

**THE DESIGN, CONSTRUCTION AND TESTING OF A MOBILE  
ESSENTIAL OIL DISTILLATION UNIT**

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## ABSTRACT

Steam distillation is the most widely accepted process for the large scale production of volatile essential oils from herbaceous material and is also regarded as the standard practice throughout the flavour and fragrance industry. A mobile essential oil distillation unit for the extraction of oils from herbaceous materials would be extremely valuable to the essential oil industry in South Africa. Using a mobile platform, the extraction technology could be taken to rural areas where essential oil crops are grown in order to extract and then analyse the oils produced. Existing systems in South Africa are static distillation units which are usually owned by commercial growers that are generally positioned large distances away from the rural areas.

The objective of this project was to design, construct and test a prototype mobile distillation unit for the extraction of essential oils from herbaceous materials. The unit was to have a charge vessel capacity of approximately 250 kg of plant material and should be able to perform in-field distillations in areas where electricity is not available. A literature review on all the essential oil extraction methods, the theory behind steam distillation and oil isolation and the effect that each of the distillation components have on the distillation process was performed. A small test distillation unit was set up in a laboratory in order to investigate the effects of varying steam flow rates on the distillation time, oil yield and oil quality.

A double charge vessel unit was designed, constructed and mounted onto a frame which in turn was fixed onto a trailer to be hauled by a light delivery vehicle (LDV). The steam generator with all its ancillary equipment was fixed onto a separate trailer. The unit could thus be easily transported and in-field distillations on various crops could be conducted.

Field distillations were conducted with two crop types, namely rose geranium and lemon grass. Satisfactory results were obtained as the oil yields were within the expected oil yield range.

I wish to certify that the work reported in this dissertation is my own unaided work except where specific acknowledgement is given. In addition I wish to declare that this dissertation has not been submitted for a degree at any other university.

Signed:  \_\_\_\_\_

C.E. Talanda

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## 1. INTRODUCTION

Essential oils can be given a simple definition as the predominantly volatile and odorous fraction isolated by some physical process from vegetative materials (Latta, 1999). They are used as sources of aromatic and flavouring chemicals in food, industrial, and pharmaceutical products. The oils are largely composed of terpene hydrocarbons with their oxygenated derivatives, benzenoid compounds and also compounds containing nitrogen and sulphur, which are not that common (Charles and Simon, 1990). The differences in aroma among plant types result from differences in the volatility and quantity of certain chemicals in the volatile oil (Veljkovic and Stankovic, 1993). The essential oil compositions of plants vary due to genetic and environmental factors that influence genetic expression (Bernath, 1986) and the composition also varies with the developmental stage of the plant and extraction methods (Burbott and Loomis, 1967).

Among all types of plants in the world, only about 700 plants are considered aromatic, and are therefore significant for the production of essential oils. Besides the limited source of supply, the small volumes of essential oils that are contained in each aromatic plant make the oil even more valuable (Davis *et al.*, 1997). The yield of essential oils from plants varies widely and the broad range is 0.05 – 18.0 % of mass, with most oil yields less than 1 % (Sankarikutty and Narayanan, 1993). Essential oils are thus generally high value, low volume commodities, making them attractive crops to grow and process for smallholder farmers and farmers in remote communities where transport problems prevent them from marketing high volume cash crops (Axtell and Fairman, 1992).

The extraction procedure is the key controlling step in obtaining the best quantity and quality of essential oils (Davis *et al.*, 1997). Factors such as the type of plants, chemical make up of oil, location of oil in the plant (root, bark, wood, branch, leaf, flower, and/or seed) all need to be considered prior to extraction. Due to the low yield and high value of the oil it is of vital importance that the proper extraction method is used in order to extract as much oil as possible from the plant material. It is necessary to process most of the plant material rapidly after

harvesting, as oil could be lost due to evaporation once harvested (Sankarikutty and Narayanan, 1993).

South Africa has a diverse range of climatic areas that suit a large variety of oil bearing plants. The country also accommodates a range of indigenous plants from which essential oils are produced that are used internationally. Essential oil production in South Africa is not only suitable for commercial farming, but is also ideally suited to emerging farmers in rural communities requiring economic upliftment. Research and training programmes for the production of essential oil crops by emerging farmers in rural areas have been established, but one of the major barriers is the unavailability of extraction technology required for the extraction and evaluation of the oils produced in these areas.

Therefore, the main objective of this project, which was proposed and funded by the Agricultural Research Council–Institute for Agricultural Engineering (ARC-ILI), was to design, construct and evaluate a mobile essential oil distillation unit that can be used to extract essential oils from herbaceous materials. The unit had to be mobile so that it can be transported to the areas where the essential oil crops are produced. Further objectives were that the unit should have a plant charge capacity of approximately 250 kg, an efficient mechanism for loading and removing plant material from the vessel and should be able to operate in areas where electricity is not available.

In this dissertation a review of the various essential oil extraction techniques focussing primarily on the theory of oil isolation by steam distillation and on the components of a steam distillation system is contained in Section 2. The assembly and testing methodology of a small laboratory distillation system used to investigate various distillation parameters, the design, construction and assembly of the mobile distillation unit components and the field testing methodology of the unit are described in Section 3. The results obtained from the laboratory distillations and the field distillations conducted with the mobile unit are presented and discussed in Section 4. The dissertation is then ended with conclusions and recommendations for future research contained in Section 5.

## 2. ISOLATING ESSENTIAL OILS FROM PLANT MATERIAL

Essential oils are situated in oil sacs in various plant components and are isolated by comminution (shredding) and the action of heat, water and solvents. Distillation, selective solvent extraction, and mechanical expression are the three general basic methods employed for essential oil isolation. Carbon dioxide extraction and enflourage are two further methods used. Each of these isolation methods will be discussed in this section.

### 2.1 Solvent Extraction

Solvent extraction is used for recovering essential oils consisting of non-volatile compounds (Burbott and Loomis, 1967). A fresh solvent is circulated through an extraction vessel into which the plant material is placed. The solvent draws out the essential oil from the plant material and after each cycle it becomes more enriched with the plant volatiles until extraction is complete (Lawrence, 1995).

The solvents are then evaporated to leave a thick residue known as concrete. Concretes can be used in household care products but are converted into an alcohol soluble volatile concentrate, known as an absolute when used in perfumery. The solvents used should have low boiling points, be free of odour and impurities and should also be inert towards the oil constituents. Examples include butane, pentane, hexane, petroleum ether (60-80 °C), benzene and toluene (Lawrence, 1995).

This method is preferred by perfumers as solvent extracted oils are generally considered to reflect the natural odour of the plant more accurately than other extraction methods. For aromatherapy use, oils produced in this way have the disadvantage that some of the solvent will remain in the oil (Weiss, 1997).

## **2.2 Expression**

This method is also known as cold pressing. It is commonly used in the production of oils from citrus rinds such as lemon, orange, grapefruit, tangerine and mandarin. The rinds of the fruits are chopped, then ground and pressed to force out the essential oil components found in the skin, producing a mixture of both juice and oil concurrently. The mixture is then allowed to settle and the collected oil is then washed with a spray of water and then separated by means of a centrifuge (Sankarikutty and Narayanan, 1993).

## **2.3 Carbon Dioxide Extraction**

Carbon dioxide expression is a high-pressure extraction method with supercritical or liquefied carbon dioxide. The high pressure causes the carbon dioxide to liquefy and in this form it has the ability to form a solution with the volatile components of the plant material. The solution is then drained off and de-pressurized, leaving behind a deposit of pure essential oil.

It is a relatively new expensive technique for obtaining essential oils without thermal or hydrolytic deterioration. An advantage of this method is that the product obtained from this method has no signs of solvent residues (Sankarikutty and Narayanan, 1993). This extraction process is especially suited for the extraction of oils with low molecular weights (Lawrence, 1995).

## **2.4 Enfluerage**

This technique is used for producing aromatic oils from flowers that contain minute quantities of aromatic compounds and that are too delicate to withstand other processing conditions such as heat and steam (Lawrence, 1995). Layers of flowers are laid on trays of specially prepared fat, which absorbs the small quantities of oil from the petals. The flower layers are removed and renewed until the fat is saturated with oil. This mixture of fat and oil is known as pomade. The oils are then extracted from the pomade with an alcohol solvent (solvent extraction). This method

is highly labour intensive, but the products are of extremely high quality, and the technique will continue to be used while manufacturers are prepared to pay a premium for its products (Weiss, 1997).

## **2.5 Distillation**

The most popular physical method for the isolation of volatile essential oils is distillation. With distillation, the plant material is placed in a distillation vessel and steam is passed through the plant material. As the steam condenses on the plant material, the oil glands are ruptured due to the release of its latent heat. The molecules of these volatile oils then escape from the plant material and evaporate. Once the vapour containing both water and the volatile oil compounds is condensed, the oil either floats on top of the water or sinks to the bottom, depending on its density, and can thus be separated (Lawrence, 1995).

The essential oil of a plant consists of many compounds that generally boil between 150 °C and 300 °C. If attempts are made to remove these compounds by dry distillation, many will decompose and the oil will be ruined. The compounds are however steam volatile and can be distilled out of the vegetal materials at below 100 °C as explained in Section 2.6.2 (Axtell and Fairman, 1992).

The preparation, the packing of the material into the charge vessel and the rate/type of distillation must be determined for the particular essential oil crop (Axtell and Fairman, 1992). It must be noted that the balance of plant constituents varies from day to day as the plant develops further and that there is not only one profile for a particular oil (Barson, 2002). Variables such as the plant variety, geographical origin, climate, soil type, rainfall, period of harvest, moisture content and distillation conditions also have an effect on the oil content and chemical composition (Guenther, 1948).

There are four basic types of essential oil distillation that will be reviewed:

1. Hydrodistillation
2. Water and steam distillation

3. Steam distillation
4. Vacuum distillation

### **2.5.1 Hydrodistillation**

Hydrodistillation is the most commonly used method in rural areas, as it is simple and inexpensive (Weiss, 1997). The principle of this form of distillation is to boil a suspension of an aromatic plant material in water. The vapour then passes through a condenser and the oil, which is immiscible with the water, is then separated (Lawrence, 1995). Most of these distillations are performed in rural areas where no access to a steam boiler is possible. The temperature in the still body is raised to boiling by direct firing often using spent residues. In stills where the water is boiled by direct contact with the fire, the water present in the still must always be in excess to last throughout the distillation; otherwise the plant material can overheat and burn (Guenther, 1948).

The quality of the produced oil is directly related to the skill of the operator, not only in managing the still, but also in selecting and preparing the raw material (Weiss, 1997). Oils produced by this form of distillation tend to have a lower quality than oils produced by the other methods for the following reasons (Lawrence, 1995):

- Oxygenated compounds like phenols have a tendency to partially dissolve in the still water so their complete removal by distillation is not possible.
- Some of the oil components like esters are sensitive to hydrolysis while others like acyclic monoterpene hydrocarbons are susceptible to polymerization.
- As hydrodistillations tend to be small, it takes a long time to accumulate significant quantities of oil. Hence good quality oil is often mixed with bad quality oil, thus reducing the grade.
- Water distillation is a slower process than the other methods and is consequently less energy efficient.

### **2.5.2 Water and steam distillation**

This is an improved method, in that the charge vessel contains a perforated grid which keeps the plant material above the water level. This reduces the capacity of the still but affords a better quality of oil. The water is boiled below the grid and the wet steam passes through the plant charge. Like hydrodistillation, it is widely used in the rural areas as it does not require more capital expenditure than water distillation. The design of the equipment is also very similar to the water distillation equipment (Lawrence, 1995).

It is important in both water/steam and steam distillation (discussed below) that the plant material is evenly and not too tightly packed so that the steam can move uniformly through the plant material. Over packing of the still can cause the steam to force holes through the plant material and hence parts of the material will not be exposed to steam. Consequently the volatile compounds will remain in the plant material in these areas and hence a low yield will be obtained (Axtell and Fairman, 1992).

### **2.5.3 Steam distillation**

This is the most advanced type of distillation where steam is provided directly from a separate boiler and has been shown to be more efficient than water/steam distillation (Whish, 1996). Again the plant material is supported by a perforated grid under which the steam is injected through a steam manifold. An advantage with a satellite steam generator is that the steam flow rate can be readily controlled and the temperature of the steam in contact with the plant material will be no higher than 100 °C, when the steam is not superheated. The heat with which the plant material will come into contact is acceptable and should not cause any thermal degradation of the essential oils distilled (Lawrence, 1995). Further advantages of this type of 'dry' steam distillation are that the distillation times are faster, energy consumption is also lower than the other distillation methods and steam is also available rapidly and continuously after starting up (Axtell and Fairman, 1992).

Steam distillation is the most widely accepted process for the production of essential oils on the large scale and is also regarded as the standard practice throughout the flavour and fragrance industry. The only drawback of steam distillation is the high capital expenditure needed to build such a system (Lawrence, 1995).

A further form of steam distillation is where the steam enters the still at the top and passes down through the charge. Oil and water vapour condense on coils fixed below the bottom grid within the still and oil and water are then separated in the usual way. With the steam moving in the same direction as the liquid flow, its ability to vaporise the oil is impaired as the lower levels of the charge tend to become flooded by freely moving surface liquid (Denny, 1999).

Experiments carried out by Mr A. E. Dann at the Natural Resources Institute in the U.K., show this type of steam distillation uses greater quantities of steam and returns smaller oil yields (Denny, 1999). This method has however been found to be suitable for distilling dry materials where problems associated with plant juices are minimal, and also for seed oils (Sankarikutty and Narayanan, 1993).

#### **2.5.4 Vacuum distillation**

Vacuum distillation obtains oil from plant material under a vacuum varying in intensity depending on the material. This technique allows very accurate control of distillate since it can be adjusted according to the boiling points of the various oil constituents (Weiss, 1997).

This type of distillation is usually adopted when redistilling oils to improve a particular property or concentrate a particular fraction of the oil i.e. rectifying oils. It is very rarely used to distil oil directly from the plant material (Sankarikutty and Narayanan, 1993).

Since steam distillation is the most popular and widely accepted process and also regarded as the standard method by the flavour and fragrance industry, it will be investigated further. Before discussing the way in which the oils are isolated from herbaceous materials by steam distillation,

it is essential to look at the laws and the thermodynamic properties that govern the isolation process.

## **2.6 Fundamentals of Steam Distillation**

When distilling oils from plant materials, the process is mainly influenced by heat, particularly latent heat, and also by pressure, specifically vapour pressure. Any relationship that exists between these two properties is important and it is thus necessary to discuss these two subjects, as well as summarise some of the laws related to them:

- The First Law of Thermodynamics states that:  
“Energy (heat) cannot be either created or destroyed in a closed system” (Lawrence, 1995).
- The Second Law of Thermodynamics states that:  
“Heat cannot be transferred from a cooler system to a hotter system by any self sustaining, continuous process” (Lawrence, 1995).
- Fourier’s Law of Heat Conduction states that:  
“Heat will be conducted from one plane surface to another at a rate proportional to the area of contact and to the magnitude of the temperature difference between the two surfaces” (Denny, 1999).
- Fick’s Law of Diffusion states that:  
“The rate that a substance will pass a unit area in a unit time as it diffuses through a substance, is proportional to the negative concentration gradient of the substance” (Denny, 1999)

### **2.6.1 Latent heat**

The molecules of any liquid are in continuous motion at speeds dependant on temperature. The amount of energy or heat required per unit mass, to vaporise the liquid is known as the latent heat of vaporisation. If any vapour is condensed back into the liquid state, the energy of the molecules

is correspondingly reduced, and the characteristic quantity of latent heat is given out (Lewin, 1963).

### 2.6.2 Vapour pressure

Within a temperature controlled, constant volume, closed system the molecules in the vapour phase above a liquid will reach a point where they are in equilibrium with those in the liquid phase. When the temperature increases, the number of molecules in the vapour phase increase, and as a result the vapour pressure increases. When the system is in equilibrium, the vapour is saturated and the pressure exerted by the vapour is termed the saturated vapour pressure. If the volume of a closed system is then increased while the temperature remains constant, the number of molecules in the vapour state will increase. A slight cooling because of the latent heat of vaporization and a corresponding reduction in vapour pressure will occur (Denny, 1999).

The latent heat that is required for evaporation to occur is taken from the liquid body, and if this energy is not replaced, the liquid will cool, the vapour pressure will reduce and hence the rate of evaporation will decrease. The rate of evaporation can be kept constant if heat is supplied from an outside source and is equal to the amount of heat lost, resulting in a stable temperature and hence stable vapour pressure (Denny, 1999).

From the discussion above it can be deduced that the saturated vapour pressure of a specific liquid at a certain temperature is the boiling point of that liquid, when the surrounding pressure is the same as its saturated vapour pressure (Denny, 1999). If the surrounding pressure is higher than the saturated vapour pressure, the temperature of the liquid will need to be raised to increase its vapour pressure to match the surrounding pressure for the liquid to boil and evaporate.

The boiling temperatures of most essential oil components exceed that of water due to their low vapour pressures. When a mixture of two or more immiscible liquids occurs, the boiling point for the mixture of the immiscible liquids is when the sum of their vapour pressures is the same as the surrounding pressure. For example Linalool, which is a compound present in various essential oils, has a vapour pressure of 3.706 kPa at 99.5 °C, while at the same temperature water has a

vapour pressure of 97.619 kPa. As a result the mixture will exist at a vapour pressure of 101.325 kPa, which is the standard atmospheric pressure (Lawrence, 1995).

A sample of Linalool will usually boil at a temperature of 198 °C and similarly water will boil at 100 °C at atmospheric pressure. When the two immiscible samples coexist, and once their vapours have reached saturation, the boiling temperature will drop to 99.5°C. Heterogeneous liquids thus boil at a temperature below the boiling point of the lowest boiling constituent (Lawrence, 1995).

When the liquid is completely evaporated and more heat is applied, the vapour molecules will reach a point where they can no longer attain equilibrium. The vapour increases in temperature becoming higher than that of the same saturated vapour exerting the same pressure and this is how superheated steam is formed (Lawrence, 1995). When superheated steam enters a still, it would immediately take up water from the charge of plant material and as a result, tends to dry it out. It can be useful to dry out certain moist plant surfaces to a limited extent, but apart from drying, superheated steam is not suitable for distilling essential oils (Guenther, 1948).

By vaporising mixtures of water and essential oils, the temperature will always be maintained lower than the boiling point of water at the same total pressure and, in this way, damage and decomposition of the essential oils by overheating is prevented. When however the steam is superheated, pressure and temperature are no longer dependant and hence the vapour temperature can vary at a constant pressure. As a result, the characteristic safeguard against overheating common with saturated steam is no longer operative (Guenther, 1948).

### **2.6.3 Steam**

The correct steam wetness fraction is very important for the efficient distillation of oil from plant materials, although the optimum level varies considerably with the individual nature and condition of the plant material or charge. It is important to match the wetness fraction of the steam to the absorptive capacity of the plant materials surface. For absorptive plant materials, wet steam can be produced. For less absorptive plant materials, one can generate steam under

relatively high pressure and cause it to dry out slightly as it expands to a lower pressure on entering the charge vessel (Denny, 1999).

The most important feature of the steam supply is the generating rate and the magnitude of the distillate flow per square meter of the charge cross-sectional area. According to Denny (1999), the steam flow rate should be between 2 and 4 litres per minute per square meter of plant material or charge cross-sectional area. This forms an upward draft that prevents downward flow of juices from the collapsing plant cells termed reflux flow. If the rate of displacement is lower than the range mentioned above refluxing will occur, which can result in oil losses of up to 30%. Refluxing can usually be detected by the accumulation of dark coloured water in the bottom of the still. It must be noted that there will always be some accumulation of water in the bottom of the charge vessel due to the initial condensation of vapours on the cold plant material and charge vessel walls at start up.

Having discussed the basic thermodynamic properties, it is now possible to focus on the oil isolation procedure from the plant charge within the charge vessel by steam distillation.

## **2.7 Principles of Oil Isolation through Steam Distillation**

For the oil to change from the liquid phase to the vapour phase, it must receive latent heat, which within a field still, comes from the condensing steam. Consequently, the temperature of the steam within the still must be higher than the temperature at which the oil boils in the presence of water on the surface of the plant charge. If this was not the case, there would not be a temperature gradient to transfer the latent heat from the condensing steam to vaporise the oil droplet (2<sup>nd</sup> law of Thermodynamics) (Lawrence, 1995).

At the point of evaporation of the oil off the plant surface, the concentration of the oil vapour and the proportion of the total pressure it exerts will be at a maximum. Hence the local temperature will be the lowest that can cause the mixture of water and oil to boil under the prevailing pressure. As the oil vapour is dispersed through the steam in the general vapour space, the concentration of the oil vapour is reduced, and hence its share of the total vapour pressure is also

reduced. The steam thus needs to exert a greater vapour pressure than it did at the point of evaporation of the oil and the temperature in the general vapour space is therefore higher than that existing at the point of evaporation of the oil (Denny, 1999).

It is this temperature difference between the general vapour space and the point of evaporation of the oil that is the vital principle of the distillation process. This temperature gradient makes the transference of latent heat from the steam to the vaporising oil possible, according to Fourier's law. The magnitude of this temperature gradient governs the rate at which the oil receives the latent heat and boils away from the surface of the plant charge (Denny, 1999).

The oil can thus only evaporate as fast as the latent heat of vaporisation can be supplied. Hence for every type of oil and set conditions, there is a balance point for the maximum obtainable oil content of the distillate vapour in the general vapour space. Any further enrichment of oil would lower the temperature of the general vapour space and hence reduce the gradient. This would retard the rate at which the heat is delivered to the oil and reduce the rate of evaporation until the equilibrium was restored. This automatic control system is referred to as feedback (Denny, 1999).

One of the most important factors that affect the temperature gradient, at which feedback controls the ratio of oil to water in the condensate, is the varied action of different plant charge surfaces with the wetness fraction carried by steam i.e. the ability of the plant charge to remove the moisture from the steam. A further important consequence of feedback is that the amount of steam required to pass in order to complete distillation, depends on the amount of oil to be recovered, and is independent on the actual mass of plant material in the still (Denny, 1999).

Essential oils isolated from plants may either be superficial (surface born) or subcutaneous (beneath the outer surface) oils. The location of the oil affects the oil isolation process.

### **2.7.1 Distillation of superficial oils**

Superficial oils are those of which the entire oil content of the plant material is borne on the outer surface of the plant material. A plant bearing superficial oils is characterised by having

distillation times that are inversely proportional the distillate flow rate when distillations are performed (Lawrence, 1995).

During the distillation of superficial oils, the steam rises through the charge vessel and moistens the leaves of the plant charge by condensing on them and raising their temperature correspondingly by surrendering its latent heat during condensation. On the surface of the plant material, numerous oil patches occur because of the ruptured oil glands. Around each of these oil patches an oil and water interface occurs. As the temperature around this interface increases, the additive affect of the vapour pressures of the two immiscible liquids at the interface will cause them to boil. Hence, the oil saturated vapour rises from all the interface points so that the vapour above the entire oil droplet will be saturated with oil vapour as depicted in Figure 2.1. Saturated steam vapour will still readily condense on the mini-puddles of water on the plant materials surface that have direct contact with the oil droplets, releasing its latent heat to vaporise the oil with which the water has contact (Lawrence, 1995).

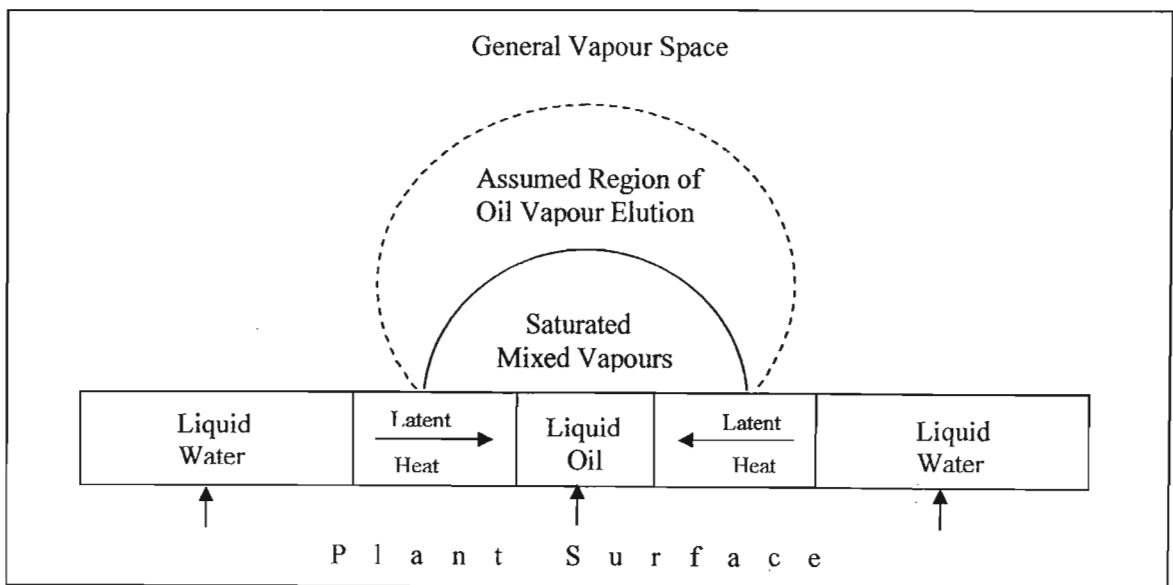


Figure 2.1 Illustration of heat transference at the water/oil interface (Denny, 1999).

Evaporation of the oil can only occur at the edges of the oil patches, and these perimeters recede during distillation until the oil patch has been exhausted. The rate at which the oil will vaporise depends on the rate that fresh steam is delivered and also on the rate at which the oil saturated

vapour is physically removed from the oil-water interface. The size of the heat transferring annulus is important for efficient distillation as the rate that the heat is delivered to the oil is proportional to its area. The magnitudes of this area and of the temperature gradient are the principal items affecting the rate that a given flow of steam can deliver latent heat and reduce the radii of the oil patches (Denny, 1999).

The way steam and vaporised oil move through the plant charge bearing superficial oils is illustrated in Figure 2.2. Assume that a plant charge has a number of hypothetical layers 1cm thick with steam supposedly delivered to the charge at a rate sufficient to raise one layer to boiling point in seven seconds and also to evaporate all the oil from one layer in seven seconds. As the lowest layer starts to boil, the mixture of oil and water vapour will rise and condense on the next layer above it for a period of seven seconds. When the second layer starts to boil, the vapour will consist of the oil that condensed onto it from the layer below, plus the oil vaporising off its own surface for seven seconds and the amount of steam flow for seven seconds (Denny, 1999). This process will then continue up the still.

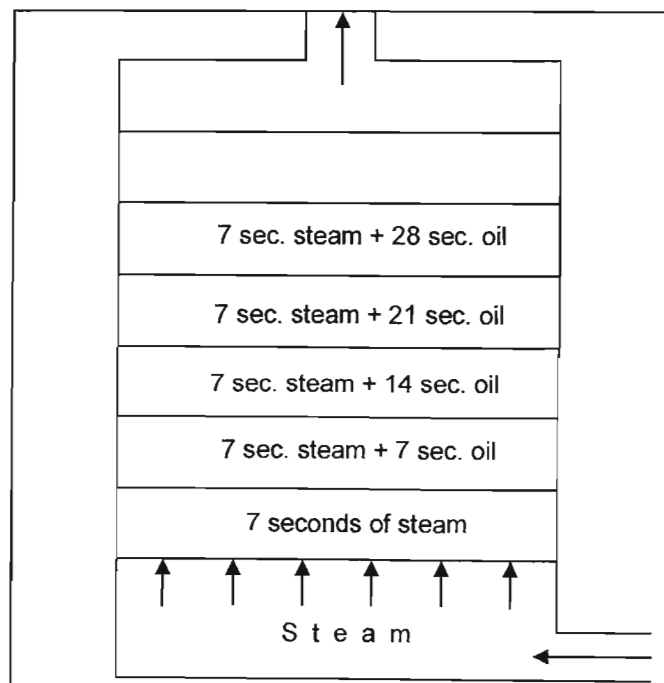


Figure 2.2 Illustration of the steam/oil movement during steam distillation (after Denny, 1999).

As more layers release their oil, the sizes of the oil patches increase further up the still. Consequently the top layers will take the longest to distil. As a result the oil extraction time is based on the time that it takes to exhaust the oil from the top layer of the plant charge. The parameters related to the oil isolation or extraction time are discussed in the following section. It is important to note that the surface of the material must be able to absorb the oil condensing on it without becoming saturated. This is a problem with plant materials that have a high moisture content and partial wilting of these plants prior to distillation, helps to increase their absorptive capacity (Lawrence, 1995).

### 2.7.2 Superficial oil extraction time

The method for estimating the extraction time of superficial oils that is discussed in this section was obtained from Denny (1999). For plants bearing superficial oils, the steam rate of displacement and the height of the charge are related to the extraction time by two crop specific characteristic parameters:

- The basic time ( $t$ ), which is the time required to distil the oil from a natural oil patch formed from a single bursting gland on the bottom layer of plant material in the still, with no additive oil from layers below. The basic time is proportionally dependant on the size of the oil patch and the relationship between the basic time and the surface area of the oil patch  $a$  ( $a$ ) is illustrated in Equation 2.1 below.

$$t = E^{-1} \cdot r = E^{-1} \left( \frac{a}{\pi} \right)^{\frac{1}{2}} \quad (2.1)$$

where;  $t$  = Basic time (min)  
 $r$  = Oil patch radius (cm)  
 $a$  = Area of neutral surface oil patch ( $a = \pi r^2$  in  $\text{cm}^2$ )  
 $E$  = Constant of proportionality (cm/min)

- The ratio  $s$  (in cm), which is the ratio of the area that the basic surface oil patch from the bottom layer bears ( $a$  in  $\text{cm}^2$ ) to the area increment that is added per unit increase in charge height ( $\delta a$  in  $\text{cm}^2/\text{cm}$ ) i.e.

$$s = \frac{a}{\delta a} \quad (2.2)$$

The complete extraction time can be calculated by adding a second term to Equation 2.1 to account for the increase in oil patch size with an increase in charge height as shown below:

$$T = E^{-1} \left( \frac{a + H \cdot \delta a}{\pi} \right)^{\frac{1}{2}} \quad (2.3)$$

where;  $T$  = Extraction time (min)  
 $H$  = Charge height (cm)

Making the constant of proportionality ( $E$ ) the subject of Equation 2.1, the area of a neutral oil patch ( $a$ ) the subject of Equation 2.2 and then substituting both into Equation 2.3 yields the following equation:

$$t = T \sqrt{\frac{s}{s + H}} \quad (2.4)$$

In order to derive  $t$  and  $s$  for a specific crop, test distillations need to be conducted. At least three carefully recorded distillations need to be conducted in the same vessel, each with different charge heights but equal packing densities and similar steam flow rates. For each distillation, approximately two-minute fractions of distillate should be sampled in separate containers and the amount of oil and water in each must be recorded. The average water and the average oil flow per minute, divided by the charge cross sectional area i.e. in  $\text{kg}/\text{min}/\text{m}^2$  can then be calculated. The total oil yield of each distillation must also be recorded.

A graph of oil produced against elapsed time should be plotted for each distillation, and the curve should be extended until it is asymptotic with the time axis as illustrated in Figure 2.3. At this point, the y-value stays constant as no more oil is extracted and is termed the estimated virtual exhaustion oil content (EVE). The extraction time to reach the EVE (approximately 42 min in Figure 2.3) together with the charge height can be substituted into Equation 2.4 for each of the distillations. The two unknown parameters ( $t$  and  $s$ ) can then be derived by solving two of the equations simultaneously. An advantage of doing more than two distillations is that the two derived parameters can be used to calculate the extraction times and charge heights of the other distillations, and hence the accuracy of the derived parameters can be assessed.

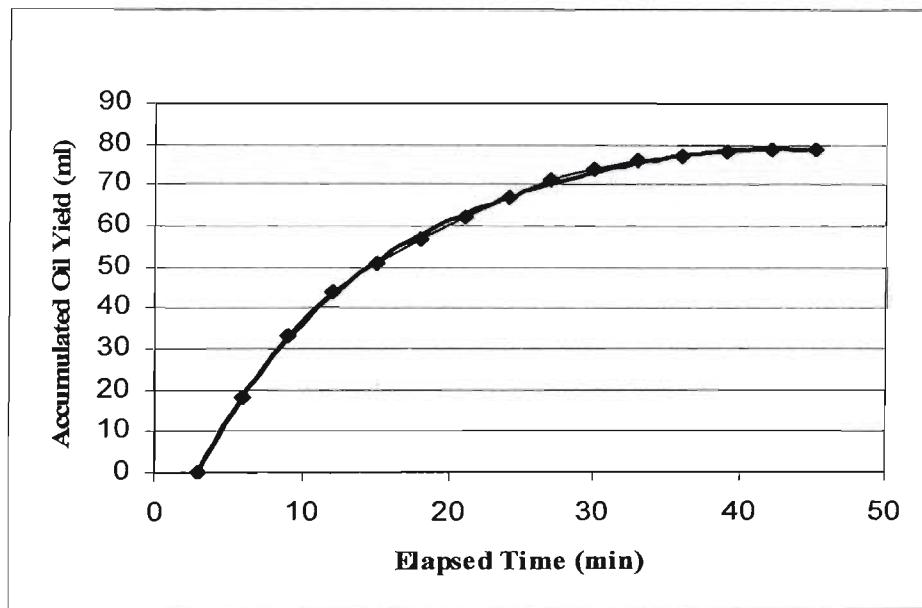


Figure 2.3 Oil produced against elapsed time for a rosemary distillation conducted on the test distillation unit.

### 2.7.3 Distillation of subcutaneous oils

Subcutaneous oils are those which occur beneath the outer surface of the herbaceous material, and which are not exposed on the surface of the plant material when loaded into the charge vessel (Denny, 1999).

hence increasing the diffusion rate (Fick's Law) (Denny, 1999). A method for calculating the extraction time for subcutaneous oils will now be discussed.

#### 2.7.4 Subcutaneous oil extraction time

The method for estimating the extraction time of subcutaneous oils that is discussed in this section was obtained from Denny (1999). The relationship between the extraction time and charge height for subcutaneous oil crops varies from the superficial oil relationship. This is mainly because the movement of the oil from within the plant material to the top of the charge vessel differs from the superficial oils. The extraction time ( $T$ ) for subcutaneous oils is given by the general relation:

$$T = t + H_{virt} \cdot \delta \quad (2.5)$$

- where;  $T$  = The extraction time at 95% estimated virtual exhaustion (EVE) (min)  
 $t$  = The basic time for a given flow of steam to extract oil from a typical subcutaneous oil gland at the bottom of the charge (min)  
 $\delta$  = Increment parameter or time increment per unit of virtual height (min/cm)  
 $H_{virt}$  = The virtual height of the charge, is the height (cm) of a number of layers of plant material that each contain the same amount of oil as one layer of unit thickness and area in a standard charge of typical material (explained on Page 22)

The second term in Equation 2.5 accounts for the increase in oil quantity for each unit increase in height due to condensation of the oil vapours from below. The formula that relates the effect of a change in steam flow rate on the subcutaneous oils extraction time is shown below.

$$Z = R \cdot X^{\frac{2}{3}} \quad (2.6)$$

- where;  $Z$  = Factor of change in oil recovery rate

- $X$  = Factor of change in steam flow rate over the plant material surface  
 $R$  = Diffusion lag factor compared with that for a given steam displacement rate

The fluids diffusing through the parent plant material do not increase their rate of flow in full proportion to the accelerating force due to the increase in steaming rate. As a result, the diffusion lag factor ( $R$ ) is present in Equation 2.6.

In order to determine the lag factor for the specific subcutaneous oil crop, two test distillations with charge vessels of the same size need to be conducted at different steam flow rates. The following symbols can be assigned to the variables that need to be determined for the two scenarios;  $W_o$  = mass of steam to pass at the original flow rate  $\dot{M}_o$  for time  $T_o$  and  $W_n$  = mass of steam to pass at a new flow rate  $\dot{M}_n$  for time  $T_n$ . From these results, obtained from the distillations, it is possible to calculate the values of  $Z$  and  $X$ , represented in Equation 2.6 as follows:

$$Z = \frac{T_o}{T_n} \quad (2.7)$$

$$X = \frac{\dot{M}_o}{\dot{M}_n} \quad (2.8)$$

Once the parameters  $Z$  and  $X$  have been determined, Equation 2.6 can be used to calculate the value of  $R$ . By conducting several test distillations with the steam flow rate varied by a different factor for each, it is possible to plot a curve of the lag factor for the specific crop against the factor of change in steam flow rate as illustrated in Figure 2.4 below.

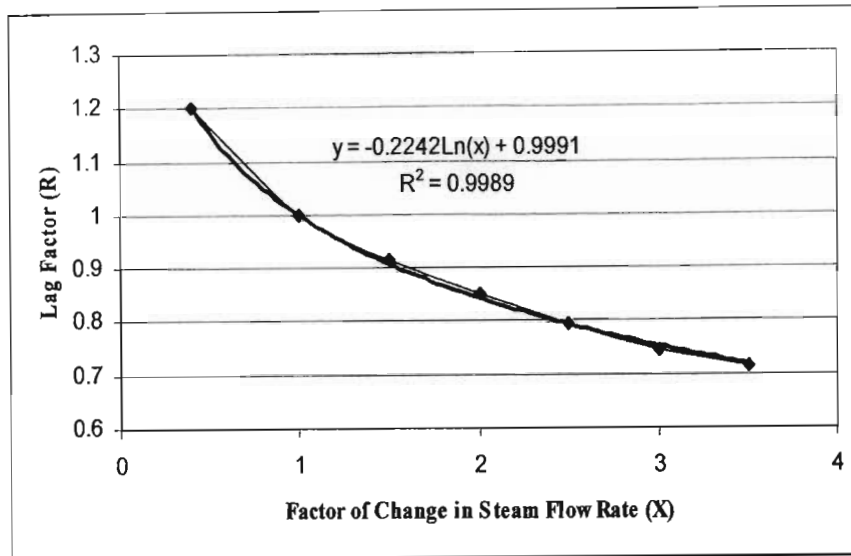


Figure 2.4 Relation between the lag factor and the factor of change in steam flow rate for *eucalyptus polybractea* (adapted from Denny, 1999).

It is again necessary to conduct carefully recorded test distillations (same way as for superficial oil crops) in order to determine the time increment parameter ( $\delta t$ ) and the basic time ( $t$ ) of Equation 2.5 for the specific crop.

Curves of oil produced against clock time need to again be plotted for each of the distillations and the distillation end-points are estimated as a percentage of the estimated virtual exhaustion oil content (95% EVE) for practical purposes. The extraction times are thus taken as the distilling time to reach the 95% EVE level.

In order to determine the virtual charge heights, the charge height of one distillation is taken as the standard (say the first distillation). The oil yield from this distillation is then divided by its charge height (oil yield/height) in order to determine the amount of oil (ml) present per centimetre of height i.e. ml/cm. For the standard distillation, the virtual charge height is the same as the actual charge height. The virtual charge height ( $H$ ) of the second distillation can be calculated by dividing the oil yield obtained by the value of oil yield/height calculated for the first distillation. The virtual height for the other distillations can be calculated in exactly the same manner.

The extraction times (from the test distillations) and the calculated virtual heights for each of the distillations should be substituted into Equation 2.5. The two unknown parameters ( $t$  and  $\delta$ ) can then be calculated by solving two of the equations simultaneously.

The preparation of the plant material prior to distillation can have an effect on the distillation time, the oil yield and chemical composition of the extracted oil. This will now be discussed.

### **2.7.5 Preparation of the plant material prior to distillation**

The preparation of plant material for distillation varies with the material to be distilled (Axtell & Fairman, 1992). Many plant materials (dried roots, seeds, woods) can be effectively kept for several months without significant loss of essential oil content if the room is cool, dark, dry and free from air circulation (Barson, 2002).

For plants bearing superficial oils in thick leaves, actual evaporation of the volatile oil through the walls of the plant tissue cannot take place readily because the oil must first be brought to the surface through hydrodiffusion. This is however not the case with flowers and leaves with thin walls as the volatile oil can easily diffuse through the thin walls and then evaporate from the plant materials surface. Mainly volatile components with high boiling points are affected by this process (Guenther, 1948).

Alternatively, with plant material containing high water content, it is sometimes advantageous to dry the plant material prior to the distillation process. When leaves of such plants, for example mint, are steamed, their surfaces become flooded by liquids from collapsing aqueous cells, as well as condensation from the steam. The oil glands collapse, but the oil floats on the water and no intermingling between the oil and water can take place. Instead a thin circumferential line of contact between the oil and water is formed around the perimeter of the surface oil patch. As a result, distillation is exceedingly slow because the transference of latent heat is minimal without mixing at the interface. The yield of oil is also poor, usually due to the strong reflux flow washing down to the bottom of the still. If the plant material has however previously been wilted to a moisture content of about 25 %, the material will have a slightly absorptive surface on which

the oil and water will intermingle at the interface and hence distillate will be rich in oil and the extraction more rapid (Denny, 1999).

For subcutaneous oils, the volatile oil loss from storing the plant material before comminution is not as significant as in the case of comminuted material. Therefore, if a delay in the distilling of the plant material cannot be avoided, the plant material should be stored in its natural condition (Guenther, 1948).

It can thus be deduced that proper storage and preparation of plant material prior to distillation is vital as it can have an effect on both the yield and the efficiency of the distillation. The extracted oil yields and the distillation times are also affected by the way the plant material is packed into the charge vessel. The plant material packing densities are thus important for oil recovery and will now be discussed.

#### 2.7.6 Packing densities and charging methods

The method of charging a still with plant material and then discharging it are extremely important, as it affects the amount of labour involved and also the production of the system. Any labour saving device will increase the economy of the process in the final calculation (Guenther, 1948). Hence there has been a drive towards increasing the charge weights, by stamping the herb into the still with the assistance of steam to soften the plant material. Studies (using lavender) have however shown that the abovementioned practice is not economical as shown in Table 2.1.

Table 2.1 Effects of steam assisted charge packing for lavender (adapted from Denny, 1999).

	Dry Pack	Steam Pack
Mass of Herb (kg)	420	425
Extraction Time (min)	21.43	25.34
Yield Oil per kg Herb (ml)	10.58	9.61
Distillate Ratio oil:water	1 : 17.41	1 : 19.68

By comparing the results of the two charges in Table 2.1 above, it can be seen that the steam pressed charge took 18% longer to distil, yielded 9% less oil and consumed 13% more steam. It can thus be deduced that when the density of dry plant material is increased without steam assistance, both the extraction time and the oil yield increases (Denny, 1999). A uniform packing density of plant material into a charge vessel will be an advantage because if the material is not packed uniformly, the steam will find easy passages through the less dense section of the material. As a result, the oil will not be isolated from the more dense areas that received no steam (Guenther, 1948).

It can thus be concluded that tight, even, dry packing of the herb charge saves time, fuel and increases the oil yield. Each of the components of a distillation unit, as well as the influence that they have on the quality and yield of the essential oils will now be investigated.

## **2.8 Distillery Components**

To design a distillery the following components need to be taken into consideration:

- The charge vessel
- Condenser
- Boiler
- Oil separator

### **2.8.1 Charge vessel**

The charge vessel is basically a tank with some means of injecting steam at the bottom of the tank in a way that allows uniform distribution, such as a manifold (Barson, 2002). The charge vessel usually contains a grid plate that supports the plant material, under which the steam manifold is fitted (Whish, 1996).

The charge vessel should be constructed in such a way that it is easy to charge and discharge. One of the methods used to minimise downtime required for loading and emptying after distillation is a basket that is placed in the charge vessel. The plant material is placed in the basket, which is then positioned inside the charge vessel. A tipping mechanism or hoist is often used to discharge the material easily from the charge vessel (Lawrence, 1995).

A suitable drain valve should be fitted to the bottom of the charge vessel. This makes it possible to drain the condensed vapours that accumulate in the bottom of the vessel. The drain can also serve as an outlet for the wash water, when cleaning the still (Guenther, 1948). In order to avoid steam leaks between the still pot and its top cover, which would result in oil losses, they must be held tightly together with a suitable gasket. The charge vessel should also be insulated in order to reduce condensation and hence heat losses within the charge vessel (Axtell and Fairman, 1992).

A critical parameter in the design of the charge vessel is the steam capacity of the boiler. The size of the boiler can be determined once the oil content and the amount of steam needed to displace this oil are known (Denny, 1999). When calculating the dimensions of the charge vessel one should also keep in mind not only that some plant materials are very voluminous, but also that during distillation the mass often swells and expands by up to a third of its original volume (Guenther, 1948). The designed height of the still pot in relation to its width depends on the porosity of the plant material that the distillation unit will be used for. The height to width ratio for porous material can be greater than for materials that are less porous as the amount of moisture that the material will need to absorb increases higher up the charge vessel (Guenther, 1948).

Stainless steel (Grade 304 or 316) is usually used to construct the still as it is durable, the chemical constituents of the essential oils do not react with stainless steel and it is easy to clean after each distillation. Certain materials have the tendency to absorb a little essential oil, which cannot be removed and as a result, a certain odour will always adhere to the still, which can easily spoil the odour of another type of oil and hence reduce its value (Guenther, 1948).

### **2.8.1.1 Effect of charge height on the distillation process**

The manner, in which the charge height influences the oil content of the distillate, differs slightly between superficial and subcutaneous oils, but in both cases it is entirely independent of the charge diameter. The charge diameter only influences the speed of the steam through the charge at a given steam flow rate (Denny, 1999).

With subcutaneous oils, the amount of surface oil at the top of the still increases proportionally with charge height. Hence the factor, by which the concentration of the oil inside the plant material is greater than that at the surface at that level, is reduced for each successive layer from which the steam has already gathered oil. As a result the rate of diffusion will decline and the extraction time of the top layer, which is that of the charge as a whole, will increase accordingly (Denny, 1999).

For the superficial oils, as the charge height increases, the oil content of the mixing vapour in the general vapour space will tend to rise along with it. The oil then exerts a greater share of the total pressure, and hence the temperature of the general vapour space is reduced. As the saturation temperature on the plant materials surface stays constant, the temperature gradient decreases. This causes the rate of evaporation to also decrease and hence an increase in distillation times for an increase in charge height (Denny, 1999).

The vapours pass through the condenser once they exit the charge vessel. The condenser thus forms a vital component of a distillation unit and the functioning of the existing condenser designs will now be discussed.

### **2.8.2 Condenser**

The function of the condenser is to convert all the steam and the accompanying oil vapours into liquid. It is important that the condensate is cooled to the temperature at which the oil and water will best separate in order to decrease the separation time.

With the simplest type of condenser, the vapours pass through a coiled tube contained in a water bath supplied with running cold water, and the condensate is obtained at the bottom of the condenser tube as illustrated in Figure 2.5(a) (Axtell and Fairmen, 1992). A disadvantage is that the flow of vapours through the system is often hampered, thus creating a back pressure (Guenther, 1948).

A far more efficient type of condenser is the multi-tubular type (Figure 2.5 (b)) in which a series of parallel tubes are mounted inside a cylindrical jacket through which the cooling water is passed. This design provides a large surface area for cooling in relation to its volume (Axtell and Fairmen, 1992). The main differences between the two types of condenser discussed are summarised in Table 2.2.

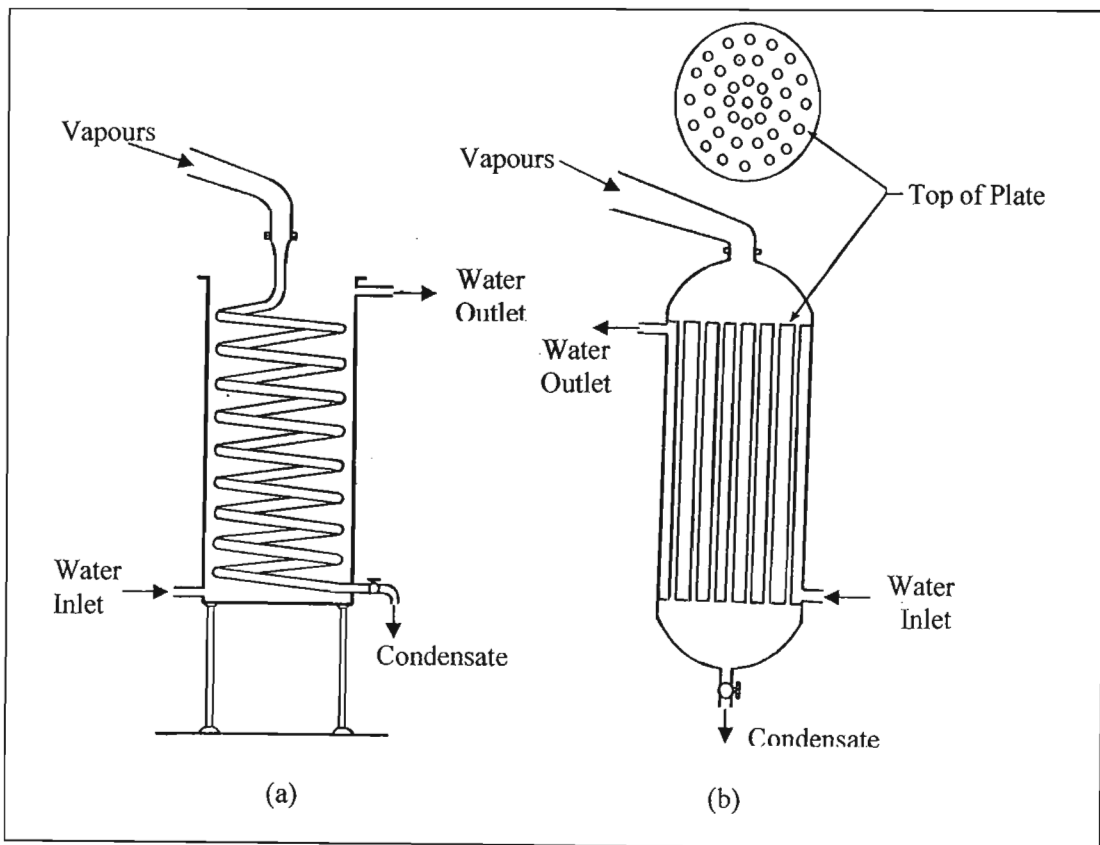


Figure 2.5 Typical (a) Coil Condenser and (b) Tubular Condenser (Guenther, 1948).

The main design requirements for such a tubular condenser, given by Denny (1999), are as follows:

- There must be no tendency for laminar flow on either side of the tube wall for efficient heat transfer.
- The hot distillate and the cold water travelling in opposite directions must pass each other at a maximum practical speed. This means that there must be minimum idle space between the tube bundle and the enclosing shell wall.
- The cross-sectional area of entry and passage through the unit must allow for the expulsion of air when distillation starts.
- It must be easy to clean the tubes and also to disassemble and reassemble the unit.
- The unit must also be durable and relatively easy to construct.

Table 2.2 Differences between coiled tubular and multiple tube condensers (adapted from Lawrence, 1995).

<b>Coiled Tubular Condenser</b>	<b>Multiple Tube Condenser</b>
Easy to make	Difficult to make (requires shop manufacture)
Poor heat transfer	Good heat transfer
High pressure build-up during distillation	No pressure build-up during distillation
Needs tank of water with sparse use of running water. Non-economical water use	Needs running water. Economical use of water
Large in physical size	Smaller in size

The required condenser size depends on the rate of distillate flow, the acceptable pressure under the still lid when air is being expelled at the start of each distillation, the temperature and quantity of the available cooling water, the desired exit temperature of the condensate and the factors affecting the properties of the heat transfer through the tube wall and the dissolved liquid (Denny, 1999). A condenser should be designed slightly larger than required as the condenser surface must be large enough to cool the distillate sufficiently, even at a very high rate of distillation.

Slow distillation has many disadvantages, such as hydrolysis of esters, wetting and conglomeration of the plant charge. These can cause a reduction in oil yield (Guenther, 1948).

In the next two sections the method for sizing the connecting pipe, through which the vapours pass from the charge vessel to the condenser and the design calculations for sizing the condenser itself will be discussed.

### **2.8.2.1 Condenser connecting pipe**

Pipes through which steam flows need to be sized to accommodate the steam flow at the required pressure. When pipes are oversized, the pipes, valves, fittings will be more expensive than necessary and a greater volume of condensate will be formed due to the greater heat loss. This means that either more steam trapping will be required to remove the moisture or wet steam will be delivered to the point of use. If however the pipes are undersized a lower pressure will be available at the point of use and there is also a risk of steam starvation (Anon, 2004).

Steam pipes can be sized by using Figure 2.6 below. The temperature and pressure at which the steam will move through the pipe can be used to obtain point A. The steam flow rate through the pipe can be selected from the bottom right figure and a line should then be projected horizontally from A to the steam flow rate (B). Pressure losses occur as the vapours enter a pipe, pass through a bend and/or a globe valve and due to friction on the pipe walls. The pressure losses for various pipe sizes are represented in Table 2.3 below. The expected pressure loss through the pipe, per 100m of length, can be selected from the y-axis on the top figure (D). The required pipe size can then be obtained from the intersection of projecting a line horizontally from D and vertically from B.

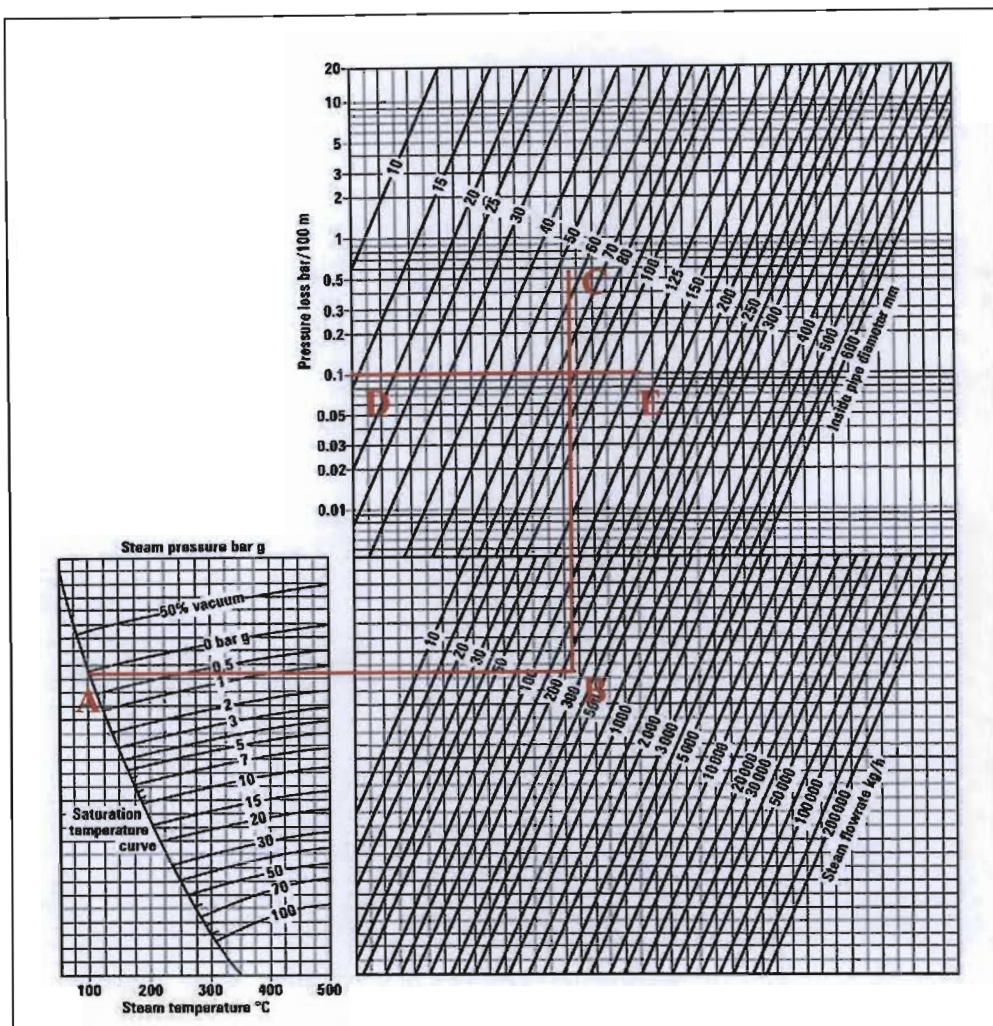


Figure 2.6 Steam pipe sizing graph (Anon, 2004).

Table 2.3 Pressure losses for the specified pipe sizes (adapted from Denny, 1999)

Pipe size (mm)	33.4	48.3	60.3	88.9	114.3	141.3
Pipe entry (m)	0.683	1.442	2.182	4.087	6.154	8.528
90° bend (m)	0.457	0.960	1.454	2.725	4.103	5.688
Globe valve (m)	0.683	1.442	2.182	4.087	6.154	8.528

### 2.8.2.2 Condenser size

Heat transfer equipment typically operates on the principle that one fluid receives heat from a second fluid, which is usually separated by a solid retaining wall. In designing such equipment, the temperatures at which the two fluids are to enter and leave the equipment are usually known. The rate of heat transfer between the retaining wall and the fluid per unit area of the retaining wall and per degree temperature difference between the surface of the wall and the main body is given by (Frank, 1978):

$$\dot{q}_{tot} = AU.\Delta T \quad (2.9)$$

$$\Delta T = (\alpha' - \alpha'') / \ln(\alpha' / \alpha'') \quad (2.10)$$

- where:  $\dot{q}_{tot}$  = heat flow from one fluid, across a barrier to the next fluid (kJ/h)  
 $A$  = barrier area across which the heat flow occurs (m<sup>2</sup>)  
 $U$  = overall condenser coefficient (kJ/h.m<sup>2</sup>.K)  
 $\Delta T$  = logarithmic mean temperature difference (K) between vapours and moving coolant  
 $\alpha'$  = the temperature difference between the hot vapours and entering coolant (K)  
 $\alpha''$  = the temperature difference between the hot vapours and exiting coolant (K)

Both condensing and cooling of the distillate takes place inside the same tube with such condensers as shown in Figure 2.7. Hence the total required tube area consists of the tube area required for condensing the vapours and the tube area required for cooling the condensate. The overall condenser coefficient is thus divided into  $U_{cond(ensing)}$  and  $U_{cool(ing)}$ .

The processes taking place in the condenser are illustrated in Figure 2.7. The first portion of the condenser is where the vapours are condensed and condensed vapours are then cooled in the second portion. The coolant flows in the opposite direction to the vapours and enters the condenser at the end where the condensed vapours leave the condenser. The coolant first reduces the temperature of the condensed vapours and as a result, the coolant itself will have increased in

temperature after passing through the cooling phase and entering the condensing phase. During the entire condensing phase the vapours are at their entering temperature, usually around 373 K. They are still at the abovementioned temperature at the point where they condense at the hypothetical changeover point, but are then liquids entering the cooling phase (Denny, 1999).

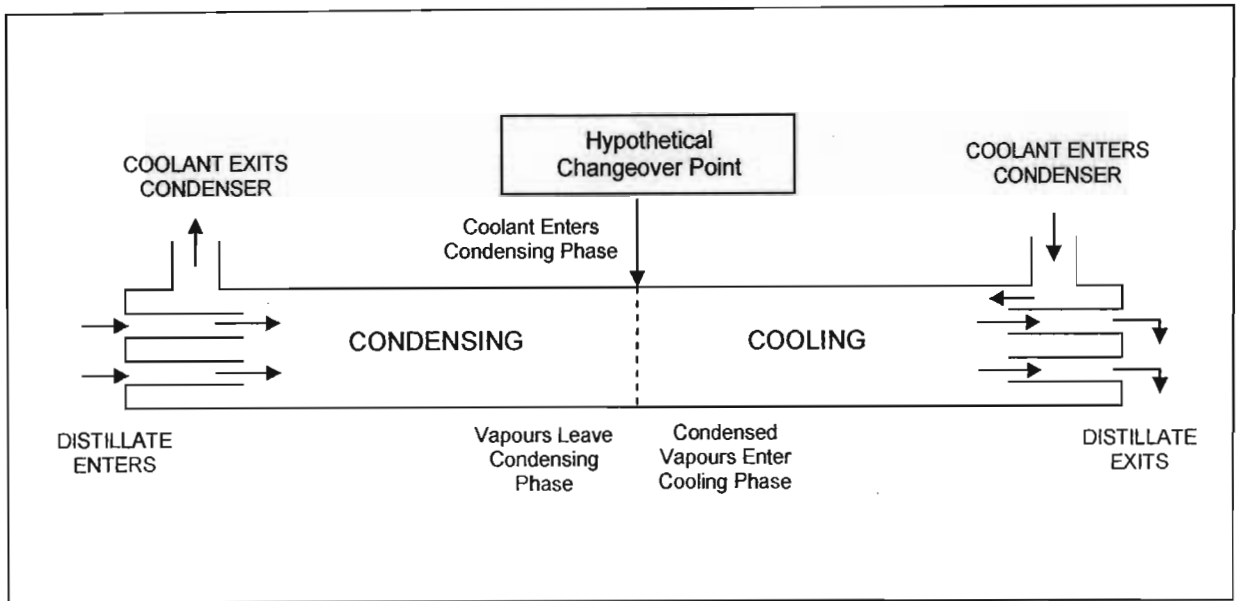


Figure 2.7 Condensing and cooling phases inside a condenser tube (after Denny, 1999).

There are thus two logarithmic mean temperatures to calculate, one for cooling and the other for condensing. The cooling phase is calculated first in order to calculate the rise in temperature of the coolant and hence the temperature at which the cooling water enters the condensing phase will be known. The distillate flow rate (steam flow rate), distillate entering temperature, required distillate exit temperature, coolant flow rate and its entering temperature are all known. The heat removed cooling the distillate from where it enters the cooling stage to its exit temperature can be calculated by Equation 2.11 below (Holman, 1989).

$$\dot{q}_{cool} = \dot{m} \cdot C_p \cdot \Delta t \quad (2.11)$$

where:  $\dot{q}_{cool}$  = heat removed cooling the distillate (kJ/h)

$\dot{m}$  = distillate flow rate (kg/h)

$C_p$  = the specific heat of water (4.187 kJ/kg.K)

$\Delta t$  = the temperature difference between the temperature of the distillate liquid when it enters the cooling stage and its exiting temperature (K)

The increase in coolant temperature can then be calculated by making  $\Delta t$  the subject of Equation 2.11 and dividing the heat removed by the coolant water flow rate multiplied by the specific heat of water. From the calculated increase in temperature, the cooling water temperature at the hypothetical changeover point can be determined by adding it to the cooling water entering temperature.

At the end of the cooling phase the logarithmic change in temperature ( $\Delta T_{cool}$ ) can be calculated from Equation 2.10, where now:

$\delta t'$  = temperature of distillate liquid entering cooling phase – the coolant water temperature at the end of cooling

$\delta t''$  = distillate liquid exit temperature – coolant water entering temperature

Values of  $U_{cool}$  for typical operating conditions have been determined as 1839.76 kJ/h.m<sup>2</sup>.K for near horizontal tubes and 2146.38 kJ/h.m<sup>2</sup>.K for vertical tubes (Denny, 1999). Once  $\dot{q}_{cool}$ ,  $U_{cool}$ ,  $\Delta T_{cool}$  have been determined it is possible to calculate the surface area required for cooling the distillate to the desired exit temperature by Equation 2.9. The latent heat removed in the condensing phase can be calculated by:

$$\dot{q}_{cond} = \dot{m} \cdot Q_{Ls} \quad (2.12)$$

where:  $\dot{q}_{cond}$  = latent heat removed condensing (kJ/h)

$\dot{m}$  = distillate flow rate (kg/h)

$Q_{Ls}$  = latent heat of steam at working pressure from steam tables (kJ/kg)

The coolant enters the condensing phase at the temperature calculated earlier. The rise in the coolant temperature is then calculated in exactly the same way as the cooling stage and hence the coolant exit temperature can be derived. The  $\Delta T_{cond}$  of Equation 2.9 for the condensing phase can be calculated from Equation 2.10, where now:

- $\delta'$  = temperature of distillate vapour entering condensing phase – the coolant water temperature at the start of the condensing phase
- $\delta''$  = temperature of distillate exiting the condensing phase – coolant water exiting temperature

The value of  $U_{cond}$  for thin walled tubes can be calculated as follows (Denny, 1999):

$$\frac{1}{U} = \frac{d''}{h' \times d'} + \frac{L}{K} + \frac{1}{h''} \quad (2.13)$$

where:  $U$  = the overall coefficient for heating or cooling, in this case  $U_{cond}$  (kJ/h.m<sup>2</sup>.K)

$h'$  = film coefficient for the inside of tube (kJ/h.m<sup>2</sup>.K)

$h''$  = film coefficient for the outside of tube (kJ/h.m<sup>2</sup>.K)

$L$  = thickness of the tube wall (m)

$K$  = thermal conductivity of the tube wall (kJ/h.m.K)

$d'$  = inside diameter of tube (m)

$d''$  = outside diameter (m)

For grade 304 stainless steel,  $K$  is 58.57 kJ/h.m.K at 373 K and for vapour condensing on the inside of the tube at 373 K,  $h'$  is 61 325.25 kJ/h.m<sup>2</sup>.K (Denny, 1999). The base factor for the film coefficient on the coolant side of the wall ( $h''$ ) is dependant on the temperature of the film. This temperature is taken as the mean of the inside tube wall and the average of the coolants entering and exit temperatures for both condensing and cooling. The tube wall temperature is the temperature of the vapour entering the condenser, as during the entire condensing phase the vapours are at this temperature. Since the coolant entering and exit temperatures have been

determined it is possible to determine the mean coolant temperature, hence the film temperature can be calculated and the base factor can be obtained from Figure 2.8 below.

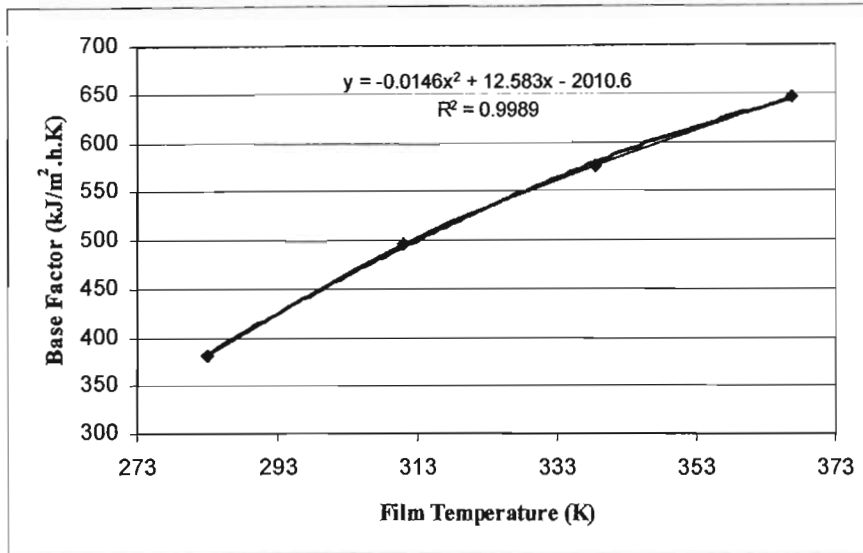


Figure 2.8 Base factors at varying film temperatures (adapted from Denny, 1999).

Condensers usually have a series of baffles that redirect the coolant water through the unit to maximise the heat transfer. For baffled tubular condensers the base factor must be corrected for the velocity of the fluid between the tubes, given by the formula:

$$V = \frac{3.28M}{(d_s - n.d'')L} \quad (2.14)$$

- where:  $V$  = the velocity of the fluid between the tubes (m/s)  
 $M$  = total flow of coolant (m<sup>3</sup>/s)  
 $d_s$  = internal diameter of the condenser shell (m)  
 $d''$  = outside diameter of tubes (m)  
 $n$  = number of tubes across the widest diameter  
 $L$  = distance between baffles (taken as the shell diameter)

The coolant velocity between the tubes can be determined from the chosen tube and shell dimensions and the coolant flow rate. This coolant velocity can then be used to obtain the correction factor from the curves represented in Figure 2.9. The base factor obtained from Figure 2.8 must then be multiplied by this correction factor in order to obtain the coolant film coefficient  $h''$ . As all the variables are known, it is now possible to calculate  $U_{cond}$  using Equation 2.13.

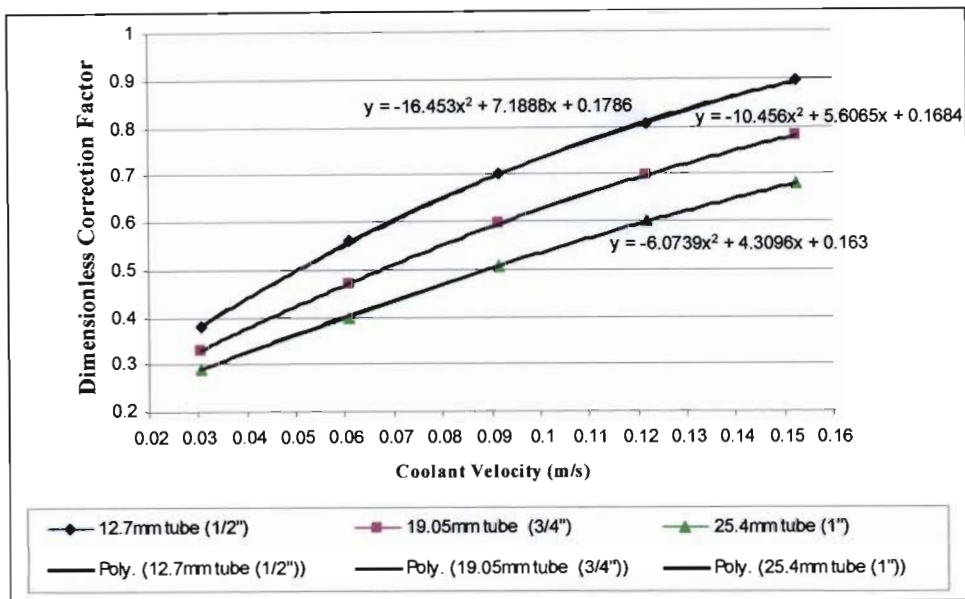


Figure 2.9 Correction factors for the base film coefficients at different coolant velocities (adapted from Denny, 1999).

Since  $U_{cond}$ ,  $\Delta T_{cond}$  and  $\dot{q}_{cond}$  have been calculated, it is possible to calculate the tube area required for condensing (Equation 2.9). Thus, the total area required is the sum of the areas calculated for cooling and condensing. This total area can be divided by the surface area for the specific tube bundle per meter of length, in order to determine the required condenser length.

### 2.8.2.3 Condenser backpressure

There is a large volume of air in the charge vessel at the start of a distillation. This air will be expelled through the condenser tubes as the distillation process starts. Air is denser than steam

and hence the backpressure in the charge vessel will be greater with air than with steam. It is necessary to estimate the backpressure for lid sealing purposes.

Since air is about 1.5 times as dense as steam, its speed along the tubes will be 0.67 times that of steam. The backpressure ( $P_{back}$ ) can be estimated by multiplying the pressure loss along the tube ( $\Delta P_{tube}$ ) for pure steam by  $0.67^2 = 0.45$  and adding it to the pressure drop in the connecting pipe ( $\Delta P_{pipe}$ ) as illustrated in Equation 2.15 below (Denny, 1999). The pressure loss through the tube can be calculated by subtracting the pressure loss through the connecting pipe from the charge vessel operating pressure.

$$P_{back} = \Delta P_{pipe} + (\Delta P_{tube} \times 0.45) \quad (2.15)$$

### 2.8.3 Oil separator

The oil separator is the most critical component in overall product recovery and the profitability of the plant (Barson, 2002). The condensate flows from the condenser into the separator and its function is to achieve a quick and complete separation of the oil from the condensed water. The volatile oil and water are mutually insoluble and due to the differences in their specific gravities, the two liquids form two separate layers. The position of the oil layer (top or bottom) depends on the specific gravity of the oil. Since the total volume of water condensed will always be larger than the oil, it is necessary to remove the hydrosol (the water component) continuously during the distillation process (Guenther, 1948).

Oil and water often do not separate immediately in the oil separator. The distillate or condensate must therefore not flow into the separator too rapidly and any turbulence must be avoided to decrease separation time. As a result the condensate should enter the separator at the bottom with a curved outlet (Figure 2.10) in order to create a swirl and hence a vortex, which prevents airlocks from forming at the entry pipe (Denny, 1999). Turbulence is also reduced by the baffle, which is an inner cylinder sealed to the bottom of the separator.

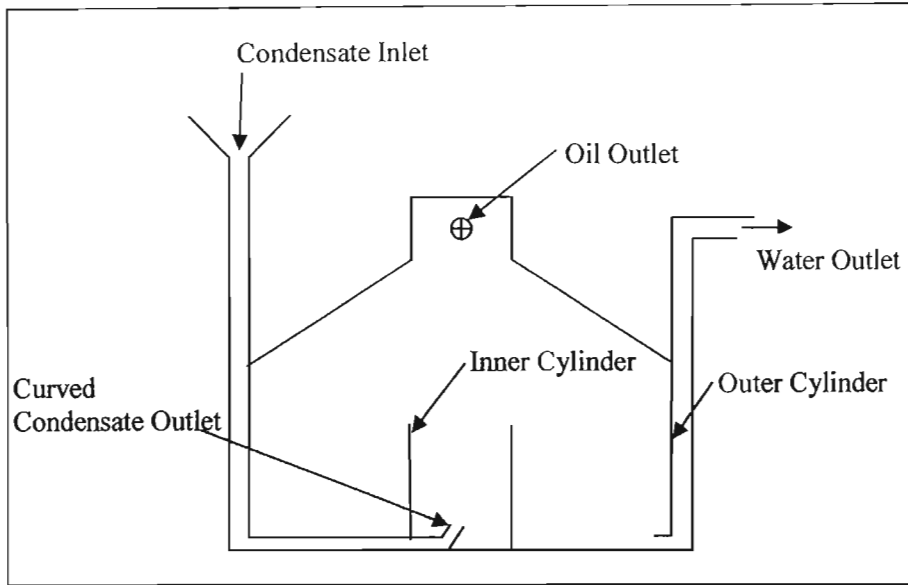


Figure 2.10 Typical oil separator for oils less dense than water (after Lawrence, 1995).

A separator should be designed or sized in such a way that there is enough time for the solution to settle and the oil to separate, while the hydrosol continuously discharges from the outlet at the bottom and the oil continuously pours out of the top outlet (Lawrence, 1995). In this way, distillation can continue without the separator limiting the process. If the separator is sized too small and there is not significant time for the oil to separate, some of the oil will flow out with the hydrosol and hence will be lost.

Materials used to construct separators are usually glass and stainless steel. Rubber tubing or stoppers cannot be used because rubber, being partly soluble in essential oils, gives the oils an objectionable odour (Guenther, 1948).

### 2.8.3.1 Fundamentals of oil separation from water

The separation of essential oil from water in the condensate stream following distillation is influenced greatly by the relative densities of the oil and the water. Where there are significant differences between the densities, separation will be much faster and more complete, leading to a greater recovery of oil (Porter and Lammerink, 1993). Oils with a specific gravity only slightly

below that of water do not readily separate from the distillation water (Guenther, 1948). Separation of such oils would benefit from increasing the temperature of the condensate. This is because as the temperature of the solution increases, the density of the oil decreases more rapidly than that of the water. As a result, the differences between the relative densities of the two solutions increase, promoting the oils separation (Porter and Lammerink, 1993).

The increased temperature will reduce the density of the oil and will also cause the viscosity of the condensate water to decrease more significantly than that of the oil, thereby removing another barrier that could prevent the minute oil particles from coalescing into droplets which will readily separate from water (Lawrence, 1995). It is due to this reduction in the condensate water viscosity that improves the separation of oils that are denser than water when the temperature is increased, despite their density having moved closer to that of water (Denny, 1999). To separate the oil from the water, the temperature in the separator must be maintained to give sufficient time for the droplets of oil to either rise or sink (Lawrence, 1995).

The sizes of the oil drops also have an effect on the rising velocities of oil. As the droplets decrease in size, interfacial tension (a measurement of the cohesive energy present at an interface arising from the imbalance of forces between molecules at an interface) becomes predominant over buoyancy, caused by the difference in densities, and the small droplets can remain trapped in the water or hydrosol. As a result, the hydrosol flowing from the oil separator usually contains some small quantity of the volatile oil in solution or suspension, with the quantity depending on the solubility and specific gravity of the various oil constituents. With steam distillation, hydrosol containing a significant quantity of oil should be pumped or injected into a separate still for redistillation in order to isolate some of the remaining oils. This procedure is not usually carried out, but depends on the value and magnitude of the oils still present. The process of recovering the oil from the water is known as cohobation (Guenther, 1948).

### **2.8.3.2 Separator Design**

The distillate flow rate and the speed at which the oil rises through water need to be taken into account when designing a separator. A separator should be designed in such a way that the oil

particles rise faster than the hydrosol moving downwards within it for the known distillate flow rate and temperature.

The distillate enters the separator through a curved outlet within the inner cylinder (Figure 2.10). According to Denny, (1999) the inner cylinder should be sized to accommodate the first three minutes of the distillate flow, as the distillate exit temperature is lower at the start of a distillation than for the remainder of the distillation process. Hence the distillate can gradually increase in temperature within the inner vessel before entering the main separator area when starting up the next distillation. The diameter of the inner cylinder can be selected and the required accumulated distillate volume for three minutes of flow is determined from the distillate flow rate. The height of the inner cylinder is calculated by dividing the accumulated volume by the cross sectional area of the inner cylinder.

The outer cylinders cross sectional area (Figure 2.10) must be large enough for the distillate to decant from the inner cylinder at a speed slower than the speed of the oil rising from below at the distillate temperature. In this way the oil will be able to rise to the top of the separator. The rising oil speeds at various temperatures for the four listed crops are shown in Figure 2.11. The area must therefore be larger than the area calculated by dividing the distillate volumetric flow rate by the rising speed of the specific oil type, to ensure a lower decanting speed from the inner cylinder. It must be noted that the cross sectional area of the inner cylinder must be added to the calculated outer area as it forms part of the total area. The required diameter can then be derived from the calculated total outer area.

The relative heights of the oil and hydrosol (water component) outlets must be so that the both the oil and the hydrosol can continually discharge. The relative density of the oil is less than hydrosol and hence the hydrosol outlet will have to be slightly higher than the oil outlet with the actual height difference depending on the relative density of the oil. The relative density differs for unlike oils, and the actual depth of hydrosol in the horizontal part of the hydrosol outlet changes due to the varying distillate flow rate caused by surges from the boiler. As a result the height of the hydrosol outlet should be adjustable (Denny, 1999). Once the oils have been

separated from the hydrosol, it is important that they are prepared for storage and also stored correctly in order to prevent any degradation in oil quality as discussed below.

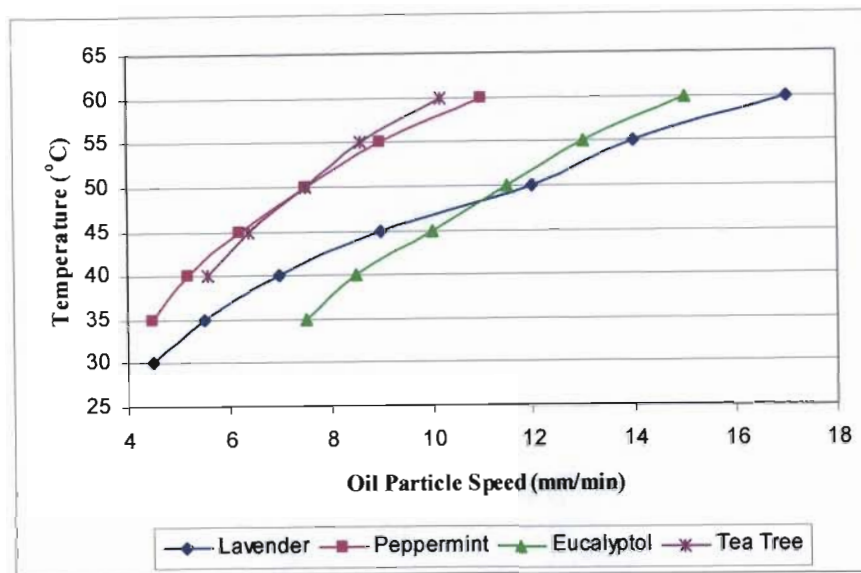


Figure 2.11 Oil particle speeds at certain temperatures for lavender, peppermint, eucalyptol and tea tree oil (adapted from results obtained from Denny, 1999).

### 2.8.3.3 Storage and packaging

Most essential oils can be stored for long periods under suitable conditions. They should be free of water, not in contact with air or direct sunlight and they should also be kept cool (Axtell and Fairman, 1992).

Once the oil has been separated, it should be filtered in order to remove any debris that has found their way into the oil. To prevent the oil from any aging reactions, the small amounts of dissolved water present in the oil should be removed. This is done by filtering the oil through a bed of anhydrous sodium sulphate (Lawrence, 1995). Oils generally show no cloudiness when dry (Axtell and Fairmen, 1992). Even when the oils are stored dry, in clean new drums, inferior odours or flavours may develop in the product due to the action of oxygen present (oxidation) in the small amount of air between the oil and the top of the drum. This can be minimised by replacing the air with dry nitrogen gas (Denny, 1999).

Tinted glass containers are often used to store small amounts of oil while larger quantities are invariably stored in metal drums. Mild steel drums that are lined with epoxy resin are very popular. When second-hand drums are used, it is important that they are thoroughly cleaned and dried before being filled with essential oil (Axtell and Fairmen, 1992).

Freshly distilled oils often possess some still odours that are unpleasant. These generally disappear after several weeks of storage. Some oils gradually improve in storage and acquire a fuller more rounded aroma (Axtell and Fairmen, 1992).

#### **2.8.3.4 Oil quality**

Conditions of distillation must be carefully established and controlled according to the nature of the raw material to ensure optimum yields of quality oil. Extended periods of distillation adversely affect the oil quality and also the cost of distillation. The yield of the oil depends on efficient separation of the essential oil from the steam/oil condensate (Sankarikutty and Narayanan, 1993).

The produced oil should meet the international standards of odour, chemical composition and overall quality (Lawrence, 1995). The uniformity of the odour or flavour between different batches of produced oil is also important to the buyer as the formula cannot be changed with every fresh drum received. There will however always be some variation in the produced oils, although this may be minimised where clonal control of the herbaceous source is being practised. Attention must also be paid to the purity of the oil, and hence an effort should be made to try and minimise the contamination of impurities as far as possible (Denny, 1999).

Commercial essential oils are required to comply with sets of standards (e.g. International Standards Organisation (ISO)). The standards are numerical values of refractive index, optical rotations, ester values, freezing points, acid values, carbonyl values, phenol contents and chemical compositions with lower and/or upper limits. Oils whose numerical values fall within

the accepted range can find their way into the market and can be used in whatever industry it is needed (Baser, 1995).

#### **2.8.4 Boiler**

The size of the boiler will depend on the amount of steam required to adequately remove the oil from the still charge (Guenther, 1948). According to Denny (1999), the steam flow rate should be at least between 2 and 4 litres per minute per square meter of charge cross sectional area. Boilers in field distillation units generally have a working pressure of about 7 bar, with the stills operating at atmospheric pressure (Guenther, 1948).

There are many different types of boilers available such as electrode, coal fired, diesel fired, gas fired, and heavy furnace oil boilers. Certain factors influence the type of boiler selected such as the size of the required unit, cost of fuel, existing infrastructure on the farm, location of the farm, future expansion plans and personal preference of the farm management. Each boiler type is also available in different sizes with steam flow rates produced under a variety of pressure ranges. Boilers operating at high pressures (approximately 7 bar or higher) are used to attain higher temperatures rather than merely to force the steam through the plant material contained in the distillation unit. Higher temperatures and pressures penetrate the plant material more effectively with less condensation in the still. As a result, these boilers can be more efficient with regards to distillation, by shortening the distillation time for certain crop types (Guenther, 1948).

Steam tends to become superheated when it is produced at high pressure in the boiler and enters the charge vessel at a far lower pressure. When the steam is superheated, the temperature within the charge vessel will no longer stay close to the boiling point of water, but will rise to the temperature of the superheated steam. It is thus essential for the operator to monitor the temperature within the charge vessel to avoid overheating.

With subcutaneous oils, most of the oil is only vaporised once it has diffused to the outside of the plant material. Diffusion can however only take place if hot water is present at the surface of the material and is thus slowed down or stopped when the charge is completely dried by the

superheated steam. With superficial oils, the oils start evaporating from the plant materials surface at the points where intermingling of the oil and water occurs. Superheated steam will again hinder the extraction process especially with plant materials containing low moisture contents (Guenther, 1948).

It is advisable to start of the distillations with steam produced at lower pressures. The pressure can then be increased towards the end of the distillation, when the oil content present in the plant material decreased, and when mainly the high boiling constituents of the essential oil remain in the plant material (Guenther, 1948).

### **3. MOBILE DISTILLATION UNIT COMPONENT DESIGN METHODOLOGY**

When designing a complete essential oil distillation system, it is vital that the entire systems components are in balance with each other. This means that the boiler must be suitably sized to supply the necessary amount of steam to process the required amount of plant material per day. Consequently, the condenser must be able to condense all the vapours at the maximum steam flow rate to the desired temperature and the separator should be designed to accommodate the maximum condensate flow and allow for complete oil separation. The entire steam distribution system must also be able to accommodate maximum steam flow at the required pressure range. In order to explore the various distillation parameters, a test distillation apparatus was constructed.

#### **3.1 Laboratory Test Distillation**

A small test distillation apparatus was set up in the School of Chemical Engineering main laboratory at the University of KwaZulu-Natal in Durban (shown in Figure 3.1). This equipment was used to become familiarised with the distillation process and to investigate the effects of varying steam flow rates on the distillation time, oil yield and oil quality.

##### **3.1.1 Apparatus components**

The distillation unit was constructed from glassware available in the School of Chemical Engineering and the entire unit was mounted onto a frame. The charge vessel was sized to hold a significant amount of plant material so that measurable quantities of oil could be extracted from the crops at 2, 3 or 4 minute time intervals, depending on the steam flow rate.

A glass cylinder with an inner diameter of 220 mm and a height of 700 mm was used as the charge vessel. The cylinder had glass caps fixed on either ends of it with bolts. The bottom cap had two openings, one for the steam inlet and the second had a drain tap for draining the reflux flow that collected in the bottom of the vessel. The top cap had an opening connected to the

condenser. A piece of gauze, 215 mm in diameter, was positioned at the bottom of the cylinder to support the plant material. A rod with a handle, which extended above the cylinder top, was fitted to lift the gauze with the distilled plant material out of the cylinder after a distillation as shown in Figure 3.2. The entire charge vessel, together with the end caps, was insulated with 25 mm thick Fiberflax insulation material in order to reduce the amount of condensation and hence reduce the reflux flow inside the charge vessel.

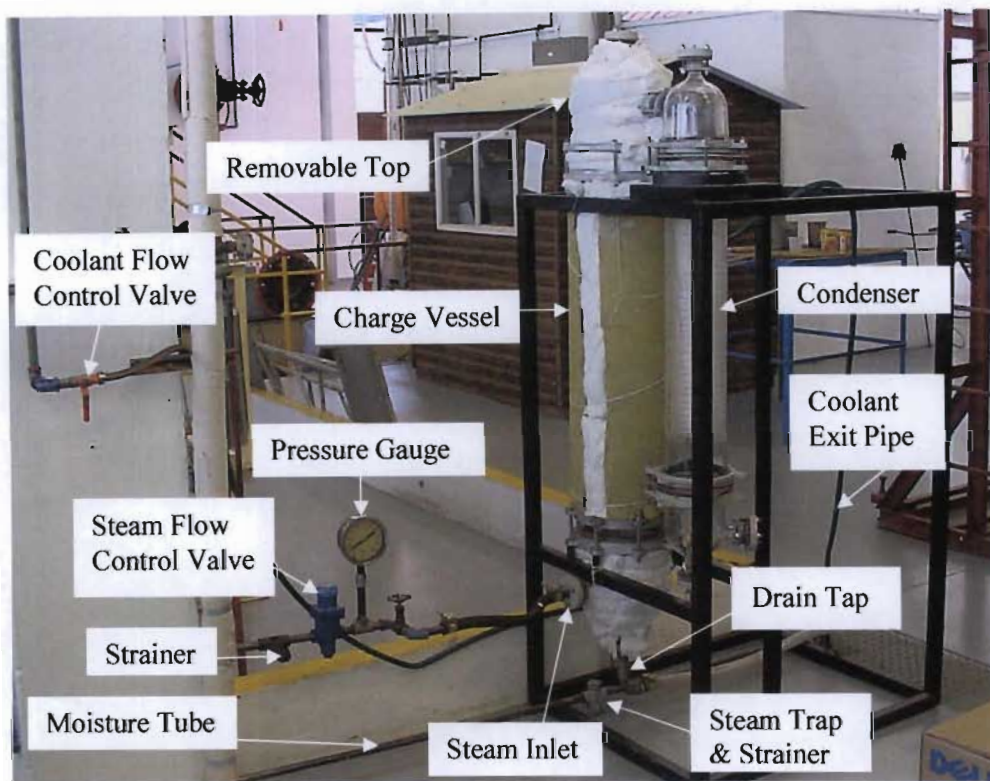


Figure 3.1 Test distillation apparatus.

A coiled glass condenser with two glass caps bolted on either end was used. The vapours were transferred from the vessel to the condenser through the two connected top caps. The vapours entered the general space of the condenser while the coolant water flowed through the coil from the bottom to the top of the condenser. A control valve was fitted to vary the coolant flow rate. The bottom cap had a distillate outlet pipe mounted to it. The top caps of both the charge vessel and the condenser had to be removed to charge and remove plant material from the charge vessel (Figure 3.2).



Figure 3.2 Removing charge from the test distillation apparatus.

Steam was delivered to the still by a steam line connected to one of the steam outlet points in the laboratory. The steam line consisted of a strainer, steam flow control valve and a pressure gauge, as shown in Figure 3.1. The purpose of the strainer was to remove any small unwanted particles such as pipe scale and rust, which would damage the steam flow control valve if they entered it (Anon, 2004).

A tube was positioned below the steam line to the distillation unit (Figure 3.1). All the moisture that had condensed in the steam delivery pipes could accumulate in the lower moisture tube before entering the steam line to the vessel and as a result the steam could enter the charge vessel in a dry state. Moisture or condensate in the steam line can lead to increased wear and tear of control valves, to leakages and also have a negative effect on the distillation process if not removed. A thermodynamic steam trap and strainer combination was fitted into the moisture tube. A steam trap is an automatic valve that opens to air and condensate and hence is responsible for removing condensate from a steam line (Anon, 2004). The accumulated moisture in the lower tube could thus be discharged by the steam trap.

### 3.1.2 Methodology and test procedure

Test distillations were performed with lemon grass and rosemary. In order to prevent reflux flow from occurring, the steam flow rate should be kept between 2 kg/min/m<sup>2</sup> and 4 kg/min/m<sup>2</sup>, which respectively amounted to 4.8 kg/h and 9.5 kg/h for the test unit (Denny, 1999). Three steam flow rates were used for testing, two within the abovementioned range and a third at a higher rate.

The lowest steam flow rate that could successfully be used for distilling was 6 kg of steam per hour. This was due to the performance limitations of the boiler. The boiler generates steam until the upper pressure limit was reached and then it would cut out until the pressure reduced to the lower limit. As a result, the steam supply to the various outlet points in the lab would fluctuate. When the steam flow rate for the distillation unit was set to an average below 6 kg/h, very little distillate would exit the distillation unit when the boiler reached the lower half of its pressure range and hence a lower steam flow rate was not used.

The three selected steam flow rates for the testing procedure were approximately 6, 8.5 and 12 kg/h. At least two distillations, with precisely the same packing density and mass of plant material, were conducted for each flow rate with each crop. The testing procedure for each distillation was conducted as follows:

1. Crops were harvested on the morning of distillation and the weather conditions were recorded.
2. The distillation unit was calibrated to the required steam flow rate.
3. The vessel was charged with a known mass of plant material to maximum capacity. The lemon grass was cut to lengths of approximately 120 mm for easier charging.
4. Once the top glass caps were fastened after charging, the distillation was started and the heating time was measured. The heating time is the time from when the distillation was started till the first drops of condensate flowed out of the condenser.
5. Samples were taken at 2, 3 or 4 minute intervals, depending on the steam flow rate. The amount of oil and hydrosol for each sample was measured.
6. Each sample was emptied into a separating funnel.

7. At the end of the distillation, the oil was separated from the hydrosol and the total oil yield was noted.
8. All the reflux flow was drained from the charge vessel and measured.
9. The total plant material was weighed after distillation and the change in mass was calculated.
10. A sample of fresh and distilled plant material was weighed and then placed in a drying oven. The dried samples were then weighed and the moisture content of each was calculated.
11. A curve of oil produced against the elapsed time was plotted.
12. The distillation or extraction time was estimated from the curve.
13. A gas chromatographic (GC) analysis was performed on the extracted oil.

The weather conditions were noted as the weather prior to the harvest has an effect on the oil yield. It is recommended that the crops are harvested only after they have received a couple of hours of sunlight. This is mainly to reduce the moisture content of the plant material caused by dew. The increased moisture content can increase the distillation time and also increase the amount of reflux, depending on the absorptive capacity of the plant material. With some crops there are some valuable chemical components whose percentages increase when the plant warms up and hence it is advantageous to harvest the crops only once they have warmed up (Figueiredo, 2004).

### **3.2 Design Considerations**

The mobile distillation unit had to have with a charge capacity of approximately 250 kg of plant material and an efficient charging and discharging mechanism in order to minimize labour requirements.

From the start of the project it was clear that electricity would be needed for such a system, as even a diesel fired steam generator requires electricity to ignite the burner fuel and also to run the electric motors that drive the feed water pump and fan. Electricity would further be needed for

the lifting mechanism and the condenser cooling system (discussed in Section 3.3.1 and Section 3.7.2 respectively). Hence a generator would be required.

The distillation unit was to be mounted onto a frame, which in turn was fixed onto a double axle 2 ton trailer. The steam generator with all its accessories and the generator were mounted onto a separate frame, which in turn was mounted onto a second trailer. The design procedure was split into the following categories:

- Charge vessel and loading mechanism
- Steam generator and steam distribution system
- Condenser and water cooling system
- Separator
- Electrical system

The materials used for the construction are of vital importance. Care had to be taken to ensure that the vapours containing volatile oils, as well as the extracted oils in liquid form are only in contact with stainless steel (Grade 304 or 316) or glass as essential oils are extremely corrosive. Stainless steel and glass are also easy to clean after each distillation, as they have no absorptive properties. Other materials have the tendency to absorb a little essential oil, which cannot be removed and as a result a certain odour will always adhere to the still. This can easily spoil the fragrance of a different type of oil with the next distillation and hence reduce its value (Guenther, 1948). It is recommended that Teflon is used as the material for gaskets (Denny, 1999). Teflon is a polytetrafluoroethene (PTFE) plastic that is extremely corrosion resistant, has no absorptive properties and has a temperature index greater than 400 °C and consequently will not melt (Maistry, 2004).

The basic properties of 304 and 316 stainless steel are (SASSDA, 1999):

- Excellent corrosion resistance.
- Easily cleanable and has an excellent hygiene factor for product purity, and non-contamination nor tainting of foodstuffs occurs.
- Excellent welding properties.
- Good elevated high temperature strength.

- Excellent fabrication properties.

Grade 316 stainless steel has a higher resistance to pitting and crevice corrosion in chloride environments. Grade 304 stainless steel (cheaper than Grade 316) was however suitable for the distillation system, as the material is not exposed to a severe chloride environment (Mortimer, 2004). Typical applications for Grade 304 stainless steel are (SASSDA, 1999):

- Food processing equipment i.e. beer brewing, milk processing and wine making
- Kitchen sinks, equipment and appliance
- Chemical containers and screens for mining, quarrying & water filtration
- Heat exchangers
- Threaded fasteners and springs

The design of the charge vessel as well as its loading mechanism will be discussed in the following section. Technical drawings of all the designed components are represented in APPENDIX D.

### **3.3 Charge Vessel**

The charge vessel is a tank into which the plant material is placed for distilling. A charge vessel should be sized to accommodate the required mass of plant material and should also have a suitable charging and discharging mechanism to minimize the downtime associated with the process.

As mentioned earlier, the vessel had to be designed to accommodate approximately 250 kg of plant material. Two charge vessels were used, with the intention that the one vessel can cool down and be emptied and reloaded respectively while the other vessel is distilling, thus increasing the productivity of the unit (discussed in Section 4.2).

The vessels were also sized in such a way to make efficient use of material used to construct them in order to minimise off cuts. The dimensions of the trailer onto which the vessels were

mounted also had to be taken into account. An average packing density of  $300 \text{ kg/m}^3$  (Denny, 1999), together with the abovementioned factors was used to suitably size the charge vessels illustrated in Figure 3.3 below. The charge vessels were manufactured from 2 mm thick Grade 304 stainless steel sheets and were mounted onto a constructed frame. Detailed technical drawings of the complete charge vessel with all the attachments and the frame onto which both vessels were mounted are illustrated in Figures D.1 and Figure D.2 respectively (APPENDIX D). The calculations for sizing the section members of the frame are contained in APPENDIX A.

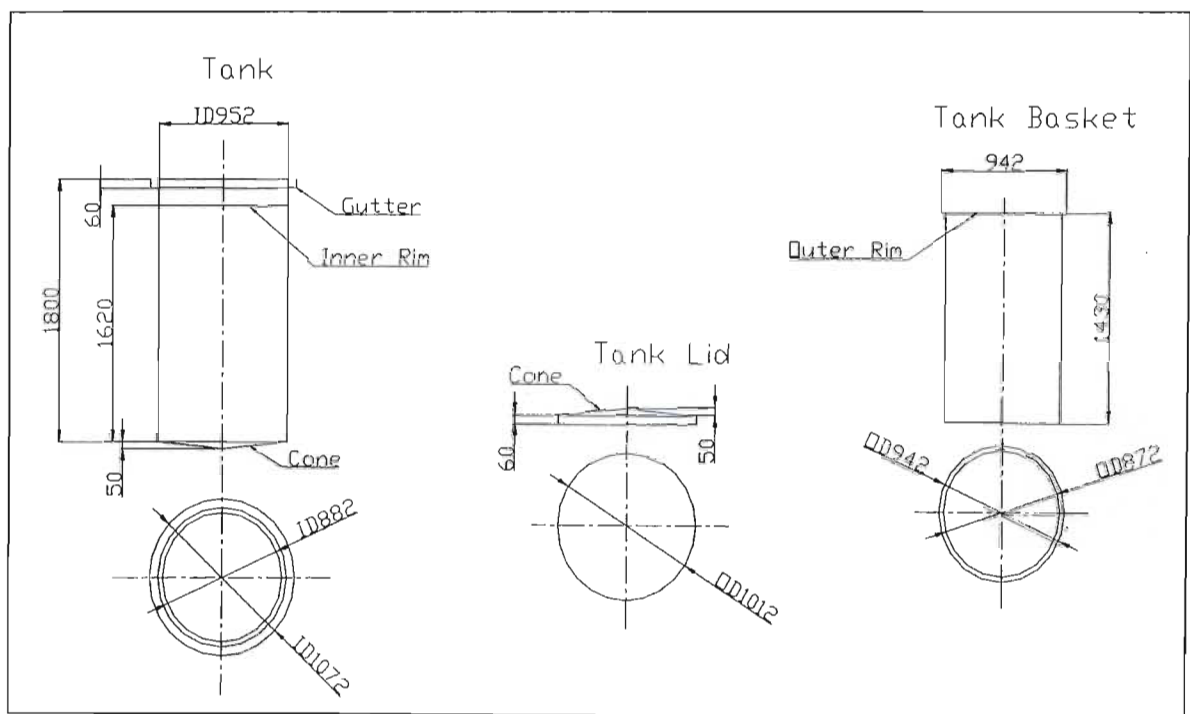


Figure 3.3 Dimensions of charge vessel, basket and lid without ancillary components.

The next step was to design an efficient charging and discharging system for the two charge vessels.

### 3.3.1 Charging and discharging mechanism

A basket mechanism was used for charging and discharging the charge vessel with plant material. With this method, a basket filled with plant material supported by a grid at the bottom of the basket, is placed inside the vessel as shown in Figure 3.4. An inner rim was fixed to the inside of

the charge vessel as shown in Figure 3.3, which supports the basket top when positioned inside the vessel. The basket was manufactured from the same material as the charge vessel. Both the outer rim of the basket and the inner rim of the charge vessel were however manufactured from 4.5 mm Grade 304 stainless steel. Thicker material was used as the weight of the entire basket loaded with plant material is supported by these rims.

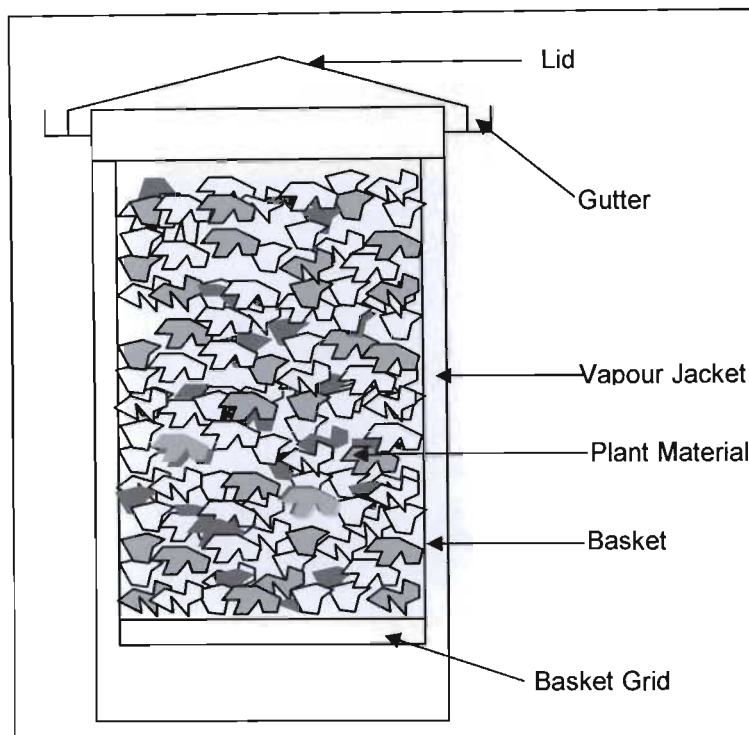


Figure 3.4 Charge vessel with inserted basket.

Once a distillation is complete, the entire basket is lifted out of the vessel, by means of a crane fitted with an electric hoist that is powered by a generator (illustrated in Figure 3.5). The basket is then emptied and reloaded next to the still at ground level. Two eye bolts were mounted adjacent to each other on top of the basket onto which a chain can be connected for lifting. An advantage with this system is that plant material need not to be lifted up to the top of the charge vessel for loading.

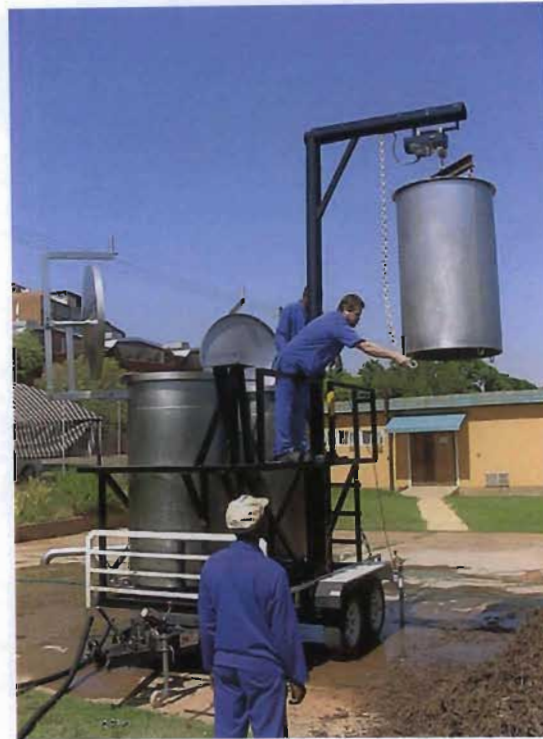


Figure 3.5 Basket lifted from charge vessel.

When a charged basket is positioned inside the charge vessel, a 40 mm gap exists between the outer basket and tank wall. This gap is filled with steam during distillation and acts as an insulation layer (Figure 3.4). This insulation vapour jacket reduces the amount of condensation that could take place on the inner walls of the basket and hence reduces reflux flow and consequently oil losses. It was thus not necessary to insulate the tank with insulation material. A disadvantage of this method is that the charge vessel accommodates less plant material due to the reduction in cross sectional area.

The lid outer rim of each vessel is positioned in a water-filled gutter that surrounds the top of the vessel (when the lid is closed), creating a water seal as illustrated in Figure 3.4. The water seal also acts as a safety valve through which the pressure can be relieved if it rises above the designed pressure, due to blockages or a closed valve. Generally hinged or threaded clamps are used to clamp the lid to the vessel with a gasket for sealing. A disadvantage with the conventional method is that a large amount of time is lost when opening and closing the vessel.

Due to the position of the lids outer rim in the gutter, a suitable lid opening mechanism had to be designed to ensure that the lid of each charge vessel could be easily opened and closed with a minimum time requirement. The lid mechanism illustrated in Figure 3.6, had to be designed so that the lid first lifts out of the gutter when the handle is lifted, in order for its rim to clear the outer wall of the gutter, before it can pivot about the hinge fixed to the charge vessel. Three hinging points were needed for the mechanism to work. The dimensions of the sections used for both the basket and the components of the lid mechanism are shown in Figure D.1 (APPENDIX D). The crane is an essential component for the charging and discharging mechanism and hence the productivity of the unit will be affected by the functioning of it.



Figure 3.6 Charge vessel lid with opening mechanism.

### 3.3.2 Crane design

Failure of the crane when loaded could be fatal to the operator of the system and it is for this reason that a safety factor of 3 was used in the design calculations illustrated in APPENDIX A.

A bush was constructed from Nylatron GSM bushing stock. The Nylatron GSM material was selected due to its excellent wear resistance, self lubricating and low friction characteristics (Minne, 2004). The bush was machined to fit inside the bottom of the crane tubing. The inside of

the bush was then fitted over a shaft machined from bright steel (Figure 3.7) that was welded to the base of the frame. This formed a bearing which enabled the crane to rotate. The calculations for checking both the strength of the bush and the force required to rotate the crane are shown in APPENDIX A. A thrust bearing was also considered but was decided against due to the cost and maintenance (greasing) requirements. A thrust bearing also has extremely low friction characteristics and some friction is needed in order for the operator to stop the crane when rotating with a load.

The crane was supported higher up by a plate that was fixed to the distillation unit frame in order to reduce its effective length for bending. The plate had a hole machined through its centre through which the crane passes. Three grooves were machined into the plate 120° apart to accommodate three bearings that can be adjusted to support the crane with minimal play as shown in Figure 3.8 below.



Figure 3.7 Nylatron GSM bush with bright steel shaft.

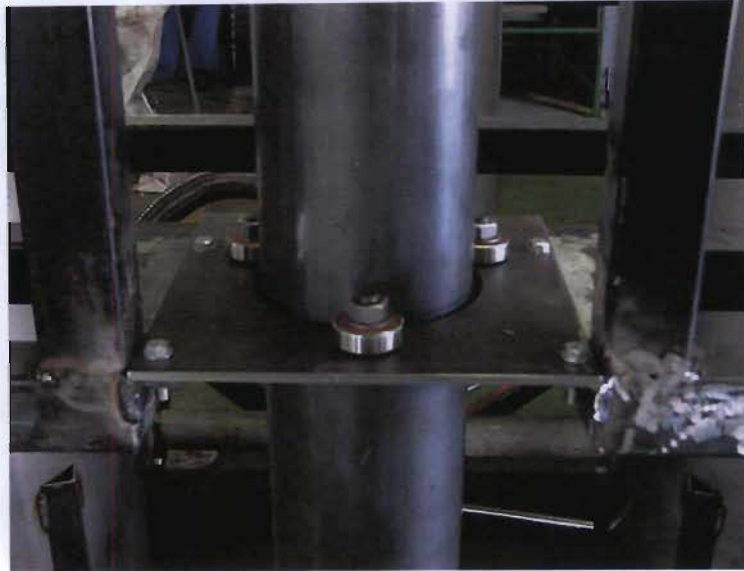


Figure 3.8 Crane top support plate.

In order to reduce the height of the crane for transport purposes, hinges were installed. This made it possible for the crane to fold down and the unit could thus be easily transported without excessive height limitations (Figure 3.9). The crane was supported by a saddle that formed part of the frame when folded down. When the crane was folded down into the transporting position it was difficult to raise it again due to its weight. A pillar to which the electric hoist can be connected was constructed on the opposite end of the frame. The hitching point of the pillar is higher than the hinging point of the crane and hence the electric hoist could be used to raise and also lower the crane into the two different positions.

At the point where the crane is hinged, a plate was welded on either pipe ends to which the hinges were fixed. The two plates were bolted together when the crane was in the operating position, to prevent the crane from folding down. Three support girders were welded in on either sides of the plate for support and two handles were also fixed to the crane in order for the operator to rotate the crane as shown in Figure 3.10. Detailed technical drawings of the crane

structure, the bush and shaft and the top support plate are illustrated in Figures D.3, Figure D.4 and Figure D.5 respectively (APPENDIX D).



Figure 3.9 Mobile distillation unit with the crane in the transporting position.



Figure 3.10 Crane hinge point with the support girders and handles.

Before the condenser for the distillation system could be designed, it was necessary to first determine the quantity of steam flow required to distil the designed mass of plant material and hence the quantity of steam flow that will need to be condensed.

### **3.4 Sizing the Steam Generator**

A steam generator should be sized in such a way that the required amount of plant material can be processed in a certain time, as a distillery's handling capacity is proportional to the mass of steam available per hour (Denny, 1999).

A distillation production model that can calculate the production of a distillation system based on the distillery size, steam flow rate, type of crop and time taken to recharge the still was set up in Microsoft Excel. With this model, it was possible to explore the effects that both different steam flow rates and also discharging and recharging times have on the production of an existing distillery. The calculation methods of the two models, one for superficial oil crops and the second for subcutaneous oil crops, are illustrated below. All the equations used in both the models have either been taken directly or adapted from Denny, 1999.

#### **3.4.1 Superficial oil crop model calculations**

An illustration of the superficial oil crop model with the equations used is represented in Figure B.1 (APPENDIX B). All the shaded cells in the model are the variables that need to be entered. The first step was to calculate the mass of the basket, as well as the mass of the plant material that the basket can hold. The total mass of the charge vessel basket with the bottom grid could be calculated by knowing that the density of stainless steel is  $8000 \text{ kg/m}^3$  (SASSDA, 1999). The mass of plant charge that the basket can accommodate was calculated by assuming a packing density of  $300 \text{ kg/m}^3$  (Denny, 1999). The amount of steam condensed to raise the temperature of the plant material from  $20 \text{ }^\circ\text{C}$  to  $100 \text{ }^\circ\text{C}$  and to raise the steel from  $20 \text{ }^\circ\text{C}$  to  $80 \text{ }^\circ\text{C}$  was estimated by Equations 3.1 and 3.2 respectively.

$$Q_{L_{cond}} = m.C_p.\Delta T \quad (3.1)$$

$$m_s = \frac{Q_{L_{cond}}}{Q_{L_s}} \quad (3.2)$$

- where;  $Q_{L_{cond}}$  = Latent heat removed during condensing (kJ)  
 $m$  = Mass (kg) of material (steel or plant material)  
 $C_p$  = Specific heat capacity of the material (kJ/kg.K)  
           Stainless steel = 0.885 kJ/kg.K (Perry and Green, 1997)  
           Plant material = 3.347 kJ/kg.K (Denny, 1999)  
 $\Delta T$  = Change in material temperature (K)  
 $m_s$  = Mass of steam condensed to raise the materials temperature (kg)  
 $Q_{L_s}$  = Latent heat of steam = 2255.59 kJ/kg at 100 °C (Perry and Green, 1997)

The total mass on the hoist was taken as the sum of the total amount of steam condensed to raise the temperatures of both the plant material and the steel, the mass of the plant material and the mass of the basket.

Three test distillations with lavender were conducted by Denny (1999) with the aim of estimating both the basic time ( $t$ ) and the increment parameter ( $s$ ) by the method discussed in Section 2.7.2. The following results were obtained from the tests:

- Basic time ( $t$ ) is 9.719 min with a steam displacement rate ( $\dot{m}_{test}$ ) of 3.09 kg/min/m<sup>2</sup> and an oil flow ( $q_{oil}$ ) of 9.75 ml/min/m<sup>2</sup> of charge cross sectional area.
- The increment parameter ( $s$ ) is 40.946 cm when the oil content of the lavender ( $Y_{test}$ ) was 9.09 ml/kg of plant material.

These parameters had to be adapted to the proposed distillation system, as the test distillations from which the parameters were derived were conducted in a system with different charge vessel dimensions and steam flow rates. For the rate of steam flow of the new still ( $\dot{m}_{calc}$ ) to be

equivalent to that of the test distillation steam displacement rate, the test steam displacement rate (3.09 kg/min/m<sup>2</sup>) had to be multiplied by the cross sectional area of the new still. The basic time (9.719 min) remained unchanged as rate of steam flow had been adjusted to be equivalent to that of the test distillations. If the expected yields differ from the yields obtained with the test distillation apparatus, the value of the ratio ( $s$ ) needs to be adjusted as shown in Equation 3.3 below.

$$s_{new} = s \left( \frac{Y_{test}}{Y_{expected}} \right) \quad (3.3)$$

where;  $s_{new}$  = Incremental parameter for new still

$Y_{test}$  = Test distillation oil yield (ml/kg)

$Y_{expected}$  = Expected oil yield (ml/kg)

Referring to Equation 3.3, if the expected yield is greater than the test yield, the value of  $s_{new}$  will be less than the original  $s$ . The reason for this is that the incremental area ( $\delta a$ ) that is added for each unit of increase in charge height will be greater due to the higher oil yield. Since  $s = a / \delta a$ , the value of  $s$  will decrease.

Having determined both the parameters  $s$  and  $t$  for the new still and knowing its actual height, it was possible to calculate the extraction time ( $T$ ) for lavender distillations with the new still by using Equation 2.4 in Section 2.7.2.

The time required to heat both the plant material and the steel ( $T_h$ ) was calculated by dividing the total amount of steam required to heat the two materials (calculated using Equation 3.2) by steam flow rate of the new still adapted from the test distillations ( $\dot{m}_{calc}$ ). The total distillation time ( $T_{tot}$ ) is then equal to the sum of the heating and extraction time.

The total time that the charge will be processed with the available steam flow rate of the proposed distillation system could be calculated as follows:

$$T_{distl} = T_{tot} \left( \frac{\dot{m}_{calc}}{\dot{m}_{avbl}} \right) \quad (3.4)$$

where;  $T_{distl}$  = Total distillation time with available steam flow rate (min)

$T_{tot}$  = Total distillation time with steam flow rate adapted from test distillation (min)

$\dot{m}_{calc}$  = Steam flow rate adapted from test distillations for the proposed system (kg/min)

$\dot{m}_{avbl}$  = Available steam flow rate (kg/min)

The total production of the distillation system was calculated by:

$$M_{prod} = \frac{m_c T_{day}}{\left( \frac{T_{distl} + T_{d\&r}}{60} \right)} \quad (3.5)$$

where;  $M_{prod}$  = The amount of plant material that can be processed in one working day (kg)

$m_c$  = The mass of plant material that the vessel holds (kg)

$T_{day}$  = Amount of working hours in a day (h)

$T_{distl}$  = Time to complete one distillation (min)

$T_{d\&r}$  = Time taken to discharge and recharge the basket (min)

Different values for the available steam flow rate could then be entered into the model, and the effect on the distillation time and production of the system for each entered value could then be monitored. The subcutaneous oil crop production model varies from the superficial oil crop production model as discussed in the next section.

### 3.4.2 Subcutaneous oil crop model calculations

An illustration of the superficial oil crop model with the equations used is represented in Figure B.2 (APPENDIX B) and the shaded cells are again the variables that need to be entered into the model. The mass and capacity of the cartridge, the mass of the plant charge and the amount of steam required to heat up both the materials is calculated in precisely the same manner as for the superficial oil crop model.

From *eucalyptus polybractea* test distillations that were conducted by Davis of Sydney the following results were obtained for the basic time ( $t$ ) and increment parameter ( $\delta$ ) by the method discussed in Section 2.7.4 (Denny, 1999):

- Basic time ( $t$ ) is 18.27 min with a steam displacement rate ( $\dot{m}_{test}$ ) of 1.361 kg/min/m<sup>2</sup> of charge cross sectional area.
- Increment Parameter ( $\delta$ ) is 0.411 min/cm when the oil content of *eucalyptus polybractea* is ( $Y_{Mtest}$ ) 15.09 ml/kg and ( $Y_{Vtest}$ ) 37.3 ml/m<sup>2</sup>.cm layer.

These parameters had to be adapted to the proposed distillation system. The test displacement rate ( $\dot{m}_{test}$ ) and oil content ( $Y_{Vtest}$ ) were multiplied by the proposed stills cross sectional area in order to determine the equivalent rate of steam flow ( $\dot{m}_{calc}$ ) in kg/min and oil content ( $Y_{calc}$ ) in ml/cm for the proposed still. The virtual height ( $H_{virt}$ ) of the proposed still was obtained by dividing the known or expected total oil yield (at 95 % EVE) by  $Y_{calc}$ . The basic time ( $t$ ) and the increment parameter ( $\delta$ ) from the test distillation results remained unchanged.

The extraction time ( $T$ ) of the proposed distillation unit could then be calculated (Equation 2.5 in Section 2.7.4) as the basic time ( $t$ ), increment parameter ( $\delta$ ) and virtual height ( $H_{virt}$ ) had all been determined for the proposed distillation unit and crop. If the available steam flow rate differs to the flow rate equivalent to the test distillations, then the calculated extraction time had to be adjusted by Equation 3.6 below.

$$T = \left( \frac{t + H.\delta t}{Z} \right) = \left( \frac{t + H.\delta t}{R.X^{\frac{2}{3}}} \right) \quad (3.6)$$

- where;  $Z$  = Factor of change in speed of oil recovery per unit time  
 $X$  = Factor of change in steam flow rate over the plant material surface  
 $R$  = Diffusion lag factor compared

The value of the steam speed factor ( $X$ ) was calculated by dividing the actual available steam flow rate ( $\dot{m}_{avbl}$ ) by the test equivalent steam flow rate of the proposed still ( $\dot{m}_{calc}$ ). The only remaining unknown variable was the lag factor and was obtained from Figure 2.4 in Section 2.7.4. From the figure it can be seen that it varies between 0.7 and 1.2 as the change in steam flow rate ( $X$ ) decreases from 3.5 to 0.5 respectively, for the specific eucalyptus crop. As both  $X$  and  $R$  had been determined, it was possible to calculate actual extraction time of the system.

The heating time ( $T_h$ ) required to heat up both the plant material and the metal was calculated by dividing the total mass of steam required to heat up the materials ( $m_s$ ) by the available steam flow rate ( $\dot{m}_{avbl}$ ). Hence the total distillation time could be calculated by adding the extraction time and the heating time. The total daily production of the distillation system was then calculated the same way as for the superficial oil crop with Equation 3.5. From the steam requirement calculation models for the two crop types it was possible to get an indication of the amount of steam flow required for the designed distillation unit. It was thus possible to size the required steam generator for the system.

### 3.5 Steam Generator and Feed Water Treatment Plant

A diesel fired steam generator that can produce 250 kg/h of steam at a pressure of up to 10 bar was acquired for the system. The decision on the size of the steam generator was based on the steam requirements calculation model results as discussed in Section 4.2. The steam generator with some of the ancillary equipment is shown in Figure 3.12.

With the acquired steam generator, the feed water passes from the feed water tank through a tube wound up into serial connected tube coils. The diesel is pumped through an atomiser and the fine vapours are ignited, producing a flame that heats the tube coils. The feed water inside the tube coil is heated up to the boiling point temperature inside the coil and then evaporates. Steam generation is thus a once through process unlike other systems, where there is a certain level of water within the boiler (Bouillart, 2004). Advantages of the discussed system are (Anon, 2004):

- They are easy to operate and boiler authorisation requirement is not necessary as there is no water level in the unit, making it safe.
- The amount of water treatment required is minimal. Only softened water is needed and no other chemicals need to be added to the feed water.
- Start-up is quick and steam is available at full pressure within a few minutes.
- Steam is only drawn when required and hence no storage is required.
- They are compact and hence suitable for mobile units.

Softening of the feed water prior to entering the steam generator is necessary as it affects the quality of the produced steam as well as the efficiency and life of the steam generator itself. If hard feed water enters the steam generator, scaling of the heat transfer surfaces will occur within the tube coil of the steam generator, reducing heat transfer through the tube walls as well as the life of the tube. In extreme cases, local hot spots can occur, leading to mechanical damage or even tube failure (Anon, 2004). The water should thus first pass through an ion exchange water softening plant before it enters the feed water tank and then the boiler (Bouillart, 2004).

The softening plant consists of a resin tank containing a bed of ion exchange resin that removes the calcium and magnesium hardness from the water. The bed is originally charged by passing 7 – 12 % of brine (sodium chloride or salt) through it leaving the resin rich in sodium ions. As the water passes through the resin tank, the calcium and magnesium hardness is exchanged with an equivalent amount of sodium (Figure 3.11) that possesses no hardness properties (Anon, 2004). The water softening plant used for the acquired steam generator is shown in Figure 3.12.

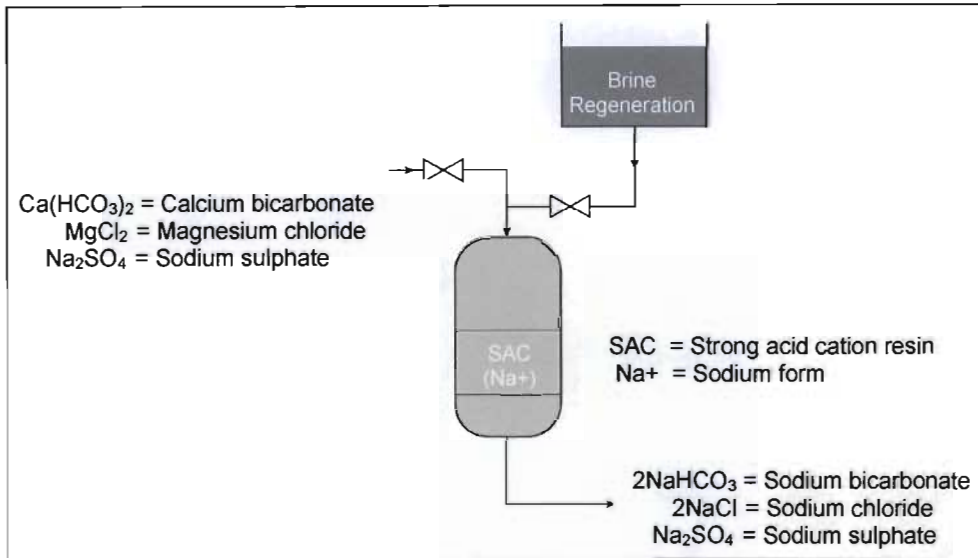


Figure 3.11 Ion exchange water softening plant (after Anon, 2004).

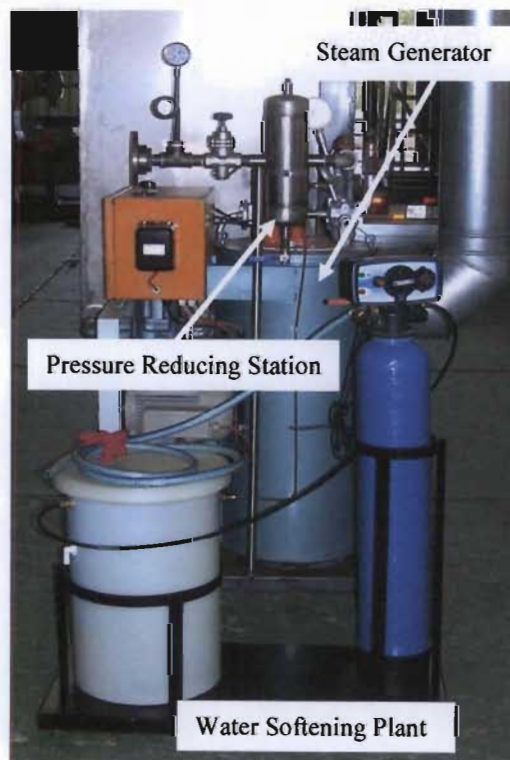


Figure 3.12 Steam generator with pressure reducing station and water softening plant.

It is recommended that the feed water is preheated before it enters the steam generator. It is usually preheated in the feed water tank by injecting a quantity of the produced steam into the water when starting up. While the system is in operation, the hot condensate from the steam traps within the system is returned to the feed water tank to maintain the raised feed water temperature. This would reduce the energy required by the steam generator to produce steam. A further advantage of preheating the feed water is that the amount of oxygen in the water is reduced. Oxygen present in the feed water can cause pitting within the tube coil and hence reduces its lifespan (Bouillart, 2004).

The feed water tank was designed to accommodate 240 litres of water. It was manufactured from 1.6 mm thick 3CR12 stainless steel sheet material, as it is cheaper than Grade 304 and the tank is not exposed to a highly corrosive environment. A decision was made not to preheat the feed water even though the energy consumption of the steam generator will be higher and there is a risk of shell damage. This was mainly due to the high cost of a steam injector and the steam line to the feed water tank. By referring to Figure 3.13 it can also be seen that the reduction in oxygen content when the water temperature is increased from 25 °C to 60 °C is approximately 34 %. At 60 °C there is still oxygen present and hence there is still a possibility that pitting will occur within the shell. The tube coil will be monitored during the course of the season and if signs of excessive pitting occur in the future, a preheating system will be installed.

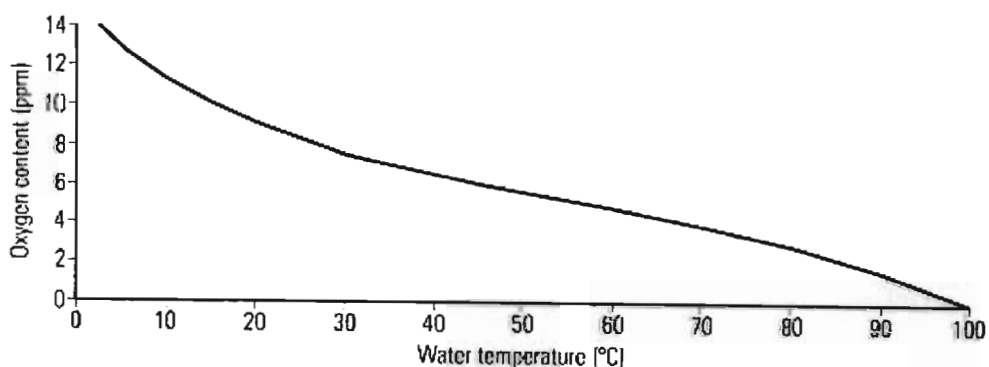


Figure 3.13 Water temperature versus oxygen content (after Anon, 2004).

The diesel consumption of the steam generator is 22 litres per hour (Bouillart, 2004). A 250 litre diesel tank was manufactured from 1.6mm mild steel sheeting as diesel has lubricating properties

and hence costly stainless steel is not necessary. With a tank capacity of 250 litres, the distillation unit will be able to operate for approximately 11 hours without refuelling.

Once steam is produced, the steam needs to be distributed from the steam generator to the charge vessel. The steam distribution system together with all the components is discussed below.

### **3.6 Steam Distribution System**

A suitable steam distribution system was designed to convey the steam from the steam generator to the distillation unit. On entering the steam chamber at the bottom of the charge vessel, the steam needs to be evenly distributed by a steam manifold, in order for it to move uniformly through the loaded plant material.

A pressure reducing valve (PRV) was fitted in the steam line so that the steam pressure can be reduced before it enters the distillation unit. In this way the temperature of the steam is also reduced. The PRV was sized to reduce the pressure from 10 bar to approximately 1 bar or lower. It is important to make sure that there is no water present in the steam when it flows through the costly PRV as it can damage it (Anon, 2004). For this reason, a separator was fitted before the PRV. The function of the separator is to remove any moisture from the steam line (Frankel, 1996). The separator has a steam trap fitted to the bottom of it, which is responsible for dispersing the accumulated water. The accumulated water is good quality hot water and is usually returned to the feed water tank. With large steam distribution systems with multiple steam traps, the hot water returned from all the steam traps is usually sufficient to maintain the feed water at a high temperature (Beckley, 2004). In order to prevent any scale or other unwanted objects from entering the steam line and damage components such as the steam trap and PRV, a strainer was installed at the steam generators outlet.

The layout of a typical pressure reducing station is represented in Figure 3.14. An isolation valve is positioned before and after the PRV, which is mainly for maintenance purposes. A pressure gauge is fitted on either side of the PRV so that the pressure on either side can be monitored. The function of the safety valve is to prevent over pressurising of the steam line for safety purposes.

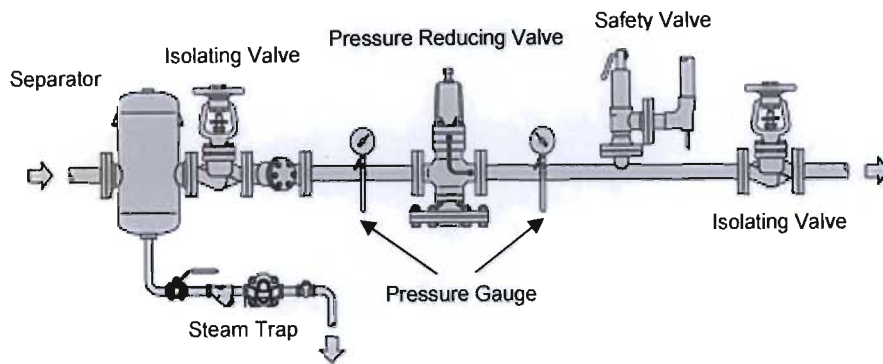


Figure 3.14 Standard pressure reducing station (Anon, 2004).

The pressure reducing station fitted to the steam generator is illustrated in Figure 3.12. The steam is conveyed from the end of the pressure reducing station to the charge vessel via a 50 mm flexible stainless steel hose. The steam line from the steam generator has a diameter of 25 mm and is then expanded to 50 mm below the PRV due to the reduction in pressure. The flexible hose was split into two parts, the one is flanged to the end of the pressure reducing station and the other is flanged to the three-way control valve that directs steam flow to either of the two charge vessels. The trailer containing the distillation unit and the trailer containing the steam generator could then be parked alongside each other and the flexible hoses could be connected.

A three-way plug valve (88.9 mm diameter) was installed to direct the flow to either of the charge vessels. The steam enters the charge vessel through a steam manifold that evenly distributes the steam. The steam manifold shown in Figure 3.15 below, was constructed from 76.2×2 mm stainless steel tube that was rolled into two half circles that were then welded together. The largest pipe size that generally can be rolled is 76 mm, which is why the pipe diameter decreased from 88.9 mm to 76.2 mm at the steam manifold. Holes through which the steam enters the charge vessel were drilled on the inside of the ring. A technical drawing of the steam manifold is represented in Figure D.1 in APPENDIX D.



Figure 3.15 Steam manifold.

The steam then moves through the perforated grid and up through the plant material. The vapours exit the charge vessel at the top and are then directed down to the condenser by a second 88.9 mm three-way plug valve. The condensed vapours then leave the condenser and enter the separator. It is important that all the vapours are condensed when the distillate leaves the condenser, if not oil will be lost to the atmosphere.

### 3.7 Condenser Design

A multi-tubular condenser was designed for the distillation system according to the method discussed in Section 2.8.2.2. The condenser design had three constraints:

- The vapour had to be condensed and cooled to an optimum separating temperature of approximately 45 °C (Denny, 1999).
- The coolant water leaving the condenser had to enter the cooling tower at a temperature below 55 °C as specified by the cooling tower manufacturers. A temperature above 55 °C affects the viscosity and hence the bonding ability of the resin in the fibreglass cooling tower. Cooling towers that can cool water entering at temperatures higher than 55 °C are available but require greater capital outlay.

- The coolant velocity between the tubes should not be greater than approximately 0.15 m/s. The heat transfer from the cooling water to the condensing tubes and hence the vapours, is directly proportional to the coolant velocity between the tubes. If the coolant velocity between the tubes is too high, the turbulence of the cooling water could hamper the heat transfer capacity and hence reduce the effectiveness of the condenser.

The first step was to size the pipe through which the vapours move from the charge vessel to the condenser. Once the pipe size was calculated, the size of the opening of the condenser was known and the required condenser size was then determined.

### 3.7.1 Condenser connecting pipe and backpressure

Figure 2.6 in Section 2.8.2.1 was used to size the connecting pipe. The operating pressure of the vessel is close to atmospheric pressure, but will build up slightly to form the necessary pressure gradient to force the vapours through the connecting pipe and then the condenser. The maximum pressure assumed to build up inside the vessel was 1400 Pa (0.014 bar), the temperature of the steam is approximately 100 °C and the maximum steam flow rate to occur is 250 kg/h. An acceptable pressure loss through the connecting pipe was taken as 480 Pa (0.0048 bar) (Denny, 1999). The pressure loss and the length of the connecting pipe, with the associated losses added to its length (illustrated in Table 2.3) were used to calculate the expected pressure loss per 100 m length.

The above results were used to select a suitable pipe size from Figure 2.6. An 88.9 mm (3" nominal bore) pipe was chosen. The 88.9 mm is larger than required (80 mm), but in this way would still be suitable if the pressure within the charge vessel were slightly lower than expected.

Since both the operating pressure of the charge vessel and the pressure loss through the connecting pipe were known, it was possible to estimate the backpressure due to expelling air at the start of the distillation (894 Pa) by substituting the variables into Equation 2.15. Once the

entire distillation unit had been constructed, the backpressure was measured as 1010 Pa, which was slightly higher than estimated.

### 3.7.2 Condenser size

A condenser design model was set up in Microsoft Excel using the condenser design method discussed in Section 2.8.2.2 and is illustrated in Figure B.3 (APPENDIX B). The model could be used to determine the required condenser length for different shell diameters, tube numbers, tube sizes and coolant flow rates for it to perform within the required constraints. The effect of varying the coolant flow rate on distillate exit temperature, coolant exit temperature and coolant velocity between the tubes could also be monitored for different coolant and steam flow rates for a set condenser size.

The model could thus be used to design a condenser that would perform within the required parameters. A 42 tube condenser with 19.05 mm tubes (1.2 mm wall thickness), a shell diameter of 356 mm, coolant baffles 400mm apart and a length of 830 mm was found to be sufficient. A detailed technical drawing of the condenser is shown in Figure D.6 (APPENDIX D). The results were checked with a simpler calculation method also obtained from Denny (1999) in order to verify them. The calculated length from the second method was slightly less and hence a condenser with dimensions obtained from the condenser design model was constructed. A vapour baffle was positioned inside the inlet cone to the condenser with the function of dispersing the vapours so that the vapours pass through all the condenser tubes. Diagrams of the vapour and cooling water baffles are represented in Figure D.6 (APPENDIX D).

Curves representing the distillate exit temperatures, the coolant water exit temperatures and the coolant velocity between the tubes for various coolant flow rates were plotted for the condenser at steam flow rates of 225, 180 and 150 kg/h and are illustrated in Figure 3.16, Figure 3.17 and Figure 3.18 respectively. Different steam flow rates were used in the design to ensure that the condenser would be able to perform satisfactorily if the steam flow rate was varied.

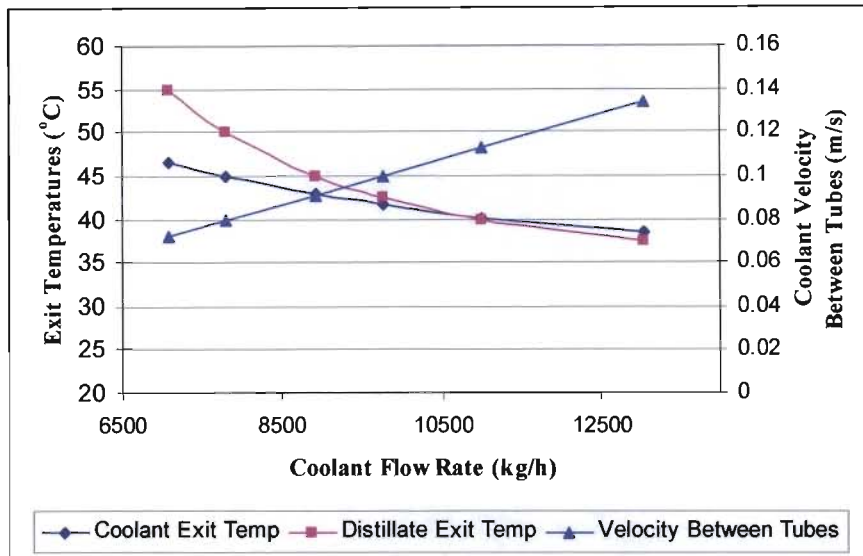


Figure 3.16 Coolant exit temperature, distillate exit temperature and coolant velocity curves for a steam flow rate of 225 kg/h and a coolant entering temperature of 28 °C.

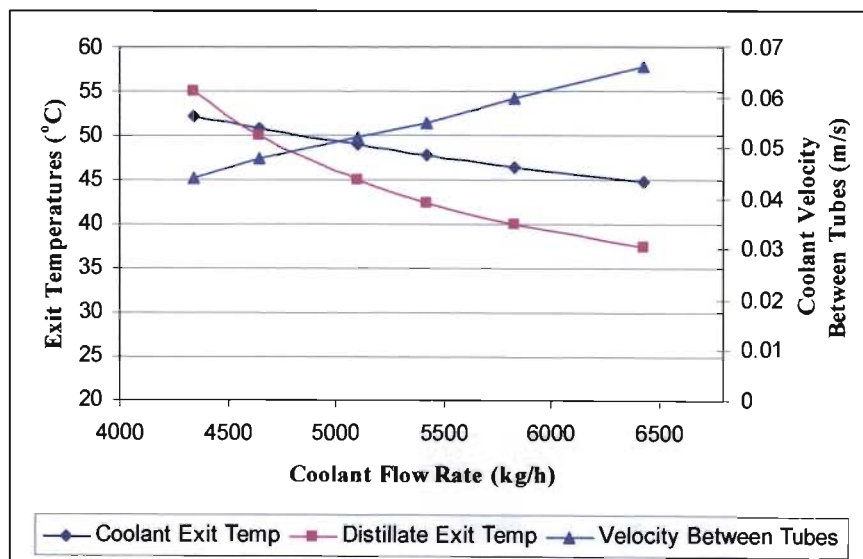


Figure 3.17 Coolant exit temperature, distillate exit temperature and coolant velocity curves for a steam flow rate of 180 kg/h and a coolant entering temperature of 28 °C.

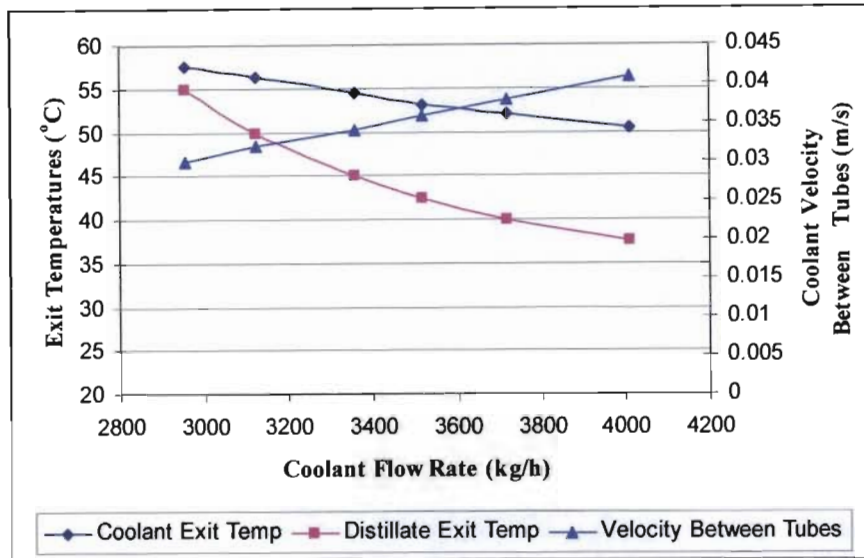


Figure 3.18 Coolant exit temperature, distillate exit temperature and coolant velocity curves for a steam flow rate of 150 kg/h and a coolant entering temperature of 28 °C.

From the figures it can be seen that for a fixed distillate exit temperature the required coolant flow rate decreases when the steam flow rate is reduced. The coolant exit temperature increases when the coolant flow rate decreases. For a steam flow rate of 150 kg/h and a distillate exit temperature of 45 °C, the coolant exit temperature is just below 55 °C. Hence for steam flow rates of 150 kg/h or lower, the coolant exit temperature will be around the maximum or higher if the coolant flow rate was less than 3300 kg/h. Even though steam flow rates lower than 150 kg/h should not be required for the system, higher coolant flow rates could reduce the coolant exit temperature if required. This would however also reduce the distillate exit temperature and increase the required oil separation time (Section 2.8.3.1). The separator was designed for a steam flow rate of 250 kg/h and hence additional separation time will be available for lower steam flow rates. A deduction can thus be made that according to the model, the designed condenser will perform within the constraints discussed in Section 3.7 for the various steam flow rates.

Once the required condenser size had been determined, it was possible to size the coolant water pump. From Figure 3.16 it can be seen that a coolant flow rate of approximately 9000 kg/h is required to condense and cool the distillate to a temperature of 45 °C with a steam flow rate of

225 kg/h as shown in Figure 3.16. Since the accuracy of the model was not known, the coolant pump was sized to be larger than required and can pump at a maximum flow rate of 15000 kg/h with a dynamic head of 11.6 m. The cooling tower was only sized after the entire system had been constructed and the performance of the condenser had practically been determined.

### 3.8 Separator Design

The required separator dimensions were calculated according to the method discussed in Section 2.8.3.2. A maximum flow rate of 225 kg/h was used and the lowest oil particle speed through water at a temperature of 45 °C (6 mm/s) was selected from Figure 2.11. The dimensions of the separator designed according to the above parameters are shown in Figure 3.19 below with a volume of 220 l (excluding the top neck). If one were to distil lavender at a lower steam flow rate of 160 kg/h for example, one distillation would take approximately 30 minutes according to the results obtained from the steam requirement calculation model illustrated in Figure 4.9. For one complete distillation approximately 80 l of distillate would have condensed which means that two distillations would not fill the separator and hence no oil discharge would occur after two distillations. The separator would thus not be suitable for distillations set at a low steam flow rate.

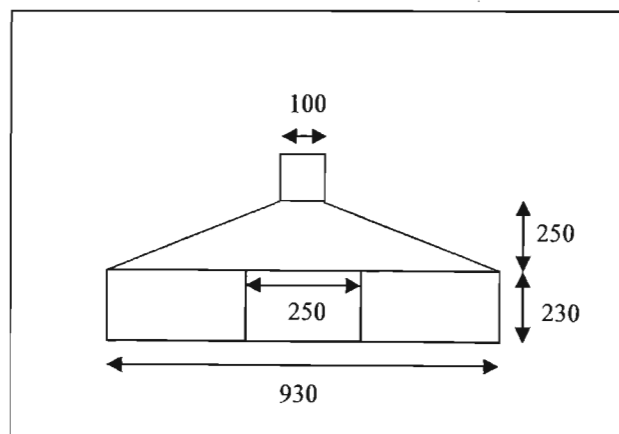


Figure 3.19 Dimensions of the separator designed for a distillate flow rate of 225 kg/h and an oil particle speed of 6 mm/s.

The separator was thus redesigned for a lower steam flow rate of 200 kg/h and an oil particle speed of 9 mm/s, even though oils with slower particle speeds would be distilled in the future. The design method discussed in Section 2.8.3.2 assumes that there is a continuous distillate flow into the separator and with the designed system, the distillate flow will seize after each distillation, for the time required to switch the two valves to the second vessel plus the heating time of the new vessel. As a result, the outer cylinder of the separator could theoretically be designed for the hydrosol speed to exceed the oil particle speed in the opposite direction by a small magnitude, since the oil particles could make up the lost height when distillation stops. Hence it was deduced that the designed separator would be able to separate oils efficiently, even when the oil particle speed is slower than the speed used in the design calculations.

An illustration of the final separator designed according to the discussed parameters is shown in Figure 3.20 and a technical drawing is illustrated in Figure D.7 (APPENDIX D). The inner cylinder is lower than the outer cylinder in order to reduce the volume of the separator. The volume of the separator is 112 l and would have been 193 l if the outer cylinder extended to the bottom of the inner cylinder.



Figure 3.20 The designed separator.

In order to vary the height of the hydrosol outlet as discussed in Section 2.3.8.2, a pipe formed into an s-bend was threaded to the horizontal outlet point. A clear glass bottle was fitted to the top of the separator so that the oil level and oil outlet point could be observed while adjusting the hydrosol outlet. The legs were fitted with screw feet that can be adjusted for levelling the separator on uneven surfaces. Once the distillation procedure was complete, the separator could be drained by unscrewing the drain plug fitted to the t-piece at the bottom of the funnel pipe. The drain plug could also be used to drain water when rinsing the separator after distilling.

### **3.9 Construction, Assembly and Commissioning**

All the components of the distillation unit were constructed at the Agricultural Research Council –Institute for Agricultural Engineering (ARC-ILI) workshop. Components that required accurate cutting from stainless steel sheet metal were outsourced for laser cutting and the rolling of cylinders and cones for the condenser, charge vessels and separator were also outsourced. Once all the stainless steel components were constructed and assembled, they were chemically pickled and passivated in the welded areas in order to prevent corrosion from taking place in these chromium depleted areas. Pickling is the removal of high temperature scale and any adjacent low chromium layer of metal from the surface by chemical means. Passivation is the treatment of the area with acid (usually nitric acid) solutions or pastes in order to remove the contaminants and promote the formation of an oxide film which increases the corrosion resistance (SASSDA, 1999).

The system was commissioned once the distillation unit and the steam generating and distribution system had been assembled and mounted onto the trailers as illustrated in Figure 3.21 below. The baskets were charged with plant material, as it affects the pressure below and above the basket within the charge vessel. The Pressure will be higher below the basket, due to the resistance to the steam flow caused by the packed plant material. There will pressure drops as the vapour moves through the plant material and hence the pressure will be lower above the basket than without plant material.



Figure 3.21 Complete mobile essential oil distillation system.

On start-up steam leaked through the vessel lid water seal. The 50 mm deep water seal was thus not deep enough to accommodate the condenser back pressure. In order to overcome the problem it was decided to insert a seal into the gutter and the charge vessel lid was then clamped down by means of a turnbuckle fixed to the distillation unit frame. The material selected for sealing was EPDM closed cell foam. A second option was to increase the depth of the gutter but was declined due to the cost and time required for the modification. High temperature rubber seals were also fixed to the charge vessel inner rims that support the baskets. These prevent steam from escaping the vapour jacket through the charge vessel inner rim and the basket outer rim and hence all the produced steam will be forced to move through the plant material.

A safety u-tube that acts as a pressure relief valve had to be installed on each vessel. The u-tube would be filled with water which would be blown out to relieve the pressure if it rises above the designed pressure within the vessel due to blockages or a closed valve. In order to determine the required u-tube height, the back pressure was measured while the system was running (excluding the cooling water) with the installed seal and a maximum pressure of 1010 Pa (103 mm) was measured. The u-tubes were made higher than required (350 mm) and were installed at the top of both charge vessels as shown in Figure D.1 (APPENDIX D). In order to monitor the temperature and pressure above and below the plant charge within the charge vessel during distillations,

sockets for inserting pressure and temperature gauges were installed at the top and bottom side of both charge vessels. Field tests were conducted once all the modifications had been completed.

### **3.10 Field Testing**

Rose geranium and lemon grass were available for field testing. The cooling tower had not yet been acquired and hence the distillations were conducted on the ARC-ILI premises where cooling water could be pumped from a reservoir.

The one charge vessel was fitted with a pressure and temperature gauge at the steam inlet and a temperature gauge at the vapour outlet of the charge vessel. In this way the steam condition at the inlet to the charge vessel and the vapour outlet temperature could be monitored. At least two distillations were conducted for each crop type. The testing procedure for each distillation was conducted as follows:

1. Crops were harvested on the morning of distillation.
2. The basket was loaded and weighed to determine the mass of plant material.
3. The distillation was started and the heating time was recorded.
4. The pressure and temperature readings on the boiler, pressure reducing station and charge vessel were recorded once the distillation was running.
5. The extraction time was recorded.
6. After each distillation the oil was tapped from the separator and the total oil yield was noted.
7. The plant material was weighed after distillation and the change in mass was calculated.
8. The accumulated reflux flow in the bottom of the charge vessel was measured.

The lemon grass was obtained from a different grower and area as the lemon grass obtained for the laboratory distillations. As a result the chemical compositions of the extracted oils would differ and there would be no value in comparing gas chromatographic (GC) analyses results for the two samples. The operating conditions within the charge vessel could be monitored during the distillations to ensure that no thermal decomposition of the oil takes place. It was not possible to take samples of the distillate at fixed intervals as large measuring cylinders (5.5 l for 2 min

intervals) were not available. As a result oil production curves could not be constructed for the distillations as the amount of oil and hydrosol present at fixed time interval could not be determined. These curves are usually only plotted for lab test distillations as it is easier to deal with the small volumes due to the low distillate flow.

## 4. RESULTS AND DISCUSSION

The results and discussions of the laboratory distillations, the steam requirement calculation model, the distillation unit condenser performance and field testing of the designed distillation unit are discussed in this section.

### 4.1 Laboratory Distillations

The laboratory distillations were conducted with lemon grass and rosemary. The lemon grass was freshly harvested on the same day as distillation and each batch came from the same field. The rosemary had been harvested the previous season, was dried and then passed through a hammer mill in order to separate the needles from the woody twigs. Both crops had been grown organically and hence no chemical fertilisers or pesticides had been used. They were fertilised with chicken litter and sprayed with kakiebos.

Due to the time consumed harvesting the crops, calibrating the test distillation unit, charging and discharging the unit, taking measurements and cleaning the unit after the last distillation it was only possible to conduct two distillations for each steam flow rate in one day. The results obtained from the distillations for the three steam flow rates for both crops are discussed below.

#### 4.1.1 Lemon grass

The lemon grass plants used for the test distillations were approximately three years old. For each distillation 5.5 kg of fresh lemon grass cut to 120 mm lengths, was packed into the charge vessel with a packing density of 207 kg/m<sup>3</sup>.

Two days before the lemon grass was harvested for the 13.3 kg/h steam flow rate distillation tests, the weather was inclement and the day prior to harvesting became sunny in the afternoon. The lemon grass was exposed to approximately two hours of sunlight before harvesting in the morning. Two distillations were performed with a steam flow rate of 13.3 kg/h. The distillation

curves are illustrated in Figure 4.1 below and the average results from the two distillations are contained in Table 4.1 below. It must be noted that the oil yields were calculated on a mass basis i.e. kg of oil per kg of plant material.

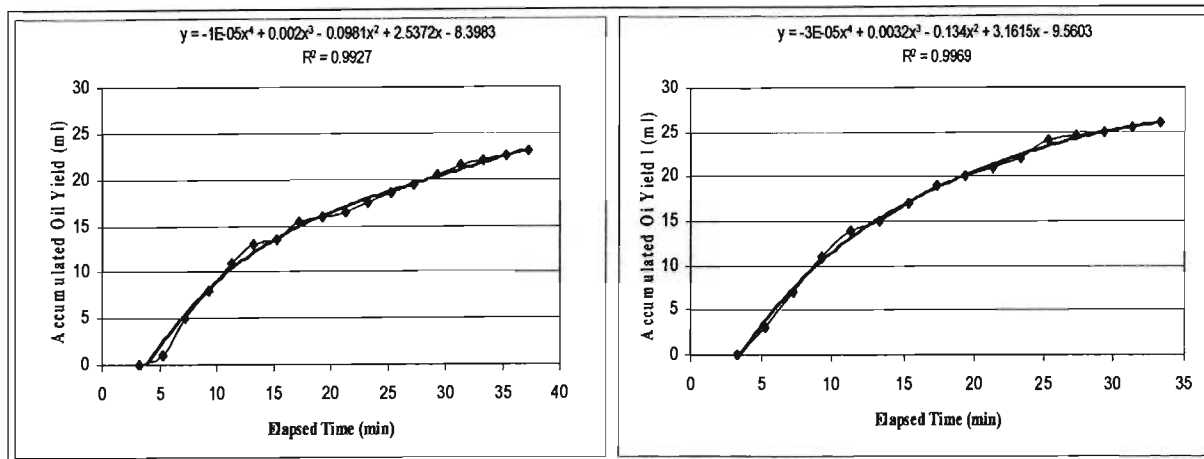


Figure 4.1 Lemon grass oil yield curves for an average steam flow rate of 13.3 kg/h.

Table 4.1 Average lemon grass results for 13.3 kg/h steam flow.

Average Steam Flow (kg/h)	13.3
Average Yield (%)	0.41
Average Reflux Flow per Distillation (ml)	430
Average Distilling Time (min)	35.26
Lemon grass moisture content prior to distillation (%)	72.97

The weather on the days prior to the 8.9 kg/h and 6.2 kg/h distillation tests were both sunny and then overcast on morning of harvesting with no rain. The crops were harvested from the same field and the distillation curves and average results obtained are shown in Figure 4.2 and Table 4.2 for the 8.9 kg/h distillations and in Figure 4.3 and Table 4.3 for the 6.2 distillations.

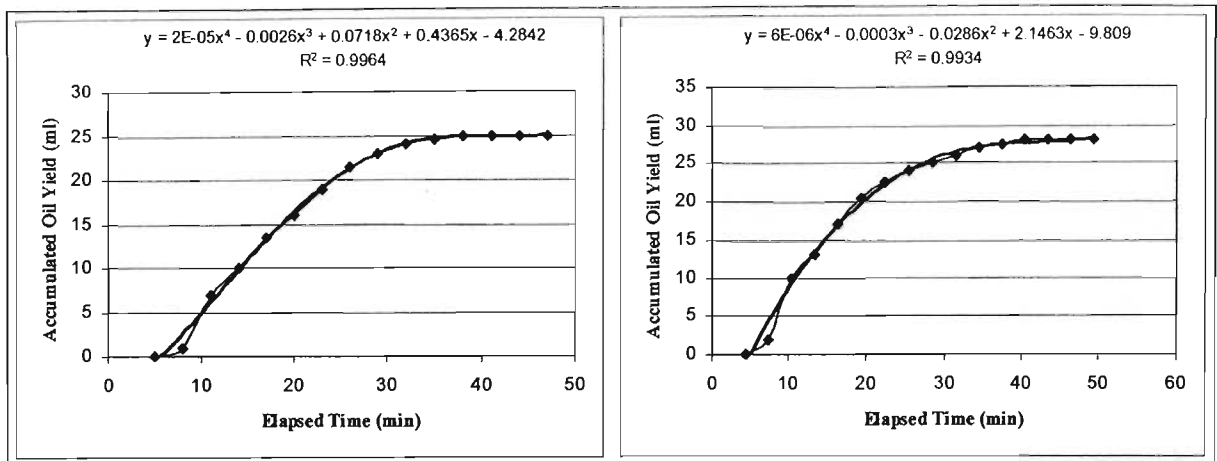


Figure 4.2 Lemon grass oil yield curves for an average steam flow rate of 8.9 kg/h.

Table 4.2 Average lemon grass results for 8.9 kg/h steam flow.

Average Steam Flow (kg/h)	8.9
Average Yield (%)	0.49
Average Reflux Flow per Distillation (ml)	530
Average Distilling Time (min)	48.16
Lemon grass moisture content prior to distillation (%)	72.00

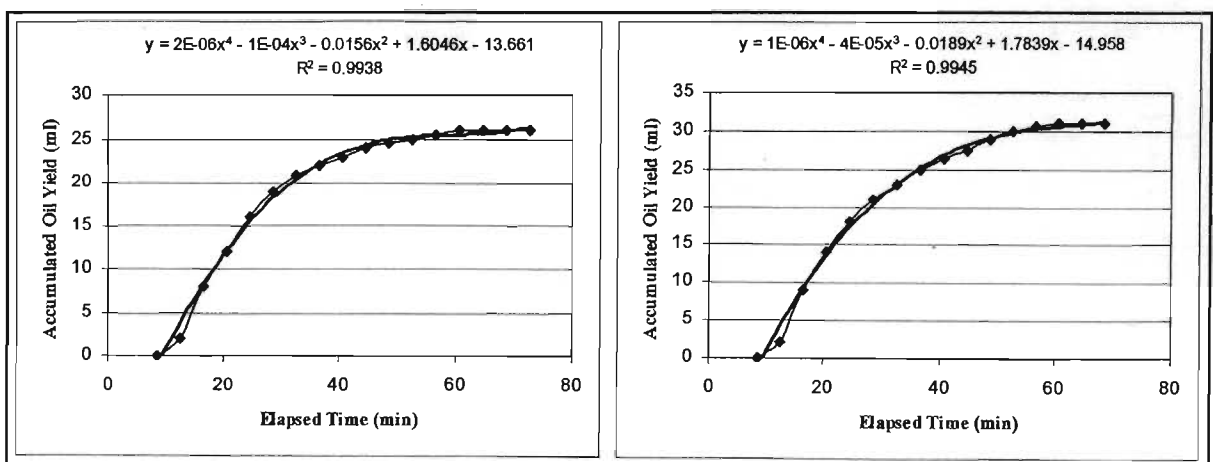


Figure 4.3 Lemon grass yield curve for an average steam flow rate of 6.2 kg/h.

Table 4.3 Average lemon grass results for 6.2 kg/h steam flow.

Average Steam Flow (kg/h)	6.2
Average Yield (%)	0.52
Average Reflux Flow per Distillation (ml)	802.5
Average Distilling Time (min)	70.46
Lemon grass moisture content prior to distillation (%)	74.07

For all the distillations that were conducted, there were no signs of “rat holes” that are usually formed by steam finding easier paths through plant material. This indicates that the material was evenly packed and hence the steam could move up evenly through the plant material. After distilling the plant material was very moist and hence heavier than when loaded into the charge vessel. The average increase in mass of the plant material after distilling was calculated as 10.01%.

The lemon grass distillation time and oil yield results from Table 4.1, Table 4.2 and Table 4.3 are represented in Figure 4.4 below. The oil yields obtained were above the normal lemon grass yield range of 0.2 – 0.4 % (Sankarikutty and Narayanan, 1993). From Figure 4.4 it can be seen that the average distillation time increased with a decrease in steam flow rate, while the oil yield increased. With a reduction of steam flow from approximately 13 kg/h to 9 kg/h there is a 37 % increase in distillation time and also a significant increase in oil yield (19.5 % yield increase). With a further reduction of steam flow to approximately 6 kg/h, there is a similar increase in distillation (32 %) time while the increase in oil yield is only 6 %. The moisture content of the plant material for each of the distillations was similar and hence would have had no effect on the distillation times.

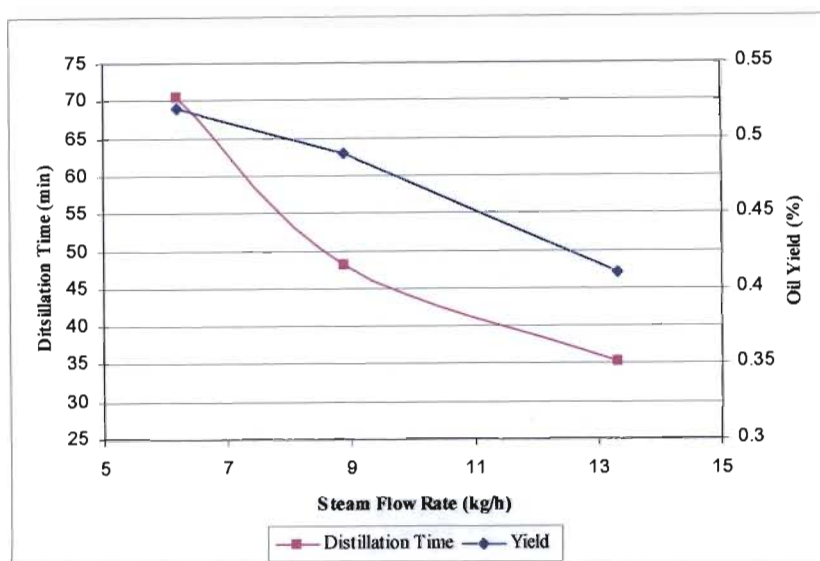


Figure 4.4 Lemon grass extraction times and oil yields for the tested steam flow rate range.

The average reflux flow for each distillation was higher for lower steam flow rates as can be observed from Table 4.1, Table 4.2 and Table 4.3. Since reflux flow is associated with oil losses, it is interesting to note that the oil yield increased with a decrease in steam flow rate, while the reflux flow also increased. In other words, even though the oil losses due to the reflux flow were higher, the yields still increased. It can thus be deduced that there was still oil left in the plant material for the higher steam flow rates and hence the distillations were ended too early. This deduction is supported by comparing Figure 4.1 to Figure 4.3. It can be seen that the distillation end points were not reached for the distillations conducted with steam flow rates of approximately 13 kg/h as the curve for each distillation does not end asymptotically with the time axis, as is the case with the lower steam flow rate distillation curves. This explains the large drop in yield from 9 kg/h to 13 kg/h of steam flow in Figure 4.4.

From the oil yield curves it can be seen that most of the oil is extracted in the first half of the distillation and the amount of oil extracted per unit time is far less near the end of the distillation. At high steam flow rates the distillate ratio, which is the ratio of oil to hydrosol, is lower than for distillations of the same crop with lower steam flow rates if samples are taken at fixed time intervals. This made it extremely difficult to determine the end point for the high steam flow rate distillations, as it was difficult to observe whether there was still oil present in the samples near

the end. As a result, no oil could have been recorded for the samples near then end of the distillation even though some oil was still being extracted at small quantities. The oil yield curves constructed from the sample records would then look like the end point was reached, even though not all the oil had been extracted. It is due to this reason that there was such a large decrease in oil yield for an increase in steam flow rate as the distillations were ended too early.

Due to the low yields and value of the oil, it is vital that the distillation end point is reached. With certain oils, such as vetiver or angelica root oil, the most valuable constituents are extracted at the end of the distillation process (highest boiling fractions). In cases like this, the distillation must be prolonged for hours, even though it seems as though no further oil is extracted towards the end of the operation. If this is not done, the valuable constituents with a high boiling point will be lacking in the oil and hence greatly reduce its value (Guenther, 1948).

From the gas chromatographic (GC) analysis results of the lemon grass oils produced with the three steam flow rates illustrated in Table C.1 (APPENDIX C) it can be seen that there is not a significant difference between the chemical constituents of the produced oil for each steam flow rate. The chemical composition of the produced oil can also be affected by the temperature of the condensate exiting the condenser (De Figueiredo, 2004). The condensate temperature for the test distillations was not consistent for the three steam flow rates. The varying condensate exit temperature and the chemical composition of the oil within the plant itself would have contributed to the minor differences in chemical compositions of the oil.

It can thus be deduced that the distillation steam flow rate has a major effect on the distillation time with shorter distillation times for higher steam flow rates and no effect on the chemical composition of the extracted oils. The oil yield decreased for an increase in steam flow rate, but this was because the distillations ended too early. The effect of different steam flow rates on the distillation time, yield and oil composition for rosemary will now be discussed.

#### 4.1.2 Rosemary

The rosemary distillations were conducted in precisely the same manner as the lemon grass distillations. The crops were harvested the previous season and then dried. The charge vessel was packed with 5 kg of dried rosemary with a packing density of  $188 \text{ kg/m}^3$ . The reason for this low packing density is because the crop had been dried, thus containing a low moisture content.

All the rosemary was harvested from the same field. The distillation curves for the various steam flow rates are illustrated in Figure 4.5, Figure 4.6 and Figure 4.7 and the average results obtained from the distillations are shown in Table 4.4, Table 4.5 and Table 4.6 below.

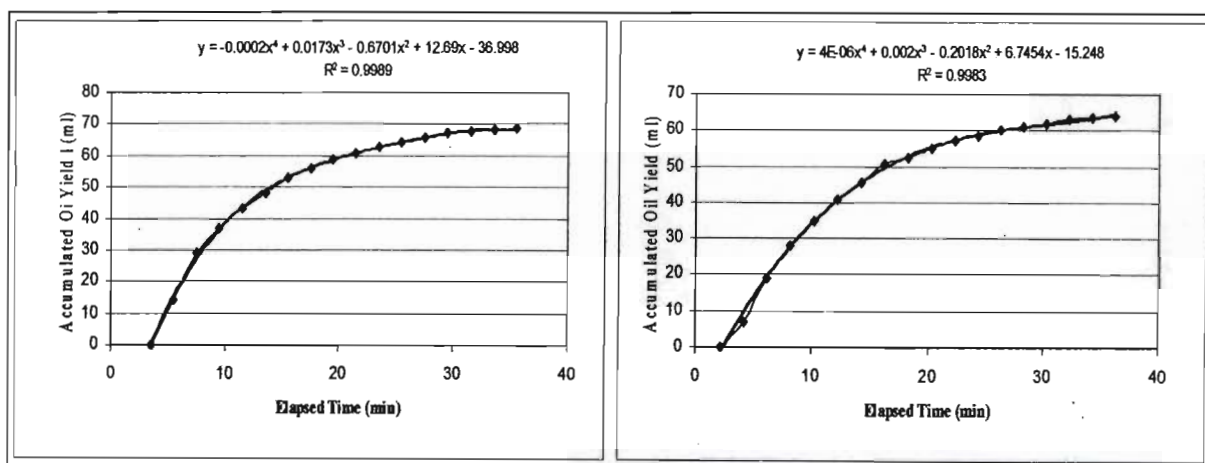


Figure 4.5 Rosemary oil yield curves for an average steam flow rate of 11.1 kg/h.

Table 4.4 Average rosemary results for 11.1 kg/h steam flow.

Average Steam Flow (kg/h)	11.1
Average Yield (%)	1.52
Average Reflux Flow per Distillation (ml)	525
Average Distilling Time (min)	35.9
Rosemary moisture content prior to distillation (%)	8.11

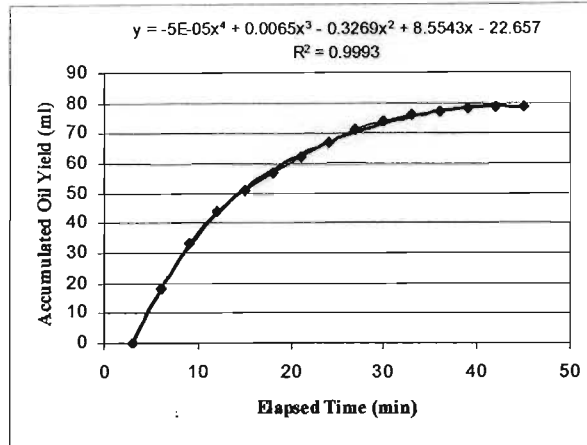


Figure 4.6 Rosemary oil yield curve for a steam flow rate of 8.7 kg/h.

Table 4.5 Average rosemary results for 8.7 kg/h steam flow.

Average Steam Flow (kg/h)	8.8
Average Yield (%)	1.58
Average Reflux Flow per Distillation (ml)	330.0
Average Distilling Time (min)	45
Rosemary moisture content prior to distillation (%)	12.82

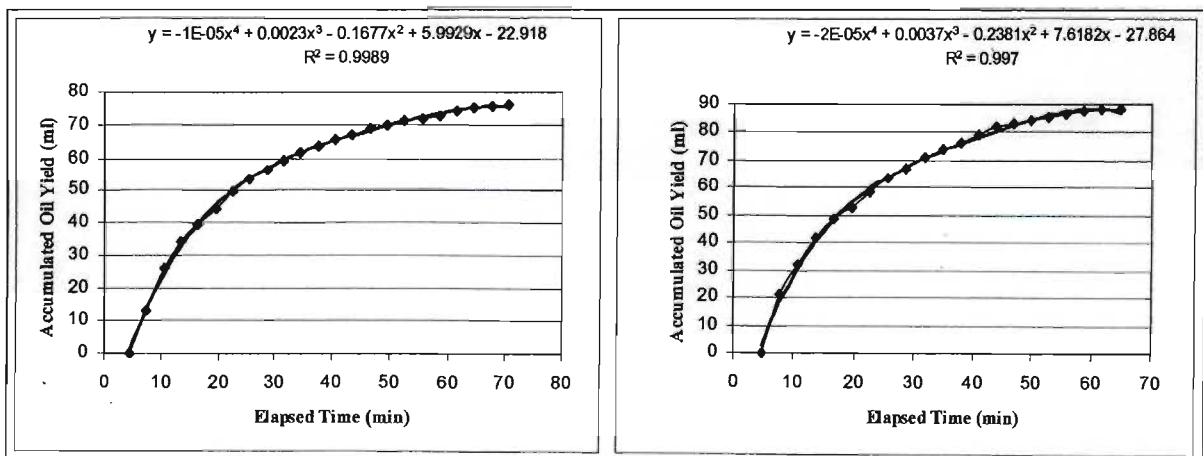


Figure 4.7 Rosemary oil yield curves for an average steam flow rate of 6.1 kg/h.

Table 4.6 Average rosemary results for 6.1 kg/h steam flow.

Average Steam Flow (kg/h)	6.1
Average Yield (%)	1.65
Average Reflux Flow per Distillation (ml)	645
Average Distilling Time (min)	67.6
Rosemary moisture content prior to distillation (%)	9.53

There were no signs of “rat holes” present in the distilled plant material for all the conducted distillations. Due to the low moisture content and physical properties of the rosemary, the plant material was less moist than the lemon grass after distilling. The average increase in mass was measured as only 4.67 %.

The rosemary distillation time and oil yield results from Table 4.4, Table 4.5 and

Table 4.6 are represented in Figure 4.8 below. From the Figure it can be seen that the average distillation or extraction time increased with a decrease in steam flow rate, while the oil yield increased. With a reduction of steam flow from approximately 11 kg/h to 9 kg/h there is a 25% increase in distillation time while the increase is more significant when the flow rate is reduced from approximately 9 kg/h to 6 kg/h (50 % increase). The oil yield increase for the same reductions in steam flow is more or less consistent at 10 %.

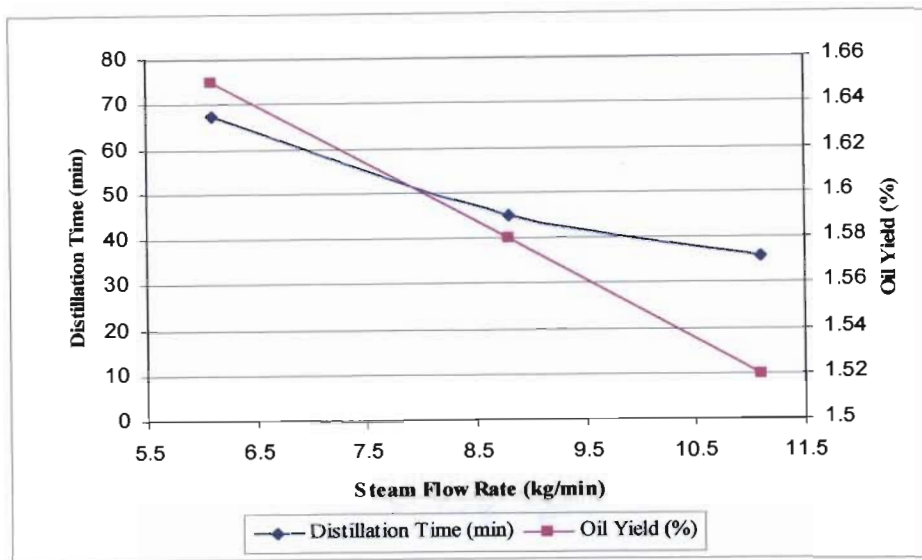


Figure 4.8 Rosemary extraction times and oil yields for the tested steam flow rate range.

The GC analysis results of the rosemary oils produced with the three steam flow rates are illustrated in Table C.2 (APPENDIX C). It can be seen that the chemical constituents of the oil produced for each steam flow rate are again very similar. The same factors as those discussed at the end of Section 4.1.2 would have contributed to the minor differences in the chemical composition of the rosemary oils.

It can thus be deduced that variations in steam flow rate again have an effect on the distillation times while the chemical composition of the oils remain unchanged. The oil yield decreased for higher steam flow rates as the distillations were again ended too early. The results obtained from the steam requirement calculation models for superficial and subcutaneous oils are discussed below.

## 4.2 Steam Requirement Calculation Model

The dimensions of the proposed distillation system were entered into both superficial and subcutaneous models. The entered values were a charge vessel height of 1.4 m, a diameter of 0.872 m and a packing density of  $300 \text{ kg/m}^3$ , giving a maximum charge mass of 250.8 kg. The distillation times for both lavender and *eucalyptus polybractea* crops were then calculated for

various steam flow rates. The results obtained for both crops are shown in Figure 4.9 and Figure 4.10 respectively.

The results are based on the crop material that was used in the test distillations conducted by Denny (lavender) and Davis (*eucalyptus polybractea*). Hence the actual distillation times for the various steam flow rates might differ slightly in practice as the crop and steam characteristics will not be exactly the same as those of the test distillations.

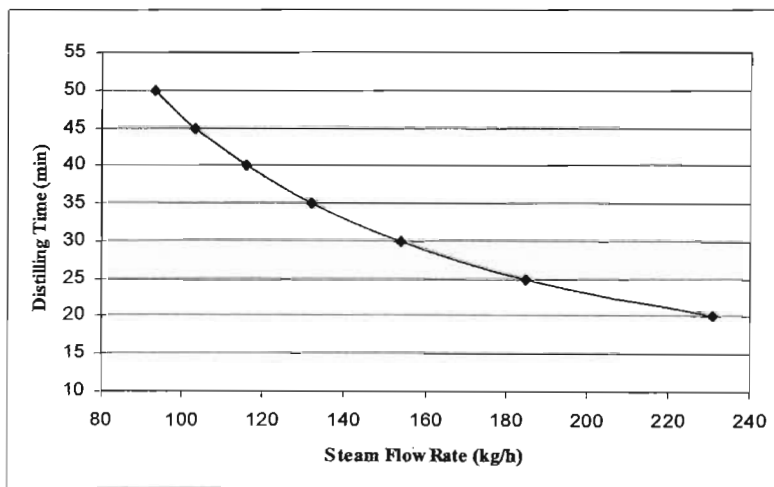


Figure 4.9 Distillation time at various steam flow rates for lavender with the proposed distillation system with an expected yield of 1%.

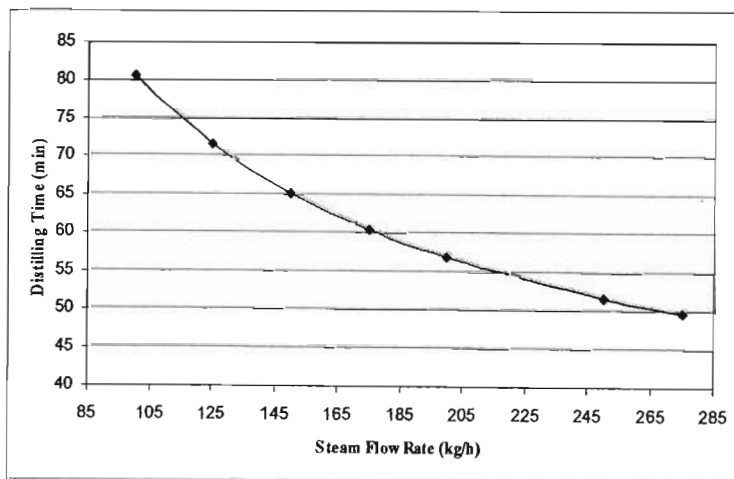


Figure 4.10 Distillation time at various steam flow rates for *eucalyptus polybractea* with the proposed distillation system with an expected yield of 1.45%.

The distillation times for superficial lavender oils are less than the subcutaneous *eucalyptus* oils for the same steam flow rate, as illustrated in Figure 4.9 and Figure 4.10. This was expected, as the oils first need to diffuse to the surface of the plant material before being exposed to steam with subcutaneous oils. The distillation times for various crops with a certain distillation system will depend on the location of the oil glands, the quantity of the oil present, the vapour pressure and hence volatility of the oil, and also the condition of the herb (Guenther, 1948).

A steam flow rate of 140 kg/h would be sufficient for distilling lavender in the proposed distillation unit, as a distillation would be completed in less than 33 minutes (Figure 4.9). A *eucalyptus polybractea* distillation would take approximately 67 minutes to reach its end with the same flow rate and hence a higher steam flow rate would be more suitable. The steam flow rate should be at least between 2 and 4 kg/min/m<sup>2</sup> of charge cross-sectional area which is equivalent to 86 and 171 kg/h for the proposed system (Denny, 1999). It is thus advisable that the selected steam generator for the proposed system should be able to produce at least 170 kg/h.

The smallest diesel fired steam generators that are commonly available are units that produce 250 kg/h of steam. Smaller units are not commonly available but can be specially manufactured on order. It was found that a smaller specially made unit was approximately the same price as a standard 250 kg/h unit. The steam flow rate of a 250 kg/h unit can be reduced if a lower steam flow rate is required, as is the case with lavender and hence a 250 kg/h steam generator that produces steam at up to 10 bar pressure was acquired.

The time taken to charge and discharge the basket has a major effect on the production of the distillation unit per day. The effects of this on the proposed systems daily production for both lavender and *eucalyptus polybractea* are shown in Figure 4.11 below. The curves were plotted using results obtained from the two crop models and a packing density of 300 kg/m<sup>3</sup>. It must be noted that steam flow rates of 152 kg/h for lavender and 250 kg/h for *eucalyptus polybractea* were entered, which yielded distillation times of 37 min and 53 min respectively.

Assuming an efficient discharging and charging system, a realistic time taken to remove the basket from the charge vessel, load and replace it into the charge vessel would be approximately

15 – 20 minutes. When two vessels are used, as is the case with the proposed distillation system, both the vessels can be loaded with charged baskets. As soon as the distillation of the one vessel is completed, the steam supply can be switched to the second vessel and hence distillation can continue while the basket from the first vessel is recharged and reloaded into the charge vessel. Assuming that the basket recharging time is less than the distillation time itself, the only time lost would be on the switch over from the one vessel to the other.

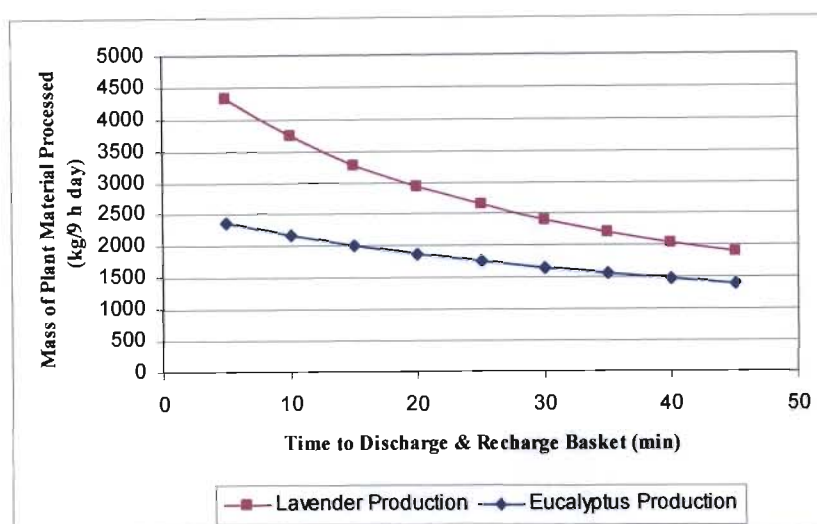


Figure 4.11 Effects of time taken to discharge and recharge baskets on the production of lavender (distillation time 37 min) and *eucalyptus polybractea* (distillation time 53 min) for a 9 hour day.

With the designed system, two three-way valves were used, one to divert the steam flow to either of the distillation vessels and the other to divert the vapours from the vessel to the condenser. The time needed to reset the two valves will be approximately 5 minutes. The increased amount of material that can be processed when this downtime is reduced from 20 minutes to 5 minutes is approximately 1300 kg for lavender and 500 kg for *eucalyptus*. There is thus a significant effect on the production of the system and the addition of a second vessel is thus a viable option.

In order to verify the results obtained from the steam requirement calculation model for superficial oils (Section 3.4.1), the parameters of an existing distillation system used for distilling lavender were entered into the steam requirement calculation model for lavender and the results

were compared. The system had a charge vessel height of 1.4 m, a diameter of 0.8 m, charge mass of approximately 120 kg (packing density of 120 kg/m<sup>3</sup>), a steam flow rate of 40 kg/h and took approximately 1 h to distil. The distillation time calculated by the model was 37 min. It must be noted that the system parameters were only approximate figures obtained from a grower.

It was not possible to obtain any further results from systems used to distil lavender or *eucalyptus* in order to further verify the superficial and subcutaneous oil crop models discussed in Sections 3.4.1 and 3.4.2. There was also no time available to experimentally determine accurate results from the existing systems. The model results were however discussed with experienced people in the distilling field and it was agreed that the results seemed accurate. It was also decided that further verification of the model would also not influence the decision on the acquisition of the steam generator since it was larger than required, and the chances of needing higher steam flow rates than it can supply would be very small. Since the required steam generator size had been determined, it was now possible to design the condenser for a maximum steam flow rate.

### 4.3 Condenser Performance

Once the entire distillation unit had been constructed and commissioned, the condenser was tested by generating a fixed steam flow rate through the system and then varying the coolant flow rate. The distillate and coolant exit temperatures were measured for each set coolant flow rate. Both the distillate and the coolant exit temperatures were then obtained from the condenser model for the measured coolant flow rates. The results were compared by constructing observed versus predicted scatter plots for both coolant and distillate exit temperatures as displayed in Figure 4.12 and Figure 4.13 below. A 1:1 line, representing the ideal relationship was inserted in both drawings.

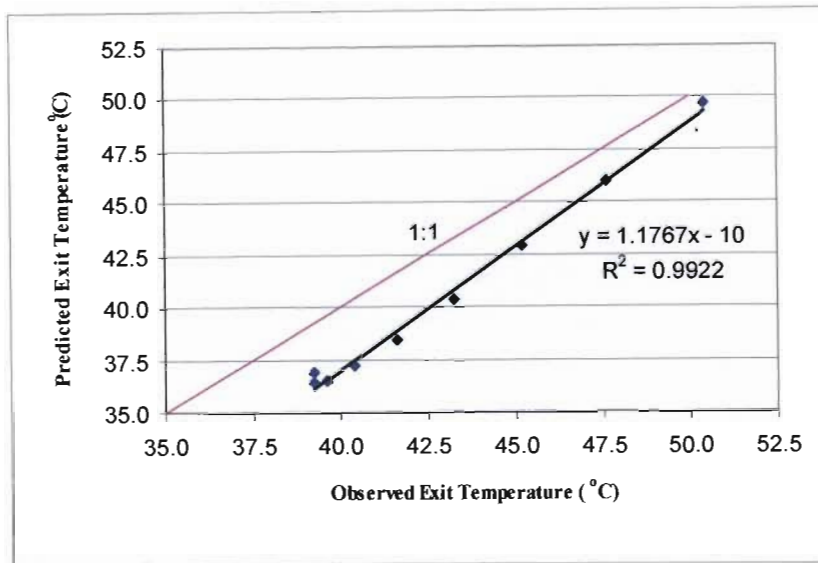


Figure 4.12 Predicted coolant exit temperatures against observed coolant exit temperatures.

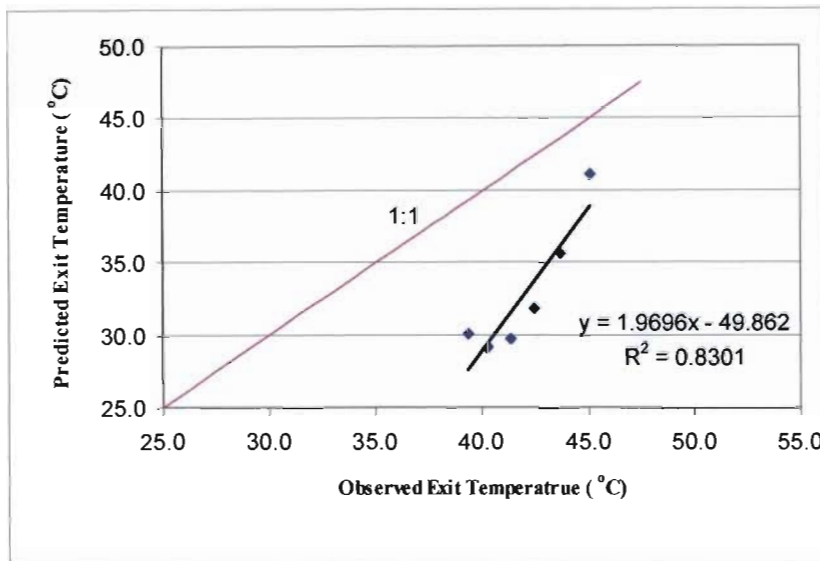


Figure 4.13 Predicted distillate exit temperatures against observed distillate exit temperatures.

The distillate exit temperature was measured for a maximum coolant flow rate and the coolant flow rate was then reduced till the required distillate temperature for separation (45 °C) was obtained. The distillate exit temperature at maximum coolant flow rate was just below 40 °C and that is why distillate exit temperature results were obtained over such a short temperature range illustrated in Figure 4.13. Although relative values for both the coolant and distillate exit

temperatures compare well, the results obtained are positioned below the ideal relationship line as shown in Figure 4.12 and Figure 4.13. It can thus be deduced that the model underestimates both coolant and distillate temperatures.

The steam flow rate through the system (162 kg/h) was at the boilers maximum capacity and at a pressure of 6 bar. This means that 88 kg of water was released through the steam trap connected to the separator per hour (250 kg/h – 162 kg/h). This implies that a great deal of water passed through the boiler without evaporating. The amount of this water can be reduced by increasing the operating pressure. These types of steam generators are however designed to always have some water passing through it in order to ensure that superheated steam is not produced.

From the test results, the required heat removal to reduce the coolant water back to its entering temperature (26 °C) for the different flow rates ranged between 103 kW and 126 kW. A cooling tower with a heat rejection capacity greater than 126 kW would be suitable as the required heat removal would be slightly higher if the operating pressure of the steam generator were increased.

#### **4.4 Field Testing**

The rose geranium was harvested on the morning when the distillations were conducted. This was the first cutting of the season for the field and hence there was a great deal of dry matter (plants killed by winter frost) present in the plant material and hence a low yield was expected. Four distillations were conducted with an average charge mass of 122 kg per distillation. The plant material was compacted in the basket while charging. The baskets were not filled to maximum capacity due to the amount of material available. The maximum charge mass was 158 kg and it is estimated that the basket could be packed to a maximum mass of 200 kg with a packing density of 205 kg/m<sup>3</sup> for rose geranium of the same moisture content. It can thus be deduced that the packing density of 300 kg/m<sup>3</sup> used in the steam requirement calculation model is slightly high. The average results obtained from the distillations are represented in Table 4.7 below.

Table 4.7 Average results for rose geranium distillations.

Average Steam Flow (kg/h)	152
Average Yield (%)	0.12
Average Reflux Flow per Distillation (l)	47.5
Average Distilling Time (min)	64

The oil yield obtained is within the expected range of 0.1 – 0.2 % (Sankarikutty and Narayanan, 1993). Average yields of 0.1 % were obtained from the crops of the same field for the previous season and hence it can be deduced that good yields were obtained even though there was a large percentage of dry material present. The average heating time was 7 min for each distillation.

The steam was generated at a pressure of 8 bar and the pressure was reduced to 0.12 bar before entering the charge vessel. The pressure in the steam chamber in the bottom of the charge vessel was 0.03 bar and the steam temperature was 99 °C. It could thus be deduced that the steam was not superheated and hence the oil quality would not be reduced by thermal decomposition. The vapour temperature at the charge vessel outlet remained at 99 °C.

The lemon grass was also harvested on the morning when the distillations were conducted. A great deal of dry matter was present in the plant material as it was again the first cutting for the season. Two distillations were conducted with an average charge mass of 127 kg per distillation. Due to the limited amount of material available on the day, the baskets were not filled to maximum capacity. The maximum charge mass was 137 kg. The average results obtained from the distillations are represented in Table 4.7 below.

Table 4.8 Average results for lemon grass distillations.

Average Steam Flow (kg/h)	158
Average Yield (%)	0.39
Average Reflux Flow per Distillation (ml)	26.1
Average Distilling Time (min)	49

The oil yield obtained (Table 4.8) was surprisingly high when considering the large amount of dry matter present and was within the upper limit of the expected oil yield range 0.2 – 0.4 % (Sankarikutty and Narayanan, 1993). The average heating time for the distillations was 7 min.

Samples of the hydrosol discharging from the separator were taken during the distillations and no sign of oil globules present in the hydrosol occurred. A deduction can thus be made that the separator is suitably sized, allowing for sufficient separating time. Even though the steam flow rates were similar, the average extraction time per distillation was longer for rose geranium than lemon grass as shown in Table 4.7 and Table 4.8. The location of the oil glands within the plant material, the volatility of the oil and the condition of the plant material (moisture content) are the factors that influence the difference in distillation times (Denny, 1999).

More reflux flow accumulated in the bottom of the charge vessel for the rose geranium distillations than the lemon grass distillations and hence a deduction can be made that the rose geranium had a lower absorptive capacity than the lemon grass. Finally it can be concluded that the mobile distillation unit is a suitable system for extracting essential oils from herbaceous materials as satisfactory results were obtained from the distillations.

## 5. SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

The essential oil industry is an attractive one for small-scale farmers in remote areas due to the high value and low volume products produced, which have a long shelf life when stored properly. The quality and the yield of the oil is largely dependant on the extraction process as well as the plant material itself and the preparation of the plant material prior to distillation. Steam distillation is the most widely accepted process for the production of volatile essential oils from herbaceous materials and is regarded as the standard process by the flavour and fragrance industry.

In order to maximise the yield and minimise the distillation time it is important that the herbaceous materials are harvested, prepared and stored correctly prior to distillation. The material should be prepared according to the location of the oil in the plant material, the moisture content and the absorptive capacity of the herbaceous material. Storage of plant material before comminution also offers some hazard to the loss of volatile oil, and should be stored in its natural condition to minimise oil losses.

The packing requirements for efficient distillation with regards to minimising fuel usage are tight and even dry packing of the herbaceous material into the still. Steam assistance to soften plant material for greater packing density has been proven to increase fuel usage and reduce the yield, even though the still is filled with more plant material.

All the components of a distillation system should be designed such that they can be easily constructed and also disassembled for cleaning purposes. Suitable materials such as stainless steel, glass and Teflon should be used for construction as they do not have any absorptive properties and are resistant to corrosive essential oils.

From the results obtained from the laboratory distillations it can be deduced that higher steam flow rates reduced the distillation times while the chemical composition of the oils remain unchanged. A reduction in oil yield occurred for higher steam flow rates, but this was due to the fact that the distillations were ended too early, and hence not all the oil had been extracted from

the plant material. An indication of the distillation time for various steam flow rates for the proposed distillation system was obtained from the steam requirement calculation models for both superficial (lavender) and subcutaneous (*eucalyptus polybractea*) crops. The model was also used to simulate the effects of downtime due to reloading on the productivity of the designed system.

The condenser design model was used to design the multi-tubular condenser. Once constructed and assembled, the performance of the condenser was tested and the results were compared to those predicted by the model. The predicted distillate and coolant exit temperatures were lower than the measured temperatures. A conclusion can thus be made that the condenser model is suitable for designing multi-tubular condensers but underestimates coolant and distillate exit temperatures for the tested coolant flow rates.

A mobile essential oil distillation system that is capable of extracting essential oils from herbaceous materials in areas where electricity is not available was developed and tested with good results. The unit consisted of a double charge vessel system mounted onto a trailer with a crane mounted on the distillation unit frame to enable efficient charging and discharging of the charge vessel with plant material. A petrol generator and a diesel fired steam generator with its ancillary components were mounted onto a second trailer. The trailers could thus be positioned alongside one another and the steam line from the steam generator could then be connected to the charge vessels. The generator is responsible for supplying electricity to the motor driving the feed water pump, the electric hoist, the cooling water pump and the cooling tower fan motor. The cooling tower through which the cooling water for the condenser is circulated, which had not yet been acquired due to financial reasons, and the separator could be loaded on one of the vehicles towing the trailers.

Field tests were conducted with rose geranium and lemon grass and yields of 0.12 % and 0.39 % were obtained respectively. The yields fitted well into the expected yield range of 0.1 % – 0.2 % for rose geranium and 0.2 % - 0.4 % for lemongrass and were higher than the yields obtained for both crops the previous season with a different system, even though large quantities of dry matter were present in both crops. There were no signs of oil globules present in the hydrosol

discharging from the separator, hence indicating that the separator has been correctly sized for the system, as complete separation was taking place. Finally a conclusion can be made that all the project objectives were met and that excellent results were obtained from the field tests that were conducted.

A minor consideration that needs to be mentioned for the future use of the distillation system is that during the test distillations it was noticed that the EPDM closed cell foam seals inserted into the water gutter were showing signs of wear. Any further wear due to distillations in the future might impair the sealing ability and it is recommended that they are replaced with a stronger rubber capable of withstanding high temperatures. A further recommendation is that the hot water separated from the steam and discharged via the steam trap should be returned to the feed water tank. Up to approximately 80 l/h of water is separated and hence would not only reduce the water usage of the system, but also heat the feed water and thus reduce the energy consumption of the steam generator if it were returned.

It is recommended that an analysis of the total operating costs of the mobile essential oil distillation unit should be conducted and also compared to a similar sized static electric unit. When both the operating cost and the production capacity of the system have been determined, it would be possible to determine what percentage of the entire essential oil production cost would account for the distillation process. As the distillation process is one of the major factors affecting the cost of essential oil production, future research on ways to reduce the distillation times and hence distillation costs would be extremely valuable to the industry. The effects of increasing the operating pressure of the charge vessel and also varying the wetness fraction of the produced steam on the distillation times and oil quality should thus be investigated for the various crop types.

It is further recommended that distillations of all the major essential oil crops grown in South Africa are conducted in the future and that the distillation times of each crop type should be experimentally determined for the set operating conditions. In this way the operator of the system could have an idea of the distillation time and hence distillations can be terminated at the right times, thus optimising the distillation process.

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## 7. APPENDICES

### APPENDIX A

APPENDIX A covers the structural design aspects of the frame and crane of the distillation unit.

#### DISTILLATION UNIT FRAME DESIGN CALCULATIONS

The base of the frame had to be sized to be able to withstand the forces subjected to it when loading the entire unit onto a trailer. The loads on the base members of the frame were determined by distributing the weights of the crane, distillation unit and the self weight of the frame as point loads on the base members. The members were then sized according to the member subjected to the worst loading case. When loading the frame, it would be lifted on the one end and then the opposite end would then either be positioned on the ground or trailer. Hence two supports were put in place on either end. The worst loading scenario is shown in Figure A.1 below.

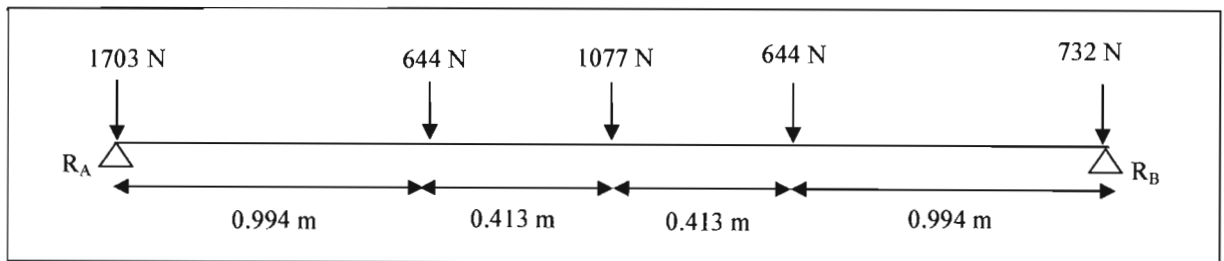


Figure A.1 Worst loading case for base member of frame.

The reaction forces were calculated as:

$$R_A = 2885.5 \text{ N}$$

$$R_B = 1914.5 \text{ N}$$

The shear force diagram and the bending moment diagram for the loading conditions illustrated above are depicted in Figure A.2 and Figure A.3 below.

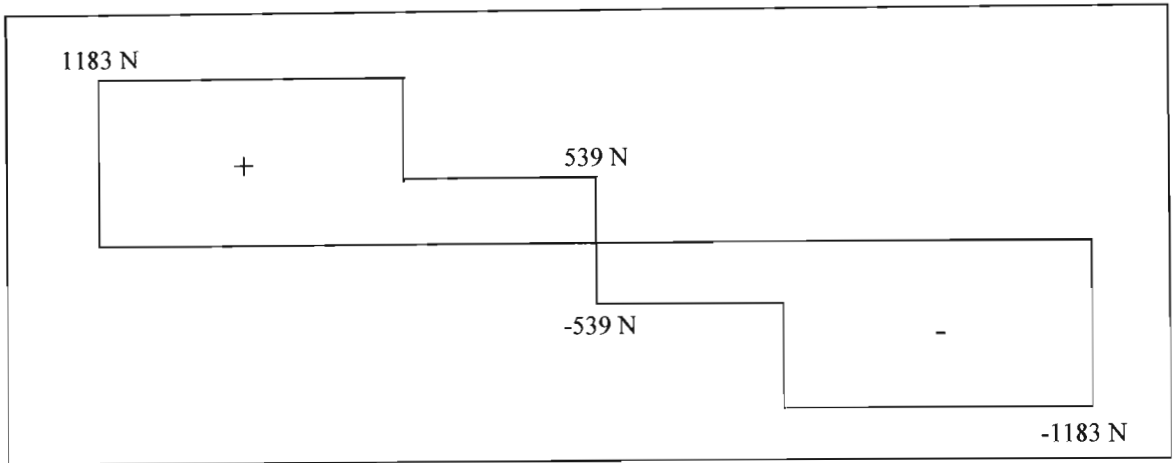


Figure A.2 Shear force diagram for applied loads.

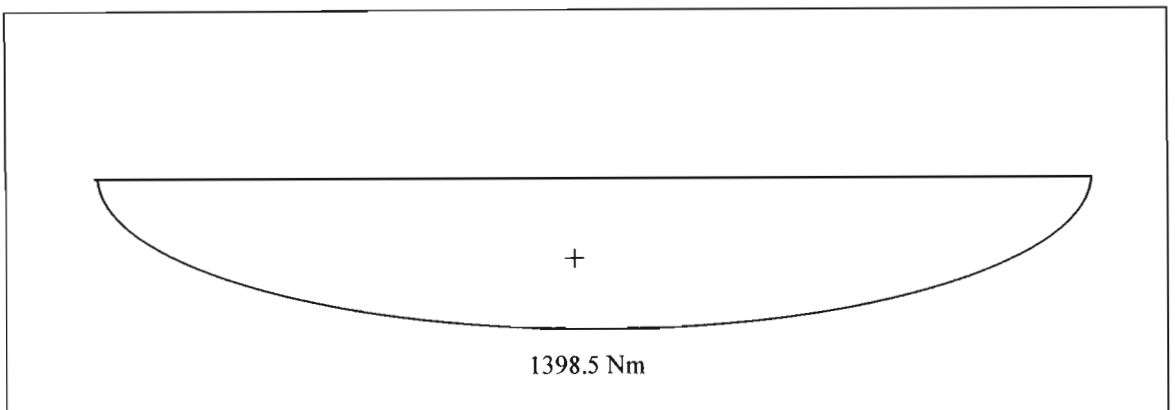


Figure A.3 Bending moment diagram derived from the shear force diagram.

The required section modulus was calculated by the following formula:

$$S = \text{Section Modulus} = (\text{Maximum Moment}) / (\text{Allowable Stress}) = M_{\max} / F_u$$

$$M_{\max} = 1398.5 \text{ Nm}$$

$$F_u = 250 \text{ MPa}$$

Hence:

$$S = 5.594 \times 10^{-6} \text{ m}^3$$

The safety factor for the design calculation of the frame was determined by taking the following into account:

- The amount of times the distillation unit will be offloaded and reloaded onto the trailer. This could occur regularly as there is not a trailer designated to the distillation unit.
- The fact that a chance exists that the frame could not be lifted on the intended lifting points when loading, could cause stresses of different magnitude within the members.
- The quality of the workmanship.
- The danger to human life if failure were to occur.

A safety factor of 1.75 was chosen and the required section modulus became:

$$S_{sf} = (M_{max}/f) * 1.75$$

$$= 9.790 \times 10^{-6} \text{ m}^3$$

The section modulus was calculated for various square hollow sections with the following formula:

$$S_{section} = (\text{Moment of Inertia} / \text{Distance from Neutral Axis to Outer Surface}) = I / c$$

For 50 × 50 × 4 mm square hollow section (values of I obtained from “South African Steel Construction Handbook”):

$$I = 0.229 \times 10^{-6} \text{ m}^4$$

$$c = 25 \times 10^{-3} \text{ m}$$

Hence:

$$S_{section} = 9.160 \times 10^{-6} \text{ m}^3$$

$$< 9.790 \times 10^{-6} \text{ m}^3 \text{ and therefore too small}$$

For 50 × 50 × 5 mm square hollow section (values of I obtained from “South African Steel Construction Handbook”):

$$I = 0.257 \times 10^{-6} \text{ m}^4$$

$$c = 25 \times 10^{-3} \text{ m}$$

Hence:

$$S_{\text{section}} = 10.280 \times 10^{-6} \text{ m}^3$$

$$> 9.790 \times 10^{-6} \text{ m}^3 \text{ and therefore suitable}$$

The  $50 \times 50 \times 5$  mm square section suitable section but it is not a standard size and hence only available on special order at a high price. As a result a larger standard ( $76 \times 76 \times 2$  mm) section was chosen, which was cheaper, lighter and has a section modulus of  $14.423 \times 10^{-6} \text{ m}^3$ .

### DISTILLATION UNIT CRANE DESIGN CALCULATIONS

The load on the crane includes the mass of the basket, the plant material when wet (after distilling) and the mass of the lifting hoist itself. In order to size the members, the maximum bending moment  $M$  (shown in Figure A.4) needed to be determined.

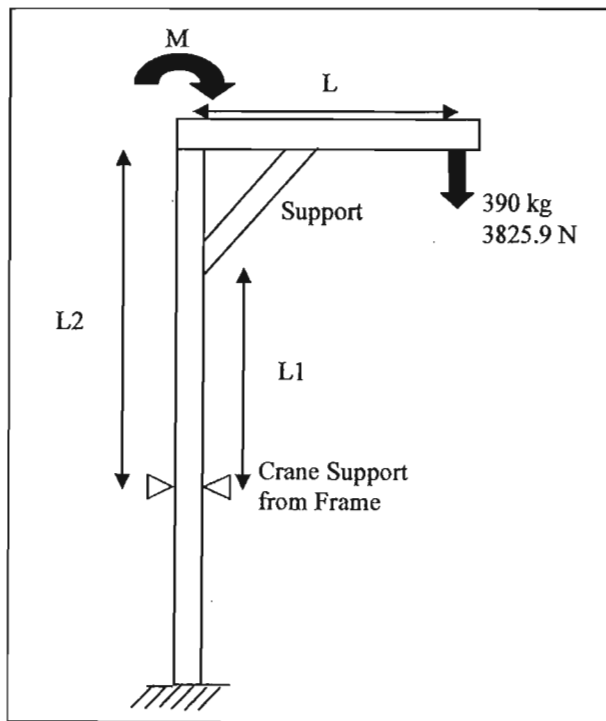


Figure A.4 Load on crane.

The maximum moment was calculated by:

$$M_{\max} = \text{Force} * \text{Lever Arm} = F.L$$

$$F = 3825 \text{ N}$$

$$L = 1.5 \text{ m}$$

Hence:

$$M_{\max} = 5738.9 \text{ Nm}$$

The required section modulus was calculated by the following formula:

$$S = \text{Section Modulus} = (\text{Maximum Moment}) / (\text{Allowable Stress}) = M_{\max} / F_u$$

$$M_{\max} = 5738.9 \text{ Nm}$$

$$F_u = 250 \text{ MPa}$$

Hence:

$$S = 22.956 \times 10^{-6} \text{ m}^3$$

The same factors as the distillation unit frame design were taken into consideration when deciding on the safety factor for the design calculations. A safety factor of 3 was selected as the crane would be used up to 12 times a day and failure of it could be fatal to operator of the system.

The required section modulus thus became:

$$\begin{aligned} S_{sf} &= (M_{\max}/f)*3 \\ &= 68.868 \times 10^{-6} \text{ m}^3 \end{aligned}$$

The section modulus was calculated for various round hollow sections with the following formula:

$$S_{\text{section}} = (\text{Moment of Inertia} / \text{Distance from Neutral Axis to Outer Surface}) = I / c$$

For 127 × 4 mm round hollow section (values of I obtained from “South African Steel Construction Handbook”):

$$I = 2.93 \times 10^{-6} \text{ m}^4$$

$$c = 63.5 \times 10^{-3} \text{ m}$$

Hence:

$$S_{\text{section}} = 46.142 \times 10^{-6} \text{ m}^3$$

$$< 68.868 \times 10^{-6} \text{ m}^3 \text{ and therefore too small}$$

For 140 × 5 mm round hollow section (values of I obtained from “South African Steel Construction Handbook”):

$$I = 4.84 \times 10^{-6} \text{ m}^4$$

$$c = 70 \times 10^{-3} \text{ m}$$

Hence:

$$S_{\text{section}} = 69.143 \times 10^{-6} \text{ m}^3$$

$$> 68.868 \times 10^{-6} \text{ m}^3 \text{ and therefore suitable}$$

Hence the 140 × 5 mm round hollow section was selected. The next step was to determine whether the deflection of the vertical member of the crane under load is acceptable. The deflection was calculated by:

$$\delta = (\text{Maximum Moment} \times \text{Length}^2) / (2 \times \text{Young's Modulus} \times \text{Moment of Inertia})$$

$$= (M_{\text{max}} \times L^2) / (2 \times E \times I)$$

For the chosen section (140 × 5 mm) the deflection was calculated as follows:

$$M_{\text{max}} = 5738.9 \text{ Nm}$$

$$L = 2.99 \text{ m (from top of crane to crane support on frame as shown in Figure A.4)}$$

$$E = 207 \times 10^9 \text{ Pa}$$

$$I = 4.84 \times 10^{-6} \text{ m}^4$$

Hence:

$$\begin{aligned}\delta &= 0.0256 \text{ m} \\ &= 25.6 \text{ mm}\end{aligned}$$

The calculated deflection is acceptable, particularly when considering that a safety factor of 3 has been used. A support (50 × 50 × 2 mm square hollow section) was positioned between the horizontal and vertical member of the crane as depicted in Figure A.4, in order to reduce the stresses on the weld at the vertical/horizontal joint. The support was positioned at approximately 30 ° from the vertical. This further reduced the effective length for bending to L1 (2.24 m) and hence the calculated deflection decreased to:

$$\begin{aligned}\delta &= 0.0144 \text{ m} \\ &= 14.4 \text{ mm}\end{aligned}$$

### CRANE BUSH DESIGN CALCULATIONS

The chosen bush material was Nylatron GSM (reasons discussed in Section 3.3.2). The material needed to be checked whether it can withstand the loading it will be exposed to. The worst loading condition will be the load used in the crane design calculations, plus the weight of the crane itself. The surface area (A) of the material required to carry the designed load was calculated by:

$$A_{\text{req}} = \text{Load} / \text{Design Loading Limit of Material} = L / DL$$

$$L = 5161.1 \text{ N}$$

$$DL = 20 \times 10^6 \text{ Pa (Material properties sheet from suppliers)}$$

Hence:

$$\begin{aligned}A_{\text{req}} &= 2.581 \times 10^{-4} \text{ m}^2 \\ &= 258 \text{ mm}^2\end{aligned}$$

The contact area where the load is transferred onto the bush is 2121 mm<sup>2</sup>, hence the bush will be strong enough. The next concern was the force that will be required to turn the crane manually when under load. This will be the force required to overcome friction ( $F_{\text{fric}}$ ) and is calculated by:

$$F_{\text{fric}} = \text{Material Coefficient of Friction} * \text{Load} = \mu.L$$

$$\mu = 0.2 \text{ (Material properties sheet from suppliers)}$$

$$L = 5161.1 \text{ N}$$

Hence:

$$F_{\text{fric}} = 1033.2 \text{ N}$$

$$= 105.2 \text{ kg}$$

Handles (300 mm long) were fitted to the crane thus reducing the force required to rotate the crane due to the lever arm. The force that needs to be applied to the handle could be determined from the torque required to rotate the crane, which was calculated by:

$$T = \text{Frictional Force} * \text{Lever Arm} = F_{\text{fric}} \cdot S$$

$$S = 0.07 \text{ m (Distance from centre to outside of } 140 \times 5 \text{ mm crane member)}$$

Hence:

$$T = 72.3 \text{ Nm}$$

The force that needs to be applied to the handles was calculated by using the torque equation above and making  $F$  the subject of the equation.

$$S = 0.3 \text{ m (Handle length)}$$

$$T = 72.3 \text{ Nm}$$

Hence:

$$F_{\text{handles}} = 241 \text{ N}$$

$$= 24.5 \text{ kg}$$

A force of approximately 12 kg needs to be applied on each handle in opposite directions.

APPENDIX B

	A	B	C	D	E	F	G	H
1	<b>Superficial Oil Under ATM Pressure (Lavender clone MS)</b>							
2	<i>Note: The shaded values are the variables that need to be entered</i>							
3								
4	<b>Mass and Capacity of Cartridge</b>							
5	Charge Height (m)	<i>H</i>	1.400					
6	Charge Diameter (m)	<i>D</i>	0.872					
7	H/D ratio	<i>HD</i>	1.606					
8	X-sectional Area (m <sup>2</sup> )	<i>A</i>	0.597					
9	Mass of 2mm Cartridge Wall (kg)		61.4					
10	Mass of Outer and Inner Rim (kg)		7.0					
11	Mass of Grid (kg)		18.0					
12	Mass of Basket (kg)	<i>m<sub>b</sub></i>	86.4					
13	Mass of Outer Tank (kg)	<i>m<sub>t</sub></i>	113.0					<i>(only parts with steam contact)</i>
14								
15	<b>Mass of Charge</b>							
16	Packing Density (kg/m <sup>3</sup> )	<i>ρ</i>	300					
17	Specific Heat of Plant Charge (kJ/kg.K)	<i>C<sub>pp</sub></i>	3.347					
18	Specific Heat of S/steel (kJ/kg.K)	<i>C<sub>ps</sub></i>	0.885					
19	Latent heat of Steam (kJ/kg)	<i>Q<sub>LS</sub></i>	2256					<i>(Steam table)</i>
20	Rise in Plant Charge Temperature (K)	<i>ΔT<sub>p</sub></i>	80					
21	Rise in S/steel Temperature (K)	<i>ΔT<sub>s</sub></i>	60					
22								
23	Cartridge Volume (m <sup>3</sup> )	<i>V</i>	0.836					
24	Charge Mass at Max Load (kg)	<i>m<sub>c</sub></i>	250.8					<i>m<sub>c</sub> = ρ.V</i>
25	Steam Required to Raise Plant Charge to 100 °C (kg)	<i>m<sub>sc</sub></i>	29.8					<i>m<sub>sc</sub> = (m<sub>c</sub>.C<sub>pp</sub>.ΔT<sub>p</sub>)/Q<sub>LS</sub></i>
26	Steam Required to Raise Basket to 80 °C (kg)	<i>m<sub>sb</sub></i>	2.0					<i>m<sub>sb</sub> = (m<sub>b</sub>.C<sub>ps</sub>.ΔT<sub>s</sub>)/Q<sub>LS</sub></i>
27	Mass of Basket (kg)	<i>m<sub>b</sub></i>	86.4					
28	Total Mass on Hoist (kg)	<i>m<sub>tot</sub></i>	369.0					<i>m<sub>tot</sub> = m<sub>c</sub> + m<sub>sc</sub> + m<sub>sb</sub> + m<sub>b</sub></i>
29	Steam Required to Raise Outer Tank to 80 °C (kg)	<i>m<sub>st</sub></i>	2.7					<i>m<sub>st</sub> = (m<sub>t</sub>.C<sub>ps</sub>.ΔT<sub>s</sub>)/Q<sub>LS</sub></i>
30								
31	<b>Data from Test Distillations (Denny Experiment)</b>							
32	Basic time (min)	<i>t</i>	9.719					
33	Steam Rate of Displacement (kg/min/m <sup>2</sup> )	<i>m'<sub>test</sub></i>	3.090					
34	Terminal Oil Flow (ml/min/m <sup>2</sup> )	<i>q<sub>oil</sub></i>	9.750					
35	Increment Parameter (cm)	<i>s</i>	40.946					
36	Oil Yield (ml/kg)	<i>Y<sub>test</sub></i>	9.090					
37								
38	<b>Adapting Parameters to Proposed System</b>							
39	Steam Flow Rate for New Still (kg/min)	<i>m'<sub>calc</sub></i>	1.845					<i>m'<sub>calc</sub> = m'<sub>test</sub>.A</i>
40	Steam Flow Rate for New Still (kg/h)	<i>m'<sub>calc</sub></i>	110.7					
41	Expected Oil Yield (ml/kg)	<i>Y<sub>exp</sub></i>	10					
42	Actual Height of Still (cm)	<i>H</i>	140					
43	New Increment Parameter (cm)	<i>s<sub>new</sub></i>	37.2					<i>s<sub>new</sub> = s.(Y<sub>test</sub>/Y<sub>exp</sub>)</i>
44	Actual Available Steam Flow Rate (kg/h)	<i>m'avbl</i>	154					
45	Actual Available Steam Flow Rate (kg/min)	<i>m'avbl</i>	2.567					
46								
47	<b>Determine Proposed System Production</b>							
48	Extraction time (min)	<i>T</i>	23					<i>T = t.(1+(H/s<sub>new</sub>))<sup>1/2</sup></i>
49	Heating time (min)	<i>T<sub>h</sub></i>	19					<i>T<sub>h</sub> = (m<sub>sc</sub> + m<sub>sb</sub> + m<sub>st</sub>)/m'<sub>calc</sub></i>
50	Total Time (min)	<i>T<sub>tot</sub></i>	42					<i>T<sub>tot</sub> = T + T<sub>h</sub></i>
51	Time that Charge must be Processed (min)	<i>T<sub>dist</sub></i>	30					<i>T<sub>dist</sub> = T<sub>tot</sub>.(m'<sub>calc</sub>/m'avbl)</i>
52	Discharging and Recharging Time (min)	<i>T<sub>d&amp;r</sub></i>	15					
53	Working Hours per Day (h)	<i>T<sub>day</sub></i>	9					
54	System Production per Day (kg of plant material)	<i>m<sub>prod</sub></i>	3006					<i>M<sub>prod</sub> = m<sub>c</sub>.T<sub>day</sub>/((T<sub>dist</sub> + T<sub>d&amp;r</sub>)/60)</i>
55	Number of Distillations	<i>N<sub>dist</sub></i>	12					<i>N<sub>dist</sub> = T<sub>day</sub>/((T<sub>dist</sub> + T<sub>d&amp;r</sub>)/60)</i>

Figure B.1 Superficial crop model.

	A	B	C	D	E	F	G
1	<b>Subcutaneous Oil Under ATM Pressure (Eucalyptus)</b>						
2	<i>Note: The shaded values are the variables that need to be entered</i>						
3							
4	<b>Mass and Capacity of Cartridge</b>						
5	Charge Height (m)	$H_c$	1.4				
6	Charge Diameter (m)	$D$	0.872				
7	H/D ratio	$H/D$	1.606				
8	X-sectional area (m <sup>2</sup> )	$A$	0.597				
9	Mass of 2mm cartridge wall (kg)		61				
10	Mass of Outer and Inner Rim (kg)		7				
11	Mass of Grid (kg)		18				
12	Mass of Basket (kg)	$m_b$	86				
13	Mass of Outer Tank (kg)	$m_t$	113		<i>(only parts with steam contact)</i>		
14							
15	<b>Mass of Charge</b>						
16	Packing Density (kg/m <sup>3</sup> )	$\rho$	300				
17	Charge Volume (m <sup>3</sup> )	$V$	0.836				
18	Charge Mass (kg)	$m_c$	251		$m_c = \rho \cdot V$		
19							
20	<b>Data from Test Distillations (Davis Experiment)</b>						
21	Oil Yield (ml/kg)	$Y_{trasc}$	15.090				
22	Oil Yield (ml/m <sup>2</sup> .cm)	$Y_{vtrasc}$	37.300				
23	Steam Rate of Displacement (kg/min/m <sup>2</sup> )	$m'_{trasc}$	1.361				
24	Basic Time (min)	$t$	18.270				
25	Charge Height Increment Factor (min/cm)	$st$	0.411				
26							
27	<b>Adapting Parameters to Proposed System</b>						
28	Adapted Oil Yield (ml/cm)	$Y_{calc}$	22.3		$Y_{calc} = Y_{vtrasc} \cdot A$		
29	Actual Still Height (cm)	$H_s$	140				
30	Expected Oil Yield (ml/kg)	$Y_{exp}$	14.5				
31	Estimated Virtual Exhaustion (ml)	$EVE$	3637		$EVE = m_c \cdot Y_{exp}$		
32	95% Estimated Virtual Exhaustion (ml)	$95\%EVE$	3455		$95\%EVE = EVE \cdot 0.95$		
33	Virtual Height (cm)	$H_{virt}$	155		$H_{virt} = 95\%EVE / Y_{calc}$		
34	X-sectional Area (m <sup>2</sup> )	$A$	0.597		$A = V/H_c$		
35	Adapted Steam Flow Rate for New Still (kg/min)	$m'_{calc}$	0.813		$m'_{calc} = m'_{trasc} \cdot A$		
36	Actual Available Steam Flow Rate (kg/h)	$m'_{avbl}$	250				
37	Actual Available Steam Flow Rate (kg/min)	$m'_{avbl}$	4.167				
38	Factor of Change in Flow Rate	$X$	5.126		$X = m'_{avbl} / m'_{calc}$		
39	$X^{2.0}$	$X^{2.0}$	2.973				
40	Lag Factor	$R$	0.633		<b>Figure 3.8</b>		
41							
42	<b>Determine Proposed System Production</b>						
43	Extraction Time (min)	$T$	82		$T = t + (H_{virt} \cdot dt)$		
44	Extraction Time Adjusted (min)	$T_{adj}$	44		$T_{adj} = T / (R \cdot X^{2.0})$		
45	Specific Heat of S/steel (kJ/kg.K)	$C_{ps}$	0.885				
46	Specific Heat of Plant Charge (kJ/kg.K)	$C_{pp}$	3.347				
47	Latent Heat of Steam (kJ/kg)	$Q_{LS}$	2256				
48	Rise in Plant Charge Temp (K)	$D_{tp}$	80				
49	Rise in S/steel Temp (K)	$D_{ts}$	60				
50	Steam Required to Raise Plant Charge to 100 °C (kg)	$m_{sc}$	30		$m_{sc} = (m_c \cdot C_{pp} \cdot DT_p) / Q_{LS}$		
51	Steam Required to Raise Basket to 80 °C (kg)	$m_{sb}$	2		$m_{sb} = (m_b \cdot C_{ps} \cdot DT_s) / Q_{LS}$		
52	Steam Required to Raise Outer Tank to 80 °C (kg)	$m_{st}$	3		$m_{st} = (m_t \cdot C_{ps} \cdot DT_s) / Q_{LS}$		
53	Total Steam Mass (kg)	$m_{stot}$	34				
54	Heating Time (min)	$T_h$	8		$T_h = m_{stot} / m'_{avbl}$		
55	Total time for Distillation (min)	$T_{tot}$	52		$T_{tot} = T_{adj} + T_h$		
56	Discharging and Recharging Time (min)	$T_{d&r}$	5				
57	Working Hours per Day (h)	$T_{day}$	9				
58	System Production per Day (kg of material)	$M_{prod}$	2381		$M_{prod} = m_c \cdot T_{day} / ((T_{dist} + T_{d&r}) / 60)$		
59	Number of Distillations	$N_{dist}$	9.5		$N_{dist} = T_{day} / ((T_{dist} + T_{d&r}) / 60)$		

Figure B.2 Subcutaneous crop model.

	A	B	C	D	E	F	G
1	<b>Condenser Design Model</b>						
2	<i>Note: The shaded values are the variables that need to be entered</i>						
3							
4	<b>Film Coefficient Calculation</b>						
5	Number of tubes	<i>N</i>	42				
6	Condenser shell diameter (m)	<i>d<sub>s</sub></i>	0.356				
7	Numer of tubes across widest diameter	<i>n</i>	7				
8	Distance of coolant baffles apart (m)	<i>L</i>	0.4				
9	Tube outside diameter (m)	<i>d''</i>	0.01905				
10	Tube inside diameter (m)	<i>d'</i>	0.01665				
11	Tube wall thickness (m)	<i>l</i>	0.00120				
12	Total external area of tubes per m of length (m <sup>2</sup> /m length)	<i>a</i>	2.514				
13	Distillate entering temperature (K)	<i>td<sub>enter</sub></i>	373				
14	Distillate exiting temperature (K)	<i>td<sub>exit</sub></i>	318				
15	Coolant entering temperature (K)	<i>tc<sub>enter</sub></i>	301				
16	Distillate flow rate(kg/h)	<i>m'</i>	150				
17	Coolant flow rate (kg/h)	<i>M'</i>	3360				
18							
19	<b>Cooling Distillate</b>						
20	Heat removed cooling distillate (kJ/hr)	<i>q'<sub>cool</sub></i>	34541			$q'_{cool} = m'.C_p \cdot \Delta t$	
21	Increase in coolant temperature after cooling (K)	$\Delta t_{cool}$	2.5			$\Delta t_{cool} = q'_{cool} / M'.C_p$	
22	Coolant temperature after cooling (K)	<i>tc<sub>cool</sub></i>	303.5				
23	At end of cooling (K)	$\delta t'$	70			$\delta t' = tc_{enter} + \Delta t_{cool}$	
24	At start of cooling (K)	$\delta t''$	17			$\delta t'' = td_{exit} - tc_{enter}$	
25	Logarithmic change in temperature after cooling (K)	$\Delta T_{cool}$	37.3			$\Delta t_{cool} = (\delta t' - \delta t'') / \ln(\delta t' / \delta t'')$	
26							
27	<b>Condensing</b>						
28	Latent heat of vapours/steam (kJ/kg)	<i>Q<sub>LS</sub></i>	2255.59				
29	Heat removed condensing (kJ/h)	<i>q'<sub>cond</sub></i>	338339			$q'_{cond} = m'.Q_{LS}$	
30	Increase in coolant temperature after condensing (K)	$\Delta t_{cond}$	24.1			$\Delta t_{cond} = q'_{cond} / M'.C_p$	
31	Coolant exit temp after condensing (K)	<i>tc<sub>cond</sub></i>	327.5				
32	At end of condensing (K)	$\delta t'$	69.5			$\delta t' = td_{enter} + \Delta t_{cool}$	
33	At start of condensing (K)	$\delta t''$	45.5			$\delta t'' = td_{enter} - tc_{cond}$	
34	Logarithmic change in temperature after condensing (K)	$\Delta T_{cond}$	56.7			$\Delta t_{cool} = (\delta t' - \delta t'') / \ln(\delta t' / \delta t'')$	
35							
36	<b>Sizing Condenser</b>						
37	Arithmetic avg of coolant body temp condensing (K)	<i>t<sub>avg</sub></i>	315			$t_{avg} = (t_{cool} + t_{cond}) / 2$	
38	Coolant film temp (K)	<i>t<sub>film</sub></i>	344			$t_{film} = (td_{enter} + t_{avg}) / 2$	
39	Base Factor (Figure 3.21)	<i>BF</i>	12047				
40	Coolant velocity between tubes (m/s)	<i>V</i>	0.034			$V = 3.28M' / (ds - d'')L$	
41	Correction factor (Figure 3.22)	<i>CF</i>	0.35				
42	Corrected base factor (kJ/m <sup>2</sup> .h.K)		4202				
43	Film Coefficient for outside of tube (kJ/m <sup>2</sup> .h.K)	<i>h''</i>	4202			$h'' = BF \cdot CF$	
44	Film Coefficient for inside of tube (kJ/m <sup>2</sup> .h.K)	<i>h'</i>	61325				
45	Thermal conductivity of tube wall (kJ/m <sup>2</sup> .h.K)	<i>K</i>	58.6				
46	1 / U <sub>cond</sub>	1 / U <sub>cond</sub>	0.00028			$1/U_{cond} = (d''/h'd') + (L/K) + (1/h'')$	
47							
48	O'all coefficient for condensing (kJ/m <sup>2</sup> .h.K)	<i>U<sub>cond</sub></i>	3608				
49	Tube area required for condensing (m <sup>2</sup> )	<i>A<sub>cond</sub></i>	1.7			$A_{cond} = q'_{cond} / U_{cond} \cdot \Delta T_{cond}$	
50	O'all coeff for cooling (90 hori & 105 vert) (kJ/m <sup>2</sup> .h.K)	<i>U<sub>cool</sub></i>	2146.4				
51	Tube area required for cooling (m <sup>2</sup> )	<i>A<sub>cool</sub></i>	0.4			$A_{cool} = q'_{cool} / U_{cool} \cdot \Delta t_{cool}$	
52	Total area required (m <sup>2</sup> )	<i>A</i>	2.1			$A = A_{cond} + A_{cool}$	
53	Length of condenser (m)	<i>L</i>	0.830			$L = A/a$	

Figure B.3 Condenser design model.

## APPENDIX C

### G.C ANALYSES RESULTS

Table 7.1 GC analysis of lemon grass oil produced with the three tested steam flow rates.

Constituents	Oil %		
	6.2 kg/h	8.9 kg/h	13.3 kg/h
Alpha-pinene	nd	nd	nd
Myrcene	12.75	10.69	10.95
1,8 Cineole	Nd	nd	nd
Limonene	Nd	nd	nd
Ocimene	0.24	0.21	0.21
Linalol	0.74	0.65	0.64
Linalyl acetate	nd.	nd	nd
Neral	32.22	32.69	30.22
Alpha-trepeniol	Nd	nd	nd
Geranial	44.76	46.93	45.20
Geranelyl acetate	0.21	0.23	0.17
Citronelol	0.25	0.28	0.21
Citral *	75.77	74.88	75.42

**\*Citral content is a sum of Neral + Geranial**  
 nd. Not detected

Comments: Your lemon grass is of the citrus type.

Table 7.2 GC analysis of rosemary oil produced with the three tested steam flow rates.



KwaZulu-Natal  
Department of Agriculture & Environmental Affairs

**CERTIFICATE OF ANALYSIS**

Number : EO/R 280404-60/61/62

Sample Name : **Rosemary oil**

Date of analysis : 03/06/04

Constituents	Rosemary oil (%)		
	6.1 kg/h	8.8 kg/h	11.1 kg/h
Alpha-Pinene 155	14.91	15.59	15.77
Camphene 160.5	5.24	5.37	5.50
Beta-pinene 158.3	1.35	0.42	1.41
Beta-myrcene 171.5	1.91	1.50	2.00
Limonene	4.12	4.47	4.28
Cineole 176.4	7.54	7.63	7.37
p-myrcene	0.88	1.86	0.92
Thujone 201.0	0.11	Nd	0.13
Camphor 209.2	15.45	17.14	15.10
Linalol 198.3	0.28	0.20	0.29
Bornyl acetate	9.34	7.13	9.14
Beta-caryophelene 260.5	0.97	1.11	0.92
Terpinene-4-ol	0.03	0.04	0.04
Alpha-terpeniol	18.49	17.10	17.88
Borneol 212	1.39	1.63	1.38
Verbenone	0.03	nd	nd

nd not detected

**N.B. Results may not be used for litigation**

## **APPENDIX D**

### **TECHNICAL DRAWINGS OF THE DESIGNED COMPONENTS**

Figure D.1 Distillation charge vessel with attachments.

Figure D.2 Distillation unit frame.

Figure D.3 Crane.

Figure D.4 Crane base bush and shaft.

Figure D.5 Crane support

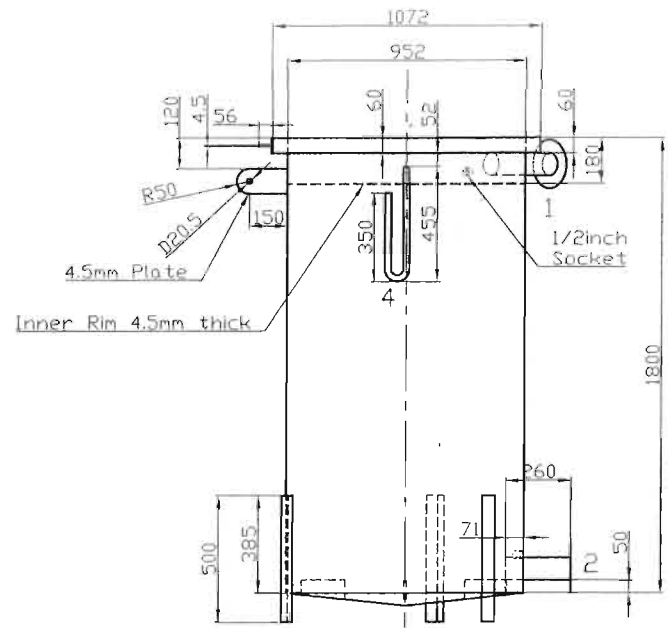
Figure D.6 Condenser.

Figure D.7 Separator.

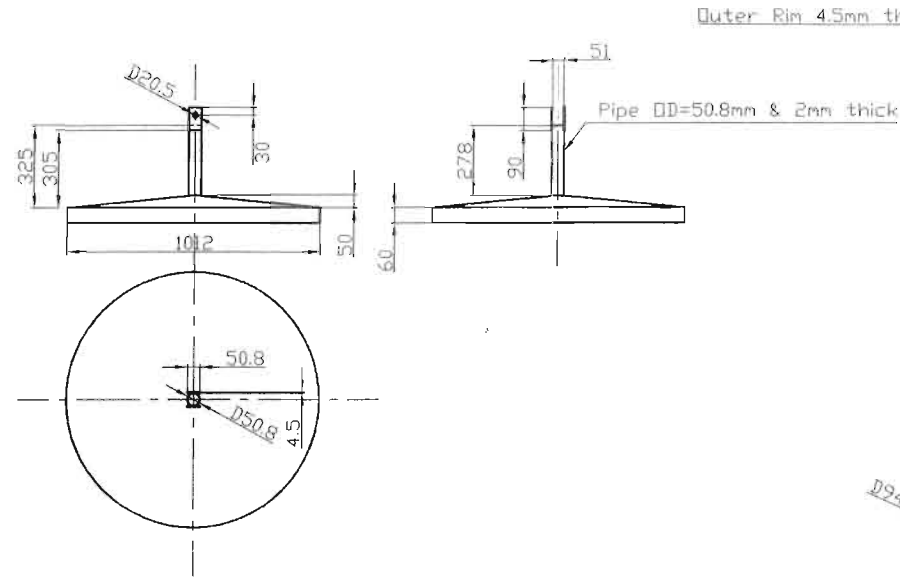
# COMPLETE DISTILLATION CHARGE VESSEL

NOTE:  
Material is Grade 304 Stainless Steel  
Material is 2mm thick unless specified

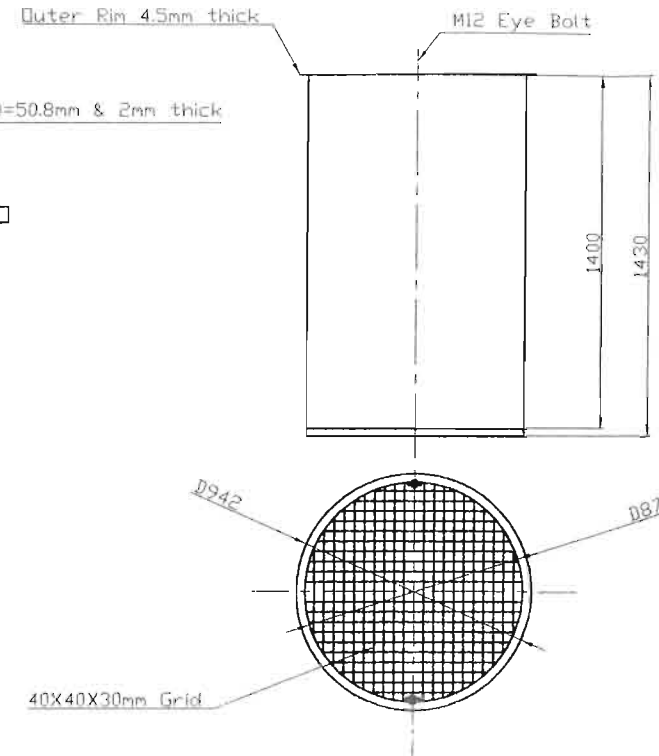
Charge Vessel



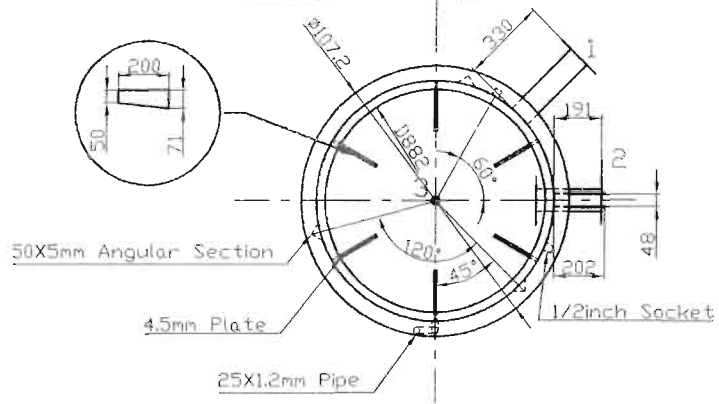
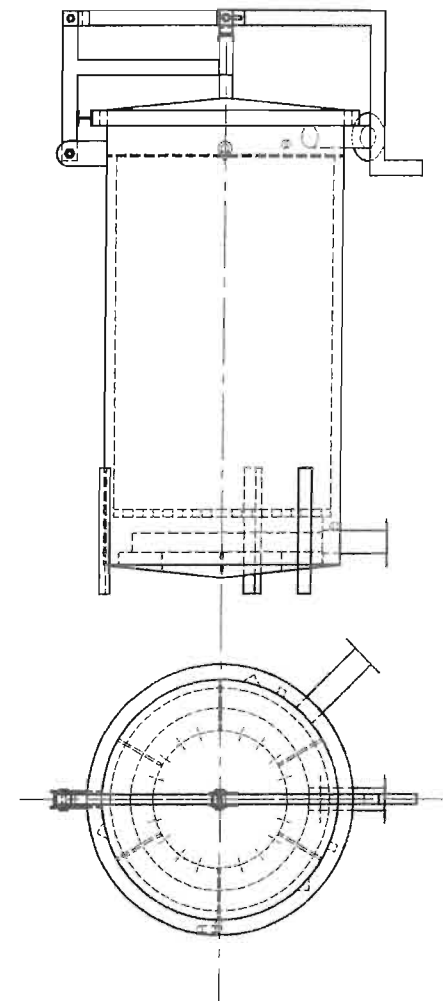
Charge Vessel Lid



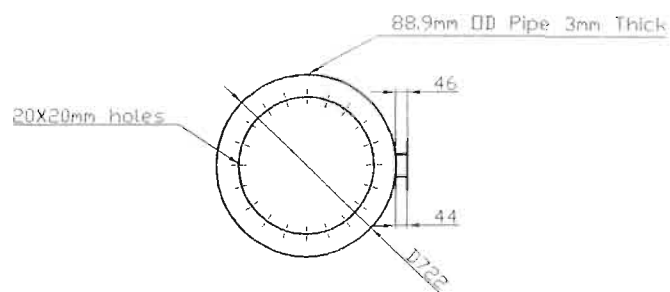
Charge Basket



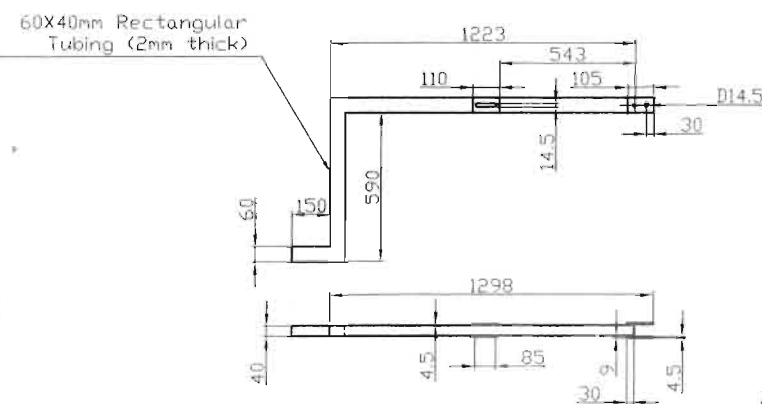
Assembly Drawing



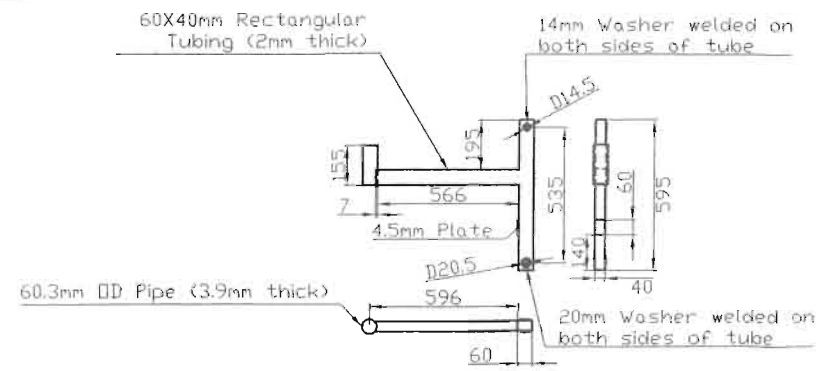
Steam Inlet Manifold



Vessel Lid Handle



Lid Handle Support

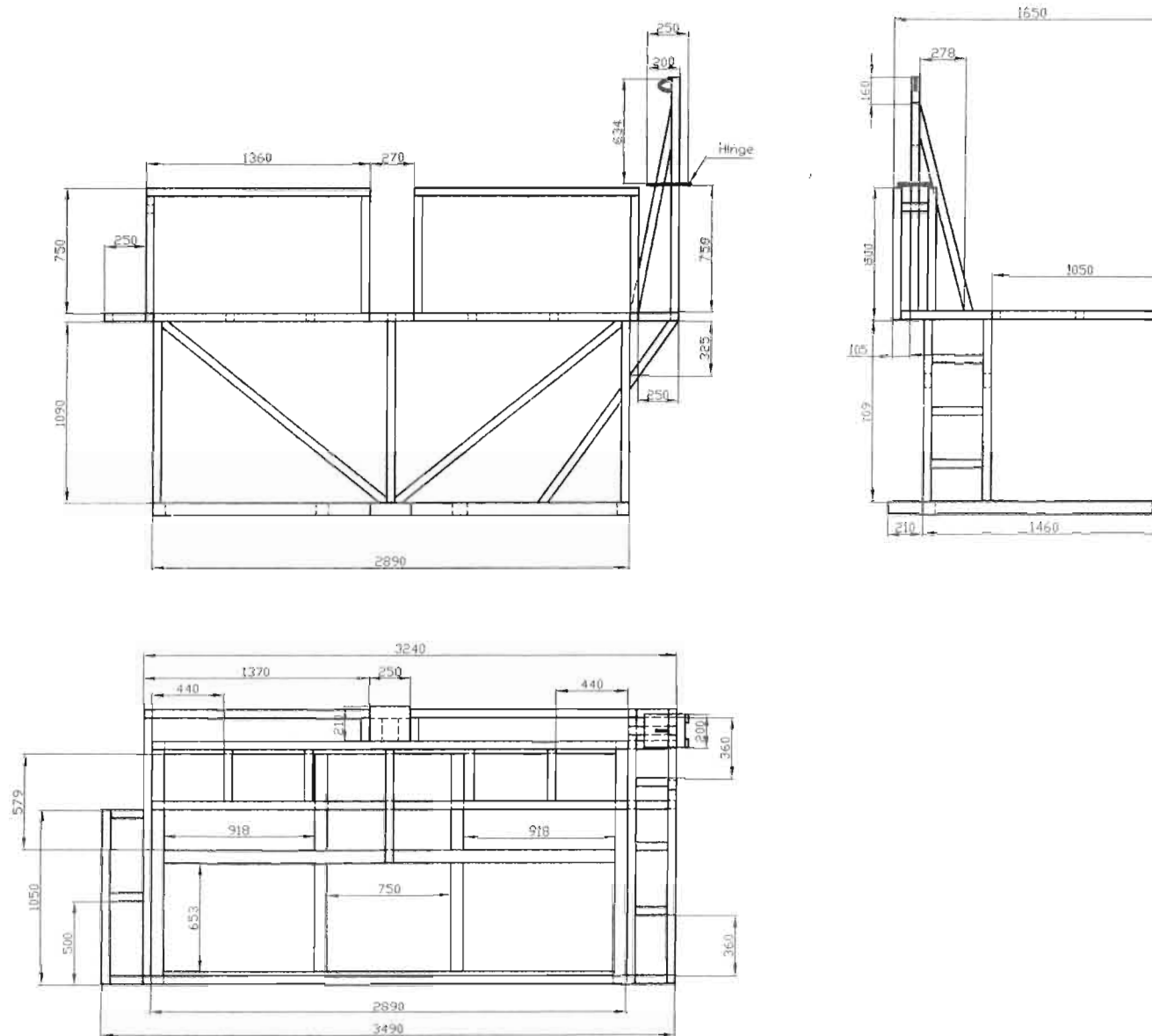


1. Vapour Outlet (88.9x3mm Pipe)
2. Steam Inlet (88.9x3mm Pipe)
3. Drain Hole
4. Safety U-tube (25x1.2mm Pipe)

INSTITUTE FOR AGRICULTURAL ENGINEERING AGRICULTURAL RESEARCH COUNCIL		INSTITUUT VIR LANDBOU-INGENIEURSWESSE LANDBOUWONDERSONDING	
DESIGNER : C. Tolomide	DRAWING NO. : 202		SCALE : 1:30
DATE : 20-12-2004	PROJECT NO. : 1512		DATE : 20-12-2004
DISTILLATION CHARGE VESSEL WITH ATTACHMENTS		D.1	

# DISTILLATION UNIT FRAME

Note:  
 Frame Base Material: 76X76X2mm square tubing  
 Remainder of Frame: 50X50X2mm square tubing



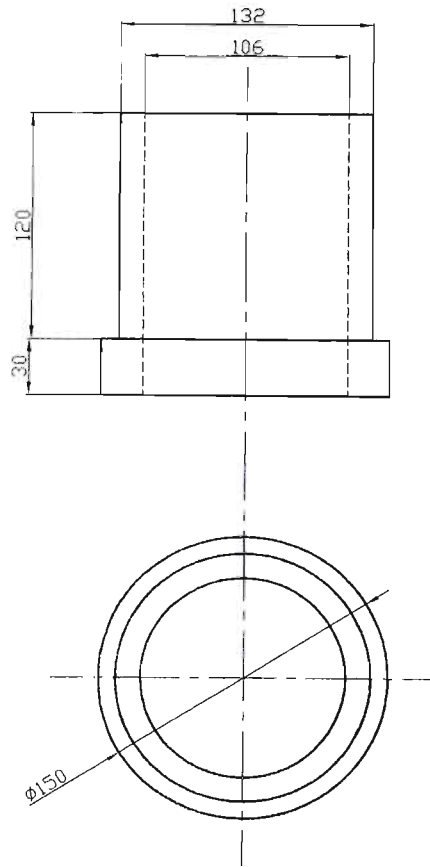
INSTITUTE FOR AGRICULTURAL ENGINEERING AGRICULTURAL RESEARCH COUNCIL		INSTITUUT VIR LANDBOU-INGENIEURSWETENSCAP LANDBOUWINGENIEURSWETENSCAP	
DESIGNED BY: C. T. F. M. S. J.	DRAWN BY: C. T. F. M. S. J.		SCALE: 1:10
PROJECT LEADER: C. T. F. M. S. J.			DATE: 20-12-2004
CHECKED BY: C. T. F. M. S. J.			TECH. NO.: 10112
APPROVED BY: C. T. F. M. S. J.			D.2



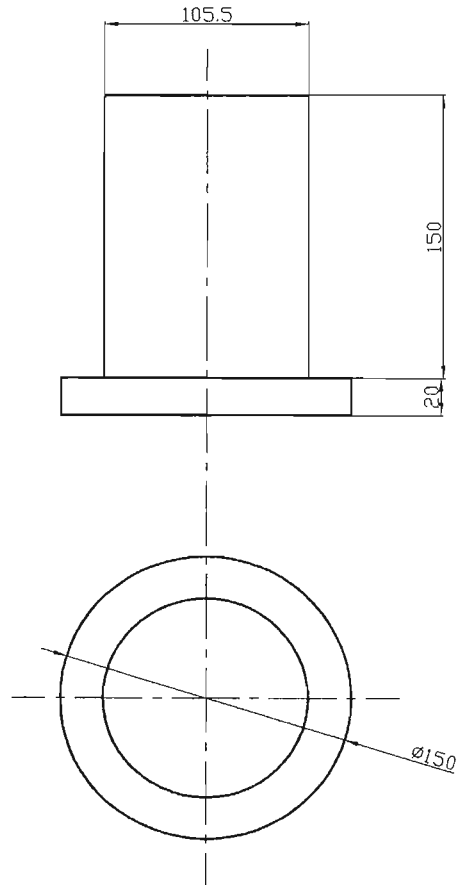
# CRANE BUSH AND SHAFT


Note:  
 Bush Material is Nylatron GSM  
 Shaft Material is Bright Mild Steel

Bush



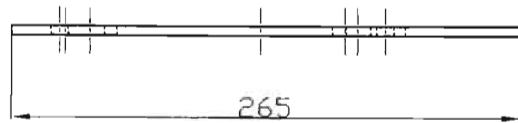
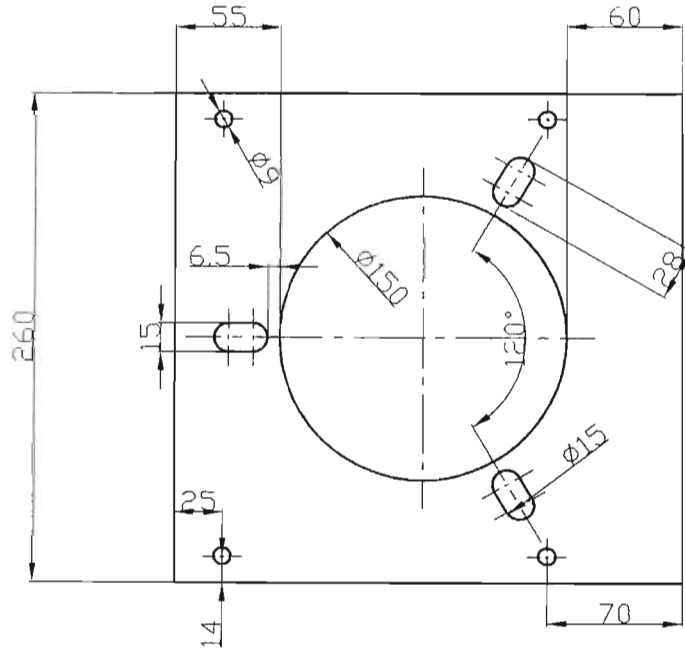
Shaft




INSTITUTE FOR AGRICULTURAL ENGINEERING AGRICULTURAL RESEARCH COUNCIL			INSTITUUT VIR LANDBOU-INGENIEURSWESE LANDBOONAVORSINGSRAAD	
GETEKEN : C.Talanda DRAWN : C.Talanda		<h2>CRANE BUSH AND SHAFT</h2>		SKAAL SCALE : 1:4
ONTWERP : C.Talanda DESIGN : C.Talanda				DATUM DATE : 20-12-2004
PROJ. LEIER PROJ. LEADER :		W.P. No. : 202 W.P. Nr. :	Proj No.: NS12	TEK. NR . DRAWING NO. D.4
NAGESIEN CHECKED :		GOEDGEGEUR : APPROVED		

# CRANE SUPPORT

Note:  
Material is 5mm mild steel

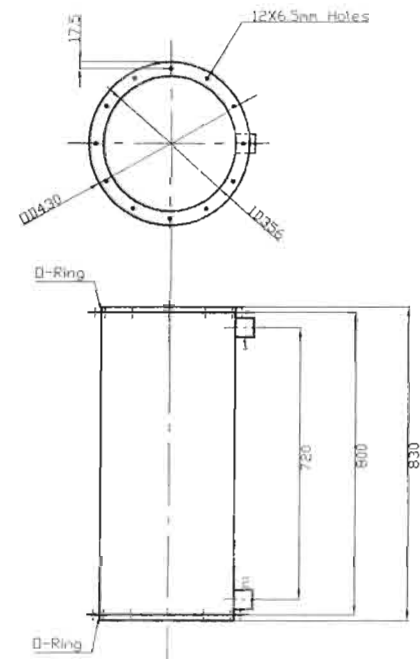


INSTITUTE FOR AGRICULTURAL ENGINEERING AGRICULTURAL RESEARCH COUNCIL		 INSTITUUT VIR LANDBOU-INGENIEURSWESE LANDBOUAVORSINGSRAAD	
GETEKEN : C.Talanda DRAWN : C.Talanda		<h2>CRANE SUPPORT</h2>	
ONTWERP : C.Talanda DESIGN : C.Talanda			
PROJ. LEIER : PROJ. LEADER :		WP No. : 202 WP Nr. :	Proj No: NS12
NAGESIEN : CHECKED :		GOEDGEKEUR : APPROVED :	TEK. NR : DRAWING NO.  D.5
		SKAAL : 1:4 SCALE :	DATUM : 20-12-2004 DATE :

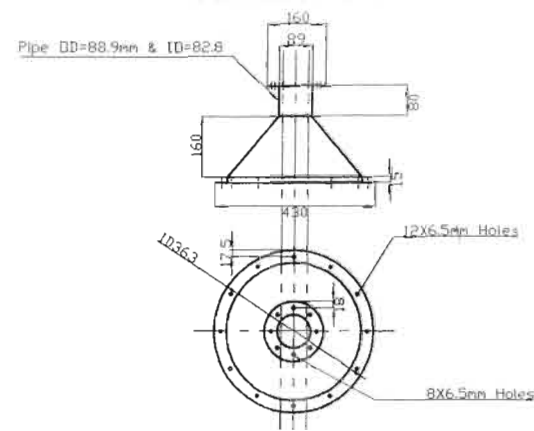
# 42 TUBE CONDENSER

NOTE:  
Material is Grade 304 Stainless Steel  
Flanges are 4.5mm thick  
Other material is 2mm thick

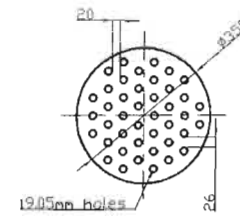
Condenser Shell



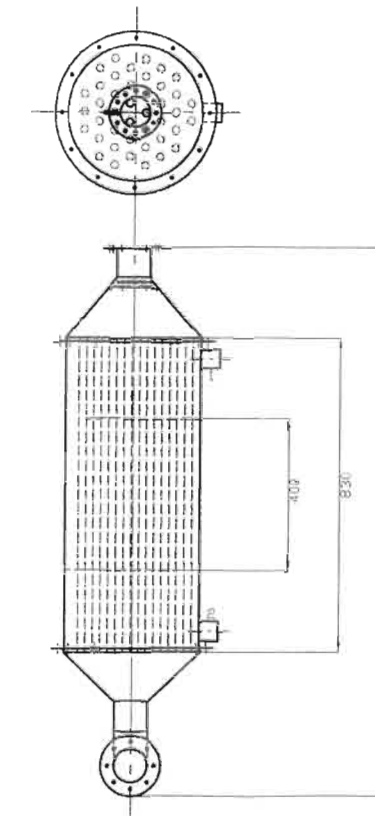
Condenser Inlet



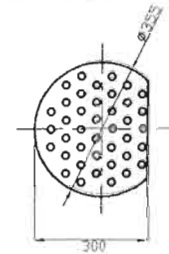
Shell End Piece (2 of)



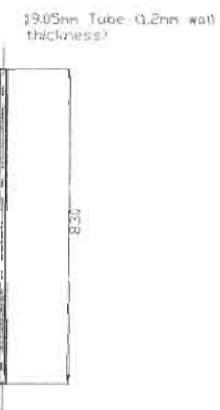
Assembly Drawing



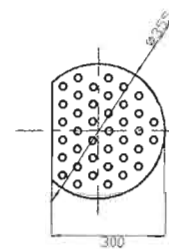
Condenser Baffle



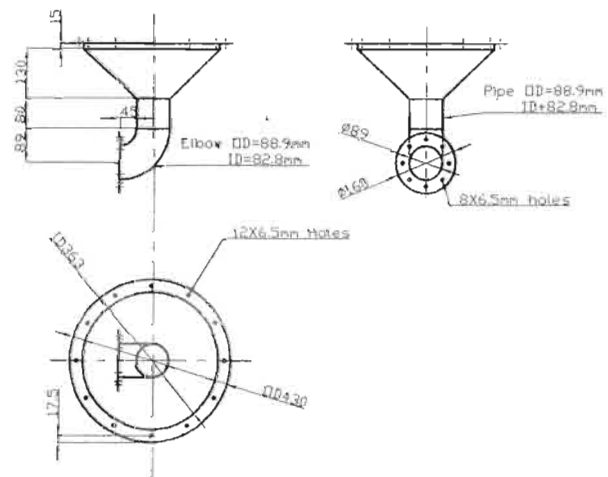
Diameter Tubes (42 of)



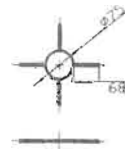
Condenser Baffle



Condenser Outlet



Inlet Steam Diffuser

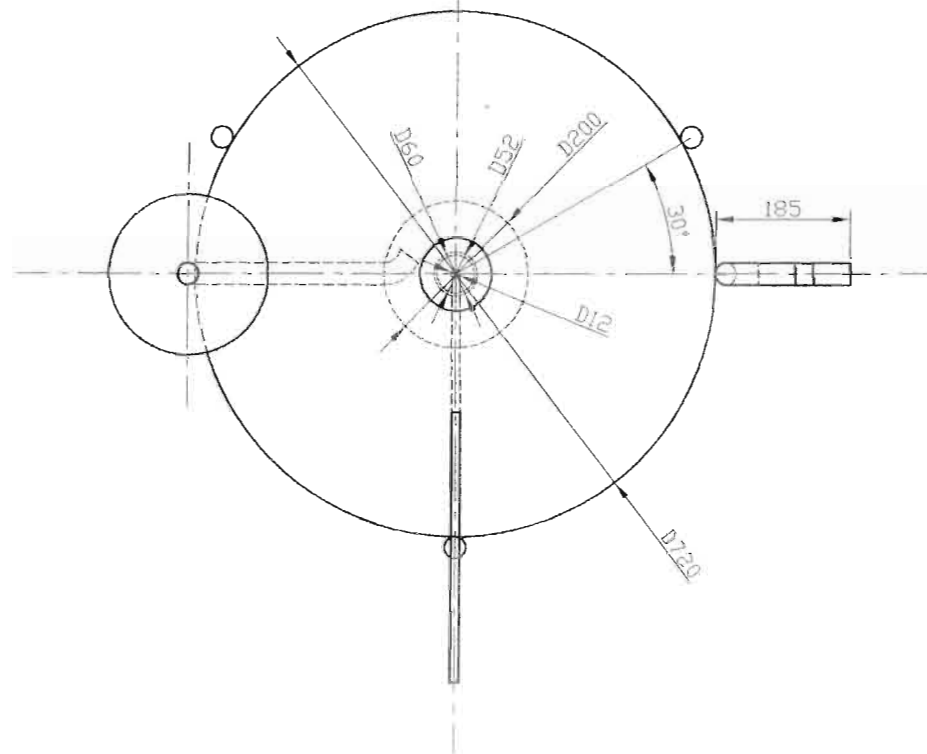
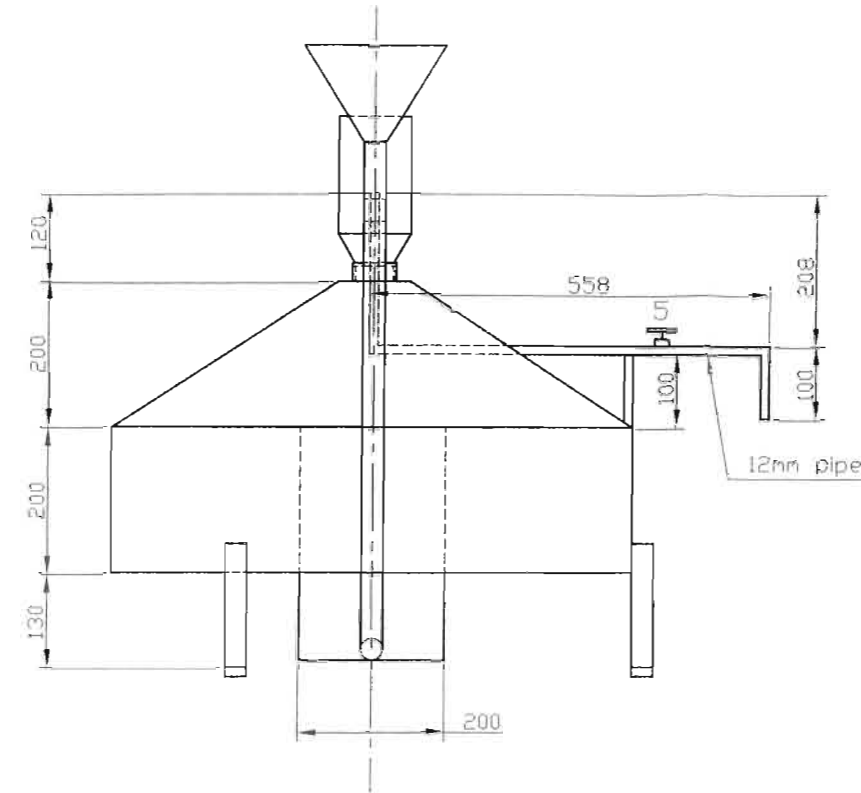
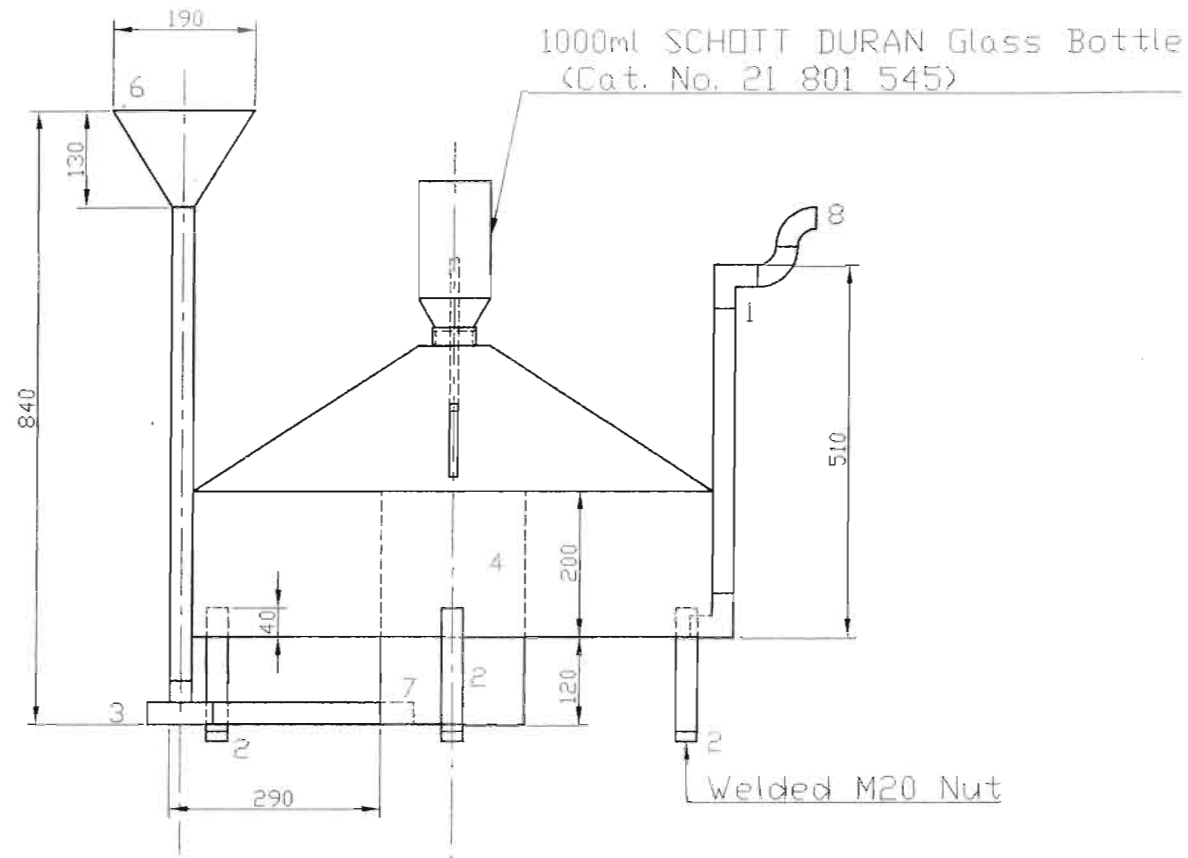


1. 1.5inch Socket
2. 1.5inch Socket
3. Cooling Water Baffle
4. Cooling Water Baffle

INSTITUTE FOR AGRICULTURAL ENGINEERING AGRICULTURAL RESEARCH COUNCIL		INSTITUUT VIR LANDBOU-INGENIEURSWESSE (LANDBOUWONKORINGSRAAD)	
DESIGNER : G. Takanda	SCALE : 1:20	CONDENSER	
DRAWN : G. Takanda	DATE : 20-12-2004		
TRIAL LEAD :	TEK. NR. : DIBBING 101	D.6	
APPROVED :	NO. : 202	REV. : NS12	

# SEPARATOR

NOTE:  
Material is Grade 304 Stainless Steel  
Material is 2mm thick unless specified



1. Water discharge 32mm elbow
2. 32mm  $\square$  Pipe Legs with welded M20 nut at end
3. Water discharge 32mm pipe tee with drain plug
4. Inner cylinder 200mm diameter
5. Oil discharge tap (12mm)
6. Distillate inlet funnel with 32mm pipe
7. Distillate curved outlet
8. S-bend outlet (2X32mm elbows welded end on end)

INSTITUTE FOR AGRICULTURAL ENGINEERING AGRICULTURAL RESEARCH COUNCIL		INSTITUUT VIR LANDBOU-INGENIEURSWESSE LANDBOUUNIVERSITEITSRAAD	
DESIGNER: G. Venter	SEPARATOR		SCALE: 1:100
DRAWN: C. Venter			DATE: 20-12-2004
PROJECT LEADER:	APPROVED:	REF No.: 202	PROJ No.: NS12
NUMERICAL QUESTION:			TEX. No.: DRAWING NO. D.7