following area of essential oil development.

i. Know-how in odour assessment technique

ii. Fractionation and conversion of isolates into other important compounds.

iii. Product development and fragrance formulations.

iv. Improvement in field distillation specially for fuel economy.

v. Improvement of the planting materials for higher yields.

INFLUENCE OF SOME FACTORS ON THE FORMATION OF BY-PRODUCTS IN THE SYNTHESIS OF PSEUDO-IONONE

DO LINH CUONG

Pseudo-ionone is obtained as an intermediate product in the manufacturing process of ionones. It is formed by the condensation of the olefenic terpene aldehyde citral with acetone under the action of various reagents.

This condensation was mentioned by Tiemann in 1893. In 1923, Rene Sornet (1) carried out this reaction in a large scale with sodium hydroxide and an excess of acetone. According to Budnitskata (2), a reaction yield of 30-40% is obtained when this condensation occurs in the presence of glycerine. The Dupont's patent suggested the use of monochloracetone as a catalyst at low temperature (3).

Previously, in some countries, most of the commercially manufacturing process used alkaline reagent with the concentration of 40-45% and at a temperature of 25 to 30°C.

Recently, the use of low concentration of alkali is preferred, but at higher temperatures.

In 1963, Bogacheva reached a high yield by using 2-5% alkaline concentration at 50°C (4).

In brief, the research studies on the synthesis of pseudo-ionone from citral and acetone have mentioned the substantive use of alkaline reagents at various concentrations and temperatures. Under these different conditions, different kinds of by-products are formed. The separation and re-use of these by-products are economical problems.

In this paper, concern is made for the influence of th sodium hydroxide concentration on the formation of by-products in the synthesis of pseudo-ionone from citral and acetone.

Experiments were carried out in a stirrer vessel with temperature control. The reaction mixture is distilled at reduced pressure to separate the various fractions. The products were then analysed by SVET 100 gas chromatograph.

Based on the amount of unreacted citral and the amount of reacted citral and formed pseudo-ionone, we figure out the amount of citral used for forming by-products.

Table 9: Influence of NaOH concentration on the formation of by-products at 25°C.

Concentration of NaOH (%)	1	5	15	25	35	45	55
% Citral used to form by-products	3,2	7,5	15,1	20,2	26,8	32.4	38,3

With constant concentration of sodium hydroxide, reaction temperature is altered and the influence of temperature on the formation of by-products is observed.

Table 10: Influence of alkaline reagent on the formation of by-products at various temperatures.

	Temperature °C				
% Citral	20	30	40	50	60
NaOH concentration of 4%	6,4	7,8	9,8	12,7	15,2
NaOH concentration of 40%	26,4	30,2	34,3	39,1	45,3

The following conclusions can be made from the above observations:

1. Comparison between a reaction with low alkaline concentration and another one with high alkaline concentration showed the same yield of pseudo-ionone:

If the reaction mixtures are distilled at reduced pressure to receive the same low boiling point fraction, the quantity of fraction in the first reaction is much more than in the second one.

At low alkaline concentration, a chain rupture of citral molecule is thought to have occured. Based on the boiling points of the fraction in distilling, methylheptenone is considered as the main by-product resulting from this rupture.

2. After pseudo-ionone fraction is distilled out completely, an adhesive gum residue is obtained at the bottom of the distilling flask. For a same yield of pseudo-ionone, the higher alkaline concentration is used, more residue is obtained. Besides the condensation of citral molecules themselves, another condensation might occur between 2 mols of citral and a mol of acetone, resulting in the formation of dicitrilideneacetone.

3. Comparison between a reaction with low alkaline concentration, at high temperature and another one with a high alkaline concentration, at low temperature:

   With a low alkaline concentration (about 3-5%) at high temperature (abount 50-60°C), less by-products are obtained than expected in a process with high concentration at low temperature.

   In brief, the mentioned alkaline reagent affects the condensation reaction of citral with acetone in the formation of different types of by-products. It is therefore recommended to select the most suitable catalyst in order to decrease the amount of by-products and subsequently to increase the amount of pseudo-ionone.

REFERENCES

1. Rene Sornet, Technique Industrielle des Parfumes synthetiques. 70 Paris (1923)
2. E.V. Bunitskaja, Chemical Abstracts. Vol 44, 5849 (1950).
3. William Poucher, Parfumes, Cosmetiques et Savons. Tome 1, 95, Paris (1951).
4. K.T. Bogacheva, Chemical Abstracts. Vol 58, 4413, (1963).

Aromatic Plant Resources of the Country:

Local Name	Botanical Name
- **Species most abundant:**	
Java citronella	Cymbopogon winterianus
Turpentine	Pinus Spp.
Japanese mint	Mentha arvensis
Star anise	Ilicium verum
Cinnamon	Cinnamomum loureirii
Eucalyptus	Eucalyptus citriodora
- **Species less abundant:**	
Lemongrass	Cymbopogon citratus
Litsea	Litsea cubeba
Vetiver	Vetiveria zizanoides

Trade in Essential OIls/Fragrances.

- Imports: fragrances from Givaudan, Quest, Kelkar Co.
- Exports: essential oils, mainly Java citronella oil
- Names and addresses of leading traoing houses/corporations:

Imexco	23 Nguyen Hue St. HoChiMinh City
Generalimex	342 Ben Chuong Duong HoChiMinh City

Industrial Production:

- Types of essential oils produced locally: citronella oil, cinnamon oil, Japanese mint oil, Turpentine oil, Eucalyptus oil, Star anise oi, Cajeput oil.
- Equipment used: locally fabricated.

R & D institutions involved in the Industry:

Institution	Address	Contact Person	Main Activities
Essential Oil Centre	124 To Hien Thanh Street HoChiMinh City	Pr. Dao Van Luong	Research on essential oils
Enteroil	Nghia Do, Tu Liem, Ha Noi	Dr. Tran Trong Vong	Producing essential oils

Institute of Chemistry	2 Mac Dinh Chi Street HoChiMinh City	Pr. Ho Si Thoang	Analysis and synthesis of essential oils

Institution/Firms Utilizing Fragrance Materials:

Name of institution	Industry	Quantitative estimate	Source of Fragrance Material	
			Imported	Locally Formulated
South Vietnam Detergent Company	Detergent, soap	100 tons/year	x	x
LIPACO	Cosmetic	40 tons/year	x	
VINABICO	Food	80 tons/year	x	
Saigon Tooth paste Enterprise	Toothpaste	50 tons/year	x	

SICHUAN LEMON BASE CONSTRUCTION AND PROCESS

DR. NIE SHU CHENG

In China, Lemon has been cultured for sixty years and there were just a few planted in the suburbs of Chongquing and Chengdu in 1920. Eureka Lemon was introduced by the U.S. in the later part of 1938. In 1950, due to local demand a large area of Sichuan was palnted with Lemon. Sichuan is the main plantation area for citrus in China and considered as the most adaptive area for citrus growing in the world. Since then, lemon was one of the major citrus fruits exported to other countries which started in 1960. Around 860 tons of fruits were produced in 1965 and only a small quantity of fresh lemons were being sold and the rest were utilized in the production of essential oils whereby we succeeded in the development of the extraction equipment and its process. Hence, cold-ground lemon oil utilized for aromatic indsutries leads to the increasing demand of lemon oil. In 1970, the Ministry of Light Industry invested in the expansion of the lemon plantation to provide the aromatic industries with enough fragrance materials. Up to now, 2.6 million of lemon trees have been planted in Sichuan and some of the trees begin to bear fruits. Yields in 1988 and 1989 are 4,000 tons and 5,500 tons of fruits respectively. It is estimated that yield will be 7,000 tons in 1990 and 80% of the total production will be processed into essential oils and the rest are marketed as fresh fruits. The oils that were produced in 1989 is 12 tons and the demand for these oils increases year by year. The local demand is around 70 tons per year which local manufacturers cannot provide, so importation of these oils is needed. Lemons are chiefly utilized for oil processing in China as compared with other developed countries. The rest of its parts like the juice, seed and pulp are considered as waste products which pollute our environment.

We have systematically conducted researches on the cultural aspects of the plant, harvesting techniques, process of extraction and the suitable equipment needed for higher yield of essential oil. With ZH79 volatile oil apparatus which is being fabricated by our Institute, we were able to determine the content of essential oil from the fresh fruits which is 5.0-5.7

kg per ton. This yield can be attained if the fruits are harvested during the 10th to 30th November. Based on these results, we processed the fruits on the said period. We also make use of a fabricated grinder and centrifuge (made in Hangzhou) to obtain cold-ground lemon oil. When processing, we took care in the treatment of fresh fruits prior to grinding. These include the volume of spraying water, grading of the fruit according to size and soaking of the fruits with lime water at pH 12 for 2 hrs. and the time required for grinding. The purpose of soaking the fruits with lime water is to increase the presence of peel cells to eleocytes so that essential oil will spurt when punctured.

The fruits are then washed with water after treatment with lime water. In grinding the fruits, one ton of spraying water at pH 7.0-7.5 is used for every hour per one ton of fruit and 25 kgs. of fruits are ground for 20 secs. to 3 mins. depending on the size. There were some modifications made in the old grinder to improve its efficiency. A sparse prong seta plate was utilized instead of a tetra bevel plate. It is a single grinding plate and the rate was increased up to 90%. The cost of this type of grinder is 17% cheaper than that of the old type. Percent recovery of oil also increases (3.5-4.5%). Furthermore, studies were also made on the utilization of the juice, peel and seeds. These were applied in the manufacture of jam strips, jellies, beverages, edible oils, cosmetics, etc. These products are widely accepted by the local consumers and are sold well. Lastly, studies on the lemon pectin is being carried out in our institute.

Workshop made the following recommendations addressed to UNIDO and the Governments of member states

	Addressed to UNIDO	Addressed to Governments
1. **HUMAN RESOURCES DEVELOPMENT**		
Human Resources Development in all the major facets of the industry is considered a necessity for developing countries.	+	+
To this end, UNIDO should take steps to continue initiating and sponsoring symposia, workshops, in-plant group training programmes specifically related to the following areas: a. Agrotechnology b. Process technologies, i.e. extraction, distillation, fractionation, derivatization c. Quality Assessment d. Sensory methods of compounding and formulation practices.		
2. **INFORMATION AND PUBLICATIONS:**		
a. Updating of the previously published UNIDO Publication titled "Information Sources on Essential Oil Industries", which should include aspects such as equipment and manufacturers, listing of producers, producing countries & markets, journals, books, reports, etc. A directory on research establishments/institutions and experts.	+	
b. Publishing of practical manuals related to specific aspects of the industry such as: i) fractional distillation ii) sensory evaluation & formulation methodology of flavours and fragrances iii) standard design options for field distillation and fractionation distillation units iv) essential oil derived isolates, their synthetic modifications and analogues		
c. Developing video or computer-aided courses on various aspects of the industry		

	Addressed to	
	UNIDO	**Governments**
3. RESEARCH AND DEVELOPMENT		
- R & D efforts of developing countries need to be reinforced and strengthened in order to sustain national industrial development in this sector	+	+
- Initiation of a programme of systematic search for new plant-derived fragrances and utilization of underutilized or non-utilized aromatic plant resources.		
- Steps should be taken to encourage and assist wherever feasible the development of herbaria and (collection of live plants) industrially utilizable aromatic plants in each developing country.		
- Creation of a network R & D institutions which includes facilities for processing of aromatic plants and quality control, etc. was recognized as a desirable feature in promoting Technical Cooperation among Developing Countries (TCDC).		
- Utilization and Management of Waste		
4. POLICY INITIATIVES		
Government initiatives were considered necessary in respect of the folloiwng aspects so as to sustain this socially relevant rural employment oriented industry		+

- safeguarding endangered species
- preventing indiscriminate exploitation of the spontaneous flora
- encouraging systematic crop-wise cultivation
- setting up of an essential oil promotion and development board
- instituting export promotion incentives with respect to value-added products which have hitherto been exported in the raw form
- provision of assistance to entrepreneurs in order to develop small and medium scale industries

Annex A

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4-8 February 1991
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