UNIVERSITY OF
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# TOPIC 23: URBAN MINING OF RARE EARTH ELEMENTS FROM RARE EARTH MAGNETS – EVALUATION OF AN EXTRACTION COLUMN DESIGN

MODULE: LABORATORY PROJECT - ENCH4LA

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# STATEMENT OF AUTHORSHIP

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# ABSTRACT

Over the recent years there has been an increasing demand for technological devices as the 4th industrial revolution accelerates. These devices utilize rare earth elements (REE) such as neodymium, europium and samarium which act as vital components in devices such as smartphones and digital cameras. With the fairly short lifespan of these devices, the waste electronic and electrical equipment (WEEE) are disposed of in landfill sites. Thus, WEEE forms one of the fastest growing waste streams globally. Therefore, it is imperative that methods of recycling WEEE are researched and implemented. Liquid- liquid extraction (LLE) is one method that can be used to recycle the metal present in WEEE.

Liquid-liquid extraction involves the use of a solvent to extract a solute from a binary mixture and can be performed using various types of extraction devices. This work focused on the use of a vibrating plate extraction column (VPE) to perform liquid-liquid extraction. This extraction equipment was chosen since it can operate at higher frequencies and lower amplitudes than other equipment. It was originally required that the efficiency of the VPE column should be investigated when removing the valuable rare earth metal neodymium from an aqueous solution. However, due to the limited availability of neodymium, a different system of ethanol-cyclohexane-water was chosen. This system was assumed to adequately replicate the expected column behavior for the rare earth metal system. The aim of this research was to re-commission the VPE column and to investigate the performance of the VPE column using the cyclohexane-water-ethanol system

Four experimental runs were performed, whereby the VPE column was operated in batch mode for two runs and semi-batch mode for the other 2 runs. The samples obtained during experimental runs were analyzed using a gas chromatograph. The effect of frequency, solvent to feed ratio and the mode of operation on the effectiveness of separation and the percentage of ethanol extracted were investigated. The number of equilibrium stages for separation was also obtained by stepping off on the ternary diagram for the system. Results indicated that using a higher frequency for the batch runs, of approximately 15 Hz, allowed for better mixing with a larger percentage of ethanol extracted (64.268%). Likewise, a higher solvent to feed ratio, which was used for semi-batch run 2b, allowed more solvent to be available to perform the extraction and thus led to a higher percentage of ethanol extracted (83.360%). For all runs, less than 1 equilibrium stage was obtained for the separation. It was deduced that operating the column in semi-batch mode allowed for efficient contacting of the phases due to the presence of a recycle stream and was thus a more effective mode of operation than batch mode.

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# NOMENCLATURE

| Symbol | Definition | Units |
|---|---|---|
| $A_i$ | Peak area of component *i* from gas chromatograph | Dimensionless |
| E | Extract mass or flow rate | kg or kg/h |
| F | Feed mass or flow rate | kg or kg/h |
| $m_i$ | Mass of component *i* | kg |
| $n_i$ | Number of moles of component *i* | mols |
| R | Raffinate mass or flow rate | kg or kg/h |
| S | Solvent mass or flow rate | kg or kg/h |
| $x_i$ | Mass fraction | Dimensionless |

# CHAPTER 1: INTRODUCTION

Rare earth elements (REE) are a group of 17 metals with unique properties. These elements are difficult to mine since their concentrations in ore are not economically viable, however there is an abundance of rare earth deposits on the earth's surface (Somaran, 2013). The mining of rare earth metals leads to various environmental issues, such as soil erosion and acidification (Ives, 2013). REE are utilized as components in electrical devices, since it has unique properties that are crucial in the proper functioning of these devices. For instance, europium is a REE that is utilized in television sets to produce images in colour (Somaran, 2013).

The majority of global REE deposits are located in China, which forms 36% of the world's deposits (Bunting, 2018). China is imposing restrictions worth $110 billion on exports of REE to other countries (Morrison, 2019). The explosion in the demand of REEs, accompanied by the tariff hikes imposed by China, has lead to an increase in the price of REEs globally. The increase in demand for REEs globally is shown in Figure 1-1.

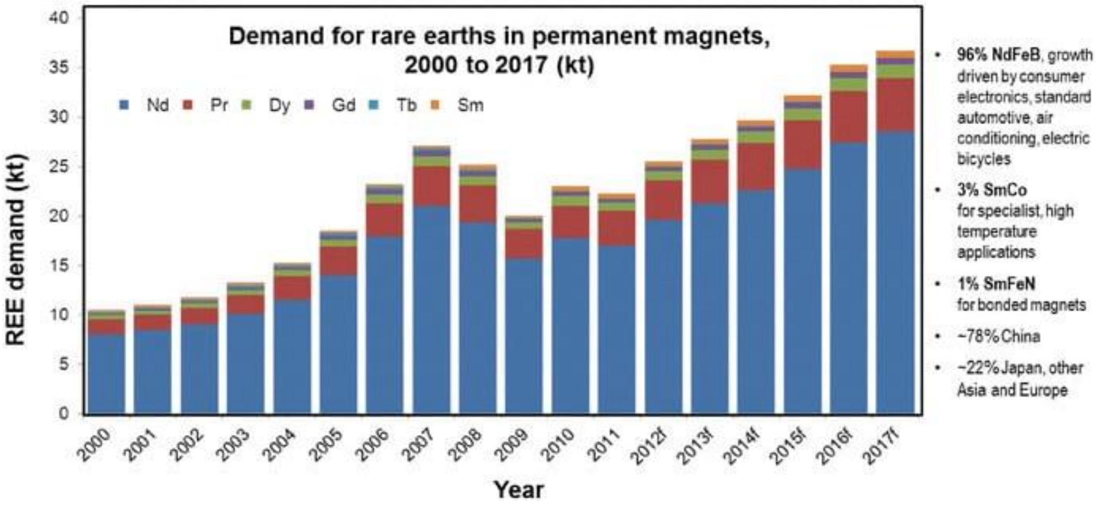

**Figure 1-1: Increase in the demand of REE in recent years (Walton, et al., 2017)**

Rare earth elements are also mined in South Africa at the Steenkampskraal mine, which is located in the Western Cape (NS Energy, 2020). The mine has the highest grade of REEs globally. The most valuable metal mined at this mine is neodymium, which was valued at $49 763 per metric ton in 2020 (Garside, 2021). Neodymium can be alloyed with iron and boron to form very strong permanent magnets, which can be used in a variety of electrical devices such as loudspeakers, mobile phones and computer hard drives (Menad & Seron, 2017).

In this digital age, there is an increasing demand for technological devices such as computers and mobile phones. This has subsequently resulted in the large-scale production of electrical and electronic equipment, which are disposed of in landfill sites upon reaching the end of their lifespan (Tuncuk, et al., 2012). Waste electronic and electrical equipment (WEEE) is one of the fastest growing waste streams globally and has drastic environmental impacts since toxic substances from WEEE can leak into the water supply (Vaccari, et al., 2019)

In recent years, there has been a spike in the amount of WEEE generated globally, with 20-50 million metric tons of WEEE generated in the world annually (Tuncuk, et al., 2012). Since August 2016, light bulbs have been restricted from being disposed of in landfill sites in South Africa, while the prohibition on the disposal of other types of WEEE in landfill sites is effective as of 2021 (EnviroServ Waste Management, 2021). WEEE can be utilized as a secondary source of rare earths, due to the high content of metals used in electrical equipment. Therefore, it is crucial that methods of recycling WEEE are researched and implemented, which also allows the reuse of valuable rare earths such as neodymium. Methods of recycling WEEE include the use of gravity, magnetic or electrostatic separation to remove rare earths from other components (Tuncuk, et al., 2012).

One attractive option of recycling WEEE is liquid-liquid extraction (LLE), also known as solvent extraction. Liquid-liquid extraction is a unit operation that involves the use of a solvent to extract a solute from a binary mixture (Seader & Henley, 2011). When thermodynamic equilibrium between the phases is reached, the extraction process is completed (Naidoo, 2012). LLE can be used to extract valuable rare earth metals from waste mixtures using equipment such as extraction columns.

LLE is an important separation method in various chemical industries, such as petroleum, metallurgy and waste management (Usman, et al., 2008). It is preferred due to the low requirement of energy to perform the separation (Usman, et al., 2008), as heat is generally not required to affect the separation. Previous work by Gruber and Carsky (Gruber & Carsky, 2020) focused on the extraction of REEs from magnets using LLE. Those results showed that a purity of 99% was obtainable, highlighting LLE as a viable method of extracting rare earths.

To improve the separation efficiency in LLE processes, the use of vibrating plate columns have been proposed in literature. Prochazka and his co-workers (Lo & Prochazka, 1983) designed the first vibrating plate extraction column (VPE) which operates via the reciprocating movement of an assembly of sieve plates to improve mixing and mass transfer. This type of column exhibits a plate design that has perforations to allow for the flow of the dispersed phase, and downcomers for the flow of the continuous phase. The VPE plates are advantageous over other plates, since these plates can be utilized at higher frequencies and lower amplitudes (Lo, et al., 1992). Previous experimental work was performed on the VPE column by (Naidoo, 2012) and (Rathilal, 2010) to investigate the effect of solvent- to – feed ratio and agitation level on the performance of the column.

This work originally required the efficiency of the VPE column to be investigated when removing the valuable rare earth metal neodymium from an aqueous solution of nitric acid and kerosene. However, due to the limited availability of neodymium, and the column requiring a re-commissioning after prolonged storage, a different system of ethanol-cyclohexane-water was chosen, which is assumed to adequately replicate the expected column behavior for the rare earth metal system.

This system was chosen due to the availability and low costs of the chemicals, as well as the extensive range of LLE data available. These chemicals are also not highly toxic and corrosive, and thus the materials of construction of the column were not affected (Joshi & Adhikar, 2019). This system of cyclohexane, water and ethanol was used to commission the column for further experiments involving the extraction of neodymium that is part of an ongoing research project at the Thermodynamics Research Unit.

## 1.1.Aim and objectives

The aim of this research was to re-commission the VPE column and to investigate the performance of the VPE column using the cyclohexane-water-ethanol system. The objectives included:

i. Cleaning, leak tests, reconnecting electrical components of the VPE column and evaluating the operability of the column
ii. Investigating the effect of crucial parameters, such as solvent-to-feed ratio and agitation level, on the performance of the column via measurements with the cyclohexane, ethanol and water system.

Various experimental runs were performed where the column was operated in batch and semi-batch mode, and different key parameters were altered for each run. Samples were removed from the column and gas chromatography was utilized to obtain the composition of components in the streams/phases.

# CHAPTER 2: THEORETICAL BACKGROUND

This chapter presents a concise view on the fundamentals of liquid-liquid extraction and extraction equipment used at bench and pilot scale.

## 2.1. Liquid – Liquid Extraction (LLE)

Liquid- liquid extraction, also known as solvent extraction or partitioning, is the method of removing a solute from a mixture by contacting it with a solvent in which the solute is miscible. Liquid- liquid extraction, along with distillation, form two of the most important industrial separation processes (Thornton, 1992). When thermodynamic equilibrium between the phases is reached, the transfer of solute is terminated. Liquid-liquid extraction processes are performed at room temperature and atmospheric pressure (Humphrey & Keller, 1997).

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Liquid-liquid extraction systems consist of the following constituents (Rathilal, 2010):

- Solute – the material that is removed from the carrier by the solvent. It is miscible with the solvent and carrier.
- Solvent- the fluid that removes solute from carrier. It is immiscible/ partially immiscible with the carrier.
- Carrier – the fluid that remains after the solute has been extracted.

The feed which enters liquid-liquid extraction equipment contains the solute and the carrier. The phases exiting the liquid-liquid extraction columns are the extract and raffinate phases. The extract phase is the liquid phase that contains the removed solute, while the raffinate phase is the exiting liquid phase that is carrier- rich (Seader & Henley, 2011). The light phase, which is less dense, travels to the top of the extraction column where it accumulates (Rathilal, 2010). The heavy phase, which is denser, flows to the bottom of the column where it accumulates (Rathilal, 2010). The dispersed phase is the phase that forms due to the splitting up of an intact phase of liquid into droplets (Visual Encyclopedia of Chemical Engineering, 2020), while the continuous phase is the bulk fluid phase that flows without the splitting of the liquid into droplets (Rathilal, 2010).

## 2.2. Applications of LLE

Industrial applications of LLE include the use of LLE to recover heavy metals, separate aromatics from hydrocarbons, purify antibiotics and for metallurgical purifications (Talebi, et al., 2018). Liquid-liquid extraction can be a valuable alternative when processes such as distillation, evaporation and crystallization are too expensive or are unfeasible (Rathilal, 2010). ✓ It is preferred over distillation for the following separations (Seader & Henley, 2011):

- Separation of liquids that have close boiling points or close melting points
- Separation of liquids that have a low relative volatility
- Separation of azeotropic mixtures
- Separation of heat- sensitive materials
- Separation of mixtures according to chemical type instead of relative volatility
- Extraction of contaminants that are available in low concentrations

## 2.3.Advantages and Disadvantages of LLE

The following advantages of LLE were obtained from (Humphrey & Keller, 1997):

- LLE can be used for the separation of components that have close boiling points or that form azeotropes.
- LLE can be used to separate heat sensitive components, such as in pharmaceutical industries, since the process occurs at ambient temperatures.
- The separation of large volumes of liquid can occur without the consumption of large amounts of energy.
- LLE can be used for separation according to chemical type, instead of relative volatility.
- Many chemical methods use up reagents, thus LLE may be a more cost-effective alternative for these methods.
- LLE is an attractive substitute to distillation under high vacuum and low temperature conditions
- LLE can be utilized in the separation of materials with high boiling impurities, such as in aqueous solutions.

The following disadvantages of LLE were obtained from (Humphrey & Keller, 1997):

- There are additional cost expenses since the solvent will need to be recovered using a solvent-recovery system.
- The final product may be contaminated since it contains the solvent.
- Due to the requirement that the solvent must be immiscible with the original solution, the solvent chosen will be chemically different and thus expensive materials of construction may be utilized to prevent corrosion.
- Large volumes of solvent are required for efficient mass transfer, which may increase capital costs.
- There is also a requirement of solvent storage tanks and distribution systems.

## 2.4.Counter-current liquid-liquid extraction

The feed and the solvent enter extraction columns or equipment from opposite sides, otherwise known as counter-current extraction, which thus leads to intense contact between the material in the feed and solvent (Seader & Henley, 2011). The solvent can be a pure component or a mixture. This also leads to improved mass transfer and efficiency. Figure 2-1 indicates the counter-current extraction process that is used in various chemical processes.

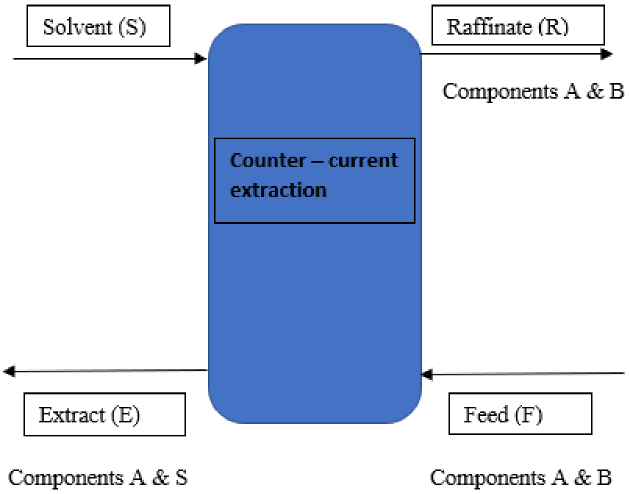

**Figure 2-1: Counter-current liquid-liquid extraction process (adapted from (Seader & Henley, 2011))**

## 2.5.Classification of extractors

The methods used for dispersing phases and the creation of counter-flow arrangements can be employed to classify extractors. The different phases can contact either by gravity or by centrifugal force (Lo & Baird, 1994). Figure 2-2 shows the categorization of different extraction columns.

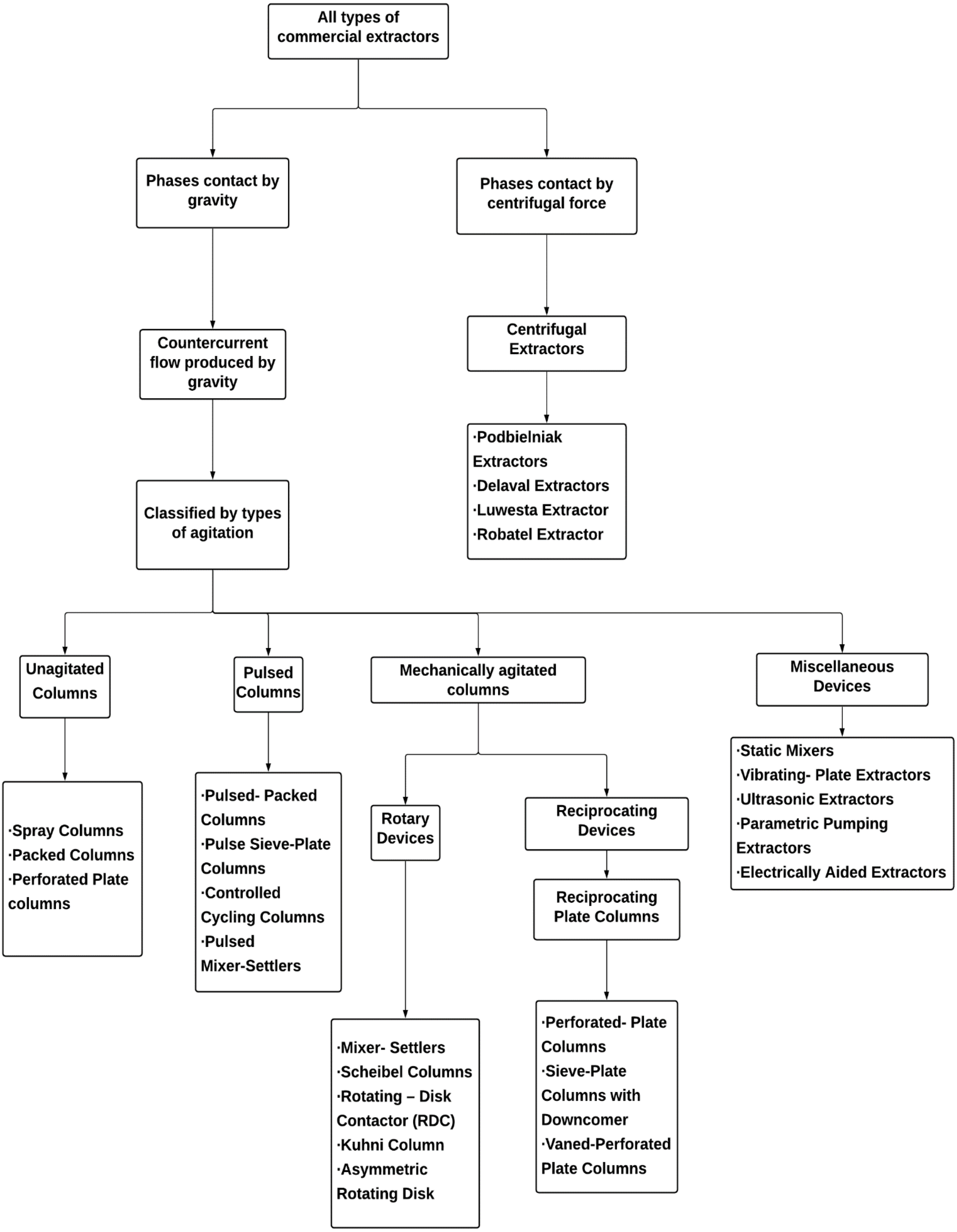

**Figure 2-2: Classification of commercial extractors (taken from (Lo & Baird, 1994))**

## 2.6. Comparison of the different extractors

The following advantages and disadvantages of different extractors are obtained from (Seader & Henley, 2011) and (Naidoo, 2012):

***Table 2-1: Comparison of the different extractors***

| Type of equipment | Advantages | Disadvantages |
|---|---|---|
| Mixer - Settlers | • Efficient contacting<br>• Allows for wide flow ratio<br>• Optimal flexibility<br>• Able to operate with high viscosity liquids<br>• Many stages available<br>• High-stage efficiency | • Large holdup<br>• Large floor space required<br>• High power costs<br>• High investment<br>• Requirement of interstage pumping |
| Centrifugal extractors | • Low holdup capacity<br>• Short holdup time<br>• Small floor space<br>• Operates with low density difference between phases<br>• Small amounts of solvent required | • High operating costs<br>• High start-up costs<br>• Maintenance is costly<br>• Limited stages in a single unit (between 2-7 stages) |
| Continuous, counterflow contactors (no mechanical agitation) | • Low initial cost<br>• Low operating cost<br>• Simple construction | • Limited throughput with low density difference<br>• Cannot operate with high flow ratios<br>• Difficult scale-up<br>• Low efficiency depending on operating conditions<br>• High headroom |
| Continuous, counterflow contactors (mechanical agitation) | • Relatively low cost<br>• Many stages possible<br>• Good dispersion | • Cannot operate with high flow ratios<br>• Limited throughput with low density difference<br>• Unable to handle emulsifying systems |

## 2.7. Description of the vibrating plate extraction (VPE) column

Reciprocating plate extraction columns (RPC) function by moving an assembly of sieve plates which forms a reciprocating motion (Humphrey & Keller, 1997). Three different types of RPCs are used in industrial processes, which were created by the Union of Soviet Socialist Republics. These include the Karr column, the vibrating plate extraction column and the vaned-perforation plate column (Dijk, 1935).

The vibrating plate extraction (VPE) column was created by Prochazka and his team in Czechoslovakia in 1971 (Prochazka, et al., 1971). This type of reciprocating plate extraction column exhibits a plate design which consists of small perforations for the dispersed phase to flow through, and downcomers for the flow of the continuous phase (Lo, et al., 1992). The VPE consists of a stack of plates that are installed on a shaft which is manoeuvred by a vibrating motor to form reciprocating movements, that allows the system in the column to mix (Lo, et al., 1992). VPE plates can function at higher frequencies and lower amplitudes than other RPCs. The VPE column is most similar to a continuous, counterflow contactor with mechanical agitation.

## 2.8. Advantages and disadvantages of the VPE column

The advantages of a vibrating plate extraction column are indicated as follows (Prochazka, et al., 1971):

- The VPE can handle high flow rates, which leads to an increased efficiency.
- Easy scale-up of the VPE.
- Construction and maintenance are simple.
- The column can be modified to handle a variety of system properties.
- Capable of functioning in both the mixer-settler regime and in the emulsion regime.
- Segmental downcomers permit larger throughput.

The disadvantages of a vibrating plate extraction column are indicated as follows (Prochazka, et al., 1971):

- Since the column is susceptible to impurities, it is not ideal for liquid mixtures containing solids.
- Axial mixing is increased with greater energy requirements, which minimizes the column's total effectiveness.
- Entrainment issues may occur due to small droplets.

- Temperature control is difficult due to the height of the column. Large amounts of energy would be required for temperature control.

### 2.9.Modes of Operation

The VPE column can be operated in the following modes (Seader & Henley, 2011):

- Batch: material enters the VE column at the beginning and is only removed at the end of the run.
- Semi-batch: material periodically enters the VPE column and material periodically exits the column. This can include a recycle stream.
- Continuous mode: material flows into the VPE column via the feed and solvent streams and material is removed from the VPE column continuously via the extract and raffinate streams.

### 2.10.Equations

#### Mass balance on extraction column

When the column is operated in continuous mode, feed (F) and the solvent (S) are pumped into the column, while the extract (E) and the raffinate (R) exit the column. The mass balance on the extraction column is shown as follows (Seader & Henley, 2011):

$$\mathrm{F} + \mathrm{S} = \mathrm{E} + \mathrm{R} \tag{1}$$

The following equation indicates the ethanol balance over the extraction column:

$$\mathrm{x}_{\text{ethanol,feed}}\,\mathrm{F} = \mathrm{x}_{\text{ethanol,extract}}\mathrm{E} + \mathrm{x}_{\text{ethanol,raffinate}}\mathrm{R} \tag{2}$$

#### Percentage of solute extracted

The percentage of solute extracted can be calculated as follows (Seader & Henley, 2011):

$$\%\ \text{solute extracted} = \frac{\mathrm{x}_{\text{feed}}\,(\mathrm{n}_{\text{feed}}) - \mathrm{x}_{\text{raffinate}}(\mathrm{n}_{\text{raffinate}})}{\mathrm{x}_{\text{feed}}(\mathrm{n}_{\text{feed}})}\ \text{x}\ 100 \tag{3}$$

Where $x_{feed}$ is the fraction of solute in the feed, and $x_{raffinate}$ is the fraction of solute in the raffinate.

### 2.11. Parameters affecting efficiency and separation of the VPE column

The following parameters affect the rate of mass transfer, separation and hence efficiency of the VPE (Rathilal, 2010):

- Temperature
- Solvent-to-feed ratio
- Agitation level
- Solvent selection
- Physical properties of the components of the system (density, viscosity, etc.)

### 2.12. Solvent selection

The amount of solute that is extracted is dependent upon the solvent that is selected, and this therefore affects the extent and efficiency of the column. It was required that the solvent chosen should fulfil the following criteria (Humphrey & Keller, 1997):

- The solvent chosen should be immiscible with the carrier but should be miscible with the solute. This prevents the need of having a further separation system to recover the solvent from the raffinate.
- The solvent should be widely available and should have a reasonable cost, since large volumes of solvent may be required for extraction of the solute. This would also minimize costs of replacing solvent due to losses.
- The solvent should not be highly flammable or toxic, for health and safety considerations.
- The solvent should not be highly corrosive as this would increase costs of material of construction for the column.
- A significant density difference between the extract and raffinate phases would allow for higher capacities to be obtained. The solvent should not have a high viscosity since this can lead to subsequent problems in pumping and dispersion, which decreases the rate of mass transfer.
- A high value of the distribution coefficient is required, since this signifies that the solvent has a high affinity for the solute which improves the performance of the column.
- The solvent, carrier and solute must produce a high separation factor since this reduces the number of equilibrium stages required. The separation factor, or selectivity, is defined as the ratio of the activity coefficients of the solute- solvent and carrier-solvent pairs at infinite dilution. ✓

## 2.13.Types of ternary systems

The Gibbs equilateral triangle is used for the plotting of the ternary diagrams in this work. The corners of the diagram indicate the pure constituents of the system, while the edges show the binary mixtures of the components. The points within the triangle charaterize the mixture of all 3 components (Thornton, 1992). The most common ternary system is the Type I system shown in Figure 2-3. This was the type of system considered in this work. Outside the phase envelope, there is only one phase; however, within the phase envelope there are 2 liquid phases. This system occurs when the carrier and the solvent are immiscible, and there is complete miscibility between the carrier-solute and solvent-solute pairs. Type I systems have a plait point where the tie-lines converge and where the two liquid phases have similar compositions (Seader & Henley, 2011). The plait point is a critical point at which no separation can be obtained using those compositions (Humphrey & Keller, 1997). The tie-lines on the diagram are used to connect the extract and the raffinate phases and their intersection with the binodal curve indicate the compositions for the liquid-liquid phases upon splitting.

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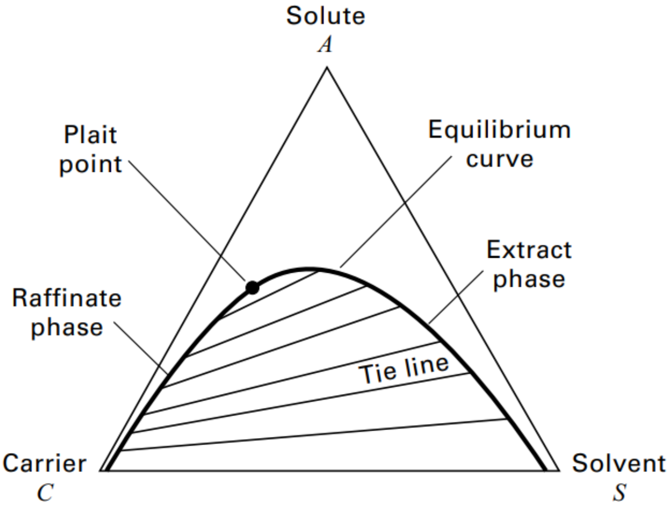


**Figure 2-3: Ternary liquid-liquid Type I system (Seader & Henley, 2011)**

The type II system depicted in Figure 2-4 indicates immisicibility between the solute and solvent (Humphrey & Keller, 1997). It was required that for viable liquid-liquid extraction processes to occur, the compositions of the ternary system had to be within the two phase region (Humphrey & Keller, 1997).

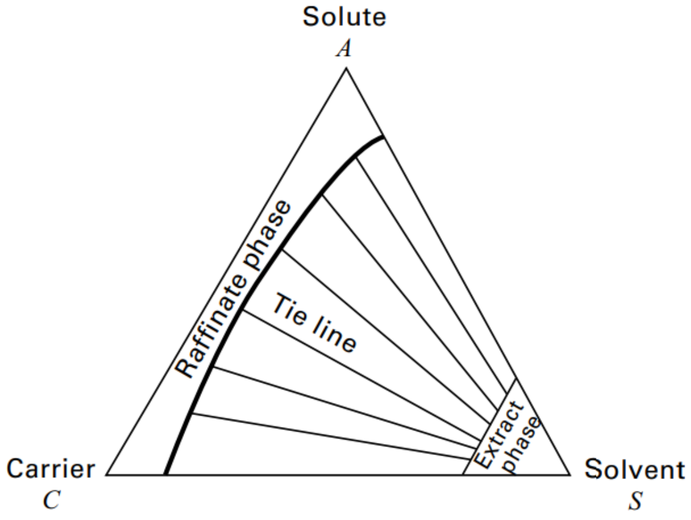
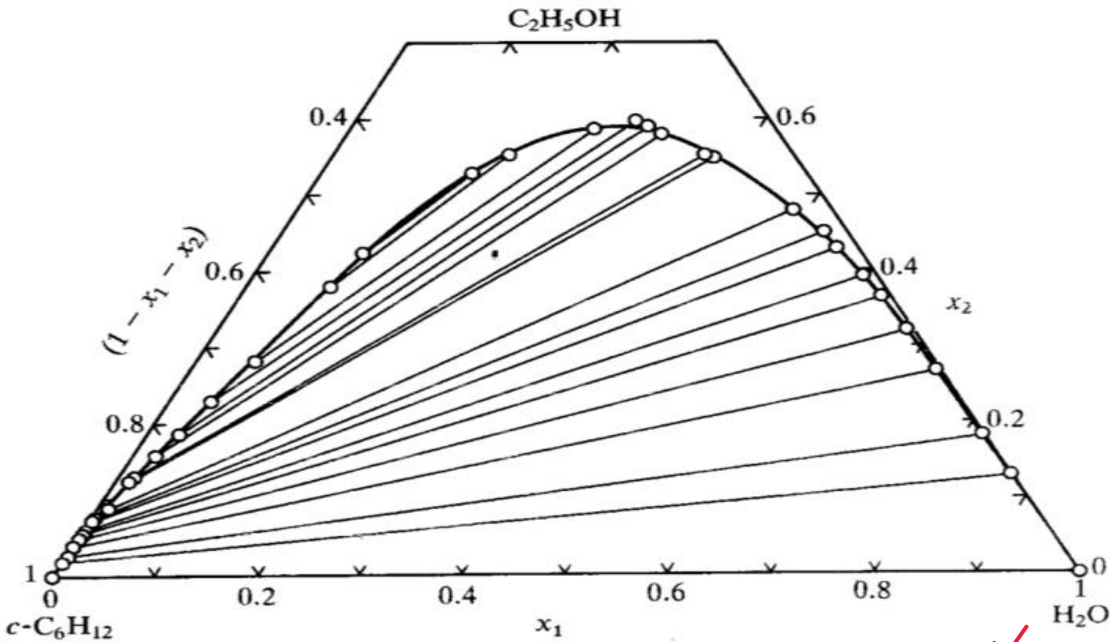


**Figure 2-4: Ternary liquid-liquid Type II system (Seader & Henley, 2011)**

As mentioned, the ethanol-cyclohexane-water system utilized in this work is a type I system. The ternary diagram for the system is depicted in Figure 2-5. $x_1$ represents the mole fraction of water, $x_2$ represents the mole fraction of ethanol and $(1 - x_1 - x_2)$ represents the mole fraction of cyclohexane (Moriyoshi, et al., 1991). Water has a higher selectivity for ethanol than cyclohexane, therefore water was chosen as the solvent.



✓

**Figure 2-5: Ternary phase diagram for the ethanol-cyclohexane-water system in mole fractions at 298.15K and 0.1 MPa (Moriyoshi, et al., 1991)**

# CHAPTER 3: EXPERIMENTAL WORK

## 3.1.Experimental system

The cyclohexane-ethanol-water system was chosen due to the availability and low costs of the chemicals, as well as the extensive range of LLE data available. These chemicals are also not highly toxic and corrosive, and thus the materials of construction of the column were not affected. Ethanol was the component that was extracted. Cyclohexane and water are immiscible due to the difference in polarity of the cyclohexane and water molecules. Water has stronger hydrogen bonds and is polar, while cyclohexane is non-polar and has weaker London dispersion forces (Moriyoshi, et al., 1991). According to the ternary diagram in Figure 2-5, water was more efficient in extracting ethanol than cyclohexane, therefore water was chosen as the solvent and cyclohexane was the carrier. Table 3-1 shows the properties of the system components.

**Table 3-1: Properties of the system components**

| | Cyclohexane | Ethanol | Deionized Water | Acetone |
|---|---|---|---|---|
| Chemical formula | $C_6H_{12}$ | $C_2H_5OH$ | $H_2O$ | $C_3H_6O$ |
| CAS no. | 110-82-7 | 64-17-5 | 7732-18-5 | 67-64-1 |
| Molar mass (g/mol) | 84.16 | 46.07 | 18.02 | 58.08 |
| Density (g/cm$^3$) | 0.780 | 0.790 | 0.998 | 0.784 ✓ |
| Purity (wt%) | ≥ 99.5% | ≥ 99.5% | - | ≥ 99.5% |
| Supplier | Honeywell Riedel-de Haën | Sigma- Aldrich | Elga | Sigma- Aldrich |

The conductivity of deionized water used in experiments was 0.069 μS/cm.

### 3.2.Experimental set-up

The experimental set-up of the VPE column is depicted in Figure 3-1 and Figure 3-2.

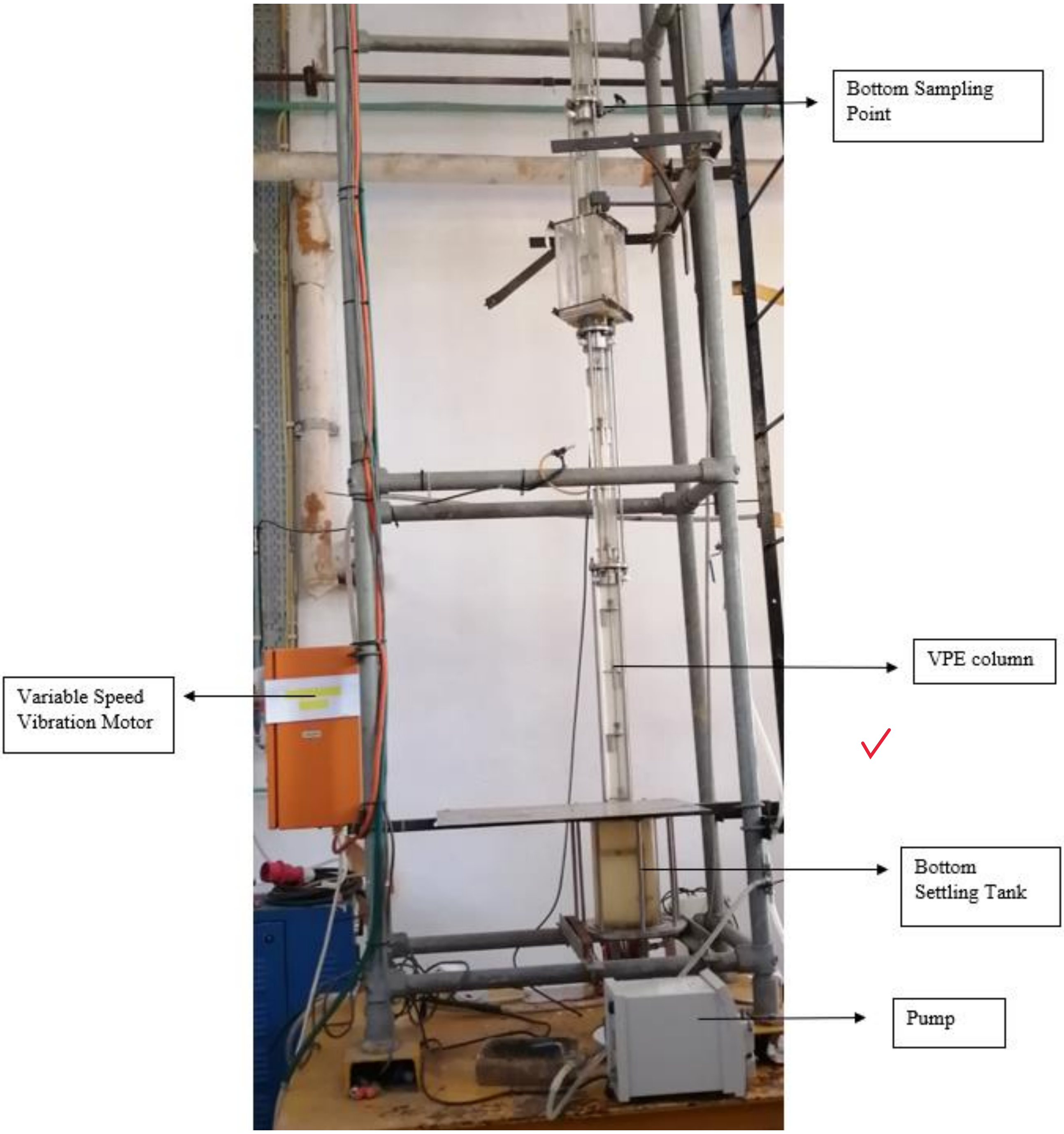


**Figure 3-1: Experimental set up of the bottom of the VPE column**

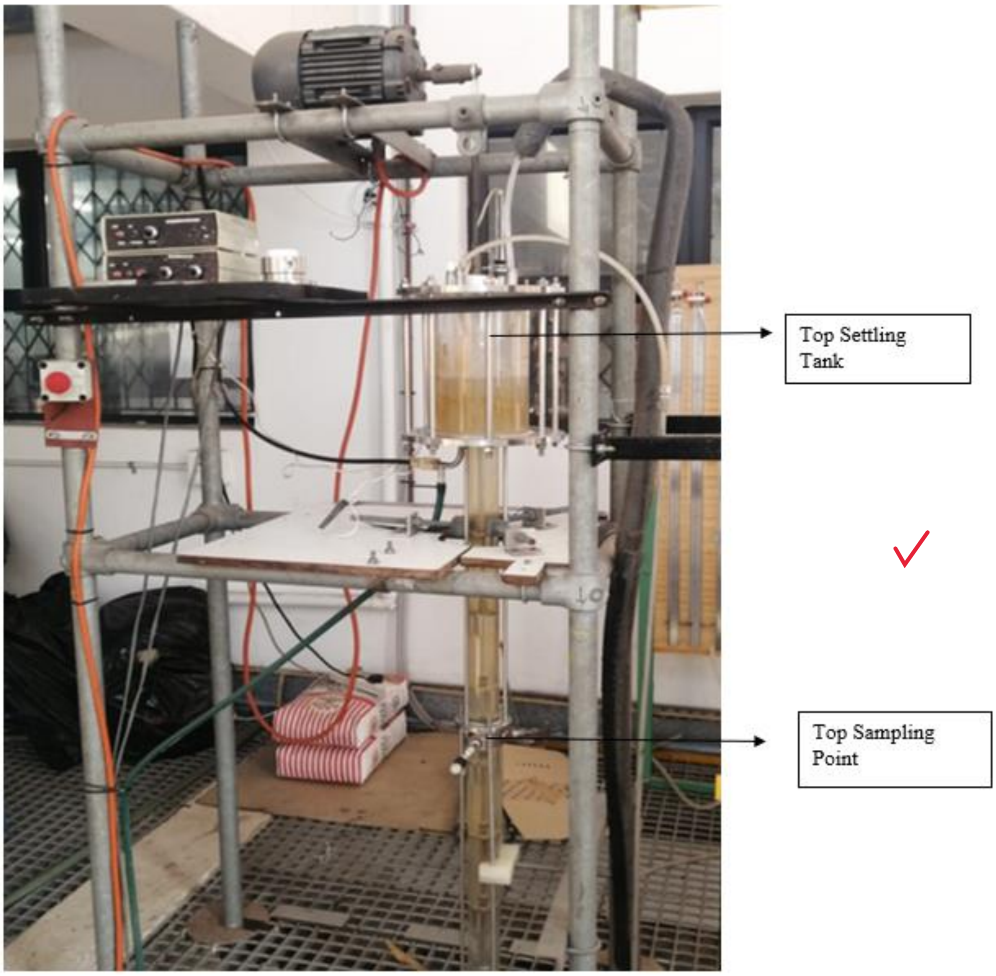


Figure 3-2: Experimental set up of the top of the VPE column

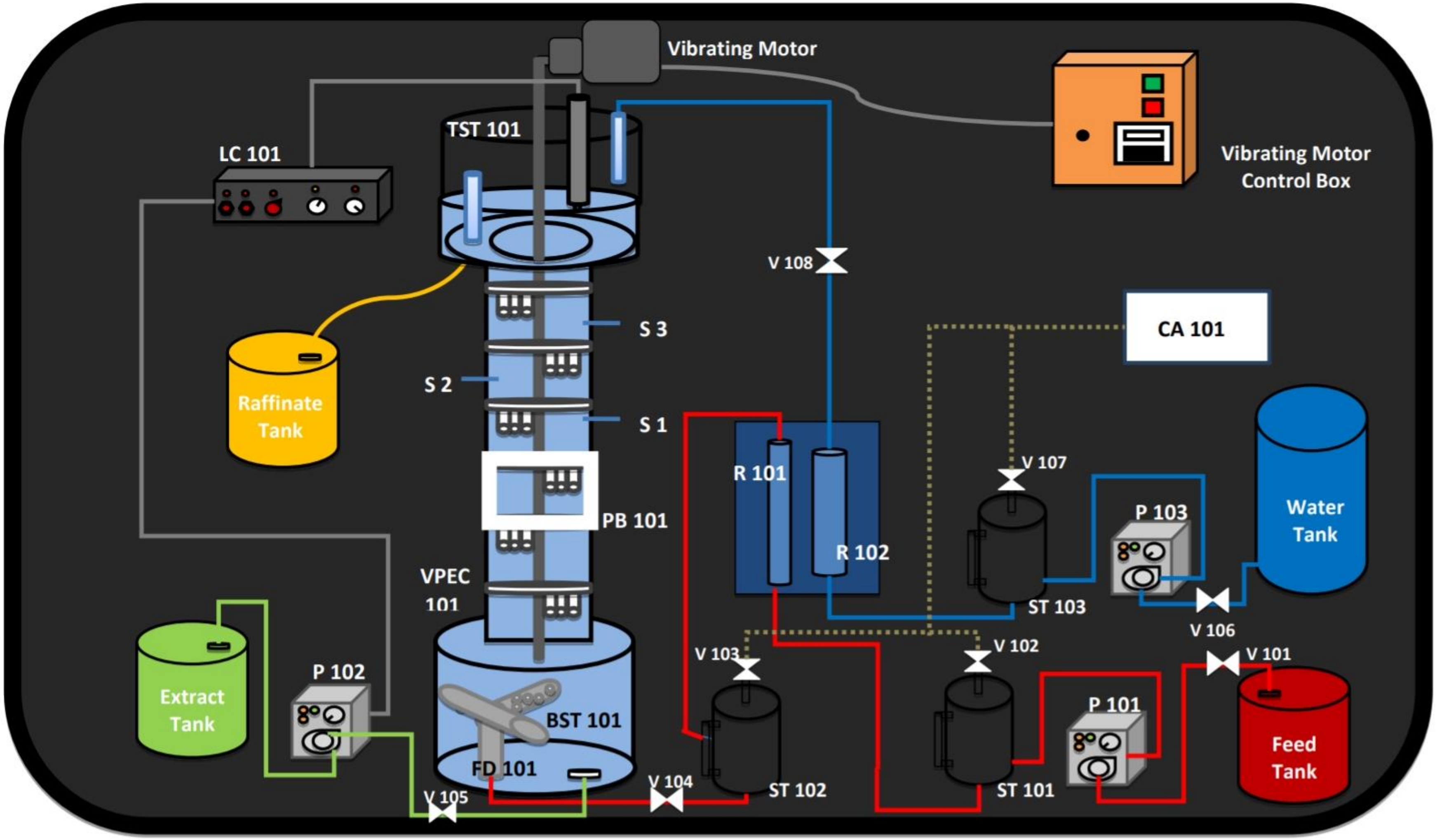


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Figure 3-3: Schematic of VPE column taken from (Naidoo, 2012)

## 3.3.Description of equipment

The following equipment was used in the experiment:

- 1 litre measuring cylinder
- 1 $\mu$L injector
- Feed drums
- Kern weighing balance
- Thirty 18 ml sampling vials
- VPE column

The ancillaries of the VPE column are described below. For a full description of the equipment associated with the VPE column, please refer to (Rathilal, 2010) and (Naidoo, 2012).

### 3.3.1 Extraction Column

The specifications of the vibrating plate extraction column are provided in Table 3-2.

**Table 3-2: VPE column specifications (Naidoo, 2012)**

| | |
|---|---|
| Inner Diameter (mm) | 47.7 |
| Outer Diameter (mm) | 58.7 |
| Thickness of glass walls(mm) | 5.7 |
| Number of sections | 8 |
| Length of each section (mm) | 550 |
| Effective height of column (m) | 4.76 |
| Cross sectional area of the column (m) | $1.787 \times 10^{-3}$ |

✓

### 3.3.2 Perforated plates

The plates in the extraction column are constructed of stainless steel and consisted of small perforations for the movement of the dispersed phase, and downcomers for the movement of the continuous phase (Rathilal, 2010). Each plate consists of 26 perforations and 3 downcomers, as illustrated in Figure 3-4. Table 3-3 shows the specifications of the perforated plate.

**Table 3-3: Perforated plate specifications (Naidoo, 2012)**

| | |
|---|---|
| Perforated plate diameter (mm) | 47.4 |
| Perforated plate thickness (mm) | 2 |
| Number of perforations | 26 |
| Hole (perforation) diameter (mm) | 2.98 |
| No. of downcomers per plate | 3 |
| Downcomer diameter (mm) | 10.9 |
| Downcomer length (mm) | 43.3 |
| Cross- sectional area of plate ($m^2$) | $1.764 \times 10^{-3}$ |

✓

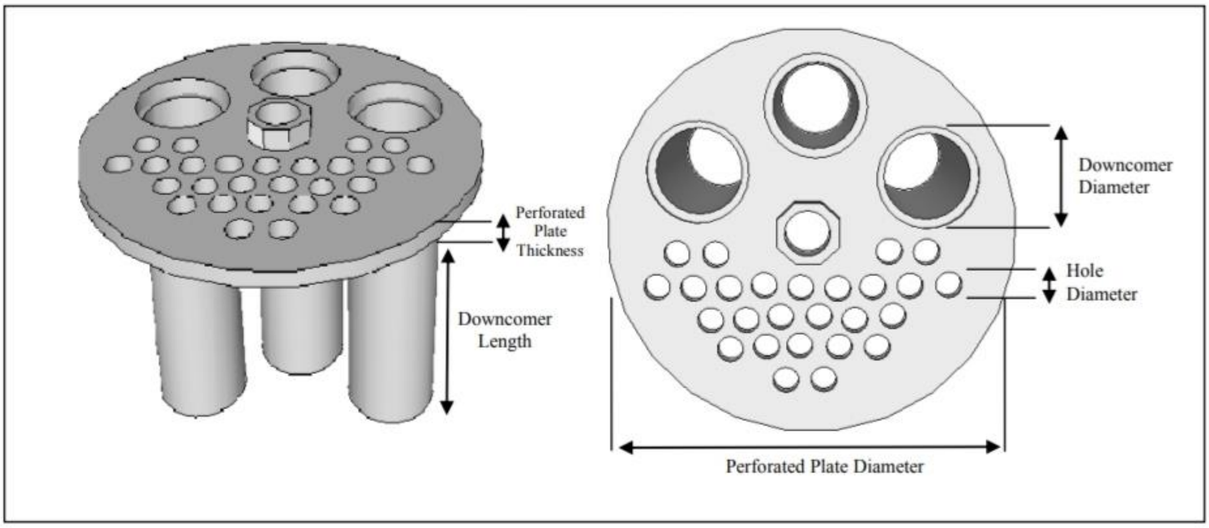


**Figure 3-4: Perforated plate in VPE column obtained from (Naidoo, 2012)**

The plates are assembled on a central shaft in the VPE column, with the downcomers on each successive plate arranged on opposite ends. This positioning of the plates permits the continuous phase to flow across the perforations of each plate (Naidoo, 2012). The liquids which form the continuous and dispersed phases is dependent upon the wettability of the liquid with the extraction column internals.

### 3.3.3 Vibration motor

A variable speed vibration motor is used to reciprocate the plates in the VPE column, by driving an adjustable yoke above the extraction column which was connected to the central shaft (Naidoo, 2012). The perforated plates are assembled along the central shaft. The vibration motor operated with the specifications as follows: 220V, power of 0.75 kW and 3.37 amp. The agitation level, which is the product of frequency and amplitude, is varied by adjusting the frequency of the vibration motor (Rathilal, 2010).

### 3.3.4 Peristaltic pump

One peristaltic pump was used to pump the feed mixture and the solvent consecutively into the column. The peristaltic pump used was Heidolph PD5106. The specifications of this pump are depicted in Table 3-4. Figure 3-5 depicts the peristaltic pump used in the experiment.

**Table 3-4: Heidolph PD5106 pump specifications (Naidoo, 2012)**

| | |
|---|---|
| Minimum speed (rpm) | 24 |
| Maximum speed (rpm) | 600 |
| Maximum flow rate (l/h) | 160 |

**Figure 3-5: Peristaltic pump**

The flow rate of liquid into the column can be adjusted by changing the speed of the pump. The pump is able to pump liquid both into and out of the column, since it could be operated in both clockwise and anti-clockwise directions (Naidoo, 2012). ✓

### 3.3.5 Settling tanks

Two settling tanks, which permitted the separation of the phases, are located above and below the VPE column. The interface between the organic and aqueous phases was sustained in the top settling tank, since the dispersed phase was the light phase (Sincuba, et al., 2015). The feed distributor, which is utilized for the dispersion of the dispersed phase, is located in the bottom settling tank (Naidoo, 2012). The top settling tank is shown in Figure 3-6.

**Figure 3-6: Top settling tank**

### 3.3.6 Sampler

Three sample points are located at different lengths along the column. The location of the sampler points along the column is shown in Table 3-5.

**Table 3-5: Sampler locations (Naidoo, 2012)**

| Sampling point | 1 | 2 | 3 |
|---|---|---|---|
| Distance (m) of the sampler points from the bottom of VPE column | 2.03 | 3.22 | 4.42 |

### 3.3.7 Gas chromatograph

The Shimadzu Gas Chromatograph shown in Figure 3-7 was used to obtain the composition of the samples attained from the column. An injector was used to inject a 1 $\mu$L sample into the packed column in the gas chromatograph. Carrier gas (He) transported the sample through the column. The GC is fitted with a thermal conductivity detector. Figure 3-6 shows the gas chromatograph specifications.

**Table 3-6: Gas Chromatograph Specifications (Naidoo, 2012)**

| | |
|---|---|
| Name | Shimadzu GC-2014 |
| Injector Temperature (℃) | 200 |
| Column Pressure (kPa) | 0.4 |
| Column Flow (ml/min) | 25 |
| Column Name | Chromosorb WHP SE 30 |

| | |
|---|---|
| Column Pressure (kPa) | 0.4 |
| Column Flow (ml/min) | 25 |
| Column Temperature (℃) | 180 |
| Column Length (m) | 3 |
| Column Inner Diameter (mm) | 3 |
| Detector Temperature (℃) | 250 |

**Figure 3-7: Shimadzu Gas Chromatograph**

## 3.4. Experimental Procedure

The column was run in batch mode and semi-batch mode. On the days the experiments were conducted, the temperatures were approximately 25.4 ℃ and the surrounding pressure was 99.481 kPa. The temperatures obtained using PT-100 Probe with ±0.05℃ uncertainty, while the pressures were obtained using Mensor CPC 3000 with ±0.025% (full-scale) uncertainty.

### 3.4.1. Batch mode

1. The feed solution of 21.50 wt. % ethanol and 78.50 wt. % cyclohexane was prepared. This constituted 2.2 litres of ethanol and 8.2 litres of cyclohexane.
2. The feed pump was started, and the speed of the feed pump was set to 100 rpm.
3. The solvent (water) was first pumped into the column through the top.

4. Thereafter the feed solution of cyclohexane and ethanol was pumped into the column, also through the top. The same pump was used to pump both the solvent and the feed into the column consecutively.
5. When all the solution was fed into the column, the pump was then stopped. A total volume of 11.8 litres was fed into the column.
6. The frequency was adjusted to 10 Hz for run 1a and 15 Hz for run 1b in order to vary the agitation level (product of frequency and amplitude).
7. A period of time was allowed for an assumed steady state to be reached (previous experiments carried out by (Rathilal, 2010), showed that 45 minutes was adequate). It was assumed that compositions after 45 minutes would remain constant. For this experiment, an hour was allowed for steady state to be reached.
8. The vibrations were then stopped and the solution in the column was allowed to settle for another hour.
9. The samples of solution at the sampler points along the column were then obtained, as well as the sample of the feed.
10. The samples obtained at different lengths along the column were thereafter analysed using gas chromatography analysis to establish the amount of ethanol present.

### 3.4.2. Semi-batch mode

1. The feed solution of ethanol and cyclohexane was prepared in specific ratios of:
   - 54% ethanol and 46 wt. % cyclohexane for run 2a
   - 52.04% ethanol and 47.96wt. % cyclohexane for run 2b

   8.06 litres of ethanol and 7 litres of cyclohexane were used for run 2a, while 5.74 litres of ethanol and 5.29 litres of cyclohexane were used for run 2b.
2. The pump was started after checking the solvent valve was opened and the speed of the pump was set to 100 rpm.
3. The solvent (water) was fed into the column through the top. 3 litres of water were fed into the column for run 2a, and 4 litres of water was fed into the column for run 2b.
4. The feed valve was then opened and the valve for the solvent was closed. The feed solution of cyclohexane and ethanol was then fed into the column through the bottom using the feed pump. Enough solution was prepared so that the overflow line was reached. This solution overflowed and was collected in the feed drum and was then fed back into the column via the bottom inlet, so that the solution was recycled. A volume of 2 litres was kept constant in the feed drum, and a total volume of 18 litres was used for the semi-batch runs.

5. A period of time (15 min) was allowed for the system to stabilize, as well as for mixing to occur with the vibration set to 15 Hz.
6. After 15 minutes, the mixture from the drum was sampled (approximately 5-10 ml was withdrawn for each sample) and this was continued for 1 hour over 10 min intervals.
7. Samples were removed from two sampling points along the column.
8. The vibrations were then stopped. The feed pump was then switched off and the column was drained.
9. The samples were stored in the fridge and then analysed using gas chromatography in order to obtain the amount of solute (ethanol) present in the feed drum and at the different points along the column.

### 3.4.3. Gas Chromatograph operation procedure

1. The gas flow was turned on and the flow rate was set to 30 ml/min.
2. The oven was then turned on and the detector and injector temperatures were set to 200°C.
3. The column temperature was then set to 180°C.
4. When the oven temperature stabilized, the detector filament was then turned on.
5. The samples were then removed from the fridge and approximately 1-4g of acetone was added to the samples.
6. The samples were then injected into the gas chromatograph.
7. After the sample analyses were complete, the gas chromatograph was shut down. The detector was switched off, and the gas flow rate was set to 3ml/min.
8. The detector, injector and column temperatures were then set to 25°C, and the system was allowed to cool.
9. When the detector temperature cooled to below 50°C, the gas was switched off.

### 3.4.4. Calibration of the Gas Chromatograph Detector

The gas chromatograph detector was calibrated by injecting binary mixtures of each component with an internal standard. In this manner, the ratios of the component to a known amount of standard could be evaluated to solve for the unknown compositions. Cyclohexane and water are immiscible therefore it was required that a miscible solvent be added to the sample before injection into the gas chromatograph. Acetone was added as the internal standard. Mixtures of ethanol (1)-acetone (4), cyclohexane (2)-acetone(4) and water (3) – acetone (4) were prepared in specific ratios. The samples were weighed using a Kern weighing scale, which had a readability of 0.0001g and a precision of ±0.00048g. These mixtures were prepared in concentrated and dilute regions of each component, in the ranges of $\frac{x_4}{x_i}$ between 0 and 1.2, and the ranges of $\frac{x_i}{x_4}$ between 0 and 1.2, where $x_4$ is the composition of acetone. These samples were well mixed and then injected into the gas chromatograph, after which the peaks showing each component were outputted and the peak areas were then obtained. The ratio of these areas was then plotted against the mass fraction ratios, which were compared to determine the accuracy of the calibrations. The calibration graphs were used to obtain the mass of the components in the samples obtained from experiments, by using the ✓ ratio of the peak areas to obtain the mass fraction ratios.

# CHAPTER 4: RESULTS AND DISCUSSION

This chapter presents the results and discussion, beginning with the GC detector calibrations, and uncertainties of the measured variables. The results from the separation of the ternary system with the compositions using the VPE column is reported thereafter, with a discussion of the performance of the column based on the percentage extracted. The number of equilibrium stages obtained for each separation is also presented.

## 4.1.Calibration of the gas chromatograph detector

Calibrations were performed for the systems of ethanol- acetone, water- acetone and cyclohexane- acetone. The masses of the components in each sample used for calibration are shown in Table A- 1, Table A- 2, and Table A- 3in appendix A for the 3 different systems. Two different peaks were obtained from the gas chromatograph for the two components in each system. The samples were injected twice to ensure repeatability and to obtain the uncertainty in calibration. The calibration graphs, shown in Appendix C, were obtained by plotting the ratio of the areas of each component with acetone against the ratio of the mass fractions.

***Table 4-1: Uncertainties in calibration of the gas chromatograph detector***

| | Gradient of calibration graph in acetone- dilute region | Inverse slope (acetone- dilute region) | Gradient of calibration graph in acetone-rich region | Uncertainty of calibration (acetone – dilute region) | Uncertainty of calibration (acetone –rich region) |
|---|---|---|---|---|---|
| Ethanol- acetone | 1.0717 | 0.9330 | 0.9558 = (1/1.0462) | 0.28% | 0.69 % |
| Water- acetone | 0.8519 | 1.1738 | 1.115= (1/0.8969) | 2.80% | 1.26% |
| Cyclohexane - acetone | 0.7238 | 1.3816 | 1.3878= (1/0.7206) | 6.62%<br>✓ | 3.45% |

Table 4-1 presents the response ratios ($A_i/A_j$vs $x_i/x_j$) or factors for the binary calibrations with the aforementioned systems. The trendline fit of the calibration graphs was grounded at the origin and the gradient of the trendline was obtained. From Table 4-1, it can be deduced that the inverse of the gradient of the calibration graph for each system in the acetone dilute region is similar to the gradient of the calibration graph in the acetone rich region, correct to one decimal place. This criterion shows that the calibrations were performed accurately. The calibration graphs were used to obtain the masses of each component in the samples taken from the column during experimental runs.

The uncertainty of the calibrations is shown in the final two columns in Table 4-1. The error for cyclohexane and acetone is significantly higher than other systems. This is attributed to the fact that the temperature of the oven in the gas chromatograph had to be changed from 200℃ to 180℃ when using this system since the peaks of different components were overlapping. These uncertainties can also be attributed to equipment such as the mass balance which took long periods of time to stabilize. Acetone, which was used as the internal standard, has a relatively low boiling point of 56 °C, and thus the samples had to be stored in the refrigerator to ensure the acetone did not evaporate. Higher room temperatures on the day the experiment was conducted may have resulted in acetone evaporating and would thus affect the results and contribute to higher uncertainties.

## 4.2.Batch runs 1a and 1b

Samples of the feed, extract and raffinate were taken at the end of the batch run, after 1 hour each was allowed for mixing and settling. The continuous phase was the aqueous phase which flowed to the bottom of the column, whereas the organic phase formed the dispersed phase. The VPE column was vibrated at frequencies of 10Hz and 15Hz for runs 1a and 1b respectively. These frequencies were chosen since too high frequencies cause flooding in the column, and too low frequencies do not allow for efficient mixing (Naidoo, 2012). A solvent to feed ratio of 1:1.5 was used for both runs 1a and run 1b. Table 4.2 presents the compositions of samples obtained from batch run 1a and 1b.

**Table 4-2: Compositions of samples obtained from batch run 1a and 1b**

| Mass fraction | Batch run 1a | Batch run 1b |
|---|---|---|
| $x_{ethanol,feed}$ | 0.49 | 0.49 |
| $x_{ethanol,extract}$ | 0.390 | 0.457 |
| $x_{ethanol,raffinate}$ | 0.261 | 0.209 |

Table 4-2 indicates that the mass fraction of ethanol in the extract was higher for run 1b than run 1a. This is due to a higher frequency being used for run 1b, which allows for better mixing and therefore a greater rate of mass transfer.

### 4.3. Experiments- Semi-batch runs

#### 4.3.1. Semi-batch run 2a

For the semi-batch runs, samples of the feed, extract and raffinate were taken over 10-minute intervals after allowing mixing to occur for 15 minutes. The VPE column was vibrated at 15 Hz and a solvent to feed ratio of 1:4 was used for run 2a. Figure 4-1 depicts the change in the composition of ethanol in the extract, raffinate and feed over time.

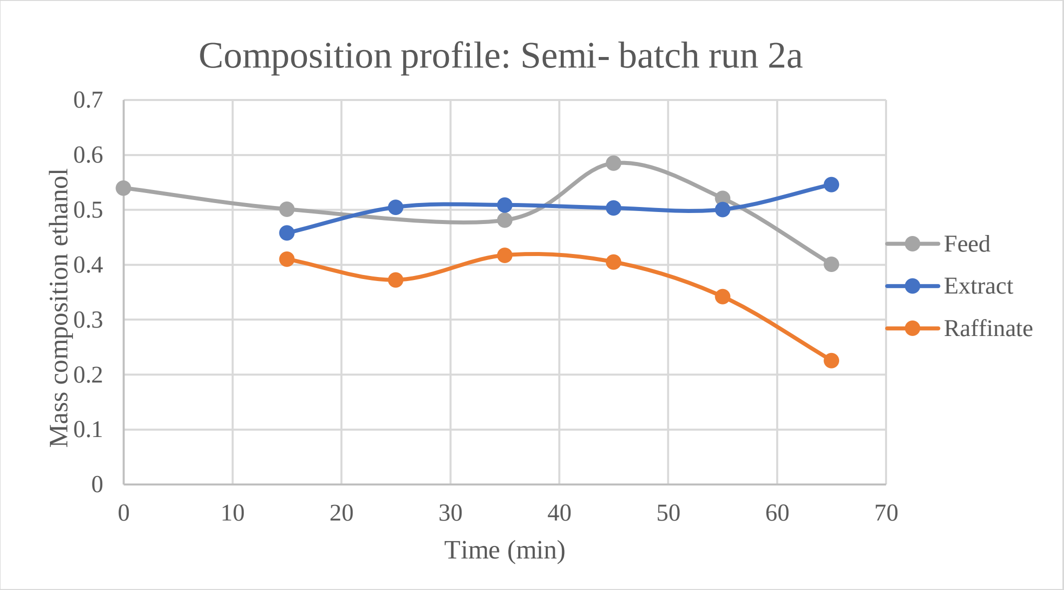


**Figure 4-1: Composition profile for semi- batch run 2a**

The composition of ethanol in the extract increased from 0.458 at the beginning of the run to 0.546 at 65 minutes, since the mass transfer of ethanol from the carrier (cyclohexane) to the solvent (water) occurred during this time interval. The composition of ethanol in the raffinate was expected to decrease over time. The composition in the raffinate initially decreases at 25 minutes, and thereafter increases to 0.418 at 35 minutes. This can be attributed to errors such as incorrect sampling, as well as the presence of immiscibility between the different phases within the sample. This would lead to inaccurate compositions being obtained from the gas chromatograph. The composition of ethanol in the raffinate thereafter decreases to 0.226 at 65 minutes, which indicates that ethanol was extracted by water and therefore decreases the amount of ethanol present in the carrier.

A volume of 2 litres was maintained in the feed drum during semi-batch operation. It was observed that the composition of ethanol in the feed decreased within the 0-35 minute interval but increased to 0.585 at 45 minutes. This is due to experimental errors such as the storage vessel not being shaken thoroughly before sampling, and immiscible samples being obtained from the feed drum. Insufficient acetone could have been added, hence not allowing the sample to dissolve properly which leads to immiscibility in the sample. Inaccurate results could have also been obtained due to the incorrect sampling of the feed solution, since a pipette was used to obtain solution from a large feed vessel that was obstructed by pipes and the silicon tubing of the pump. Inaccurate results could have also been obtained due to the incorrect sampling of the feed solution, since a pipette was used to obtain solution from a large feed vessel that was obstructed by pipes and the silicon tubing of the pump.

Overall, the increase of the ethanol in the extract, with the decrease of the solute in the feed and raffinate are observed and were expected trends.

### 4.3.2. Semi-batch run 2b

The experimental procedure for run 2a was repeated for run 2b. Figure 4-2 depicts the change in the composition of ethanol in the extract, raffinate and feed over time. A solvent to feed ratio of 1:3 was used for run 2b, and vibrations occurred at a frequency of 15Hz.

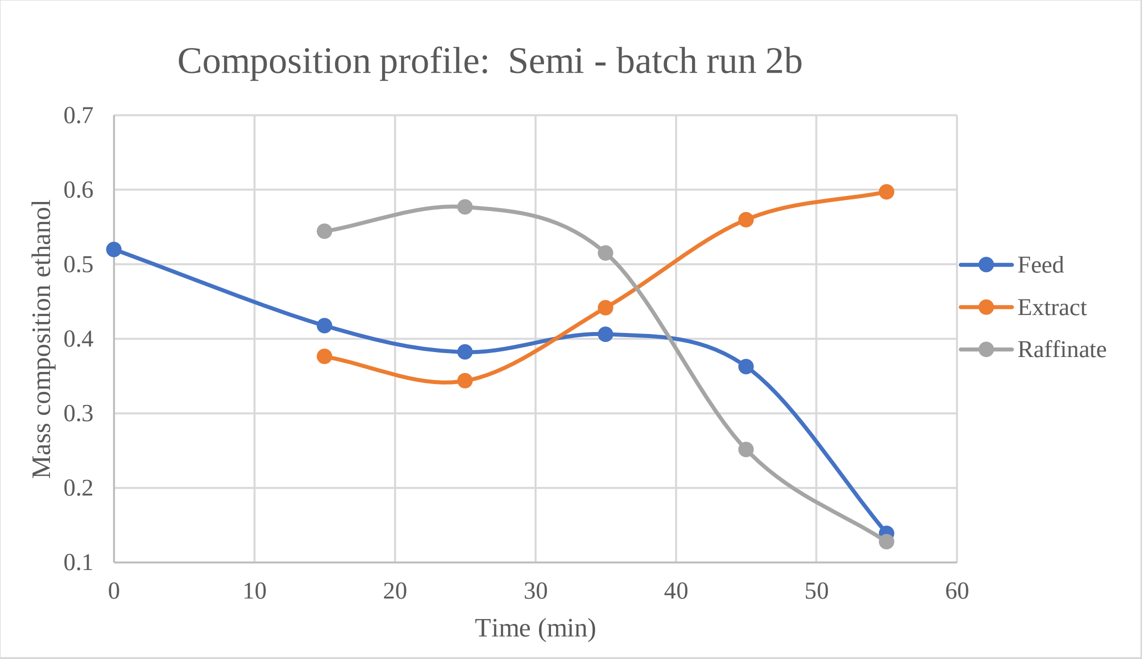


**Figure 4-2: Composition profile for semi- batch run 2b**

The composition of ethanol in the extract was expected to increase with time. However, as shown in Figure 4-2, the composition of ethanol in the extract decreased at 25 minutes, and thereafter increased to 0.597 ethanol at 55 minutes. The composition of ethanol in the raffinate decreased from 0.544 to 0.128 during the run; however, at 25 minutes, the composition of ethanol in the raffinate increased to 0.577. These observations are similar to the previous run.

These discrepancies can be attributed to immiscible samples taken from the column, which would therefore lead to inaccurate results. According to the graph of the extract ethanol composition, steady state was approached at 55 minutes. In order for steady state to be attained, the column would need to be vibrated for a period longer than 55 minutes. The ethanol feed composition decreased at 25 minutes and remained constant until a sharp decrease in the composition in the 45-55 minute interval. This sharp decrease could be due to immiscible samples being drawn from the feed vessel, as well as the fact that the storage vessel was not shaken before sampling. Final extract compositions of 0.547 and 0.597 were obtained for runs 2a and 2b respectively. A higher solvent to feed ratio was used for run 2b, therefore more solvent was available to extract ethanol from cyclohexane and thus a slightly higher ethanol extract composition was obtained for run 2b than for run 2a.

### 4.4.Percentage of ethanol extracted

Material balances over the VPE column were used to obtain the moles of ethanol in the feed and raffinate. The sample calculation for percentage extracted is depicted in Appendix B. The performance of the VPE column was evaluated using the percentage of ethanol extracted. The equation for the percentage extracted is shown in Chapter 2 as equation (2). Table 4.3 presents the percentage of ethanol extracted for all experimental runs performed.

**Table 4-3: Percentage of ethanol extracted**

| | $n_{ethanol,feed}$ (mol) | $n_{ethanol,raffinate}$ (mol) | Percentage ethanol extracted (%) |
|---|---|---|---|
| Run 1a (Batch) | 63.383 | 25.058 | 60.466 |
| Run 1b (Batch) | 63.383 | 22.648 | 64.268 |
| Run 2a (Semi-batch) | 138.054 | 26.696 | 80.663 |
| Run 2b (Semi-batch) | 124.596 | 20.733 | 83.360 |

When the VPE column was operated in semi-batch mode, it was observed that a greater degree of mixing and mass transfer occurred since phases were distributed uniformly throughout the column. Therefore, higher percentages of ethanol were extracted for the semi-batch runs, as shown in Table 4-3.

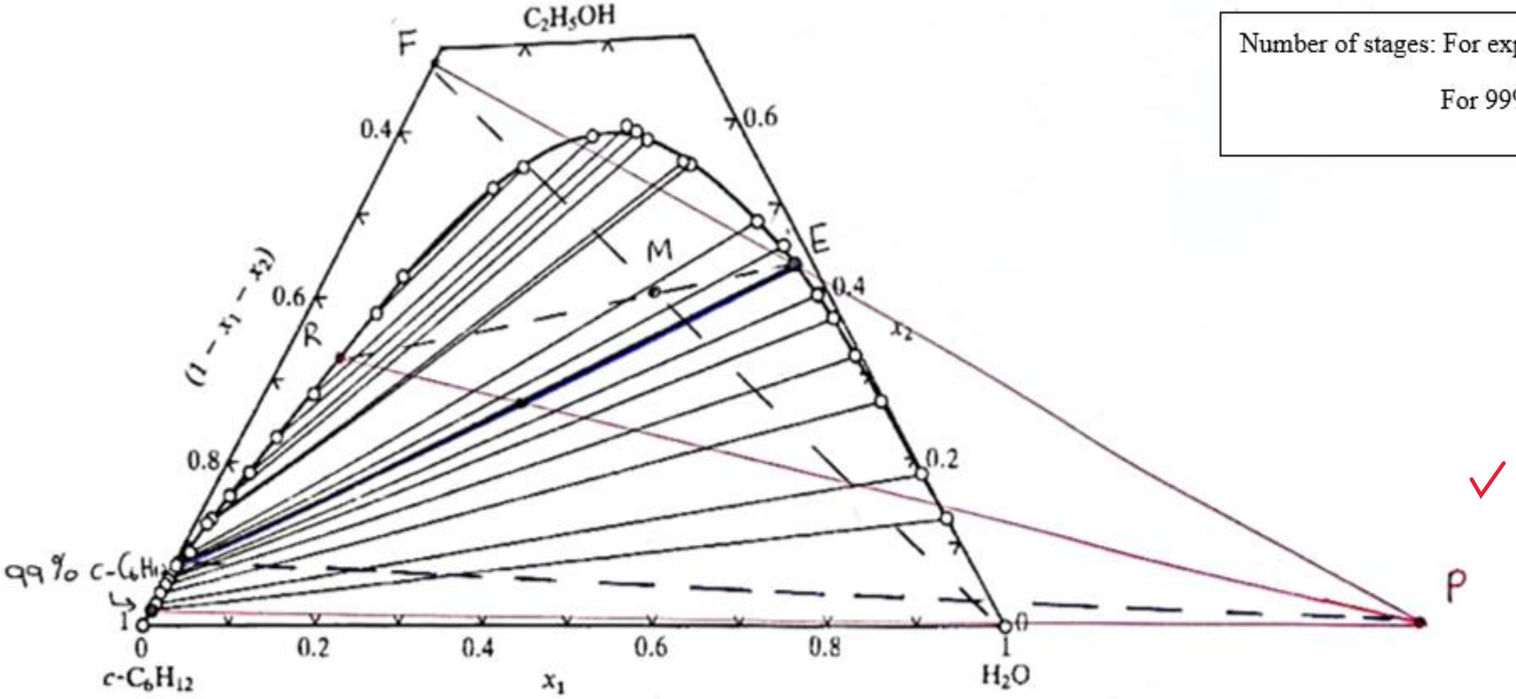
It was observed that operating the VPE column in batch mode did not allow for efficient mixing since the solution in the column appeared murky on the top of the column and clear on the bottom. Thus, lower percentages of ethanol were extracted for the batch runs. The lowest percentage of ethanol (60.466%) was extracted for batch run 1a, which can be attributed to the fact that the VPE column was vibrated at a frequency of 10 Hz, whereas for the other runs, the column was vibrated at a frequency of 15 Hz. Higher frequencies effect better mixing and mass transfer of ethanol from the carrier (cyclohexane) to the solvent (water). The approximate 4% improvement in the extraction of ethanol between runs 1a and 1b can be attributed to the slightly improved mixing. Although a higher mass solvent to feed ratio of 1: 1.5 was used for the batch runs 1a and 1b, the mode of operation did not allow for efficient contacting of the phases since the extract and raffinate phases were not continually removed, and no recycle stream was present.

Similarly, for the semi-batch mode, a higher percentage of ethanol (83.360%) was extracted for run 2b when compared to run 2a. The reproducibility of the result with a difference of 3.4% between runs 2a and 2b is quite good. The improvement in run 2 is also due to a higher solvent to feed ratio of 1:3 being used for run 2b whereas a solvent to feed ratio of 1:4 was used for run 2a.

## 4.5. Calculation of equilibrium stages

The comparison of the number of equilibrium stages obtained for each run and the number of stages required to obtain 99 mol% cyclohexane in the raffinate are shown in Table 4-4. The stages that were stepped off on the ternary diagram for the ethanol-cyclohexane-water system are shown in Appendix C. Note that the graphs shown in Appendix C are in mol% for the compositions, while the data in previous tables in Appendix C reported the compositions in weight %. The data was converted to a consistent format. The number of equilibrium stages required is dependent on the solvent to feed ratio and agitation level used in each run, as well as the mode of operation (Naidoo, 2012).

Figure 4-3 shows the equilibrium stages that were stepped off for semi- batch run 2a. The feed, extract and raffinate points are shown on the diagram. The Hunter Nash graphical equilibrium stage method was used to obtain the equilibrium stages for each experimental run (Seader & Henley, 2011). The alternate use of the steep tie lines and operating lines, which are joined to the operating point P, were used to step off the equilibrium stages (Seader & Henley, 2011). It can be seen that the feed concentrations (around 68 mol% ethanol), are in fact quite high in ethanol. However due to the volume of chemicals, and lack of cyclohexane, these runs were performed regardless of the higher ethanol in the concentration in the feed. The purpose of the experiments were to show the distribution of the solute and gain understanding in the operation of the column.



Number of stages: For experimental raffinate composition: 0.5 stages

For 99% cyclohexane in the raffinate: 1.7 stages

**Figure 4-3: Equilibrium stages for semi-batch run 2a stepped off on ternary diagram**

**Table 4-4: Equilibrium stages for the cyclohexane-ethanol-water system**

| | **Number of equilibrium stages obtained from experiment** | **Number of stages required to obtain 99 mol% cyclohexane in the raffinate** |
|---|---|---|
| **Batch run 1a** | 0.75 | 2 |
| **Batch run 1b** | 0.72 | 1.8 |
| **Semi- batch run 2a** | 0.5 | 1.7 |
| **Semi- batch run 2b** | 0.45 | 1.5 |

The number of stages obtained for batch run 1a were the highest, while the number of stages obtained for semi- batch run 2b were the lowest. Less than 1 stage was obtained for all 4 runs. Batch run 1a was performed at the lowest frequency of 10 Hz. The degree of separation achieved from the experiments corresponds to 0.75 theoretical stages. For the same mixture, 2 theoretical stages would be required to achieve 99% cyclohexane in the raffinate. The stages stepped off on the ternary diagram for this run is shown in Figure C- 7. Batch run 1b was performed using a frequency of 15 Hz. The degree of separation corresponded to a lower number of 0.72 stages, which is shown in Figure C- 8. For the same experimental run, to achieve 99% cyclohexane in the raffinate, 1.8 theoretical stages were obtained. Therefore, using a higher frequency allowed for better mixing and mass transfer to occur, which resulted in less stages being attained for separation.

A mass solvent to feed ratio of 1:4 was used for semi-batch run 2a. The degree of separation achieved corresponded to 0.5 stages for separation which is shown in Figure 4-3. Semi-batch run 2b used a solvent to feed ratio of 1:3 and 0.45 stages for separation was obtained, as depicted in Figure C- 9. Therefore, a higher solvent to feed ratio allowed more solvent (water) to be available for the extraction of ethanol and thus required less stages for separation. ✓

From these results, it can be deduced that operating the column in semi-batch mode allowed for efficient separation to occur since less equilibrium stages were obtained when compared to the stages stepped off when operating the column in batch mode. The composition of 99 mol% cyclohexane in the raffinate represents the optimum composition that can be obtained in the raffinate. Thus, more stages were required to obtain this extraction of ethanol since the compositions of cyclohexane in the raffinate for the experimental runs were less than 99%. The most equilibrium stages stepped off for a composition of 99 mol% cyclohexane in the raffinate were obtained for batch run 1a as shown in Table 4-4, while the least number of stages was obtained for semi- batch run 2b.

# CHAPTER 5: CONCLUSION

The system of cyclohexane, ethanol and water was used to commission the column in this work since the chemicals were cheaper than the initial proposed systems, and readily available in the quantities necessary.

Four experimental runs were performed, whereby the VPE column was operated in batch mode for two runs and semi-batch mode for the other two runs. Samples were drawn from the sampling points on the VPE column and from the feed vessel. The effect of solvent to feed ratio, frequency and the mode of operation on the effectiveness of the separation was investigated.

The gas chromatograph was calibrated to minimize uncertainties and to obtain the calibration plots, which were used to obtain the mass fractions of the components in each sample drawn from the VPE column. The uncertainties for calibration obtained were ±1%, except for the cyclohexane—acetone system which displayed the highest error of 6.62%.

Batch run 1a was performed using a frequency of 10Hz and a low percentage of 60.466% of ethanol was extracted. Batch run 1b was performed by vibrating the VPE column at a frequency of 15Hz and 64.268% of ethanol was extracted. It is observed that a higher frequency of 15 Hz allowed for more efficient mixing and mass transfer.

The semi- batch runs were performed using a frequency of 15 Hz. Solvent to feed ratios of 1:4 and 1:3 were utilized for semi- batch runs 2a and 2b respectively. A higher percentage of 83.360% of ethanol was extracted for run 2b, while 80.663% ethanol was extracted for run 2a. Subsequently, a higher solvent to feed ratio allows for more ethanol to be extracted since there is more water was available to perform the extraction.

Approximately 0.75 and 0.72 stages were obtained after stepping off on the ternary diagram for batch runs 1a and 1b respectively, while 0.5 and 0.45 stages were obtained for semi-batch runs 2a and 2b respectively. Therefore, operating the column in semi- batch mode requires less stages, and it can be concluded that semi-batch mode is more effective than batch mode and allows for a better rate of mass transfer of ethanol from the carrier. Less than 1 stage for separation was obtained for all four runs.

There were some discrepancies in the concentration profiles obtained for the semi-batch runs. These were due to experimental errors such as immiscible samples being drawn from the column, and uncertainties due to calibration. The feed storage vessel was not agitated before sampling, which also affects the accuracy of the results. Recommendations for the sample withdrawal technique are proposed to overcome these errors.

# CHAPTER 6: RECOMMENDATIONS

- For the system investigated, the use of a feed with ethanol (composition <15%) should be tested.
- Additional experiments are proposed to investigate the effect of varying frequencies on the effectiveness of separation when operating the VPE column in semi- batch mode.
- More experiments are also required to investigate the effect that different solvent to feed ratios have on the effectiveness of separation when operating the VPE column in batch mode.
- The VPE column should also be operated in continuous mode (with both phases being in counter current flow) and the results thereof compared to other modes of operation. This will require greater quantities of chemicals which may likely be expensive.
- When calibrating the gas chromatograph, internal standards other than acetone should be investigated since acetone has a relatively low boiling point and thus evaporates very quickly. This should decrease the uncertainty and error associated with the composition reporting, and prevents immiscibility in samples.
- Experiments should be run for a longer period of time, possibly 2 hours, when operating the column in semi- batch mode to investigate when steady state is reached.
- The number of actual stages in the VPE column and the efficiency of the separation when varying different parameters and using different modes of operation should be investigated.
- Parameters such as tray spacing and temperature also affect separation effectiveness and thus more experiments are required to investigate the effect of these parameters.

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# APPENDIX A: RAW DATA

**Table A- 1: Calibrations for ethanol- acetone system**

| Acetone rich region | | | | Ethanol rich region | | | |
|---|---|---|---|---|---|---|---|
| Component Masses for calibration | | Areas obtained from gas chromatograph | | Component Masses for calibration | | Areas obtained from gas chromatograph | |
| ethanol (g) | acetone (g) | $A_{1,ethanol}$ | $A_{4,acetone}$ | ethanol (g) | acetone (g) | $A_{1,ethanol}$ | $A_{4,acetone}$ |
| 0.1153 | 1.4534 | 81309 | 913514 | 1.4003 | 0.1765 | 831687.9 | 133408 |
| | | 78614.7 | 886029.9 | | | 867635.6 | 139464.9 |
| | | | | | | | |
| 0.4461 | 1.1247 | 286630.1 | 708347.7 | 0.9655 | 0.6086 | 604096.5 | 399088.6 |
| | | 267599.8 | 680872.5 | | | 593446.6 | 396187.7 |
| | | | | | | | |
| 0.6549 | 0.9173 | 393527.2 | 572550.3 | 0.7367 | 0.8359 | 434648.8 | 522099.3 |
| | | 402785.1 | 591435.5 | | | 453512.4 | 542417.2 |

✓

## Table A- 2: Calibrations for water- acetone system

| Acetone rich region | | | | Water rich region | | | |
|---|---|---|---|---|---|---|---|
| Component Masses for calibration | | Areas obtained from gas chromatograph | | Component Masses for calibration | | Areas obtained from gas chromatograph | |
| water (g) | acetone (g) | $A_{2,water}$ | $A_{4,acetone}$ | water (g) | acetone (g) | $A_{2,water}$ | $A_{4,acetone}$ |
| 0.0475 | 1.5307 | 45536.5 | 919134.7 | 1.4143 | 0.4558 | 916268.1 | 303374.3 |
| | | 45815.9 | 953718.4 | | | 994001.9 | 301598.7 |
| | | | | | | | |
| 0.2168 | 1.3975 | 145864.9 | 873322.7 | 0.6539 | 1.0538 | 504154.2 | 683547.5 |
| | | 144217.1 | 877633.4 | | | 470804.3 | 669493.3 |
| | | | | | | | |
| 0.6549 | 1.5680 | 252552.2 | 812272 | 0.4253 | 1.2336 | 320804.5 | 800817.9 |
| | | 255219.3 | 797808.1 | | | 334870 | 808590.8 |

✓

### Table A- 3: Calibrations for cyclohexane- acetone system

| Acetone rich region | | | | Cyclohexane rich region | | | |
|---|---|---|---|---|---|---|---|
| Component Masses for calibration | | Areas obtained from gas chromatograph | | Component Masses for calibration | | Areas obtained from gas chromatograph | |
| cyclohexane (g) | acetone (g) | $A_{3,cyclohexane}$ | $A_{4,acetone}$ | cyclohexane (g) | acetone (g) | $A_{3,cyclohexane}$ | $A_{4,acetone}$ |
| 0.1983 | 1.3684 | 12632.7 | 91210.8 | 1.4580 | 0.1006 | 87110.3 | 11049.6 |
| | | 11556.8 | 91862.4 | | | 93582.1 | 13235.3 |
| | | | | | | | |
| 0.6570 | 0.9068 | 121802.5 | 251782.4 | 1.1602 | 0.4003 | 634479.9 | 250679.6 |
| | | 125203.5 | 255329.8 | | | 796802.2 | 398149.8 |
| | | | | | | | |
| 0.8843 | 0.6781 | 491502.8 | 485291.6 | 0.9634 | 0.5984 | 487167.8 | 372686.6 |
| | | 410607.7 | 446943.6 | | | 438783.7 | 354460.6 |

✓

*Table A- 4: Feed quantities used for each run*

|  |  | Component | Mass (kg) | Mass composition | Density $\left(\frac{kg}{dm^3}\right)$ | Volume (l) |
|---|---|---|---|---|---|---|
| Semi-batch run 2a | Feed | Ethanol | 6.36 | 0.54 | 0.789 | 8.06 |
|  |  | Cyclohexane | 5.42 | 0.46 | 0.779 | 6.96 |
|  | Solvent | Water | 2.99 |  | 0.997 | 3 |
| Semi- batch run 2b | Feed | Ethanol | 5.74 | 0.52 | 0.789 | 7.27 |
|  |  | Cyclohexane | 5.29 | 0.48 | 0.779 | 6.79 |
|  | Solvent | Water | 4 |  | 0.997 | 4.01 |
| Batch run 1a and 1b | Feed | Ethanol | 2.92 | 0.49 | 0.789 | 3.71 |
|  |  | Cyclohexane | 3.04 | 0.51 | 0.779 | 3.91 |
|  | Solvent | Water | 4.17 |  | 0.997 | 4.19 |

**Table A- 5: Sample masses and areas obtained from gas chromatograph for batch runs 1a and 1b**

| | Sample point | Mass without acetone | Mass acetone | Total mass | $A_{1,ethanol}$ | $A_{2,water}$ | $A_{3,cyclohexane}$ | $A_{4,acetone}$ |
|---|---|---|---|---|---|---|---|---|
| Batch Run 1a | Feed | 10.8611 | 2.0204 | 12.8815 | 393156 | 78446.6 | 262417.4 | 241189.4 |
| | Extract (bottom) | 12.0792 | 2.098 | 14.1772 | 846343 | 198393.6 | 0 | 278852.6 |
| | Middle | 11.1588 | 1.0028 | 12.1616 | 283974.4 | 0 | 591385.8 | 24314.4 |
| | Raffinate (top) | 7.9976 | 2.0016 | 9.9992 | 106991 | 3362 | 694042.2 | 169573.5 |
| | | | | | | | | |
| Batch Run 1b | Extract (bottom) | 12.0972 | 2.098 | 14.1952 | 315214.6 | 174937.5 | 0 | 195308.5 |
| | Middle | 10.1588 | 2.0028 | 12.1616 | 6494.4 | 1239.9 | 686081.5 | 160042 |
| | Raffinate (top) | 7.9776 | 2.0026 | 9.9802 | 98260.8 | 0 | 665376.2 | 204063.6 |

**Table A- 6: Sample masses and areas obtained from gas chromatograph for semi-batch run 2a**

| Time | Sample point | Mass without acetone | Mass acetone | Total mass | $A_{1,ethanol}$ | $A_{2,water}$ | $A_{3,cyclohexane}$ | $A_{4,acetone}$ |
|---:|---|---:|---:|---:|---:|---:|---:|---:|
| 0 | Feed | | | | | | | |
| 15 | | 0.3414 | 0.1124 | 0.4538 | 460076.7 | 28019.5 | 172488 | 323773.4 |
| 35 | | 3.7288 | 1.0037 | 4.7325 | 405112.6 | 25877.4 | 263492 | 242820.2 |
| 45 | | 0.3754 | 0.1184 | 0.4938 | 463674.8 | 19944.6 | 216810.9 | 267957.9 |
| 55 | | 0.8252 | 0.3045 | 1.1297 | 390960.5 | 15532.2 | 251758.1 | 296702.2 |
| 65 | | 1.0994 | 0.503 | 1.6024 | 325882.2 | 13724.2 | 209766.2 | 388990.9 |
| | | | | | | | | |
| 15 | Bottom point | 8.0283 | 2.0856 | 10.1139 | 371880.2 | 76776.3 | 299475.8 | 226120.9 |
| 25 | | 3.8748 | 1.0021 | 4.8769 | 393216.9 | 16503.7 | 324258.4 | 215805.7 |
| 35 | | 4.3786 | 1.0076 | 5.3862 | 404664.5 | 25773.2 | 332376.8 | 196093 |
| 45 | | 6.1587 | 1.0253 | 7.184 | 414614 | 26289.1 | 328123.3 | 146941.9 |
| 55 | | 3.0571 | 0.7956 | 3.8527 | 422469.5 | 27119.3 | 288996.8 | 235284.8 |
| 65 | | 6.0525 | 1.0155 | 7.068 | 461646.9 | 27060.1 | 313278.7 | 151944 |
| | | | | | | | | |
| 15 | Top point | 9.542 | 2.507 | 12.049 | 299330.2 | 18249.2 | 299330.2 | 205388.7 |
| 25 | | 7.9417 | 1.507 | 9.4487 | 311614.6 | 13377.5 | 311614.6 | 170094.2 |
| 35 | | 1.37 | 0.5018 | 1.8718 | 310164.5 | 12436.1 | 245359.7 | 291628 |
| 45 | | 4.4648 | 1.0045 | 5.4693 | 335070.6 | 11783.5 | 310164.5 | 199393.8 |
| 55 | | 5.2866 | 1.0073 | 6.2939 | 285182.4 | 16322.5 | 335070.6 | 170242.3 |
| 65 | | 3.162 | 1.0106 | 4.1726 | 250028.5 | 19335.6 | 285182.4 | 370050.3 |

### Table A- 7: Sample masses and areas obtained from gas chromatograph for semi- batch run 2b

| Time | Sample point | Mass without acetone | Mass acetone | Total mass | $A_{1,ethanol}$ | $A_{2,water}$ | $A_{3,cyclohexane}$ | $A_{4,acetone}$ |
|---|---|---|---|---|---|---|---|---|
| 0 | Feed | | | | | | | |
| 15 | | 1.7762 | 0.4032 | 2.1794 | 232808.9 | 28955.4 | 621198.2 | 135597 |
| 25 | | 2.5514 | 1.0065 | 3.5579 | 183494 | 18702.9 | 572757.3 | 198132 |
| 35 | | 3.8953 | 1.0485 | 4.9438 | 395709.9 | 475321.9 | 16792.3 | 280977.8 |
| 45 | | 1.6295 | 0.5134 | 2.1429 | 258485 | 9347.7 | 624644.1 | 240586.2 |
| 55 | | 6.3056 | 2.693 | 8.9986 | 101534.4 | 73052.4 | 391387.1 | 326180 |
| | | | | | | | | |
| 15 | Bottom point | 5.8187 | 4.8132 | 10.6319 | 209280.2 | 102929.7 | 187957.9 | 481417.5 |
| 25 | | 7.2829 | 3.5577 | 10.8406 | 299111.8 | 131904 | 139736.1 | 444842 |
| 35 | | 2.2918 | 1.451 | 3.7428 | 263729.6 | 121665 | 165225.7 | 395384.9 |
| 45 | | 5.722 | 0.9734 | 6.6954 | 563686.5 | 271832.3 | 63317.6 | 183536.9 |
| 55 | | 5.1192 | 1.0097 | 6.1289 | 573045.8 | 70930.3 | 33060.2 | 202835 |
| | | | | | | | | |
| 15 | Top point | 7.8076 | 4.8057 | 12.6133 | 341733.7 | 125124.5 | 113142 | 404432.7 |
| 25 | | 3.3766 | 1.0127 | 4.3893 | 524086.6 | 216555.6 | 33279.1 | 292022.7 |
| 35 | | 6.1833 | 3.6283 | 9.8116 | 343580.9 | 140037.6 | 142589.4 | 409575.9 |
| 45 | | 2.8897 | 1.0254 | 3.9151 | 207972.7 | 207972.7 | 43742.2 | 307008.5 |
| 55 | | 7.7332 | 4.8081 | 12.5413 | 78863.7 | 136217 | 918863.7 | 401824.2 |

# APPENDIX B :SAMPLE CALCULATIONS

The sample calculations for the top sampling point (raffinate) for semi- batch run 2 at 55 minutes are shown in this section.

## B.1. Calibration Uncertainty

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The sample calculation for the uncertainty of the ethanol-acetone system in the acetone rich region is shown as follows:

The peak areas of ethanol and acetone were obtained from the gas chromatograph. The sample was injected twice to allow for repeatability.

First injection:

$$A_{1,ethanol} = 81\ 309$$

$$A_{4,acetone} = 913514$$

Second injection:

$$A_{1,ethanol} = 78\ 614.7$$

$$A_{4,acetone} = 886029.9$$

The ratio of these areas were then calculated. Since this sample was in the acetone rich region, the ratio $\frac{A_1}{A_4}$ was used.

First injection:

$$\frac{A_1}{A_4} = \frac{81309}{913514} = 0.0890$$

Second injection:

$$\frac{A_1}{A_4} = \frac{78614.7}{886029.9} = 0.0887$$

The standard deviation of the above ratios was calculated using the standard deviation function on excel STD.P. The standard deviation obtained was $1.4\ x10^{-4}$.

The average of these two ratios was also calculated:

$$Average = \frac{0.0890 + 0.0887}{2} = 8.885\ x10^{-2}$$

The error in calibration was then computed as follows:

$$Percentage\ error = \frac{standard\ deviation}{average} x100 = \frac{1.4x10^{-4}}{8.885x10^{-2}} x100 = 0.157$$

## B.2. Compositions of each component in samples

The calibration plots were used to obtain the masses of each component present in the samples. The procedure is outlined as follows:

The sample was weighed using a mass balance. The vial which contained the sample had a mass of 16.387g while the mass of the sample was 7.733g.

Acetone was added to the samples obtained from the column as the internal standard and to dissolve any immiscibility between the phases. The mass of acetone added to the top sample at 55 minutes was 4.808g.

The composition of acetone in the sample was then calculated:

$$x_{4,acetone} = \frac{m_{acetone}}{m_{total}} = \frac{4.808}{7.733 + 4.808} = 0.383$$

The peak areas obtained from the gas chromatograph for each component are indicated below:

$A_{1,ethanol} = 78863.7$

$A_{2,water} = 26217$

$A_{3,cyclohexane} = 918863.7$

$A_{4,acetone} = 401824.2$

### Composition of ethanol

The ratio of the areas of ethanol and acetone was then calculated. If $A_{1,ethanol} > A_{4,acetone}$, then the ratio $\frac{A_{4,acetone}}{A_{1,ethanol}}$ was used and the corresponding plot in the ethanol rich region, however if $A_{4,acetone} > A_{1,ethanol}$, then the ratio $\frac{A_{1,ethanol}}{A_{4,acetone}}$ was used and the corresponding plot in the acetone rich region was used. This procedure was repeated for all components.

Since $A_{4,acetone} > A_{1,ethanol}$, the ratio of $\frac{A_{1,ethanol}}{A_{4,acetone}}$ was used:

$$\frac{A_{1,ethanol}}{A_{4,acetone}} = \frac{78863.7}{401824.2} = 0.196$$

The corresponding calibration plot for the ethanol- acetone system in the acetone rich region as shown in Figure C- 1: Calibration of GC with ethanol- acetone system in the acetone rich region was then used to obtain the mass fraction ratios of ethanol and acetone. The gradient of the calibration plot was calculated to be 0.956.

The ratio of the mass fractions was obtained using the straight-line equation y =mx +c as follows:

$$\frac{A_{1,ethanol}}{A_{4,acetone}} = m.\frac{x_{1,ethanol}}{x_{4,acetone}}$$

$$\frac{x_{1,ethanol}}{x_{4,acetone}} = \frac{0.196}{0.956} = 0.205$$

The composition of ethanol in the sample was then calculated since the mass fraction of acetone in the sample was already known.

$$\frac{x_{1,ethanol}}{0.383} = 0.205$$

$$x_{1,ethanol} = 0.0785$$

The mass of ethanol in the sample was thereafter computed.

$$m_{ethanol} = x_{1,ethanol} * m_{total} = 0.0785 * 12.541 = 0.984g$$

<u>Composition of water</u>

Similarly, the masses of the other components in the sample were computed using the corresponding plots in Appendix C. Since $A_{4,acetone} > A_{2,water}$, the ratio of $\frac{A_{2,water}}{A_{4,acetone}}$ was used:

$$\frac{A_{2,water}}{A_{4,acetone}} = \frac{26217}{401824.2} = 0.0652$$

The corresponding calibration plot for the water- acetone system in the acetone rich region as shown in Figure C- 3 was then used to obtain the mass fraction ratios of water and acetone. The gradient of the calibration plot was calculated to be 1.115.

The ratio of the mass fractions was obtained using the straight-line equation y =mx +c as follows:

$$\frac{A_{2,water}}{A_{4,acetone}} = m.\frac{x_{2,water}}{x_{4,acetone}}$$

$$\frac{x_{2,water}}{x_{4,acetone}} = \frac{0.0652}{1.115} = 0.0585$$

The composition of water in the sample was then calculated since the mass fraction of acetone in the sample was already known.

$$\frac{x_{2,water}}{0.383} = 0.0585$$

$$x_{2,water} = 0.0224$$

The mass of water in the sample was thereafter computed.

$$m_{water} = x_{2,water} \ * m_{total} = 0.0224 * 12.541 = 0.281g$$

Composition of cyclohexane

Since the compositions of the other 3 components in the sample are known, the composition of cyclohexane can now be calculated:

$$x_{3,cyclohexane} = 1 - (x_{1,ethanol} \ + x_{2,water} + x_{4,acetone})$$

$$= 1 - (0.0785 + 0.0224 + 0.383) = 0.516$$

The mass of ethanol in the sample was thereafter computed.

$$m_{ethanol} = x_{1,ethanol} \ * m_{total} = 0.516 * 12.541 = 6.471g$$

$m_{cyclohexane} = 6.471g$

Acetone was not in the original sample obtained from the column and thus the original compositions of cyclohexane, ethanol and water were calculated.

$x_{ethanol} = \frac{0.984}{7.733} = 0.127$

$x_{water} = \frac{0.281}{7.733} = 0.0036$

$$x_{cyclohexane} = \frac{6.471}{7.733} = 0.836$$

These compositions were then plotted on the composition profile for the semi- batch runs.

B.3. Percentage Extracted

In order to calculate the percentage extracted, a mole balance had to be carried out over the VPE column.

The total mass of the feed was 11.03kg while the mass of ethanol in the feed was 5.74kg. The mass fraction of ethanol in the feed was then computed:

$$x_{ethanol,feed} = \frac{m_{ethanol,feed}}{m_{feed}} = \frac{5.74}{5.74 + 5.29} = 0.520$$

The mass fraction of ethanol in the raffinate was previously calculated to be 0.127, while the mass fraction of ethanol in the extract at the end of the run (55 minutes) was obtained as 0.634. The following ethanol balance and overall balance were solved simultaneously:

$$x_{ethanol,feed}F = x_{ethanol,raffinate}R + x_{ethanol,extract}E$$

$$0.520F = 0.127R + 0.634E$$

There was no ethanol in the solvent and thus the solvent term was neglected. Solving the above equation for E yields:

$$E = \frac{0.520F - 0.127R}{0.634}$$

The overall balance is shown as follows:

$$F + S = 4 + 11.03 = 15.03$$

$$F + S = 15.03 = E + R$$

Substituting for E and solving for R:

$$\frac{5.74 - 0.127R}{0.634} + R = 15.03$$

Therefore, R=7.473kg and E=7.557kg.

The number of moles of ethanol in the feed was then calculated:

$$n_{ethanol,feed} = \frac{m_{ethanol,feed}}{M_{ethanol}} = \frac{5.74}{46.070} = 0.126\ kmol$$

The number of moles of ethanol in the raffinate was also calculated:

$$n_{ethanol,raffinate} = \frac{m_{ethanol,raffinate}}{M_{ethanol}} = \frac{0.127\ (7.473)}{46.070} = 0.0206\ kmol$$

Therefore, the percentage extracted was computed as follows:

$$Percentage\ extracted = \frac{n_{feed} - n_{raffinate}}{n_{feed}} x\ 100$$

$$= \frac{0.126 - 0.0206}{0.126}\ x\ 100$$

$$= 83.651\ \%$$

# APPENDIX C : ADDITIONAL RESULTS

## C.1. Calibration plots

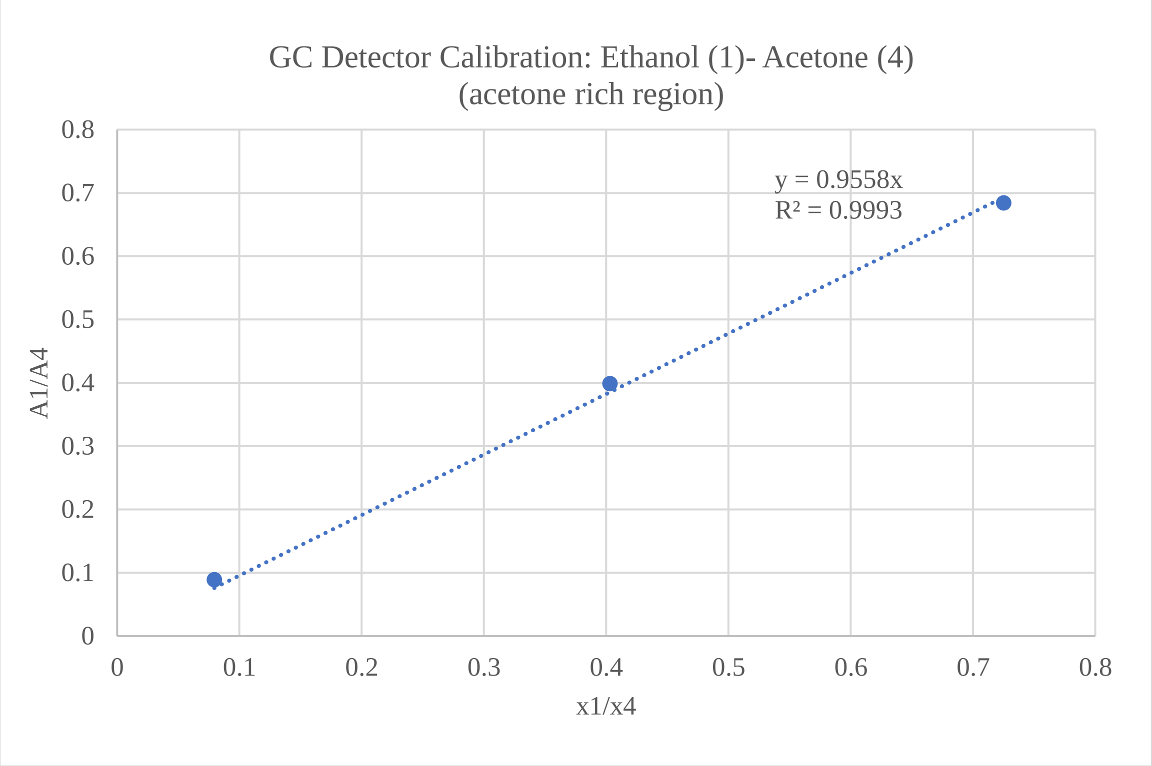

**Figure C- 1: Calibration of GC with ethanol- acetone system in the acetone rich region**

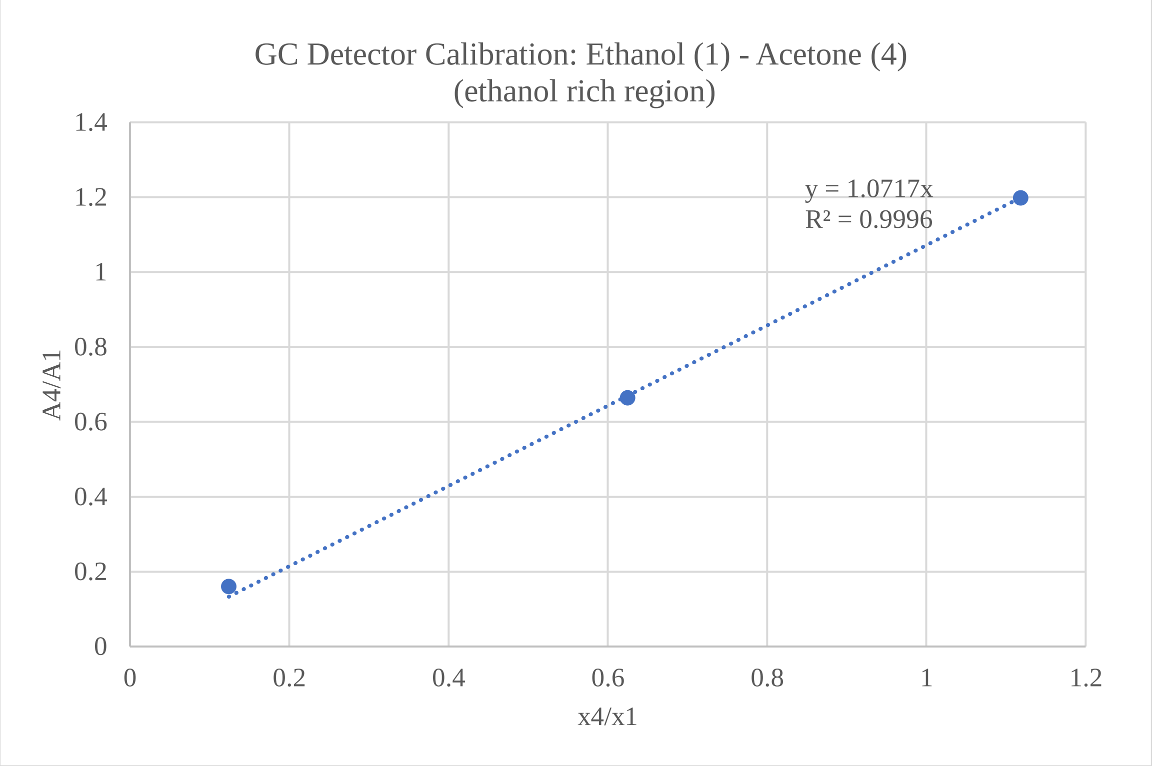

**Figure C- 2: Calibration of GC with ethanol- acetone system in the ethanol rich region**

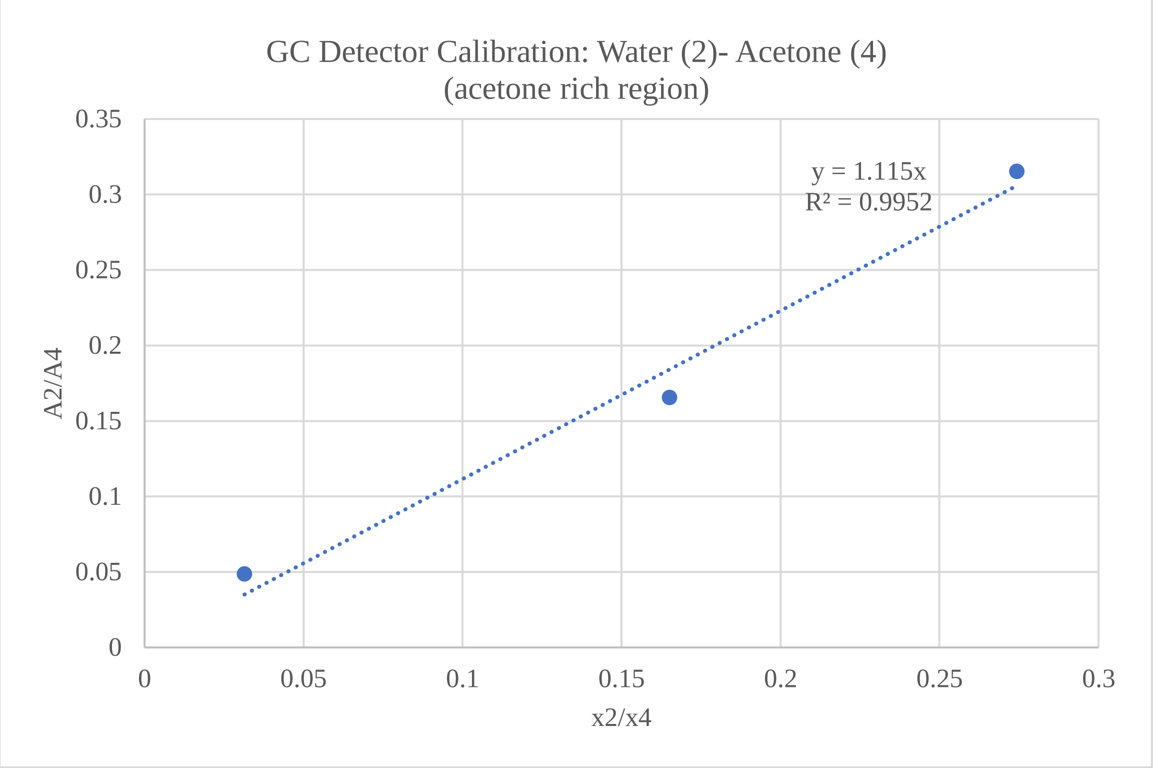

**Figure C- 3: Calibration of GC with water- acetone system in the acetone rich region**

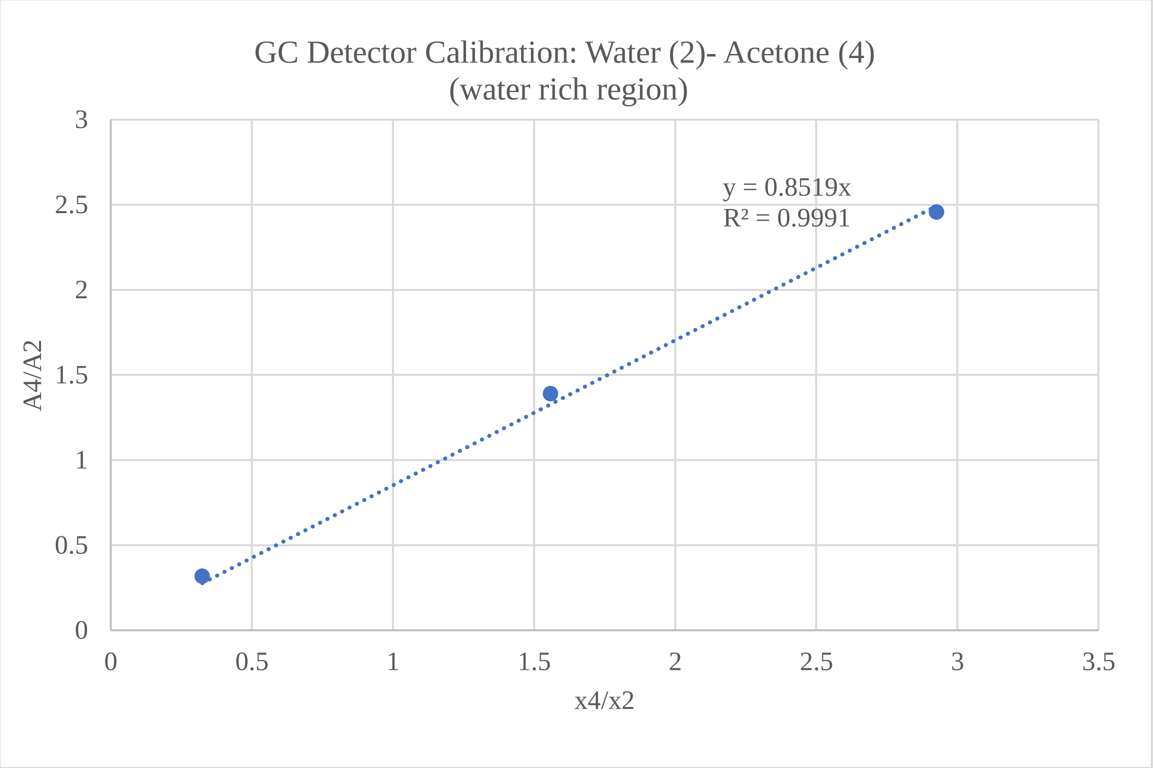

**Figure C- 4: Calibration of GC with water- acetone system in the water rich region**

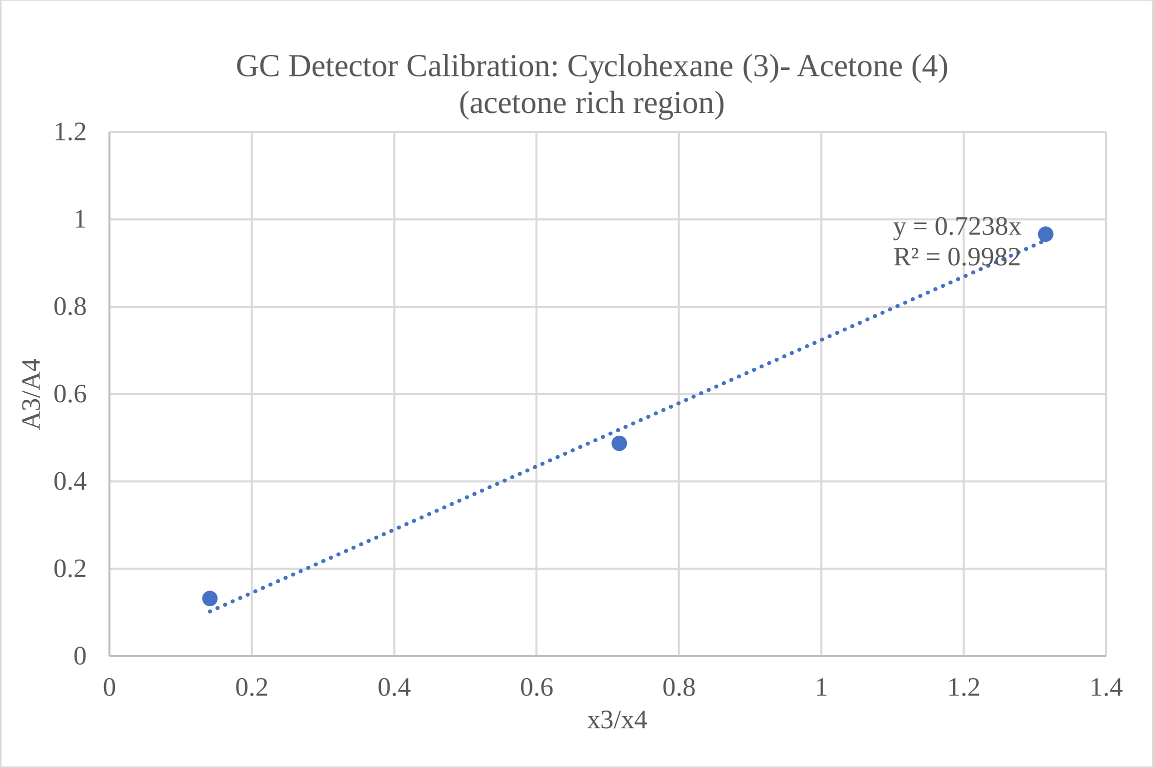

**Figure C- 5: Calibration of GC with cyclohexane- acetone system in the acetone rich region**

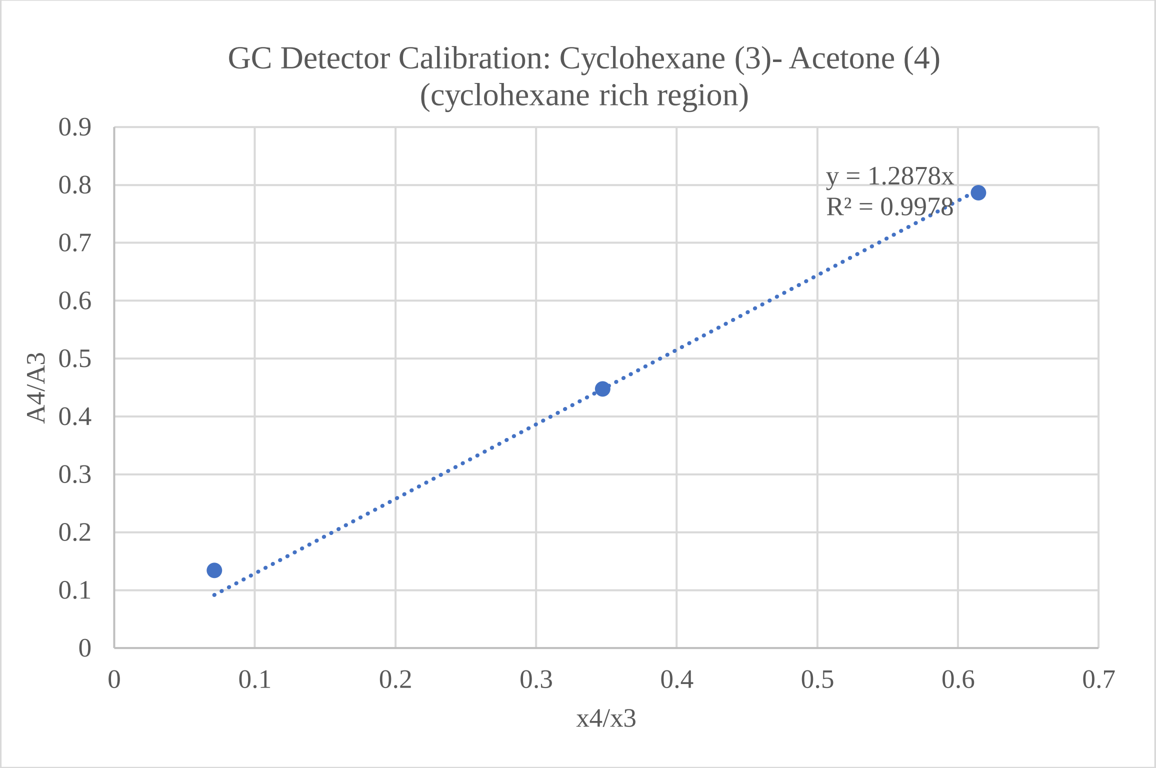

**Figure C- 6: Calibration of GC with cyclohexane- acetone system in the cyclohexane rich region**

C.3. Calibration Uncertainties

**Table C- 1: Calibration uncertainty for ethanol- acetone system in acetone rich region**

| $x_1/x_2$ | $A_{1,ethanol}$ | $A_{4,acetone}$ | $A_1/A_4$ | Standard Deviation | Average | Error | % Error |
|---|---|---|---|---|---|---|---|
| 0.1 | 81309 | 913514 | 0.089 | | | | |
| | 78614.7 | 886029.9 | 0.089 | 0.000 | 0.089 | 0.002 | 0.157 |
| 0.5 | 286630.1 | 708347.7 | 0.405 | | | | |
| | 267599.8 | 680872.5 | 0.393 | 0.006 | 0.399 | 0.015 | 1.457 |
| 0.9 | 393527.2 | 572550.3 | 0.687 | | | | |
| | 402785.1 | 591435.5 | 0.681 | 0.003 | 0.684 | 0.005 | 0.460 |
| | | | | | | Average % error | 0.691 |

**Table C- 2: Calibration uncertainty for ethanol- acetone system in ethanol rich region**

| $x_2/x_1$ | $A_{1,ethanol}$ | $A_{4,acetone}$ | $A_4/A_1$ | Standard Deviation | Average | Error | % Error |
|---|---|---|---|---|---|---|---|
| 0.1 | 831687.9 | 133408 | 0.160 | | | | |
| | 867635.6 | 139464.9 | 0.161 | 0.000 | 0.161 | 0.001 | 0.104 |
| 0.5 | 604096.5 | 399088.6 | 0.661 | | | | |
| | 593446.6 | 396187.7 | 0.668 | 0.003 | 0.664 | 0.005 | 0.525 |
| 0.9 | 434648.8 | 522099.3 | 1.201 | | | | |
| | 453512.4 | 542417.2 | 1.196 | 0.003 | 1.199 | 0.002 | 0.215 |
| | | | | | | Average % error | 0.281 |

**Table C- 3: Calibration uncertainty for water- acetone system in acetone rich region**

| $x_2/x_4$ | $A_{2,water}$ | $A_{4,acetone}$ | $A_2/A_4$ | Standard Deviation | Average | Error | % Error |
|---|---|---|---|---|---|---|---|
| 0.1 | 45536.5 | 919134.7 | 0.050 | | | | |
| | 45815.9 | 953718.4 | 0.048 | 0.001 | 0.049 | 0.015 | 1.541 |
| 0.5 | 145864.9 | 873322.7 | 0.167 | | | | |
| | 144217.1 | 877633.4 | 0.164 | 0.001 | 0.166 | 0.008 | 0.814 |
| 0.9 | 252552.2 | 812272 | 0.311 | | | | |
| | 255219.3 | 797808.1 | 0.320 | 0.004 | 0.315 | 0.014 | 1.424 |
| | | | | | | Average % error | 1.260 |

**Table C- 4: Calibration uncertainty for water- acetone system in water rich region**

| $x_4/x_2$ | $A_{2,water}$ | $A_{4,acetone}$ | $A_4/A_2$ | Standard Deviation | Average | Error | % Error |
|---|---|---|---|---|---|---|---|
| 0.1 | 916268.1 | 303374.3 | 0.331 | | | | |
| | 994001.9 | 301598.7 | 0.303 | 0.014 | 0.317 | 0.044 | 4.362 |
| 0.5 | 504154.2 | 683547.5 | 1.356 | | | | |
| | 470804.3 | 669493.3 | 1.422 | 0.033 | 1.389 | 0.024 | 2.383 |
| 0.9 | 320804.5 | 800817.9 | 2.496 | | | | |
| | 334870 | 808590.8 | 2.415 | 0.041 | 2.455 | 0.017 | 1.662 |
| | | | | | | Average % error | 2.802 |

**Table C- 5: Calibration uncertainty for cyclohexane- acetone system in cyclohexane rich region**

| $x_4/x_3$ | $A_{4,acetone}$ | $A_{3,cyclohexane}$ | $A_4/A_3$ | Standard Deviation | Average | Error | % Error |
|---|---|---|---|---|---|---|---|
| 0.1 | 11049.6 | 87110.3 | 0.127 | | | | |
| | 13235.3 | 93582.1 | 0.141 | 0.007 | 0.134 | 0.054 | 5.436 |
| 0.5 | 250679.6 | 634479.9 | 0.395 | | | | |
| | 398149.8 | 796802.2 | 0.500 | 0.052 | 0.447 | 0.117 | 11.689 |
| 0.9 | 372686.6 | 487167.8 | 0.765 | | | | |
| | 354460.6 | 438783.7 | 0.808 | 0.021 | 0.786 | 0.027 | 2.722 |
| | | | | | | Average % error | 6.616 |

**Table C- 6: Calibration uncertainty for cyclohexane- acetone system in acetone rich region**

| $x_3/x_4$ | $A_{4,acetone}$ | $A_{3,cyclohexane}$ | $A_3/A_4$ | Standard Deviation | Average | Error | % Error |
|---|---|---|---|---|---|---|---|
| 0.1 | 91210.8 | 12632.7 | 0.139 | | | | |
| | 91862.4 | 11556.8 | 0.126 | 0.006 | 0.132 | 0.048 | 4.803 |
| 0.5 | 251782.4 | 121802.5 | 0.484 | | | | |
| | 255329.8 | 125203.5 | 0.490 | 0.003 | 0.487 | 0.007 | 0.677 |
| 0.9 | 485291.6 | 491502.8 | 1.013 | | | | |
| | 446943.6 | 410607.7 | 0.919 | 0.047 | 0.966 | 0.049 | 4.872 |
| | | | | | | Average % error | 3.451 |

## C.4. Compositions of samples from runs

**Table C- 7: Batch run 1a extract and raffinate compositions**

| | $x_{ethanol}$ | $x_{water}$ | $x_{cyclohexane}$ |
|---|---|---|---|
| Feed | 0.490 | 0 | 0.510 |
| Extract | 0.390 | 0.303 | 0.307 |
| Raffinate | 0.261 | 0.021 | 0.718 |

**Table C- 8: Batch run 1b extract and raffinate compositions**

| | $x_{ethanol}$ | $x_{water}$ | $x_{cyclohexane}$ |
|---|---|---|---|
| Feed | 0.490 | 0 | 0.510 |
| Extract | 0.457 | 0.503 | 0 |
| Raffinate | 0.209 | 0 | 0.791 |

✓

## Table C- 9: Feed, extract and raffinate compositions for semi-batch run 2a

| Time | Sample point | Mass of ethanol | Mass of water | Mass of cyclohexane | Mass of sample | $x_{ethanol}$ | $x_{water}$ | $x_{cyclohexane}$ |
|---|---|---|---|---|---|---|---|---|
| 0 | Feed | 6.360 |  | 5.420 | 0.000 | 0.540 | 0.000 | 0.460 |
| 15 |  | 0.171 | 0.009 | 0.167 | 0.341 | 0.501 | 0.026 | 0.489 |
| 35 |  | 1.795 | 0.096 | 1.795 | 3.729 | 0.481 | 0.026 | 0.481 |
| 45 |  | 0.220 | 0.008 | 0.214 | 0.375 | 0.585 | 0.021 | 0.571 |
| 55 |  | 0.430 | 0.014 | 0.420 | 0.825 | 0.521 | 0.017 | 0.509 |
| 65 |  | 0.441 | 0.016 | 0.441 | 1.099 | 0.401 | 0.014 | 0.401 |
|  |  |  |  |  |  |  |  |  |
| 15 | Bottom point | 3.676 | 0.635 | 3.676 | 8.028 | 0.458 | 0.079 | 0.458 |
| 25 |  | 1.957 | 0.069 | 1.957 | 3.875 | 0.505 | 0.018 | 0.505 |
| 35 |  | 2.228 | 0.119 | 2.228 | 4.379 | 0.509 | 0.027 | 0.509 |
| 45 |  | 3.100 | 0.165 | 3.100 | 6.159 | 0.503 | 0.027 | 0.503 |
| 55 |  | 1.531 | 0.082 | 1.531 | 3.057 | 0.501 | 0.027 | 0.501 |
| 65 |  | 3.307 | 0.162 | 3.307 | 6.053 | 0.546 | 0.027 | 0.546 |
|  |  |  |  |  |  |  |  |  |
| 15 | Top point | 3.916 | 0.200 | 5.427 | 9.542 | 0.410 | 0.021 | 0.569 |
| 25 |  | 2.959 | 0.106 | 4.877 | 7.942 | 0.373 | 0.013 | 0.614 |
| 35 |  | 0.572 | 0.019 | 0.779 | 1.370 | 0.417 | 0.014 | 0.569 |
| 45 |  | 1.809 | 0.053 | 2.603 | 4.465 | 0.405 | 0.012 | 0.583 |
| 55 |  | 1.808 | 0.087 | 3.392 | 5.287 | 0.342 | 0.016 | 0.642 |
| 65 |  | 0.714 | 0.047 | 2.400 | 3.162 | 0.226 | 0.015 | 0.759 |

### Table C- 10: Feed, extract and raffinate compositions for semi-batch run 2b

| Time | Sample point | Mass of ethanol | Mass of water | Mass of cyclohexane | Mass of sample | $x_{ethanol}$ | $x_{water}$ | $x_{cyclohexane}$ |
|---|---|---|---|---|---|---|---|---|
| 0 | Feed | 5.740 | | 5.290 | | 0.520 | 0.000 | 0.480 |
| 15 | | 0.742 | 0.077 | 0.957 | 1.776 | 0.418 | 0.043 | 0.539 |
| 25 | | 0.975 | 0.085 | 1.491 | 2.551 | 0.382 | 0.033 | 0.584 |
| 35 | | 1.583 | 16.175 | 3.862 | 3.895 | 0.406 | 4.152 | 3.559 |
| 45 | | 0.591 | 0.018 | 1.020 | 1.630 | 0.363 | 0.011 | 0.626 |
| 55 | | 0.877 | 0.541 | 4.888 | 6.306 | 0.139 | 0.086 | 0.775 |
| | | | | | | | | |
| 15 | Bottom point | 2.189 | 0.923 | 2.707 | 5.819 | 0.376 | 0.159 | 0.465 |
| 25 | | 2.503 | 0.946 | 3.834 | 7.283 | 0.344 | 0.130 | 0.526 |
| 35 | | 1.013 | 0.400 | 0.879 | 2.292 | 0.442 | 0.175 | 0.383 |
| 45 | | 3.204 | 36.500 | 3.982 | 5.722 | 0.560 | 6.379 | 5.939 |
| 55 | | 3.057 | 0.317 | 1.745 | 5.119 | 0.597 | 0.062 | 0.341 |
| | | | | | | | | |
| 15 | Top point | 4.248 | 1.333 | 2.226 | 7.808 | 0.544 | 0.171 | 0.285 |
| 25 | | 1.948 | 0.674 | 0.755 | 3.377 | 0.577 | 0.199 | 0.224 |
| 35 | | 3.184 | 1.113 | 1.886 | 6.183 | 0.515 | 0.180 | 0.305 |
| 45 | | 0.727 | 0.623 | 1.540 | 2.890 | 0.251 | 0.216 | 0.533 |
| 55 | | 0.987 | 0.281 | 6.465 | 7.733 | 0.128 | 0.036 | 0.836 |

C.5. Equilibrium stages on  ethanol-cyclohexane-water ternary diagram

Number of stages: For experimental raffinate composition: 0.75 stages

For 99% cyclohexane in the raffinate: 2 stages

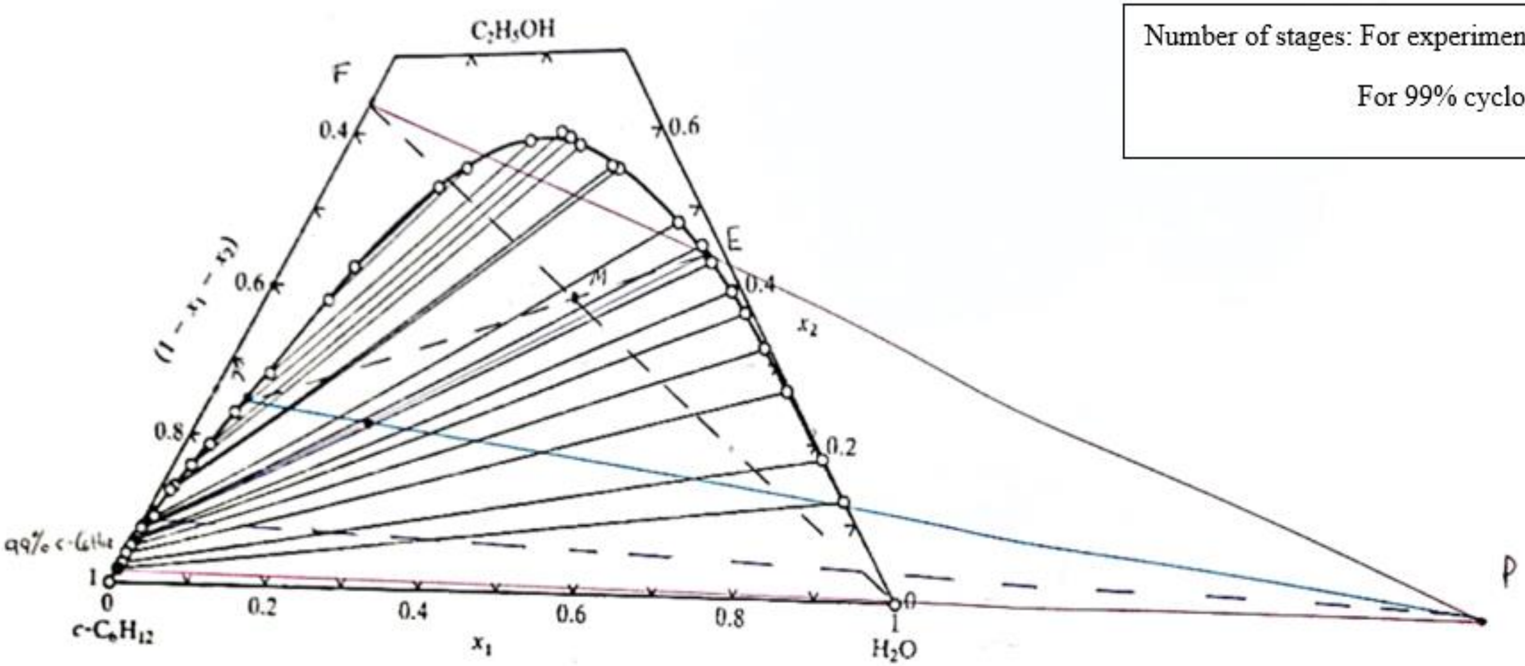

**Figure C- 7: Equilibrium stages for batch run 1a stepped off on ternary diagram**

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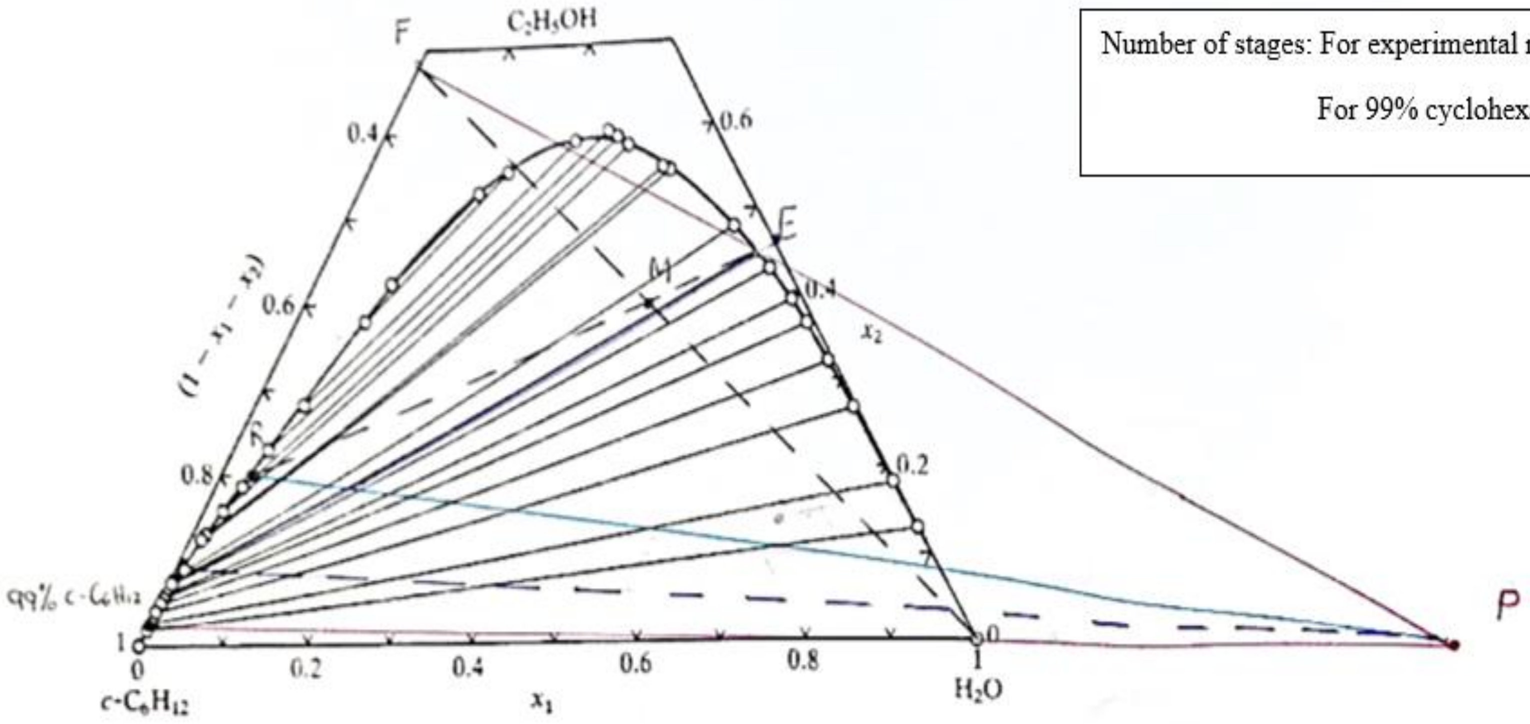


Number of stages: For experimental raffinate composition: 0.72 stages

For 99% cyclohexane in the raffinate: 1.8 stages

**Figure C- 8: Equilibrium stages for batch run 1b stepped off on ternary diagram**

Number of stages: For experimental raffinate composition: 0.45 stages

For 99% cyclohexane in the raffinate: 1.5 stages

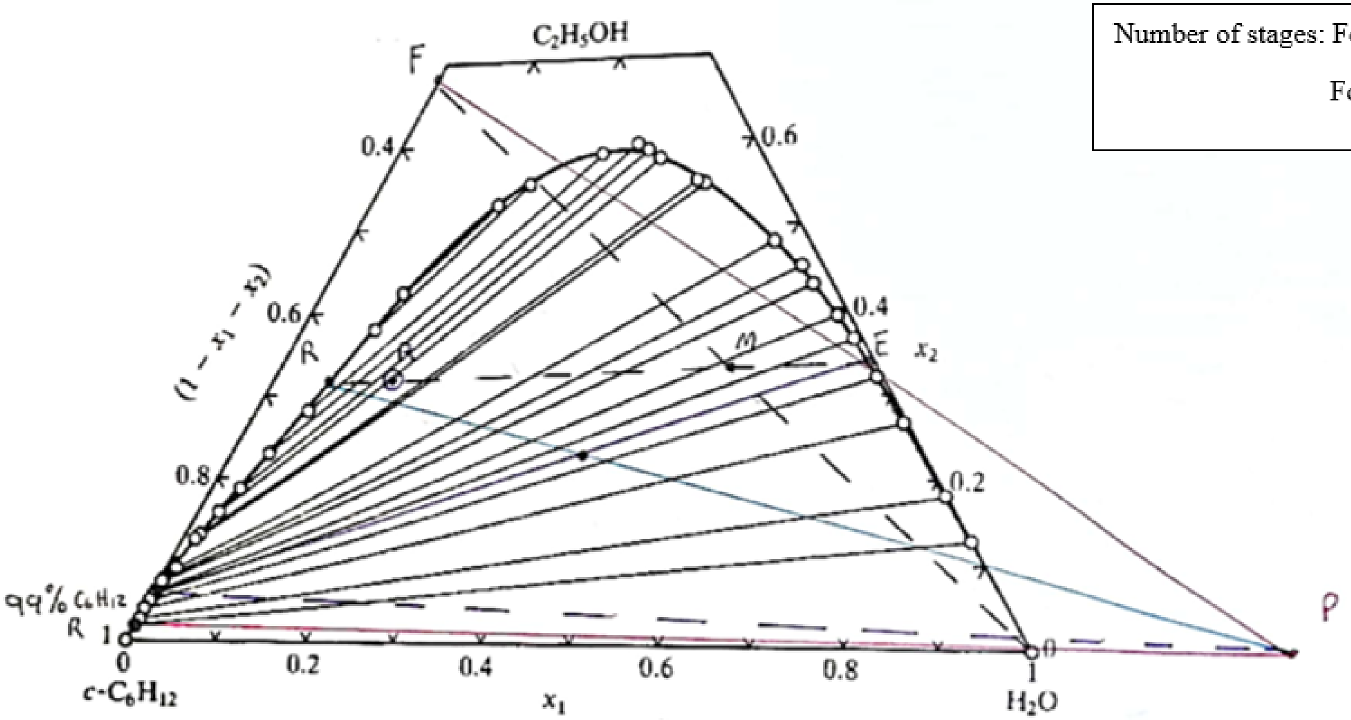


**Figure C- 9: Equilibrium stages for semi-batch run 2b stepped off on ternary diagram**

# APPENDIX D: MATERIAL AND SAFETY DATA SHEETS

## Safety Data Sheet

according to 29CFR1910/1200 and GHS Rev. 3

**Effective date** : 12.20.2014

Page 1 of 8

## Cyclohexane

### SECTION 1 : Identification of the substance/mixture and of the supplier

| | |
|---|---|
| **Product name** : | **Cyclohexane** |
| **Manufacturer/Supplier Trade name**: | |
| **Manufacturer/Supplier Article number**: | **S25292A** |

**Recommended uses of the product and uses restrictions on use**:

**Manufacturer Details**:

AquaPhoenix Scientific
9 Barnhart Drive, Hanover, PA 17331

**Supplier Details**:

Fisher Science Education
15 Jet View Drive, Rochester, NY 14624

**Emergency telephone number**:

Fisher Science Education    Emergency Telephone No.: 800-535-5053

### SECTION 2 : Hazards identification

**Classification of the substance or mixture**:

**Flammable**

**Health hazard**

**Irritant**

**Environmentally Damaging**

Flam Liq. 2
Skin irrit, cat 2
STOT SE 3
Asp. Tox. 1
Aquatic AcTox. 1
Aquatic ChrTox. 1

**Signal word** :Danger

**Hazard statements**:
Highly flammable liquid and vapour
Causes skin irritation
May cause drowsiness or dizziness
May be fatal if swallowed and enters airways
Very toxic to aquatic life with long lasting effects

**Precautionary statements**:

If medical advice is needed, have product container or label at hand
Keep out of reach of children
Read label before use
Keep container tightly closed
Wash ... thoroughly after handling
Avoid release to the environment
Do not eat, drink or smoke when using this product
Keep away from heat/sparks/open flames/hot surfaces. No smoking
Ground/bond container and receiving equipment
Use explosion-proof electrical/ventilating/light/.../equipment
Use only non-sparking tools
Take precautionary measures against static discharge
Use only outdoors or in a well-ventilated area
Wear protective gloves/protective clothing/eye protection/face protection
Avoid breathing dust/fume/gas/mist/vapours/spray
IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing
Collect spillage
IF ON SKIN: Wash with soap and water
IF ON SKIN (or hair): Remove/Take off immediately all contaminated clothing. Rinse skin with water/shower
Take off contaminated clothing and wash before reuse
If skin irritation occurs: Get medical advice/attention
IF SWALLOWED: Immediately call a POISON CENTER or doctor/physician
Specific treatment (see ... on this label)
Do NOT induce vomiting
In case of fire: Use ... for extinction
Store in a well ventilated place. Keep container tightly closed
Store in a well ventilated place. Keep cool
Store locked up
Dispose of contents/container to ...

**Other Non-GHS Classification**:

**WHMIS**

B2

D2B

**NFPA/HMIS**

NFPA SCALE (0-4)

| | |
|---|---|
| Health | 2 |
| Flammability | 3 |
| Physical Hazard | 0 |
| Personal Protection | X |

HMIS RATINGS (0-4)

## SECTION 3 : Composition/information on ingredients

| **Ingredients:** | | |
|---|---|---|
| CAS 110-82-7 | Cyclohexane | >95 % |
| | | Percentages are by weight |

## SECTION 4 : First aid measures

**Description of first aid measures**

**After inhalation:** Move exposed individual to fresh air. Loosen clothing as necessary and position individual in a comfortable position.Seek medical advice if discomfort or irritation persists.

**After skin contact:** Wash affected area with soap and water. Rinse/flush exposed skin gently using water for 15-20 minutes. Seek medical attention if irritation persists or if concerned.

**After eye contact:** Protect unexposed eye. Rinse/flush exposed eye(s) gently using water for 15-20 minutes. Remove contact lens(es) if able to do so during rinsing. Seek medical attention if irritation persists or if concerned.

**After swallowing:** Rinse mouth thoroughly. Do not induce vomiting. Have exposed individual drink sips of water. Seek medical attention if irritation, discomfort or vomiting persists.

**Most important symptoms and effects, both acute and delayed:**

Irritation, Nausea,Headache, Shortness of breath.;

**Indication of any immediate medical attention and special treatment needed:**

If seeking medical attention, provide SDS document to physician.

## SECTION 5 : Firefighting measures

**Extinguishing media**

**Suitable extinguishing agents:** Foam. Dry chemical powder. Carbon dioxide. If in laboratory setting, follow laboratory fire suppression procedures. Use appropriate fire suppression agents for adjacent combustible materials or sources of ignition

**For safety reasons unsuitable extinguishing agents:** Water may be ineffective because it may not cool the material below its flash point.

**Special hazards arising from the substance or mixture:**

Combustion products may include carbon oxides or other toxic vapors.Risk of ignition. Flammable. Vapors may travel to source of ignition and flash back. Containers may explode when heated.

**Advice for firefighters:**

**Protective equipment:**

**Additional information (precautions):** Move product containers away from fire or keep cool with water spray as a protective measure, where feasible.

## SECTION 6 : Accidental release measures

**Personal precautions, protective equipment and emergency procedures:**

Wear protective equipment. Use respiratory protective device against the effects of fumes/dust/aerosol. Keep unprotected persons away. Ensure adequate ventilation.Keep away from ignition sources. Protect from heat.Stop the spill, if possible. Contain spilled material by diking or using inert absorbent. Transfer to a disposal or recovery container.

**Environmental precautions:**

Prevent from reaching drains, sewer or waterway. Collect contaminated soil for characterization per Section 13

**Methods and material for containment and cleaning up:**

If in a laboratory setting, follow Chemical Hygiene Plan procedures.Collect liquids using vacuum or by use of absorbents. Place into properly labeled containers for recovery or disposal. If necessary, use trained response staff/contractor.

**Reference to other sections:**

## SECTION 7 : Handling and storage

**Precautions for safe handling:**

Prevent formation of aerosols. Follow good hygiene procedures when handling chemical materials. Do not eat, drink, smoke, or use personal products when handling chemical substances. Avoid splashes or spray in enclosed areas.Use only under chemical fume hood. Wear personal protective equipment. Wash hands after handling. Avoid contact with skin, eyes, and clothing, Do not breathe in vapors or spray mist. Keep away from open flames, hot surfaces and sources of ignitions. Use only non-sparking tools. Use explosion proof equipment. Take precautionary measures against static discharge.

**Conditions for safe storage, including any incompatibilities:**

Provide ventilation for containers. Avoid storage near extreme heat, ignition sources or open flame. Store away from foodstuffs. Store away from oxidizing agents.Store in cool, dry conditions in well sealed containers. Store with like hazards. Keep in a flammables area.

## SECTION 8 : Exposure controls/personal protection

| | |
|---|---|
| **Control Parameters:** | 110-82-7, Cyclohexane , ACGIH TLV 100 ppm TWA<br>110-82-7 , Cyclohexane , NIOSH 1300 ppm IDLH (10% LEL)<br>110-82-7, Cyclohexane, NIOSH 300 ppm TWA; 1050 mg/m3 TWA |
| **Appropriate Engineering controls:** | Emergency eye wash fountains and safety showers should be available in the immediate vicinity of use/handling.Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapor or mists below the applicable workplace exposure limits (Occupational Exposure Limits-OELs) indicated above.Use only under fume hood. |
| **Respiratory protection:** | Not required under normal conditions of use. Use suitable respiratory protective device when high concentrations are present. Use suitable respiratory protective device when aerosol or mist is formed. For spills, respiratory protection may be advisable. |
| **Protection of skin:** | The glove material has to be impermeable and resistant to the product/ the substance/ the preparation being used/handled.Selection of the glove material on consideration of the penetration times, rates of diffusion and the degradation. |
| **Eye protection:** | Safety glasses with side shields or goggles. |
| **General hygienic measures:** | The usual precautionary measures are to be adhered to when handling chemicals. Keep away from food, beverages and feed sources. Immediately remove all soiled and contaminated clothing. Wash hands before breaks and at the end of work. Do not inhale gases/fumes/dust/mist/vapor/aerosols. Avoid contact with the eyes and skin. |

## SECTION 9 : Physical and chemical properties

| | | | |
|---|---|---|---|
| **Appearance (physical state,color)**: | Clear, colorless liquid. | **Explosion limit lower**:<br>**Explosion limit upper**: | Not Determined<br>Not Determined |
| **Odor**: | Sweet | **Vapor pressure**: | 104 mbar @ 20C |
| **Odor threshold**: | Not Determined | **Vapor density**: | 2.90 (Air = 1.0) |
| **pH-value**: | Not Determined | **Relative density**: | 0.770 |
| **Melting/Freezing point**: | 6.5 C | **Solubilities**: | Insoluble |
| **Boiling point/Boiling range**: | 81C | **Partition coefficient (n-octanol/water)**: | Not Determined |
| **Flash point (closed cup)**: | - 17.99 °C ( - 0.38 °F) - closed cup | **Auto/Self-ignition temperature**: | Not Determined |
| **Evaporation rate**: | 6.1 (Butyl Acetate) | **Decomposition temperature**: | Not Determined |
| **Flammability (solid,gaseous)**: | Not Determined | **Viscosity**: | a. Kinematic:Not Determined<br>b. Dynamic: Not Determined |
| **Density**: Not Determined | | | |

## SECTION 10 : Stability and reactivity

**Reactivity:**
**Chemical stability:**No decomposition if used and stored according to specifications.
**Possible hazardous reactions:**Vapours may form explosive mixture with air.
**Conditions to avoid:**Store away from oxidizing agents, strong acids or bases. Incompatible materials,excess heat. Keep away from open flames, hot surfaces and sources of ignition.
**Incompatible materials:**Strong acids.Strong bases.Strong oxidizing agents.
**Hazardous decomposition products:**Carbon oxides (CO, CO2).

## SECTION 11 : Toxicological information

| | | |
|---|---|---|
| **Acute Toxicity**: | | |
| **Dermal**: | >2000 mg/kg | Dermal LD50 Rabbit |
| **Inhalation**: | 13.9 mg/L 4 h | Inhalation LC50 Rat |
| **Oral**: | >5000 mg/kg | Oral LD50 Rat |
| **Chronic Toxicity**: No additional information. | | |
| **Corrosion Irritation**: No additional information. | | |
| **Sensitization**: | | Causes eye and skin irritation. |
| **Single Target Organ (STOT)**: | | No additional information. |
| **Numerical Measures**: | | No additional information. |

| | |
|---|---|
| **Carcinogenicity**: | No additional information. |
| **Mutagenicity**: | Mutagenic effects have occurred in microorganisms. |
| **Reproductive Toxicity**: | No additional information. |

## SECTION 12 : Ecological information

**Ecotoxicity**

**Freshwater Algae**: 72 Hr EC50 Desmodesmus subspicatus: >500 mg/L

**Freshwater Fish**: 96 Hr LC50 Pimephales promelas: 3.96 - 5.18 mg/L [flow-through]

**Freshwater Fish**: 96 Hr LC50 Pimephales promelas: 23.03 - 42.07 mg/L [static]

**Freshwater Fish**: 96 Hr LC50 Lepomis macrochirus: 24.99 - 44.69 mg/L [static]

**Freshwater Fish**: 96 Hr LC50 Poecilia reticulata: 48.87 - 68.76 mg/L [static]

**Persistence and degradability**: Readily degradable in the environment.
**Bioaccumulative potential**:
**Mobility in soil**: Aqueous solution has high mobility in soil.
**Other adverse effects**:

## SECTION 13 : Disposal considerations

**Waste disposal recommendations**:

Product/containers must not be disposed together with household garbage. Do not allow product to reach sewage system or open water.It is the responsibility of the waste generator to properly characterize all waste materials according to applicable regulatory entities (US 40CFR262.11). Consult federal state/ provincial and local regulations regarding the proper disposal of waste material that may incorporate some amount of this product.

## SECTION 14 : Transport information

**UN-Number**

1145

**UN proper shipping name**

Cyclohexane

**Transport hazard class(es)**

**Class:**
3 Flammable liquids

**Packing group**:II
**Environmental hazard**:
**Transport in bulk**:
**Special precautions for user**:

## SECTION 15 : Regulatory information

**United States (USA)**

**SARA Section 311/312 (Specific toxic chemical listings)**:

Acute, Fire

**SARA Section 313 (Specific toxic chemical listings)**:

110-82-7 Cyclohexane

**RCRA (hazardous waste code)**:

None of the ingredients is listed

**TSCA (Toxic Substances Control Act)**:

All ingredients are listed.

**CERCLA (Comprehensive Environmental Response, Compensation, and Liability Act)**:

110-82-7 Cyclohexane 1000 lbs

**Proposition 65 (California)**:

**Chemicals known to cause cancer**:

None of the ingredients is listed

**Chemicals known to cause reproductive toxicity for females**:

None of the ingredients is listed

**Chemicals known to cause reproductive toxicity for males**:

None of the ingredients is listed

**Chemicals known to cause developmental toxicity**:

None of the ingredients is listed

**Canada**

**Canadian Domestic Substances List (DSL)**:

All ingredients are listed.

**Canadian NPRI Ingredient Disclosure list (limit 0.1%)**:

None of the ingredients is listed

**Canadian NPRI Ingredient Disclosure list (limit 1%)**:

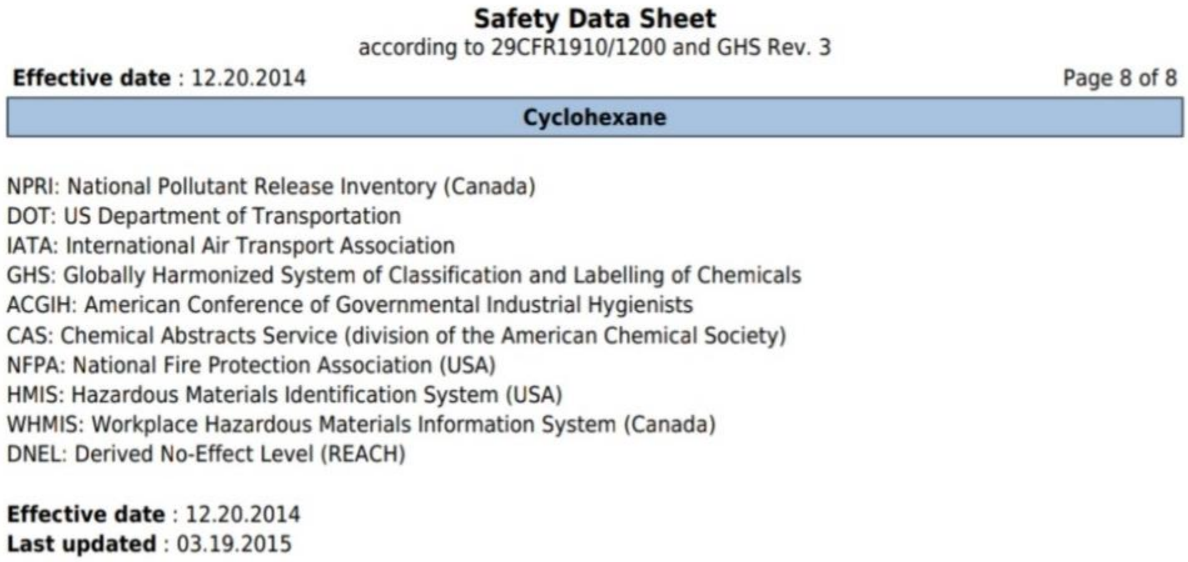
None of the ingredients is listed

## SECTION 16 : Other information

This product has been classified in accordance with hazard criteria of the Controlled Products Regulations and the SDS contains all the information required by the Controlled Products Regulations.Note:. The responsibility to provide a safe workplace remains with the user.The user should consider the health hazards and safety information contained herein as a guide and should take those precautions required in an individual operation to instruct employees and develop work practice procedures for a safe work environment.The information contained herein is, to the best of our knowledge and belief, accurate.However, since the conditions of handling and use are beyond our control, we make no guarantee of results, and assume no liability for damages incurred by the use of this material.It is the responsibility of the user to comply with all applicable laws and regulations applicable to this material.

**GHS Full Text Phrases**:

**Abbreviations and acronyms**:
IMDG: International Maritime Code for Dangerous Goods
PNEC: Predicted No-Effect Concentration (REACH)
CFR: Code of Federal Regulations (USA)
SARA: Superfund Amendments and Reauthorization Act (USA)
RCRA: Resource Conservation and Recovery Act (USA)
TSCA: Toxic Substances Control Act (USA)



**Safety Data Sheet**

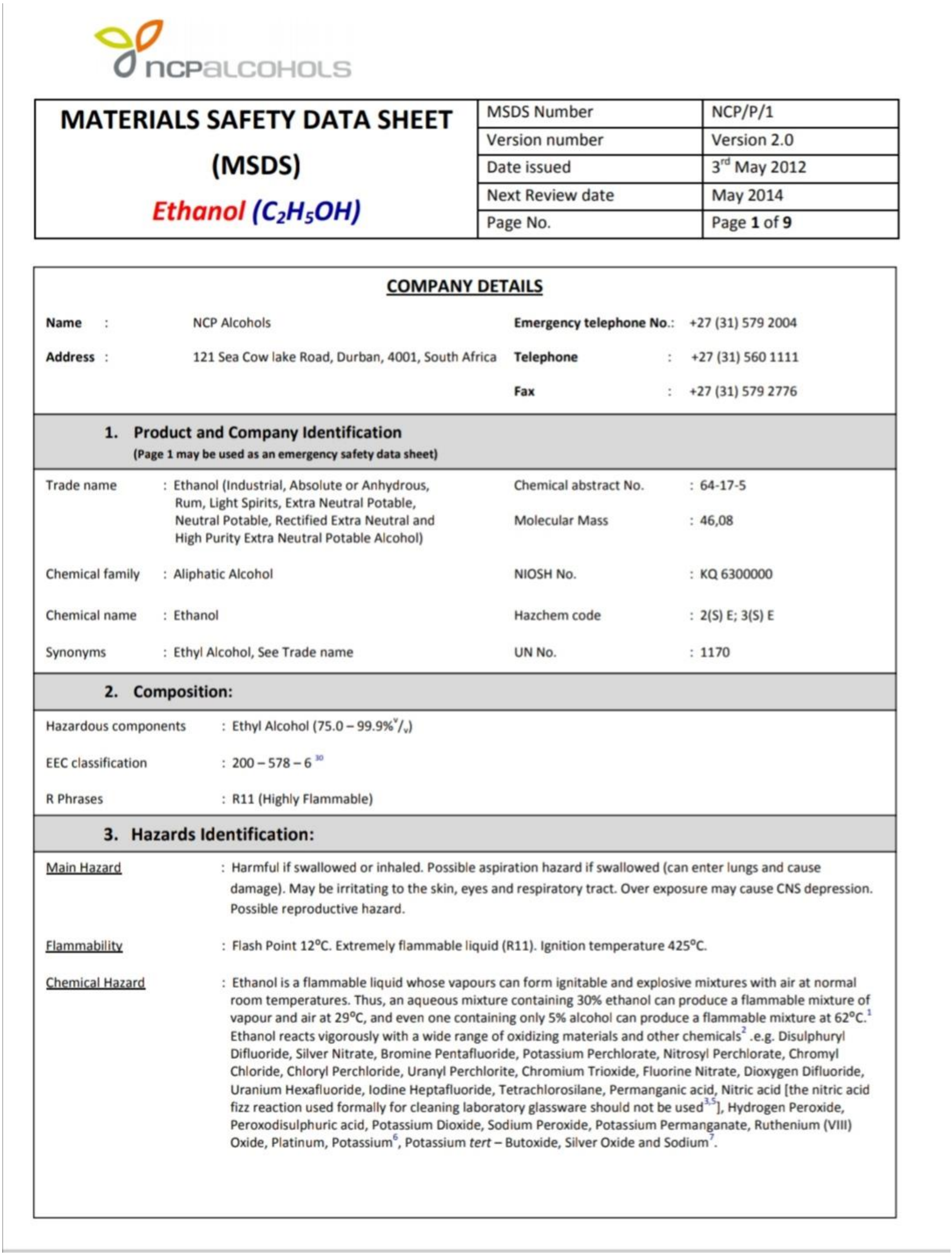
according to 29CFR1910/1200 and GHS Rev. 3

**Effective date** : 12.20.2014 Page 8 of 8

**Cyclohexane**

NPRI: National Pollutant Release Inventory (Canada)
DOT: US Department of Transportation
IATA: International Air Transport Association
GHS: Globally Harmonized System of Classification and Labelling of Chemicals
ACGIH: American Conference of Governmental Industrial Hygienists
CAS: Chemical Abstracts Service (division of the American Chemical Society)
NFPA: National Fire Protection Association (USA)
HMIS: Hazardous Materials Identification System (USA)
WHMIS: Workplace Hazardous Materials Information System (Canada)
DNEL: Derived No-Effect Level (REACH)

**Effective date** : 12.20.2014
**Last updated** : 03.19.2015



ncpalcohols

| MATERIALS SAFETY DATA SHEET (MSDS) Ethanol ($C_2H_5OH$) | MSDS Number | NCP/P/1 |
|---|---|---|
| | Version number | Version 2.0 |
| | Date issued | 3rd May 2012 |
| | Next Review date | May 2014 |
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## COMPANY DETAILS

| | | | |
|---|---|---|---|
| **Name** : | NCP Alcohols | **Emergency telephone No.**: | +27 (31) 579 2004 |
| **Address** : | 121 Sea Cow lake Road, Durban, 4001, South Africa | **Telephone** : | +27 (31) 560 1111 |
| | | **Fax** : | +27 (31) 579 2776 |

## 1. Product and Company Identification

**(Page 1 may be used as an emergency safety data sheet)**

| | | | |
|---|---|---|---|
| Trade name | : Ethanol (Industrial, Absolute or Anhydrous, Rum, Light Spirits, Extra Neutral Potable, Neutral Potable, Rectified Extra Neutral and High Purity Extra Neutral Potable Alcohol) | Chemical abstract No.<br>Molecular Mass | : 64-17-5<br>: 46,08 |
| Chemical family | : Aliphatic Alcohol | NIOSH No. | : KQ 6300000 |
| Chemical name | : Ethanol | Hazchem code | : 2(S) E; 3(S) E |
| Synonyms | : Ethyl Alcohol, See Trade name | UN No. | : 1170 |

## 2. Composition:

| | |
|---|---|
| Hazardous components | : Ethyl Alcohol (75.0 – 99.9%$^v/_v$) |
| EEC classification | : 200 – 578 – 6 [30] |
| R Phrases | : R11 (Highly Flammable) |

## 3. Hazards Identification:

| | |
|---|---|
| Main Hazard | : Harmful if swallowed or inhaled. Possible aspiration hazard if swallowed (can enter lungs and cause damage). May be irritating to the skin, eyes and respiratory tract. Over exposure may cause CNS depression. Possible reproductive hazard. |
| Flammability | : Flash Point 12°C. Extremely flammable liquid (R11). Ignition temperature 425°C. |
| Chemical Hazard | : Ethanol is a flammable liquid whose vapours can form ignitable and explosive mixtures with air at normal room temperatures. Thus, an aqueous mixture containing 30% ethanol can produce a flammable mixture of vapour and air at 29°C, and even one containing only 5% alcohol can produce a flammable mixture at 62°C.[1] Ethanol reacts vigorously with a wide range of oxidizing materials and other chemicals[2] .e.g. Disulphuryl Difluoride, Silver Nitrate, Bromine Pentafluoride, Potassium Perchlorate, Nitrosyl Perchlorate, Chromyl Chloride, Chloryl Perchloride, Uranyl Perchlorite, Chromium Trioxide, Fluorine Nitrate, Dioxygen Difluoride, Uranium Hexafluoride, Iodine Heptafluoride, Tetrachlorosilane, Permanganic acid, Nitric acid [the nitric acid fizz reaction used formally for cleaning laboratory glassware should not be used[3,5]], Hydrogen Peroxide, Peroxodisulphuric acid, Potassium Dioxide, Sodium Peroxide, Potassium Permanganate, Ruthenium (VIII) Oxide, Platinum, Potassium[6], Potassium *tert* – Butoxide, Silver Oxide and Sodium[7]. |

Biological Hazard : Ethanol is rapidly oxidized in the body to acetaldehyde, then to acetate, and finally to carbon dioxide and water; un-oxidized alcohol is excreted in the urine and expired in the air.[8, 9]

Reproductive hazard : Some evidence of foetotoxicity[26-28] and teratogenicity[29] has been observed in experimental animals treated with high doses of ethanol during gestation. Alcohol may induce spontaneous abortions, may impair fertility, may cause harm to the unborn child and may cause harm to breast fed babies. The reproductive hazards have been determined after repeated excessive consumption of ethanol; these effects are not likely to occur through exposure below the Occupational Exposure Limits in the working environment.

Health effects – eyes : Moderately irritating. Exposure to liquid, vapours, fumes or mist may cause irritation. Direct contact may cause irritation, redness, pain, corneal inflammation and possible corneal damage.

Health effects – skin : Repeated or prolonged contact may result in defatting, redness, pain, itching, inflammation, cracking and possible secondary infection. Repeated skin contact may result in allergic skin reaction in a very small proportion of individuals.

Health effects – ingestion : Large doses lead to alcohol poisoning while repeated ingestion can lead to alcoholism. Alcohol abuse and dependence can have a profound effect on work performance and tendency to accidents at work.[11-13] The presence of denaturants, e.g. Methanol, pyridines, and benzene in industrial alcohol greatly increase the toxicity on ingestion. Ethanol drinking is also suspected of increasing the toxic effect of other chemicals encountered in the laboratory and the workplace by inhibition of their metabolism or excretion[14]; e.g. 1, 1, 1 –Trichloroethane[15], Xylene, Trichloroethylene and Dimethylformamide[16], Benzene[17] and Lead.[18, 19] May cause harmful central nervous system effects. Effects may include excitation, euphoria, headache, dizziness, drowsiness, blurred vision, fatigue, tremors, convulsions, loss of consciousness, coma, respiratory arrest and death. Severe acute intoxication may cause Hypoglycaemia, Hypothermia and extensor rigidity. Prolonged or frequent contact may result in liver injury.

Health effects – inhalation : Intoxicating if continuously inhaled for a long period of time. Occupational Exposure Limits (8-hour reference period) 1000ppm (1900$mg/m^3$).[30]

Carcinogenicity : Long-term consumption of alcoholic beverages demonstrates an increase in the occurrence of breast cancer and colorectal cancer. Malignant tumours of the oral cavity, Pharynx, Larynx, Oesophagus and Liver is also causally related to the consumption of alcoholic beverages.[31] Some studies[20, 21] have shown an excess incidence of laryngeal cancer over the expected from exposure to synthetic alcohol, with Diethyl Sulphate probably being the causative agent.

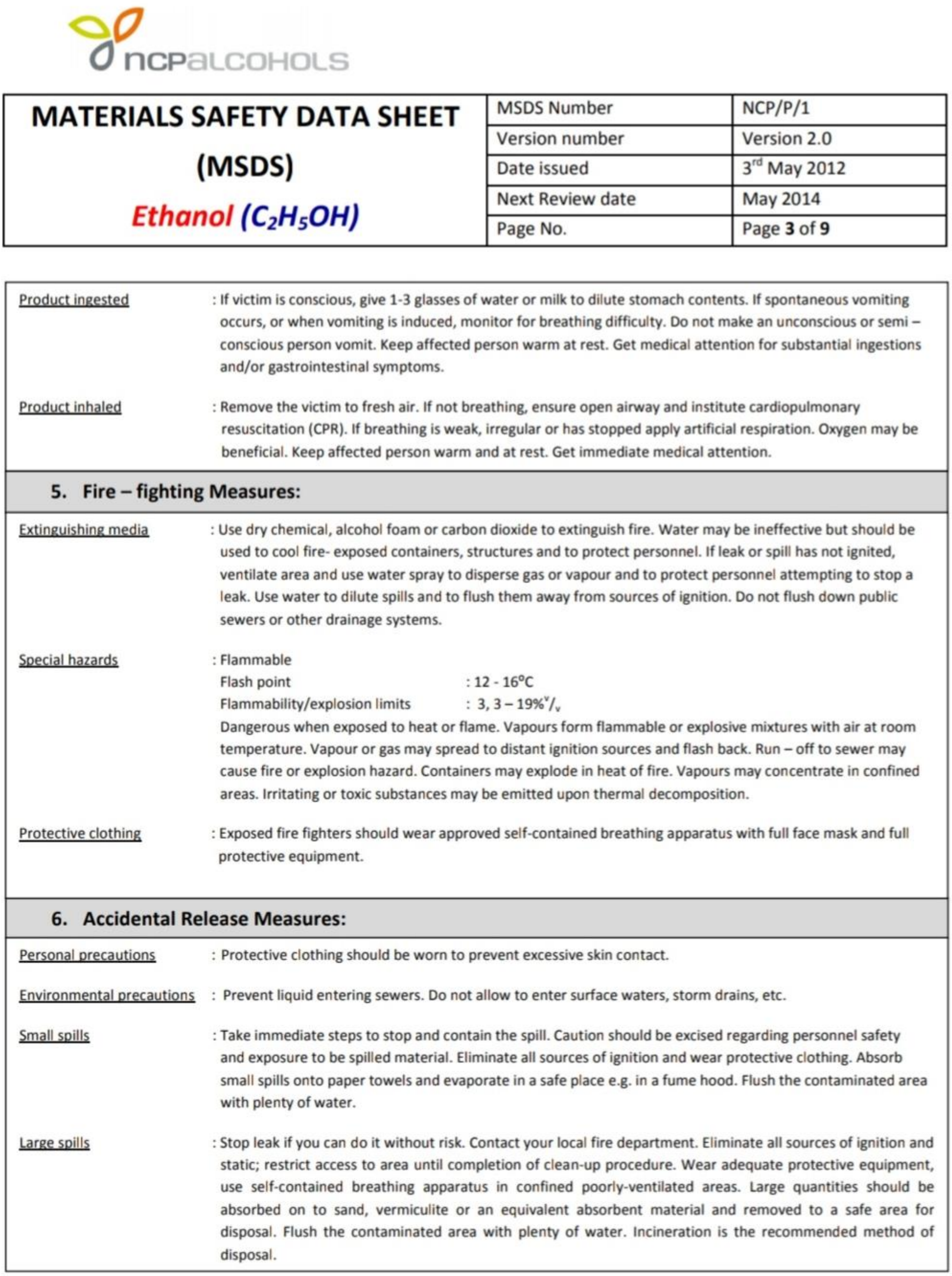
Mutagenicity : Ethanol has been found to be non- mutagenic in the *Salmonella* microsome test,[22] but some transient mutagenic changes have been observed in male, but not female, mice treated with rather large doses.[23-25] Ethanol is mutagenic in man via its first metabolite, Acetaldehyde. Acetaldehyde induces chromosomal aberrations, sister-chromatid exchanges and cross-links between DNA strands.[32]

Neurotoxicity : Over exposure may cause Central Nervous System (CNS) depression.

## 4. First – aid Measures:

Product in eye : Flush immediately with water or neutral saline solution for at least 15 minutes. Seek medical attention.

Product on skin : Remove contaminated clothing and rinse contaminated area with soap and water. If skin irritation persists seek medical attention.



| | |
|---|---|
| Product ingested | : If victim is conscious, give 1-3 glasses of water or milk to dilute stomach contents. If spontaneous vomiting occurs, or when vomiting is induced, monitor for breathing difficulty. Do not make an unconscious or semi – conscious person vomit. Keep affected person warm at rest. Get medical attention for substantial ingestions and/or gastrointestinal symptoms. |
| Product inhaled | : Remove the victim to fresh air. If not breathing, ensure open airway and institute cardiopulmonary resuscitation (CPR). If breathing is weak, irregular or has stopped apply artificial respiration. Oxygen may be beneficial. Keep affected person warm and at rest. Get immediate medical attention. |

## 5. Fire – fighting Measures:

| | |
|---|---|
| Extinguishing media | : Use dry chemical, alcohol foam or carbon dioxide to extinguish fire. Water may be ineffective but should be used to cool fire- exposed containers, structures and to protect personnel. If leak or spill has not ignited, ventilate area and use water spray to disperse gas or vapour and to protect personnel attempting to stop a leak. Use water to dilute spills and to flush them away from sources of ignition. Do not flush down public sewers or other drainage systems. |
| Special hazards | : Flammable<br>Flash point : 12 - 16°C<br>Flammability/explosion limits : 3, 3 – 19%$^v/_v$<br>Dangerous when exposed to heat or flame. Vapours form flammable or explosive mixtures with air at room temperature. Vapour or gas may spread to distant ignition sources and flash back. Run – off to sewer may cause fire or explosion hazard. Containers may explode in heat of fire. Vapours may concentrate in confined areas. Irritating or toxic substances may be emitted upon thermal decomposition. |
| Protective clothing | : Exposed fire fighters should wear approved self-contained breathing apparatus with full face mask and full protective equipment. |

## 6. Accidental Release Measures:

| | |
|---|---|
| Personal precautions | : Protective clothing should be worn to prevent excessive skin contact. |
| Environmental precautions | : Prevent liquid entering sewers. Do not allow to enter surface waters, storm drains, etc. |
| Small spills | : Take immediate steps to stop and contain the spill. Caution should be excised regarding personnel safety and exposure to be spilled material. Eliminate all sources of ignition and wear protective clothing. Absorb small spills onto paper towels and evaporate in a safe place e.g. in a fume hood. Flush the contaminated area with plenty of water. |
| Large spills | : Stop leak if you can do it without risk. Contact your local fire department. Eliminate all sources of ignition and static; restrict access to area until completion of clean-up procedure. Wear adequate protective equipment, use self-contained breathing apparatus in confined poorly-ventilated areas. Large quantities should be absorbed on to sand, vermiculite or an equivalent absorbent material and removed to a safe area for disposal. Flush the contaminated area with plenty of water. Incineration is the recommended method of disposal. |

## 7. Handling and Storage:

Suitable material : Ethanol is not corrosive to metals and may be stored in stainless steel, mild steel or aluminium containers. Ethanol may also be stored in HDPE containers.

Handling/ storage precautions : Ground lines and equipment used during transfer to reduce the possibility of static spark-initiated fire or explosion. Store in approved flammable liquid storage containers. Keep containers tightly closed as this material readily absorbs moisture. Store away from incompatible materials. Store in a cool, dry well-ventilated area away from sparks, flames and other sources of ignition. Eliminate all sources of static electricity. Use non –sparking electrical and ventilation systems. Storage criteria: Flammable Liquid store

## 8. Exposure Control / Personal Protection:

Occupational exposure limits :

| Country | 8 Hour – TWA Hygiene Limit | STEL |
|---|---|---|
| US (OSHA) | 1900 mg/m$^3$ (1000ppm) | None |
| US (ACGIH) | 1900 mg/m$^3$ (1000ppm) | None |
| Germany (MAK)* | 960 mg/m3 (500ppm) | Peak limit cat. II,1 |
| UK (OES) | 1920 mg/m$^3$ (1000ppm) | None |
| Slovak Republic | 960 mg/m3 (500ppm) | 1920 mg/m3 (1000ppm) (30 min, 4x per shift) |
| Czech Republic | 1000 mg/m$^3$ | 3000 mg/m$^3$ |

Engineering control measures : Engineering control methods to reduce hazardous exposures are preferred. General methods include mechanical ventilation (dilution and local exhaust), process or personnel enclosure, control of process conditions and process modification (e.g. substitution of a less hazardous material). Administrative controls and personal protective equipment may also be required. Use a non-sparking, grounded ventilation system separate from other exhaust ventilation systems. Exhaust directly to the outside. Supply sufficient replacement air to make up for air removed by exhaust system.

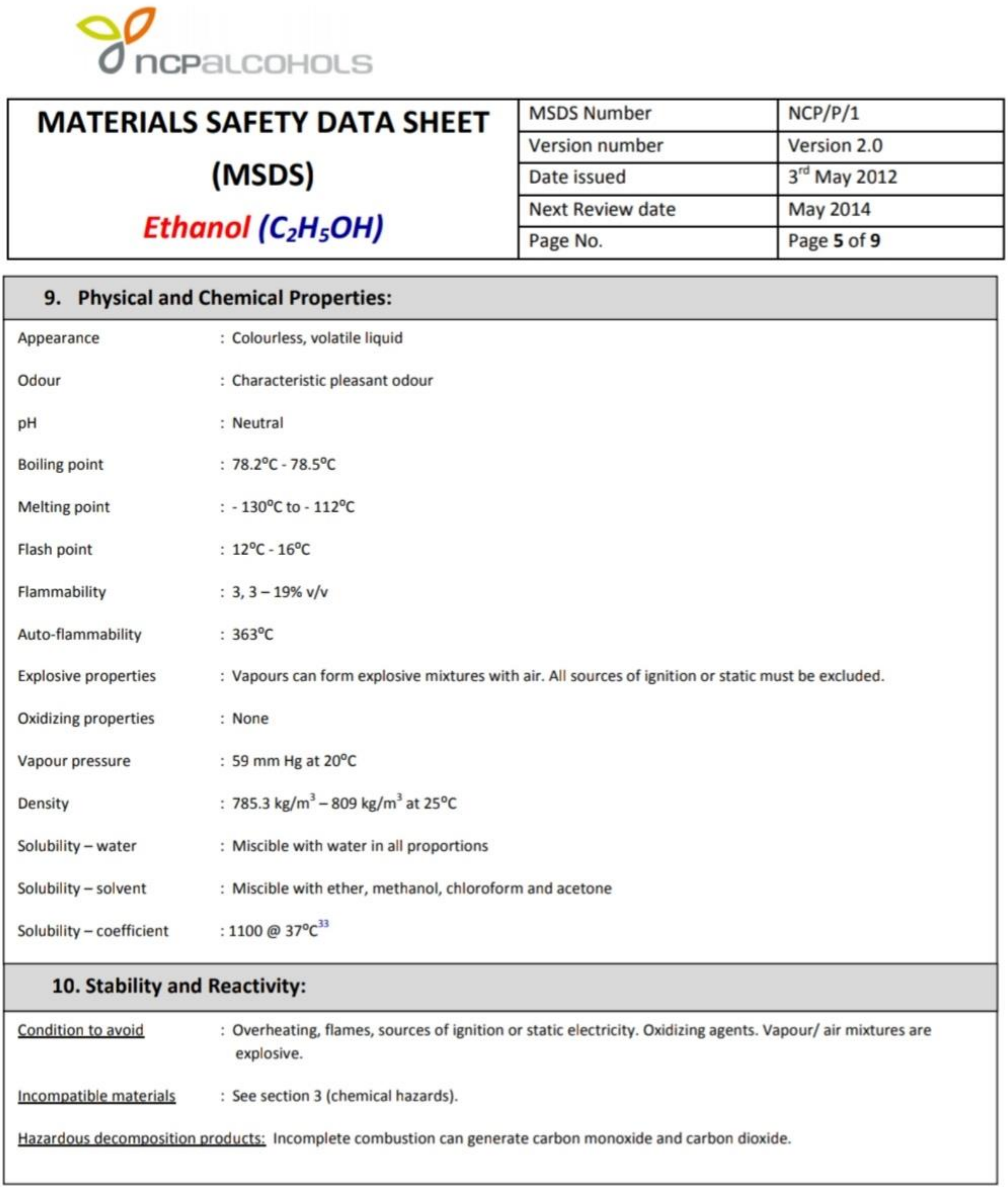
Personal protection – respiratory : If exposure limits are exceeded or if irritation is experienced, an approved respirator for organic vapours is generally acceptable. For high concentrations and for oxygen-deficient atmospheres, use approved air-supplied respirator. Full respiratory protection should be readily available in case of spillage.

Personal protection – hand : Rubber (Butyl) or neoprene gloves are recommended.

Personal protection – eye : Prevent eye contact with this material. Wear chemical tight safety goggles where eye exposure is reasonably probable. Provide an eyewash station immediately accessible to the work area. Contact lenses should not be worn when working with this chemical.

Personal protection – skin : Avoid skin contact. When working with this substance, wear appropriate chemical protective gloves. Depending upon conditions of use, additional protection may be necessary such as face shield, apron, etc.

Other protection : Provide a safety shower immediately accessible to the work area.



ncpalcohols

| MATERIALS SAFETY DATA SHEET (MSDS) Ethanol ($C_2H_5OH$) | MSDS Number | NCP/P/1 |
|---|---|---|
| | Version number | Version 2.0 |
| | Date issued | 3rd May 2012 |
| | Next Review date | May 2014 |
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## 9. Physical and Chemical Properties:

Appearance : Colourless, volatile liquid

Odour : Characteristic pleasant odour

pH : Neutral

Boiling point : 78.2°C - 78.5°C

Melting point : - 130°C to - 112°C

Flash point : 12°C - 16°C

Flammability : 3, 3 – 19% v/v

Auto-flammability : 363°C

Explosive properties : Vapours can form explosive mixtures with air. All sources of ignition or static must be excluded.

Oxidizing properties : None

Vapour pressure : 59 mm Hg at 20°C

Density : 785.3 kg/$m^3$ – 809 kg/$m^3$ at 25°C

Solubility – water : Miscible with water in all proportions

Solubility – solvent : Miscible with ether, methanol, chloroform and acetone

Solubility – coefficient : 1100 @ 37°C[33]

## 10. Stability and Reactivity:

Condition to avoid : Overheating, flames, sources of ignition or static electricity. Oxidizing agents. Vapour/ air mixtures are explosive.

Incompatible materials : See section 3 (chemical hazards).

Hazardous decomposition products: Incomplete combustion can generate carbon monoxide and carbon dioxide.

## 11. Toxicological Information:

Acute toxicity : Short-term hazards

Acute oral toxicity Ethanol : LD$_{50}$ rat: 7,060 mg/kg; literature value
Acute inhalation toxicity Ethanol : LC$_{50}$ rat: 66,000 mg/l; literature value; 4 h
Acute dermal toxicity Ethanol : LDLo rabbit: 20,000 mg/kg; literature value

NOAEL - 2400 mg/kg (2%) - for rats
LOAEL – 3600 mg/kg (3%) - for rats

Skin and eye contact : Redness, pain (refer to Section 3 for further information)

Chronic toxicity : Refer to Section 3

Carcinogenicity : Refer to Section 3

Mutagenicity : Refer to Section 3

Neurotoxicity : Refer to Section 3

Reproductive hazards : Refer to Section 3

## 12. Ecological Information:

Aquatic toxicity – fish : In high concentration it harms fish and plankton; LC$_{50}$ (fish, 96 hours) – 15.3 mg/L (Pimephales promelas)

Aquatic toxicity – daphnia : Threshold for deleterious effects in small crustaceans upwards of 7.800 mg/l; EC$_{50}$ (Daphnia, 48 hours) – 5012 mg/L (Ceriodaphnia dubia)

Aquatic toxicity – algae : Toxic threshold concentration: *Pseudomonas putida* upwards of 6.500mg/l, *Scenedesmus quadricauda* upwards of 5.000mg/l, *Microsystis aeruginosa* upwards of 1.450ml/L IC$_{50}$ (algae, 72 hours) – 275 mg/L

Biodegradability : This product is readily biodegradable. Ethanol is widely recognized as being readily biodegradable in the environment as it is both a metabolite of and nutrient for microbes. There are no persistent

Bio – accumulation : This product in not expected to bio accumulate through the food chains in the environment. The very low log KOW of –0.31 is indicative of a low bioaccumulation potential.

Mobility : This product is likely to volatize rapidly into the air because of its high vapour pressure. The product is poorly absorbed onto soils or sediments. Adsorption coefficient (K$_{OC}$) solid phase/liquid phase = 1 (highly mobile)

German wgk : 1 (low hazard to water)

## 13. Disposal Considerations:

Disposal methods : Only under conditions approved by local authorization. See also Section 6.

Disposal of packaging : Empty containers may contain flammable and hazardous residues. Always obey hazard warnings.

## 14. Transport Information:

UN No : 1170

Substance Identity No : UN 1170

ADR/RID class : 3

ADR/RID item No : 3(b)

ADR/RID hazard identity No : 3

IMDG – shipping name : Ethanol

IMDG – class : 3.2

IMDG – packaging group : II

IMDG – marine pollutant : Not a marine pollutant

IMDG – EMS No : F-E, S-D

IMDG – MFAG table No : 3074

IATA – shipping name : Ethanol Solutions

IATA – class : 3

IATA – subsidiary risk(s) : Flammable liquid

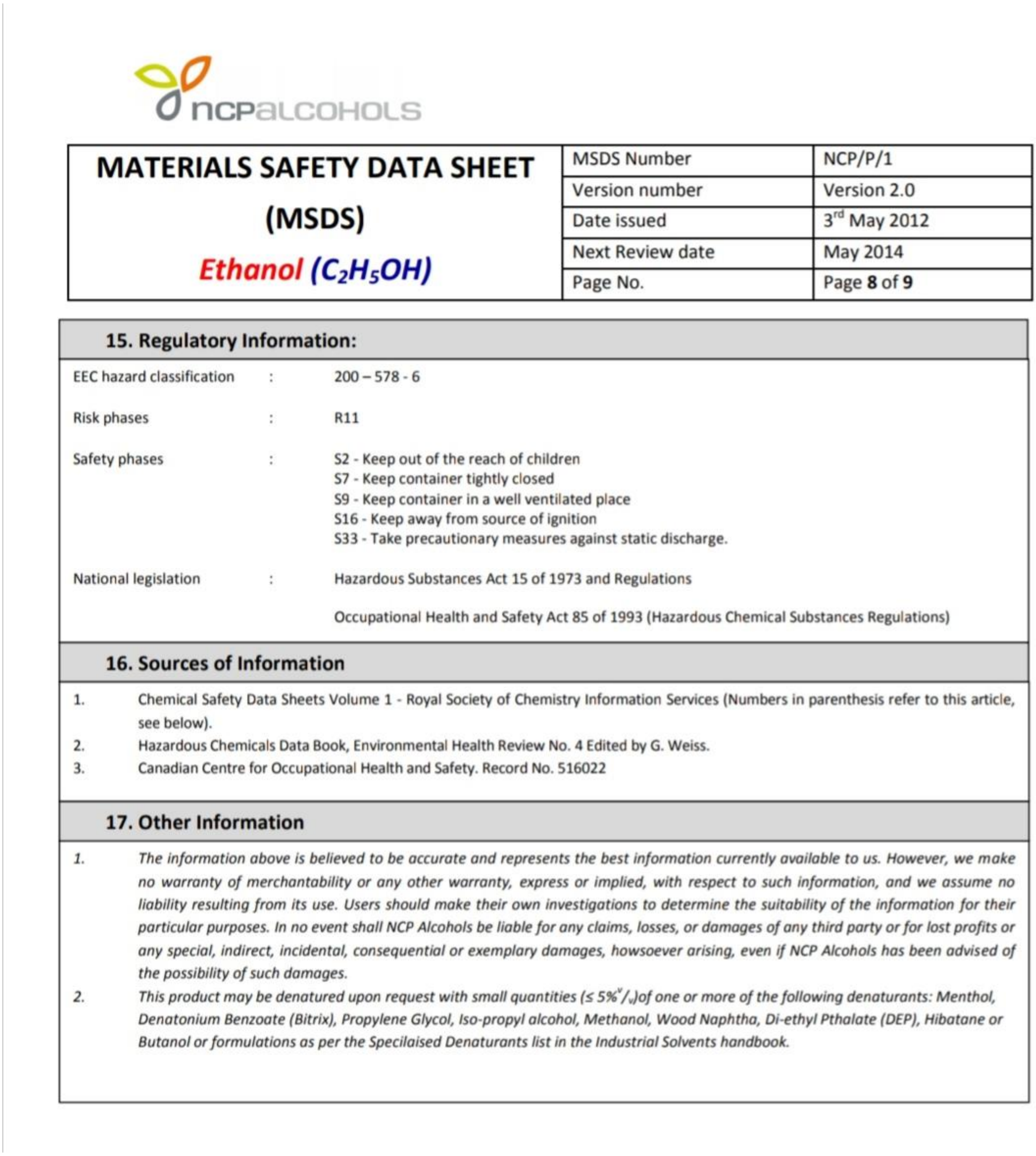
ADNR – class : UN –No.:1170; Class 3, Packaging Group II

UK – description : Not available

UK- emergency action class : Not available

UK – classification : Not available

Tremcard No : 1170



ncPalcohols

# MATERIALS SAFETY DATA SHEET

# (MSDS)

## Ethanol ($C_2H_5OH$)

| | |
|---|---|
| MSDS Number | NCP/P/1 |
| Version number | Version 2.0 |
| Date issued | 3rd May 2012 |
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## 15. Regulatory Information:

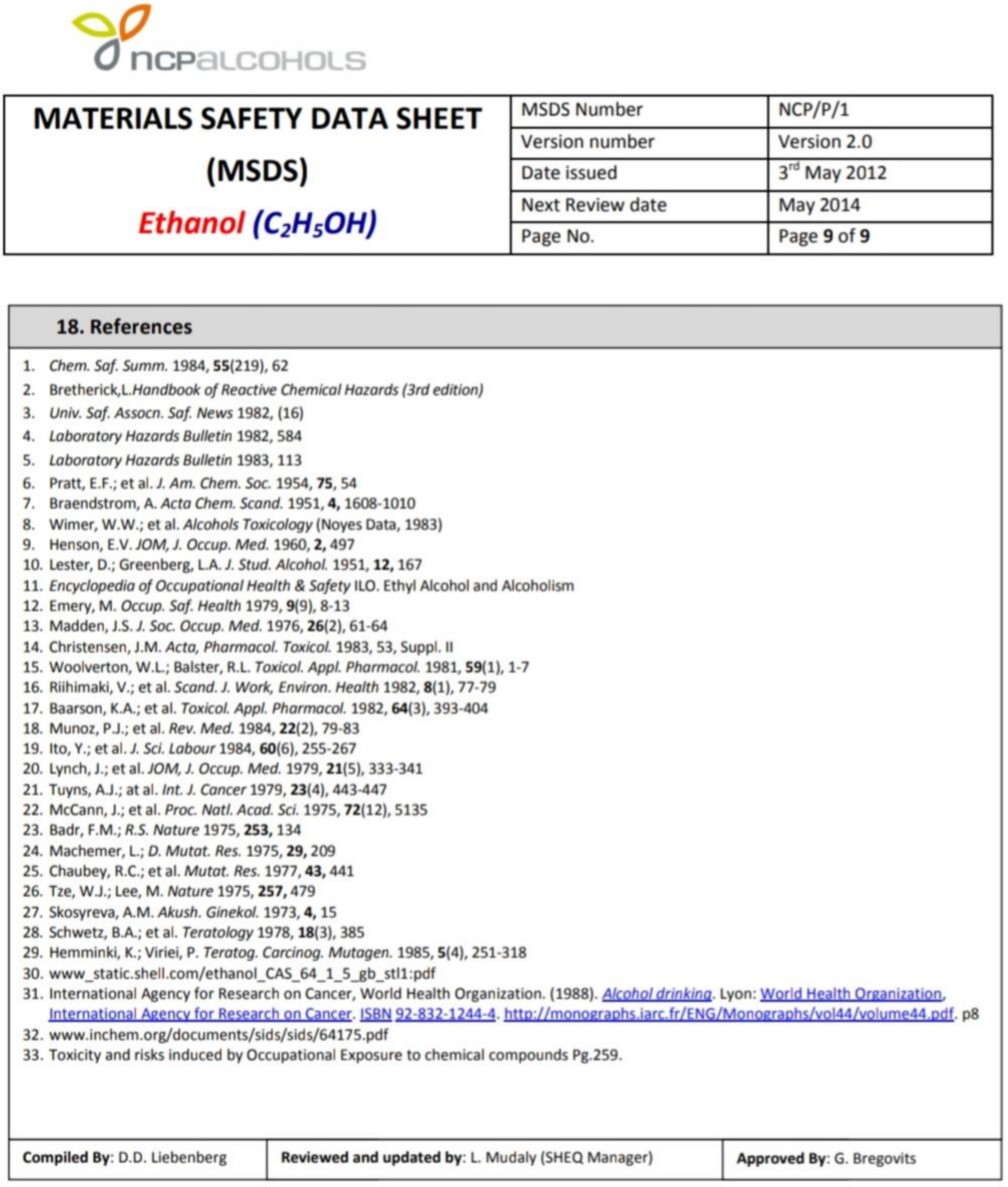
| | | |
|---|---|---|
| EEC hazard classification | : | 200 – 578 - 6 |
| Risk phases | : | R11 |
| Safety phases | : | S2 - Keep out of the reach of children<br>S7 - Keep container tightly closed<br>S9 - Keep container in a well ventilated place<br>S16 - Keep away from source of ignition<br>S33 - Take precautionary measures against static discharge. |
| National legislation | : | Hazardous Substances Act 15 of 1973 and Regulations<br>Occupational Health and Safety Act 85 of 1993 (Hazardous Chemical Substances Regulations) |

## 16. Sources of Information

1. Chemical Safety Data Sheets Volume 1 - Royal Society of Chemistry Information Services (Numbers in parenthesis refer to this article, see below).
2. Hazardous Chemicals Data Book, Environmental Health Review No. 4 Edited by G. Weiss.
3. Canadian Centre for Occupational Health and Safety. Record No. 516022

## 17. Other Information

1. *The information above is believed to be accurate and represents the best information currently available to us. However, we make no warranty of merchantability or any other warranty, express or implied, with respect to such information, and we assume no liability resulting from its use. Users should make their own investigations to determine the suitability of the information for their particular purposes. In no event shall NCP Alcohols be liable for any claims, losses, or damages of any third party or for lost profits or any special, indirect, incidental, consequential or exemplary damages, howsoever arising, even if NCP Alcohols has been advised of the possibility of such damages.*
2. *This product may be denatured upon request with small quantities ($\leq 5\%^{v}/_{v}$)of one or more of the following denaturants: Menthol, Denatonium Benzoate (Bitrix), Propylene Glycol, Iso-propyl alcohol, Methanol, Wood Naphtha, Di-ethyl Pthalate (DEP), Hibatane or Butanol or formulations as per the Specilaised Denaturants list in the Industrial Solvents handbook.*



ncpalcohols

# MATERIALS SAFETY DATA SHEET (MSDS)

## Ethanol ($C_2H_5OH$)

| | |
|---|---|
| MSDS Number | NCP/P/1 |
| Version number | Version 2.0 |
| Date issued | 3rd May 2012 |
| Next Review date | May 2014 |
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## 18. References

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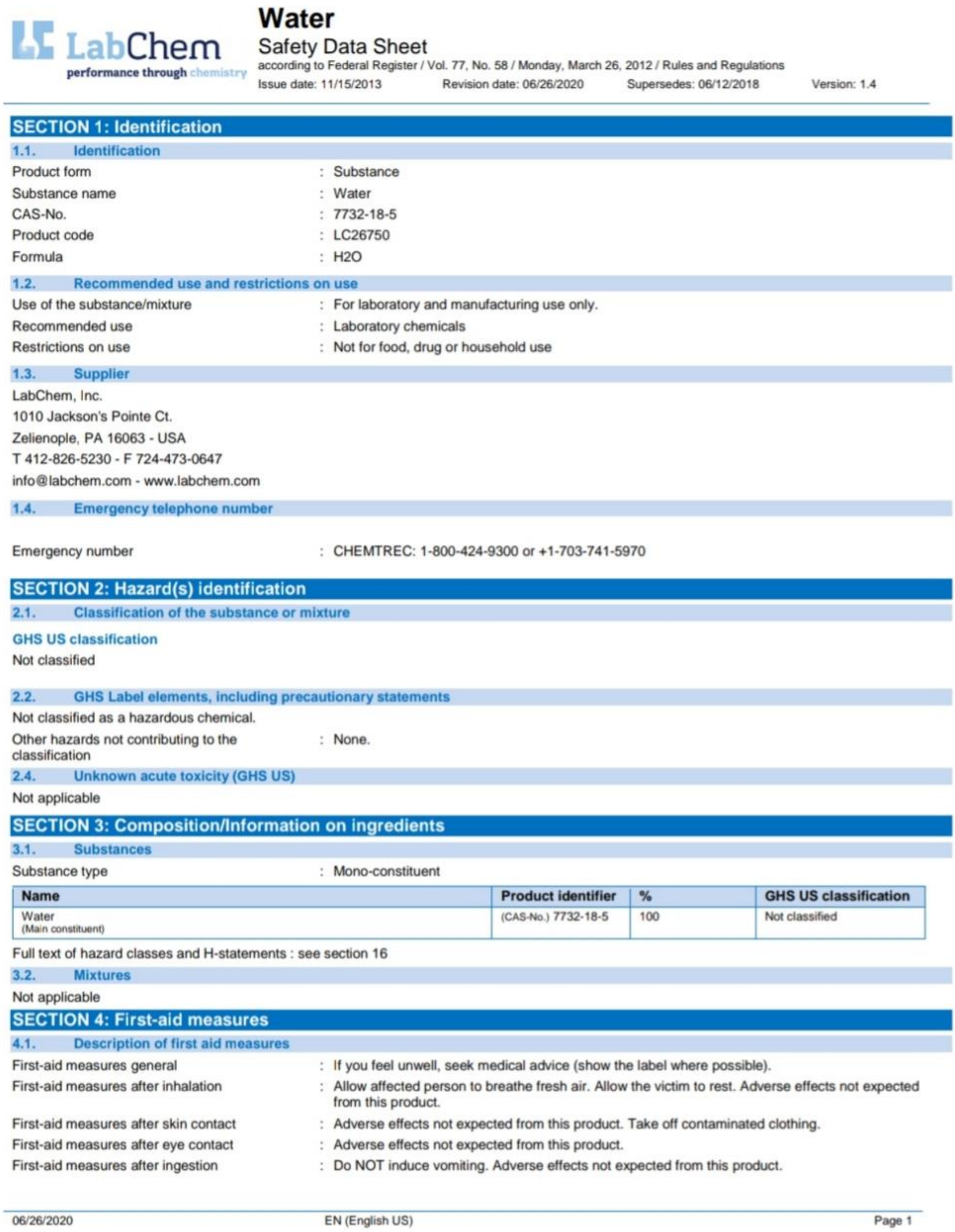
33. Toxicity and risks induced by Occupational Exposure to chemical compounds Pg.259.

| | | |
|---|---|---|
| **Compiled By**: D.D. Liebenberg | **Reviewed and updated by**: L. Mudaly (SHEQ Manager) | **Approved By**: G. Bregovits |



LabChem
performance through chemistry

# Water

## Safety Data Sheet

according to Federal Register / Vol. 77, No. 58 / Monday, March 26, 2012 / Rules and Regulations
Issue date: 11/15/2013 Revision date: 06/26/2020 Supersedes: 06/12/2018 Version: 1.4

## SECTION 1: Identification

### 1.1. Identification

Product form : Substance
Substance name : Water
CAS-No. : 7732-18-5
Product code : LC26750
Formula : $H_2O$

### 1.2. Recommended use and restrictions on use

Use of the substance/mixture : For laboratory and manufacturing use only.
Recommended use : Laboratory chemicals
Restrictions on use : Not for food, drug or household use

### 1.3. Supplier

LabChem, Inc.
1010 Jackson's Pointe Ct.
Zelienople, PA 16063 - USA
T 412-826-5230 - F 724-473-0647
info@labchem.com - www.labchem.com

### 1.4. Emergency telephone number

Emergency number : CHEMTREC: 1-800-424-9300 or +1-703-741-5970

## SECTION 2: Hazard(s) identification

### 2.1. Classification of the substance or mixture

**GHS US classification**

Not classified

### 2.2. GHS Label elements, including precautionary statements

Not classified as a hazardous chemical.

Other hazards not contributing to the classification : None.

### 2.4. Unknown acute toxicity (GHS US)

Not applicable

## SECTION 3: Composition/Information on ingredients

### 3.1. Substances

Substance type : Mono-constituent

| Name | Product identifier | % | GHS US classification |
|---|---|---|---|
| Water<br>(Main constituent) | (CAS-No.) 7732-18-5 | 100 | Not classified |

Full text of hazard classes and H-statements : see section 16

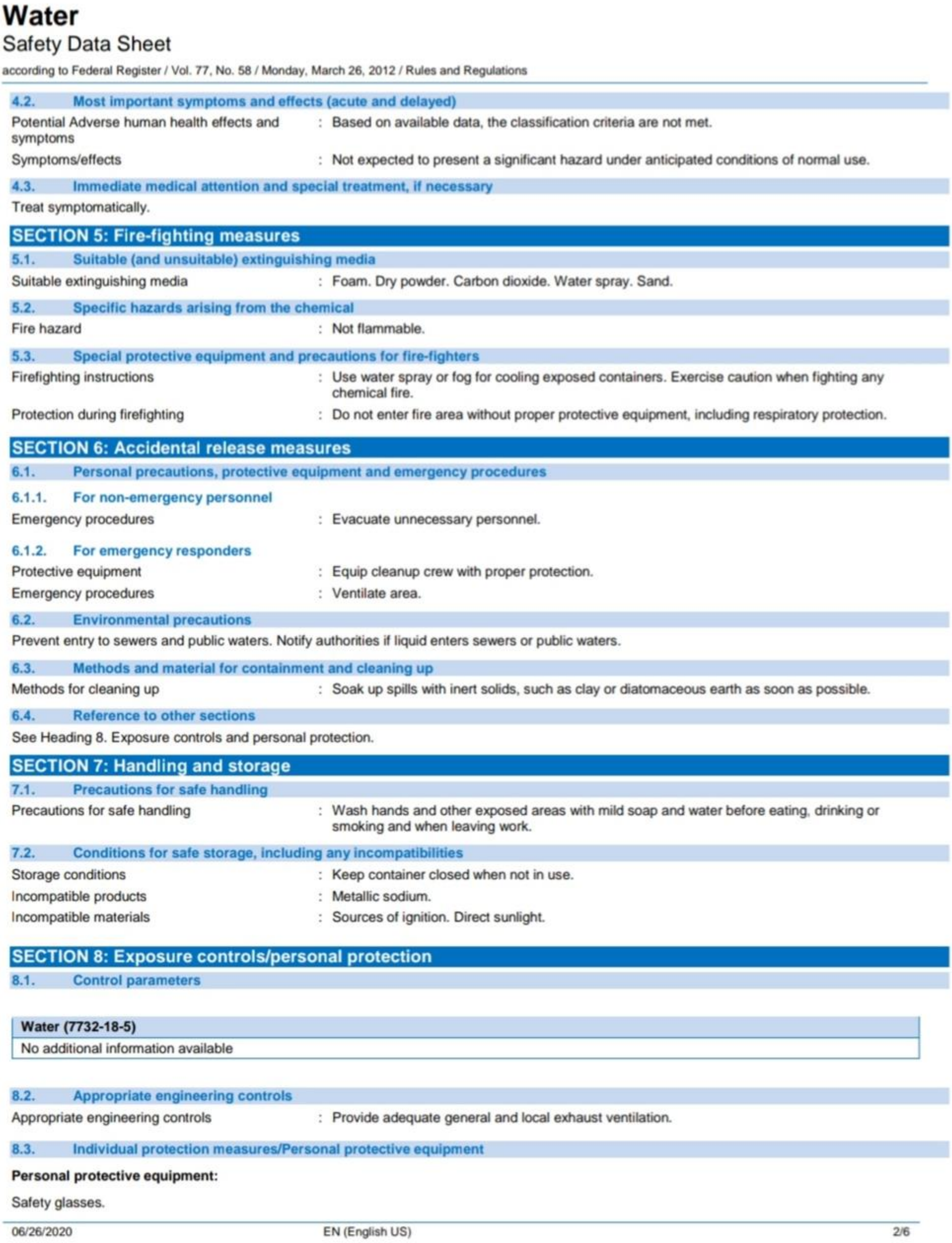
### 3.2. Mixtures

Not applicable

## SECTION 4: First-aid measures

### 4.1. Description of first aid measures

First-aid measures general : If you feel unwell, seek medical advice (show the label where possible).
First-aid measures after inhalation : Allow affected person to breathe fresh air. Allow the victim to rest. Adverse effects not expected from this product.
First-aid measures after skin contact : Adverse effects not expected from this product. Take off contaminated clothing.
First-aid measures after eye contact : Adverse effects not expected from this product.
First-aid measures after ingestion : Do NOT induce vomiting. Adverse effects not expected from this product.



**Water**

Safety Data Sheet

according to Federal Register / Vol. 77, No. 58 / Monday, March 26, 2012 / Rules and Regulations

### 4.2. Most important symptoms and effects (acute and delayed)

| | |
|---|---|
| Potential Adverse human health effects and symptoms | : Based on available data, the classification criteria are not met. |
| Symptoms/effects | : Not expected to present a significant hazard under anticipated conditions of normal use. |

### 4.3. Immediate medical attention and special treatment, if necessary

Treat symptomatically.

## SECTION 5: Fire-fighting measures

### 5.1. Suitable (and unsuitable) extinguishing media

| | |
|---|---|
| Suitable extinguishing media | : Foam. Dry powder. Carbon dioxide. Water spray. Sand. |

### 5.2. Specific hazards arising from the chemical

| | |
|---|---|
| Fire hazard | : Not flammable. |

### 5.3. Special protective equipment and precautions for fire-fighters

| | |
|---|---|
| Firefighting instructions | : Use water spray or fog for cooling exposed containers. Exercise caution when fighting any chemical fire. |
| Protection during firefighting | : Do not enter fire area without proper protective equipment, including respiratory protection. |

## SECTION 6: Accidental release measures

### 6.1. Personal precautions, protective equipment and emergency procedures

#### 6.1.1. For non-emergency personnel

| | |
|---|---|
| Emergency procedures | : Evacuate unnecessary personnel. |

#### 6.1.2. For emergency responders

| | |
|---|---|
| Protective equipment | : Equip cleanup crew with proper protection. |
| Emergency procedures | : Ventilate area. |

### 6.2. Environmental precautions

Prevent entry to sewers and public waters. Notify authorities if liquid enters sewers or public waters.

### 6.3. Methods and material for containment and cleaning up

| | |
|---|---|
| Methods for cleaning up | : Soak up spills with inert solids, such as clay or diatomaceous earth as soon as possible. |

### 6.4. Reference to other sections

See Heading 8. Exposure controls and personal protection.

## SECTION 7: Handling and storage

### 7.1. Precautions for safe handling

| | |
|---|---|
| Precautions for safe handling | : Wash hands and other exposed areas with mild soap and water before eating, drinking or smoking and when leaving work. |

### 7.2. Conditions for safe storage, including any incompatibilities

| | |
|---|---|
| Storage conditions | : Keep container closed when not in use. |
| Incompatible products | : Metallic sodium. |
| Incompatible materials | : Sources of ignition. Direct sunlight. |

## SECTION 8: Exposure controls/personal protection

### 8.1. Control parameters

| **Water (7732-18-5)** |
|---|
| No additional information available |

### 8.2. Appropriate engineering controls

| | |
|---|---|
| Appropriate engineering controls | : Provide adequate general and local exhaust ventilation. |

### 8.3. Individual protection measures/Personal protective equipment

**Personal protective equipment:**

Safety glasses.

06/26/2020 EN (English US) 2/6

**Eye protection:**

Chemical goggles or safety glasses

**Respiratory protection:**

None necessary.

**Personal protective equipment symbol(s):**

**Other information:**

Do not eat, drink or smoke during use.

## SECTION 9: Physical and chemical properties

### 9.1. Information on basic physical and chemical properties

| | |
|---|---|
| Physical state | Liquid |
| Color | Colorless |
| Odor | None. |
| Odor threshold | No data available |
| pH | 7 |
| Melting point | 0 °C |
| Freezing point | No data available |
| Boiling point | 100 °C |
| Critical temperature | 374.1 °C |
| Critical pressure | 218.3 atm |
| Flash point | No data available |
| Relative evaporation rate (butyl acetate=1) | No data available |
| Flammability (solid, gas) | Non flammable. |
| Vapor pressure | 17.535 mm Hg |
| Vapor pressure at 50 °C | 92.51 mm Hg |
| Relative vapor density at 20 °C | No data available |
| Relative density | 1 |
| Specific gravity / density | 0.99823 g/ml |
| Molecular mass | 18 g/mol |
| Solubility | Soluble in acetic acid. Soluble in acetone. Soluble in ammonia. Soluble in ammonium chloride. Soluble in ethanol. Soluble in glycerol. Soluble in hydrochloric acid. Soluble in methanol. Soluble in nitric acid. Soluble in sulfuric acid. Soluble in sodium hydroxide solution. Soluble in propylene glycol. |
| Log Pow | No data available |
| Auto-ignition temperature | No data available |
| Decomposition temperature | No data available |
| Viscosity, kinematic | 1.004 $mm^2/s$ |
| Viscosity, dynamic | 1.002 cP |
| Explosion limits | No data available |
| Explosive properties | Not applicable. |
| Oxidizing properties | None. |

### 9.2. Other information

VOC content : 0 %

## SECTION 10: Stability and reactivity

### 10.1. Reactivity

No additional information available

### 10.2. Chemical stability

Stable under normal conditions.

### 10.3. Possibility of hazardous reactions

Not established.

### 10.4. Conditions to avoid

Extremely high or low temperatures.

### 10.5. Incompatible materials

Metallic sodium.

### 10.6. Hazardous decomposition products

Hydrogen. oxygen.

## SECTION 11: Toxicological information

### 11.1. Information on toxicological effects

Acute toxicity (oral) : Not classified
Acute toxicity (dermal) : Not classified
Acute toxicity (inhalation) : Not classified

| **Water (7732-18-5)** | |
|---|---|
| LD50 oral rat | ≥ 90000 mg/kg |
| ATE US (oral) | 90000 mg/kg body weight |

Skin corrosion/irritation : Not classified
pH: 7
Serious eye damage/irritation : Not classified
pH: 7
Respiratory or skin sensitization : Not classified
Germ cell mutagenicity : Not classified
Carcinogenicity : Not classified (Based on available data, the classification criteria are not met)
Reproductive toxicity : Not classified
STOT-single exposure : Not classified
STOT-repeated exposure : Not classified
Aspiration hazard : Not classified
Viscosity, kinematic : 1.004 mm²/s
Likely routes of exposure : Skin and eye contact.
Potential Adverse human health effects and symptoms : Based on available data, the classification criteria are not met.
Symptoms/effects : Not expected to present a significant hazard under anticipated conditions of normal use.

## SECTION 12: Ecological information

### 12.1. Toxicity

No additional information available

### 12.2. Persistence and degradability

| **Water (7732-18-5)** | |
|---|---|
| Persistence and degradability | Not established. |

### 12.3. Bioaccumulative potential

| **Water (7732-18-5)** | |
|---|---|
| Bioaccumulative potential | Not established. |

### 12.4. Mobility in soil

No additional information available

### 12.5. Other adverse effects

Other information : No other effects known.

## SECTION 13: Disposal considerations

### 13.1. Disposal methods

Waste disposal recommendations : Dispose in a safe manner in accordance with local/national regulations.

## SECTION 14: Transport information

### Department of Transportation (DOT)

In accordance with DOT

Not regulated

### Transport by sea

Not regulated

### Air transport

Not regulated

## SECTION 15: Regulatory information

### 15.1. US Federal regulations

| **Water (7732-18-5)** |
|---|
| Listed on the United States TSCA (Toxic Substances Control Act) inventory |

All components of this product are listed, or excluded from listing, on the United States Environmental Protection Agency Toxic Substances Control Act (TSCA) inventory

### 15.2. International regulations

### CANADA

| **Water (7732-18-5)** |
|---|
| Listed on the Canadian DSL (Domestic Substances List) |

### EU-Regulations

No additional information available

### National regulations

No additional information available

### 15.3. US State regulations

California Proposition 65 - This product does not contain any substances known to the state of California to cause cancer, developmental and/or reproductive harm

## SECTION 16: Other information

according to Federal Register / Vol. 77, No. 58 / Monday, March 26, 2012 / Rules and Regulations

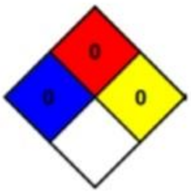
Revision date : 06/26/2020

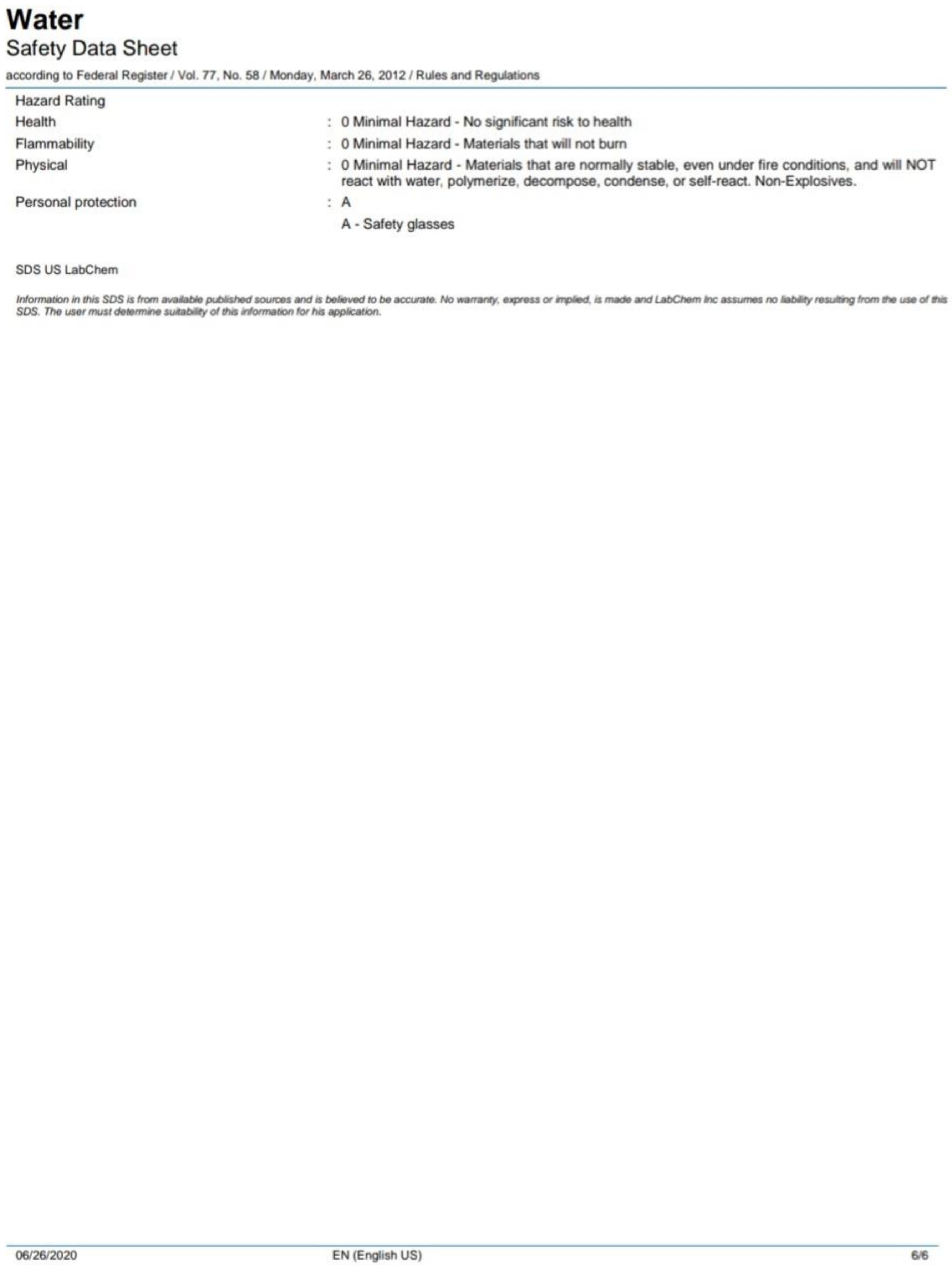
Other information : None.

NFPA health hazard : 0 - Materials that, under emergency conditions, would offer no hazard beyond that of ordinary combustible materials.

NFPA fire hazard : 0 - Materials that will not burn under typical fire conditions, including intrinsically noncombustible materials such as concrete, stone, and sand.

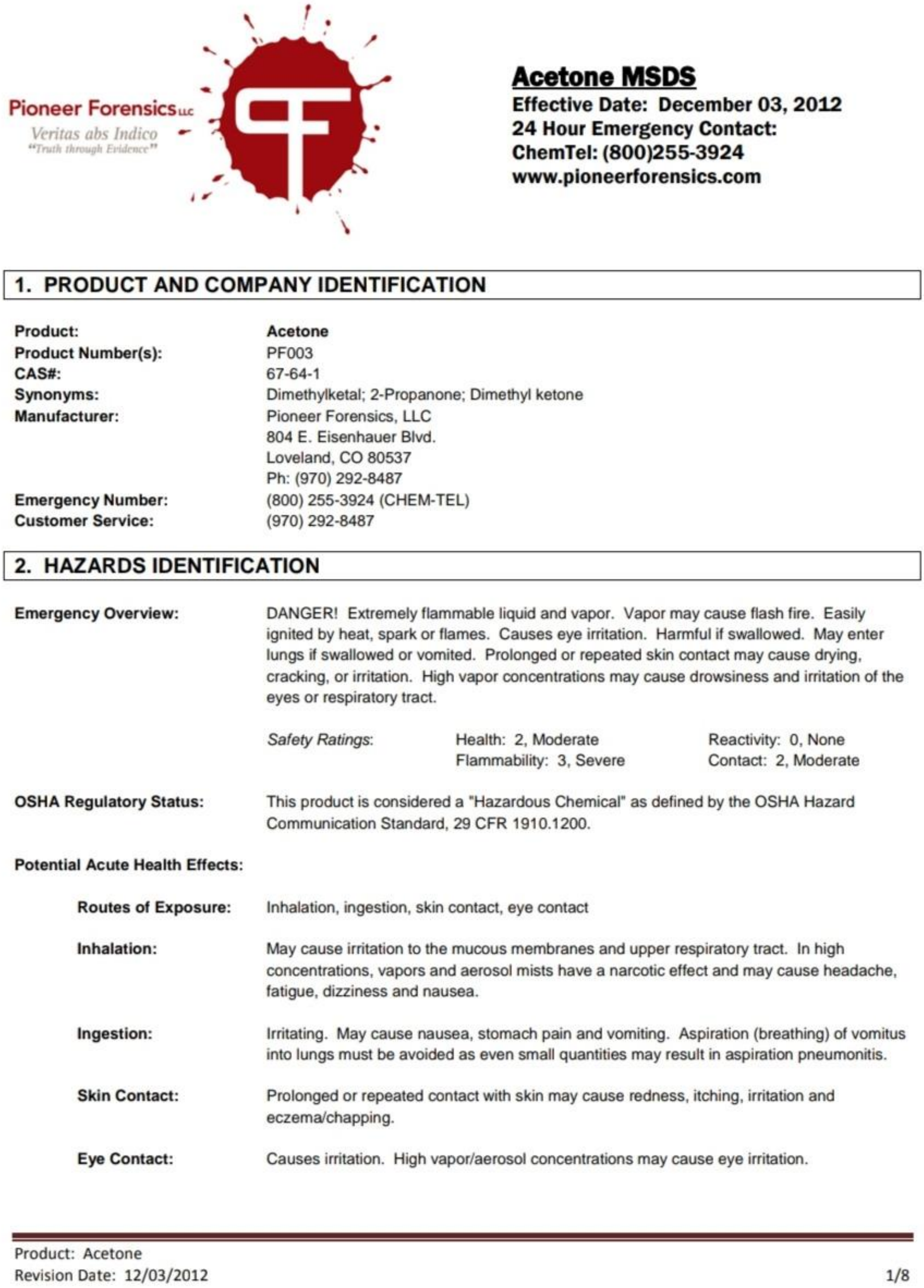
NFPA reactivity : 0 - Material that in themselves are normally stable, even under fire conditions.





| | |
|---|---|
| Hazard Rating | |
| Health | : 0 Minimal Hazard - No significant risk to health |
| Flammability | : 0 Minimal Hazard - Materials that will not burn |
| Physical | : 0 Minimal Hazard - Materials that are normally stable, even under fire conditions, and will NOT react with water, polymerize, decompose, condense, or self-react. Non-Explosives. |
| Personal protection | : A<br>A - Safety glasses |

SDS US LabChem



Pioneer Forensics LLC
Veritas abs Indico
"Truth through Evidence"

# Acetone MSDS

**Effective Date: December 03, 2012**
**24 Hour Emergency Contact:**
**ChemTel: (800)255-3924**
**www.pioneerforensics.com**

## 1. PRODUCT AND COMPANY IDENTIFICATION

| | |
|---|---|
| **Product:** | **Acetone** |
| **Product Number(s):** | PF003 |
| **CAS#:** | 67-64-1 |
| **Synonyms:** | Dimethylketal; 2-Propanone; Dimethyl ketone |
| **Manufacturer:** | Pioneer Forensics, LLC<br>804 E. Eisenhauer Blvd.<br>Loveland, CO 80537<br>Ph: (970) 292-8487 |
| **Emergency Number:** | (800) 255-3924 (CHEM-TEL) |
| **Customer Service:** | (970) 292-8487 |

## 2. HAZARDS IDENTIFICATION

**Emergency Overview:** DANGER! Extremely flammable liquid and vapor. Vapor may cause flash fire. Easily ignited by heat, spark or flames. Causes eye irritation. Harmful if swallowed. May enter lungs if swallowed or vomited. Prolonged or repeated skin contact may cause drying, cracking, or irritation. High vapor concentrations may cause drowsiness and irritation of the eyes or respiratory tract.

| *Safety Ratings*: | Health: 2, Moderate | Reactivity: 0, None |
|---|---|---|
| | Flammability: 3, Severe | Contact: 2, Moderate |

**OSHA Regulatory Status:** This product is considered a "Hazardous Chemical" as defined by the OSHA Hazard Communication Standard, 29 CFR 1910.1200.

**Potential Acute Health Effects:**

**Routes of Exposure:** Inhalation, ingestion, skin contact, eye contact

**Inhalation:** May cause irritation to the mucous membranes and upper respiratory tract. In high concentrations, vapors and aerosol mists have a narcotic effect and may cause headache, fatigue, dizziness and nausea.

**Ingestion:** Irritating. May cause nausea, stomach pain and vomiting. Aspiration (breathing) of vomitus into lungs must be avoided as even small quantities may result in aspiration pneumonitis.

**Skin Contact:** Prolonged or repeated contact with skin may cause redness, itching, irritation and eczema/chapping.

**Eye Contact:** Causes irritation. High vapor/aerosol concentrations may cause eye irritation.

**Target Organs:** Skin, respiratory system, eyes, central nervous system

**Chronic Health Effects:** Frequent or prolonged contact may defat and dry the skin, leading to discomfort and dermatitis.

**Aggravation of Medical Conditions:** Repeated or prolonged exposure to the substance can produce target organs damage. Persons with pre-existing skin disorders or eye problems may be more susceptible to the effects of the substance.

**Potential Environmental Effects:** Not classified as environmentally hazardous, however, this does not exclude the possibility that large or frequent spills can have harmful or damaging effects on the environment.

## 3. COMPOSITION AND INFORMATION ON INGREDIENTS

| Components | CAS# | Chemical Formula | Formula Weight | Hazardous | % by Weight |
|---|---|---|---|---|---|
| Acetone | 67-64-1 | $C_3H_6O$ | 58.08 | Yes | >99.5 |

## 4. FIRST AID MEASURES

**First Aid Procedures:**

**Inhalation:** Remove to fresh air. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Get medical attention if symptoms persist.

**Ingestion:** Do not induce vomiting. If vomiting occurs, keep head low so that vomit does not enter lungs. Never give anything by mouth to an unconscious person. GET MEDICAL ATTENTION IMMEDIATELY.

**Skin Contact:** Wash affected area with soap and water. Remove contaminated clothing and shoes. Wash clothing before reuse. Get medical attention if symptoms occur.

**Eye Contact:** Check for and remove contact lenses. Immediately flush eyes with gentle but large stream of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention.

**General Advice:** In the case of accident or if you feel unwell, seek medical advice immediately (show the label where possible). Ensure that medical personnel are aware of the material(s) involved and take precautions to protect themselves. Show this safety data sheet to the doctor in attendance.

**Notes to Physician:** Treat symptomatically. Keep victim under observation.

## 5. FIRE FIGHTING MEASURES

**NFPA Ratings:** Health: 2 Flammability: 3 Reactivity: 0

**Flammable Properties:** HIGHLY FLAMMABLE! Vapors may cause a flash fire or ignite explosively. Vapors may travel considerable distance to a source of ignition and flash back. Heat may cause sealed containers to explode.

**Flash Point:** -20° C (-4° F) Closed Cup

**Auto-ignition Temp:** 465° C (869° F)

| | |
|---|---|
| **Flammable Limits in Air (% by volume):** | Lower Explosion Limit: 2.6%<br>Upper Explosion Limit: 12.8% |
| **Suitable Extinguishing Media:** | Water spray, dry powder, alcohol resistant foam, carbon dioxide |
| **Unsuitable Extinguishing Media:** | Do not use a solid (straight) water stream as it may scatter and spread fire. |
| **Hazardous Combustion Products:** | Carbon monoxide, carbon dioxide |
| **Specific Hazards:** | Can be ignited easily by heat, sparks, or flame and burns vigorously. Above flash point, vapor-air mixtures are explosive within flammable limits noted above. Sealed containers may explode when heated or involved in fire. Vapor may accumulate in container headspace resulting in flammability hazard. Material is sensitive to static discharge. |
| **Special Protective Equipment For Firefighters:** | As in any fire, wear MSHA/NIOSH approved (or equivalent) self-contained positive pressure or pressure-demand breathing apparatus and full protective gear. |
| **Specific Methods:** | Use water spray to cool unopened containers. Cool containers exposed to flames with flooding quantities of water until well after the fire is out. Withdraw immediately in case of rising sound from venting safety device or any discoloration of tanks due to fire. Move containers from fire area if you can do so without risk. Some of these materials, if spilled, may evaporate leaving a flammable residue. In the event of fire and/or explosion do not breathe fumes. |

## 6. ACCIDENTAL RELEASE MEASURES

| | |
|---|---|
| **Personal Precautions:** | Ventilate area of leak or spill. Isolate hazard area and keep unnecessary and unprotected personnel away from the area of the leak or spill. Keep upwind. Keep out of low areas. Wear appropriate personal protective equipment as specified in the Exposure Control and Personal Protection Section 8. Avoid contact with eyes, skin, and clothing. Pay attention to flashback. Take precautionary measures against static discharges. |
| **Environmental Precautions:** | Prevent further leakage or spillage if safe to do so. Do not contaminate water. Avoid discharge into drains, water courses or onto the ground. In case of large spill, dike if needed. |
| **Methods for Containment:** | Remove all sources of ignition. Stop the flow of material, if this is without risk. Prevent entry into waterways, sewer, basements or confined areas. Dike the spilled material, where this is possible. |
| **Methods for Cleaning Up:** | Use spark-proof tools and explosion-proof equipment. All equipment used when handling the product must be grounded. Absorb spill with an inert material (e.g. vermiculite, dry sand, earth, cloth, fleece), and place in a suitable non-combustible container for reclamation or disposal. Do not use combustible materials, such as sawdust. Clean contaminated surface thoroughly. Never return spills in original containers for re-use. Clean up in accordance with all applicable regulations. |

## 7. HANDLING AND STORAGE

| | |
|---|---|
| **Handling:** | Do not handle or open near flame, sources of heat, or sources of ignition. Protect material from direct sunlight. Wear personal protective equipment (see section 8). Use only in well-ventilated areas. Provide sufficient air exchange and/or exhaust in work rooms. Avoid contact with skin, eyes and clothing. Do not breathe vapors or spray mist. Do not ingest. When using, do not eat, smoke, or drink. Take precautionary measures against static discharge. Keep away from incompatible materials. Handle in accordance with good |

industrial hygiene and safety practice. Wash thoroughly after handling. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquids). Observe all warnings and precautions listed for the product

**Storage:** Store in a cool, dry, ventilated area. Store away from flame, sources of ignition, heat, and incompatible materials. Store in original container. Keep containers tightly closed and upright. Keep away from food, drink and animal feedingstuffs. Keep out of the reach of children. Ground container and transfer equipment to eliminate static electric sparks. Comply with all national, state, and local codes pertaining to the storage, handling, dispensing, and disposal of flammable liquids.

## 8. EXPOSURE CONTROL AND PERSONAL PROTECTION

| | | | |
|---|---|---|---|
| **Exposure Limits:** | ACGIH: | TWA: | 500 ppm |
| | | STEL: | 750 ppm |
| | | BEL: | 50mg/L |
| | OSHA: | PEL: | 1000 ppm<br>2400 mg/m$^3$ |

**Engineering Controls:** Ensure adequate ventilation. Ventilation rates should be matched to conditions. If applicable, use process enclosures, local exhaust ventilation, or other engineering controls to maintain airborne levels below recommended exposure limits. If exposure limits have not been established, maintain airborne levels to an acceptable level. Explosion proof exhaust ventilation should be used.

**Personal Protective Equipment:**

**Eye/Face Protection:** Wear safety glasses with side shields or goggles and a face shield.

**Skin Protection:** Wear appropriate chemical resistant clothing (with long sleeves) and appropriate chemical resistant gloves.

**Respiratory Protection:** If engineering controls do not maintain airborne concentrations below recommended exposure limits (where applicable) or to an acceptable level (in countries where exposure limits have not been established), an approved respirator must be worn. Respirator type: Chemical respirator with organic vapor cartridge. Use a positive-pressure air-supplied respirator if there is any potential for an uncontrolled release, exposure levels are not known, or any other circumstances where air-purifying respirators may not provide adequate protection.

**General Hygiene Considerations:** Avoid contact with skin, eyes and clothing. When using, do not eat, drink or smoke. Always observe good personal hygiene measures, such as washing after handling the material and before eating, drinking, and/or smoking. Routinely wash work clothing and protective equipment to remove contaminants. Provide eyewash station and safety shower.

## 9. PHYSICAL AND CHEMICAL PROPERTIES

| | |
|---|---|
| **Physical State:** | Liquid |
| **Appearance:** | Transparent |
| **Color:** | Colorless |
| **Odor:** | Sweet, mint-like |
| **Molecular Formula:** | $C_3H_6O$ |
| **Molecular Weight:** | 58.08 |
| **pH:** | No information found |
| **Specific Gravity:** | 0.79 |
| **Freezing/Melting Point:** | -94.7 °C (-139 °F) |

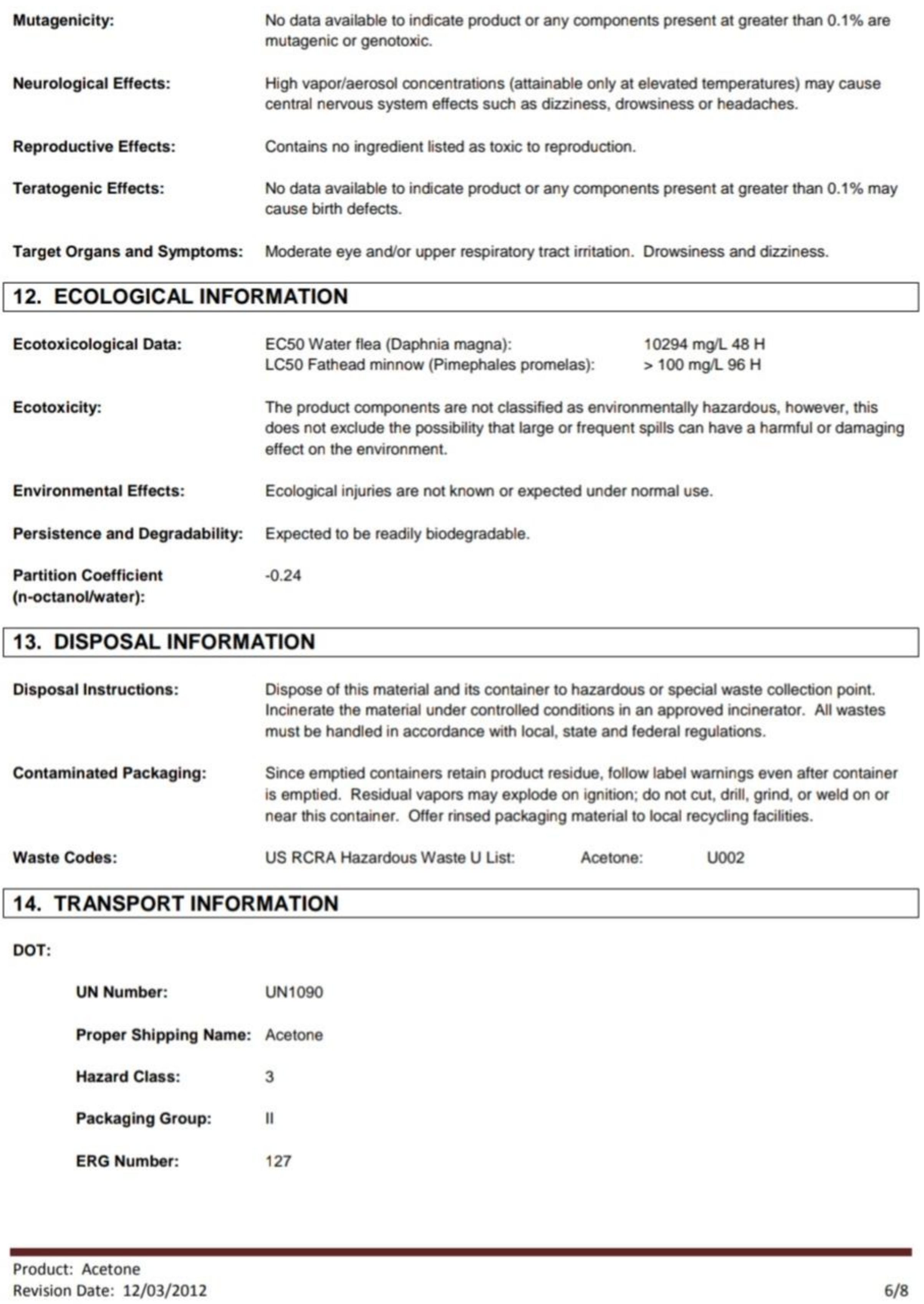
| | |
|---|---|
| **Boiling Point:** | 56.1 °C (132.8 °F) |
| **Flash Point:** | -20° C (-4° F) Closed Cup |
| **Auto Ignition Temperature:** | 465° C (869° F) |
| **Flammable Limits in Air (% by Volume):** | |
| **Upper:** | 12.8% |
| **Lower:** | 2.6% |
| **Solubility:** | Miscible with water |
| **Vapor Pressure:** | 30.93 kPa at 25°C |
| **Vapor Density:** | 2 |
| **Odor threshold (ppm):** | 62-140 ppm |
| **Evaporation Rate:** | 5.6 BuAc |
| **Partition Coefficient (n-octanol/water):** | -0.24 |

## 10. STABILITY AND REACTIVITY

| | |
|---|---|
| **Stability:** | Stable under normal conditions. |
| **Conditions to Avoid:** | Heat, flames, sparks, ignition sources, incompatibles |
| **Incompatible Materials:** | Oxidizing agents, acids, alkalis, peroxides |
| **Hazardous Decomposition Products:** | Carbon dioxide and carbon monoxide may form when heated to decomposition. |
| **Possibility of Hazardous Reactions:** | Can react vigorously, violently or explosively with incompatible materials listed above. |
| **Hazardous Polymerization:** | Will not occur. |

## 11. TOXICOLOGICAL INFORMATION

| | | |
|---|---|---|
| **Toxicological Data:** | Oral Rat LD50: | 5800 mg/kg |
| | Skin Rabbit LD50: | 20000 mg/kg |
| | Inhalation Rat LC50: | 76 mg/L 4H |
| **Acute Effects:** | Harmful if swallowed. May enter lungs if swallowed or vomited. | |
| **Local Effects:** | Causes eye irritation. Prolonged or repeated skin contact may cause drying, cracking, or irritation. High vapor concentrations may cause drowsiness and irritation of the eyes or respiratory tract. | |
| **Sensitization:** | Not a skin sensitizer. | |
| **Chronic Effects:** | Frequent or prolonged contact may defat and dry the skin, leading to discomfort and dermatitis. | |
| **Carcinogenic Effects:** | This product is not considered to be a carcinogen by IARC, ACGIH, NTP, or OSHA. | |
| | ACGIH: | A4 – Not classifiable as a human carcinogen |
| **Skin Corrosion/Irritation:** | Defatting, drying, and cracking of the skin. | |
| **Epidemiology:** | No epidemiological data is available for this product. | |



| | |
|---|---|
| **Mutagenicity:** | No data available to indicate product or any components present at greater than 0.1% are mutagenic or genotoxic. |
| **Neurological Effects:** | High vapor/aerosol concentrations (attainable only at elevated temperatures) may cause central nervous system effects such as dizziness, drowsiness or headaches. |
| **Reproductive Effects:** | Contains no ingredient listed as toxic to reproduction. |
| **Teratogenic Effects:** | No data available to indicate product or any components present at greater than 0.1% may cause birth defects. |
| **Target Organs and Symptoms:** | Moderate eye and/or upper respiratory tract irritation. Drowsiness and dizziness. |

## 12. ECOLOGICAL INFORMATION

| | | |
|---|---|---|
| **Ecotoxicological Data:** | EC50 Water flea (Daphnia magna): | 10294 mg/L 48 H |
| | LC50 Fathead minnow (Pimephales promelas): | > 100 mg/L 96 H |
| **Ecotoxicity:** | The product components are not classified as environmentally hazardous, however, this does not exclude the possibility that large or frequent spills can have a harmful or damaging effect on the environment. | |
| **Environmental Effects:** | Ecological injuries are not known or expected under normal use. | |
| **Persistence and Degradability:** | Expected to be readily biodegradable. | |
| **Partition Coefficient (n-octanol/water):** | -0.24 | |

## 13. DISPOSAL INFORMATION

| | |
|---|---|
| **Disposal Instructions:** | Dispose of this material and its container to hazardous or special waste collection point. Incinerate the material under controlled conditions in an approved incinerator. All wastes must be handled in accordance with local, state and federal regulations. |
| **Contaminated Packaging:** | Since emptied containers retain product residue, follow label warnings even after container is emptied. Residual vapors may explode on ignition; do not cut, drill, grind, or weld on or near this container. Offer rinsed packaging material to local recycling facilities. |
| **Waste Codes:** | US RCRA Hazardous Waste U List: Acetone: U002 |

## 14. TRANSPORT INFORMATION

**DOT:**

| | |
|---|---|
| **UN Number:** | UN1090 |
| **Proper Shipping Name:** | Acetone |
| **Hazard Class:** | 3 |
| **Packaging Group:** | II |
| **ERG Number:** | 127 |

## 15. REGULATORY INFORMATION

**U.S. Federal Regulations:**

**OSHA:** This product is considered a "Hazardous Chemical" as defined by the OSHA Hazard Communication Standard, 29 CFR 1910.1200.

**TSCA Inventory:** Acetone

**U.S. EPCRA (SARA Title III):**

**Sections 311/312:**

| Hazard Categories | List (Yes/No) |
|---|---|
| Section 311 – Hazardous Chemical | Yes |
| Immediate Hazard | Yes |
| Delayed Hazard | No |
| Fire Hazard | Yes |
| Pressure Hazard | No |
| Reactivity Hazard | No |

**CERCLA:** Acetone: 5000 lbs

**International Inventories:**

| Country(s) or Region | Inventory Name | On Inventory (Yes/No)* |
|---|---|---|
| Australia | Australian Inventory of Chemical Substances (AICS) | Yes |
| Canada | Domestic Substances List (DSL) | Yes |
| Canada | Non-Domestic Substances List (NDSL) | No |
| China | Inventory of Existing Chemical Substances in China (IECSC) | Yes |
| Europe | European Inventory of Existing Commercial Chemical Substances (EINECS) | Yes |
| Europe | European List of Notified Chemical Substances (ELINCS) | No |
| Japan | Inventory of Existing and New Chemical Substances (ENCS) | Yes |
| Korea | Existing Chemicals List (ECL) | Yes |
| New Zealand | New Zealand Inventory | Yes |
| Philippines | Philippine Inventory of Chemicals and Chemical Substances (PICCS) | Yes |

*A "Yes" indicates that the listed component(s) of this product comply with the inventory requirements administered by the governing country(s)

## 16. OTHER INFORMATION

**Product Use:** Laboratory and/or field reagent

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