Comparative performance of two domestic spirit stills

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Attestation of authorship

I hereby declare that this submission is my own work and that, to the best of my knowledge and belief, it contains no material previously published or written by another person (except where explicitly defined in the acknowledgments), nor material which to a substantial extent, has been submitted for the award of any other degree or diploma of a university or other institution of higher learning.

Candidate Signature: ……

Date……

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Abstract

The Turbo 500 still produced by imake Limited was designed to make home-distilled alcoholic beverages on a 23 L volume scale. The objectives of this study were to analyse the distillation performance of the Turbo 500 still, and to compare it with a competitor still, the essencia. The distillate fractions from the imake-recommended 60 to 65°C water outlet temperature treatment contained stable and high ethanol concentrations of around 90% (v/v), although the other distillation properties (distillation time, percent total recovery) were usually not as good as with a warmer 75 to 80°C cooling treatment. The stills performance comparison was done with a 15% ethanol wash. The essencia still generally produced distillates with lower ethanol concentrations and lower total ethanol recoveries. The essencia's performance was more affected by the cooling treatment than that of the T500 still. The mean theoretical plates value of the T500 was 5.08, which was higher than that of the essencia still (4.92). The gas chromatograph (GC) results of the distillates fractions from a 15% synthetic wash containing seven congeners showed essencia's recovery curves were more scattered from the ethanol recovery curve when compared with the equivalent curves of the T500 still. Ethyl acetate and acetaldehyde were better resolved than those of the other congeners by both stills. 2-Methylpropan-1-ol was better resolved by the essencia still. The GC-mass spectrometer (GCMS) results indicated that more esters were found in essencia's distillates from one of two fermentation recipes (Recipe B as recommended by imake). The reason for this is unknown. Sensory trials showed that, numerically, more panellists preferred the essencia distillate from Recipe B than the T500 distillate, but the difference was statistically insignificant (29 of 50, where 33 of 50 was $P = 0.05$). No differences were observed for Recipe A. The copper saddles (T500) and brass scourers (essencia) turned black after distillation. The results from scanning electron microscopy showed that copper, and copper and zinc (brass), became surface oxidised during distillation. Sulphur was also detected, and could be smelt after the materials were acid treated. Overall the T500 appeared to be the better still in distillation performance, but some recommendations for improvement of the T500 have been suggested.

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Chapter 1

Introduction

Alcoholic fermentation

Enzyme complexes in yeasts catalyse the conversion of simple sugars such as glucose, fructose and sucrose to lower-molecular-compounds, principally ethanol and carbon dioxide. This is the basic mechanism of alcoholic (more accurately ethanolic) fermentation. In these fermentations, yeast anaerobically metabolises sugars as a source of energy, while produced ethanol[1] and carbon dioxide are waste products (Berry & Slaughter, 2003). Alcoholic fermentation can be summarised in the below equation:

$$C_6H_{12}O_6 \rightarrow 2\ C_2H_5OH + 2CO_2$$

Depending on the biological sources of fermentable sugars, and events after fermentation is complete, the resulting liquors can be described as beers, wines and spirits.

Beer is produced after the enzyme-catalysed hydrolysis of starch from a grass grains (Trayner, 2002). Barley is the seed of the barley plant, which is the most common grain used as the raw material in brewing (Trayner, 2002) probably due to its high starch to protein ratio and low fat content. However, other grains like wheat, rice and maize can also be used, providing different beer flavours (Trayner, 2002). Figure 1 shows the brief flowchart of brewing, which describes three chemical processes: starch hydrolysis, sugar fermentation and post-maturation (Trayner, 2002).

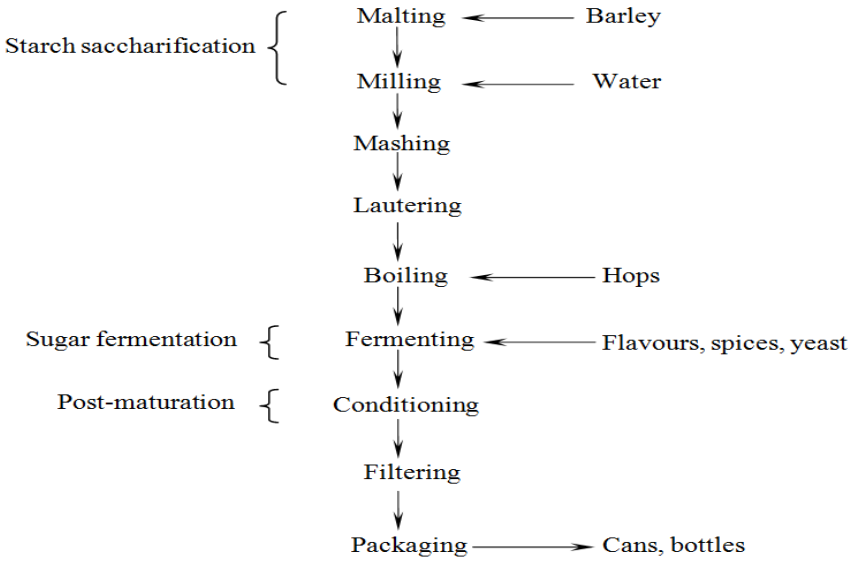

Figure 1 Flow chart of brewing process, summarized from Trayner (2002)

[1] Throughout this thesis terms alcohol and ethanol are both used for ethanol. Any other alcohols are specified, like butanol.

Wine is made from fermenting sugar-rich fruits or vegetables (Jackson, 2008). *Vitis*, the grape, is the common raw materials for winemaking (Jackson, 2008). As with beer, yeast metabolises sugars from fruits and vegetables and generates alcohol. There are three main steps in winemaking process: juice extraction, sugar fermentation (with or without yeast addition) and post-maturation. Figure 2 summarises the flowchart of winemaking (Street, 2009).

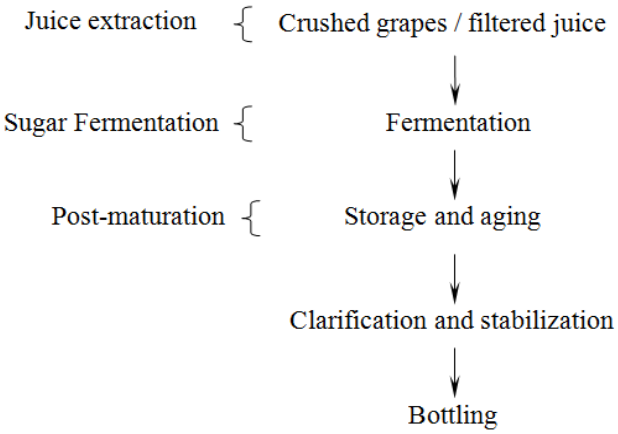

Figure 2 A flowchart of winemaking, summarised from Street (2009)

Beers and wines of every description are made, and in very many cultures. Fermentation to produce beers and wines does not yield alcohol concentrations greater than about 15%, because most yeast cannot tolerate ethanol concentration above 15%. To produce beverages with higher alcohol concentration, the fermented solution must be distilled, a process that partially separates alcohol from water, thereby concentrating the alcohol content. These high alcohol drinks are called spirits.

According to Wikipedia (2013b), "the term 'spirit' in reference to alcohol stems from Middle Eastern alchemy. These alchemists were more concerned with medical elixirs than with transmuting lead into gold. The vapour given off and collected during an alchemical process (as with distillation of alcohol) was called a spirit of the original material." Even though the original liquor placed in the still may be highly coloured (reds for many wines and browns for beer), the initial distillate is commonly colourless and clear. Subsequent to distillation the spirits, usually adjusted to about 40% (v/v[2]) ethanol, is usually flavoured and coloured in many different ways (Aylott, 2003). However, a substantial fraction of the world's spirit production is unflavoured, commonly referred to as vodka, schnapps, tequila, and in China Bai Jiu (literally white fermented spirit). Although colourless and clear, these white spirits are often far from flavourless, deriving as they do flavours from the original plant source. In

[2] v/v is subsequently implied throughout this thesis.

the case of gin, the distillation is carried out in the presence of 'botanicals', the key among them being dried juniper berries that give gin its basic gin flavour. Some white spirits, are however, redistilled many times to remove these plant-derived flavours, and other flavours derived from the yeast's activities during fermentation. Figure 3 indicates the brief flowchart of brandy production (Cantagrel & Galy, 2003).

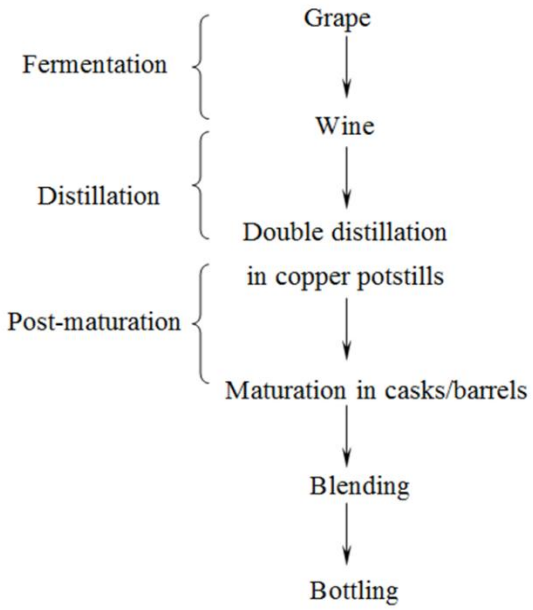

Figure 3 A brief flow chart of brandy production, summarized from Cantagrel and Galy (2003)

Spirits like whisk(e)y, brandy, dark rum, and certain tequilas develop much of their flavour and colour from storage in oak barrels. In the case of whisky from at least Scotland, the barrels used have previously been used to age fortified wines, like madeira, sherry and port wine, so those caramel flavours become part of the whisky flavour matrix (Piggott & Conner, 2003). The residues of those wines also helps colour the spirit, but the toasted oak wood from which the barrels are made also imparts the distinctive brown colour that is characteristic of these oak-aged spirits (Piggott & Conner, 2003).

The subject of this thesis is distillation in a domestic environment, which, as will be shown later, is a licit activity in New Zealand. As practised in New Zealand the objective of home distillation is to produce a relatively flavourless spirit that can be flavoured in very many ways. As noted above, fermentation to alcohol also generates a spectrum of minor products from the yeast's activity. Some of these are desirable (below a certain concentration) and others are considered undesirable. Once produced in the final fermented liquor, also called the 'wash', they can be removed only by fractionation during distillation. That issue is

researched later in this thesis, but in the meantime it is useful to describe how they are formed during fermentation.

Desirable and undesirable compounds in alcoholic beverages

Ethanol is the primary products from fermentation, and there are many types of other minor (by-) products like aldehydes, ketones, alcohols, esters, acids and carbohydrates (Jung et al., 2010). These minor products – also called congeners – are volatile and non-volatile compounds, which are partly responsible for sensory attributes (Jung et al., 2010). These subtle differences in congeners within liquor type (beer, wine, spirit) partly distinguish are drink from another (Cantagrel & Galy, 2003).

However, some of these congeners are responsible for off-flavours in liquors, which are considered flaws in the flavour profiles (Jung et al., 2010). Their concentration depending on how the liquors are made, arising from variations in fermentable material, yeasts, temperatures and other factors (Nykänen, 1986), for example variations in distillation. Greizerstein (1981) indicated that the undesirable congeners mainly exist in darker liquors, such as aged tequila, brandy, whiskey and red wine, while clear liquor, including gin, vodka and white rum, the concentrations of congeners is lower.

The ways of producing several congeners are generated and reviewed as follows:

Acetaldehyde is an intermediate in the formation of glucose to ethanol (Tadege, Dupuis, & Kuhlemeier, 1999). In this process, glucose is firstly catalyzed by yeast enzymes to form two pyruvate compounds. The two pyruvate compounds release two carbon dioxide molecules and form two acetaldehyde molecules in the anaerobic condition. Subsequently the two acetaldehydes are reduced by 2NADH to form two ethanol molecules (Figure 4) (Barnett, 2005; Tadege et al., 1999). The concentration of acetaldehyde depends on the activity of pyruvate decarboxylase by different types of yeast (Then & Radler, 1970).

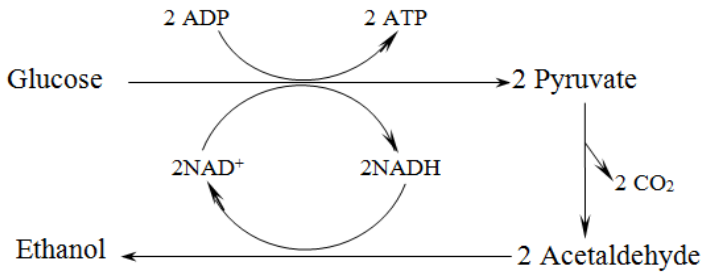

Figure 4 The formation pathway of acetaldehyde in fermentation, summarised from Then and Radler (1970)

Methanol is formed from the decomposition of pectin in plant cells (Jackson, 2008). Pectin is partly composed of methyl esters of galacturonic acid (indicated in the formula below) (Pieper, Oplustil, & Barth, 1980). When pectin is metabolised by yeasts' pectin esterases, the methyl ester reacts with water to produce the methanol.

$$\text{Pectin} + H_2O \xrightarrow{\text{Pectinase}} \text{Methanol} + \text{Pectic acid}$$

Fusel alcohols (fusel oils) is a general name for congener alcohols that contain more than two carbon atoms, typically contains propan-1-ol, butan-1-ol, butan-2-ol, 2-methylpropan-1-ol (isobutanol), 2-methylbutan-1-ol (active amyl alcohol) and 3-methylbutan-1-ol (iso-amyl alcohol) (Jackson, 2008; Kuhn, Vickers, Ebeler, Ahlgren, & Thorngate, 2003). Fusel alcohols are common in alcoholic beverages and contribute to spicy, hot, burning flavours (Nykänen, 1986). Ehrlich (1907) indicated that fusel alcohols are formed from transamination of amino acids to carbohydrates cited from Hazelwood, Daran, van Maris, Pronk, and Dickinson (2008) (Figure 5). Amino acids that are assimilated by the Ehrlich pathway are taken up slowly throughout the fermentation time (Hazelwood et al., 2008). Each fusel alcohol has its corresponding amino acids (Pietruszka, Pielech-Przybylska, & Szopa, 2010). Figure 6 indicated the Ehrlich pathways of 2-methylbutan-1-ol, 3-methylbutan-1-ol, propan-1-ol and 2-methylpropan-1-ol, which are derived from leucine, isoleucine, threonine and valine respectively (Boulton & Quain, 2001; Boulton., Singleton., Bisson., & Kunkee., 1999).

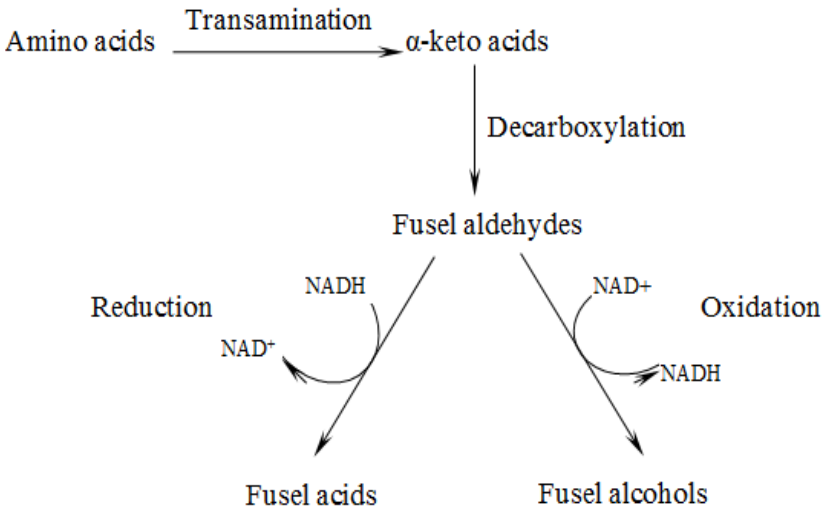

Figure 5 The formation pathway of fusel alcohols in fermentation according to the Ehrlich pathway, summarised from Pietruszka et al. (2010)

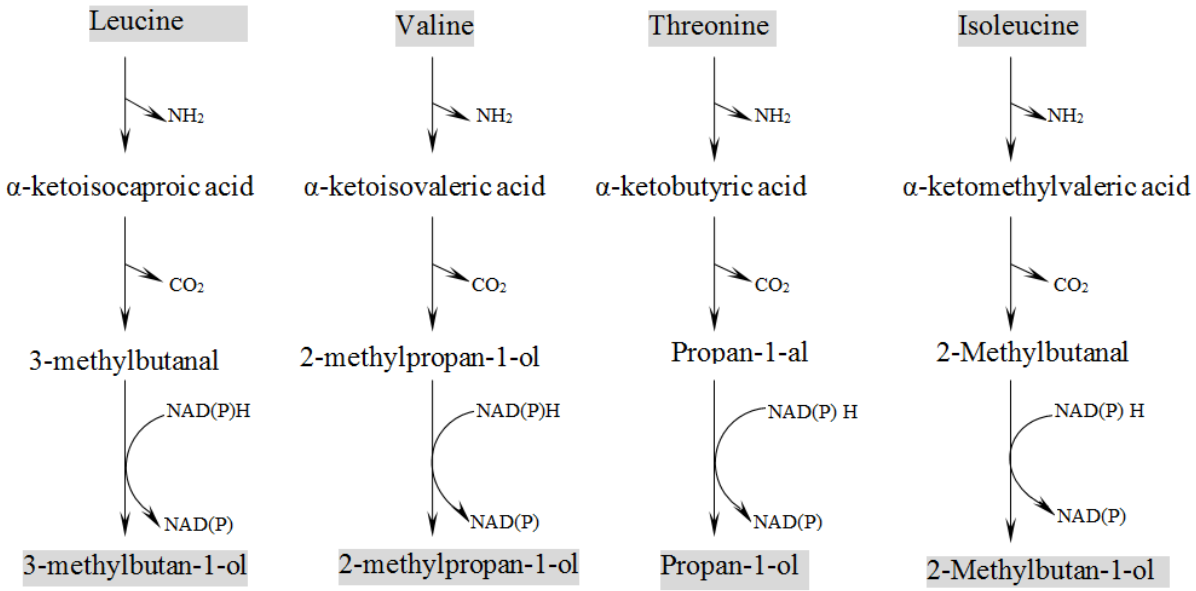

Figure 6 The formation pathway of 2-methylbutan-1-ol, 3-methylbutan-1-ol, propan-1-ol and 2-methylpropan-1-ol, summarised from Boulton. et al. (1999)

Sulphur compounds are generally considered as undesirable in liquor. They generally have lower odour thresholds than other flavour compounds; therefore, even low concentration can cause flavour problems (Mestres, Busto, & Guasch, 2000). Table 1 indicates the sensory description and thresholds of common sulphur compounds in liquor (Mestres et al., 2000).

Table 1 Sulphur compounds commonly found in wines[1]

Name of sulphur compounds	Sensory description	Detection threshold in wine (μg L^{-1})	Boiling point (°C)
Dimethyl sulphide	Quince, truffle	10.0	38
Dimethyl disulphide	Asparagus	15.0	110
Diethyl sulphide	Quince, truffle	0.9	92
Diethyl disulphide	Garlic	4.3	152
Methanethiol	Cooked cabbage	1.5	6
Hydrogen sulphide	Rotten egg	0.5	-60
2-Mercaptoethanol	Asparagus	2.0	157

[1] From Mestres et al. (2000)

The sulphur compounds in liquors can be classified into five families: sulphides, polysulphides, thiols, thioesters, and heterocyclic compounds, although no heterocylic compounds are in Table 1 (Mestres et al., 2000). The degradations of cystine, cysteine, methionine, and glutathione are the main sources of sulphur compounds (Mestres et al., 2000). Figure 7 indicates the ways sulphur compounds are generated in yeast sulphur metabolism (N Moreira, Mendes, Guedes de Pinho, Hogg, & Vasconcelos, 2008).

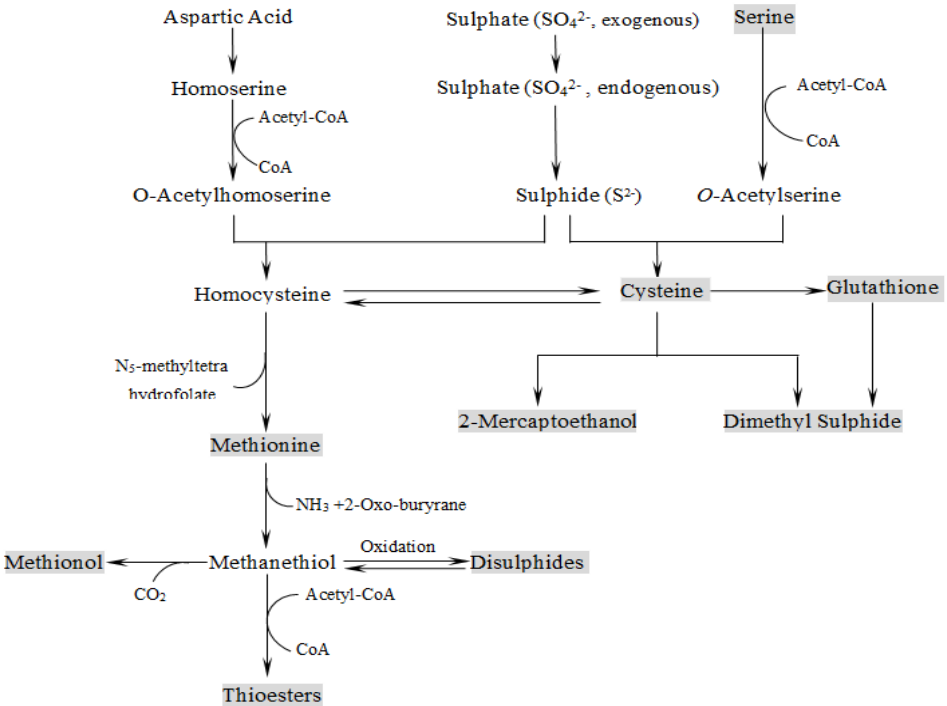

Figure 7 Yeast sulphur metabolism. Summarised from N Moreira et al. (2008)

The production of congeners can be controlled to some extent during fermentation, but once produced are integral to the liquor. However, with distilled liquors there is an opportunity to minimise their concentration in the final distilled spirit. This is because distillation is capable of fractionating compounds based on their volatilities.

Sensory analysis of beverages

The previous section discussed congeners and stated that their concentration can be minimised by fractional distillation. However, they can affect flavour in low concentrations, sometimes at concentrations below the limits of detection of chemical analytical methods. To assess their impacts on flavour requires sensory methods where people are the analytical instrument. The results from sensory analysis show the responses of consumers, which further affect the market of food products. In this way, human responses (smell, taste, touch, sights or hearing) on foods' characters are main objects sensory analysis can be evaluated and measured by sensory analysis. To beverages, appearance, aroma, taste and texture are main objects that analyzed by sensory.

This chapter will use two sensory analysis methods: triangle test and paired preference test, to detect the differences and preferences on distillates from T500 and essencia stills. The triangle test is a discrimination testing to distinguish whether there is a detectable difference among two or more products (Meilgaard, Civille, & Carr, 1999). The paired preference test compares liking in pairs, which require consumers to specify which of two foods is preferred or whether there is no preference.

Distillation theory and practice

Distillation is a physical method that separates mixtures by the components' different volatilities. The theory of distillation is based on Dalton's gas laws and their relevance to distillation is well summarised in Wikipedia (2013a) and Petlyuk (2004), the basis of the following discussion of distillation theory.

Dalton's law states that the total vapour pressure is the sum of the vapour pressures of each component in the mixture. When a multi-component liquid is heated, the vapour pressure of each component will rise, thus causing the total vapour pressure to rise. When the total vapour pressure reaches the ambient pressure (normally atmospheric), boiling occurs and liquid turns to vapour. This means that a mixture of a given composition has a single boiling point at a given pressure. At this boiling point all volatile components boil, and this mixture is called an azeotrope. Thus, an implication of the single boiling point is that lighter components never cleanly 'boil first'. For each component, its percentage in the vapour is the same as its

percentage of the total vapour pressure (Dalton's Law). Lighter components (more volatile) have a higher partial pressure and thus are concentrated in the vapour, but heavier volatile components also have a partial pressure – but lower – and also evaporate, and are less concentrated in the vapour. Therefore, in binary (and in more complex systems) one liquid component cannot be completely separated from the other liquid components by normal distillation. Table 2 shows the boiling temperature of alcohols-water azeotropic mixture at 1 atmosphere (Tanzer, Lostocco, Branham, & Bunyard, 2007). In the case of ethanol (ethyl alcohol) the azeotropic boiling point at atmospheric pressure is 78.1°C, which that deviates from the individual boiling point of ethanol (boiling at 78.4°C) and water (100°C).

Table 2 Alcohol-water azeotropes at 1 atmosphere[1]

Alcohols	Boiling point of azeotrope (°C)	Water in the azeotropic vapour (%) mole
Ethanol	78.1	4.5
Propan-2-ol	80.4	12.1
1-Propanol	87.7	28.3
2-Butanol	88.5	32.1
2-Methylpropan-1-ol	90.0	33.2
1-Butanol	92.4	38.0

[1] From Branham, Bunyard, Lostocco, and Tanzer (2006)

Distillation is fundamental spirits production from alcoholic washes. Depending on the characteristics the wash and the compounds of interest, distillation has been developed into various types: simple distillation, steam distillation, vacuum distillation, air-sensitive vacuum distillation, short path distillation, reactive distillation, fractional distillation, and continuous distillation (Petlyuk, 2004). Some of these are now discussed.

Simple distillation is the most basic method, the laboratory version of which is shown in Figure 8 (*File: Simple distillation apparatus.svg*, 2007). The more volatile compounds initially evaporate from the surface (15) more than the less volatile compounds, and condense earlier into the collector vessel (8). Simple distillation is a widely used method, which can quickly concentrate one volatile product from a mixture, and is easy to operate. However, simple distillation works best when the volatile compounds' boiling points differs markedly. The minimum temperature difference should be 25°C (Petlyuk, 2004).

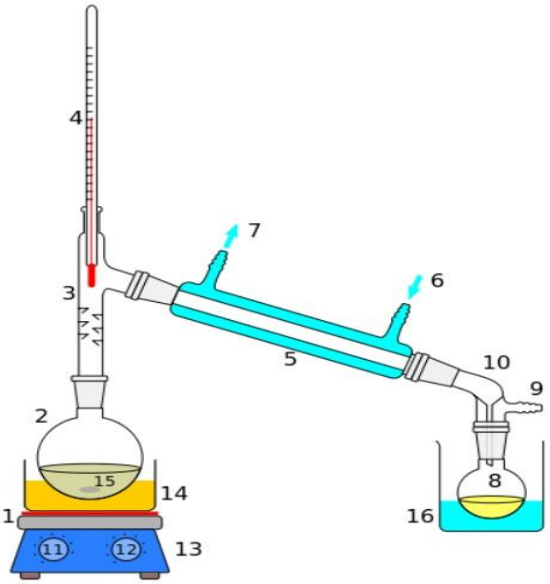

Figure 8 Simple distillation. Volatile components in the heated (11) liquid (15), vapourise and pass through the condenser (5). The distillate (8) is collected. Retrieved from *File: Simple distillation apparatus.svg 2007)*

Steam distillation uses an external steam source that passes through the material to be distilled that comprises typically plant matter or crude fat (Kister, 1992). Steam distillation is often used to distil the heat-sensitive and water-insoluble compounds, such as natural aromatic compounds (Kister, 1992).

Figure 9 shows a laboratory model of steam distillation, where plant matter (green solid) is subjected to a flow of steam (blue) and condensed into a conical receiver. The product of steam distillation is usually a two-phase liquid of organic compounds (yellow) and water (blue) (Kośmider, 2010). The final targeted products can be collected by decantation (Kister, 1992).

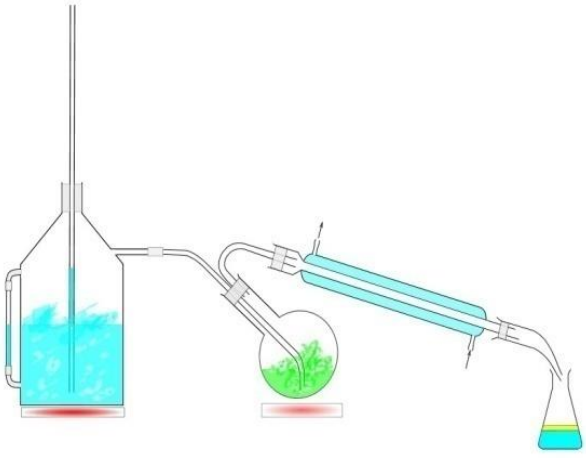

Figure 9 Steam distillation apparatus in a laboratory configuration. Retrieved from Kośmider (2010)

Vacuum distillation, is used to distil liquids that are heat sensitive or have high boiling points at atmospheric pressure (Kister, 1992). In this technique, liquids are distilled in an evacuated

environment. As the inside pressure of mixture liquids is lower than the normal atmospheric pressure, compounds boil at a low temperature and suffer no or less damage by heat (Petlyuk, 2004).

Extractive distillation is used to separate mixture components have low volatility and have similar boiling points (Kister, 1992). In this method, a suitable 'working' solvent is added in the liquid mixture (Petlyuk, 2004). The solvent must have a much higher boiling point than the components to be distilled (to the point of being essentially non-volatile), be miscible with the components, and be non-reactive with components (Petlyuk, 2004). By interacting with components, the working solvent can change the relative volatilities of the components. Thus the final mixture can be distilled more easily. The mixture of benzene and cyclohexane is a classic example of extractive distillation, where aniline is the working solvent (Kister, 1992).

Fractional distillation is used when the boiling points of mixture compounds are close to each other (less than 25°C) (Petlyuk, 2004). Figure 10 shows a laboratory setup for fractional distillation (Kershaw, 2008). The condenser of fractional distillation apparatus is packed an array of plates or trays (Kister, 1992).

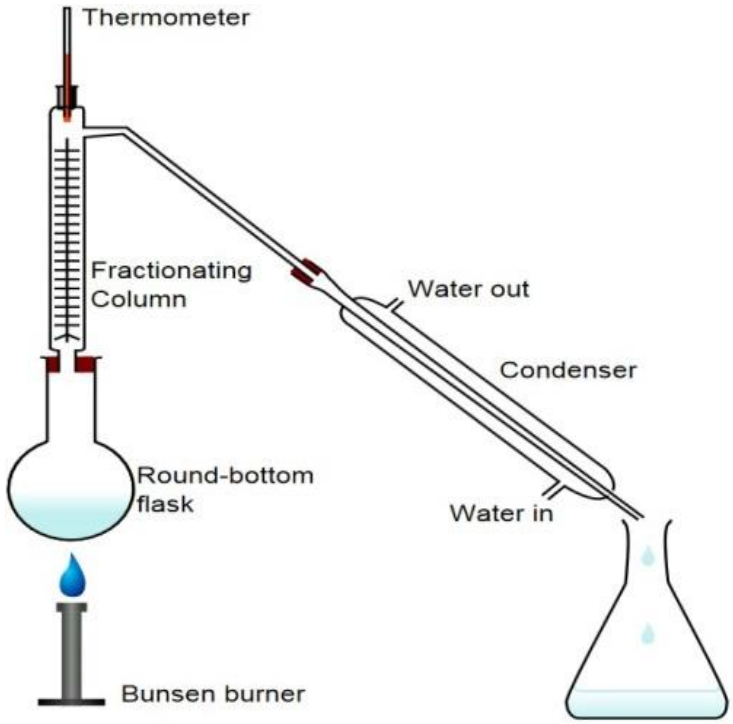

Figure 10 Fractional distillation apparatus in a laboratory configuration. The wash is heated in round-bottom flask. The vapour passes through the fractionating column and is condensed by the cooling condenser. The distillate is finally collected in the conical flask. Retrieved from Kershaw (2008)

The examples above are all batch distillation systems, while industrial distillation systems are often continuous (Padleckas, 2006a). Figure 11 is a plan of a typical continuous system. The

distillation tower contains a series of plates or trays, and the entire system uses a reflux cycle to achieve more complete separation of components.

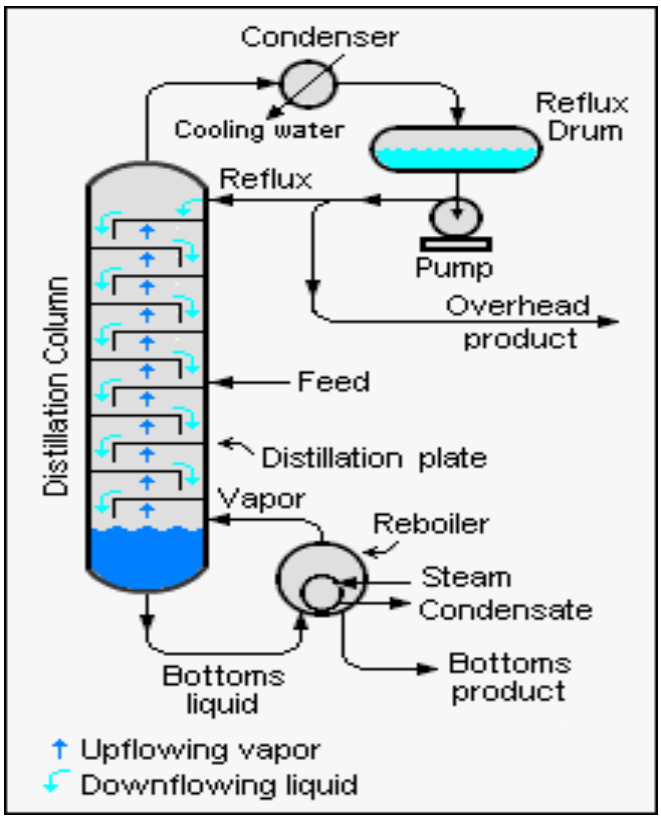

Figure 11 Diagram of a typical industrial distillation system (Websters-online-dictionary). Retrieved from Padleckas (2006a).

The flow of components in the distillation tower is shown more clearly in Figure 12, although this figure does not show the reboiling, reflux, and pumping systems. A heated mixture is fed into the column at its midpoint. For this discussion the mixture has two components, a binary distillation. Within the column, vapour of both components rises and is obstructed by a series of plates filled with so-called bubble caps that partially obstruct flow. Some condensation, mainly of the higher boiling point component, occurs here. This condensate (cooler than the vapour) is progressively drained by gravity through the series of plates, becoming progressively enriched in the higher boiling point component (HBPC). The reverse occurs for the lower boiling point component. In Figure 12, there is a partial draw off of the LBPC (Lower boiling point component), controlled by the reflux ratio that effected between the pump and column in that figure. The performance of the distillation process depends on the system of equilibrium stages and can be enhanced by providing more stages in the form of plates (Petlyuk, 2004). Therefore, more plates increase the efficiency of the separation process, and lead to a better separation (Petlyuk, 2004).

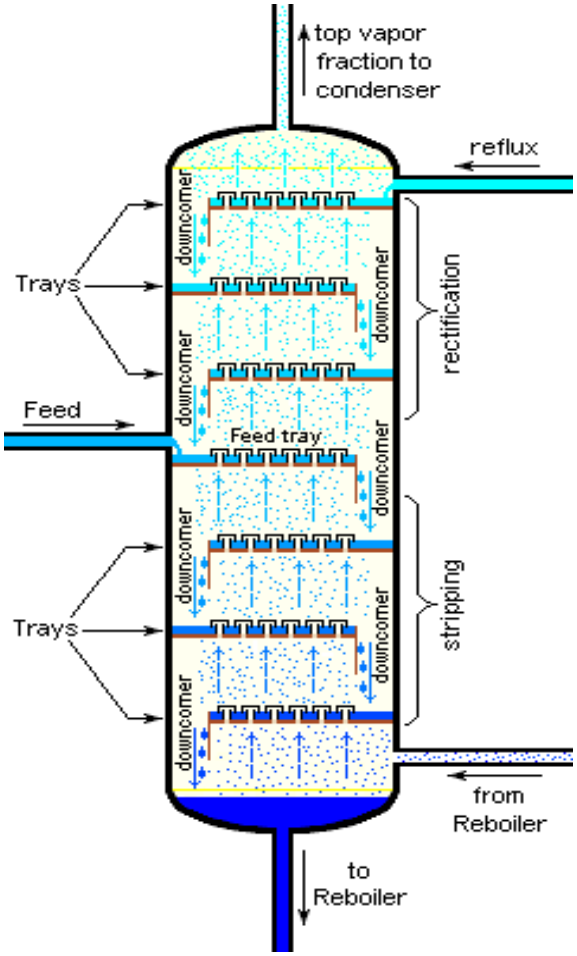

Figure 12 Chemical engineering schematic of fractionating column bubble-cap trays in a distillation tower. Retrieved from Padleckas (2006b)

In industrial use, the distillation column is sometimes filled by packing material instead of plates or trays (Kister, 1992). The packing materials are variable according to different needs. Different packing materials have different surface areas and void spaces, all affecting the performance (Kister, 1992).

The theory of distillation has developed the concept of plates. A plate is a stage in distillation where the vapour and liquid phases reach equilibrium, represented by the trays in Figure 12. In fact, the liquid-vapour equilibrium does not occur perfectly on every plate in a column, and becomes more complicated in a packed column (Petlyuk, 2004). Therefore, the concept of theoretical plates was developed to describe the true number of equilibrium in any column, plated, packed or void (Petlyuk, 2004). The number of theoretical plates is a measure of the separation efficiency of a fractionating column. The more theoretical plates, the more efficient of the column (Petlyuk, 2004).

Calculation methods of theoretical plate in fractional distillation

The Fenske equation (below), which was derived by, is used to calculate the minimum number of theoretical plates that required for the separation of a binary feed (Fenske, 1932). Fenske equation assumed the relative volatility is constant in the distillation column, which means the distillation column should be at a total reflux condition to use the Fenske equation.

$$N = \frac{\log\left[\left(\frac{X_d}{1-X_d}\right)\left(\frac{1-X_b}{X_b}\right)\right]}{\log \alpha_{avg}}$$

In the Fenske equation, N is the minimum number of theoretical plates required at total reflux. X_d is the mole fraction of more volatile component in the overhead distillate. X_b is the mole fraction of more volatile component in the bottoms. α_{avg} is the average relative volatility of the more volatile component to the less volatile component.

If the relative volatility of the less volatile component to the more volatile compound is constant from the column top to the column bottom, then α_{avg} is simply α. If not, an approximation is applied that takes the relative volatilities at the top and bottom of the column, then the following approximation will be used:

$$\alpha_{avg.} = \sqrt{(\alpha_t)(\alpha_b)}$$

α_t is the relative volatility of more volatile components to less volatile components at the top of column

α_b is the relative volatility of more volatile components to less volatile components at the bottom of column

Stills for spirit production are not always like those in Figure 11, which are designed to enrich the two components to a greater or lesser extent, such as in the petroleum industry (Kister, 1992; Petlyuk, 2004). In the spirit industry the aim is to isolate an ethanol-rich distillate from a water phase. The ethanol-rich distillate contains some water and also some congeners, as well as compounds derived from the original fermented plant material (Berry & Slaughter, 2003). In the case of vodka production the congeners and the plant-derived compounds are undesirable, but in the case of tequila for example, the plant-derived compounds at least are highly desirable because these characterise the spirit. Overall, optimal design of spirit stills is much less demanding than for the chemical-engineering industry, partly because the demands of final spirits are typically enriched in ethanol to only 40%. Thus the theoretical plate values

for spirit stills are usually less than those for chemical stills, but with the same principles for plates' calculation.

Stills for spirit production

The simplest still used for commercial spirit production is a pot still, which is used in a batch process. As the name suggests, a pot still is little more than a pot, usually made of copper, with little fractionating ability in the still head and swan neck (Figure 13). As a result of this design congeners and plant-derived compounds are included in the distillate. These can be strong flavours, but are expected in cognacs and whiskies. Figure 14 shows a typical and impressive bank of pot stills in a Scottish whisky distillery.

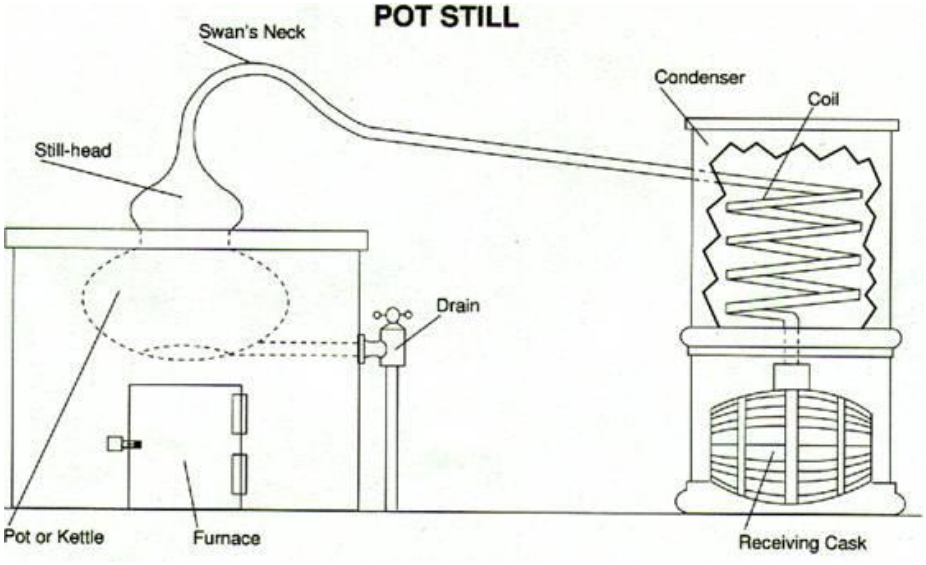

Figure 13 A schematic structure of a pot still. Retrieved from The Distillation Process 2012)

Figure 14 A bank of copper pot stills as used in a Scottish distillery, Glendronach Distillery, Speyside (Akela 2010)

The mechanism of pot still distillation is based on the theory of simple distillation (Chapter 1). The first distillate from the pot still always has lower alcohol content than required for whisky at retail because the pot still has low separation efficiency in terms of theoretical plates. Therefore, it has to be distilled at least twice to achieve sufficient alcohol, around 60% that customarily used for barrel maturation (barrel strength)(*Using a pot still* 2012). The primary distillate from the pot still is considered to be in three parts: the head, hearts and tails. The head contains the most volatile components from the wash. The head distillate is usually the least pleasant to taste and contains most of the congener methanol. The heart contains highest proportion of ethanol and the lowest proportion of undesirable congeners. The final distillate components, namely the tails, contain higher proportion of the less volatile congeners, higher alcohols for example, and plant-derived volatile matter. The heads and tails, which also contain substantial concentrations of the desirable ethanol, are redistilled to remove enough undesirable congeners to create the flavour profile required by the distillery.

The patent still, which is also named as the Coffey still, is a continuous still or column still, and is a general name for a still type characterised by two parallel columns (Figure 15). The mechanism of patent still is based on the theory of fractional distillation, discussed earlier.

Figure 15 The appearance of patent still. Retrieved from HighKing (2013)

Figure 16 shows the schematic structure of patent still, comprising column A (analyser) and column B (rectifier). The signal distillation cycle of patent still is indicated as follows:

The inside of column A contains a series of perforated diaphragms of copper plates. The cold wash enters at the top of column B, and passes through the zig-zag transfer tubes to the top of column A. Steam is pumped into the bottom of column A, and constantly rises in the column A. The descending wash meets the hot uprising steam at the perforations. Due to the lower boiling points of alcohols and other volatile congeners, the steam heats the wash and vaporises the alcohol and congeners. The counter current wash that reaches the lowest plate has thus been deprived of most alcohols and congeners. Because the temperature decreases from the bottom to the top of column A, the higher the vapours rise, the richer the vapours become in alcohol.

The alcohol, steam and the other congeners pass out the top of column A to the bottom of column B (rectifier). The escaping vapours raises in column B, contacting the cold zig-zag tube that serves as the condenser. Water in the vapour condenses before the alcohol and when the vapour reaches the top of column B, it is highly enriched in alcohol, around 95%. The less volatile components are condensed gradually in column B, and are refluxed to the top of column A for another distillation cycle.

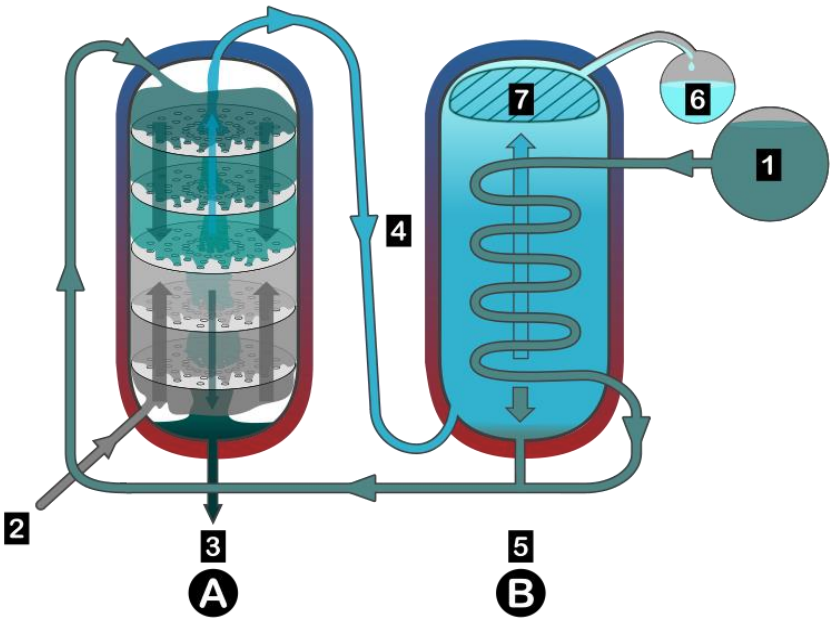

Figure 16 The schematic structure of patent still. The fermented wash (1) is pumped through the rectifier (B) to the top of analyser (A). Steam (2) enters the bottom of the analyser. The wash is heated by the uprising steam to extract alcohol and congener vapour that pass via the transfer tube (4) to the bottom of the column B. The alcoholic vapours are gradually condensed by the incoming cold wash and pass out as a concentrated alcoholic liquid. The less volatile components in the alcoholic vapours are recycled (5) and pumped in to column A for another distillation cycle

The performance of the still is controlled by the rates of steam and wash flow. By using the cold incoming wash as the condensing liquid this continuous still is economical in respect of cooling water.

Whatever still is used, from the simplest batch pot still to a continuous patent still, the act of distillation is subject to legislation, because alcohol is a drug, with varying legal status around the world.

Legislation on alcohol beverages and domestic production

The legislation of alcoholic beverages varies in different countries. The content of legislation contains definition of alcoholic beverages, restriction of supplying and selling, limitation of purchasing age, price, taxation, advertising, and alcohol-impaired driving. Alcoholic beverages are totally prohibited in Muslim countries, such as Egypt, Jordan, Libya, Arabia, Islamic Republic of Iran, Sudan and Saudi Arabia (WTO, 2004), although exemptions can apply to hotels catering to Western guests.

The definition of alcoholic beverages depends on the degree of alcohol percentage in beverages. In 2002, the WTO researched the world's alcohol policy in 181 countries representing 86% of the world's population. The legal alcohol lower limit was from 0.1% to 12% (alcohol % by volume). For 85% of countries, the lower limit was 4.5%, but in New Zealand the limit is 1.15%. Age limits for the purchase of alcohol also vary internationally. A lower limit of 18 years is very common, but with the USA being a major exception where 21 is the limit. Some countries, including China, have no age restriction.

Alcoholic beverages are subject throughout the world to a specific tax called duty. Generally the higher the alcoholic content the higher the duty, although not in perfectly linear relationship where buying alcohol as a spirit is relatively cheaper than as wine or beer. However, in New Zealand the relationship is close to linear. Whatever the duty, however, there is an incentive to avoid it. The legal way to do this is domestic production of alcohol for personal consumption, although avoidance is not the only motive for domestic production. In many countries, particularly those with Western cultural traditions, domestic production is considered a worthwhile hobby.

Domestic alcoholic production involves the manufacture of a range of alcoholic beverages for personal consumption with no commercial intent. Beer, wine, cider, sake, mead and perry are common home brew beverages, while whisky, gin and vodka are the common spirits mage after home distillation. There are several factors that have fuelled the market of domestic alcoholic beverages. First, domestic production is generally cheaper than bought equivalents, largely because of duty avoidance. Second, people can create more desirable recipes to cater for their personal tastes. Third, home-made alcoholic beverages are perceived to be more environmentally friendly, because the packaging and transport are not necessary.

The legislation on domestic production varies from country to country (WTO, 2004). Table 3 summarises the legality in several countries. Home brewing is legal in many countries, except Malaysia, Iran and India and probably other countries with a Muslim tradition not listed here. Compared with home brewing, home distillation is more restricted internationally. Home brewing and home distillation are both legal in New Zealand, Hungary and Russia.

Table 3	The legislation of home-brewing and home distillation	
	Legal for personal use	Illegal
Home-brewing	Germany, Hungary, Poland, Sweden, Ireland, Russian, UK, USA, Canada, Australia, New Zealand, Singapore, Indian, South Africa, Iran, Malaysia, Japan	Malaysia, Iran India
Home distillation	Hungary, Russia, New Zealand,	Sweden, Poland, Ireland, Japan, Malaysia, Iran, India

Brief introduction to the Turbo 500 still and the essencia express still

In New Zealand it is legal to produce home-made alcoholic beverages; a license is not needed nor is duty payable. For these reasons and for cultural reasons, home brewing and home distillation have a large following in New Zealand, particularly among males. In response to the demand several companies have been established to supply the domestic industry. Recipes, yeasts, containers, hydrometers, and specialised apparatus such as stills are the common products that are sold.

imake Limited., formerly known as Brewcraft, has been operating for 30 years in New Zealand, selling equipment and ingredients in to support the domestic industry[3]. In the liquor part of the business, imake sells around 3,000 products mainly from five brands: Mad Millie, Still Spirits, Copper Tun, Mangrove Jack's and Vintners Harvest. The aim of imake is to help people make cheaper, delicious and fresh foods and beverages at home. imake also exports to Australia, United Kingdom, USA, Canada, South Africa, Europe and China.

The Turbo 500 still (T500 still), which is a brand of Still Spirits, is the 'flagship' still that is manufactured and sold by the imake. It retails at around NZ$699. It was designed to purify ethanol from home-brewed wash, enabling the home distiller to create commercial quality, and clear spirits such as gins, rums, vodkas and whiskies. The T500 is a batch still with a fractionating column mounted on the wash tank. The fractionating column in turn leads to a conventional water-cooled condenser from which the distillate is collected.

[3] The company also supplies equipment for other domestic activities like cheesemaking.

The School of Applied Sciences, AUT University, was approached by imake to explore the T500's performance under a range of conditions. That is the major work undertaken in this thesis. At the same time imake was interested in the performance of a competing still, the essencia made by Fermanagh N.Z. Limited[4]., which is also aimed at the home spirit market. A part of this thesis is a performance comparison of the two stills that extends to sensory evaluation. The detailed performances of the T500 and essencia stills are researched in following aspects: the performance of ethanol recovery, the purifying ability of alcohol congeners, the calculation of theoretical plates, sensory tests on the distillates of both stills. Finally recommendations are made as to how the T500 might be improved.

Statistics

In this research, the distillation data typically represent only single experiment trials, because of the time limitation of a one year Master thesis. The potential for between-run variations had to be ignored. However, as will be seen, the distillation results were generally believable and could usually be interpreted from simple physical laws. Nonetheless, the lack of replication for distillation must not be forgotten, and if imake wants to verify key outcomes then replicated trials are recommended.

[4] Details of this company have been difficult to access. The New Zealand Companies Office register has not been helpful.

A guide to the thesis layout

Table 4 summarises the layout of this thesis.

Table 4 The structure of the thesis

Chapter 2	Performance of the Turbo 500 still with binary mixtures of ethanol and water
Chapter 3	Performance of the essencia express still with binary mixtures of ethanol and water
Chapter 4	Calculation of theoretical plates for the Turbo 500 still and the essencia express still
Chapter 5	Performances of T500 and essencia stills with a synthetic wash
Chapter 6	Gas chromatography and sensory analysis of distillates from two model fermentations: a comparison of stills' performances
Chapter 7	Analysis of ceramic saddles, copper saddles (T500) and scourers (essencia)
Chapter 8	Conclusion

Chapter 2

Performance of the Turbo 500 still with binary mixtures of ethanol and water

Introduction

As mentioned above, the Turbo 500 still (T500 still) is the main still that sold by imake (chapter 1.5). In response to imake's suggestions, the work in this chapter describes the distillation performance of T500 still. The distillation performance includes the analysis of ethanol concentration, kinetics of distillate collection volume and ethanol recoveries.

The structure of the T500 still

The T500 still is shown in Figure 17.

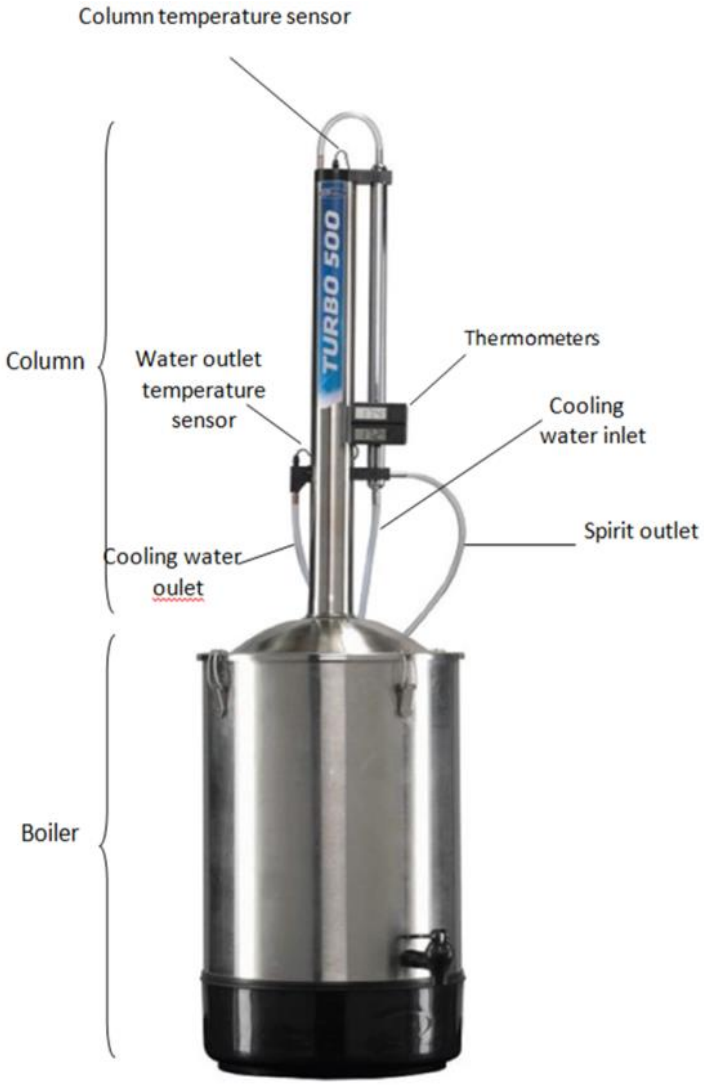

Figure 17 The T500 still

Boiler, fractionating column, and the condenser are the main three parts, mainly constructed from stainless steel. The boiler contains a 2 kW heating element at bottom to heat the wash and a waste tap to discard the wash after distillation. The heating element is not in direct contact with the wash. The exact performance of this element is affected by one other factor, the room ambient temperature. The total volume prescribed for the boiler is 23.5L. Figure 18 shows a rough sketch of cooling water flow in the column and condenser. The cooling water flows from the lower end of the condenser to the upper, thereby condensing the vapors into distillate. This drawing, supplied by imake, is not to scale, but clearly shows the path of water flow.

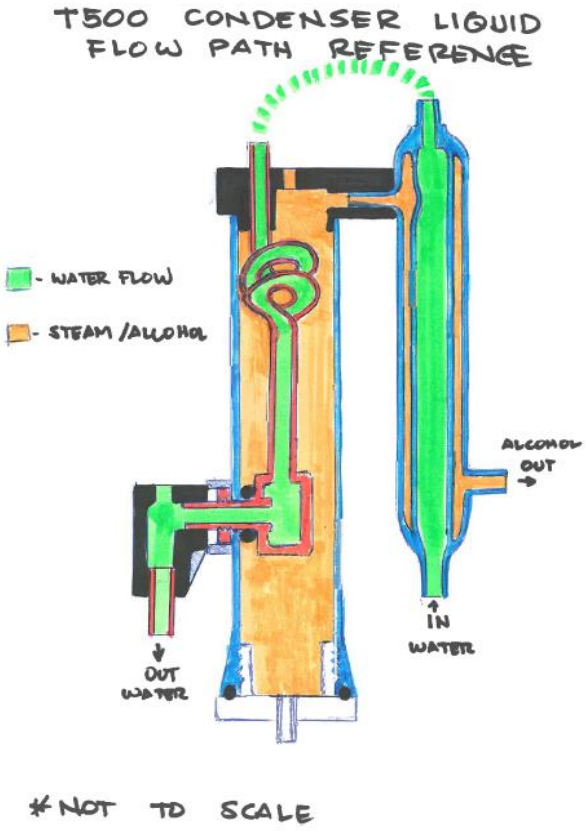

Figure 18 Sketch of water flow in the T500's column and condenser

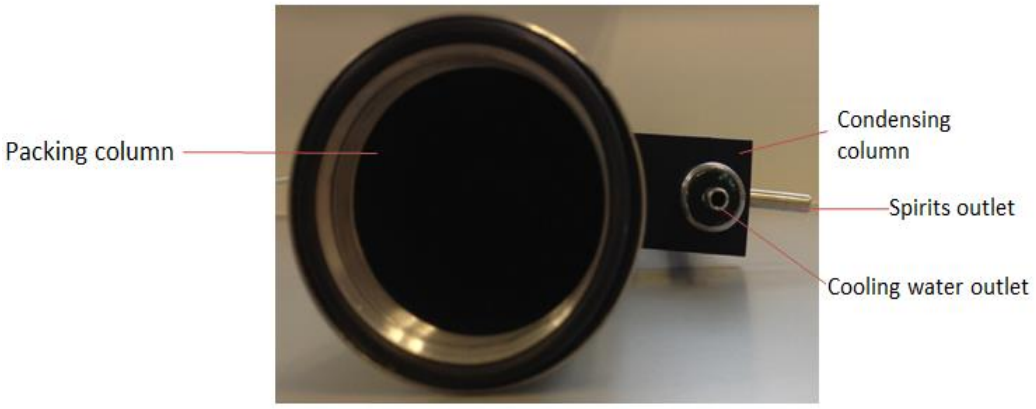

Figure 19 The graph of the looking up bottom of T500's column

The T500's column is arranged asymmetrically (Figure 19). To prepare for distillation, the T500 fractionating column – with an internal volume at around about 1100 mL – is inverted and filled first with 100 g of copper 'saddles' (so named by their shape) (Figure 2.3) (around 490 mL), with the remaining volume filled with a neutral (non-reactive) ceramic saddles (1 kg) (15mL) of unknown composition (Figure 20). Inspection of the ceramic saddles under a dissecting microscope suggested that these ceramic saddles were non-porous. Together these objects are the fractionating 'plates'.

(1) (2)

(3)

Figure 20 Ceramic saddles (1) and copper saddles (2) and representative single pieces (3)

As shown in Figure 17 there are two temperature sensors. There is the column sensor at the top of the column, and the cooling water outlet sensor at the base of the condenser. The sensor probes lead to two digital thermometers. Cooling is governed by two factors: the rate of cooling water flow and its temperature. The rate of cooling water flow is governed by a useful valve supplied with the still that fits domestic taps.

Effect of ethanol concentration on still performance

For home distillation the concentration of alcohol in the wash can range from typically 5 to 20%, although the latter concentration would be rare. A more typical range would be 8 to 15%. To cover all these possibilities it was decided to explore the performance for 5, 10, 15 and 20% alcohol. According to T500's instruction book, the cooling water outlet temperature of should be between 60 and 65°C, which can be obtained with a flow rate around 400 mL min^{-1}. This cooling condition is claimed to produce high quality spirits. However, for the purpose of comprehensive still evaluation, two other cooling temperatures were chosen: 50 to 55°C and 75 to 80°C (with matched approximate cooling water flow rates of 300 mL min^{-1} and 550 mL min^{-1}. The choice of these conditions was however imperfect – as will be shown – because tap water temperature varied with season. Thus this section describes a factorial experiment of four alcohol concentrations and three flow rates.

Materials and methods

Ethanol

Labserv^{TM} ethanol (99.5%) was obtained Thermo Fisher Scientific, Auckland. The declared density was 0.7876 g mL^{-1} (25°C).

Wash preparation

Nominally pure ethanol (99.5% ethanol) was diluted by tap water to exactly make 5, 10, 15 and 20% ethanol washes.

Factorial distillations

The date and tap water temperature were recorded before the distillation was started.

The boiler was filled to the 23.5 L mark, and the packed column was assembled on the boiler body by fastening the four clips. Cooling water is required to flow through the condenser before heating begins. During distillation, the temperatures of column and cooling water outlet were recorded in every two minutes, and the water flow rates were adjusted to ensure the cooling water temperature was in three required ranges. Thus, maintenance of the cooling

water outlet temperatures was the priority. The distillate was collected in fractions of 62 mL[5], 125 mL, and then 250 mL, 250 mL etc. by cylinders (aluminum foil covered covering the top until no further distillate was recoverable. As noted in Chapter 2 collections into measuring cylinders were always done. However, an ester note could often be smelt.

Density calibration curve of ethanol-water mixtures

Eleven standard solutions with ethanol concentrations from 0, 9.95 to 99.5% were prepared. The density of each 5 mL standard solution was measured by mass. The density-calibration curve of eleven standard solutions at two temps was created in Excel, which also calculated the polynomial regression equations and the r^2 values (Appendix 1). These equations were used to determine the ethanol concentration in 5 mL aliquots of the many distillate fractions.

Calculating the still performance

The kinetics of column and cooling water temperatures, collected volumes and ethanol recoveries were calculated by simple functions in Excel. In the graphs used to present this data the scales have been chosen to allow valid comparisons between graphs of the same family. In some cases the graphs can appear odd, but in context they are not.

Effect of ambient cooling on still performance

To explore the effect of ambient cooling on T500 still's performance, a domestic fan was placed (room temperature was 22°C) (Figure 21). The fan was produced by Martec (Sydney, Australia) (35W full power).

[5] 62 mL is not half of 125 mL, but once this error was made on the first serious distillation run, it was maintained for consistency.

Figure 21 The fan experiment

The experiment was conducted by distilling a 15% ethanol wash with a 60 to 65 °C water outlet temperature. Twenty minutes after the first distillate drops appeared, the fan was turned on for 10 min to provide an ambient draft, and then was turned off for 10 min. This cycle was repeated to the end of collection. The column temperature and cooling water outlet temperatures were recorded every two min and the water flow rates were adjusted to ensure the outlet temperature was in the 60 to 65°C range. The distillate was collected in fractions of 62 mL, 125 mL, and then 250 mL, 250 mL etc. until no further distillate was recoverable.

Results and discussion

Temperature monitoring results

The temperature results are of more technical interest than practical interest; therefore were presented in Appendix 2.

Ethanol concentrations in distillates from three cooling temperature treatments

Figure 22 shows the ethanol concentrations in distillates from a 5% ethanol wash from three cooling water outlet temperatures ranges. The distillate from the 75 to 80°C treatment contained ethanol concentrations at around 82%, lower than that of the 60 to 65°C treatment (about 90%) and the 50 to 55°C treatment (about 92%).

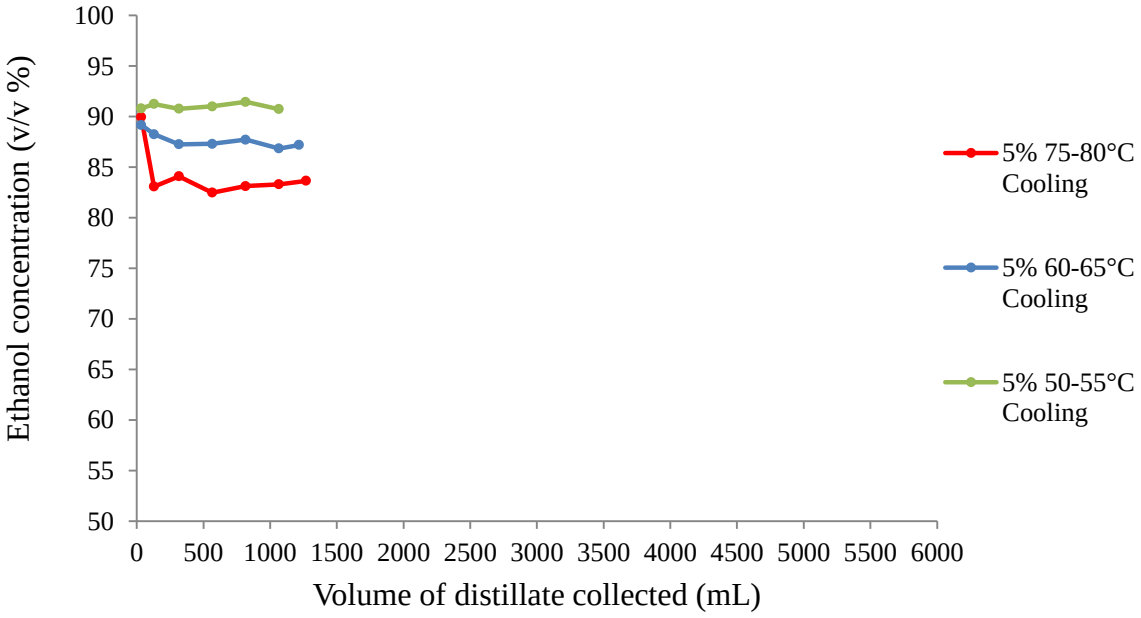

Figure 22 Ethanol concentrations in distillates of a 5% ethanol wash

According to the azeotrope theory of ethanol and water systems (Chapter 1.3), the hot vapour necessarily contains ethanol and some water. The hot vapours constantly experience vapour-liquid equilibrium stages in the T500's column, and are finally condensed and recovered by cooling. In this system, cooling water derives the heat from the hot vapour, thus resulting in changes to T500's column temperature. The cooling water temperature reflects the heat removing ability of T500's cooling system. If the cooling water outlet temperature is high, it means that the water is taking less heat out of the system than if the cooling were is cooler. (It is acknowledged that this is almost counter intuitive). Appendix 3 predicts that as cooling reduces (higher water outlet temperature) the proportion of ethanol in the vapour and therefore in the final distillate decreases, as Figure 22 shows. The differences between the column temperatures and the cooling water outlet temperatures are other way of looking at this phenomenon. In the graph of 5% ethanol that matches Figure 22, it can be seen that the temperature difference is much greater with the higher flow rates that take heat out of the system, resulting in the higher ethanol concentrations for the 60 to 65°C and 50 to 55°C treatments.

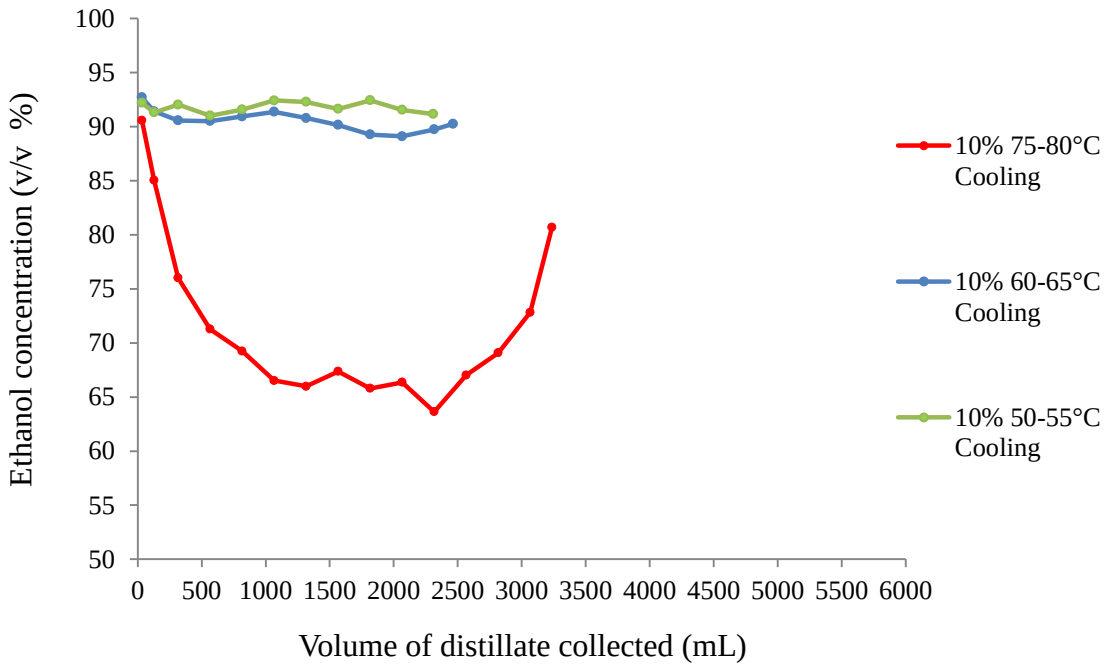

Figure 23 Ethanol concentrations in distillates of a 10% ethanol wash

Figure 23, Figure 24 and Figure 25 show the equivalent graphs for 10, 25 and 20% ethanol washes, all scaled the same way for comparison. The same general patterns were observed, except that the volume of distillate recovered obvious increased in increasing ethanol concentration in the wash. However, inspection of the curves for the four concentrations shows greatest variability with distillate volume collected in the 75 to 80°C treatments. Why is this? There is no obvious answer, but it is clear that the differences between the two temperatures were minimal and least for that treatment (Appendix 2). The still appears to be operating outside the range for it to successfully fractionate the two solvents and I was witnessing an uncontrolled boil.

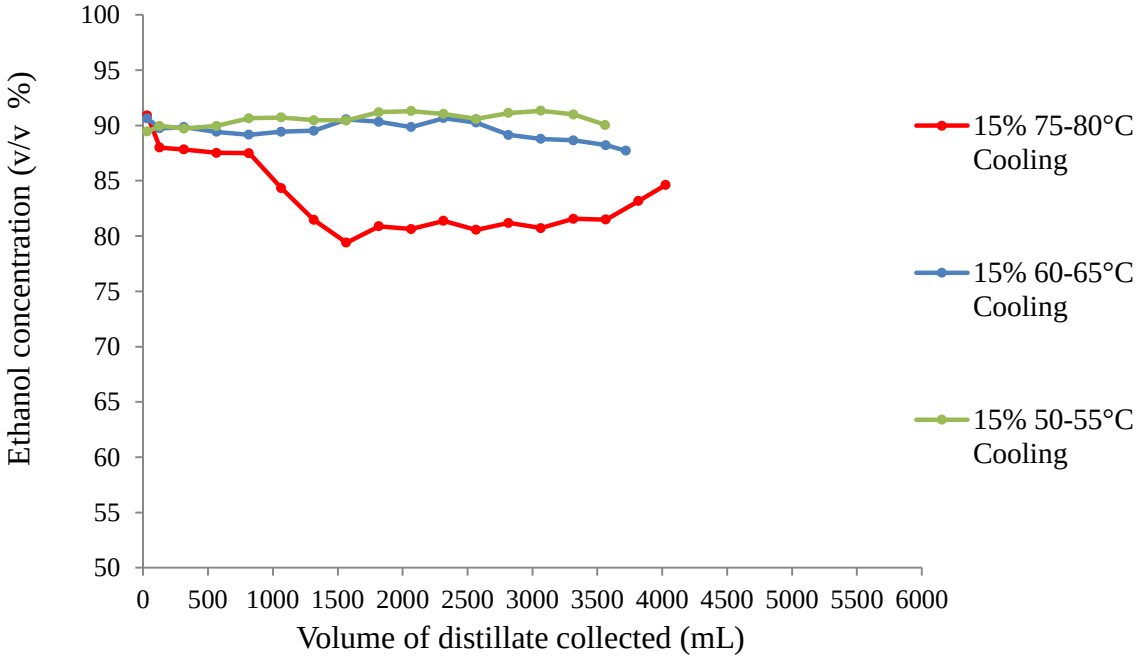

Figure 24 Ethanol concentrations in distillates from a 15% ethanol wash

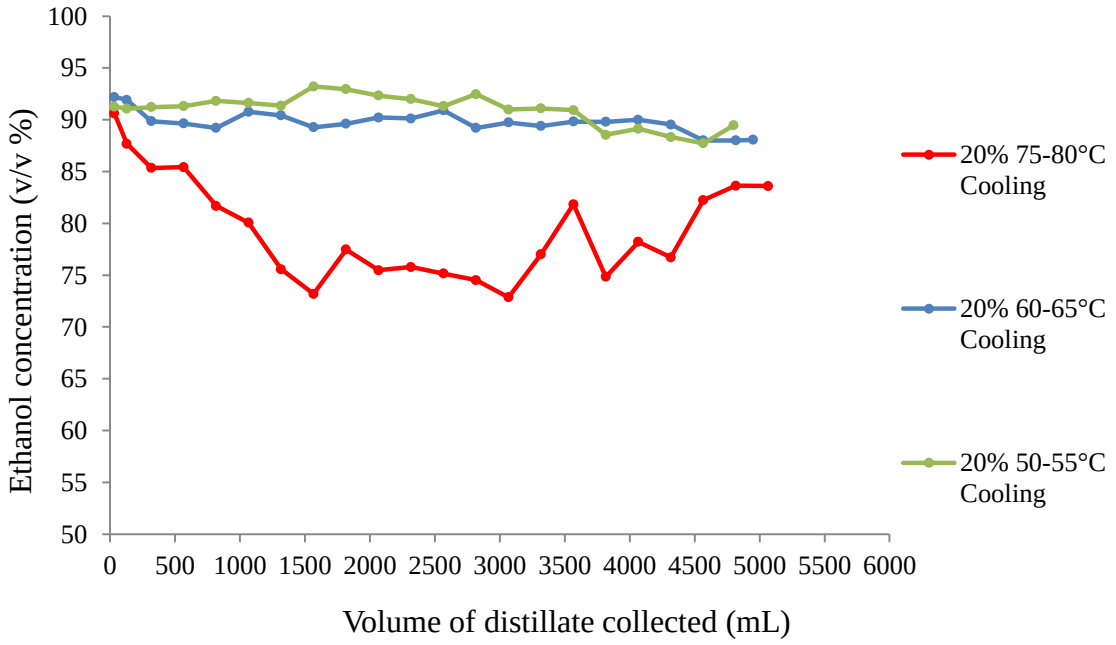

Figure 25 Ethanol concentrations in distillate from a 20% ethanol wash

All these results can be summarised as follows: Comparing the results of Figure 22 to Figure 25, the ethanol concentrations were similar at around 90% in distillates from the 50 to 55°C and 60 to 65°C cooling treatments. Ethanol concentrations in distillates from the 75 to 80°C cooling treatment were markedly lower, never exceeding 90% and dropping as low as 64%, the latter value in the 10% wash.

Kinetics of distillate collection volume

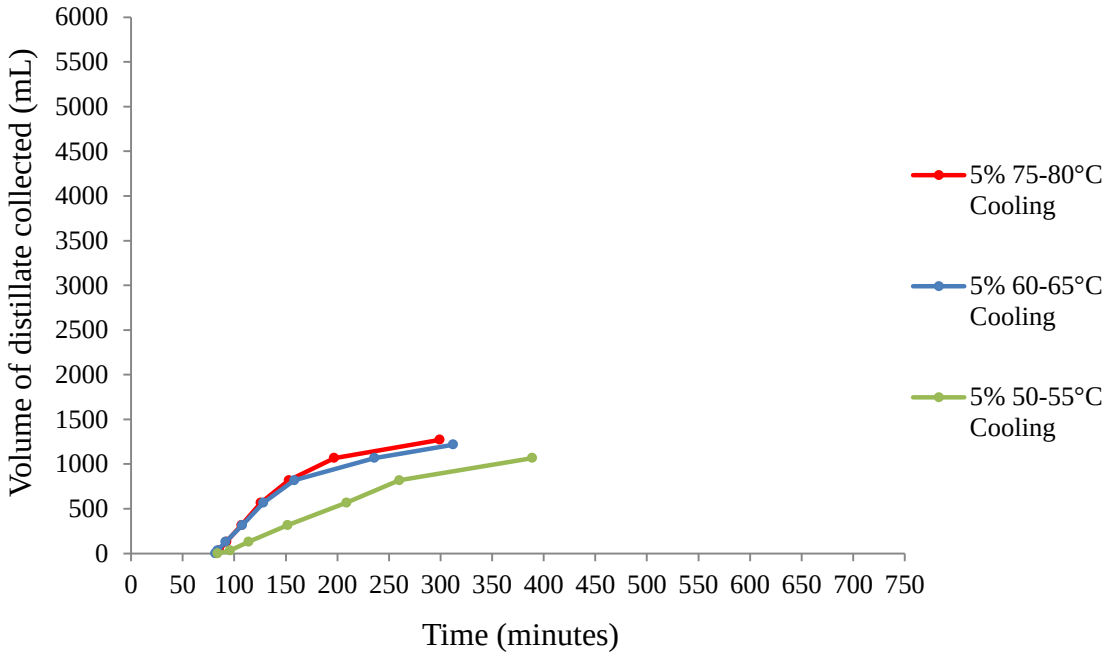

Figure 26 Kinetics of distillate volume from a 5% ethanol wash at three cooling temperatures

Figure 26 shows the kinetics of distillate volume from a 5% ethanol wash at three cooling water outlet temperatures. Although not obvious from the graph scaling, the 50 to 55°C treatment produced a total distillate volume of 1066 mL, around 180 mL less than that of the other two treatemnts (60 to 65°C treatment = 1218 mL; 75 to 80°C = 1271 mL). This was because the higher water outlet cooling water temperature condensed more water into the distillate, thereby increasing the total collected volume.

As expected, the more effective the cooling (lower cooling temperature) the longer the T500 took to collect the distillate. Thus the 75 to 80°C cooling treatment took 299 min, increasing to 389 min for the best cooled treatment, 50 to 55°C. This was because the 50 to 55°C treatment increased the heat removing effectiveness of the T500's condensing system, thus lengthening the distillate condensation time.

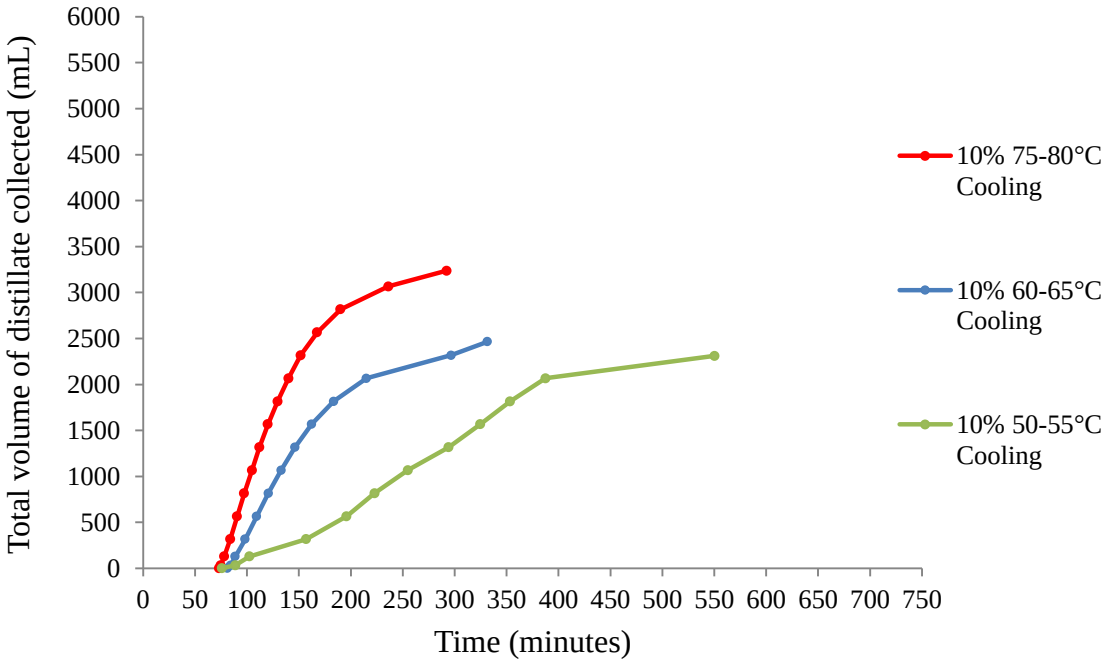

Figure 27 Kinetics of distillate volume from a 10% ethanol wash at three cooling temperatures

Figure 27, Figure 28 and Figure 29 show the equivalent data for the 10, 25 and 20% ethanol washes, all scaled the same way for comparison. As expected, the total volume collected increased with concentration of ethanol in the wash. As for the 5% wash, the more effective the cooling the longer it took to collect the distillate. Moreover, the toal volume recovered was greatest for the 50 to 55°C treatment. This was because all the ethanol was recovered along with increased volumes or water (Figure 22 to Figure 25). As comfirmation of this, the volumes collected were relatively closest in the 20% wash among three different cooling treatments.

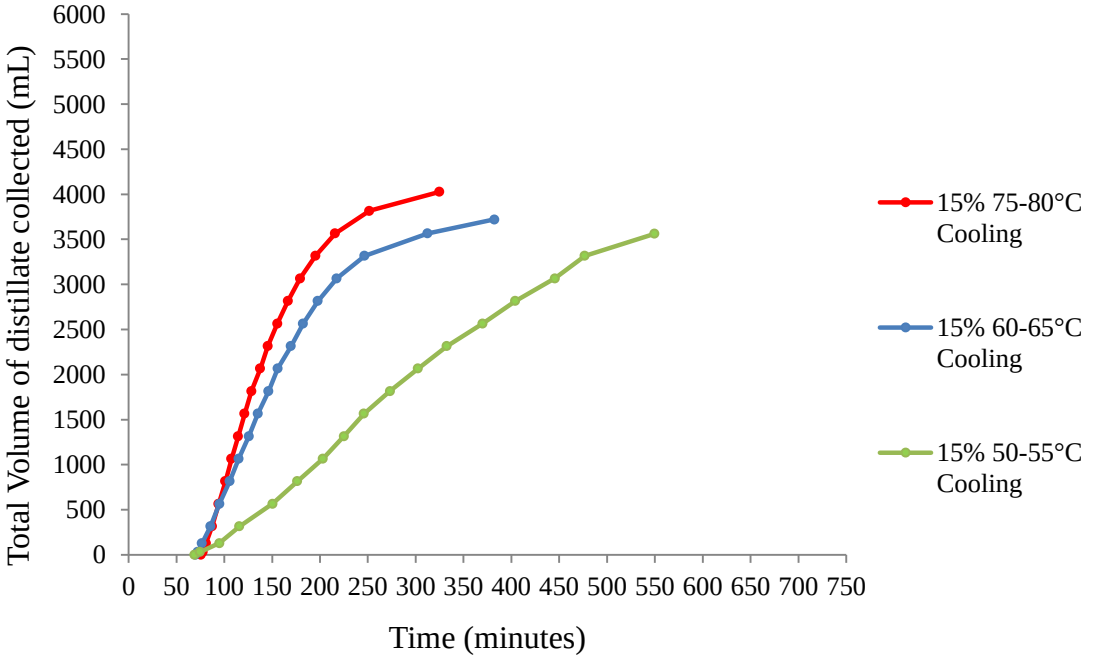

Figure 28 Kinetics of distillate volume from a 15% ethanol wash at three cooling temperatures

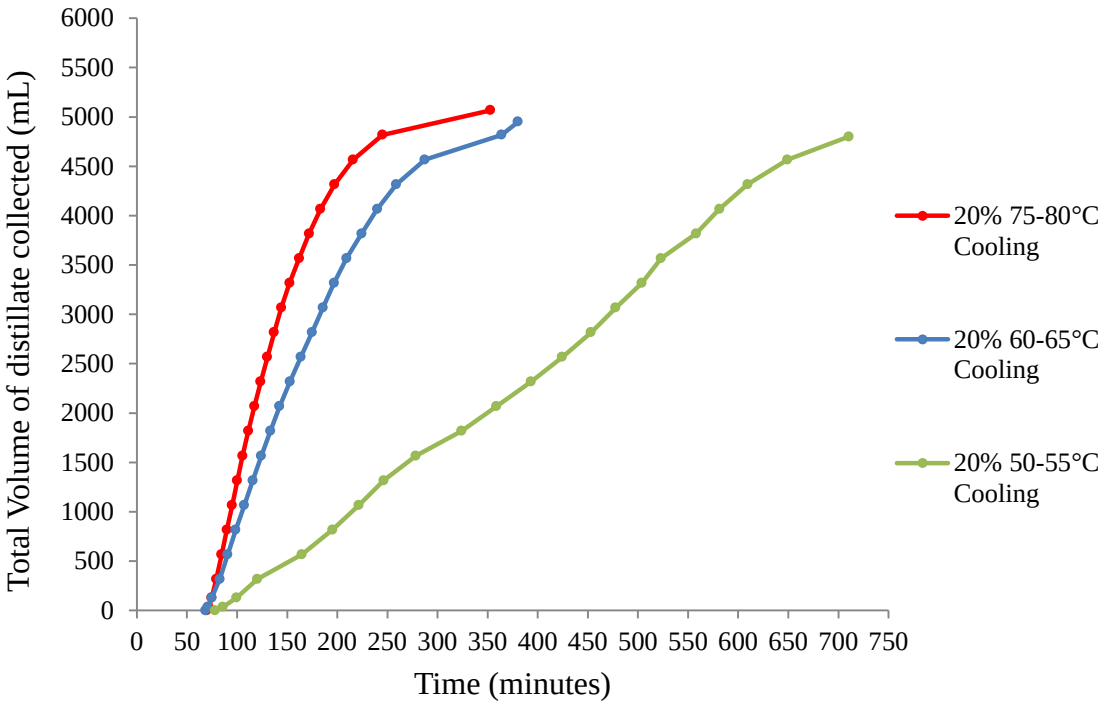

Figure 29 Kinetics of distillate volume from a 10% ethanol wash at three cooling temperatures

In summary, the higher the concentration of ethanol in the wash, the greater the total volume recovered. The more effective the cooling (50 to 55°C outlet temperature being the best) the lower the total volume collected, but the higher the ethanol concentration, as shown in the

previous section, Figure 22 to Figure 29. Overall these data presented no surprises, but provide a benchmark for imake.

Recovery of ethanol from three cooling treatments at four ethanol concentrations

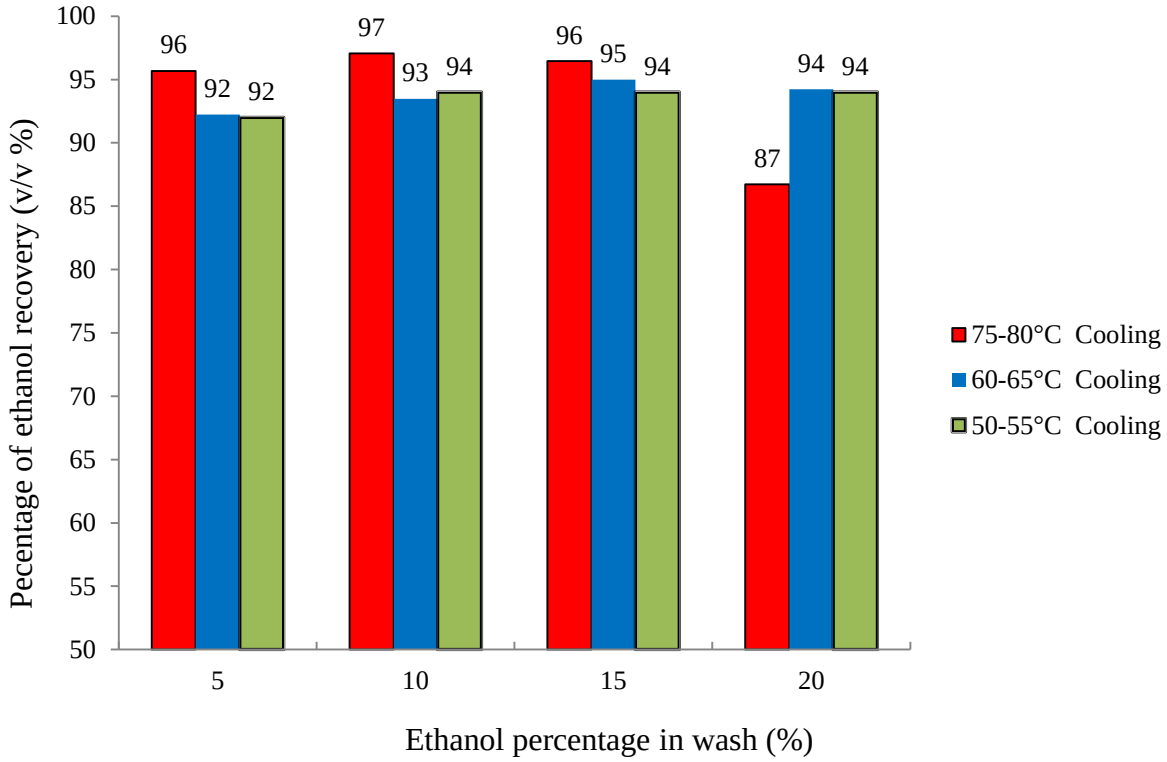

Figure 30 Ethanol recovery from the T500 still

Figure 30 shows the ethanol recoveries of the T500's 12 distillation experiments. Ethanol recoveries were similar with a minimum 92% for all distillations, except the 87% ethanol recovery from a 20% ethanol wash with the 75 to 80°C cooling treatment. The two cooler treatments – 60 to 65°C and 50 to 55°C – gave similar and consistent results. As discussed earlier, the 75 to 80°C cooling treatment resulted in the lowest ethanol concentrations in the distillate because it was operating out of its effective operating limits. However, the recovery was consistently the highest except in the 20% wash, because the column temperature was the highest, allowing more ethanol to be recovered.

Effect of ambient cooling on still performance

Although not formally reported in this thesis, the very earliest experiments showed aberrant results for the 15% ethanol wash. It was later realised that those distillations were exposed to a winter draft. Although the T500 is made of reflective stainless steel, it is otherwise non-insulated and has a large surface area relative for volume, particularly in the T500's column (comment: the column has a lot of gas phase, and the gas is easier to cool than a liquid). This experiment was designed to research the influence of a draft on the T500's performance.

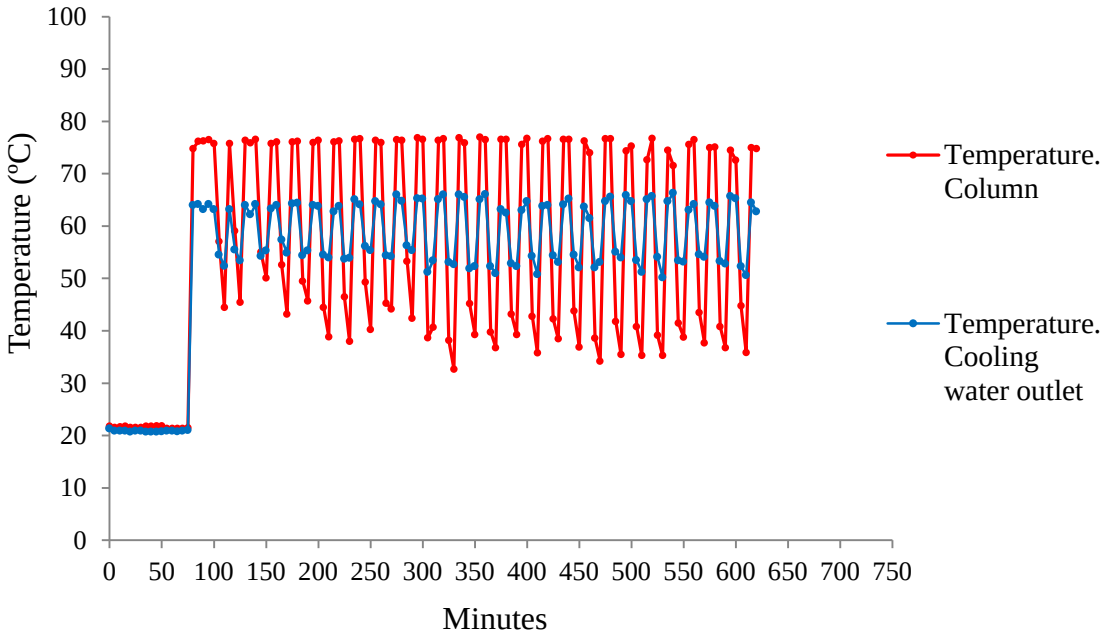

Figure 31 Temperatures changes with 15% ethanol wash in the draft .experiment, where the fan was alternately on and off for 10 min

Figure 31 shows the temperatures changes in response to the periodic cooling. The column temperature was markedly affected, with the largest difference being 43°C. The cooling water outlet temperature was less affected, but the draft caused it to routinely drop from the target range of 60 to 65°C to 50 to 55°C. These temperature changes affected the pattern of total volume recovered, as is clear from the staircase effect (Figure 32). The flow of distillate stopped gradually when fan was turned on and this resulted in an extension of collection time. The total collected volume from T500's draft distillation was 3762 mL, which was similar to the total collected volume (3721 mL) from T500's normal distillation (Figure 28). However, the distillation time was 605 min in the draft distillation, which was 223 min longer than the time taken in the equivalent normal distillation, 382 min (Figure 28). The ethanol concentrations in the recovered fractions were not measured, so ethanol recovery was not

calculated. However, since distillation continued until no more distillate was produced, it can be safely concluded that recover would be normal, that is greater than 92%.

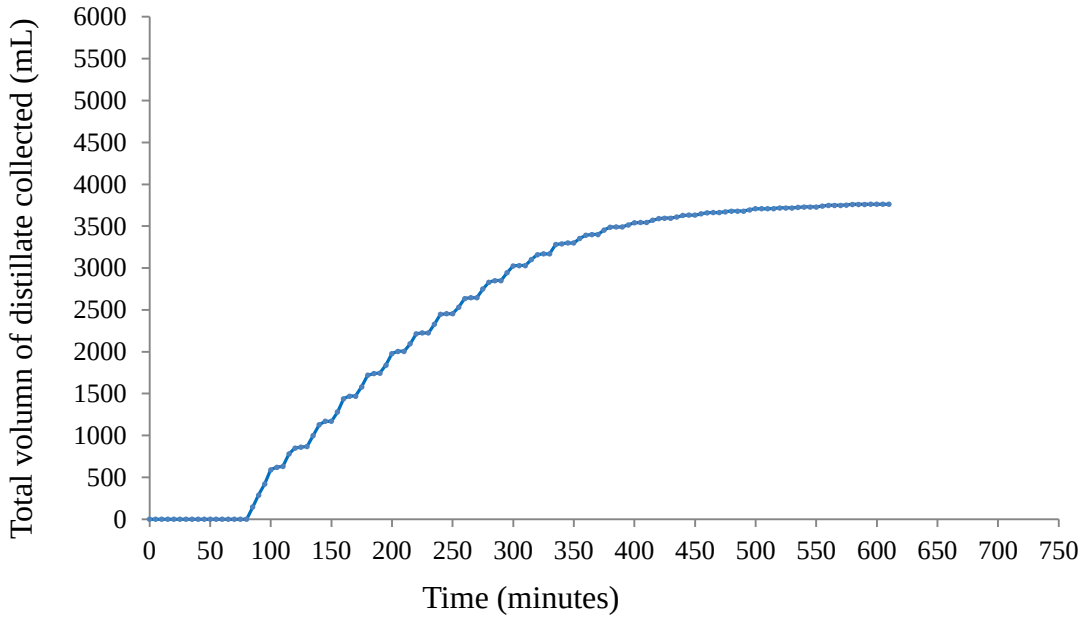

Figure 32 The kinetics of total distillate volume collected from a 15% ethanol wash with an initial 60 to 65°C water outlet temperature

In conclusion, the time taken to complete distillation was markedly increased by drafts, and effects of air movement in general may be responsible for some of the slightly variable curves shown in Figure 31 and Figure 32 for the coolest temperature treatment, 50 to 55°C.

Summary of results in this chapter

Comparing with the 50 to 55°C and 60 to 65°C cooling, the distillates from 75 to 80°C cooling treatment presented larger collection volume, faster collection time and higher ethanol recovery efficiency, but lower and unstable of ethanol contents in each fraction under same distillation conditions. The 50-55°C cooling treatments produced distillate with highest ethanol concentrations, but lowest collection volume and longest collection time. The distillates from 60 to 65°C cooling treatment contained higher and more stable ethanol concentrations at around 90%, although the other characters (volume, time and recoveries) were not as good as the 70 to 85°C cooling treatment. Therefore, the 60 to 65°C cooling treatment should be the recommended cooling treatment in distillation, which confirmed the cooling suggestion from imake. As the distillation performance of T500 still was remarkably affected by draft, imake customers are suggested to keep T500 still away from window or draft ambient when they do their home distillations.

In an ideal experimental situation, each of the distillations should be repeated to the extent of the likely variability between replicate distillations. However, each of the 12 distillations took typically three days to perform, record and analyse the data in a comparative way. To expect replication in the context of a one year Master thesis would be unrealistic given the scope of other work expected by imake. Therefore the graphs shown only represent single distillations. However, it turned out the results presented no surprises except in one case where an aberrant result was traced to distillation in a cool draft, and that itself spawned the confirmatory experiment (intermittent cooling) that was also described in this chapter.

Chapter 3

Performance of the essencia express still with binary mixtures of ethanol and water

Introduction

As noted in Chapter 1.5, the T500 and essencia stills are market competitor. imake was interested in comparing the performance of the two stills, whose main features are summarised in Table 5.

Table 5 Comparison of the T500 and the essencia stills

Property	T500	essencia	Comment
Nominal volume (L)	25	25	
Heating element (kW)	2.0	2.0?	Not identified for essencia but likely
Element in contact with wash	No	Yes	
Height of fractionation column (mm)	643	245	
Fractionation packing	Ceramic saddles plus copper saddles at top of column	Brass scourers	
Cooling within the fractionation column	An asymmetric vertical tube	A symmetrical collar	
Temperature monitoring	Column top and cooling water outlet, both digital	Column top with a conventional glass thermometer	

The structure of essencia still is shown in Figure 34. Similar to the T500 still, the essencia still composed of three main parts: the boiler, the fractionation column, and the condensor. The height of the column is 244.7mm, markedly shorter than the T500's column, 642.9mm.

The essencia's boiler contains a 2 kW (likely) heating element that is direct contact with the wash. This contrasts with the T500, where the similar element is beneath the boiler's base

and therefore not in contact. As for the T500, the essencia is also fitted with waste tap to discard the waste wash. The nominal total volume of both boiler tanks is 25 L.

The essencia's fractionating column is fitted with cooling collar that contrasts with the T500 column where cooling is achieved with a tube that descends to the cooling water outlet (Figure 33); it is arranged asymmetrically like the T500 column (Figure 19).

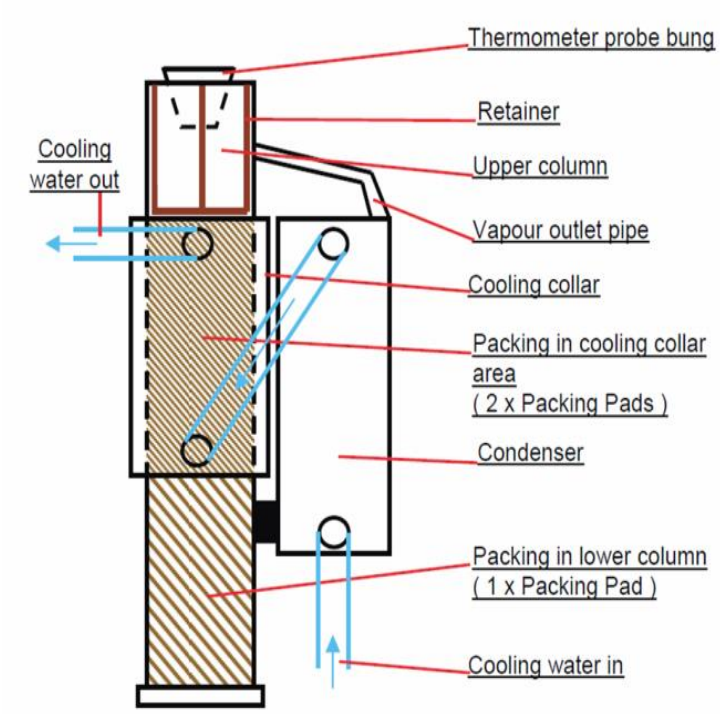

Figure 33 Sketch of water flow in essencia's column and condenser. (Source = essencia instruction booklet)

The essencia column is packed with three brass scourers similar to the familiar domestic Goldilocks (Reckitt Benckiser, Auckland) used in homes. As with the T500's ceramic lumps and copper saddles, the scourers serve two purposes, to fractionate volatiles and to trap sulphur compounds generated by fermentation and yeast autolysis. Temperature monitoring is restricted to column temperature by a conventional glass thermometer embedded in a rubber bung that fits into the top of the essencia column (Figure 33 and Figure 34).

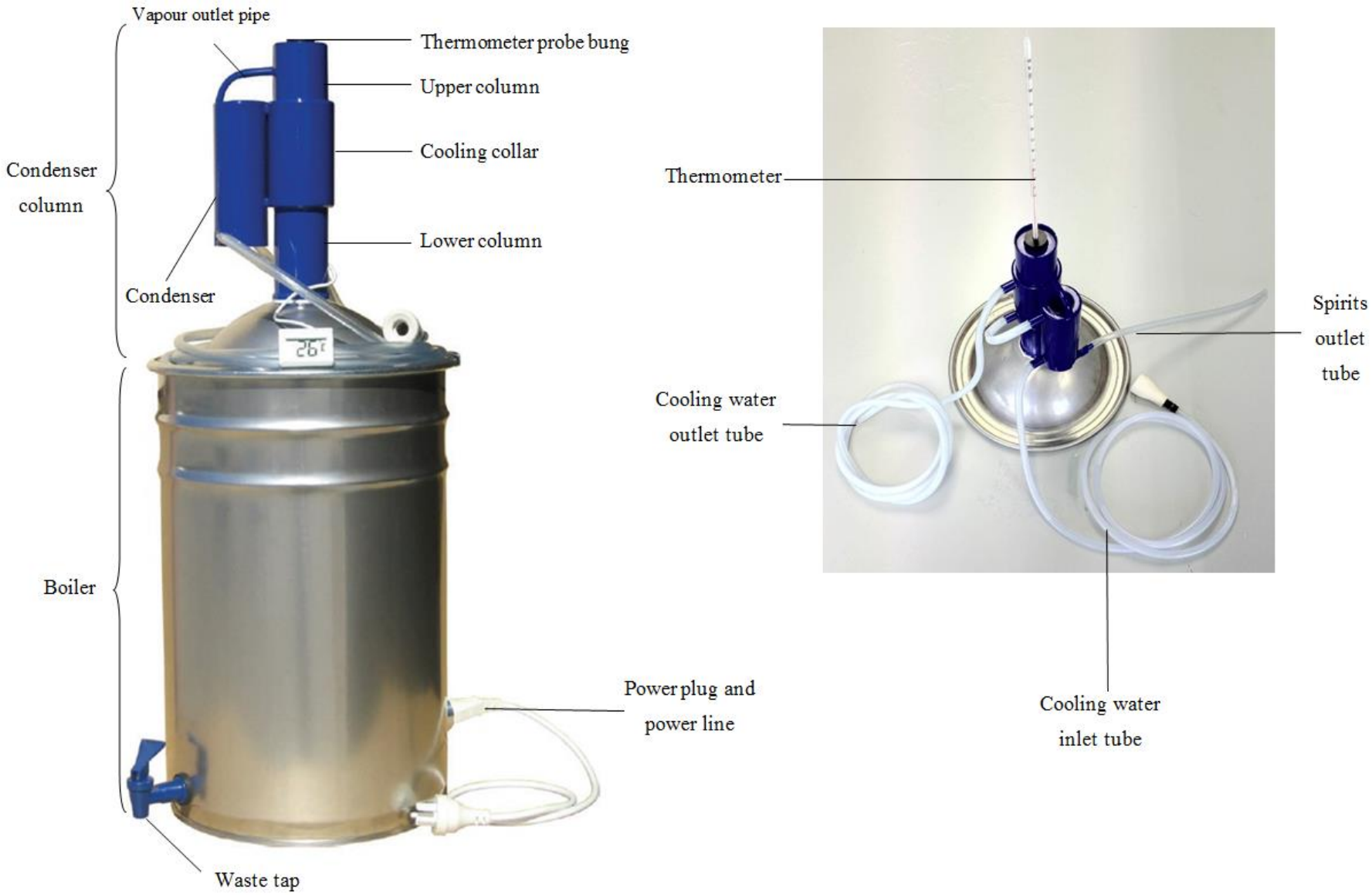

Figure 34 The essencia express still. (Source = essencia instruction booklet)

Materials and methods

In response to imake's suggestion, the distillation performance of essencia still was studied only by distilling a 15% ethanol wash with 50 to 55°C, 60 to 65°C and 75 to 80°C cooling water outlet temperatures. The cooling water temperatures were measured by a thermocouple wire that was linked to a digital thermometer. The thermocouple was inserted into the cooling water outlet tube, thereby monitoring the cooling temperature. The data obtained could be compared directly with the results obtained in Chapter 2, Figure 28.

Results and discussion for the essencia still

Temperature monitoring results

The temperature data of essenica still are presented in Appendix 4.

Ethanol concentrations in distillates from three cooling temperature treatments

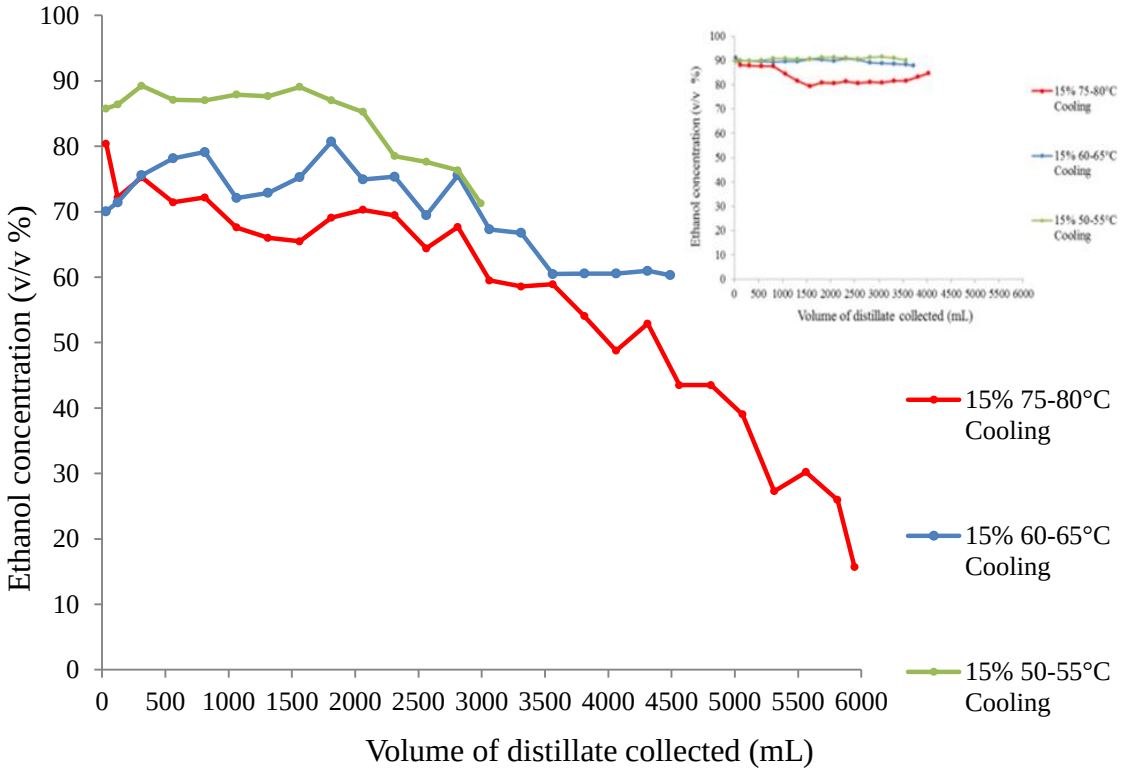

Figure 35 Ethanol concentrations in distillates from a 15% ethanol wash with the essencia still. The inset shows the comparable data from the T500 still

Figure 35 shows the ethanol concentrations in essencia's distillates from a 15% ethanol wash with three cooling water outlet temperatures ranges. Ethanol concentrations in the essencia distillate decreased steadily with increasing collection volume. Comparing this result with the equivalent T500 result (Figure 24) and a scale-matched inset in Figure 35, showed that the latter produced consistently higher ethanol concentrations.

Specifically, in 50 to 55°C cooling water outlet treatment, essencia produced distillates with ethanol concentrations ranging from 89 to 71%, which was lower than that of the T500 (around 90%); for the 60 to 65°C treatment, essencia ethanol concentrations ranged from 81 to 60%, again lower than that of T500 (around 90%). This theme continued the least effective cooling treatment – 75 to 80°C – where the ethanol concentrations in essencia's distillate dropped from 80 to 16%, larger decrease than that of T500 (from 91 to 79%). These results can be explained by reference to the column temperatures of the two stills in the two less effective cooling treatments – 60 to 65°C and 75 to 80°C. ; the essencia's column temperatures were higher than those of T500 still (Appendix 4), which distilled more water into distillate thus diluting the ethanol concentration in each fraction.

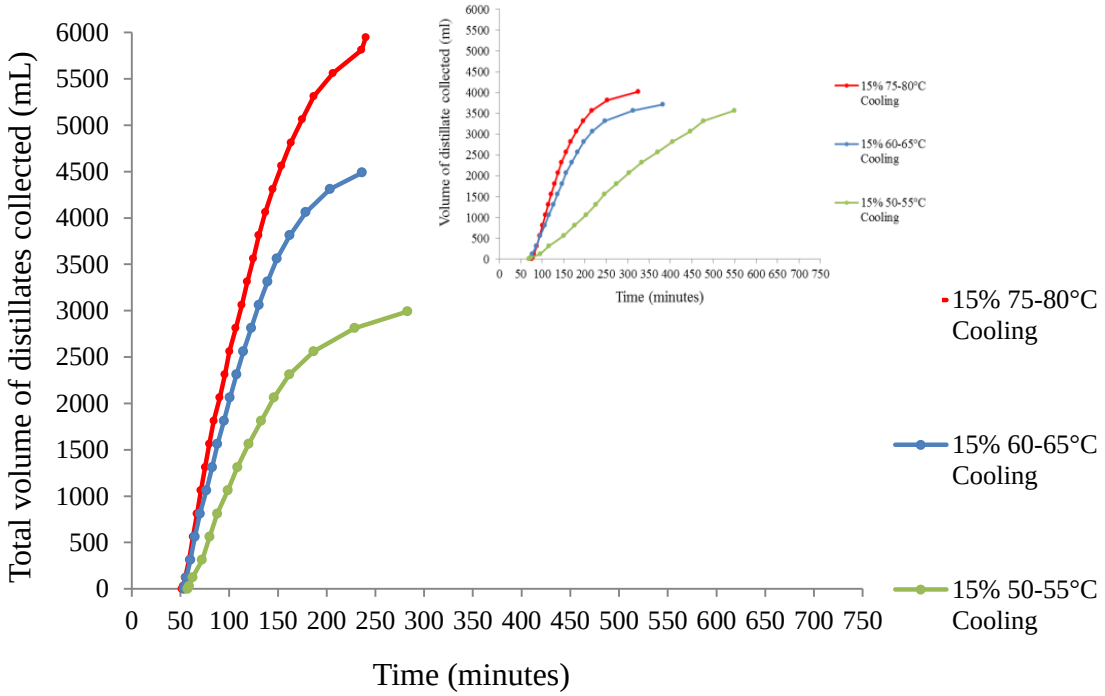

Figure 36 Kinetics of distillate volume from a 15% ethanol wash at three cooling temperatures, using the essenica still. The inset shows the comparable data from the T500 still, scaled identically.

Figure 36 shows the kinetics of essencia's distillate volume from a 15% ethanol wash with three cooling water outlet temperatures. Generally speaking, higher cooling temperatures (less effective cooling) produced larger distillate volume due to the increased water content in the distillate. This trend was much more obvious in the essencia still than the T500 (Table 6).

Table 6 Volumes of distillate produced by two stills with three cooling treatments. Values in parentheses in essencia data are the percentage differences from the T500 data

Cooling treatment	T500 (mL)	essencia (mL)
50 to 55°C (best cooling)	3563	2992 (-16)
60 to 65°C	3721	4500 (21)
75 to 80°C (worst cooling)	4029	5947 (48)

The column temperatures of essencia still were higher than those of the T500 still (Appendix 4) for the two less effective cooling treatments (6gtf0 to 65°C and 75 to 80°C) According to the azeotropic theory (Chapter 2), it can be deduced that the essencia should condense more distillate than the T500.

The total distillation times for essencia were similar for each cooling treatment (240 to 288 min), which were markedly shorter than those of the T500 under the same distillation conditions (325 to 550 min) (Figure 36 and Figure 28). Figure 37 summarises the ethanol recovery of the T500 and essencia stills from a 15% ethanol wash with three cooling temperature ranges. The ethanol recovery of essencia still was less than that of T500 still for all cooling treatments.

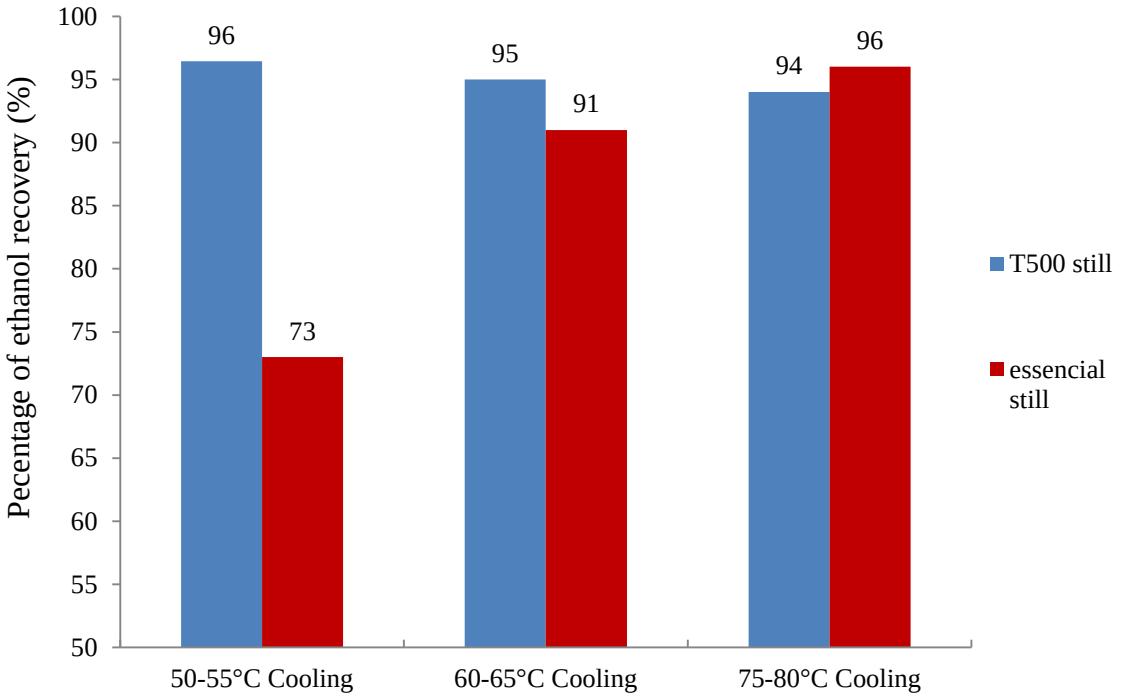

Figure 37 Ethanol recoveries from a 15% ethanol wash with the T500 and essencia stills

For the 50 to 55°C cooling treatment, the ethanol recovery for essencia was 73%, which was 23 percentage points less than that of the T500 (96%); for the 60 to 65°C treatment, the respective ethanol recovery was 91%, which was only 4 percentage points less than that of the T500 (93%); for 75 to 80°C cooling treatment, the ethanol recovery was about equal at 96 and 94%. Although ethanol concentrations are lower in essencia's distillates than that of T500, the total volume of distillates from essencia was larger (Figure 37); therefore, essencia still has higher ability to recover ethanol.

Summary of results in this chapter

The performance of essencia still was more affected by cooling temperatures than that of the T500 still, because larger difference existed in essencia's temperatures, ethanol concentrations and collected volume among different cooling treatments. With a 15% ethanol wash, the essencia still produced distillates with lower ethanol concentrations and slightly lower ethanol recoveries but faster collection time and larger collection volumes, particularly with less effective cooling (60 to 80°C range). Considering the more useful traits in distillates in terms of ethanol concentration and collected volume, the distillation performance of T500 still was more stable than that of essencia still.

Chapter 4

Calculation of theoretical plates for the Turbo 500 still and the essencia express still

Introduction

As mentioned in Chapter 1.3, the theoretical plate is a hypothetical stage of a vapour-liquid equilibrium in a fractionating distillation column, which represents the distillation efficiency of the still. In this chapter, the number of theoretical plates for the fractionating columns of T500 and essencial stills were calculated by the Fenske equation.

Materials and methods

Theoretical plates calculation

According to the Fenske equation (below), the external data for plates calculation are organized into Table 7. For each still, the data for plates calculation was obtained from a 15% ethanol wash with 60 to 65°C cooling water temperature; these conditions are as recommended by imake, and because the same cooling worked well for the essencia still (Chapter 3), this temperature range was used here. In each distillate fraction, the number of theoretical plates was calculated by equations in Excel. The final plates number were the average of all fractions.

$$N = \frac{\log\left[\left(\frac{X_d}{1-X_d}\right)\left(\frac{1-X_b}{X_b}\right)\right]}{\log\ \alpha_{avg}}$$

$\alpha_{avg} = \alpha$; (α can be got from the form of relative volatility (Appendix 5))

$X_d = N_E / (N_E+N_W)$; (N_E= mole of ethanol in distillate, N_W= mole of water in distillate)

$X_b = n_e / (n_e + n_w)$; (n_e = mole of ethanol in bottom wash, n_w= mole of water in bottom wash)

Table 7 External data used in theoretical plates calculations

Density of 99.5% ethanol ($g\ mL^{-1}$)	Density of water ($g\ mL^{-1}$)	Density of 15% ethanol wash ($g\ mL^{-1}$)	Molar mass of ethanol ($g\ mol^{-1}$)	Molar mass of water ($g\ mol^{-1}$)
0.788	1	0.984	46	18

Results and discussion

The calculations for the two stills are shown in Table 8 and Table 9.

Table 8 The factors and results of theoretical plates for the T500 still

Fraction Number	Collected vol. (mL)	Ethanol concentrations in distillates (% v/v)	Ethanol concentrations in wash tank (% v/v)	Column temperature (°C)	Mole fraction of ethanol X_d	Mole fraction of ethanol X_b	Relative volatility α[1]	Theoretical plates number
1	67.00	90.64	15.00	79.90	0.73	0.05	1.88	6.27
2	125.00	89.74	14.82	79.80	0.71	0.05	1.81	6.50
3	250.00	89.85	14.34	80.10	0.72	0.05	2.01	5.58
4	250.00	89.43	13.38	80.50	0.71	0.05	2.28	4.75
5	250.00	89.15	12.43	80.90	0.70	0.04	2.56	4.24
6	250.00	89.43	11.49	80.50	0.71	0.04	2.28	4.95
7	250.00	89.53	10.53	80.30	0.71	0.04	2.15	5.48
8	250.00	90.55	9.58	80.40	0.73	0.03	2.22	5.54
9	250.00	90.34	8.62	80.70	0.73	0.03	2.42	5.08
10	250.00	89.85	7.66	80.40	0.72	0.03	2.22	5.73
11	250.00	90.67	6.70	80.10	0.74	0.02	2.01	6.85
12	250.00	90.26	5.74	80.70	0.73	0.02	2.42	5.55
13	250.00	89.15	4.78	80.40	0.70	0.02	2.22	6.24
14	250.00	88.79	3.83	80.50	0.69	0.01	2.28	6.24
15	250.00	88.65	2.88	80.70	0.69	0.01	2.42	6.14
16	250.00	88.21	1.94	80.70	0.68	0.01	2.42	6.54
17	58.00	87.71	1.00	80.80	0.67	0.00	2.49	7.02
							Average plates number =	5.80

[1] α values were obtained from Appendix 5, by interpolating linearly between 79.8°C and 82.3°C, temperatures that covered the experimental range.

Table 9 The factors and results of theoretical plates for the essencia still

Fraction Number	Collected vol. (mL)	Ethanol concentrations in distillates (% v/v)	Ethanol concentrations in wash tank (% v/v)	Column temperature (C)	Mole fraction of ethanol X_d	Mole fraction of ethanol X_b	Relative Volatility α[1]	Theoretical plates number
1.00	67.00	70.06	15.00	80.00	0.40	0.05	1.95	3.81
2.00	125.00	71.45	14.89	80.00	0.42	0.05	1.95	3.92
3.00	250.00	75.56	14.51	80.00	0.47	0.05	1.95	4.28
4.00	250.00	78.17	13.71	80.00	0.51	0.05	1.95	4.60
5.00	250.00	79.12	12.88	81.00	0.52	0.04	2.62	3.30
6.00	250.00	72.10	12.03	81.00	0.43	0.04	2.62	2.99
7.00	250.00	72.88	11.27	80.00	0.44	0.04	1.95	4.50
8.00	250.00	75.28	10.49	80.00	0.47	0.03	1.95	4.81
9.00	250.00	80.72	9.69	80.00	0.55	0.03	1.95	5.42
10.00	250.00	74.93	8.83	80.00	0.46	0.03	1.95	5.06
11.00	250.00	75.35	8.04	80.00	0.47	0.03	1.95	5.25
12.00	250.00	69.47	7.23	80.00	0.40	0.02	1.95	4.97
13.00	250.00	75.56	6.49	80.00	0.47	0.02	1.95	5.60
14.00	250.00	67.30	5.69	80.00	0.37	0.02	1.95	5.21
15.00	250.00	66.77	4.98	80.00	0.37	0.02	1.95	5.38
16.00	250.00	60.47	4.26	80.00	0.31	0.01	1.95	5.22
17.00	250.00	60.56	3.62	80.00	0.31	0.01	1.95	5.48
18.00	250.00	60.56	2.98	80.00	0.31	0.01	1.95	5.78
19.00	250.00	60.96	2.33	80.00	0.31	0.01	1.95	6.18
20.00	106.00	60.31	1.68	80.00	0.30	0.01	1.95	6.64
							Average plates number	= 4.92

[1] α values were obtained from Appendix 5, by interpolating linearly between 79.8°C and 82.3°C, temperatures that covered the experimental range.

X_d and X_b were calculated from experimental results and the data in Table 7, where X_b showed the greatest changes as the ethanol concentration dropped in the wash tanks. Figure 38 summarizes the changes of theoretical plates during distillation by the T500 and essencia stills. The theoretical plates for the T500 still were higher than that of the essencia still, with the average plates number of 5.80 (Table 8) and 4.92 (Table 9) respectively. The generally steady increase can be traced to the increasing ability of the stills to produce highly ethanolic distillates from increasingly dilute concentrations of ethanol in the washes. Inspection of the two curves in Figure 38 shows sharp drops early in the distillations. These can be explained by small changes in column temperature that markedly affect volatility, the log of which is a divisor in the Fenske equation. The changes of column temperature may be caused by the draft ambient in the room (as mentioned in Chapter 2, the fan experiment). The performances of the stills are acutely sensitive to ambient temperature changes. Therefore, the operating stills should be kept away from drafts.

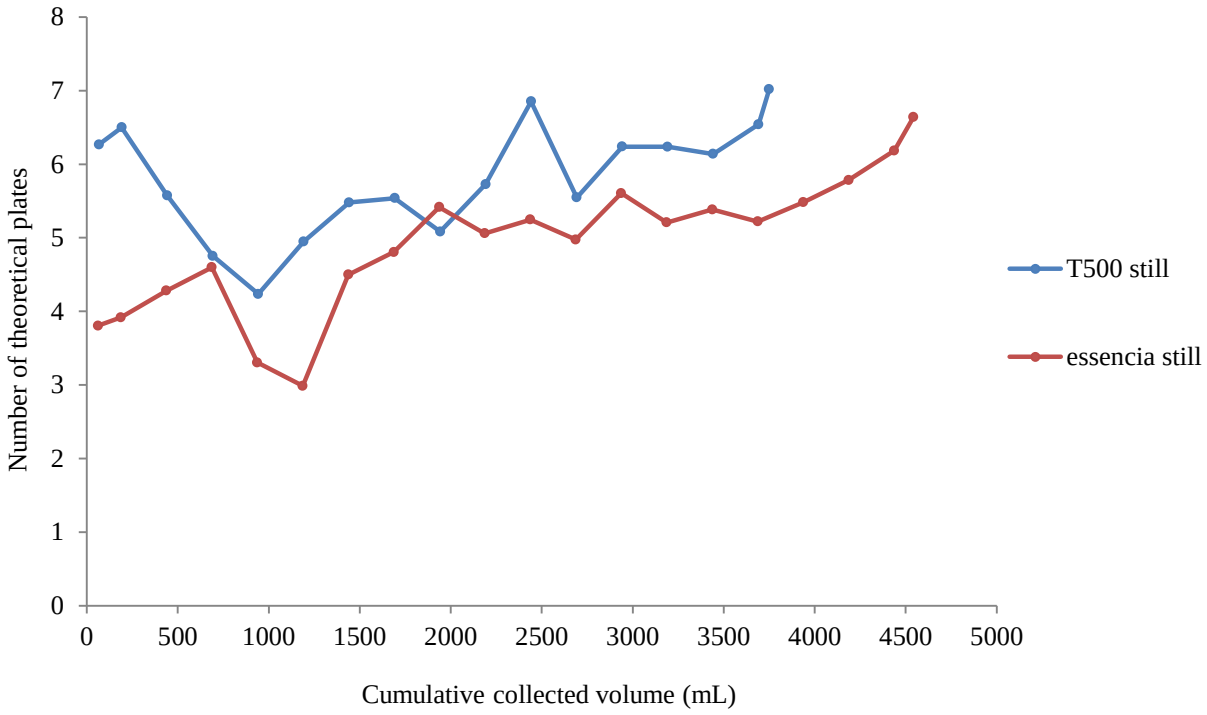

Figure 38 The changes of theoretical plates during distillation by the T500 and essencia stills

Summary of results in this chapter

According to the theory of fractionating distillation (Chapter 1.3), the more theoretical plates there are, the better the separating performance during distillation. As the number of theoretical plates for the T500 was greater than that of the essencia, this probably explains why the T500 still produced distillate with higher ethanol concentrations than that of essencia still under the same distillation conditions (Figure 35). However, it must be emphasised that the choice of 60 to 65°C cooling was arbitrary for the essencia still, and the plates' calculation will vary at other temperatures. Nonetheless, the fact remains that the T500 outperformed the essencia in terms of ethanol recovery.

Chapter 5

Performances of T500 and essencia stills with a synthetic wash

Introduction

Fermented wash contains numerous alcoholic congeners, which are considered largely unwanted congeners (Chapter 1.2). The ability of the stills to fractionate congeners is another parameter to determine with the stills. The aim in this chapter is to test these fractionating abilities. This experiment was designed to distil a synthetic wash, which was made of seven common known fermentation congeners in a 15% ethanol wash. The concentrations of seven congeners were examined by gas chromatography (GC), thereby showing the fractionating ability of the two stills.

Materials and methods

Congeners and their concentrations

The formulation of congeners was obtained from the imake company. Seven congeners were added into a 15% ethanol wash to create the synthetic wash (Table 10).

Table 10 The composition of the synthetic wash

Chemicals	Percentage (v/v %)
Water (H_2O)	84.96
Ethanol (C_2H_6O)	15.00
Acetaldehyde (CH_3CHO)	0.001
Ethyl acetate ($CH_3COOCH_2CH_3$)	0.01
Methanol (CH_4O)	0.005
Propan-1-ol ($CH_3CH_2CH_2OH$)	0.003
2-Methylpropan-1-ol (Iso-butanol) ($(CH_3)_2CHCH_2OH$)	0.015
2-Methylbutan-1-ol (Active amyl alcohol) ($C_5H_{11}OH$)	0.005
3-Methylbutan-1-ol (Iso-amyl alcohol)($C_5H_{11}OH$)	0.005

Distillation

The synthetic wash was filled into boiler at 23.5 L mark, and the packed column was assembled on the boiler body by fastening the four clips. Cooling water is required to flow through the condenser before heating begins. During distillation, the water flow rates were adjusted to ensure the cooling water temperature in 60-65°C range. Thus, maintenance of the cooling water outlet

temperatures was the priority. The distillate was collected 50 mL in first 20 fractions, and then 100mL in each continuing fractions, by cylinders (aluminum foil covered covering the top until no further distillate was recoverable.

Gas chromatography

1-Butanol was the internal standard. It was added to distillate fractions at the rate of 1% (v/v). Distallate samples were injected into a Shimadzu GC which was equipped with a flame ionization detector (FID). The GC column was a 30 metre Zebron-Wax-Plus column with 0.25 mm inner diameter and 0.25 μm film thickness. Each 0.5 μL sample of distillate containing the internal standard was injected at a split ratio of 1:100. The column injector temperature was set at 210°C. The carrier gas was hydrogen at a flow rate of 112 mL min^{-1}. Nitrogen was used as the make-up gas, which was delivered at a rate of 28 mL min^{-1}. The hydrogen and air flow rates in the FID were set at 46 mL min^{-1} and 480 mL min^{-1}, respectively. The column temperature was first held at 45°C for 4 min, then increased to 55°C at the rate of 10°C per min, at which it was held for 8 min. The detector temperature was 250°C. The method was referenced by Countryman and Kelly (2006).

Data analysis

The results were calculated in Microsoft Excel 2010. The concentration of each congener in fractional distillate samples were calculated by the following formula:

$$\text{Concentration of congener} = \frac{\text{Peak area of internal standard}}{\text{Peak areas of congener} * \text{concentration of internal standard}}$$

Results and discussion

Appendix 6 shows a typical GC trace of the congeners and the internal standard.

Total recoveries of seven congeners for T500 and essencia stills

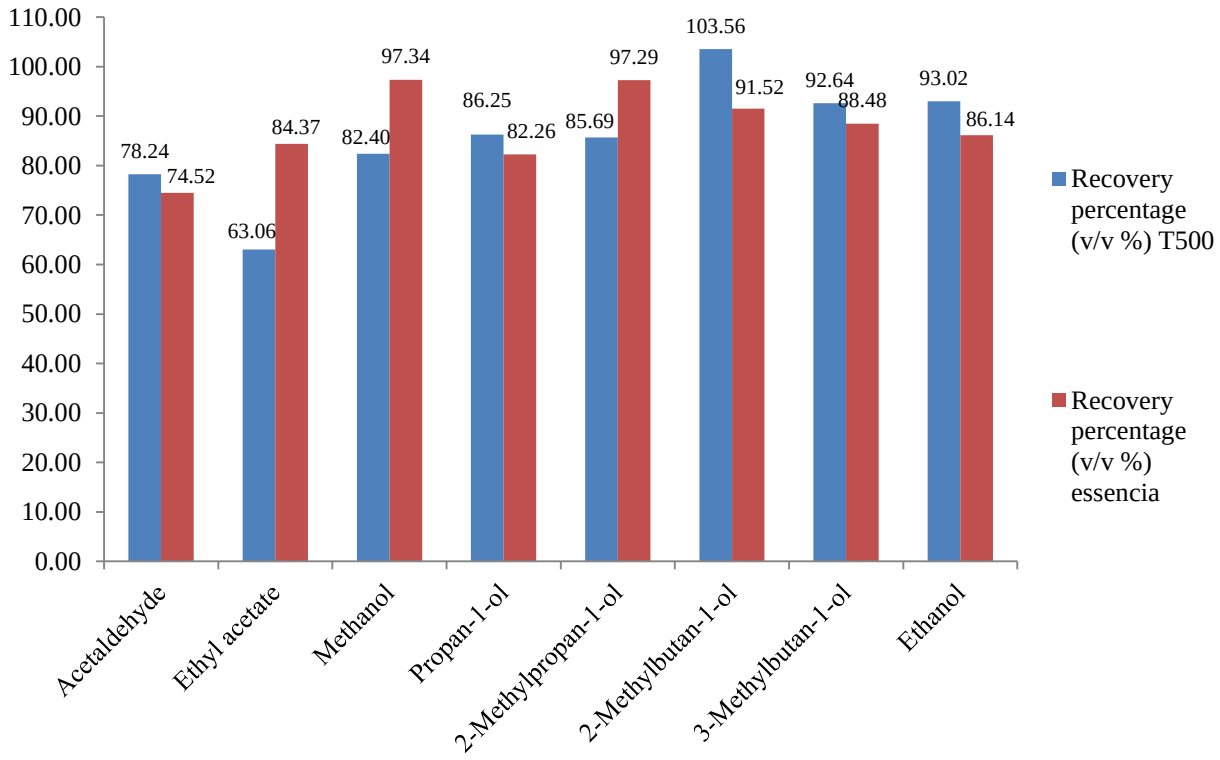

Figure 39 Total recoveries of seven congeners and ethanol from a 15% ethanol wash

Figure 39 shows that the recoveries of eight components are above 82% from both stills, except for ethyl acetate (63.1% from T500) and acetaldehyde (78.2% from T500 and 74.5% from essencia). The volatilities of acetaldehyde and ethyl acetate are higher than those of the other compounds, and may be lost during distillate collection. As noted in Chapter 2 collections into measuring cylinders were always done with aluminum foil covering the top. However, an ester note could often be smelt.

On average, the T500 had higher recovery abilities than that of the essencia. Specifically, the T500 had higher recoveries of five components: acetaldehyde, propan-1-ol, 3-methylbutan-1-ol, 2-methylbutan-1-ol and ethanol, with 4, 4, 12, 4 and 7 percentage points improvements, respectively; while the essencia has higher recoveries on only three components: ethyl acetate, methanol and 2-methylpropan-1-ol, with percentage point improvements of 21, 15 and 12 respectively. The reasons for these results are required further research**.**

Figure 39 shows the overall recovery and if the home distiller elected to produce a spirit that comprised all the distillate, then from a practical perspective the differences in final spirit flavor due to these recovery differences would be minor, and probably undetectable to the consumer. However, if the distiller elected to reject certain distillate fractions, then the fraction in which the congeners were collected would be important if they fractionated at different times during distillation. This was studied by plotting the percent of total recovery against cumulative midpoint volume.

Kinetics of recovery of eight components for T500 and essencia stills

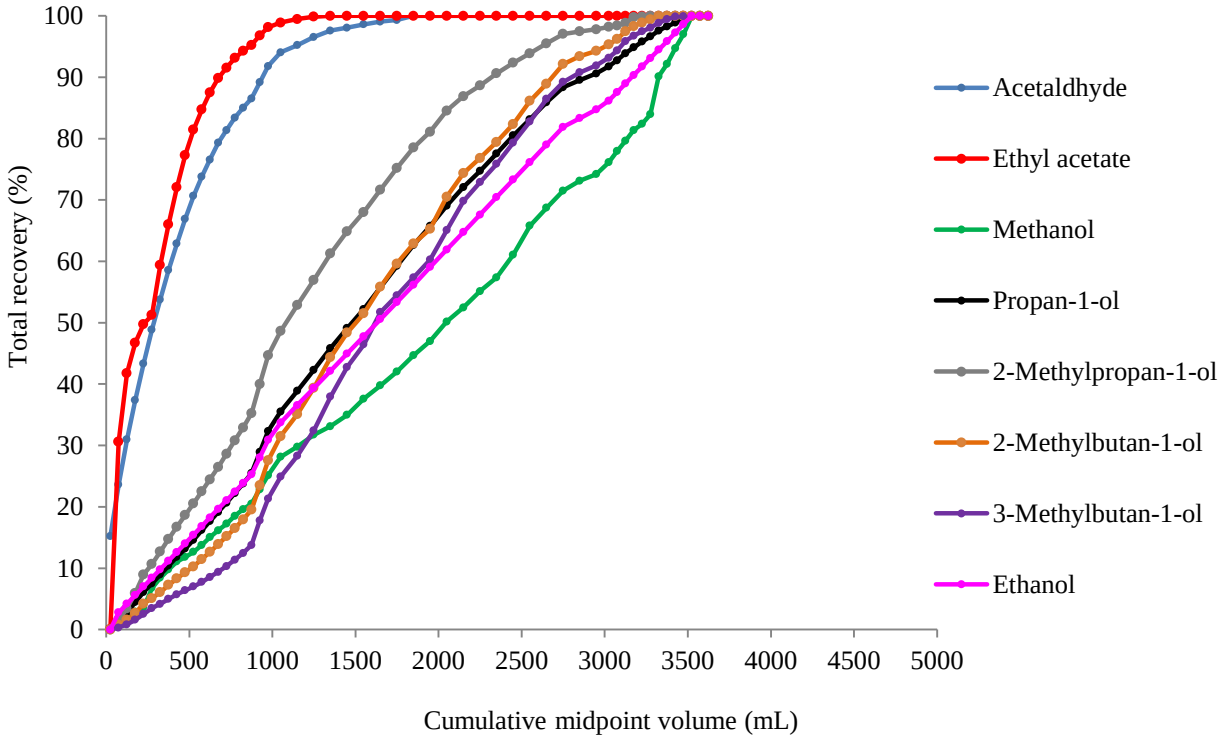

Figure 40 The kinetics of recovery of eight components from a 15% ethanol wash in the T500 still

The ethyl acetate and acetaldehyde reached their maximum recoveries at about 1350 mL and 1750 mL collected volumes, which were much earlier than those of 2-methylpropan-1-ol, 3-methylbutan-1-ol , 2-methylbutan-1-ol, propan-1-ol and methanol, all of which were maximally recovered after about 3325 mL (2-methylpropan-1-ol was the earliest of these). Under the temperature and other fractionating conditions used here, the volatilities of ethyl acetate and acetaldehyde were clearly higher than those of the others.

The recovery curves of methanol, 2-methylpropan-1-ol, propan-1-ol, 3-methylbutan-1-ol and 2-methylbutan-1-ol were closer to the ethanol recovery curve, which means these five congeners were would be difficult to remove from ethanol with the T500 still.

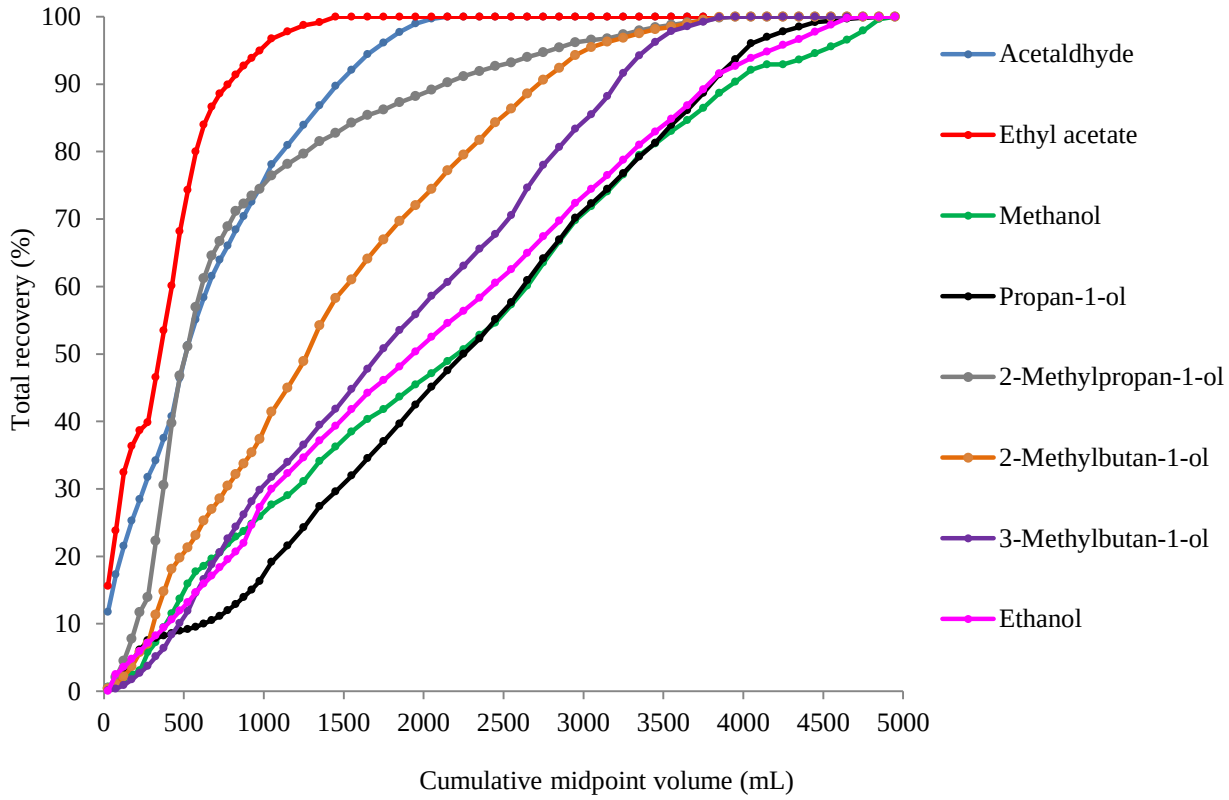

Figure 41 The kinetics of recovery of eight components from a 15% ethanol wash in the essencia still

The essencia result was more complex. Ethyl acetate and acetaldehyde reached their maximum recoveries at 1450 mL and 1950 mL collection volumes, which was earlier than those of the other cogeners. 2-methylpropan-1-ol was again the earliest at 3950 mL, and was the same as that for 3-methylbutan-1-ol.

The essencia's recovery curves of methanol, propan-1-ol, 2-methylpropan-1-ol, 3-methylbutan-1-ol and iso-amyl alcohol were more scattered from the ethanol recovery curve when compared with the equivalent curves of the T500 still. The result suggested that congeners might be better fractionated by the essencia than by the T500, in spite of the lower theoretical plates value (5.8 versus 4.92).

While that may be so, in practical terms, only a small volume of distillate should/could be discarded, because otherwise too much ethanol would be lost. It is therefore important to look at

the data another way by asking the question: what congeners are more or less removed if volumes were discarded, focusing on discarding early distillate (Table 11).

Table 11 Proportions of congeners and ethanol lost in three hypothetical discard volumes

Discard the first 75 mL	Percentage removal of total recovered	
Component	T500	essencia
Acetaldehyde	24	17
Ethyl acetate	31	24
Methanol	1	1
Propan-1-ol	1	1
2-Methylpropan-1-ol	2	2
2-Methylbutan-1-ol	1	1
3-Methylbutan-1-ol	0.3	0.4
Ethanol	3	2
Discard the first 175 mL		
Component	T500	essencia
Acetaldehyde	37	25
Ethyl acetate	47	36
Methanol	3	2
Propan-1-ol	4	4
2-Methylpropan-1-ol	6	8
2-Methylbutan-1-ol	3	4
3-Methylbutan-1-ol	2	2
Ethanol	6	5
Discard the first 475 mL		
Component	T500	essencia
Acetaldehyde	67	46
Ethyl acetate	77	68
Methanol	12	14
Propan-1-ol	13	9
2-Methylpropan-1-ol	19	47
2-Methylbutan-1-ol	9	20
3-Methylbutan-1-ol	6	10
Ethanol	14	12

Acetaldehyde and ethyl acetate can be most removed from the early distillate than that of the other congeners. Discarding the first 75 mL would remove 24 and 17% of the acetaldehyde, 31 and 24% of the ethyl acetate by the T500 and essencia stills, respectively, but lower percentages (below 3%) for the other compounds (Table 11). Thus, a first-75 mL discard from the T500 was more effective in removing acetaldehyde and ethyl acetate than the same discard from the essencia still. Discarding the first 175 mL was predictably more effective that a 75 mL discard, but the losses of other congeners and the desired ethanol were all less than 8%. A 475 mL discard was again more effective, but resulted in major losses of ethanol, 13% on average. Importantly however, a 475 mL discard removed 47% of the undesirable 2-methylpropan-1-ol from the essencia, but only 19% from the T500.

In Chapter 1, the statistical limitations of this work were discussed, and before imake recommends the first 50mL volume should be discarded, it would be useful to replicate this work, something that could not done in the time frame of the present study.

Summary of results in this chapter

Ethyl acetate and acetaldehyde were earlier reached their maximum recoveries than those of the other cogeners for both T500 and essencia stills. However, 2-methylpropan-1-ol and 3-methylbutan-1-ol is more quickly reached their maximum recoveries by the essencia still. The essencia's recovery curves were more scattered from the ethanol recovery curve when compared with the equivalent curves of the T500 still. The result suggested that congeners might be better fractionated by the essencia than by the T500, although the theoretical plates value of essencia was lower (4.92 for essencia but 5.80 for T500).

Chapter 6

Gas chromatography and sensory analysis of distillates from two model fermentations: a comparison of stills' performances

Introduction

Sulphur compounds are common undesirable congeners in liquors (Chapter 1.2). In response to imake's suggestion, work in this chapter was designed to identify possible species and concentrations of sulphur compounds in conventionally fermented distillates by the gas-chromatograph mass-spectrum (GC-MS). Distillates were prepare from two washes, both recommended by imake, using the T500 and essencia stills. This work also extends to sensory comparisons of the distillates.

Materials and methods

Fermentation

Yeast

The Turbo classic yeast was obtained from the imake, which is claimed to produce high ethanol content wash (Figure 42).

Figure 42 The Turbo Classic yeast

Fermented wash preparation

From imake's suggestions fermented washes were prepared by two recipes considered as the most popular recipes by imake customers: Recipe A contained 3 kg white sugar and 3 kg dextrose; Recipe B contained 6 kg white sugar and 2 kg dextrose.

For each recipe, two 30 L fermenters were cleaned and filled by 21 L tap water at 30°C, and then the sugars were added and dissolved. One sachet of Still Spirits Turbo Classic yeast was added into one fermenter when the temperature was below 30°C. Before the fermenters were sealed

under airlock, the gravity and temperature of each wash was measured. The washes were fermented at room temperature (round 22°C) for five days, when fermentation was considered complete. This was confirmed by specific gravity, each wash containing around 12% ethanol.

Yeast residue was removed with a clearing agent. One sachet of Still Spirits Turbo Clear (part A + part B) (Figure 43) was added into each fermented wash thereby cleaning the yeast and other debris. The wash was held for 48 hours to allow settling. At this point there were two fermenter volumes of around 20 L for each recipe, four in all. Before distillation, the pairs were self-blended to eliminate within-recipe between-tank variation.

Figure 43 The Turbo Clear sachet

Distillation of fermented wash

The fermented washes were distilled in the T500 and essencia stills, with the by now standard 60 to 65°C cooling water temperature. The distillate was collected in fractions of 50, 100, 250, 500, 500, 1000 mL, until no further distillate was recoverable. Then the distillate samples were capped and stored in a refrigerator at around 4°C.

Gas chromatography- mass spectrometry

For each native distillate, a 1µL sample was injected into gas chromatograph-mass spectrophotometer (GC-MS) at a split ratio of 1:50. The injector temperature was 200°C. The Trace GC Ultra (Thermo Scientific, USA) was coupled with a 30 m (long) x 0.3 mm (internal diameter) x 0.5 µm (film thickness), VF5 ms low bleed/MS fused-silica capillary column (5%-phenyldimethylpolysiloxane phase). The mass scan range was based on the similar research from N. Moreira, Guedes de Pinho, and Vasconcelos (2004). Detection was performed with a mass spectrophotometer (Thermo Scientific, DSQ Series, USA). Helium was used as the carrier gas of 1.5 mL min^{-1} with a constant flow rate. The oven temperature was started at 35°C with a 4

min hold time. It was then heated to 200°C at 20°C min^{-1}, and held for 2 min at 200 °C. The mass spectrometer was operated in the electron impact mode with a source temperature of 200 °C (EI at 70 eV). The temperature of the MS transfer line was 250°C. The mass acquisition range was set at full scan mode and the electron multiplier was set by the auto tune procedure. The electron multiplier scanned masses from m/z 30 to m/z 300. The acquisition was first set to the full scan model to identify the major compounds in distillates. Based on the results from the full scan model, the acquisition was then set to selective ion monitoring mode for several sulfur compounds: hydrogen sulphide, dimethyl sulphide, dimethyl disulphide, diethyl sulphide, diethyl disulphide, methanethiol and 2-mercaptoethanol. These are common sulphur compounds in alcoholic beverages, which were discussed in Chapter 1.2. In each case, the parent ion was selected for monitoring (hydrogen sulphide = 34.1 g $mole^{-1}$; dimethyl sulphide = 62.1 g $mole^{-1}$ etc.). The chromatogram was analyzed by NIST spectral library to tentatively identify the chemicals, defined as a similarity above 92%. A later search with full scan mode data was conducted for methional (104.2 g $mole^{-1}$).

Sensory triangle tests

Distillate samples from T500 and essencia were obtained from two fermentation recipes. Triangle tests were used to identify the difference between distillates from different stills but from the same recipe, A or B (Meilgaard et al., 1999).

Spirit samples were prepared for two trials: the native distillate samples and dilution distillate samples where the native distillate was diluted to 40% alcohol by distilled water. In contrast to the GC-MS work where each distillate was analysed, the triangle tests were conducted with pooled distillates, which were blended by proportion from the original fraction volumes.

For each trial of triangle test, the three wine glasses was marked by three-digit random numbers, and placed on a tray. Each 20 mL sample was place in a wine glass (Figure 44), where two samples were identical and one was different. This was done in two ways: essencia, essencia, T500 (EET), and T500, T500 and essencia (TTE).

Figure 44 A typical triangle test

Each trial involved 30 panelists, taking around 2 hours, who mainly come from the Schools of Applied Sciences and Engineering, AUT University. Panelists were asked to smell the aroma of three samples in one tray (EET for example), and to circle which one they thought was different from the other two on a question paper. If they did not know they had to guess. After taking a short break and smelling water to remove the odour of the EET trial, the panelist then moved to the TTE tray. The three glasses were randomised for position on each tray for every panelist. During the process, the gender and age of each panelist was recorded on the ballot paper. The data of the triangle test were analysed for significance, according to the table in Appendix 7.

Sensory paired preference test

The paired preference test was used to identify people's preference on distillates from the T500 and essencia stills.

The preference test only used the pooled fractions that had been diluted to 40% ethanol, because commercial liquors usually contained 40% alcohol. For each trial of paired preference test, the two wine glasses was marked by three-digit random numbers, and were placed on the tray and placed on a tray with a glass of water. Each 20 mL sample was placed in a wine glass (Figure 45), where one sample was from the T500 still and the other was from essencia, both from the same recipe, A or B.

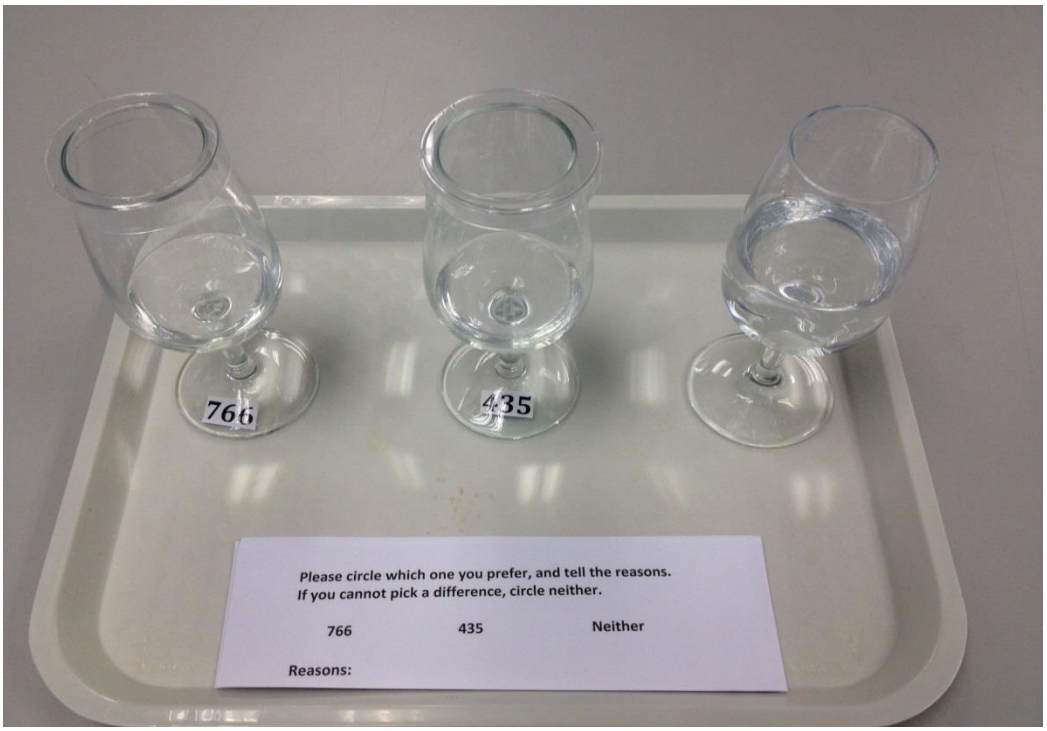

Figure 45 A typical paired preference test (water on the right)

Each trial was involved 50 panelists, taking around 1.5 hours, who mainly came from the Schools of Applied Sciences and Engineering. Panelists were asked to smell the aroma of three samples in one tray (from recipe A for example), and to circle which one they liked more. The panelist then moved to the recipe B tray after taking a short break and smelling water to remove the odour of the previous trial. If panelists had no preference they could select the 'neither' option. The two glasses were randomized for position on each tray for every panelist. Gender and age were recorded. Panelists were also asked to write the reason why they preferred their circled choice. The data of triangle test was analysed for significance, according to the table in Appendix 8.

Results and discussions

GCMS analysis for the chemical composition of each distillate fraction

The GCMS results are shown in Appendix 9. For the native distillates from Recipe A, the major chemical compositions were similar for both stills. However, more ester compounds were recovered in essencia's native distillate from Recipe B than that of the T500. The reason for this is not known. It is important to note that these results were not replicated for statistical purposes so the conclusion of more esters from essencia must be seen as a tentative result.

No sulfur compounds including methional were identified in the full scan mode, nor in the selective ion monitoring mode. Either the compounds were fully trapped by the copper (T500) or brass (essencia) and/or the concentrations of sulfur compounds were low to the point that they were below the limits of mass spectral detection. However, this does not mean that would be impossible to detect by nose, because sulphur compounds have low odour thresholds (Nathalie Moreira, Guedes de Pinho, Santos, & Vasconcelos, 2010).

The triangle test

Table 12 shows the results of the triangle test. Discrimination of native distillate was borderline significant for the TTE test of Recipe A ($P = 0.05$) but not the parallel EET test. The result is probably not important because what discrimination there was might be caused by differences in ethanol concentration in these native distillates.

Table 12 Results of a triangle test to discriminate distillate from two stills

	Comparison	Number of panelists correctly discriminating the difference for T500 and essencia stills	Statistical significance[1]
Native distillate		Recipe A	
	essencia essencia T500	10 of 30	NS
	T500 T500 essencia	15 of 30	*
		Recipe B	
	essencia essencia T500	12 of 30	NS
	T500 T500 essencia	10 of 30	NS
Diluted to 40%		Recipe A	
	essencia essencia T500	13 of 30	NS
	T500 T500 essencia	10 of 30	NS
		Recipe B	
	essencia essencia T500	14 of 30	NS
	T500 T500 essencia	17 of 30	**

[1]NS, Not Significant; *, P < = 0.05; **, P <= 0.01. The significance level was obtained from the table in Appendix 7.

When diluted to 40% ethanol, panelists could not discriminate the two stills with Recipe A. For Recipe B, the TTE test was significant (P < 0.01, 17 out 30 panelists), but not the EET test. When the date were analysed by gender and age, there were scattered examples of successful discrimination, but there were no clear patterns (Table 13 and Table 14). Overall, it is clear that odour differences between the spirits produced by the the two stills were minimal. Because odour is a major part of flavor (Meilgaard et al., 1999), it can be concluded that the spirits would taste much the same.

Table 13 Results of a triangle test to discriminate distillate from two stills, where data were analysed by gender

		Number of panellists correctly discriminating the difference for *essencia essencia T500*	Statistical signif.[1]	Number of panellists correctly discriminating the difference for *T500 T500 essencia*	Statistical signif.
Native distillate			Recipe A		
	Male	9 of 21	NS	13 of 21	**
	Female	1 of 9	NS	4 of 9	NS
			Recipe B		
	Male	6 of 20	NS	9 of 19	NS
	Female	4 of 10	NS	3 of 11	NS
Diluted to 40%			Recipe A		
	Male	6 of 19	NS	5 of 22	NS
	Female	7 of 11	*	5 of 8	NS
			Recipe B		
	Male	8 of 19	NS	12 of 20	*
	Female	6 of 11	NS	5 of 10	NS

[1]NS, Not Significant; *, P <= 0.05; **, P <= 0.01. The significance level was obtained from the table in Appendix 7

Table 14 Results of a triangle test to discriminate distillate from two stills, where data were analysed by age

		Number of panellists correctly discriminating the difference for *essencia essencia T500*	Statistical signif.[1]	Number of panellists correctly discriminating the difference for *T500 T500 essencia*	Statistical signif.
Native distillate	Recipe A				
	Age < 30	8 of 22	NS	13 of 22	*
	Age > 30	2 of 8	NS	4 of 8	NS
	Recipe B				
	Age < 30	10 of 25	NS	7 of 24	NS
	Age > 30	2 of 5	NS	3 of 6	NS
Diluted to 40%	Recipe A				
	Age < 30	8 of 21	NS	5 of 20	NS
	Age > 30	5 of 9	NS	5 of 10	NS
	Recipe B				
	Age < 30	11 of 23	NS	11 of 22	NS
	Age > 30	3 of 7	NS	6 of 8	*

[1]NS, Not Significant; *, P<=0.05. The significance level was obtained from the table in Appendix 7

The paired preference test

The preference test showed there was no significant preference between the two distillates (Table 15). However, 29 of 50 people preferred the essencia distillate from the Recipe B, a result that was close to significance, where 33 of 50 would be P = 0.05.

Table 15 Results of a paired preferences test of distillate from two stills

	Distallate name	Number of panelists preferring the T500, essencia or Neither	Statistical significance[1]
Diluted to 40%	Recipe A		
	T500	19 of 50	NS
	essencia	21 of 50	NS
	Neither	10 of 50	NS
	Recipe B		
	T500	18 of 50	NS
	essencia	29 of 50	NS
	Neither	3 of 50	NS

[1]NS, Not Significant. The significance level was obtained from the table in Appendix 8

Table 16 and Table 17 show the analysis by gender and age, and gave no new insights into preference.

Table 16 Results of a paired preference test of distillate from two stills, where data were analysed by gender

Gender	T500	Statistical signif.[1]	essencia	Statistical significance	Neither	Statistical signif.
			Recipe A			
Male	14 of 50	NS	14 of 50	NS	7 of 50	NS
Female	5 of 50	NS	7 of 50	NS	3 of 50	NS
			Recipe B			
	T500		essencia		Neither	
Male	11 of 50	NS	22 of 50	NS	2 of 50	NS
Female	7 of 50	NS	7 of 50	NS	1 of 50	NS

[1]NS, Not Significant. The significance level was obtained from the table in Appendix 8

Table 17 Results of a paired preference test of distillate from two stills, where data were analysed by age

Age	T500	Statistical signif.[1]	essencia	Statistical signif.[1]	Neither	Statistical signif.[1]
			Recipe A			
Age>30	9 of 50	NS	3 of 50	NS	6 of 50	NS
Age<30	10 of 50	NS	18 of 50	NS	4 of 50	NS
			Recipe B			
	T500		essencia		Neither	
Age>30	6 of 50	NS	10 of 50	NS	1 of 50	NS
Age<30	12 of 50	NS	20 of 50	NS	1 of 50	NS

[1]NS, Not Significant. The significance level was obtained from the table in Appendix 8

The two complete sets of written responses for the two recipes are shown in Appendix 10. These panelist comments were of interest, particularly for Recipe B, which approached significance. For Recipe B, 32 people said the distillate from the T500 had a stronger alcohol smell, while only 6 people found the distillate from essencia was stronger. Importantly, the ethanol concentrations were the same, suggesting that the panelists were sensing and judging something other than ethanol.

For those who preferred the T500 distillate, 8 of the 18 people who preferred the smell of T500 distillate claimed that this was the real smell of liquor. These 8 comprised 4 males and 4 females of whom 6 were young and 2 were older. 4 of the 18 people who preferred the T500 distillate found that essencia distillate was too strong or too sweet.

For those who preferred the essencia distillate from the recipe B, 24 of 29 people preferred it because the smell from essencia was less alcoholic, but fruity, peachy, floral, sweet and food-like, while only 2 liked essencia distillate because it had strong alcohol smell. Some of them mentioned that the strong alcohol smell from T500 distillate was like a hospital smell that that would make them sick after drinking it. The group of 24 who liked the less alcoholic smell comprised 17 males and 7 females, of whom 10 were young and 14 were older. According to the results from GCMS, greater ester compounds were found in distillates from essencia still (Recipe B). As esters usually contribute to desirable flavors in alcoholic beverages (Jackson, 2008), this should be a reason that further explained why essencia'd distillate contains fruity and floral flavor.

Referring now to Appendix 10, Recipe B, at one panelist detected a boiled potato/rice smell in the T500 distillate. This observation may be important. One characterising flavour of boiled potato is methional (kaur, Singh, & Kaur, 2009) (Figure 46), a so-called Strecker aldehyde formed in the Maillard reaction (Maillard 1923) by decarboxylation and deamination of the amino acid methionine. The important point about methional is that it a sulphur compound and although not detected in distillates, it seems possible that it is present, and was not completely trapped by the copper saddles. This is further discussed in the concluding Chapter 8.

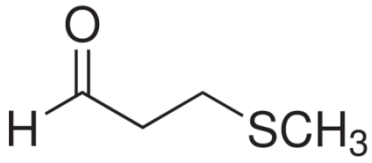

Figure 46 The formula of methional

Summary of results in this chapter

The GCMS results showed more ester compounds were recovered by essencia still (recipe B), but no sulfur compounds were detected by GCMS. The results from triangle test showed there was significant difference between diluted distillates (Recipe B) of essencia and T500. The paired preference test showed that, numerically, more people preferred the essencia's diluted distillate from the Recipe B than that of T500, although the results of preference test did not quite reach the minimum significance level of $P < 0.05$.

Chapter 7

Analysis of ceramic saddles, copper saddles (T500) and brass scourers (essencia)

Introduction

The fractionating column of T500 still comprises 100 g of copper saddles overlaying 1 kg of ceramic saddles (Chapter 2). These together provide the theoretical plates that govern still performance. In the essencia still the same job is accomplished by what appears to be brass scourers, similar, if not identical to, Goldilocks kitchen scourers as used in most New Zealand homes to clean cooking pots. The copper and brass in these two systems serve another purpose: to trap sulphur compounds form fermentation as black sulphides. In the case of copper saddles, the copper is the binding material. In the case of the brass (as judged by appearance), both copper and zinc may be involved.

As possible confirmation of this, the colour of copper saddles and brass scourers turn black after several distillations. Figure 47 and Figure 48 show the typical colour changes of copper saddles and brass scourers after distillations. This chapter describes simple measurements and experiments to research the composition of copper saddles, brass scourers, and the black deposits, as well as using a more sophisticated tool – scanning electron microscopy (SEM) – to research the phenomena. In addition, the nature of the ceramic saddles is explored by low power light microscopy and a reflux experiment.

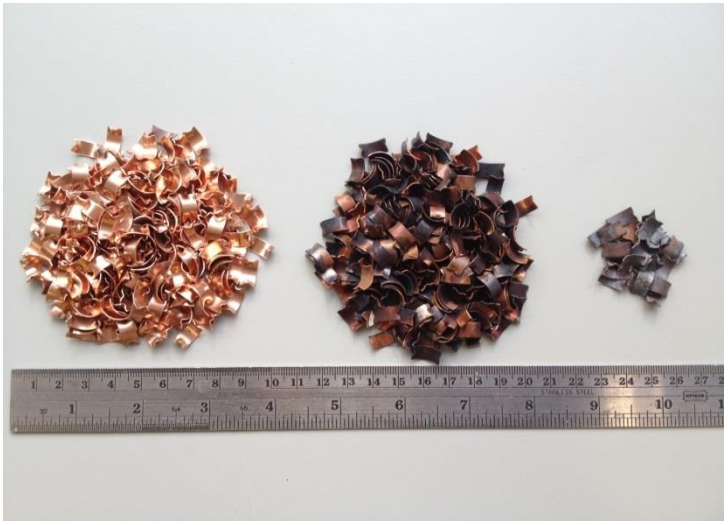

Figure 47 Colour changes of copper saddles after distillations. New copper saddles (left, golden); used copper saddles from ethanol distillation (middle, black); used copper saddles from distillation of fermented wash after many cycles (imake provided) (right, grey).

Figure 48 Colour changes of brass scourers after distillations. New brass scourers (left); used brass scourers from ethanol distillation (right)

Materials and methods

Porosity of ceramic saddles

Ceramic saddles (101 g) were paced into a round-bottomed flask, which was filled with about 100 mL ethanol wash (80% ethanol). The wash and saddles were refluxed for one hour. After reflux, the surfaces of ceramic saddles were quickly dried and immediately reweighed to find out if there was any significant moisture/ethanol uptake by the saddles.

Surface area of ceramic and copper saddles, and brass scourers

The approximate nominal surface area of the ceramic saddles was measured with the ruler shown Figure 47, in some situations by rolling the ruler edge around roughly circular surfaces. Eight saddles were measured and individually weighed to determine nominal[6] area per gram. The width and thickness of copper saddles were measured by a caliper micrometer. The width and thickness of scourers were measured by the SEM, and the mass of known lengths was determined with three-place electronic balance.

Effect of hydrochloric acid on copper saddles, and brass scourers

Solutions of 0.5 and 1.0 M HCl were added to used (blackened) copper saddles, and colour changes were observed. The headspace above the mixtures was smelt.

[6] Nominal assumes that the surface is perfectly smooth.

Scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX)

The samples were analysed by the dispersive X-ray under the SEM, which serves as an electron beam that interacted with the individual particles on the samples. SEM-EDAX is commonly used to identify the chemical composition of the sample's surface, and obtained the relative morphological information. X-ray data were processed to obtain the percentage of each measured element present in the individual particles.

A variety of samples were mounted according the SEM recommendations. A representative photograph (Figure 48) shows new and used copper saddles and brass scourer sourced from the top, middle and bottom of the scourer recovered from the essencia fractionating column after distillation. The three sites were chosen because there was a colour difference between these three sites (Figure 48).

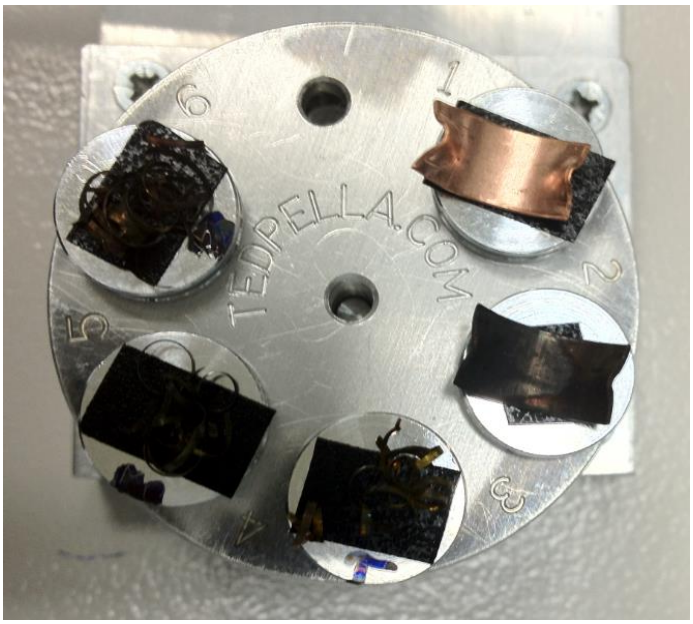

Figure 49 The samples as prepared for SEM-EDAX. New copper saddles (1), black copper saddles (2), brass top (3), brass middle (4) and brass bottom (5)

The samples were analysed for composition at an alternating current of 20.0 kV with the magnification from 300 to 10000 times. (The scourer dimensions were also determined.) The components of select sites on samples were showed by the percentages of weight and atoms. The atom percentages of components in samples were calculated in Excel.

Results and discussion

Porosity of ceramic saddles

After heating, the weight of ceramic saddles was 101 g, as the same weight of ceramic saddles before heating. This result showed the ceramic saddles did not absorb the ethanol from the wash,

which showed that the ceramic saddles were not porous. Thus, the nominal surface area reported in the next section was a useful guide to area of value in fractionation.

Effect of hydrochloric acid on new and used copper saddles and scourers

Hydrochloric acid solutions applied to new copper saddles changes the golden color of new copper saddles to pink/golden colour, and a sulphur smell was detected. Volatile sulphur compounds in the air are clearly binding to copper. This was not surprising given that in Rotorua, New Zealand, earlier telephone switching equipment required gold rather than copper contacts; silver plate will blacken on extended exposure to air, and this is due to sulphur binding.

The same solutions applied to used copper saddles restored the pink/gold colour of native copper and a distinct strong sulphur compound smell was noticed. Clearly the copper is binding sulphur compounds. Similar results were obtained with scourers (Figure 48).

Surface area of ceramic and copper saddles, and brass scourers

Table 18 shows the comparative surface area of the fractionating materials in both stills.

Table 18 Comparative surface area of the fractionating materials in the two stills

	T500	essencia
Mass of fractionating material	1 kg ceramic saddles+ 100 g copper saddles	88 g of brass scourers
Nominal[1] fractionating surface area (m^2)	0.288 (ceramic) + 0.051 (copper) + 0.080 (inner column surface area) = 0.419	0.772 (brass scourer + 0.0320 (inner column surface area) = 0.804

[1] Nominal assumes a perfectly smooth surface

This comparison suggests that the essencia should have better fractionating ability – and a higher theoretical plate value, which was not the case (Chapter 4). However, this suggestion overlooks the possibility that the true surface area of the ceramic saddles may be much greater due to an obvious roughness. Also, the height of the T500 column was 644.9 mm, more than double that of the essencia column, allowing greater scope for fractionation. The column walls may also contribute to fractionation.

The nominal fractionating surface area of essencia was 0.804, which was larger than that of T500 (0.409), which assumed that essencia should have more theoretical plates, as larger surface should provide more stages of vapor-liquid equilibrium inside the column. However, according to the results of plates calculation, the essencia has less theoretical plates than the T500. This

deduced that the number of theoretical plates should not only be determined by the surface area, but also determined by column height, packing materials and the other factors.

EDAX results

At the outset it must be pointed out that the following pictures are minute spot analyses and have no statistical basis. Thus the data are only suggestive. Figure 50, Figure 51 and Figure 52 show typical EDAX results for new copper saddles, used black copper saddles and used grey copper saddles. The black copper saddles were recovered after the binary ethanol-water distillations (Chapter 2) and the synthetic wash (Chapter 5). The grey copper saddles were supplied by imake and were recovered after distillations of fermented washes.

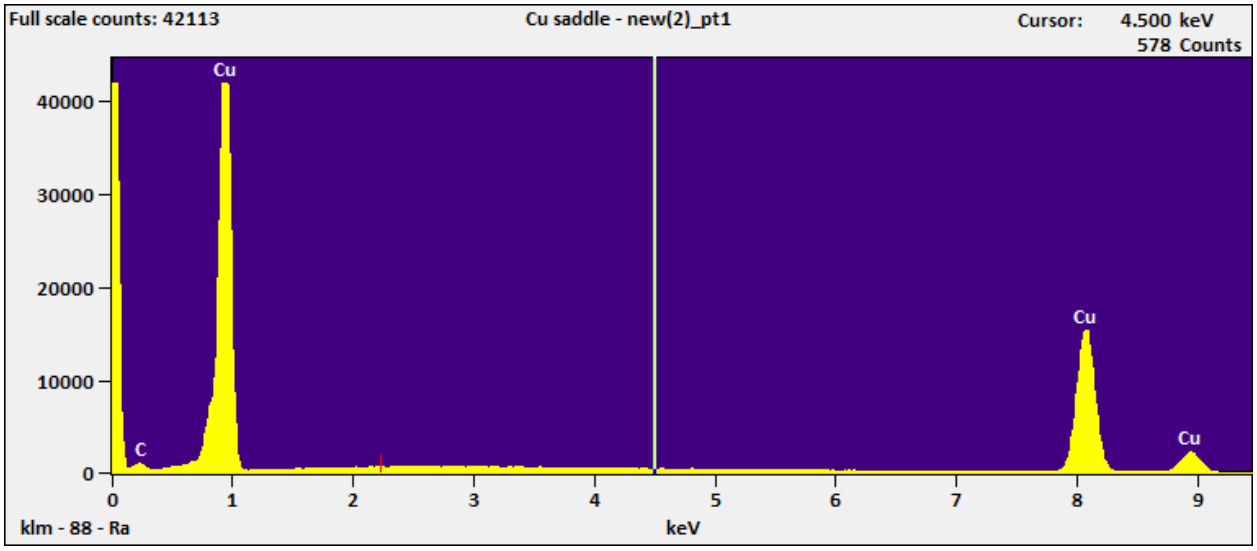

Figure 50 A typical EDAX result for new copper saddles

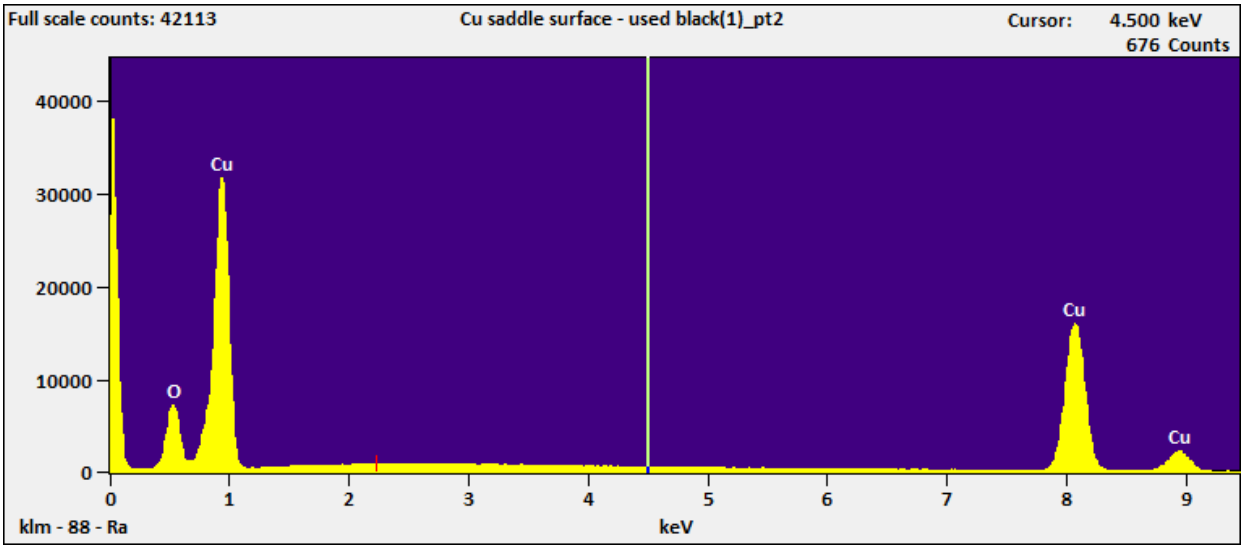

Figure 51 A typical EDAX result for used black copper saddles

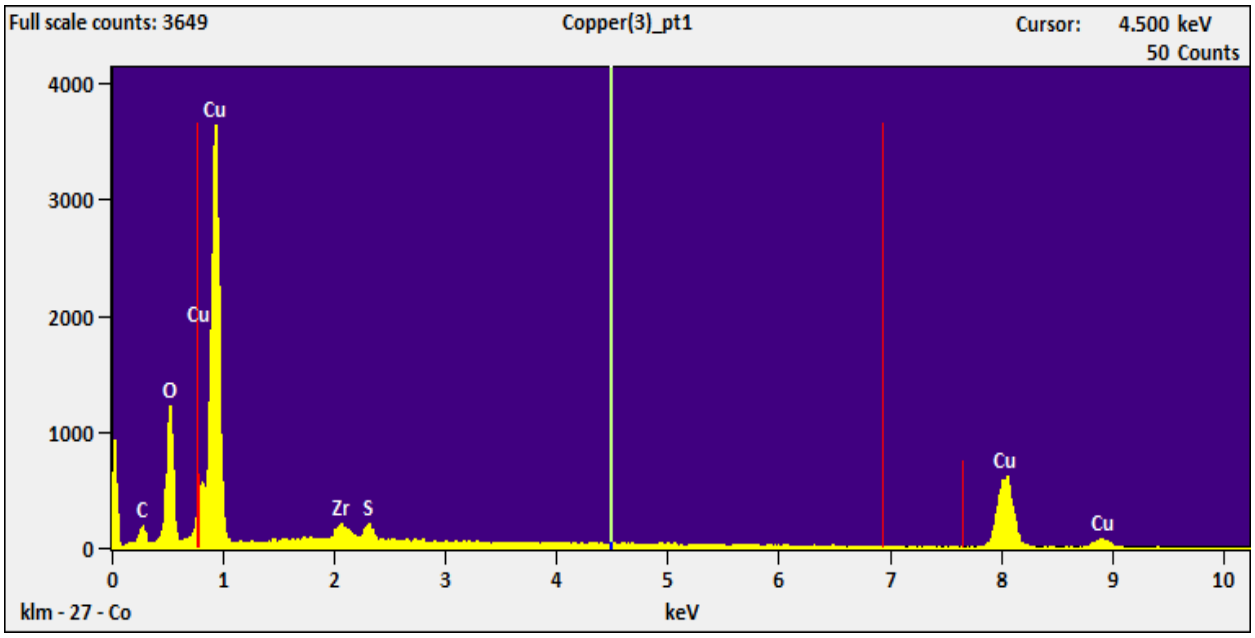

Figure 52 A typical EDAX result for used grey copper saddles

As expected, the new copper saddles were dominated by copper (Figure 50). After the binary and synthetic was distillations, oxygen became evident, but with no traces of sulphur (Figure 51). However, sulphur was detected in the grey copper saddles supplied by imake (Figure 52).

Figure 53, Figure 54 and Figure 55 show a typical EDAX result for the top, middle and bottom of brass scourers, respectively, after three binary distillations and one synthetic wash distillation.

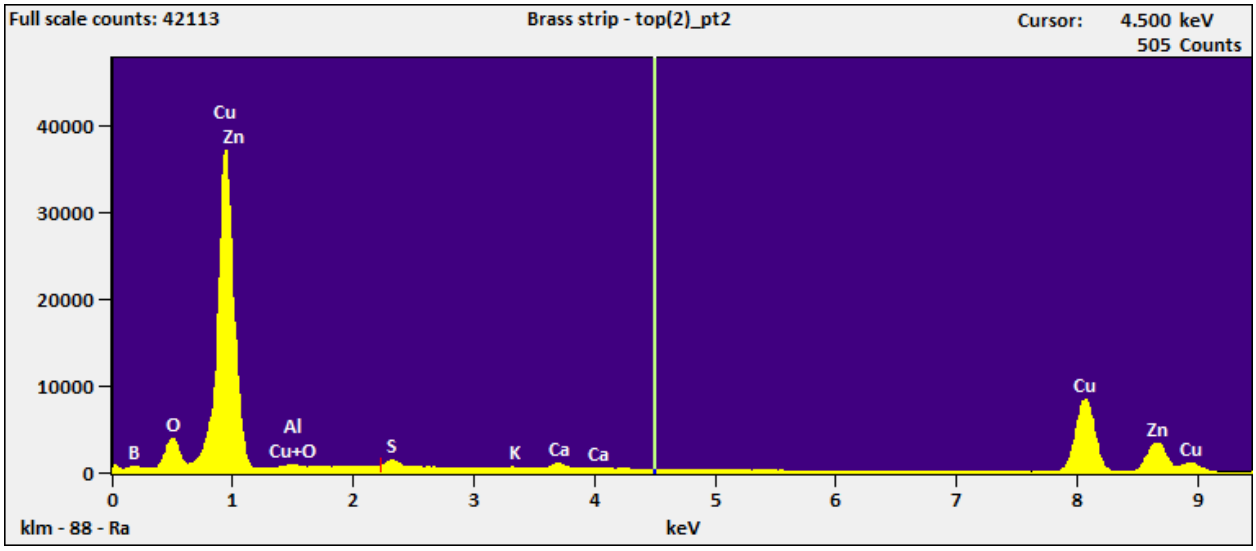

Figure 53 A typical EDAX result for used brass scourers (top)

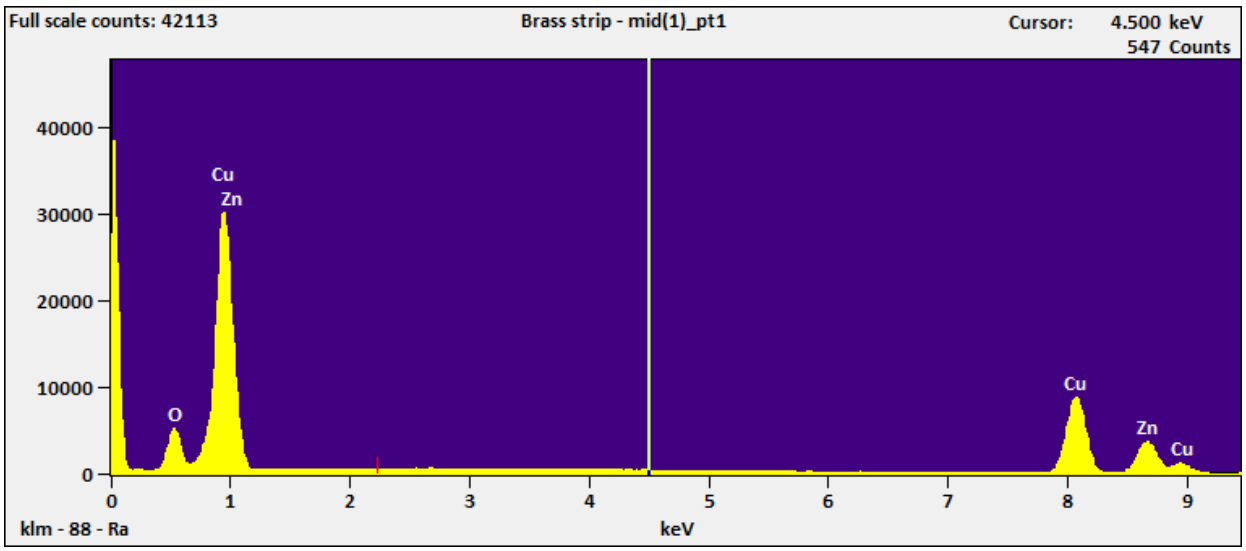

Figure 54 A typical EDAX result for used brass scourers (middle)

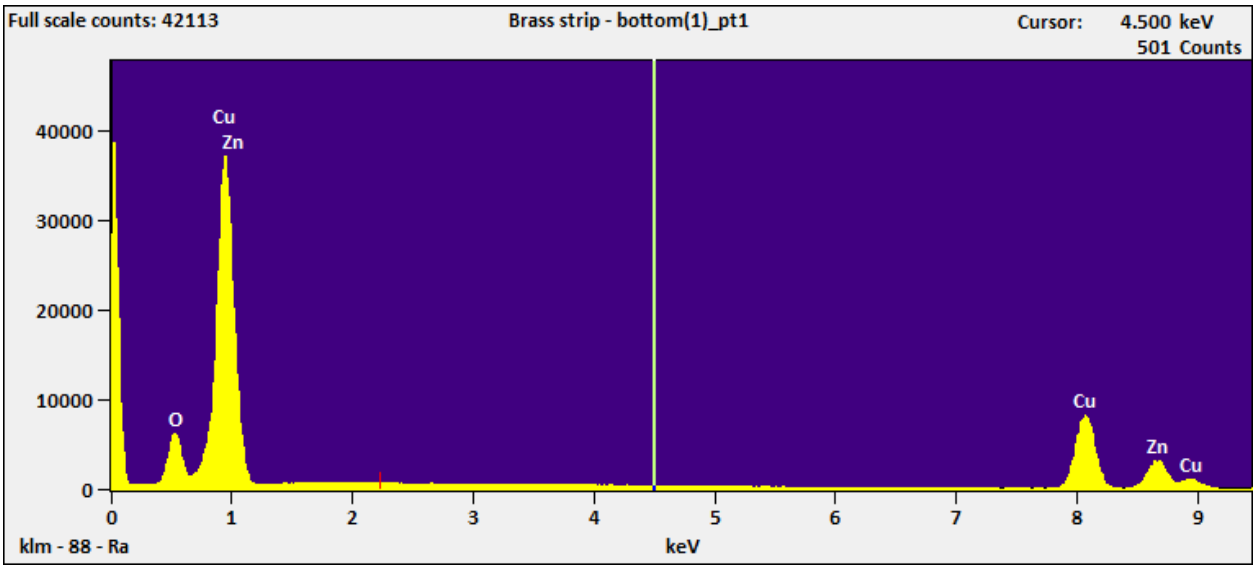

Figure 55 A typical EDAX result for used brass scourers (bottom)

Copper, zinc and oxygen were observed in all brass scourers samples, as expected. Sulphur was observed only at the top of the brass scourer column, but the non-statistical basis of this observation must be reemphasized. An SEM micrograph (Figure 56) shows two crystalline structures at the top of the brass scourer column. A search of Google images with the key words "copper oxide SEM" and "brass oxide SEM" and "zinc oxide SEM" revealed many images with similarities to Figure 7.11, although none was exactly the same as found here. The 'white' particles were most reminiscent of copper oxide, which is black to the eye. The flower-like crystals may be zinc oxide due to this reaction:

$$Zn + H_2O \longrightarrow ZnO + H_2$$

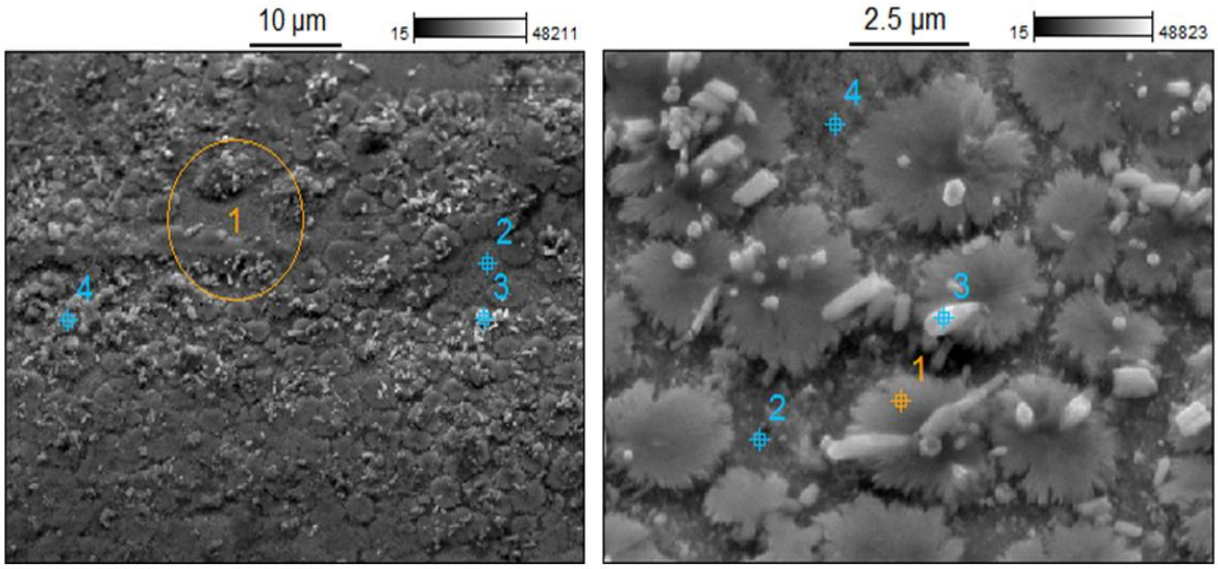

Figure 56 The SEM scanning photo of the top of brass scourers

The identification of sulphur by smell and by EDAX in grey saddles confirms their ability to bind sulphur compounds, but the greater reaction appears to be between copper and oxygen for copper saddles and between copper/zinc and oxygen for scourers. It is likely that scourers will also bind sulphur.

Summary of results in this chapter

The color of copper saddles and brass scourers turned black after distillation. The composition of new copper saddles was copper and carbon (little amount). The oxygen (rather than carbon) and copper are main composition in black copper saddles. The sulfur compound was detected in the grey copper saddles (imake provided). The used brass scourers are mainly composed by copper, zinc and oxygen. The sulfur compounds were detected in the top of brass scourers. As oxygen can be found in both used copper saddles and brass scourers, it can be deduced that the copper 1 oxide was formed during distillation.

Chapter 8

Conclusion

Main outcomes

In an ideal experimental situation, each of the distillations should be repeated to compensate for variability between distillations. However, the data in this thesis typically represent only single experiment trials, because the time limitation of a one year Master thesis. Nonetheless, the results are generally believable and could usually be interpreted from physical laws. What first follows is a summary of the main outcomes, but these must be considered in the knowledge that there was no replication to support the outcomes. After this summary, recommendations are made for further work, particularly in the matter of copper saddles.

In the factorial experiment with the T500 still involving four ethanol concentrations and three cooling rates, the distillate fractions from the imake-recommended 60 to 65°C water outlet treatment contained stable and high ethanol concentrations of around 90%, although the other distillation properties (distillation time time, percent total recovery) were usually not as good as the warmer 75 to 80°C cooling treatment. However, in the 20% ethanol washes, the total ethanol recovery from the 75 to 80°C treatment was relatively poor. The 50 to55°C cooling treatment produced distillates with similar 90% ethanol concentrations, but distillation times were excessive. Therefore, the research has confirmed that imake's recommended 60 to 65°C cooling treatment – however this may be achieved by adjusting flow rates – is useful for the domestic distiller. Warmer cooling treatments are also very effective, except where the ethanol concentration is 20%, but that is uncommon in domestic distillation. There are two other reasons why warmer cooling may also be useful. The first reason is that distillation is completed sooner, and much less water will be used (lower flow rate, less time). This can be important in localities where domestic water use is metered.

The distillation performance of the T500 still was affected by a cool draft, and a recommendation is made about this later.

The work extended to a comparison of the performance of the T500 compared with a competitor still, the essencia (Table 19). This was done with one wash, 15% ethanol in water. With this wash, the essencia still generally produced distillates with lower ethanol concentrations, which declined with time. With the two cooler cooling treatments, the essencia had lower ethanol recoveries than the T500, but this was overcome at 75 to 80°C, when the two stills had roughly equal recoveries (95 ± 1%). The performance of essencia still was more affected by the cooling

treatment than the T500 still. In this respect, the T500 is the better still because operator skill is less important.

The theoretical plates value for the T500 was 5.80, which was greater than that of the essencia (4.92). This probably explains why the T500 still produced distillate with more stable and higher ethanol concentrations than that of essencia still under the same distillation conditions.

On distillation of a synthetic wash, the gas chromatography (GC) results showed that the ethyl acetate and acetaldehyde were more quickly resolved by both T500 and essencia stills than those of the other congeners. The T500 performed better than the essencia for those two congeners. Generally however, the essencia's recovery curves were more scattered from the ethanol recovery curve when compared with the equivalent curves of the T500 still. This result suggested that congeners might be better fractionated by the essencia than by the T500. The reasons for this are unknown. However, to take advantage of this better scatter would require rejecting large volumes of distillate. As an example of this consider 2-methylpropan-1-ol. Although better resolved by the essencia, about 500 mL of distillate would have to be rejected to eliminate only half of the 2-methylpropan-1-ol; the same rejected volume from the T500 would eliminate 20%.

On distillation of the two model fermented washes (Recipe A and Recipe B), the gas chromatograph-mass spectrometer (GCMS) results showed that more esters were recovered by essencia still (Recipe B) than by the T500 still. There was no obvious difference for Recipe A. No sulphur compounds were identified after an extensive search by selective ion monitoring of likely sulphur compounds.

The results from the eight discriminatory triangle test showed, for Recipe B, there was one significant difference ($P <= 0.01$) between in distillates of essencia and T500 standardised to 40% ethanol. This was the T500 T500 essencia comparison. However, its replicate essencia essencia T500, was not significant. Other tests were either marginally significant (one test), or not significant (six). The paired preference test showed that more panelists preferred essencia's standardised distillate from the Recipe B than that from the T500, although the preference was just under minimal significance ($P =0.05$). This result was related to the greater ester recovery from the essencia's distillation of Recipe B. Overall, the differences between the two distillates were minimal.

The colour of the copper saddles and brass scourers both turned black after distillation. The results of scanning electron microscopy showed the composition of new copper saddles was dominated by copper. However, after distillation, oxygen and copper were the main elements in

black copper saddles, as well as in the grey copper saddles supplied by imake (exact history unknown). The scourers used in the essencia had the appearance of brass, and this was proved to be the case by microscopy, because both copper and zinc were detected. Similar to the copper saddles, oxygen was also found in all parts of it of the scourer array that extended the length of the column. Sulphur was detected in the grey copper saddles (imake) and at the top of brass scourers. It is possible that other parts of the scourer array, also contained sulphur, but it was not detected. However, absence of evidence is not evidence of absence. However, assuming for the moment that sulphur is concentrated at the top of the column, this suggests that the placement of copper saddles at the top of the T500 column is best, rather than elsewhere in the column. Table 1 showed the boiling points of a (non-exhaustive) number of sulphur compounds found in wine, ranging from -60 to 157°C. The design of a successful fractionating column must focus on those that are volatile in the tank temperature range, which does not exceed 90°C in the T500 in Appendix 2. Four of the seven sulphur compounds listed in Table 1 are volatile in this range and the most volatile would be expected to accumulate at the top of the column, and that is where the copper saddles are located.

Recommendations to imake

Although the T500 still performs very well, and is remarkably stable to variations in operating conditions, there are some changes that imake may want to consider to improve the still's performance in the widest possible sense:

1. The instruction document is too long, given the ease with which the still can be operated. This would simply require editing the existing document.
2. Whereas the recommended cooling water outlet temperature of 60 to 65°C works well, less effective cooling also works well, and has the added advantage of less water used and quicker distillation.
3. The still should be sheltered from cool drafts.
4. The surface area of copper saddles should be increased on the basis that the reactive area of brass (copper plus zinc) of the essencia still is roughly 15 times larger. There is no evidence in this thesis that sulphur compounds are escaping capture by the saddles. However, a poorly fermented wash may generate more sulphur compounds than usual and must be trapped to produce high quality spirits. Doubling the copper saddle mass to 200 g would start to close the gap between the T500 and the essencia, and the surface area would still contribute to the fractionating ability. Customers may resist buying more copper, but as an alternative, imake could sell citric acid so that the copper could be recycled. Thus the

citric acid would become the consumable (and the home distiller could feel virtuous by recycling the copper).

Table 19 Summary of the comparative properties of the T500 still and the competitor essencia still

Parameters	T500	essencia	Comment
Nominal volume (L)	25	25	
Volume of distilled (L)	23.5	23.5	
Heating element (kW)	2	2?	Not identified for essencia but likely
Element in contact with wash	No	Yes	
Height of fractionation column (mm)	643	245	
Cooling within the fractionation column	An asymmetric vertical tube	A symmetrical collar	
Temperature monitoring	Column top and cooling water outlet, both digital	Column top with a conventional glass thermometer	
Nature of fractionating material	Copper saddles and non-porous ceramic saddles	Fine brass wire	
Nominal fractionating surface area (m^2)	0.288 (ceramic) + 0.051 (copper) + 0.080 (inner column surface area) = 0.419	0.772 (brass scourer + 0.0320 (inner column surface area) = 0.804	
True fractionating surface area	Larger?		The ceramic saddles, although not porous may have a complex surface.
Sulphur reactive area (m^2)	0.051	0.772	Assumes Cu and Zn in scourers equally available
Ethanol concentrations in distillates	Higher	Lower	
Total distillation time	Longer	Shorter	

Total recovered distillates volume	Smaller	Larger	
Ethanol recovery	Better	Worse?	
Distillation performance was affected by the different cooling treatments	Slightly	Largely	
Theorectical plates number	5.80	4.92	
Resolution of congeners	Worst?	Best?	2-Methylpropan-1-ol better resolved by essencia
Odour of spirits	Strong alcohol smell	Fruity, floral, sweet (Possibly better)	
Preferred by most panelists	No?	Yes?	Only for the Recipe B. The preference was similar for Recipe A.

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Appendix 1

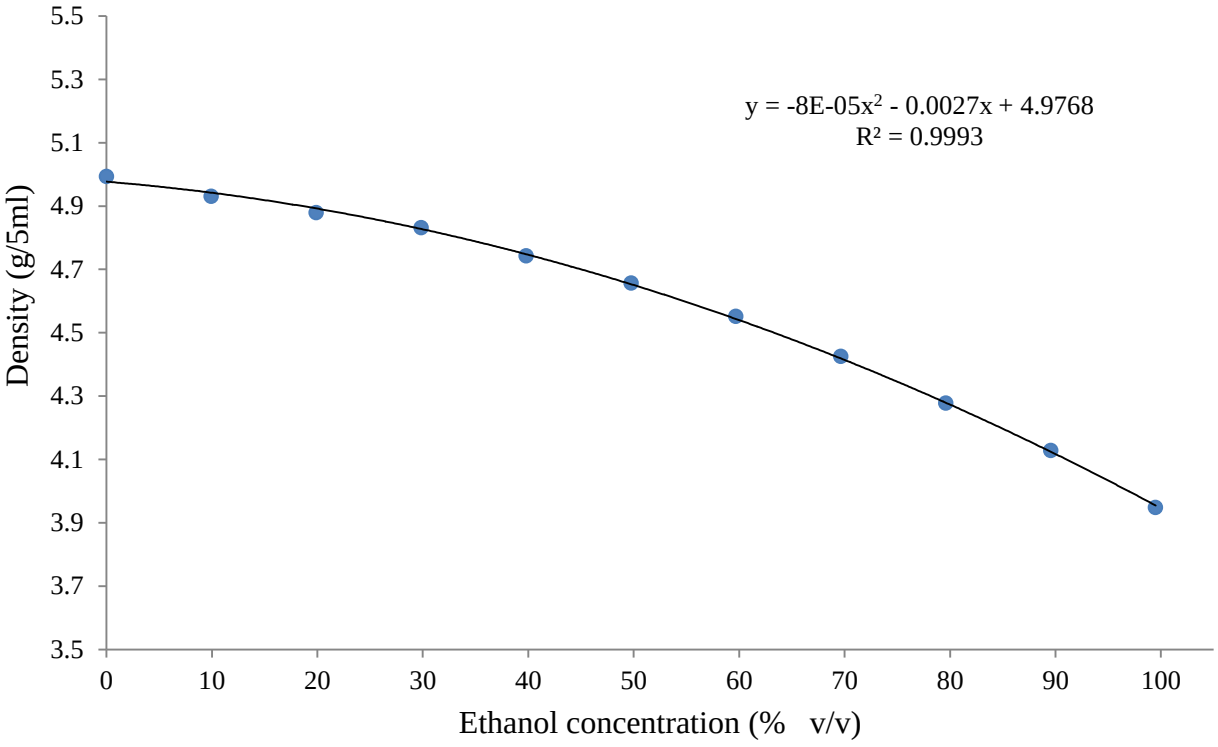

Figure 57 The ethanol concentration and density calibration curve for the T500 still

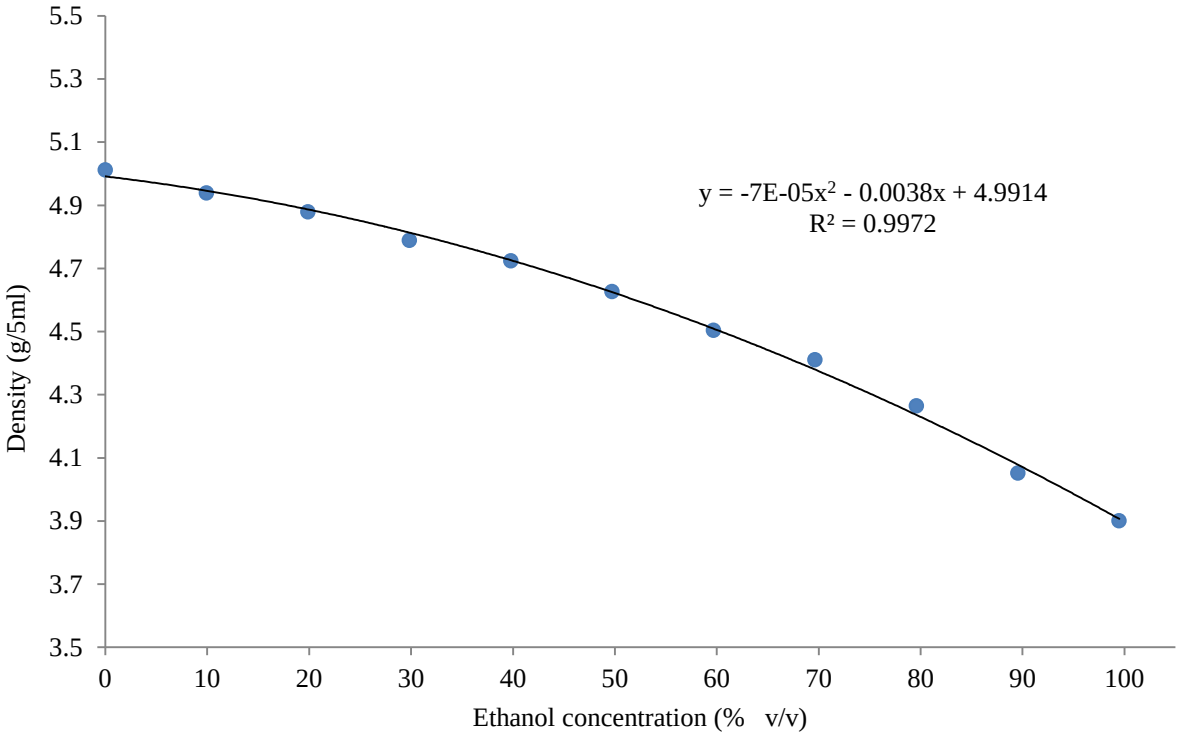

Figure 58 The ethanol concentration and density calibration curve for the essencia still

Appendix 2

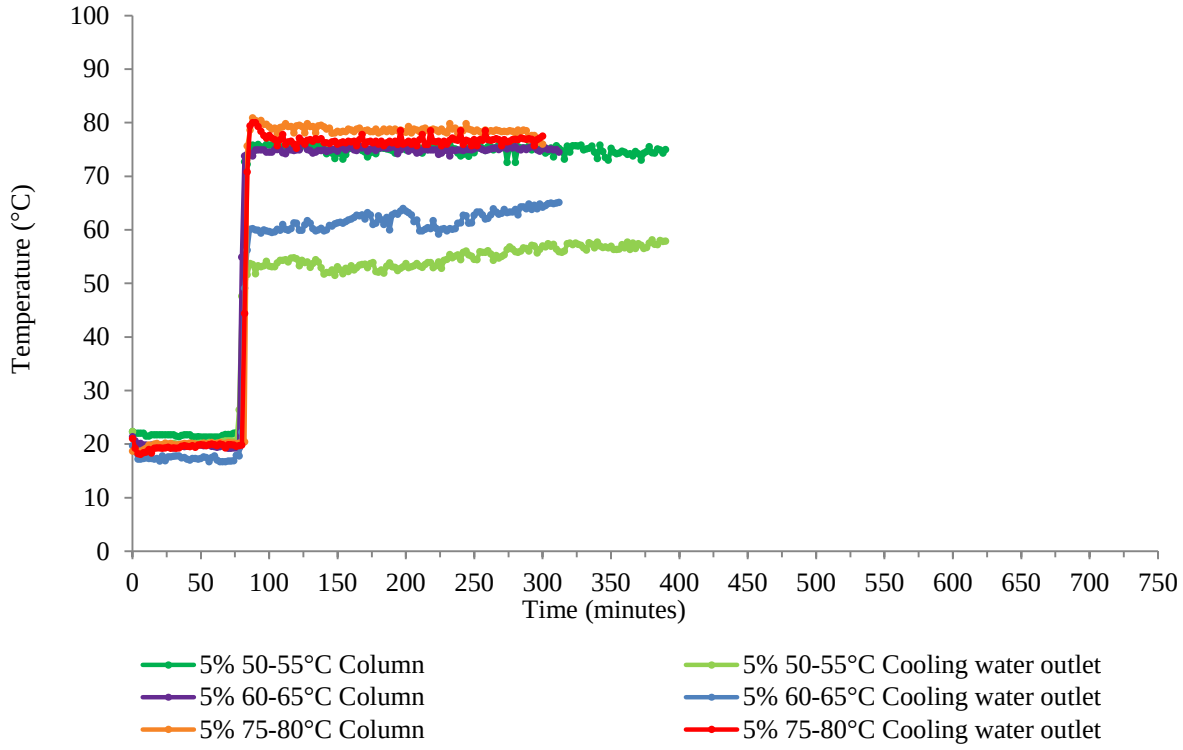

Figure 59 Kinetics of column and cooling temperatures from a 5% ethanol wash at three cooling temperatures

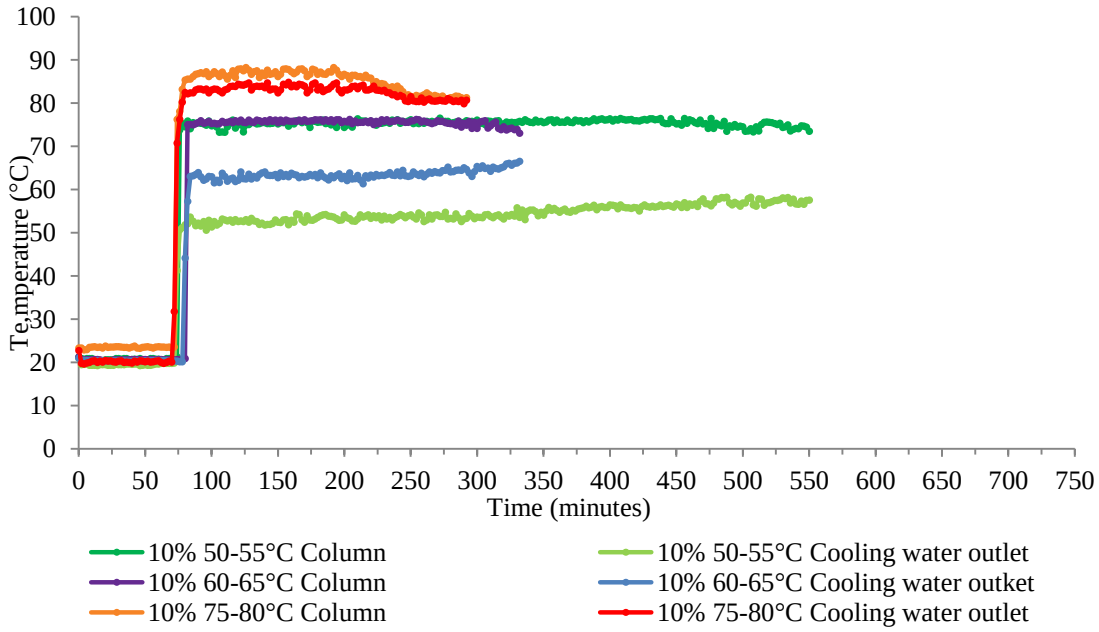

Figure 60 Kinetics of column and cooling temperatures from a 10% ethanol wash at three cooling temperatures

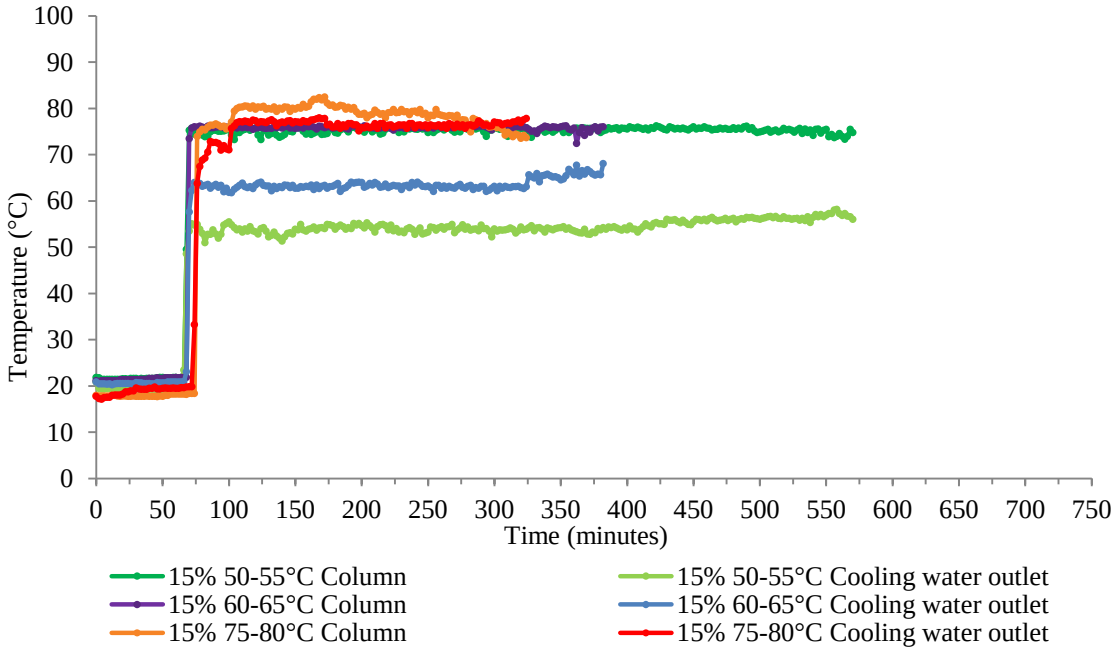

Figure 61 Kinetics of column and cooling temperatures from a 15% ethanol wash at three cooling temperatures

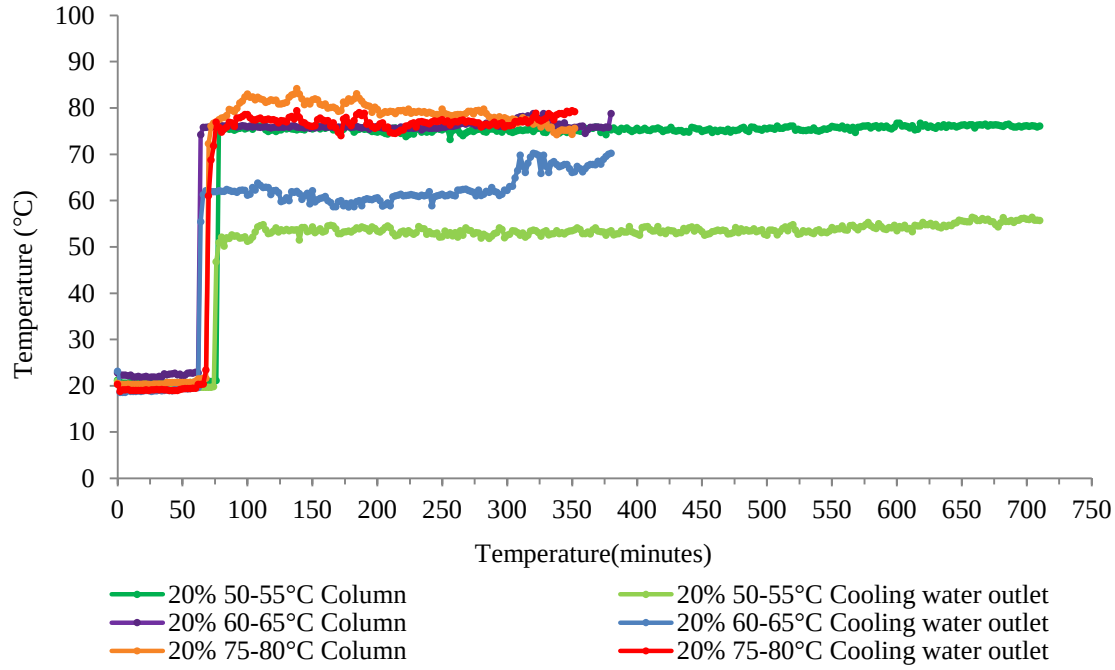

Figure 62 Kinetics of column and cooling temperatures from a 20% ethanol wash at three cooling temperatures

Appendix 3

Table 20 Vapour/liquid equilibrium data for ethanol/water mixtures at a constant pressure one atmosphere, calculated fromSeader and Kurtyka (1984)

Temperature (°C)	Mole fraction of ethanol in:		% w/w:		% v/v:	
	Liquid	Vapour	Liquid	Vapour	Liquid	Vapour
78.15	0.8943	0.8943	95.6	95.6	97.2	97.2
78.41	0.7472	0.7815	88.4	89.3	92.1	92.8
78.74	0.6783	0.7385	84.2	87.8	88.9	91.7
79.3	0.5732	0.6841	77.4	84.7	83.3	89.2
80.7	0.3965	0.6122	62.7	80.1	70.3	85.6
82.3	0.2608	0.5580	47.4	76.3	55.2	82.4
84.1	0.1661	0.5089	33.7	72.6	40.5	79.2
86.7	0.0966	0.4375	21.5	66.5	27.5	73.8
89.0	0.0721	0.3891	16.6	61.9	20.4	69.6
95.5	0.0190	0.1700	4.7	34.4	5.9	41.2
100.0	0.0000	0.0000	0.0	0.0	0.0	0.0

Appendix 4

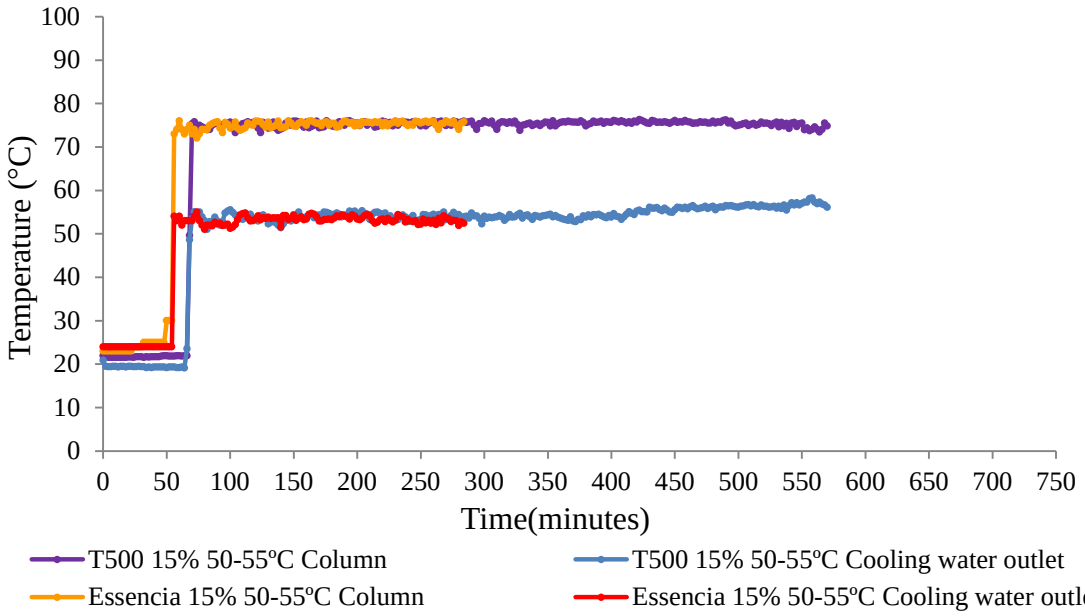

Figure 63 Kinetics of column and cooling temperatures from a 15% ethanol wash at 50 to 55°C cooling temperatures for both essencia and T500 stills

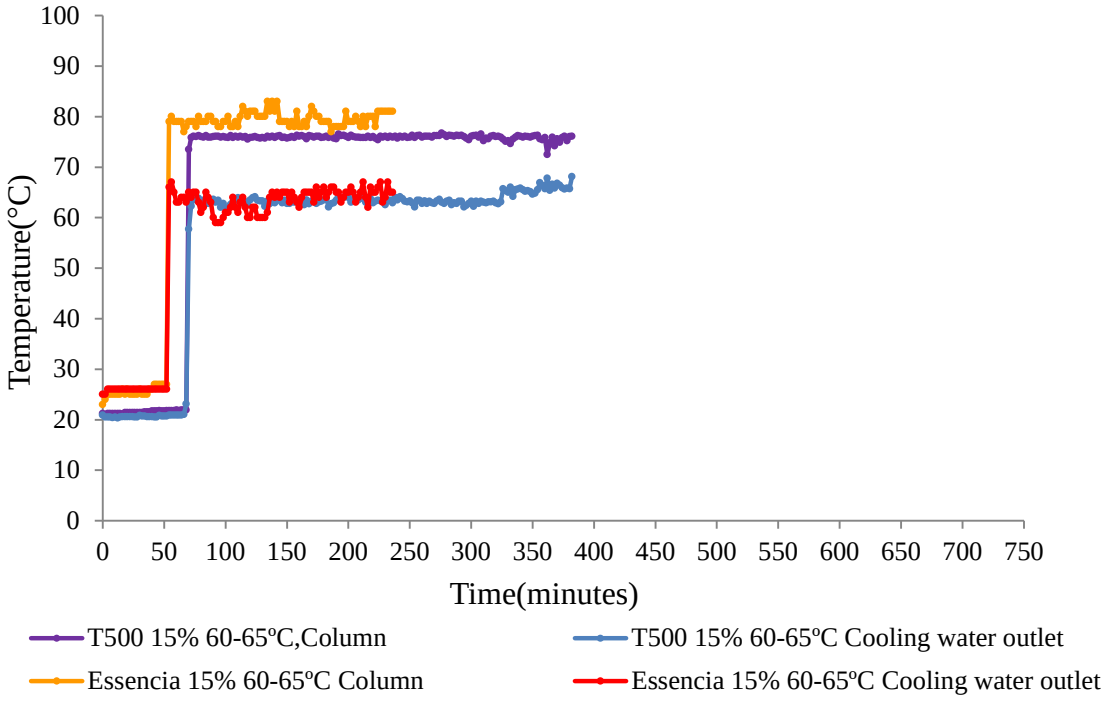

Figure 64 Kinetics of column and cooling temperatures from a 15% ethanol wash at 60 to 65°C cooling temperatures for both essencia and T500 stills

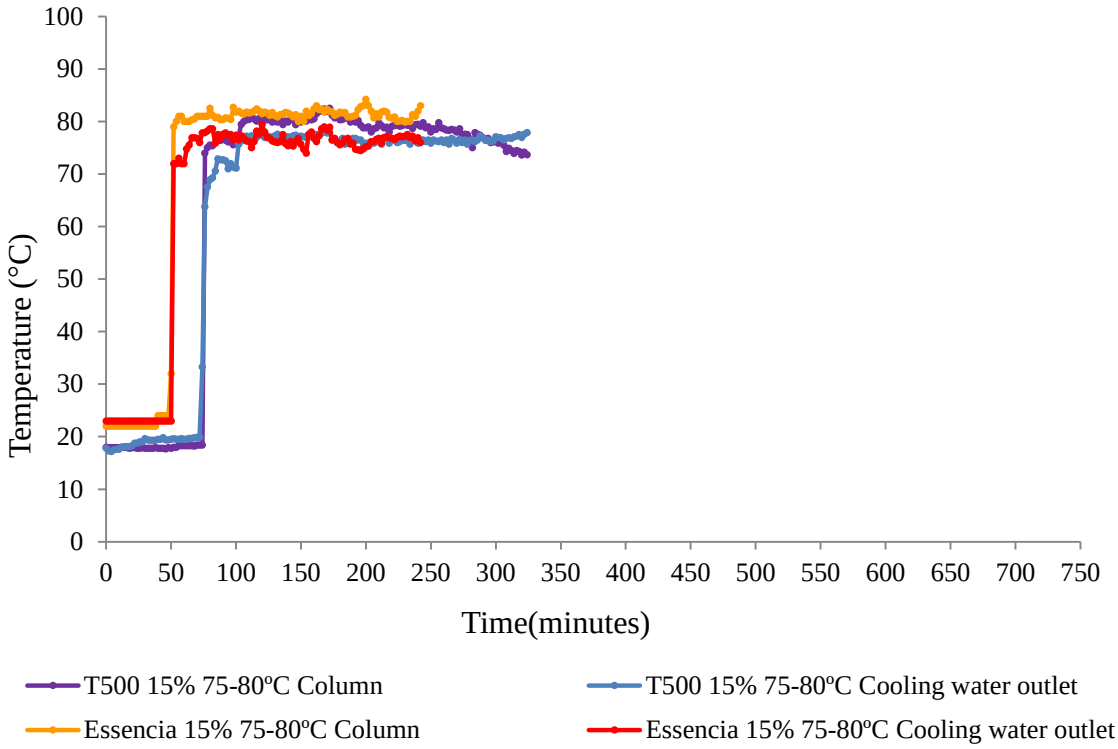

Figure 65 Kinetics of column and cooling temperatures from a 15% ethanol wash at 75 to 80°C cooling temperatures for both essencia and T500 stills

Appendix 5

Table 21 Composition and relative volatility changes of ethanol- water solution. Retrieved from http://www.separationprocesses.com/Distillation/Table01.htm

Equilibrium Temperature °C	Mole Fraction Liquid, x	Mole Fraction Vapour, y	Difference in Mole Fraction (y – x)	Relative Volatility α	Predicted y-values from α_{ave}	Percentage Error in y-values
100.0	0.0000	0.0000	0.0000	-	0.0000	-
95.5	0.0190	0.1700	0.1510	10.575	0.0709	58.28%
89.0	0.0721	0.3891	0.3170	8.197	0.2345	39.74%
86.7	0.0966	0.4375	0.3409	7.274	0.2965	32.23%
85.3	0.1238	0.4704	0.3466	6.286	0.3577	23.96%
84.1	0.1661	0.5089	0.3428	5.202	0.4398	13.58%
82.7	0.2337	0.5445	0.3108	3.920	0.5459	-0.25%
82.3	0.2608	0.5580	0.2972	3.578	0.5817	-4.25%
81.5	0.3273	0.5826	0.2553	2.869	0.6573	-12.82%
80.7	0.3965	0.6122	0.2157	2.403	0.7214	-17.84%
79.8	0.5079	0.6564	0.1485	1.851	0.8027	-22.29%
79.7	0.5198	0.6599	0.1401	1.792	0.8101	-22.76%
79.3	0.5732	0.6841	0.1109	1.612	0.8411	-22.95%
78.7	0.6763	0.7385	0.0622	1.352	0.8917	-20.75%
78.4	0.7472	0.7815	0.0343	1.210	0.9209	-17.84%
78.1	0.8943	0.8943	0.0000	1.000	0.9709	-8.56%

Appendix 6

Synthetic wash, GC trace for compounds. (Compounds' names and retention time)

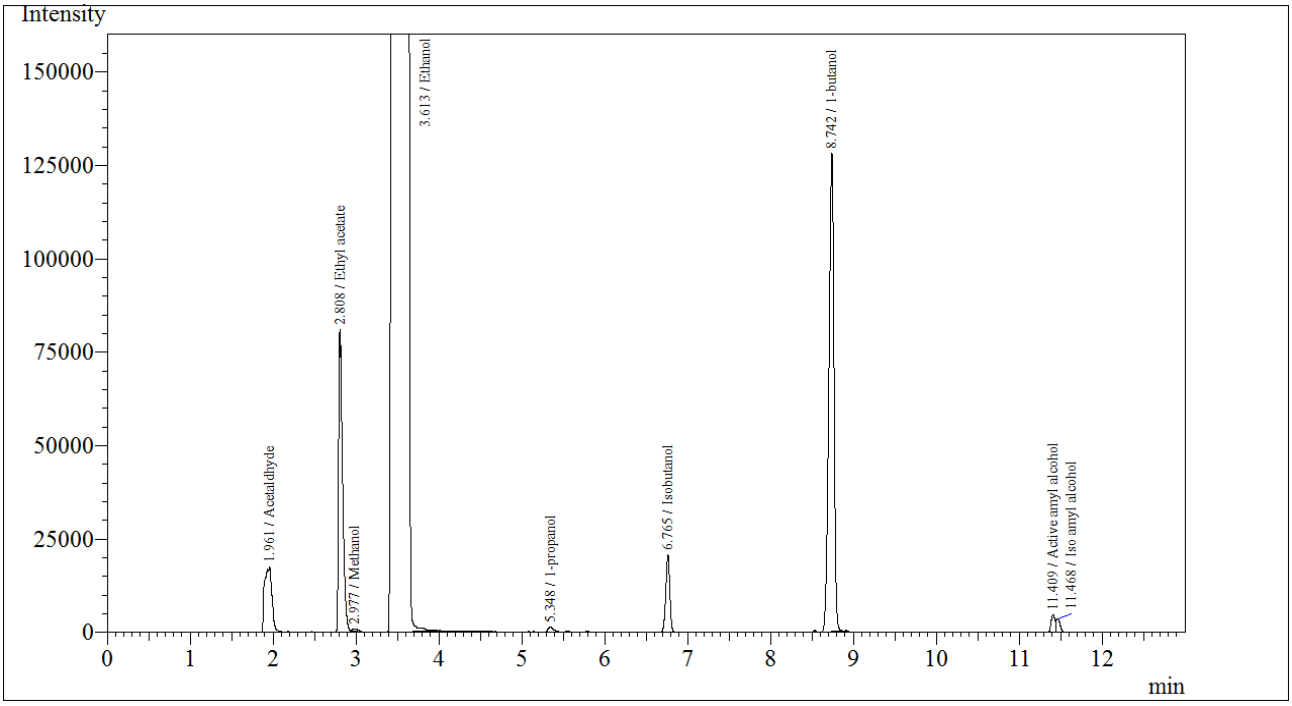

Figure 66 The gas chromatogram of distillate from synthetic wash of T500's first fraction (1st fraction)

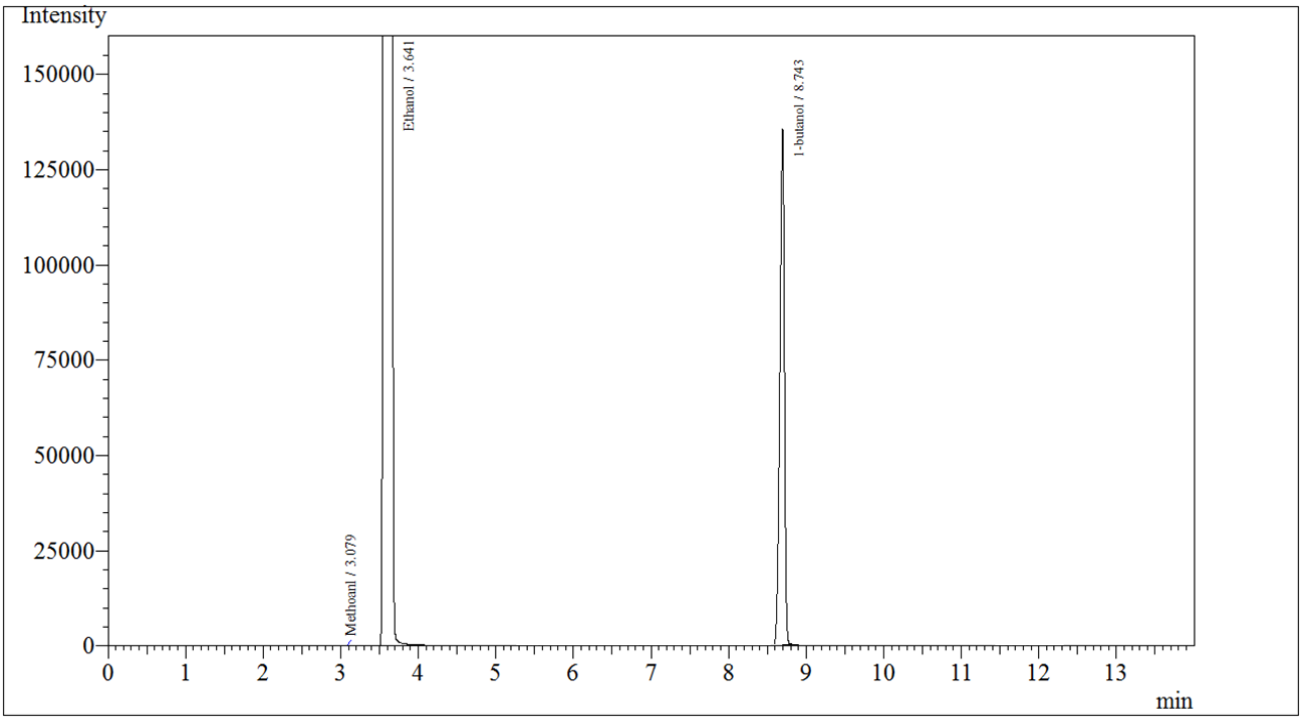

Figure 67 The gas chromatogram of distillate from synthetic wash of T500's last fraction (53rd fraction)

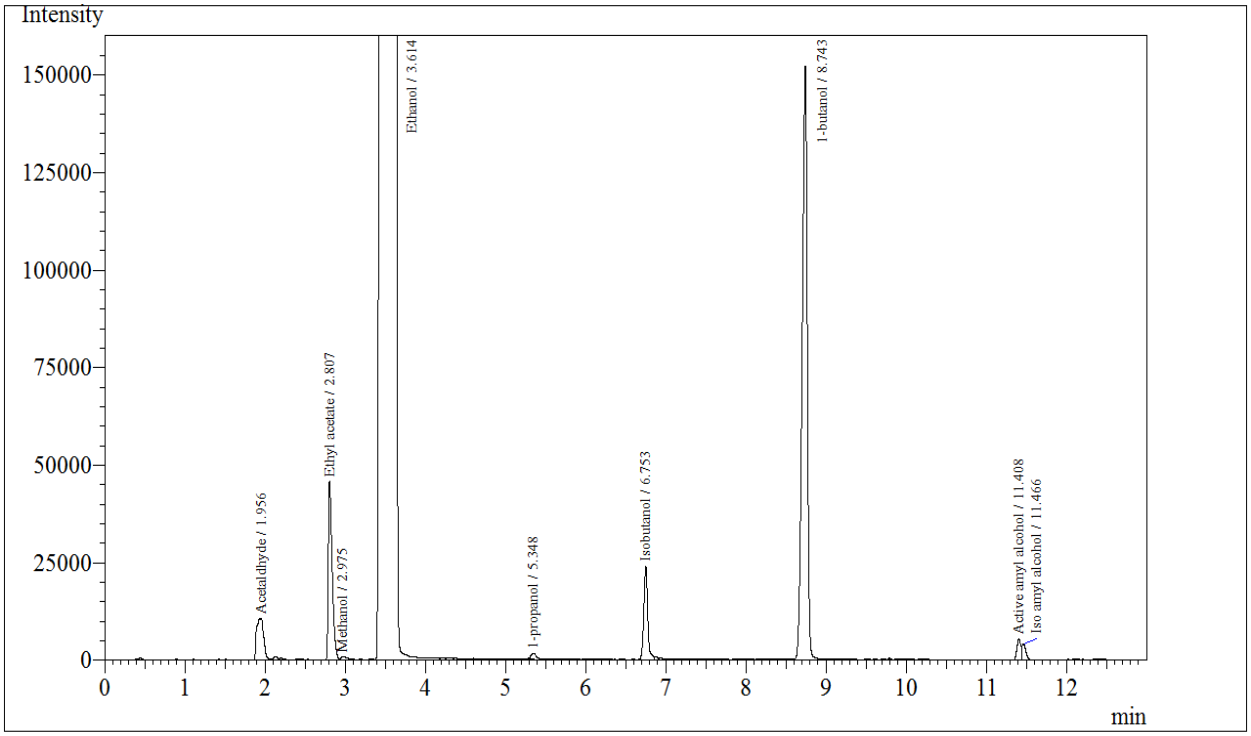

Figure 68 The gas chromatogram of distillate from synthetic wash of essencia's first fraction (1st fraction)

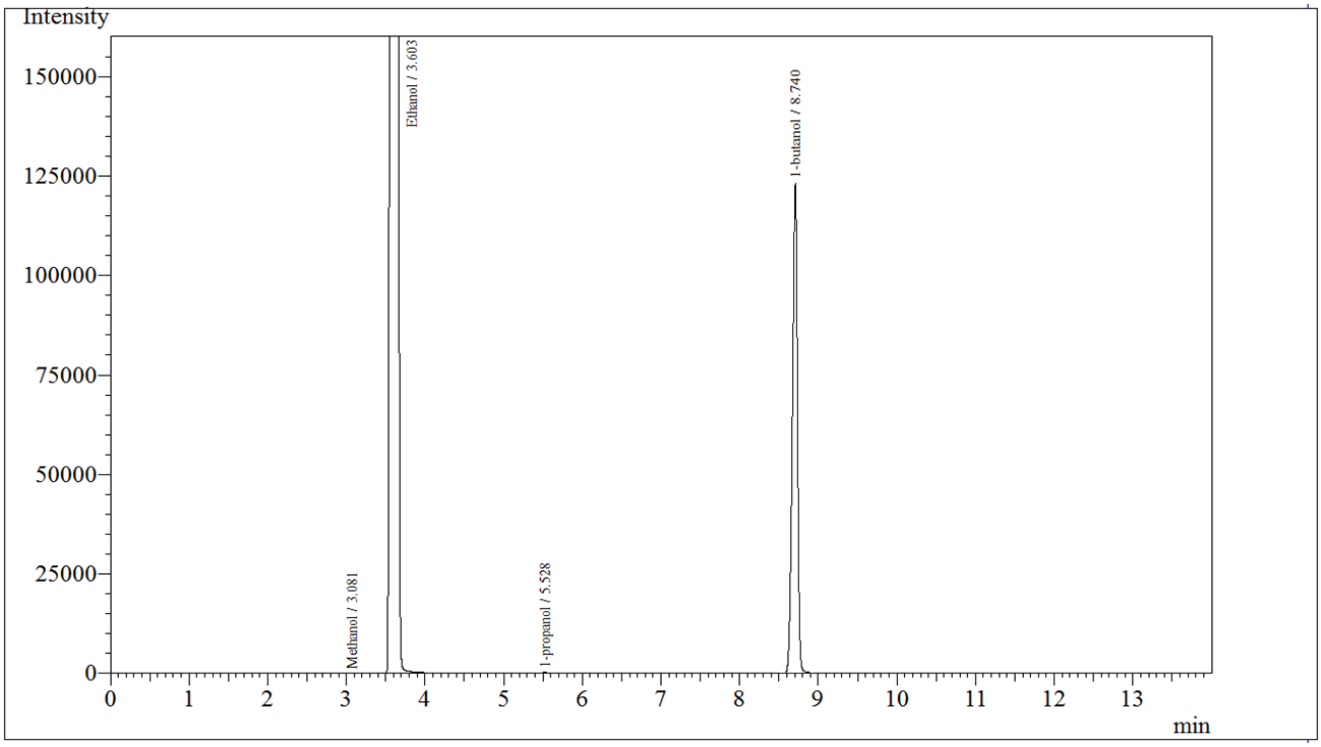

Figure 69 The gas chromatogram of distillate from synthetic wash of essencia's last fraction (60th fraction)

Appendix 7

Table 22 Minimum numbers of correct judgments to establish significance at various probability levels for the triangle test (one-tailed, p=1/3). Retrieved from Roessler, Pangborn, Sidel, and Stone (1978).

No. of trials (n)	Probability levels						
	0.05	0.04	0.03	0.02	0.01	0.005	0.001
5	4	5	5	5	5	5	
6	5	5	5	5	6	6	
7	5	6	6	6	6	7	7
8	6	6	6	6	7	7	8
9	6	7	7	7	7	8	8
10	7	7	7	7	8	8	9
11	7	7	8	8	8	9	10
12	8	8	8	8	9	9	10
13	8	8	9	9	9	10	11
14	9	9	9	9	10	10	11
15	9	9	10	10	10	11	12
16	9	10	10	10	11	11	12
17	10	10	10	11	11	12	13
18	10	11	11	11	12	12	13
19	11	11	11	12	12	13	14
20	11	11	12	12	13	13	14
21	12	12	12	13	13	14	15
22	12	12	13	13	14	14	15
23	12	13	13	13	14	15	16
24	13	13	13	14	15	15	16
25	13	14	14	14	15	16	17
26	14	14	14	15	15	16	17
27	14	14	15	15	16	17	18
28	15	15	15	16	16	17	18
29	15	15	16	16	17	17	19
30	15	16	16	16	17	18	19
31	16	16	16	17	18	18	20
32	16	16	17	17	18	19	20
33	17	17	17	18	18	19	21
34	17	17	18	18	19	20	21
35	17	18	18	19	19	20	22
36	18	18	18	19	20	20	22
37	18	18	19	19	20	21	22
38	19	19	19	20	21	21	23
39	19	19	20	20	21	22	23
40	19	20	20	21	21	22	24
41	20	20	20	21	22	23	24
42	20	20	21	21	22	23	25
43	20	21	21	22	23	24	25
44	21	21	22	22	23	24	26
45	21	22	22	23	24	24	26
46	22	22	22	23	24	25	27
47	22	22	23	23	24	25	27
48	22	23	23	24	25	26	27
49	23	23	24	24	25	26	28
50	23	24	24	25	26	26	28
60	27	27	28	29	30	31	33
70	31	31	32	33	34	35	37
80	35	35	36	36	38	39	41
90	38	39	40	40	42	43	45
100	42	43	43	44	45	47	49

Appendix 8

Table 23 Minimum numbers of agreeing judgments necessary to establish significance at various probability levels for the paired preference test (two-tailed, p=1/2). Retrieved from Roessler et al. (1978).

No. of trials (n)	Probability levels						
	0.05	0.04	0.03	0.02	0.01	0.005	0.001
7	7	7	7	7			
8	8	8	8	8	8		
9	8	8	9	9	9	9	
10	9	9	9	10	10	10	
11	10	10	10	10	11	11	11
12	10	10	11	11	11	12	12
13	11	11	11	12	12	12	13
14	12	12	12	12	13	13	14
15	12	12	13	13	13	14	14
16	13	13	13	14	14	14	15
17	13	14	14	14	15	15	16
18	14	14	15	15	15	16	17
19	15	15	15	15	16	16	17
20	15	16	16	16	17	17	18
21	16	16	16	17	17	18	19
22	17	17	17	17	18	18	19
23	17	17	18	18	19	19	20
24	18	18	18	19	19	20	21
25	18	19	19	19	20	20	21
26	19	19	19	20	20	21	22
27	20	20	20	20	21	22	23
28	20	20	21	21	22	22	23
29	21	21	21	22	22	23	24
30	21	22	22	22	23	24	25
31	22	22	22	23	24	24	25
32	23	23	23	23	24	25	26
33	23	23	24	24	25	25	27
34	24	24	24	25	25	26	27
35	24	25	25	25	26	27	28
36	25	25	25	26	27	27	29
37	25	26	26	26	27	28	29
38	26	26	27	27	28	29	30
39	27	27	27	28	28	29	31
40	27	27	28	28	29	30	31
41	28	28	28	29	30	30	32
42	28	29	29	29	30	31	32
43	29	29	30	30	31	32	33
44	29	30	30	30	31	32	34
45	30	30	31	31	32	33	34
46	31	31	31	32	33	33	35
47	31	31	32	32	33	34	36
48	32	32	32	33	34	35	36
49	32	33	33	34	34	35	37
50	33	33	34	34	35	36	37
60	39	39	39	40	41	42	44
70	44	45	45	46	47	48	50
80	50	50	51	51	52	53	56
90	56	56	56	57	58	59	61
100	61	61	62	63	64	65	67

Appendix 9

Figure 70 Chromatogram of T500's native distillate from the Recipe A (15 compounds were identified)

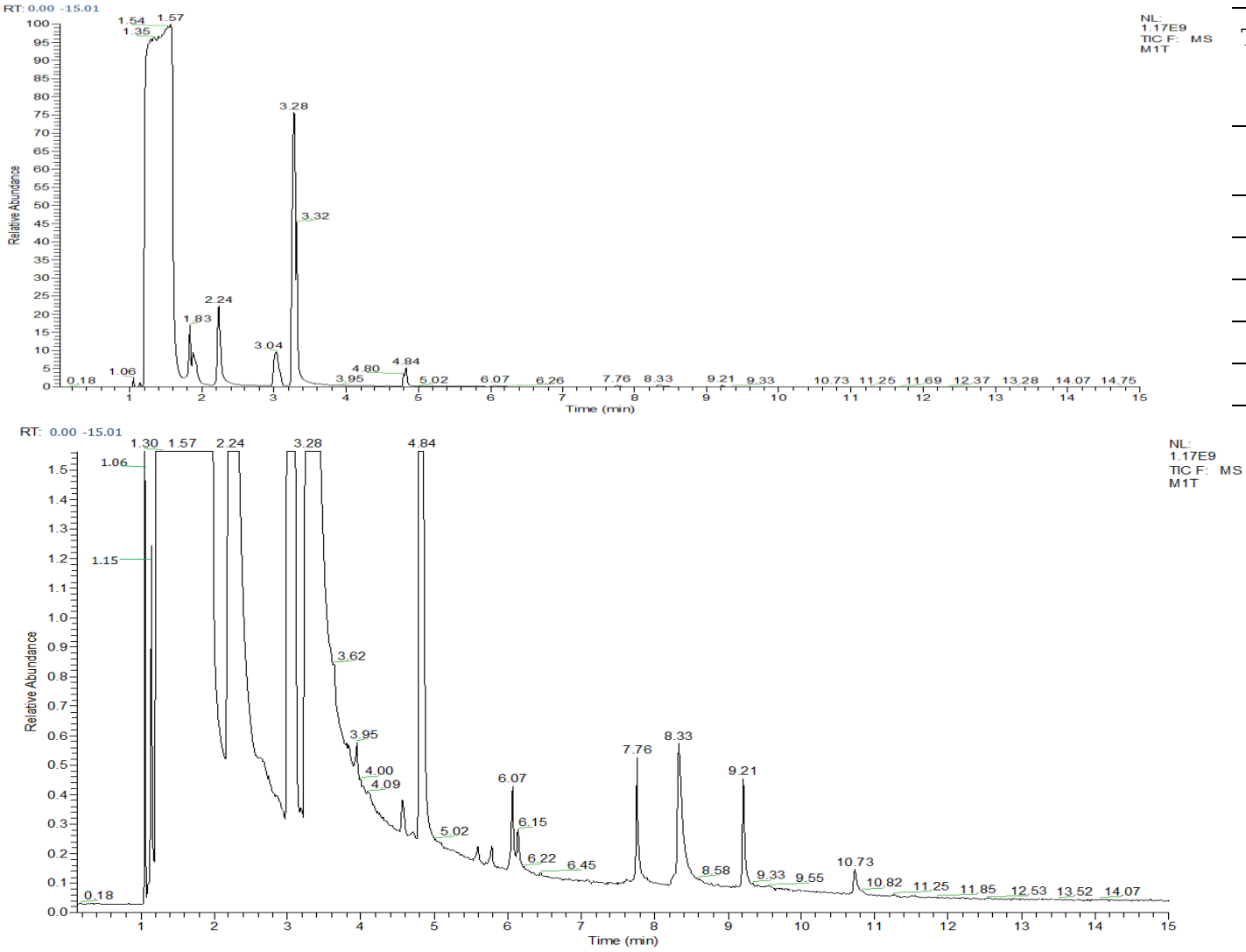

Table 24 Likely identities and retention times for T500's chromatogram (Recipe A)

Retention time (min)	Name
1.15	Acetaldehyde
1.57	Ethanol
1.83	1-Propanol
1.87	Ethyl acetate
2.24	2-Methyl-1-propanol
3.04	1-Butanol
3.28	3-Methyl-1-butanol
3.32	2-Methyl-1-butanol
4.84	3-Methyl-1-butanol acetate
5.78	Pentane
6.07	Hexanoic acid, ethyl ester
7.76	Octanoic acid, ethyl ester
8.33	Phenethyl ester
9.21	Ethyl ester
10.73	Dodecanoic acid, ethyl ester

Figure 71 Chromatogram of essencia's native distillate from the Recipe A (16 compounds were identified)

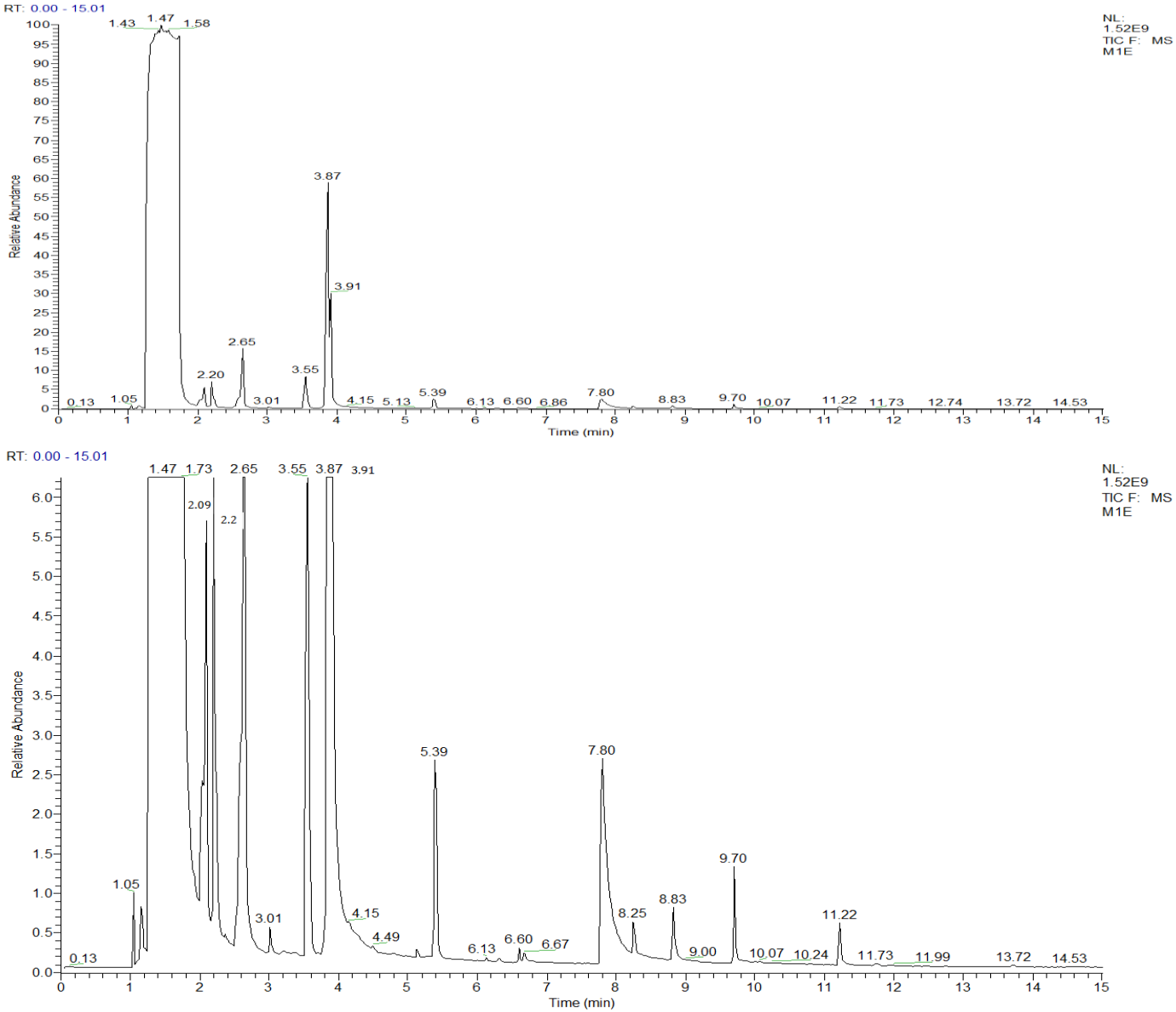

Table 25	Names and retention time for essencia's chromatogram (Recipe A)
Retention time (min)	Name
1.15	Acetaldehyde
1.47	Ethanol
2.09	1-Propanol
2.2	Ethyl Acetate
2.65	2-Methyl-1-propanol
3.01	1-Butanol
3.55	Acetal
3.87	3-Methyl-1-butanol
3.91	2-Methyl-1-butanol
5.39	3-Methyl-1-butyl acetate
6.6	Hexanoic acid, ethyl ester
7.8	2-Phenylethanol
8.25	Octanoic acid, ethyl ester
8.83	Acetic acid, 2-phenylethyl ester
9.7	Decanoic acid, ethyl ester
11.22	Dodecanoic acid, ethyl ester

Figure 72 Chromatogram of T500's native distillate from the Recipe B (12 compounds were identified)

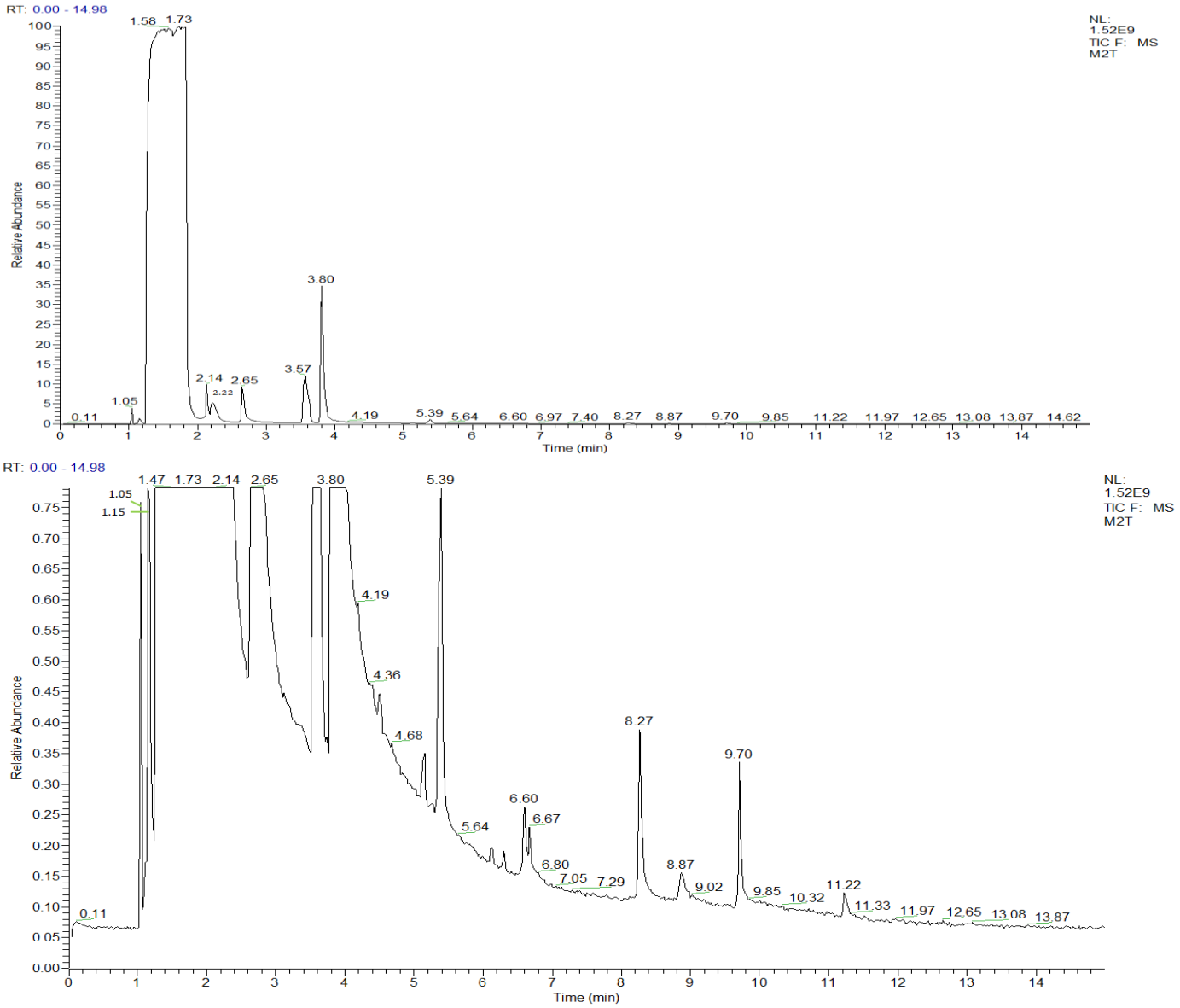

Table 26 Names and retention times for T500's chromatogram (Recipe B)

Retention time (min)	Name
1.15	Acetaldehyde
1.73	Ethanol
2.14	1-Propanol
2.22	Ethyl acetate
2.65	2-Methyl-1-propanol
3.57	Diethyl acetal
3.8	3-methylbutyl ester
5.39	3-Methyl-1-butyl acetate
6.6	Hexanoic acid, ethyl ester
8.27	Octanoic acid, ethyl ester
9.7	Decanoic acid, ethyl ester
11.22	Dodecanoic acid, ethyl ester

Figure 73 Chromatogram of essencia's native distillate from the Recipe B (19 compounds were identified)

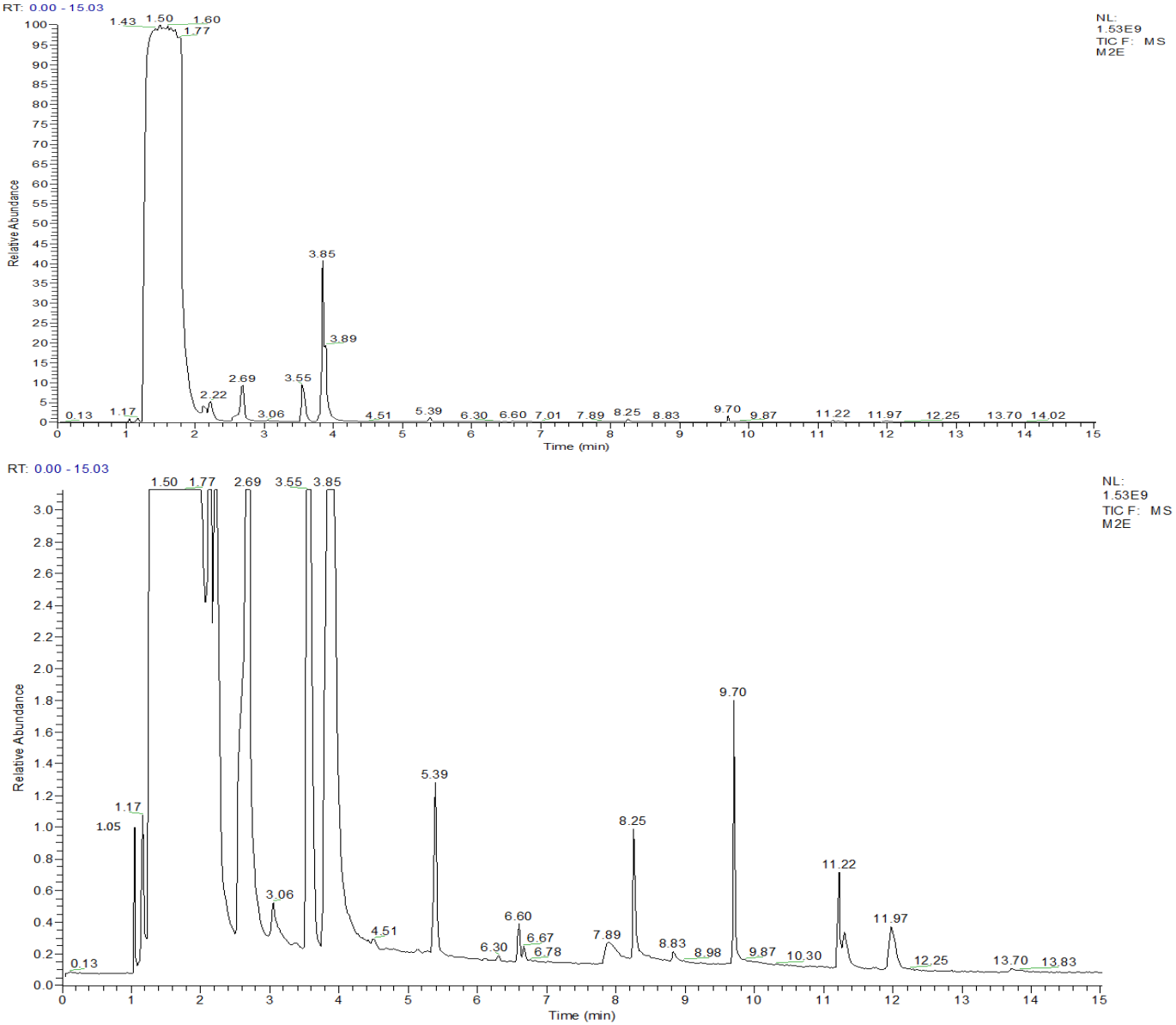

Table 27 Names and retention times for essencia's chromatogram (Recipe B)

Retention time (min)	Name
1.17	Acetaldehyde
1.5	Ethanol
2.12	1-Propanol
2.22	Ethyl Acetate
2.69	2-Methyl-1-Propanol
3.06	1-Butanol
3.55	Acetale
3.85	3-Methyl-1-butanol
3.89	2-Methyl-1-butanol
4.51	Butyric ester
5.39	3-Methylbutyl ester
6.3	1-Ethoxy-1-pentyloxyethane
6.6	Hexanoic acid, ethyl ester
7.89	Phenylethyl ethanol
8.25	Octanoic acid, ethyl ester
9.7	Decanoic acid, ethyl ester
11.22	Dodecanoic acid, ethyl ester
11.31	9-Hexadecenoic acid, ethyl ester
11.97	Hexadecanoic acid, ethyl ester

Appendix 10

Table 28 Results and comments of paired preference test on T500 and essencia's diluted distillate (Recipe A)

Panelists' ID	T500	Essencia	Neither	Gender	Age	Reason (Simply in writing)[1] [2]
5	*			F	<30	T500 is a little strong
6	*			M	<30	(T500) Not strong
9	*			M	<30	(essencia) Less strong alcohol
10	*			M	<30	(T500) Less strong
12	*			M	<30	No reason
13	*			F	<30	(Essencia) organic compounds too strong
19	*			M	<30	(T500) Smells like chocolate
22	*			M	<30	(T500) Stronger
23	*			M	<30	Essencia smells like it woulds kill me
24	*			M	<30	essenci not too strong, T500 is strong
27	*			F	>30	(T500) Besides alcohol smell, still can smell somthing aromatic
29	*			M	>30	(essencia) Less alcohol smell
33	*			M	>30	(essencia) Light alcohol smell
34	*			F	>30	(T500) Sweety smell
36	*			M	>30	(T500) Sweety smell
37	*			F	>30	(T500) Got a sweeter smell
40	*			M	>30	(essencia)not sweet, but stronger smell
46	*			M	>30	(T500) Complex taste
47	*			M	>30	(T500) Less alcohol
2		*		M	<30	Essencia is more fruity, T500 is stronger alcohol smell
3		*		F	<30	Essencia is light ethanol smell, but stron flowerly smell
7		*		M	<30	(essencia) Less intense, T500 smells like alcohol
8		*		M	<30	(essencia) smell less alcohol
11		*		M	<30	(essencia) Strong alcohol
14		*		M	<30	(essencia) Strong alcohol smell
17		*		M	<30	(essencia) Less alcohol, more aromatic
21		*		F	<30	(essencia) Sweet
25		*		M	<30	essencia not strong, T500 too strong
26		*		M	<30	(essencia) Not too strong
28		*		M	<30	(essencia) Stronger
31		*		F	<30	(essencia) mild smell
32		*		M	<30	(essencia) Sweeter
35		*		F	<30	(essencia) Fruity smell
38		*		F	<30	(essencia) Light, mild alcohol
39		*		M	<30	Essencia is lighter, T500 medicine alcohol
41		*		M	<30	(T500) Stronger
42		*		F	>30	(essencia) Strong a little bit
43		*		F	>30	(essencia) More subtle
50		*		M	>30	(essencia) Less strong
1			*	F	<30	No difference
4			*	M	<30	No difference
15			*	M	<30	No difference
16			*	M	<30	No difference
18			*	M	<30	No difference
20			*	M	>30	No difference
30			*	M	>30	No difference
44			*	F	>30	No difference
45			*	M	>30	No difference
48			*	M	>30	No difference
49			*	F	>30	No difference
In total	19	20	11			

[1] Yeallow colour means T500 was claimed stronger alcoholic smell

[2] Blue colour means essencia was claimed stronger alcoholic smell

Table 29 Results and comments of paired preference test on T500 and essencia's diluted distillate (Recipe B)

Panelists' ID	T500	Essencia	Neither	Gender	Age	Reason (Simply in writing)[1] [2]
3	*			M	<30	(Essencia) too strong
5	*			M	<30	(Essencia) too strong
10	*			M	<30	(T500) strong alcohol smell
11	*			M	<30	(T500) Smells like Tequila
13	*			M	<30	(T500) Stronger
15	*			M	<30	(T500) Alcohol smell
21	*			M	<30	No reason
23	*			M	<30	(T500) Smells chocolate
24	*			M	<30	Essencia smells very chemically
25	*			M	<30	(essencia) Fruity taste, colour better peachy
28	*			M	<30	essecia is too Sweeter
30	*			F	<30	(Essencia)too strong
33	*			F	<30	Light, not strong
34	*			F	<30	(T500) Stronger sweety
36	*			F	<30	(T500) Strong alcohol smell
37	*			F	<30	(T500) Strong alcohol smell
41	*			F	<30	(T500) Stronger
46	*			F	<30	T500 is boiled potatoes, rice, dosent like food, make me hungry
1		*		M	>30	(essencia) Stronger
2		*		M	>30	(essencia) Strong
4		*		M	>30	(T500) hospital smell, alcohol too strong
6		*		M	>30	(essencia) Lighter alcohol smell
7		*		M	>30	T500 is intense, essencia is light
8		*		M	>30	(T500) stronger
9		*		M	>30	(essencia) Less strong
12		*		M	>30	T500 has a slightly mousey smell, essencia is floral (murine)
14		*		M	>30	Less alcohol in (Essencia)
16		*		M	>30	Not strong alcohol in Essencia
18		*		M	>30	Mild (essencia)
19		*		M	>30	(essencia) Not as strong as T500
20		*		M	>30	(essencia) Less strong smell
22		*		M	>30	T500 is unpleasant
27		*		M	>30	T500 has unpleasant alcohol smell, I will be sick if I drinks this
29		*		M	>30	(essencia) Less alcohol
31		*		M	<30	(essencia)Light alcohol smell
32		*		M	<30	T500 is too strong, essencia is sweet
35		*		M	<30	(essencia) Stronger
39		*		M	<30	can not identify and prefers the taste from (T500)
40		*		F	<30	(essencia) Doesn't smell alcohol, it smells food/potato chips
42		*		F	<30	Essencia smells less alcohol
43		*		F	<30	T500 smells very strong, I dislike this !
44		*		F	<30	(essencia) Fruit flavour
45		*		F	<30	Essencia is sweet, T500 is just alcohol flavour
47		*		F	<30	(essencia)Not strong
48		*		F	<30	(essencia)Not strong
49		*		F	<30	(T500) Simple smell
50		*		F	<30	(essencia) Nail polish smell
17			*	M	>30	No difference
26			*	M	<30	No difference
38			*	M	<30	No difference
In total	18	29	3			

[1] Yeallow colour means T500 was claimed stronger alcoholic smell

[2] Blue colour means essencia was claimed stronger alcoholic smell