UNIVERSITY OF
KWAZULU-NATAL

EVALUATION OF A VIBRATING PLATE EXTRACTION COLUMN

ENCH4LA

LABORATORY PROJECT

PROJECT 23

2021

Name: Tejal Misra

Student Number: 218002638

Partner's name: Diya Singh

Partner's student number: 218016567

Supervisor/s name: Prof. Naidoo, Dr. K. Moodley, and Dr. M. Williams-Wynn

Statement of Authorship

I, Tejal Misra, declare that:

- (i) The information reported in this report, except where otherwise indicated, is my original work.
- (ii) This report has not been submitted for any degree or examination at any other university.
- (iii) This report does not contain other persons' data, pictures, graphs or other information, unless specifically acknowledged as being sourced from other persons.
- (iv) This report does not contain other persons' writing, unless specifically acknowledged as being sourced from other researchers. Where other written sources have been quoted, then:
  - a. Their words have been re-written but the general information attributed to them has been referenced;
  - b. Where their exact words have been used, their writing has been placed inside quotation marks, and referenced.
- (v) This report does not contain text, graphics or tables copied and pasted from the internet, unless specifically acknowledged, and the source being detailed in the thesis and in the references sections.

Date: 8th June 2021

Student's Signature: TMisra

✓

Abstract

Rare earth elements, such as neodymium, dysprosium, yttrium, are fundamental requisites of the technology industry, utilized in cellphones and plasma screens. However, waste electronic equipment, consisting of radioactive rare earth metals, has been proscribed from being deposited in South African landfill sites effective August 2021. Thus, it is prudent and environmentally beneficial to investigate effective methods of recycling waste electronics for the extraction of rare earth elements for repurpose. Extraction and recovery of rare earth metals is a focus study in the Thermodynamics Research Unit, funded by the Council for Scientific and Industrial Research (CSIR) and the Department of Science and Innovation (DSI). Investigations of metal separation and extraction via liquid-liquid extraction are ongoing. Liquid-liquid extraction is widely used in the chemical and petroleum industries due to the advantages that it offers over conventional distillation. Azeotropic mixtures and close-boiling point components can be easily separable via liquid-liquid extraction. The vibrating plate extraction column is a mechanically assisted agitation column in which operation can occur at high frequencies, low amplitudes, and high flow rates. This research projects focuses on the re-commissioning of a vibrating plate extraction column, and evaluating the column performance using a ternary system of ethanol (solute)-cyclohexane (carrier)-water (solvent). System selection was based on the affordability and availability of the chemicals. The evaluation of the column performance was necessary so that the extraction column can be used in future experiments for rare earth metal extraction in the Thermodynamics Research Unit. The column operated under batch and semi-batch modes to assess the effectiveness and efficiency of the separation. Furthermore, the frequency and solvent-to-feed ratio parameters were varied for the batch and semi-batch modes, respectively. It was observed that a higher vibrational frequency led to a subsequent increase in the percentage of ethanol extracted from 60.47% to 64.27% due to a greater degree of mixing. Moreover, it was determined that a higher solvent-to-feed ratio resulted in a greater percentage of ethanol extracted from 80.663% to 83.360%, which is attributed to increased solvent availability to extract ethanol from the feed mixture. The number of measured and predicted stages did not compare well for both the batch and semi-batch runs. This is likely due to insufficient time allowance for mixing to occur and steady state to be established, including the high ethanol feed ratio at the start of experiments. The results further indicated that a greater degree of separation (approximately 20% more ethanol) was achieved when the column was operated in semi-bath as opposed to batch mode.

Acknowledgements

My sincere gratitude to the following people for their assistance and guidance through this Laboratory research project:

My supervisors Prof. P. Naidoo, Dr. K. Moodley, and Dr. M. Williams-Wynn for all their assistance, advice, and guidance throughout this research project.

Mr. A. Khanyile and Ms. S. Naicker for their assistance with the experimental setup and technical support at all times.

Mr. N. Brijmohan for allowing my partner and I to use the equipment and for his invaluable assistance with the gas chromatograph.

My laboratory partner, Diya Singh, for being by my side throughout the late nights and stressful times, and for all of the help during the course of this project. ✓

Table of Contents

✓

List of Figures

✓

List of Tables

✓

Nomenclature

Symbol	**Description**	**Units**
A_i	Peak area of component i from gas chromatograph	Dimensionless
E	Mass of the extract	kg
F	Mass of the feed	kg
m_i	Mass of component i	g or kg
MM	Molar mass	g/mol
n_i	Number of moles of component i	moles
R	Mass of the raffinate	kg
S	Mass of the solvent	kg
x_i	Mass fraction of component i	Dimensionless

✓

1. Introduction

Rare earth elements (REEs) are becoming increasingly important in the technology industry due to the unique properties that the components exhibit (Lu, 2021). These elements are essential for the production of electronic, magnetic, and electrical devices such as smartphones, plasma screens, and magnets (Lu, 2021). However, the disposal of waste electronic and electrical equipment (WEEE) is increasing resulting in a significant concern of its impact in landfill sites.

Rare earth elements are found globally in ore deposits, where the most abundant ore deposits are located in China, Russia, and United States (McLeod, 2019). REEs, such as neodymium, dysprosium and praseodymium, are also mined in South Africa at the Steenkampskraal mine in the Western Cape, where neodymium exists in large quantities (Blench, 2017). Although rare earth ore deposits are considered plentiful, the concentration levels of rare earths within the ore are relatively low (Lu, 2021). Hence, there are limited rare earth elements available for the production of technological devices. Due to the similar chemical and physical properties that rare earth minerals portray, it is also difficult to extract specific rare earths from ore deposits while maintaining high product purities (99.999%). The mining of rare earths also has a detrimental impact on the environment, varying from soil erosion to pollution (Ives, 2013). Figure 1-1 displays an exponential growth in the demand for rare earths due to the role of these elements in modern technology. China is dominating in the production of rare earth elements, as seen in Figure 1-1 (King, 2020).

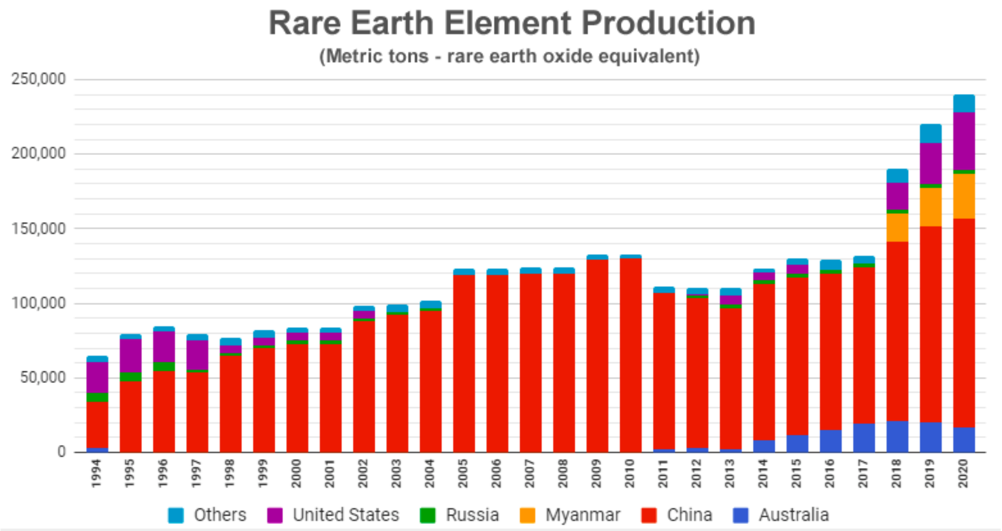


Figure 1-1*:* **Increase in the production of REEs (King, 2020***)*

Waste electronic and electrical equipment (WEEE), which contains rare earth elements, considerably fills landfill sites. WEEE consists of hazardous components that can leak toxic materials and radioactive waste into the water supply and damage the ecosystem (Vaccari, et al., 2019). Furthermore, WEEE has been banned from being deposited in landfill sites effect as of August 2021 in South Africa (EnviroServ Waste Management, 2020). Thus, it is prudent to determine effective methods of recovering rare earth elements from WEEE. One attractive process for the recovery of rare earth elements (neodymium) from WEEE is liquid-liquid extraction.

There are various separation techniques that exist to separate two or more components such as distillation, evaporation, drying, filtration, extraction etc. (Rathilal, 2010). However, liquid-liquid extraction has become an attractive separation method in cases where conventional distillation is not preferred (Seader, et al., 2011). Liquid-liquid extraction is extensively used in the petroleum, food, pharmaceutical, and chemical industries (Schmidt & Strube, 2018). Temperature-sensitive components and azeotropic mixtures can be easily separated via liquid-liquid extraction through the use of a solvent compared to distillation (Seader, et al., 2011). This separation method also does not require external energy, thus producing a more viable separation technique (Naidoo, 2012).

Various extraction equipment can be used to perform liquid-liquid extraction, such as mixer-settlers, unagitated columns including spray columns, packed columns, plate columns, and columns with mechanically assisted agitation (Seader, et al., 2011). Columns with agitation enhance mass transfer by allowing mixing to occur to improve the interaction between the components and produce fine droplets (Rathilal, 2010). The vibrating plate extraction (VPE) column incorporates mechanically assisted agitation. However, there is limited research available on the vibrating plate extraction column and the key parameters that affect the performance on the column. This column offers advantages over other types, such as the column can operate at high flow rates, high frequencies, and lower amplitudes (Lo, et al., 1992).

1.1 Aim and objectives

This research project involves re-commissioning and investigating the performance of a vibrating plate extraction column using the ethanol-cyclohexane-water system, so that the column can be fit-for-use in rare earth metal extraction research at the Thermodynamics Research Unit.

Aim: To re-commission and investigate the performance of the vibrating plate extraction (VPE) column operating on pilot scale under batch and semi-batch operation.

Objectives:

i. Perform runs on the vibrating plate extraction column using water to remove impurities in the column and to test the column’s functionality.
ii. Perform experimental runs on the vibrating plate extraction column using the ethanol-cyclohexane-water system to evaluate the operability of the column.
iii. Calibration and development of method for analysis using gas chromatography and performing uncertainty analysis.
iv. Use of the Shimadzu gas chromatograph 2014 to analyse samples obtained from the VPE column.
v. To evaluate the effect of the key parameters (vibrational frequency and solvent-to-feed ratio) on the percentage of ethanol extracted and the efficiency of the column.

The column was operated under batch and semi-batch mode to determine the method that allows for a more effective separation of ethanol (solute). A series of runs were performed on the column to obtain samples of the feed, raffinate, and extract. The samples were analyzed using gas chromatography to determine the composition of each of the components in the sample. The frequency of the column and the solvent-to-feed ratio were varied to evaluate the effect on the extent of separation and the efficiency of the column. From the results the separation efficiency was determined, and recommendations proposed for improvement, before commencing work on systems with neodymium-solvent-extractants.

2. Theoretical Background

This chapter presents an explanation of liquid-liquid extraction, selection of solvents, followed by a discussion of equipment used in similar extraction studies.

2.1 Liquid-liquid extraction

Liquid-liquid extraction is extensively used in the petrochemical, food, pharmaceutical, and chemical industries (Schmidt & Strube, 2018). Liquid-liquid extraction is based on mass transfer, where a solvent is added to extract the solute from the feed mixture (Seader, et al., 2011). The extraction of the solute from the feed occurs through diffusion, where the solute transfer is concluded after thermodynamic equilibrium is accomplished between the organic and aqueous phases (Naidoo, 2012). This separation method is advantageous compared to other separation processes due to the higher selectivity of the solvent to one of the components (solute) in the feed mixture (Seader, et al., 2011).

2.2 Uses of liquid-liquid extraction

In industry, there is growing interest in liquid-liquid extraction due to the various advantages that the process has to offer. Some of the benefits include low energy costs, higher purity products in some cases, better extractors available to perform the separation, and access to cheap solvents with higher affinity to components (Seader, et al., 2011). Liquid-liquid extraction is the preferred separation technique over distillation for (Seader, et al., 2011):

- Close-boiling point or close-melting point components.
- Temperature-sensitive components.
- The separation of azeotropic mixtures.
- The separation of mixtures using the chemical type of the components rather than the use of boiling points.
- The recovery of a substance present in relatively small amounts.

2.3 Advantages and disadvantages of liquid-liquid extraction

Liquid-liquid extraction is preferred when the use of distillation is not feasible. However, there are various advantages and disadvantages associated with liquid-liquid extraction. The advantages include the separation of azeotropic mixtures, heat-sensitive components, and components that exist in lower concentrations (Seader, et al., 2011). This separation technique is also favoured as it requires large capacities of chemicals with low energy usage while ensuring high product purities (Williams-Wynn, et al., 2020).

Despite the various advantages of liquid-liquid extraction listed above, there are a few disadvantages associated with it, such as (interchim, 2019):

- Requires large volumes of solvent, thus increasing the capital costs.
- The desired product (solute) contains the solvent; therefore, additional separation is required.

2.4 Solvent selection

A suitable solvent is a crucial requirement to ensure the effective separation of the solute from the feed mixture. The choice of solvent also determines the feasibility of the process (Seader, et al., 2011). There are various factors considered before choosing an appropriate solvent for efficient separation, such as (Seader, et al., 2011):

- The solvent should be stable, non-corrosive, and non-toxic to ensure a safe environment.
- The availability and the cost of the solvent due to the large volume required.
- The solvent should be recovered easily.
- The chosen solvent should be immiscible in the feed mixture, which allows the solute to be easily extracted from the feed mixture (solute and carrier).
- The solute should distribute in the solvent as the solvent must have a high affinity for the solute.
- The density of the solvent should be higher than that of the carrier in the feed mixture.
- Low viscosity to enhance mass transfer.

The most important factors taken into account for the solvent choice are environmental considerations and relative selectivity (Seader, et al., 2011). The solvent is evaluated using the selectivity and the partition coefficient of the solute (ethanol) related to the carrier (cyclohexane) and the solvent (water) (Seader, et al., 2011). For the ethanol-cyclohexane-water system, water has a higher affinity towards ethanol (solute). Thus, water was selected as the solvent. This is expanded further in Chapter 2.8.

2.5 Counter-Current Extraction

The configuration of counter-current extraction allows for better contact between the solvent and the feed (Seader, et al., 2011). Thus, increasing the extent of the separation and the efficiency due to better interaction. Figure 2-1 shows a counter-current extraction process where the feed comprises of the solute (A) and the carrier (B), and the pure solvent (S) is added to the column through the top. The extract is rich in solute, which consists mainly of the solute and the solvent. The raffinate is comprises of the solute and carrier.

✓

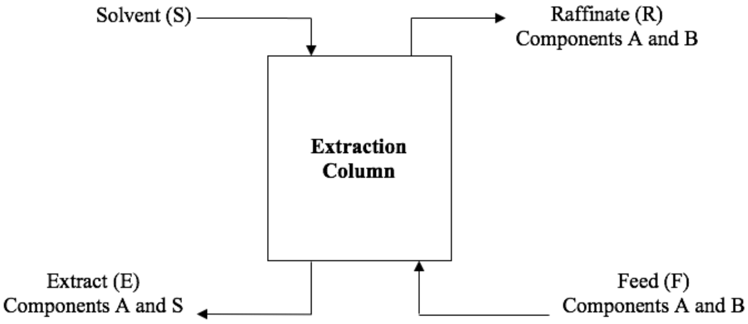


Figure 2-1: Counter-current extraction process adapted from (Seader, et al., 2011)

The extent of separation is dependent on the transfer of the solute, which only occurs effectively after thermodynamic equilibrium is established between the phases (Naidoo, 2012). The organic and aqueous phases are identified as the light and heavy phases, respectively. The light phase is the lower density phase, which moves up the column and accumulates at the top. The heavy phase is considered as the phase with a higher density, which moves down the column and collects at the bottom of the extraction column (Naidoo, 2012).

✓

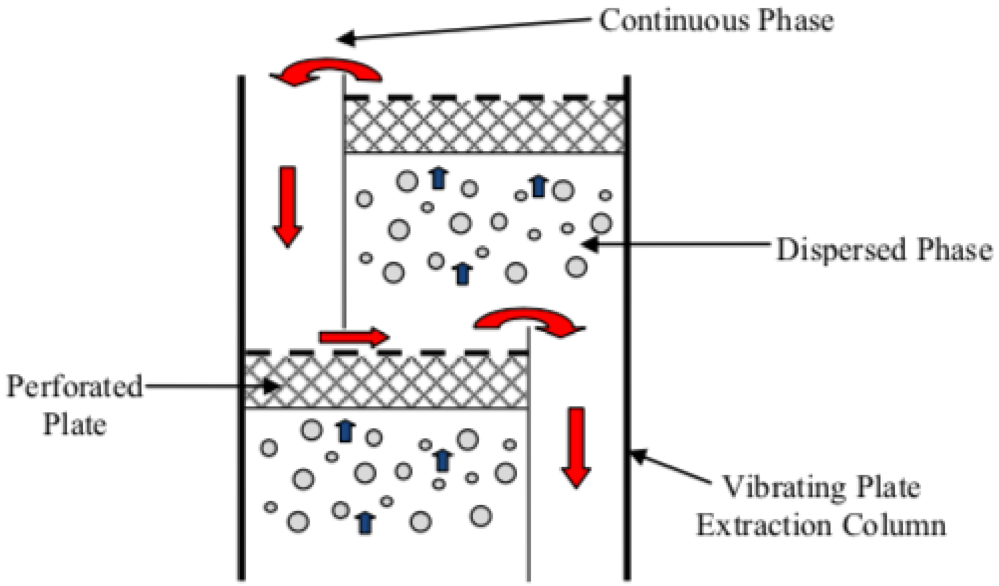


Figure 2-2: Counter-current flow of dispersed and continuous phases across the perforations in the VPE column (Fair & Humphrey, 1983).

In the column, the dispersed and continuous phases are observable, as indicated in Figure 2-2. The dispersed and continuous phases are determined according to the wettability of the components with the interior of the column (Seader, et al., 2011). The perforated plates were fabricated using stainless steel and water has a wettability to stainless steel (Naidoo, 2012). Hence, water was considered as the continuous phase (aqueous). The dispersed phase flows up the column through the perforations to form small droplets. However, the continuous phase moves down the column through the downcomer without producing droplets (Naidoo, 2012).

2.6 Types of Extraction columns

There are various equipment used to perform liquid-liquid extraction, such as mixer-settlers, spray columns, packed columns, plate columns, and columns with mechanically assisted agitation (Seader, et al., 2011). The separation of the phases occurs by gravity due to the large difference in the phase densities (Seader, et al., 2011). However, if the difference is small, then a centrifugal force is added to enhance the settling of the phases (Seader, et al., 2011). There are several advantages and disadvantages associated with each of these extraction columns, which is listed below adapted from (Seader, et al., 2011):

Table 2-1: Advantages and disadvantages of extraction columns (Seader, et al., 2011)

Type of extraction equipment	Advantages	Disadvantages
Mixer-settlers	1. High efficiency 2. Easy scale-up 3. A range of flow ratios possible 4. Good interaction between the components 5. Several stages are possible	1. High liquid holdup 2. Power costs are high 3. Requires a large floor space 4. May need interstage pumping 5. High investment
Counter-current extractors (no mechanical agitation)	1. Low initial cost 2. Low operating cost 3. Easiest extractor to construct	1. Low efficiency 2. Hard to scale-up the process 3. High headroom 4. High flow ratios cannot be controlled 5. Limited throughput with small density difference
Counter-current extractors (mechanical agitation)	1. Good distribution of the components 2. Several stages are available 3. Easy to scale-up the process 4. Reasonable operating costs	1. Cannot control high flow ratios 2. Emulsifying systems cannot be used 3. Limited throughput with small density difference

From Table 2-1 it can be seen that counter-current extractors with mechanical agitation are suitable to perform liquid-liquid extraction. This is due to the good dispersion of the components and the low costs required to operate the column. This type of extractor also only has a few disadvantages compared to mixer-settlers and extractors with no agitation.

2.7 Vibrating Plate Extraction Column (VPE)

Previous studies conducted by Vaclav and Carsky (Vaclav & Carsky, 2020), reported on the use of LLE to separate neodymium, praseodymium, and dysprosium. Vaclav and Carsky (Vaclav & Carsky, 2020) found that liquid-liquid extraction was an effective way of extracting rare earth elements from waste magnets as they achieved a product purity of at least 99% for each of the rare earth components (Vaclav & Carsky, 2020). Thus, the vibrating plate extraction column (available in Chemical Engineering laboratories) was chosen to perform liquid-liquid extraction due to the high purities and low costs obtained from similar previous studies. The vibrating plate extraction column is a form of a reciprocating plate extraction column designed by Prochazka and colleagues (Prochazka, et al., 1971). The vibrating plate extraction column consists of small perforations and downcomers to allow effortless movement of the continuous phase (Naidoo, 2012). This extraction column is specifically chosen over other extractors as it can be utilized at higher frequencies and lower amplitudes (Naidoo, 2012). However, there are still advantages and disadvantages related to the vibrating plate extraction column. The advantages are adapted from (Prochazka, et al., 1971) and (Lo, et al., 1992). The disadvantages are adapted from (Rama Rao, et al., 1991) and (Takacs, et al., 1993). This is shown in Table 2-2.

Table 2-2: Advantages and disadvantages of the VPE column.

Advantages	**Disadvantages**
1. Simple to construct the column and easy maintenance 2. Consistent and easy scale-up 3. The key parameters can be easily adjusted in the column 4. The column can operate at high flow rates, resulting in a higher extent of separation and efficiency	1. Not appropriate for feed mixtures that contain solid components 2. The occurrence of entrainment due to small droplets 3. Axial mixing can occur due to higher energy, which decreases the effectiveness of the column.

2.8 Ternary System

For this research project, a system was chosen such that it produces a type I ternary system that would replicate the behaviour of the neodymium-aqueous nitric acid-kerosene mixtures that is to be used in further research on this project. The type I, ethanol-cyclohexane-water, system was chosen to perform liquid-liquid extraction instead of the neodymium-nitric acid system (initial system) due to the variety of liquid-liquid extraction data available in literature sources, as well as the low price and the availability of these chemicals. Type I systems form a miscibility region at all points between the solute and the solvent, which is not the case for type II systems (Seader, et al., 2011). Type I systems are preferred as type II systems require large volumes of solvent for effective separation due to the tie-line data, thus increasing the expenses of the process (Seader, et al., 2011).

The following Figure 2-3 depicts the ternary diagram of the popular system of ethanol-cyclohexane-water:

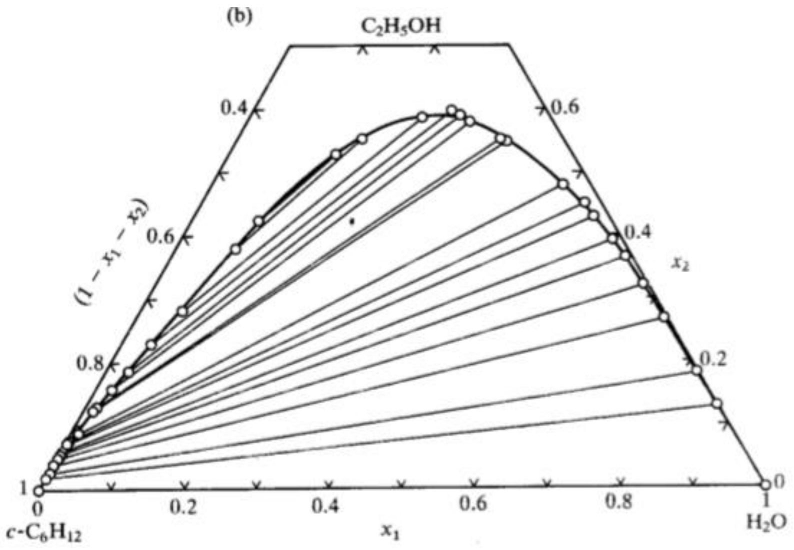


Figure 2-3: Ternary diagram of the ethanol-cyclohexane-water system (Moriyoshi, et al., 1991).

According to the tie-line data, for a (cyclohexane + ethanol) mixture, the use of water as a solvent shows a higher percentage of ethanol in the extractant phase. Water is also a suitable solvent for the system due to the ideal solvent criteria that it exemplifies, such as a higher density than the feed, the availability and inexpensive cost of water compared to other chemicals, the immiscibility of water in the feed, and the non-toxic nature of the water. In this system, ethanol is the solute, and cyclohexane is the carrier. Thus, in the feed mixture only ethanol and cyclohexane will be present. ✓

2.9 Key Parameters

The key parameters that affect the extent of the separation and the efficiency of the column are the agitation level (product of amplitude and frequency) and the solvent-to-feed ratio. The agitation level is adjusted by varying the vibrational frequency of the column. The solvent-to-feed ratio is varied by adding more solvent, thus increasing the solvent-to-feed ratio. A higher frequency and solvent-to-feed ratio should result in a greater separation efficiency of the column. ✓

2.9.1 Extent of separation

The overall material balance for the counter-current extraction column is (Seader, et al., 2011):

$$F + S = E_1 + R_N \qquad (2\text{-}1)$$

where E is the mass of the extract, R is the mass of the raffinate, F is the feed mass, and S is the solvent mass. N is the number of stages.

The ethanol mass balance is depicted below (Seader, et al., 2011):

$$Fx_{ethanol(F)} = Ex_{ethanol(E)} + Rx_{ethanol(R)} \qquad (2\text{-}2)$$

Since the mass of the feed and solvent are known, and the compositions of ethanol in the extract and raffinate can be obtained from the gas chromatograph, Equations (2-1) and (2-2) can be solved simultaneously to determine the amount of extract and raffinate in kilograms.

The extent of separation is evaluated by considering the amount of ethanol (solute) extracted. The equation required to calculate the amount of ethanol extracted adapted from (Seader, et al., 2011) utilizes the number of moles of ethanol present in the feed and the raffinate:

$$\%\ \text{ethanol extracted} = \frac{n_{feed} - n_{raffinate}}{n_{feed}} \times 100 \qquad (2\text{-}3)$$

The compositions used to determine the number of moles in the raffinate are obtained using analyses from gas chromatography, which is explained further in the experimental procedure. ✓

2.9.2 Number of Predicted and Measured Stages in the VPE Column

The vibrating plate extraction column used in this experimental work was reported to have 4 number of theoretical stages according to (Naidoo, 2012) and (Rathilal, 2010). However for this research project, the number of theoretical stages will be determined by stepping off the ternary diagram. The operating point (P) on the ternary diagram will be constructed by drawing a line through points (E_1, F) and (S, R_N) (Seader, et al., 2011). The side at which the operating point (P) is situated depends upon the slope of the tie-lines, the feed composition and the solvent-to-feed ratio (Seader, et al., 2011). The tie-lines and the operating point is utilized to step off the ternary diagram to determine the equilibrium stages. This is depicted in Figure 2-4 (Seader, et al., 2011).

✓

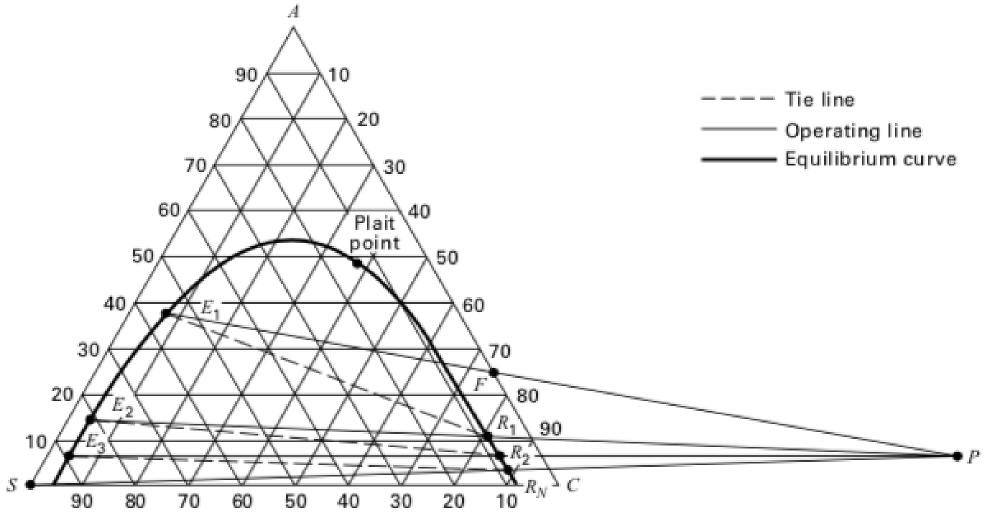


Figure 2-4: **Stepping off ternary diagram (Seader, et al., 2011)**

The measured stages will be obtained by stepping off the diagram using the extractant and raffinate compositions obtained from the gas chromatograph. However, according to the Hunter-Nash method a raffinate of 99% carrier (cyclohexane) can be obtained (Seader, et al., 2011). Thus, the predicted number of stages will be determined using the extract composition from the gas chromatograph and the raffinate of 99% carrier. The predicted and measured number of stages is an important factor, as it evaluates the variation between the stages obtained. The measured and predicted number of stages will be compared to give an indication of the performance of the column.

✓

3. Experimental Procedure

The ethanol-cyclohexane-water system was chosen as the immiscible ternary mixture to evaluate the operation of the column. The re-commissioning of the column required performing experiments to investigate the performance of the vibrating plate extraction column, so that the column can be used for future experiments conducted with the extraction of rare earth metals. This is one of the focal projects currently underway in the Thermodynamics Research Unit. Liquid-liquid extraction was the separation procedure adopted to separate ethanol (solute) from a feed mixture consisting of ethanol and cyclohexane using water as the solvent. This solvent was chosen since it resulted in a greater composition of ethanol in the extract according to the ternary diagram (Figure 2-3) as explained in Chapter 2.8. The samples obtained from the column for the raffinate and extract were analyzed using gas chromatography. With cyclohexane and water being immiscible a miscible internal standard was necessary to ensure that the sample in the sample vial did not form two liquid phases.

3.1 Materials

The chemical reagents utilized in this research project are tabulated in Table 3-3 with additional information. For further details about the properties and the hazards associated with the chemicals, the Material Safety Data Sheet is attached in Appendix G.

Table 3-1: List of chemicals with supplier details and specifications

Chemical name	Chemical formula	CAS number	Purity (wt %)	Supplier
Ethanol	C_2H_5OH	64-17-5	$\geq$ 99.5%	Sigma-Aldrich
Cyclohexane	C_6H_{12}	110-82-7	$\geq$ 99.5%	Honeywell Riedel-de Haën
Deionized water	H_2O	7732-18-5	-	ELGA
Acetone	C_3H_6O	67-64-1	$\geq$ 99.5%	Sigma-Aldrich

The conductivity of the deionized water was 0.069 μS/cm.

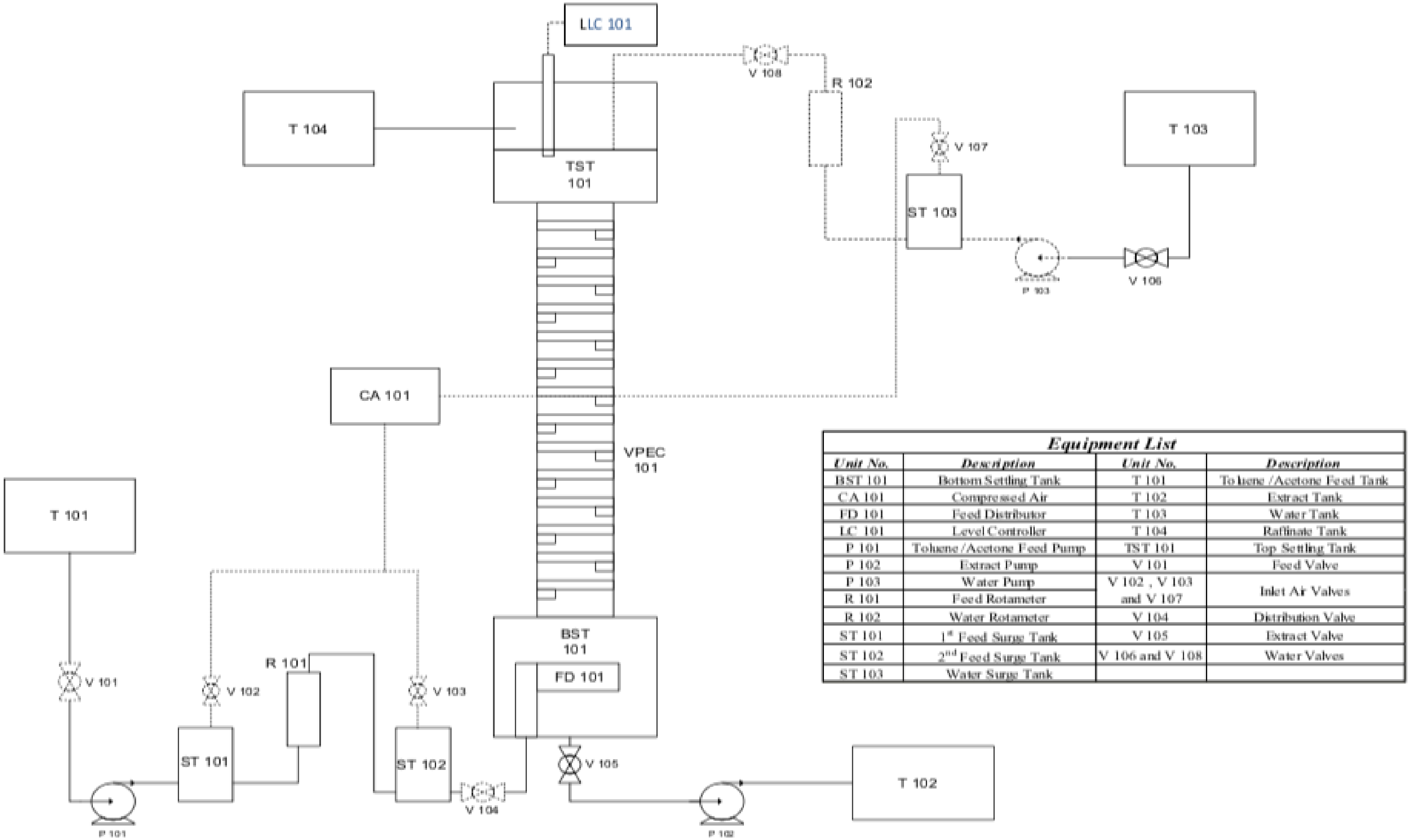
3.2 Apparatus

The equipment and resources required to conduct the research project are listed below:

- One beaker (2 L)
- One measuring cylinder (1 L)
- Vibrating Plate Extraction Column
- Two 5 L drums for the feed and solvent
- Kern mass balance
- 18 mL sample vials
- Shimadzu gas chromatograph (GC) 2014 with a Chromosorb WHP SE 30 column
- GC syringe (1 microlitre)
- Pt-100 probe
- Mensor CPC 3000

The temperature and pressure of the vibrating plate extraction column was measured using a temperature probe (Pt-100 probe) and a pressure sensor (Mensor CPC 3000), respectively, to determine the conditions of the extraction column. The uncertainties associated with the temperature probe and pressure sensor were expanded further in Chapter 4.2.

3.3 Experimental Layout



Equipment List

Unit No.	*Description*	*Unit No.*	*Description*
BST 101	Bottom Settling Tank	T 101	Toluene /Acetone Feed Tank
CA 101	Compressed Air	T 102	Extract Tank
FD 101	Feed Distributor	T 103	Water Tank
LC 101	Level Controller	T 104	Raffinate Tank
P 101	Toluene /Acetone Feed Pump	TST 101	Top Settling Tank
P 102	Extract Pump	V 101	Feed Valve
P 103	Water Pump	V 102 , V 103 and V 107	Inlet Air Valves
R 101	Feed Rotameter		
R 102	Water Rotameter	V 104	Distribution Valve
ST 101	1st Feed Surge Tank	V 105	Extract Valve
ST 102	2nd Feed Surge Tank	V 106 and V 108	Water Valves
ST 103	Water Surge Tank		

Figure 3-1: Schematic of a vibrating plate extraction column with full equipment and ancillaries (Naidoo, 2012)

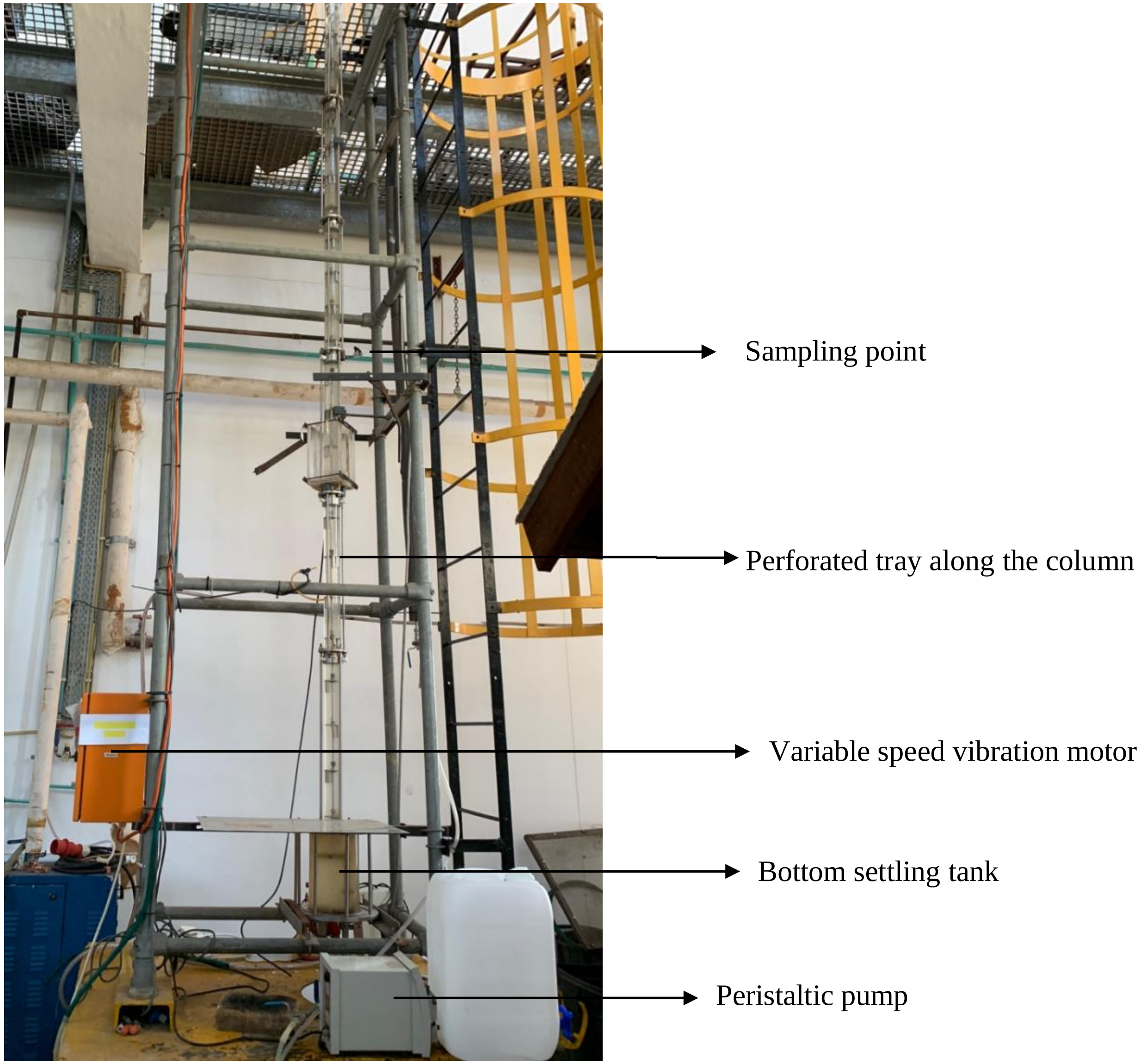


Figure 3-2: Vibrating plate extraction column

3.4 Description of the equipment

The vibrating plate column used in this project was commissioned, tested and reported in two previous studies (Rathilal, 2010) and (Naidoo, 2012). Since the column was not operated under continuous mode, not all of the equipment and ancillaries depicted in Figure 3-1 were required to conduct the experiments in this project. Hence, only the equipment associated with this research project is described in Chapter 3.4. However, for a thorough description of the vibrating plate extraction column and the units portrayed in the Chapter 3.6 of the theses, the reader is referred to the following theses: (Rathilal, 2010) and (Naidoo, 2012).

3.4.1 Vibrating Plate Extraction Column

The vibrating plate extraction column trays consist of small perforations and downcomers (perforated trays) along the column to allow effortless flow of the organic and aqueous phases (Naidoo, 2012). The following Figure 3-3 shows the downcomers and small perforations.

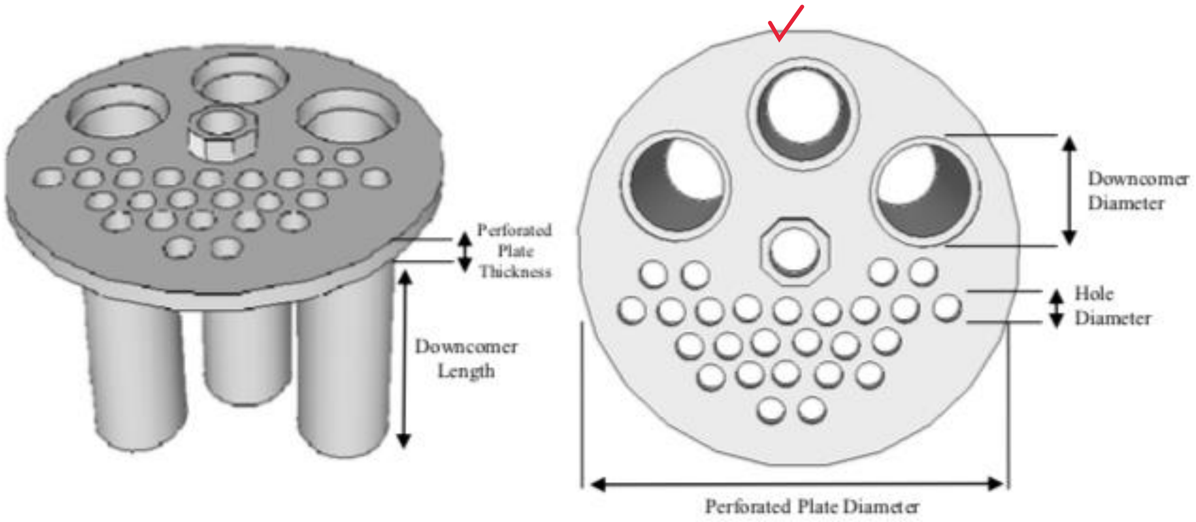


Figure 3-3: Perforated trays (Naidoo, 2012)

The specifications of the extraction column are stipulated in Table 3-2.

Table 3-2: Specifications of the Vibrating Plate Extraction Column (Naidoo, 2012)

Inner diameter (mm)	47.7
Outer diameter (mm)	58.7
Thickness of trays (mm)	5.70

Number of sections	8
Length of each section (mm)	550
Effective height of the column (m)	4.76
Cross sectional area of the column ($\mathbf{m^2}$)	1.787×10^{-3}

3.4.2 Settlings tanks

The extraction column consists of two settlings tanks located at the top and the bottom of the column. The function of the settling tanks is to maintain the partitioning of the phases and to ensure liquid-liquid interface between the dispersed and continuous phases (Naidoo, 2012). A level controller is also situated in the settling tank to maintain the interface level (Naidoo, 2012). Figure 3-4 distinctly shows the liquid-liquid interface between the dispersed (organic) and continuous (aqueous) phases, where the organic phase was at the top and the aqueous phase was situated at the bottom.

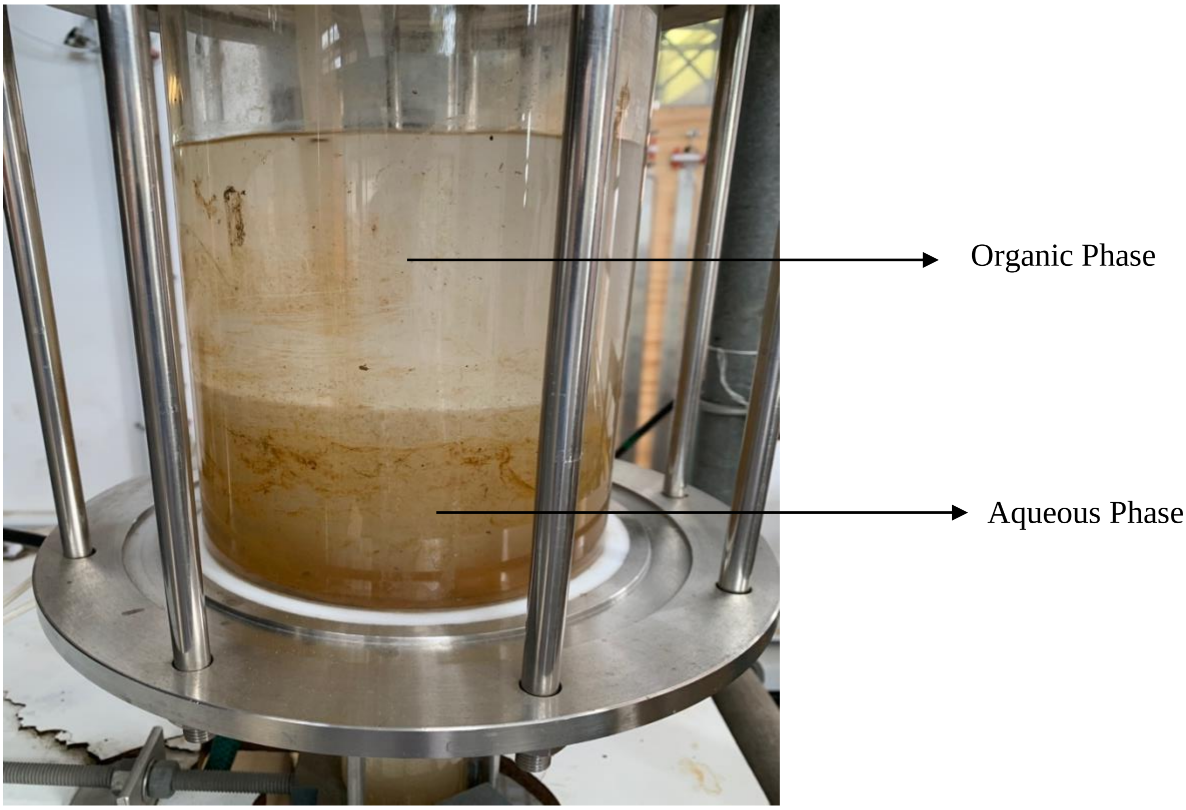


Figure 3-4: Top settling tank

Table 3-3: Specifications of the Settling Tanks (Naidoo, 2012)

Inner diameter (mm)	150
Outer diameter (mm)	160
Thickness of tank (mm)	5
Length of the tank (mm)	250
Cross sectional area of the tank (m^2)	17.671×10^{-3}

3.4.3 Variable Speed Vibration Motor

In this research project, the vibrational frequency of the column was altered while the amplitude of 3.37 remained constant. The amplitude measures the extent of the vibration. The vibrational frequency of the column was adjusted using the variable speed vibration motor, which resulted in the vibrations of the perforated plates (Naidoo, 2012). The column is known as a vibrating plate extraction column due to the reciprocations of the perforated plates (Naidoo, 2012). The variable speed vibration motor operated at a power of 0.75 kW and a voltage of 220V (Naidoo, 2012). The frequency unit is Hertz (Hz) as shown in Figure 3-5, where the vibrational frequency of the column was set to 15 Hz.

Figure 3-5: Variable speed vibration motor

3.4.4 Sampling points

Three sampling points are situated along the column to allow samples to be removed from the different phases (organic and aqueous) in the extraction column. The samples were analyzed using gas chromatography to establish the compositions of each of the components at the sampling points along the column (Naidoo, 2012). The composition at the top sampling point was assumed to be equivalent to that of the raffinate. Similarly, the composition at the bottom sampling point was assumed to be the same as the extract composition. This assumption applies if no concentration exists along the column length after good mixing and settling.

3.4.5 Peristaltic Pump

The peristaltic pump was situated at the bottom of the vibrating plate extraction column. The peristaltic pump depicted in Figure 3-6 was utilized.

Figure 3-6: Heidolph peristaltic pump

The function of the peristaltic pump is to feed the solvent and the feed mixture into the extraction column. The speed of the pump was adjusted in order to vary the flow rates. The specifications of the peristaltic pump are indicated in Table 3-4 (Naidoo, 2012).

Table 3-4: Specifications of the Peristaltic Pump (Naidoo, 2012)

Pump name	Heidolph PD5106
Maximum speed (rpm)	600
Maximum flow rate (l/h)	160
Speed used (rpm)	100
Flow rate used (l/h)	26.67

3.4.6 Gas Chromatograph

✓

A gas chromatograph was employed to analyze the samples obtained from various sampling points along the extraction column. The Shimadzu gas chromatograph 2014 shown in Figure 3-7 was utilized.

Figure 3-7: Shimadzu gas chromatograph 2014

A sample consisting of 1 μL obtained from the extraction column (raffinate and extract) was injected using a GC syringe into the septum of the column of the gas chromatograph. The carrier gas used was helium, which assisted in the movement of the sample through the packed column (Naidoo, 2012).

Table 3-5: Specifications and conditions of the gas chromatograph

Name	Shimadzu GC-2014
Injector temperature (℃)	200
Run time (minutes)	30
Column Name	Chromosorb WHP SE 30
Column pressure (kPa)	305.5
Column flow (mL/min)	30
Oven temperature (℃)	200
Column length (m)	3
Column inner diameter (mm)	2
Detector temperature (℃)	200

Binary samples of the chemicals used, each with acetone, were prepared to conduct the calibration of the gas chromatograph detector. This was done by weighing each amount of the chemical in a sample vial, using a mass balance with precision of $\pm$ 0.00048 g and a readability of 1 mg. Since the ternary system consisting of ethanol, cyclohexane, and water are immiscible, the addition of a miscible solvent (acetone used as the internal standard) was essential for the calibration of the gas chromatograph detector and the analyses of the immiscible samples obtained from the column. Acetone was chosen as the internal standard due to the availability of the chemical in the Chemical Engineering Laboratories and its compatibility with the three components.

The calibrations were performed in the dilute and concentrated regions for two different binary mixtures such as acetone-ethanol, acetone-cyclohexane, and acetone-water. Therefore, the standard mixtures of each of the components (ethanol, cyclohexane, water, and acetone) were prepared in specific mass ratios varying from 0 to 1 $\left(\frac{x_i}{x_j}\right)$ and 0 to 1 $\left(\frac{x_j}{x_i}\right)$ in the dilute and concentrated regions, respectively. The standard solutions were shaken well before injecting a

1 microlitre sample into the column of the gas chromatograph. The binary mixtures required for the calibration were evaluated twice in order to produce a reliable calibration graph and ensure accuracy of the results.

For each of the binary mixtures, two GC peak areas were obtained. The peak area ratios $\left(\frac{A_i}{A_j}\right)$ and $\left(\frac{A_j}{A_i}\right)$ in the acetone-rich and acetone-dilute regions attained from the gas chromatograph were plotted against the mass ratios of the components $\left(\frac{x_i}{x_j}\right)$ and $\left(\frac{x_j}{x_i}\right)$, respectively. These were plotted in the acetone-rich and acetone-dilute regions. The calibration plots were presented in Appendix C for each component, and acetone in the acetone-rich and acetone-dilute regions. The calibration plots were used to evaluate the composition of the components in the samples. Refer to Appendix D for detailed calculations explaining how the calibration plots were used to back calculate the ethanol composition in the original mixture.

3.5 Experimental Procedure

The column was operated in batch and semi-batch mode. Alterations on the column were done to allow for the operation in semi-batch mode, such as the solvent and feed valves were added, and a tube was also connected to the feed drum to enable the overflow mixture to be recycled back into the column through the bottom. The batch operation utilizes approximately 12 litres of chemicals. However, the semi-batch operation requires 18 litres of chemicals as the column needed to be filled to the overflow line which allows the chemicals to be recycled back into the feed drum. After the completion of the experimental runs, the samples obtained were then analyzed using gas chromatography.

3.5.1 Batch Mode

i. The feed mixture consisting of 3.71 litres of ethanol (48.99 wt. %) and 3.04 litres of cyclohexane (51 wt. %) was measured and prepared using a beaker.
ii. The peristaltic pump was switched on, and the speed of the pump was adjusted to 100 rpm.
iii. 4.19 litres of water (solvent) was first fed into the extraction column through the top via the peristaltic pump which was set to 100 rpm.

iv. The feed mixture of ethanol and cyclohexane was then pumped into extraction column through the top using the same pump (100 rpm).

v. After all the mixture was transferred into the column, the pump was switched off.

vi. The frequency of the extraction column was set to 10 Hz for the first batch run and 15 Hz for the second batch run. The frequency was varied using the variable speed vibration motor. This allowed for the first key parameter (vibrational frequency) to be altered.

vii. Sufficient time was allowed for the system to mix efficiently. Hence, one hour was allocated for the chemicals to interact.

viii. After mixing occurred, the vibrational frequency was switched off. Thereafter, another hour was allocated for the settling of the phases (organic and aqueous) in the extraction column. Settling was required for the system to stabilize otherwise the phases would be dispersed throughout the column.

ix. The samples were withdrawn in 18 mL sample vials from two sampling points along the extraction column. The withdrawal of samples from the top and bottom sampling points allowed for the analyses of the raffinate and extract, respectively. These sample vials were stored in the refrigerator to avoid disturbances in the samples.

x. The samples from the column (extract and raffinate) were then analyzed using the Shimadzu gas chromatograph 2014 to determine the composition of ethanol available in the extract and raffinate.

3.5.2 Semi-batch Mode

i. The feed mixture consisting of ethanol and cyclohexane was prepared in the following mass compositions: 54 wt. % ethanol and 46 wt. % cyclohexane for the first semi-batch run and 52.04 wt. % and 47.96 wt. % for the second semi-batch run.

ii. First, the solvent valve was opened. Thereafter, the peristaltic pump was switched on and the speed of the pump was adjusted to 100 rpm. The solvent valve allowed the solvent to be fed into the column.

iii. Three litres of water (solvent) was pumped into the extraction column through the top via the peristaltic pump at a speed of 100 rpm for the first semi-batch run. Four litres of water (solvent) was required for the second semi-batch run to allow for the variation of the solvent-to-feed ratio. Thus, the second key parameter (S/F) was altered.

iv. After all the water was transferred into the column, the solvent valve was closed, and the feed valve was opened.

v. The feed mixture consisting of ethanol and cyclohexane was then pumped through the bottom of the extraction column using the same pump (as the solvent). The solution of the solvent and feed mixture was measured and set, such that the solution reached the overflow line in the top settling tank. This enabled the mixture in the column to overflow and be transferred back into the feed drum then back into the column through the bottom inlet. This allowed the mixture to be recycled, thus resulting in semi-batch operation.

vi. The frequency of the extraction column was adjusted to 15 Hz using the variable speed vibration motor. The frequency of the column was set at 15 Hz for the first and second semi-batch runs.

vii. Sufficient time (one hour) was allocated for efficient mixing to occur and for steady state to be achieved – previous experimental work conducted by (Rathilal, 2010) and (Naidoo, 2012) proved that 45 minutes was sufficient. Hence, one hour was allocated for steady state to be established.

viii. For every 15 minutes, samples were withdrawn from the sampling points along the extraction column and the feed drum. This was done for a period of one hour. The data was used to plot the composition profiles over a period of time.

ix. After one hour, the vibrational frequency was switched off.

x. The peristaltic pump was then stopped and the mixture from the extraction column was removed.

xi. The samples obtained from the column and the feed drum were analyzed using gas chromatography to determine the composition of ethanol available in the extract, raffinate, and feed drum.

✓

3.4.3 Gas Chromatograph Procedure

i. The flow rate of the helium gas was switched on and the flow rate was adjusted to 30 mL/min.

ii. The oven was switched on. Thereafter the oven, injector and detector temperatures were set to 200℃.

iii. Once the oven and detector temperatures had increased and remained constant, the detector was switched on.

iv. The detector was allowed to stabilize.
v. The samples obtained from the column and feed drum were injected into the septum of the Chromosorb WHP SE 30 column.
vi. Once all the sample analyses were concluded, the gas chromatograph was turned off.
vii. The detector was also switched off.
viii. The gas flow rate of the carrier was then set to 3 mL/min.
ix. Thereafter, the temperature of the column, injector and detector were adjusted to 25℃ for the gas chromatograph.

3.5.4 Analyses of Samples

i. The samples obtained from the column and feed drum were removed from the fridge and were allowed to warm to ambient temperature.
ii. The mass of the vial containing the mixture sample was obtained to determine the amount of sample in the vial. The mass of the empty vial was measured and prerecorded earlier. Therefore, the mixture mass can be easily determined.
iii. A known amount of acetone (internal standard) was added to the vial. The vial was then mixed well to ensure that all of the components were miscible, and no liquid-liquid interface was formed in the vials.
iv. A 1 μL of sample was inserted into the septum of the column in the gas chromatograph.
v. Since the amount of acetone added was known, the peak area ratios were associated to the mass ratios of the components using the calibration plots to determine the composition of each of the components in the samples.

3.6 Safety and Operational Aspects

Laboratory coats, safety glasses, and safety shoes were worn at all times during experiments. Gloves were worn at all times when preparing the feed mixtures (ethanol-cyclohexane) and while withdrawing samples from the extraction column. In the event of a spillage, the chemical spill must be mopped, the safety procedure followed as indicated on the material safety data sheets (Appendix G), and the area must be cleaned. A helmet was worn when climbing the ladder along the column to withdraw samples. After chemical usage, the chemicals were disposed in a waste bottle allocated to the group. It was ensured that chemicals and liquids did not come into contact with electrical wires. Due to Covid-19, masks were worn at all times.

4. Results and Discussion

The experimental runs were performed using an ethanol-cyclohexane-water system for the batch and semi-batch operation. For the batch operation, the vibrational frequency was varied. For the semi-batch operation, the solvent-to-feed ratio along with the frequencies were also altered. The variations (vibrational frequency and S/F) and different mode of operations were utilized to evaluate effect these settings had on the percentage of ethanol (solute) extracted and the efficiency of the column. Before the analyses of the samples were conducted, the gas chromatograph detector was calibrated. The feed, extract, and raffinate samples were analysed using a Shimadzu gas chromatograph 2014 to determine the amount of ethanol present in the sample, hence was extracted in the column.

4.1 Calibration of the Gas Chromatograph Detector

The calibration of the gas chromatograph detector was conducted to determine the uncertainties associated with the equipment and to increase the reliability of the results. The samples of the binary systems in the acetone-rich region and acetone-dilute region were injected into the gas chromatograph twice to ensure repeatability and to obtain reliable calibration plots. The calibration curves were produced by plotting the area ratios against the mass fraction ratios in the dilute and concentrated ranges, as explained in Chapter 3.4.6.

Figures C-1 to C-6 in Appendix C depict the calibration curves for the binary systems, comprising, ethanol, cyclohexane, and water, and acetone in the acetone-rich region and acetone-dilute region. From these Figures, it was seen that the correlation coefficient for all of the calibration plots range from 0.995 to 0.999. The correlation coefficient evaluates the relationship between two variables (Nickolas, 2021), such as area ratios and mass fractions. A value closer to 1 represents a perfect relationship (Nickolas, 2021). Hence, the calibration curves were deemed reliable. However, there was still some error associated with it. Table 4-1 below shows the gradients and inverse gradients for each of the calibration plots, as well as the uncertainty related to the calibration plots.

Table 4-1: Response Factor and Uncertainty of GC Detector Calibration Plots

Standard sample	**Response ratio of acetone rich region**	**Response ratio of acetone dilute region**	**Percentage error of acetone rich region**	**Percentage error of acetone dilute region**
Ethanol-acetone	0.956 = (1/1.046)	1.072 = (1/0.933)	0.691	0.281
Cyclohexane-acetone	0.724 = (1/1.381)	1.288 = (1/0.776)	6.544	3.451
Water-acetone	1.115 = (1/0.897)	0.852 = (1/1.174)	1.260	2.803

As seen in Table 4-1, the inverse gradient of the calibration curve for the acetone-rich region was similar to the gradient of the acetone-dilute region. This postulates that the calibration of the gas chromatograph detector was carried out correctly and accurately. However, there was still some error related to the calibration curves. The uncertainty displayed in Table 4-1 were low for the ethanol-acetone binary mixture, well below 1%. However, the error associated with the cyclohexane-acetone and water-acetone binary systems was significantly higher, with the water-acetone at 1.2-2.8% and the cyclohexane and acetone error in the range of 3.4-6.6%. This could be due to the area peaks overlapping which resulted in the temperatures of the gas chromatograph being changed from 200℃ to 180℃. The percentage errors associated with the binary systems were fairly low. However, measurement errors and the vaporization of acetone could have been some of the errors that led to a greater uncertainty.

4.2 Batch Operation

The first experiment on the vibrating plate extraction column was batch mode, where the feed mixture entered the column first. Thereafter, the solvent was fed into the column through the top. The temperature and pressure at the base of the column was measured using a Pt-100 probe and a Mensor CPC 3000, respectively. The column operated at a temperature of approximately 25.40℃ with an uncertainty of ± 0.05℃ and a pressure of 99.481 kPa with a full-scale uncertainty of $\pm 0.025\%$.

The vibrational frequency was varied in batch runs 1 and 2 where a frequency of 10Hz and 15Hz was used, respectively. These vibrational frequencies were chosen as a low frequency would result in inefficient mixing and a high frequency would lead to flooding (Naidoo, 2012).

Inefficient mixing would result in lower percentages of ethanol extracted. Flooding greatly reduces the efficiency of the tray and has a significant impact on the product purity due to contamination (Seader, et al., 2011).

4.2.1 Percentage of Ethanol Extracted

The percentage of ethanol extracted was determined using the number of moles of ethanol in the feed and the raffinate as presented in Equation 2-2. In Appendix D, a detailed sample calculation is presented relating to the calculation of the percentage of ethanol extracted.

Table 4-2: Ethanol extracted for Batch runs 1 and 2

Operation	Number of moles of Ethanol in the feed	Number of moles of Ethanol in the raffinate	Percentage of Ethanol extracted
Batch Run 1	63.383	25.058	60.47
Batch Run 2	63.383	22.648	64.27

Shown in Table 4-2, the percentage of ethanol extracted increased from 60.47% to 64.27% as the vibration frequency increased from 10 Hz (run 1) to 15 Hz (run 2). This was due to a higher frequency in batch run 2, thus resulting in more effective mixing, mass transfer, and greater interaction between the components. This allowed for more ethanol to be transferred from the feed mixture to water (solvent). (Naidoo, 2012) and (Rathilal, 2010) also observed that a greater percentage of solute was extracted when the vibrational frequency was increased. Thus, the observations made in this research project correlate well with the expectations and to previous studies conducted.

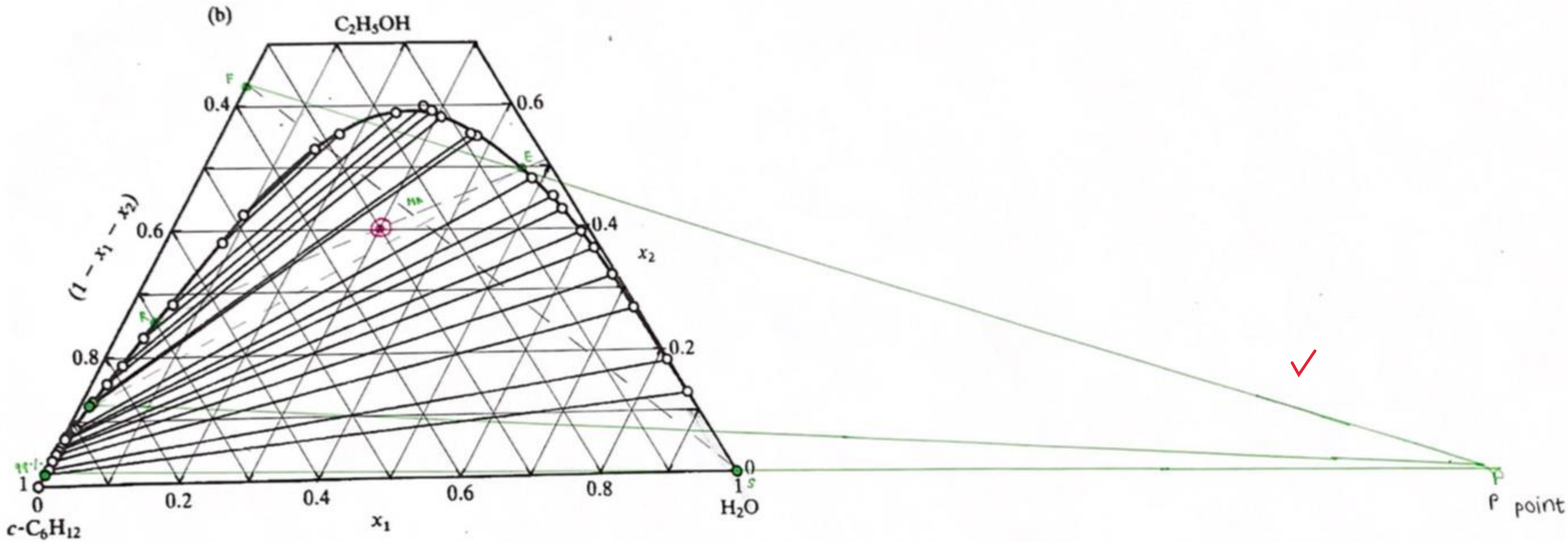
4.2.2 Number of Measured and Predicted Stages

The number of theoretical stages were obtained by applying the stepping-off technique using the ternary diagram for the ethanol-cyclohexane-water system. The ternary diagram for batch run 1 is presented in Chapter 4.2.2 as an example to indicate the stepping-off procedure on the ternary diagram. However, the remaining ternary diagrams are presented in Appendix F. The feed composition on a mole basis consisted of 0.64 ethanol and 0.36 cyclohexane. The ethanol composition in the feed (F) was quite high as indicated on the ternary diagram (Figure 4-1) due to limited cyclohexane available to reduce the composition of ethanol in the feed mixture. The mole composition of the extract sample for batch run 1 obtained from the gas chromatograph was 0.40 ethanol, 0.30 cyclohexane, and 0.30 water. This is shown as the pink point circled in

Figure 4-1. The extractant point lies within the immiscibility region in this figure. Thus, two liquid phases were present in the extract sample obtained. There were discrepancies associated with the results for the extract composition. Hence, it was decided to rather use the raffinate composition obtained from the GC analysis. A line was drawn from the raffinate composition through the incorrect extract composition, and extended to the extractant phase. This was then used to determine the desired extract composition. From Figure 4-1, it can be seen that for batch run 1 the measured number of stages obtained for the desired extract composition was 1 stage rounded up.

Using the same feed and conditions, though to reach a raffinate composition of 99% carrier, 2 theoretical stages would be required. The measured and predicted number of stages do not correspond as would be expected in an actual/real column operation. Inefficiencies in the separation lie in the column design, and tray design, while other experimental errors include sampling errors, insufficient time allowed for effective mixing and interaction to occur, which led to the splitting of phases and less ethanol extracted. The sample may have been left at room temperature for too long, thus causing disturbances in the sample and a change in composition. This is possible at higher temperature and low volatility of ethanol and acetone.

Due to time constraints, re-runs could not have been performed to improve and verify the results. The number of measured and predicted stages for batch run 2 were attained in the same manner as batch run 1. As seen in Figure F-1 for batch run 2, the extractant composition on a mole basis was 0.46 ethanol and 0.54 water. Thus, a greater mole composition of ethanol (0.46) was attained for batch run 2 as compared to batch run 1 (0.40). This was further supported by the percentage of ethanol extracted, as a greater percentage was obtained for batch run 2 compared to batch run 1 due to effective mixing and interaction of the components (discussed in Chapter 4.2.1). As seen in Figure F-1, the number of measured stages was determined to be 1 stage, while the theoretical or predicted stages to reach a 99% raffinate composition of cyclohexane was 2 stages. This is a similar result to batch run 1. It could have been that not enough time was allowed for effective mixing to occur and for the system to stabilize.



1 Stage → R

2 stages → 99%

Figure 4-1: Stepping off - Batch run 1

4.3 Semi-batch Operation

The second operation tested on the vibrating plate extraction column was semi-batch mode, where there was a recycle stream allowing chemicals to be fed back into the column via the bottom. The column operated at a temperature of approximately 25.40℃ with an uncertainty of ±0.05℃ and a pressure of 99.481 kPa with a full-scale uncertainty of ±0.025%. The solvent-to-feed ratio was altered to determine the effect on the extent of the separation in the column and the extractive efficiency. According to (Seader, et al., 2011), a higher solvent-to-feed ratio should result in a greater extent of separation.

4.3.1 Composition Profile

The composition profiles were plotted over a period of time for semi-batch runs 1 and 2 to show the trends of ethanol in the extract, raffinate, and feed. The composition profiles also determine if steady-state was established.

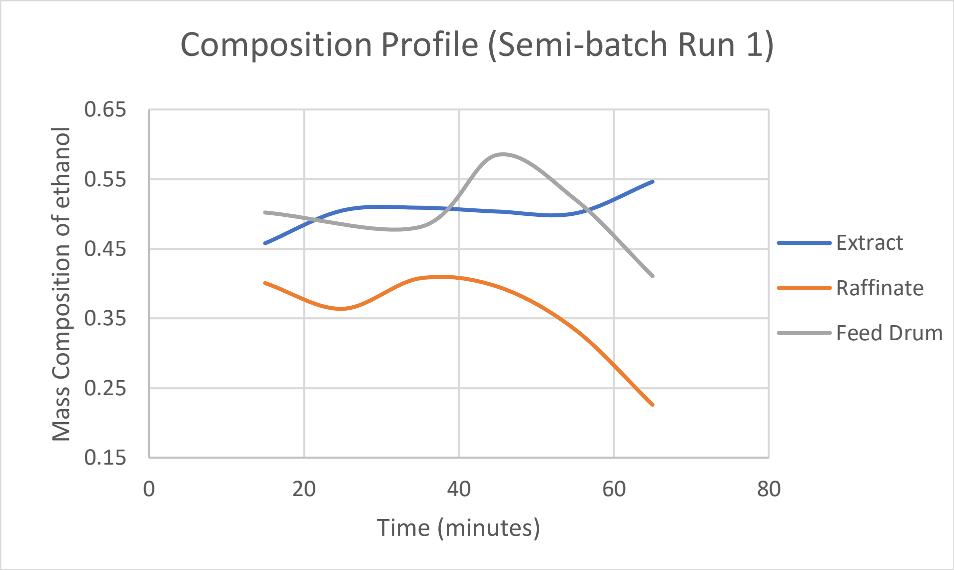


Figure 4-2: Composition Profile of Semi-batch Run 1

The ethanol composition in Figure 4-2 exhibits an increase in the extract over time, while the composition of ethanol in the raffinate and feed drum decreases. This confirmed that the experiment was performed accurately, as the trends correspond to literature. However, steady-state was not established since the composition of ethanol in the extract, raffinate, and feed did not remain constant over time. More time could be allowed for steady-state to be achieved. There was also some discrepancy associated with the sudden rise of ethanol in the feed drum at 45 minutes. This could be due to sampling errors, only a small amount of sample being obtained, and the feed drum not being well mixed.

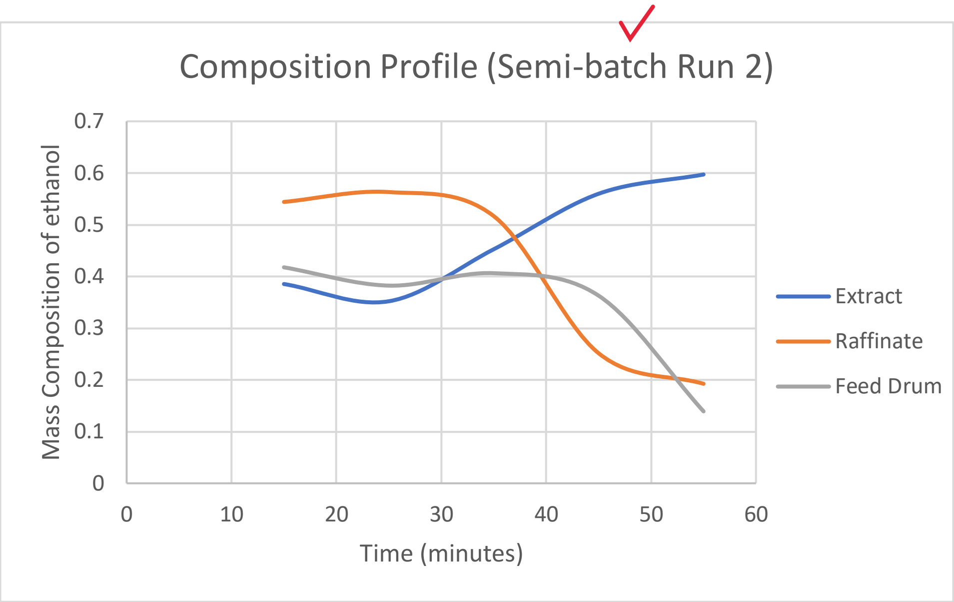


Figure 4-3: Composition Profile of Semi-batch Run 2

Figure 4-3 shows a similar trend to that of Figure 4-2 (semi-batch run 1). The composition of ethanol in the extract increases steadily, while the composition of ethanol in the raffinate and feed drum decreases. From Figure 4-2 although complete steady-state was not established, the composition of ethanol in semi-batch run 2 seemed to have reached a constant value towards the end of the time period unlike semi-batch run 1. However, both runs show the same trends in the composition of ethanol in the extract, raffinate, and feed drum.

4.3.2 Percentage of Ethanol Extracted

The percentage of ethanol extracted was determined using the number of moles of ethanol in the feed and the raffinate as presented in Equation 2-2. Refer to the Appendix D for detailed sample calculations relating to the calculation of the percentage of ethanol extracted for semi-batch run 1.

Table 4-3: Ethanol Extracted for Semi-batch runs 1 and 2

Operation	**Solvent-to-feed Ratio**	**Number of moles of Ethanol in the feed**	**Number of moles of Ethanol in the raffinate**	**Percentage of Ethanol extracted**
Semi-batch Run 1	0.254	138.054	26.696	80.663
Semi-batch Run 2	0.363	124.596	20.733	83.360

From Table 4-3, it is observed that the percentage of ethanol extracted increased from 80.663% to 83.360% as the solvent-to-feed ratios increased from run 1 to 2. A higher solvent-to-feed ratio in semi-batch run 2 resulted in a greater extent of separation, thus resulting in more ethanol being transferred from the feed mixture to water (solvent). This was due to more solvent (water) being available to extract ethanol (solute) from the feed mixture. (Naidoo, 2012) and (Rathilal, 2010) also deduced that a greater percentage of solute was extracted when the solvent-to-feed ratio was increased. Hence, the observations made in this research project corresponded to expended trends and previous experimental studies performed.

When comparing the results obtained for the semi-batch and batch runs, the percentage of ethanol extracted for the semi-batch mode was almost 20% greater than the batch mode, as indicated in Tables 4-2 and 4-3. The significant increase in the percentage of ethanol extracted for the semi-batch runs was a result of the overflow mixture being continuously fed through the bottom of the extraction column via the recycle stream. This allowed for more interaction and greater mixing between ethanol, cyclohexane, and water, which led to water extracting more ethanol from the feed (due to the higher selectivity of water towards ethanol as compared to cyclohexane). A greater degree of mass transfer occurred in the semi-batch runs due to the recycle stream.

4.3.3 Number of Predicted and Measured Stages

The number of theoretical stages were obtained by stepping off the ternary diagram for the ethanol-cyclohexane-water system. The feed composition (mole basis) of semi-batch run 1 consisted of 0.68 ethanol and 0.32 cyclohexane, which was located quite high on the ternary diagram (Figure F-2) due to the high ethanol composition in the feed. The feed composition for semi-batch run 2 was also quite high (Figure F-3). This was due to limited availability of cyclohexane to decrease the composition of ethanol in the feed mixture. The mole composition of the extract sample for semi-batch run 1 obtained from the gas chromatograph was 0.64 ethanol, 0.28 cyclohexane, and 0.08 water. In Figure F-2 (semi-batch run 1), the extractant point lies outside of the immiscibility region and the equilibrium curve. Thus, there were discrepancies associated with the results for the extract composition. Hence, a line was drawn from the raffinate composition obtained from the gas chromatograph through the mixing point to determine the desired extract composition (Figure F-2). From Figure F-2, it can be seen that for semi-batch run 1 the measured number of stages obtained for the desired extract composition was 1 stage rounded up. However, the predicted number of stages attained for semi-batch run 1 to reach a raffinate composition of 99% carrier was 2 stages. The measured and predicted number of stages were not equivalent. This could have been due to errors, such as sampling errors, not enough time allowed for effective mixing and interaction to occur.

The number of measured and predicted stages for semi-batch run 2 were attained in the same way as semi-batch run 1 which can be seen in Figure F-3. The extractant composition obtained from the gas chromatograph lies outside of the immiscibility region and the equilibrium curve. The raffinate composition obtained from the gas chromatograph lies within the immiscibility region. There were two liquid phases present in the raffinate sample due to the splitting of phases. Thus, there were errors associated with the results. This could have been due to not enough time allocated for efficient mixing and for steady-state to be established. The desired extractant composition was determined by drawing a line from the raffinate through the mixing point. This gave the desired extractant and raffinate composition. The number of measured stages was determined to be 1 stage, while the predicted stages was 2 stages just like semi-batch run 1. It could have been that not enough time was allowed for effective mixing to occur and for the system to stabilize.

4.4 Overview

The errors associated with the results, include sample withdrawal and sample analyses, as some samples contained two liquid phases. This means that inefficient mixing occurred. Other errors could have been disturbances in the samples due to evaporation, and not enough time for steady-state to be achieved in the semi-batch runs. Due to time constraints, the experimental runs could not be repeated in order to improve the results. However, the recommendations made suggest more data measurements to enhance the results.

5. Conclusion

The extraction column was re-commissioned successfully, with the column and auxiliary units modified and repaired for the operation in the two modes (batch and semi-batch).

The system ethanol-cyclohexane-water was used to perform extraction studies on the column. The calibration of the gas chromatograph detector was performed correctly and accurately, with uncertainties in the composition ranging from 0.281% to 6.544%.

The experimental runs on the extraction column were performed under batch and semi-batch mode. For the batch runs, it was observed that the percentage of ethanol extracted increased from 60.47% to 64.27% as the vibrational frequency increased from 10 to 15 Hz. For the semi-batch runs, a higher solvent-to-feed ratio resulted in a greater percentage of ethanol extracted from 80.663% to 83.360%.

The extraction column operated more efficiently under semi-batch mode, as a greater degree of separation was observed due to 20% more ethanol being extracted as compared to the batch mode.

A higher vibrational frequency and solvent-to-feed ratio led to a greater extent of separation and mass transfer.

The theoretical and measured number of stages were not equivalent due to disturbances in the samples and insufficient time allowed for mixing to occur and the system to reach equilibrium.

Improvements are proposed for future experimental runs to improve the extraction efficiency.

The VPE column was re-commissioned successfully and the performance was evaluated. The extraction column is deemed ready for use for the next phase of experiments with rare earth metal separation and recovery.

The aim and objectives of this research project were successively achieved, as the experimental runs under batch and semi-batch mode were performed on the vibrating plate extraction column.

6. Recommendations

The experiments should be repeated for both semi-batch and batch✓ modes to ensure reliability and repeatability of the results obtained. Due to time constraints this was not possible.

Additional experimental runs should be performed on the vibrating plate extraction column under batch mode, while varying the solvent-to-feed ratio to evaluate the effect on the extent of separation. This is based on the availability of chemicals.

Further experimental runs should be performed on the VPE column under semi-batch mode, while altering the vibrational frequency to determine the effect on the extent of the separation.

A greater period of time (1:30-2 h) should be allocated for the experimental runs when operating the column under the semi-batch mode to allow for steady state to be reached/achieved.

The use of continuous operation with both phases flowing counter currently is suggested to allow for greater interaction and improved degree of separation between ethanol, cyclohexane, and water. This can be compared to the batch and semi-batch modes of operation.

While acetone was used as the internal standard in this work, a different internal standard can be evaluated on the system. This is due to acetone being extremely volatile and evaporating easily in ambient conditions., which may have led to some inconsistencies.

Should it be deemed necessary, investigating the performance on the VPE column should be assessed with decreasing the tray spacing. This could ensure an increase✓ in mass transfer as recommended by Rathilal (Rathilal, 2010).

Literature reports a greater degree of mass transfer and separation for selected systems at higher temperatures. It is suggested that the performance on the VPE column can be assessed by altering the temperature of the extraction column.

References

1. Blench, T., 2017. *South Africa could play a key role in world rare earth supplies.* [Online] Available at: https://www.mining.com/web/south-africa-play-key-role-world-rare-earth-supplies/
2. EnviroServ Waste Management, 2020. *Environmental Legislation in South Africa.* [Online] Available at: https://www.enviroserv.co.za/about/legislative-framework [Accessed 24 March 2021].
3. Fair, J. & Humphrey, J., 1983. Liquid-Liquid Extraction Processes. *5th Industrial Energy Technology Conference* , Volume II, pp. 846-856.
4. Honeywell, 2014. *Safety Data Sheet.* [Online] Available at: https://us.vwr.com/assetsvc/asset/en_US/id/16131326/contents
5. interchim, 2019. *Liquid-liquid extraction: introducion to this sample preparation technique.* [Online] Available at: https://blog.interchim.com/liquid-liquid-extraction-lle-technique-sample-preparation/
6. Ives, M., 2013. *Boom in Mining Rare Earths Poses Mounting Toxic Risks.* [Online] Available at: https://e360.yale.edu/features/boom_in_mining_rare_earths_poses_mounting_toxic_risks
7. King, H. M., 2020. *REE - Rare Earth Elements and their Uses.* [Online] Available at: https://geology.com/articles/rare-earth-elements/
8. LabChem, 2009. *Material Safety Data Sheet Water, Deionized.* [Online] Available at: https://www.fieldenvironmental.com/assets/files/MSDS%20Sheets/MSDS%20Sheets%202012/3S-Water-Deionized.pdf
9. LabChem, 2018. *Acetone Safety Data Sheet.* [Online] Available at: https://www.labchem.com/tools/msds/msds/LC10420.pdf
10. Lo, T. C., Baird, M. & Rama Rao, N., 1992. The Reciprocating Plate Column - Development and Applications. *Chemical Engineering Communications,* Volume 116, pp. 67-88.
11. Lu, C., 2021. *Why Rare Earths Are the Key to Just About Everything.* [Online] Available at: https://foreignpolicy.com/2021/04/22/rare-earths-china-us-greenland-geopolitics/
12. McLeod, C., 2019. *Top Rare Earth Reserves by Country.* [Online] Available at: https://investingnews.com/daily/resource-investing/critical-metals-investing/rare-earth-investing/rare-earth-reserves-country/
13. Moriyoshi, T., Uosaki, Y., Yamakawa, T. & Takahashi, K., 1991. (Liquid+liquid) equilibria of (water+ethanol+cyclohexane) at the temperatures 298.15K and 323,15K. *The Journal of Chemical Thermodynamics*, 23(1), pp. 37-42.
14. Naidoo, P., 2012. *Investigation of the effects of the process and equipment parameters on the separation efficiency of a vibrating plate extractor,* University of KwaZulu-Natal, Durban, South Africa: MSc. Eng. Thesis, School of Engineering.
15. Nickolas, S., 2021. *What Do Correlation Coefficients Positive, Negative, and Zero Mean?.* [Online] Available at: https://www.investopedia.com/ask/answers/032515/what-does-it-mean-if-correlation-coefficient-positive-negative-or-zero.asp
16. Prochazka, J., Landau, J., Souhrada, F. & Heyberger, A., 1971. Reciprocating plate extraction column. *Britich Chemical Engineering,* 16(1), pp. 42-44.

17. Rama Rao, N. V., Vijayan, S. & Baird, M., 1991. Hydrodynamics of a Vibrating Perforated Plate Extraction Column. *The Canadian Journal of Chemical Engineering,* Volume 69, pp. 212-221.
18. Rathilal, S., 2010. *'Modeling of a vibrating plate extraction column', PhD thesis, School of Engineering, University of KwaZulu-Natal,* Durban, South Africa: s.n.
19. Schmidt, A. & Strube, J., 2018. Application and Fundamentals of Liquid-Liquid Extraction Processes: Purification of Biologicals, Botanicals, and Strategic Metals.. *Kirk-Othmer Encyclopedia of Chemical Technology,* pp. 1-52.
20. Seader, J. D., Henley, E. J. & Roper, D. K., 2011. *Separation Process Principles: chemical and biochemical operations.* 3rd Edition ed. United States of America: John Wiley & Sons, Inc..
21. Sigma-Aldrich, 2020. *Safety Data Sheet.* [Online] Available at: https://www.sigmaaldrich.com/MSDS/retriever.do?country=US&language=EN&productNumber=459836&brand=SIAL
22. Takacs, I., Beszedics, G., Fabry, G. & Rudolf, P., 1993. *Apparatus to contact liquids of different density.* United States of America, Patent No. 5194152.
23. Vaccari, M. et al., 2019. *WEEE Treatment in Developing Countries: Environmental Pollution and Health Consequences.* [Online] Available at: https://www.ncbi.nlm.nih.gov/pmc/articles/PMC6539380/
24. Vaclav, G. & Carsky, M., 2020. New technology for lanthanide recovery from spent Nd-Fe-B magnets. *South African Journal of Chemical Engineering,* Volume 33, pp. 35-38.
25. Williams-Wynn, M., Naidoo, P. & Ramjugernath, D., 2020. The distribution coefficients of Y3+ and Eu3+ between HNO3 and HDEHP. *Minerals Engineering,* 153(106285).

Appendix

Appendix A: Raw Data

Table A-1: Feed masses and volumes - Batch runs 1 & 2

Component	Mass (kg)	Volume (l)
Ethanol	2.92	3.71
Cyclohexane	3.04	3.91
Water	4.17	4.19

Table A-2: Acetone masses and GC peak areas - Batch runs 1 & 2 ✓

	Sample	Mass of the mixture without acetone (g)	Mass of acetone added (g)	Area of ethanol	Area of water	Area of cyclohexane	Area of acetone
Batch Run 1	Feed	10.8611	2.0204	393156	78446.6	262417.4	241189.4
	Extract	12.0792	2.098	846343	198393.6	0	278852.6
	Raffinate	7.9976	2.0016	106991	3362	✓ 694042.2	169573.5
Batch Run 2	Extract	12.0972	2.098	315214.6	174937.5	0	195308.5
	Raffinate	7.9776	2.0026	98260.8	0	665376.2	204063.6

Table A-3: Feed masses and volumes - Semi-batch run 1

Component	Mass (kg)	Volume (l)
Ethanol	6.36	8.06
Cyclohexane	5.42	6.96
Water	2.991	3

Table A-4: Acetone masses and GC peak areas - Semi-batch run 1

	Time (minutes)	Mass of the mixture without acetone (g)	Mass of acetone added (g)	Area of ethanol	Area of water	Area of cyclohexane	Area of acetone
Feed Drum	15	0.3414	0.1124	460076.7	28019.5	172488	323773.4
	35	3.7288	1.0037	405112.6	25877.4	263492	242820.2
	45	0.3754	0.1184	463674.8	19944.6	216810.9	267957.9
	55	0.8252	0.3045	390960.5	15532.2	251758.1	296702.2
	65	1.0994	0.503	325882.2	13724.2	209766.2	388990.9
Extract	15	8.0283	2.0856	371880.2	76776.3	299475.8	226120.9
	25	3.8748	1.0021	393216.9	16503.7	324258.4	215805.7
	35	4.3786	1.0076	404664.5	25773.2	332376.8	196093
	45	6.1587	1.0253	414614	26289.1	328123.3	146941.9
	55	3.0571	0.7956	422469.5	27119.3	288996.8	235284.8
	65	6.0525	1.0155	461646.9	27060.1	313278.7	151944
Raffinate	15	9.542	2.507	299330.2	18249.2	299330.2	205388.7
	25	7.9417	1.507	311614.6	13377.5	311614.6	170094.2
	35	1.37	0.5018	310164.5	12436.1	245359.7	291628
	45	4.4648	1.0045	335070.6	11783.5	310164.5	199393.8
	55	5.2866	1.0073	285182.4	16322.5	335070.6	170242.3
	65	3.162	1.0106	250028.5	19335.6	285182.4	370050.3

Table A-5: Feed masses and volumes - Semi-batch run 2

Component	Mass (kg)	Volume (l)
Ethanol	5.74	7.27
Cyclohexane	5.29	6.79
Water	4.00	4.01

Table A-6: Acetone masses and GC peak areas - Semi-batch run 2

	Time (minutes)	Mass of the mixture without acetone (g)	Mass of acetone added (g)	Area of ethanol	Area of water	Area of cyclohexane	Area of acetone
Feed Drum	15	1.7762	0.4032	232808.9	28955.4	621198.2	135597
	25	2.5514	1.0065	183494	18702.9	572757.3	198132
	35	3.8953	1.0485	395709.9	475321.9	16792.3	280977.8
	45	1.6295	0.5134	258485	9347.7	624644.1	240586.2
	55	6.3056	2.693	101534.4	73052.4	391387.1	326180
Extract	15	5.8187	4.8132	209280.2	102929.7	187957.9	481417.5
	25	7.2829	3.5577	299111.8	131904	139736.1	444842
	35	2.2918	1.451	263729.6	121665	165225.7	395384.9
	45	5.722	0.9734	563686.5	271832.3	63317.6	183536.9
	55	5.1192	1.0097	573045.8	70930.3	33060.2	202835
Raffinate	15	7.8076	4.8057	341733.7	125124.5	113142	404432.7
	25	3.3766	1.0127	524086.6	216555.6	33279.1	292022.7
	35	6.1833	3.6283	343580.9	140037.6	142589.4	409575.9
	45	2.8897	1.0254	207972.7	207972.7	43742.2	307008.5
	55	7.7332	4.8081	78863.7	136217	918863.7	401824.2

Appendix B: Gas Chromatograph Detector Calibration Results

Table B-1: GC peak areas and errors for ethanol-acetone (acetone-rich region)

	Ethanol	**Acetone**					
Specific Mole Ratio	**A1**	**A2**	**A1/A2**	**Standard deviation**	**Average**	**Error (Standard deviation/Average)**	**% Error**
0.10	81309	913514	0.089				
	78614.7	886029.9	0.089	0.00014	0.089	0.002	0.157
0.50	286630.1	708347.7	0.405				
	267599.8	680872.5	0.3930	0.0058	0.3988	0.0146	1.4569
0.90	393527.2	572550.3	0.6873				
	402785.1	591435.5	0.6810	0.0031	0.6842	0.0046	0.4599

Table B-2: GC peak areas and errors for ethanol-acetone (acetone-dilute region)

	Ethanol	**Acetone**					
Specific Mole Ratio	**A1**	**A2**	**A2/A1**	**Standard deviation**	**Average**	**Error (Standard deviation/Average)**	**% Error**
0.10	831687.9	133408	0.1604				
	867635.6	139464.9	0.1607	0.0002	0.1606	0.0010	0.1043
0.50	604096.5	399088.6	0.6606				
	593446.4	396187.7	0.6676	0.0035	0.6641	0.0052	0.5246
0.90	434648.8	522099.3	1.2012				
	453512.4	542417.2	1.1960	0.0026	1.1986	0.0022	0.2153

Table B-3: GC peak areas and errors for water-acetone (acetone-rich region)

	Water	Acetone					
Specific Mole Ratio	**A2**	**A4**	**A2/A4**	**Standard deviation**	**Average**	**Error (Standard deviation/Average)**	**% Error**
0.10	45536.5	919134.7	0.0495				
	45815.9	953718.4	0.0480	0.0008	0.0488	0.0154	1.5408
0.50	145864.9	873322.7	0.1670				
	144217.1	877633.4	0.1643	0.0013	0.1657	0.0081	0.8142
0.90	252552.2	812272	0.3109				
	255219.3	797808.1	0.3199	0.0045	0.3154	0.0142	1.4235

Table B-4: GC peak areas and errors for water-acetone (acetone-dilute region)

	Water	Acetone					
Specific Mole Ratio	**A2**	**A4**	**A4/A2**	**Standard deviation**	**Average**	**Error (Standard deviation/Average)**	**% Error**
0.10	916268.1	303374.3	0.331				
	994001.9	301598.7	0.303	0.014	0.317	0.044	4.362
0.50	504154.2	683547.5	1.356				
	470804.3	669493.3	1.422	0.033	1.389	0.024	2.383
0.90	320804.5	800817.9	2.496				
	334870	808590.8	2.415	0.041	2.455	0.017	1.662

Table B-5: GC peak areas and errors for acetone-cyclohexane (acetone-rich region)

	Acetone	**Cyclohexane**					
Specific Mole Ratio	**A4**	**A3**	**A3/A4**	**Standard deviation**	**Average**	**Error (Standard deviation/Average)**	**% Error**
0.10	91210.8	12632.7	0.1385				
	91862.4	11556.8	0.1258	0.0063	0.1322	0.0480	4.8030
0.50	251782.4	121802.5	0.4838				
	255329.8	125203.5	0.4904	0.0033	0.4871	0.0068	0.6774
0.90	485291.6	491502.8	1.0128				
	446943.6	410607.7	0.9187	0.0470	0.9658	0.0487	4.8717

Table B-6: GC peak areas and errors for acetone-cyclohexane (acetone-dilute region)

	Acetone	**Cyclohexane**					
Specific Mole Ratio	**A4**	**A3**	**A4/A3**	**Standard deviation**	**Average**	**Error (Standard deviation/Average)**	**% Error**
0.10	11049.6	87110.3	0.1268				
	13235.3	93582.1	0.1414	0.0073	0.1341	0.0544	5.4361
0.50	251782.4	634479.9	0.3968				
	398149.8	796802.2	0.4997	0.0514	0.4483	0.1147	11.4724
0.90	372686.6	487167.8	0.76501				
	354460.6	438783.7	0.80783	0.02141	0.78642	0.02722	2.72240

Appendix C: Calibration Plots

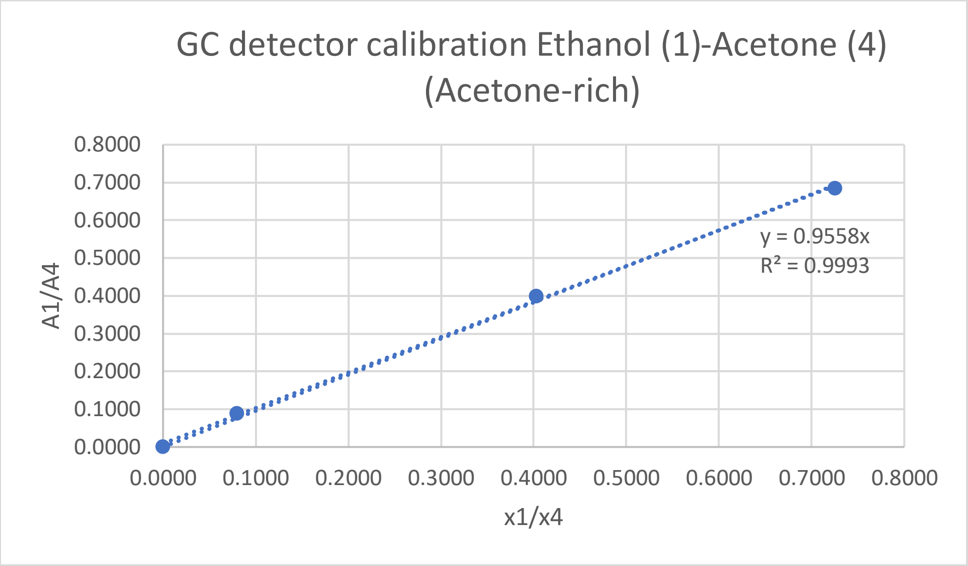


Figure C-1: GC Calibration Curve of Ethanol-Acetone (Acetone-rich)

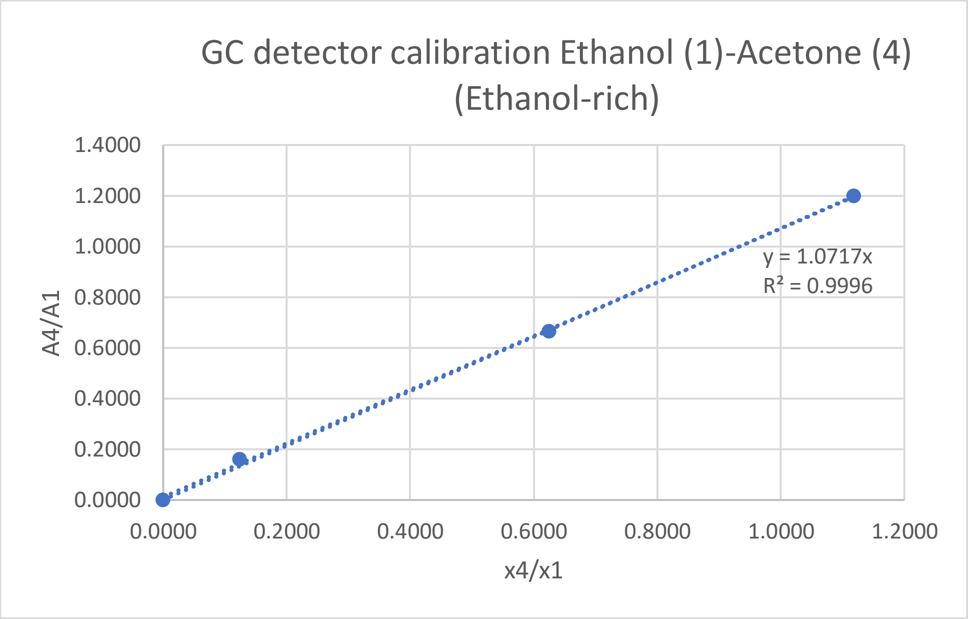


Figure C-2: GC Calibration Curve of Ethanol-Acetone (Ethanol-rich)

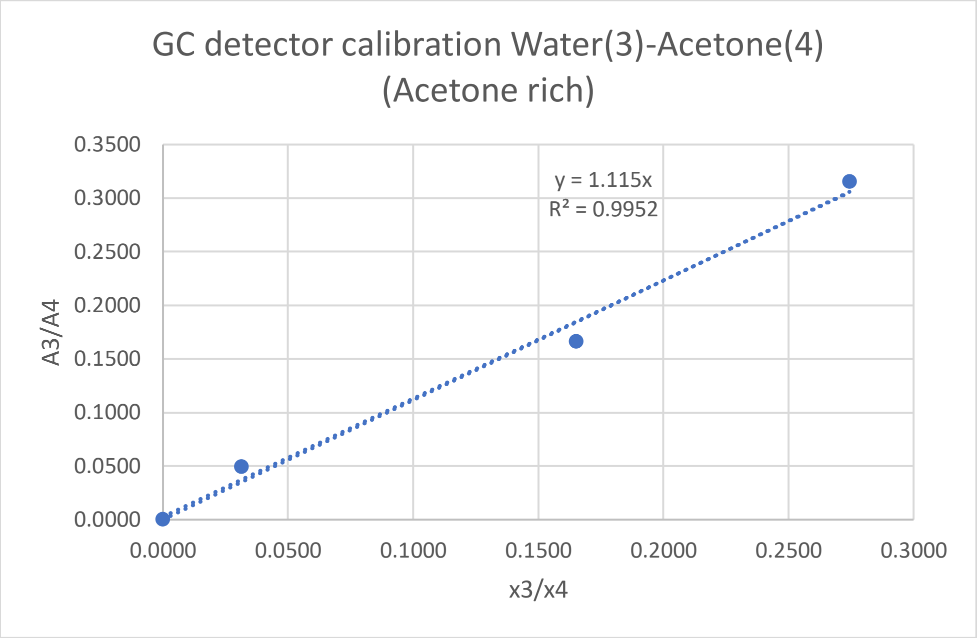
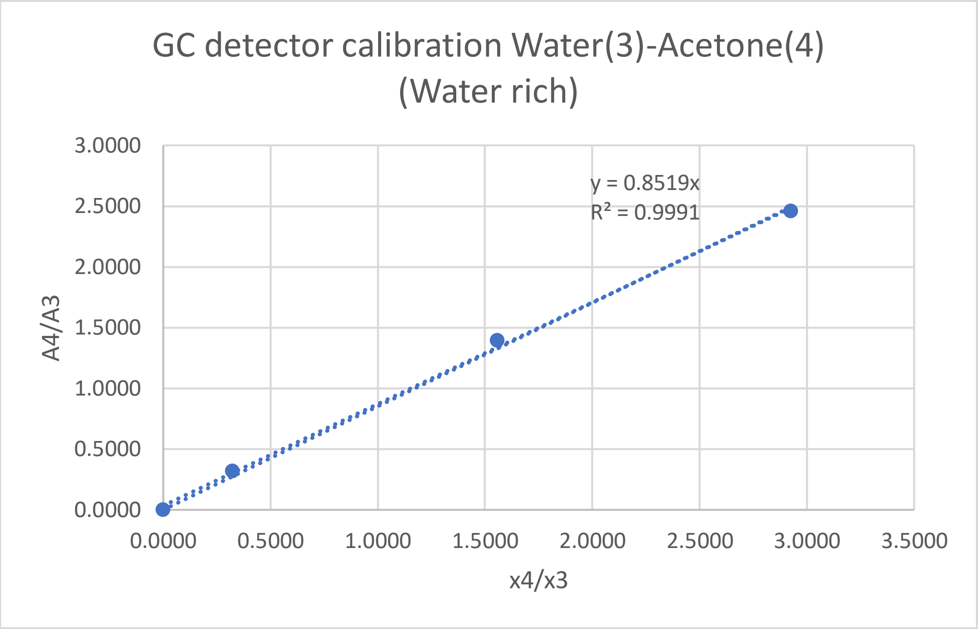


Figure C-3: GC Calibration Curve of Water-Acetone (Acetone-rich)



✓

Figure C-4: GC Calibration Curve of Water-Acetone (Water-rich)

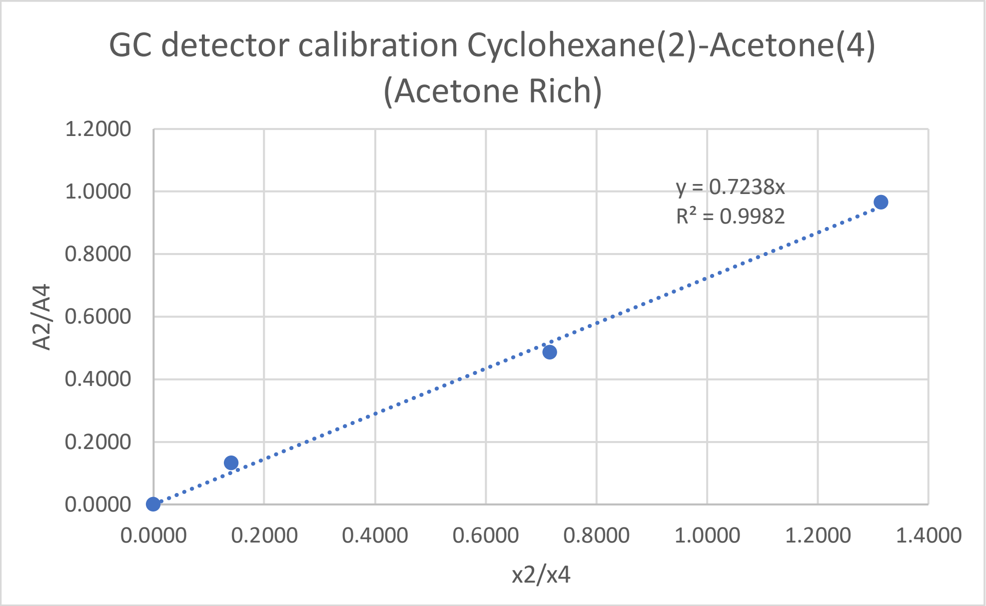
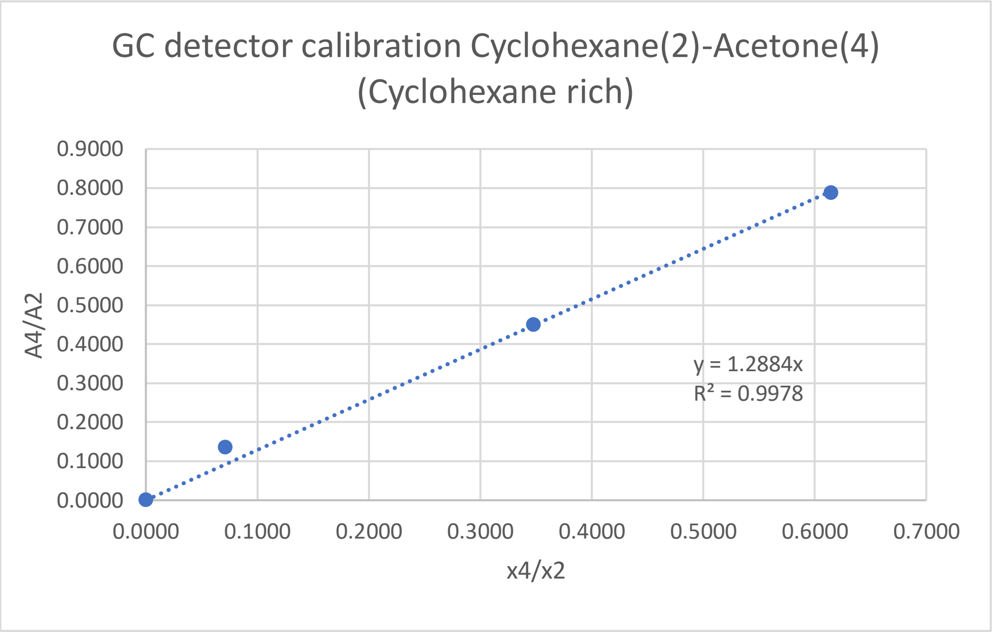


Figure C-5: GC Calibration Curve of Cyclohexane-Acetone (Acetone-rich)



✓

Figure C-6: GC Calibration Curve of Cyclohexane-Acetone (Cyclohexane-rich)

Appendix D: Sample Calculations – Semi-batch Run 1

The sample calculations presented below were for the semi-batch run 1 – Raffinate only as the same calculation procedure was employed for the extract.

As shown in Table A-3, the feed mixture consisted of 6.36 kg of ethanol and 5.42 kg of cyclohexane. The solvent consisted of 2.99 kg of water. The number of moles were calculated as follows (moles = mass/molar mass):

$$n_{ethanol} = \frac{m_{ethanol}}{M_{ethanol}} = \frac{6.36 \times 1000}{46.069} = 138.054\ moles$$

$$n_{cyclohexane} = \frac{m_{cyclohexane}}{M_{cyclohexane}} = \frac{5.42 \times 1000}{84.16} = 64.401\ moles$$

$$n_{water} = \frac{m_{water}}{M_{water}} = \frac{2.99 \times 1000}{18.02} = 165.93\ moles$$

Since the feed mixture consisted of ethanol and cyclohexane only, the **composition of the feed mixture** was now calculated on a mole basis:

$$x_{ethanol} = \frac{n_{ethanol}}{n_{ethanol} + n_{cyclohexane}} = \frac{138.054}{138.054 + 64.401} = 0.682$$

$$x_{cyclohexane} = 1 - x_{ethanol} = 1 - 0.682 = 0.318$$

The **mixing point** for semi-batch run 1 was now calculated using the moles of all three components (ethanol, cyclohexane, and water): ✓

$$x_{ethanol} = \frac{n_{ethanol}}{n_{ethanol} + n_{cyclohexane} + n_{water}} = \frac{138.054}{138.054 + 64.401 + 165.93} = 0.38$$

$$x_{cyclohexane} = \frac{n_{cyclohexane}}{n_{ethanol} + n_{cyclohexane} + n_{water}} = \frac{64.401}{138.054 + 64.401 + 165.93} = 0.18$$

$$x_{water} = \frac{n_{water}}{n_{ethanol} + n_{cyclohexane} + n_{water}} = \frac{165.98}{138.054 + 64.401 + 165.93} = 0.45$$ ✓

The mixing point was used on the ternary diagram as seen in Figures 4-1 and F-1 to F-3, where the mixing point was labelled MP.

From Table A-4, the **final raffinate** was taken at 65 minutes assuming that steady state was established. The area of ethanol was 250028.5 (A_1), and the area of acetone was 370050.3 (A_4). Since this was in the acetone-rich region, the calibration plot of ethanol-acetone in the acetone-rich region was used to determine the composition of ethanol in the raffinate (top). The area ratios were calculated, and the mass fraction ratio was obtained using the calibration plot shown in Figure C-1.

$$\frac{A_1}{A_4} = \frac{250028.5}{370050.3} = 0.676$$

The gradient of the calibration plot for ethanol-acetone in the acetone-rich region was 0.9558. Thus, the mass fraction ratio was easily calculated. Note that $\frac{A_1}{A_4} = y$ (area ratios) and $\frac{x_1}{x_4} = x$ (mass fraction ratios) in the straight-line equation shown below.

$$y = 0.9558x$$

$$0.676 = 0.9558x \;\therefore\; x = 0.707$$

Since the amount of acetone added is known, the composition of ethanol in the mixture containing ethanol, cyclohexane, water, and acetone can be determined.

Composition of acetone: $x_4 = \frac{Mass\ of\ Acetone\ added}{Mass\ of\ mixture + Mass\ of\ acetone\ added} = \frac{1.0106}{3.162+1.0106} = 0.242$

$$x_1 = x_4 \times \frac{x_1}{x_4} = 0.242 \times 0.707 = 0.171$$

The ethanol composition above was the composition after the addition of acetone. Thus, the composition of ethanol in the original mixture needed to be back calculated. The mass of ethanol in the original mixture can be determined as follows:

$$Mass\ of\ ethanol = x_1 \times m_{mixture\ with\ acetone} = 0.171 \times 4.1726 = 0.714\ g$$

✓

The composition of ethanol on a mass basis was calculated as follows:

$$x_{1(original)} = \frac{Mass\ of\ ethanol}{Mass\ of\ mixture\ without\ acetone} = \frac{0.714}{3.162} = 0.226$$

The moles of each of the components can be determined as follows:

$$n_{ethanol} = \frac{m_{ethanol}}{M_{ethanol}} = \frac{0.714}{46.069} = 0.0155\ moles$$

Finally, the mole composition of ethanol in the original mixture was now calculated:

$$x_{ethanol(original)} = \frac{n_{ethanol}}{n_{ethanol} + n_{cyclohexane} + n_{water}} = \frac{0.0155}{0.0155 + 0.0285 + 0.00262} = 0.332$$

The composition of water was then calculated using the relevant calibration plot, masses, and areas. The area of water was 19335.6 (A_2), and the area of acetone was 370050.3 (A_4). Since this was in the acetone-rich region, the calibration plot of water-acetone in the acetone-rich region (Figure C-3) was used to determine the composition of water in the raffinate (top). The area ratios were calculated, and the mass fraction ratio was obtained using the calibration plot equation shown in Figure C-3.

$$\frac{A_2}{A_4} = \frac{19335.6}{370050.3} = 0.052$$

The gradient of the calibration plot for water-acetone in the acetone-rich region was 1.115. Thus, the mass fraction ratio was easily calculated. Note that $\frac{A_2}{A_4} = y$ (area ratios) and $\frac{x_2}{x_4} = x$ (mass fraction ratios) in the straight-line equation shown below.

$$y = 1.115x$$

$$0.052 = 1.115x \ \therefore x = 0.047$$

Since the amount of acetone added is known, the composition of water in the mixture containing ethanol, cyclohexane, water, and acetone can be determined.

Composition of acetone: $x_4 = \frac{Mass\ of\ Acetone\ added}{Mass\ of\ mixture + Mass\ of\ acetone\ added} = \frac{1.0106}{3.162 + 1.0106} = 0.242$

$$x_2 = x_4 \times \frac{x_2}{x_4} = 0.242 \times 0.047 = 0.011$$

The water composition above was the composition after the addition of acetone. Thus, the composition of water in the original mixture needed to be back calculated. The mass of water in the original mixture can be determined as follows:

$$Mass\ of\ water = \ x_2 \times m_{mixture\ with\ acetone} = 0.011\ \times 4.1726 = 0.0474\ g$$

The composition of water on a mass basis was calculated as follows:

$$x_{2(original)} = \frac{Mass\ of\ water}{Mass\ of\ mixture\ without\ acetone} = \frac{0.0474}{3.162} = 0.015$$

The moles of each of the components can be determined as follows:

$$n_{water} = \frac{m_{water}}{M_{water}} = \frac{0.015}{18.02} = 0.00262\, moles$$

Since there is a **high error** associated with using the **cyclohexane-acetone** calibration plots, the composition of cyclohexane on a mass basis (in the ethanol, cyclohexane, water, and acetone mixture) can be determined by using 1 subtracted by the mass compositions of the rest of the components. Thus:

$$x_3 = 1 - (x_1 + x_2 + x_4) = 1 - (0.171 + +0.011 + 0.242) = 0.575$$

The mass of cyclohexane in the original mixture can be calculated as follows:

$$Mass\ of\ cyclohexane = \ x_3 \times m_{mixture\ with\ acetone} = 0.575\ \times 4.1726 = 2.40\ g$$

The composition of cyclohexane on a mass basis was calculated as follows:

$$x_{3(original)} = \frac{Mass\ of\ cyclohexane}{Mass\ of\ mixture\ without\ acetone} = \frac{2.40}{3.162} = 0.759$$

The moles of each of the components can be determined as follows:

$$n_{cyclohexane} = \frac{m_{cyclohexane}}{M_{cyclohexane}} = \frac{2.40}{84.16} = 0.0285\, moles$$

Finally, the **mole composition** of each of the components in the original mixture was then calculated:

$$x_{ethanol(original)} = \frac{n_{ethanol}}{n_{ethanol} + n_{cyclohexane} + n_{water}} = \frac{0.0155}{0.0155 + 0.0285 + 0.00262} = 0.332$$

$$x_{water(original)} = \frac{n_{ethanol}}{n_{ethanol} + n_{cyclohexane} + n_{water}} = \frac{0.00262}{0.0155 + 0.0285 + 0.00262} = 0.056$$

$$x_{cyclohexane(original)} = 1 - (0.332 + 0.056) = 0.611$$

The composition for each of the components in the extract (ethanol, cyclohexane, and water) can be calculated using the same calculation procedure. The results were shown in Tables D-1 and D-2.

Table D-1: Extract and raffinate compositions on a mass basis - Semi-batch run 1

Component	Extract Composition (mass basis)	Raffinate Composition (mass basis)
Ethanol	0.546	0.226
Water	0.027	0.015 ✓
Cyclohexane	0.427	0.76

Table D-2: Extract and raffinate compositions on a mole basis - Semi-batch run 1

Component	Extract Composition (mole basis)	Raffinate Composition (mole basis)
Ethanol	0.644	0.332
Water	0.081	0.056
Cyclohexane	0.275	0.611

The extract and raffinate compositions on a mole basis were presented in Tables E-1 to E-3 for the rest of the experimental runs. The mole compositions of the feed, extract, and raffinate were used to determine the number of stages on the ternary diagram. The stepping off the ternary diagram was shown in Figures 4-1, and Figures F-1 to F-3.

Using the overall mass balance and the ethanol balance (Equations 2-1 and 2-2), the number of actual moles in the raffinate and extract were now calculated to evaluate the extent of separation.

$$F + S = E + R$$

$$11.78 + 2.99 = E + R \;\; \therefore E = 14.77 - R \;\ldots\ldots\; \mathbf{1}$$

$$Fx_{ethanol(F)} = Ex_{ethanol(E)} + Rx_{ethanol(R)}$$

$$11.78(0.682) = (14.77 - R)(0.546) + R(0.226) \;\ldots\ldots\; \mathbf{2}$$

1 and **2** can be solved simultaneously to determine the amount of extract and raffinate in kilograms.

The raffinate was calculated to be 5.44 kg, while the extract was 9.33 kg. The number of moles of ethanol in the raffinate was then determined as follows:

$$n_{ethanol(R)} = \frac{Raffinate\ Mass\ \times x_{ethanol(R)}}{M_{ethanol}} = \frac{(5.44 \times 1000) \times 0.226}{46.07} = 26.686\ moles$$

Note that the composition of ethanol is on a mass basis in the equation above.

✓

The percentage of ethanol extracted was then calculated using Equation 2-3. The number of moles of ethanol in the feed as calculated at the beginning of the sample calculations was 138.054.

$$\%\ ethanol\ extracted = \frac{n_{ethanol(F)} - n_{ethanol(R)}}{n_{ethanol(F)}} \times 100 = \frac{138.054-26.686}{138.054} \times 100 = 80.67\%$$

This calculation procedure was repeated for the batch runs and semi-batch run 2 using the relevant calibration plots, masses, and GC peak area ratios. The results for the percentage of ethanol extracted were presented in Chapter 4. ✓

Appendix E: Additional Results

Table E-1: Extract and raffinate compositions on a mole basis - Batch run 1

Component	**Extract Composition (mole basis)**	**Raffinate Composition (mole basis)**
Ethanol	0.390	0.261
Water	0.303	0.021
Cyclohexane	0.307	0.718

Table E-2: Extract and raffinate compositions on a mole basis - Batch run 2

Component	**Extract Composition (mole basis)**	**Raffinate Composition (mole basis)**
Ethanol	0.457	0.209
Water	0.543	0
Cyclohexane	0	0.791

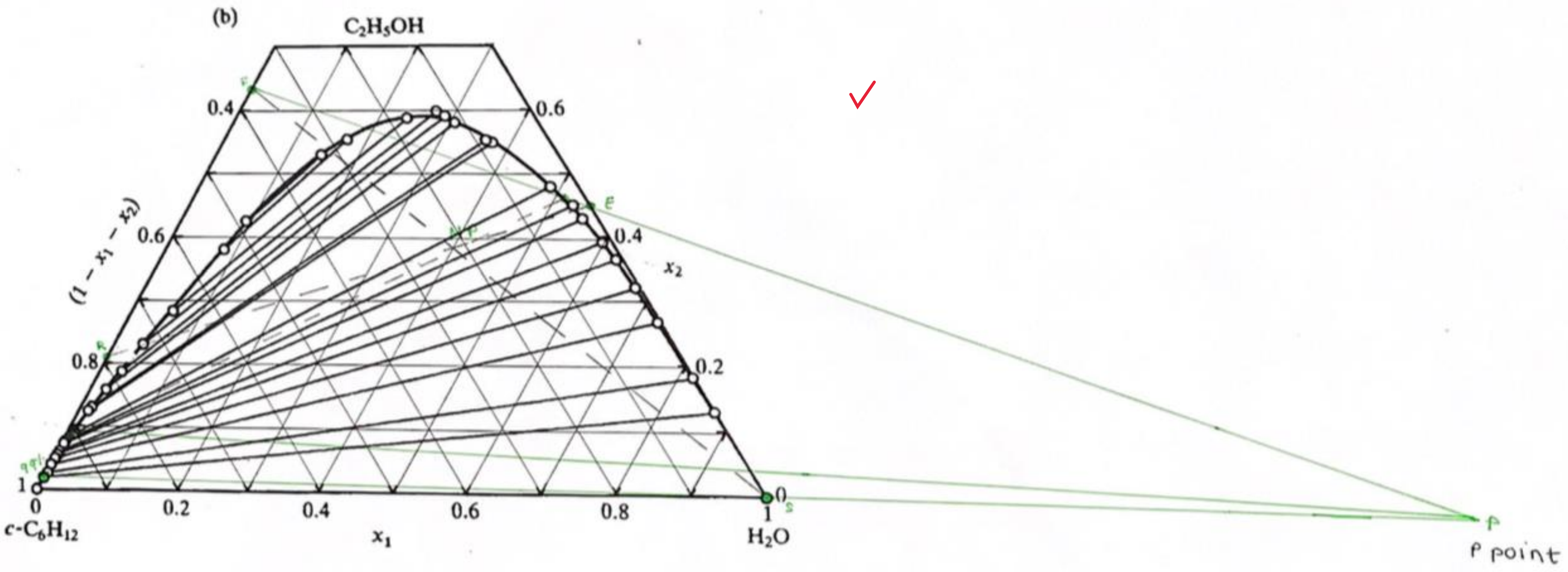
Table E-3: Extract and raffinate compositions on a mole basis - Semi-batch run 2

Component	**Extract Composition (mole basis)**	**Raffinate Composition (mole basis)**
Ethanol	0.634	0.272
Water	0.168	0.132
Cyclohexane	0.198	0.60

✓

Appendix F: Stepping Off

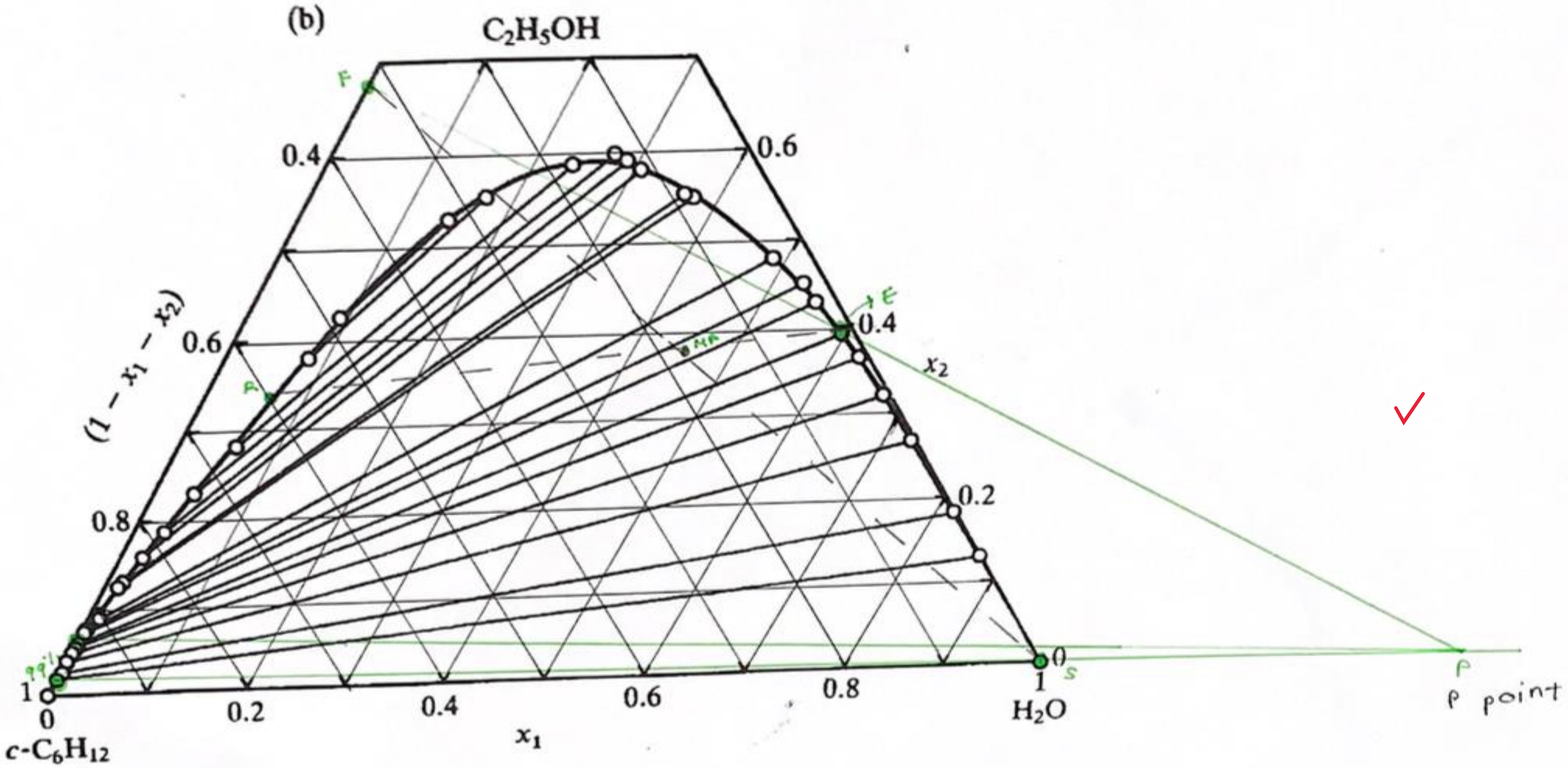
✓



1 stage → R

2 stages → 99%

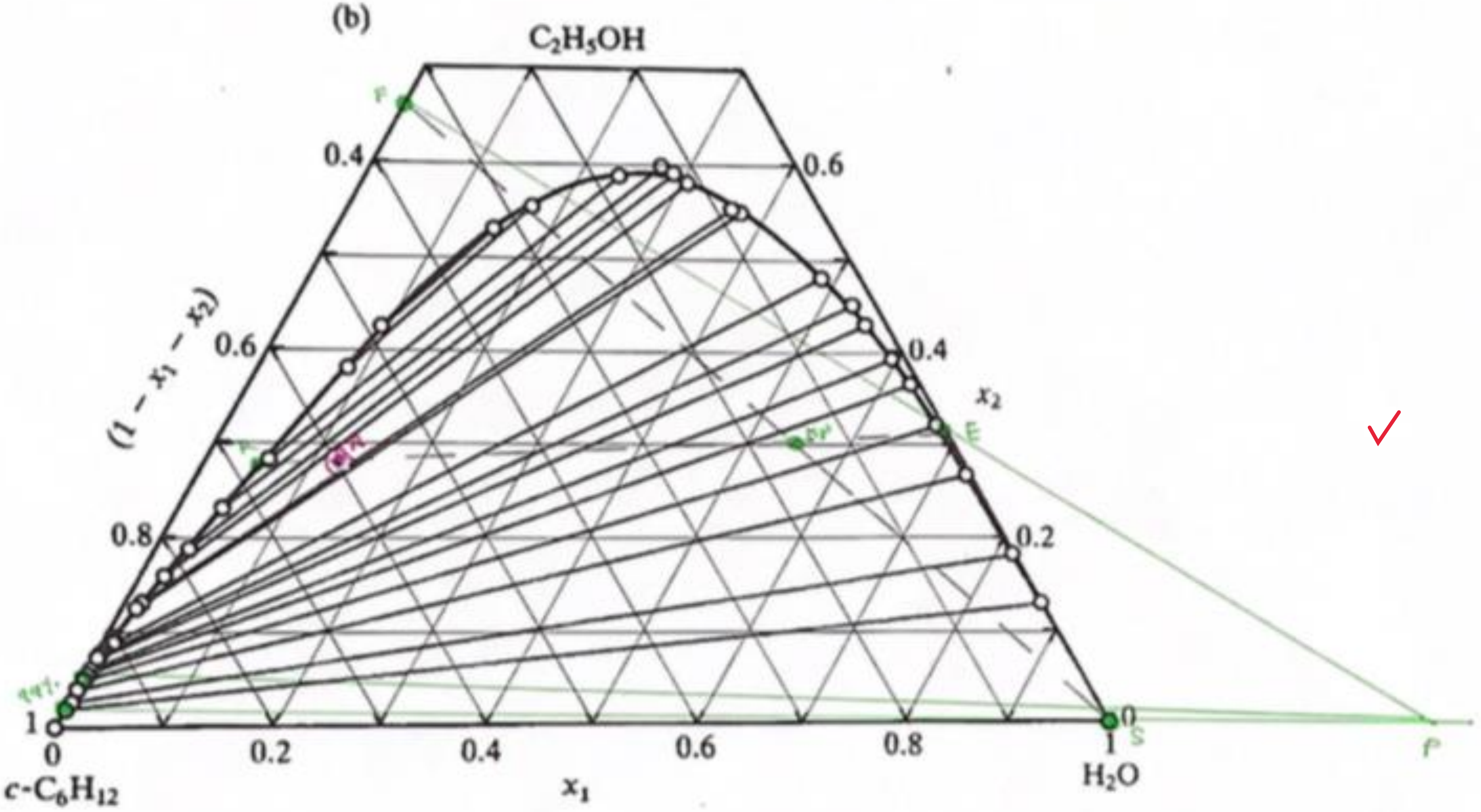
Figure F-1: Stepping off – Batch run 1



1 stage → R

2 stages → 99%

Figure F-2: Stepping off – Semi-batch run 1



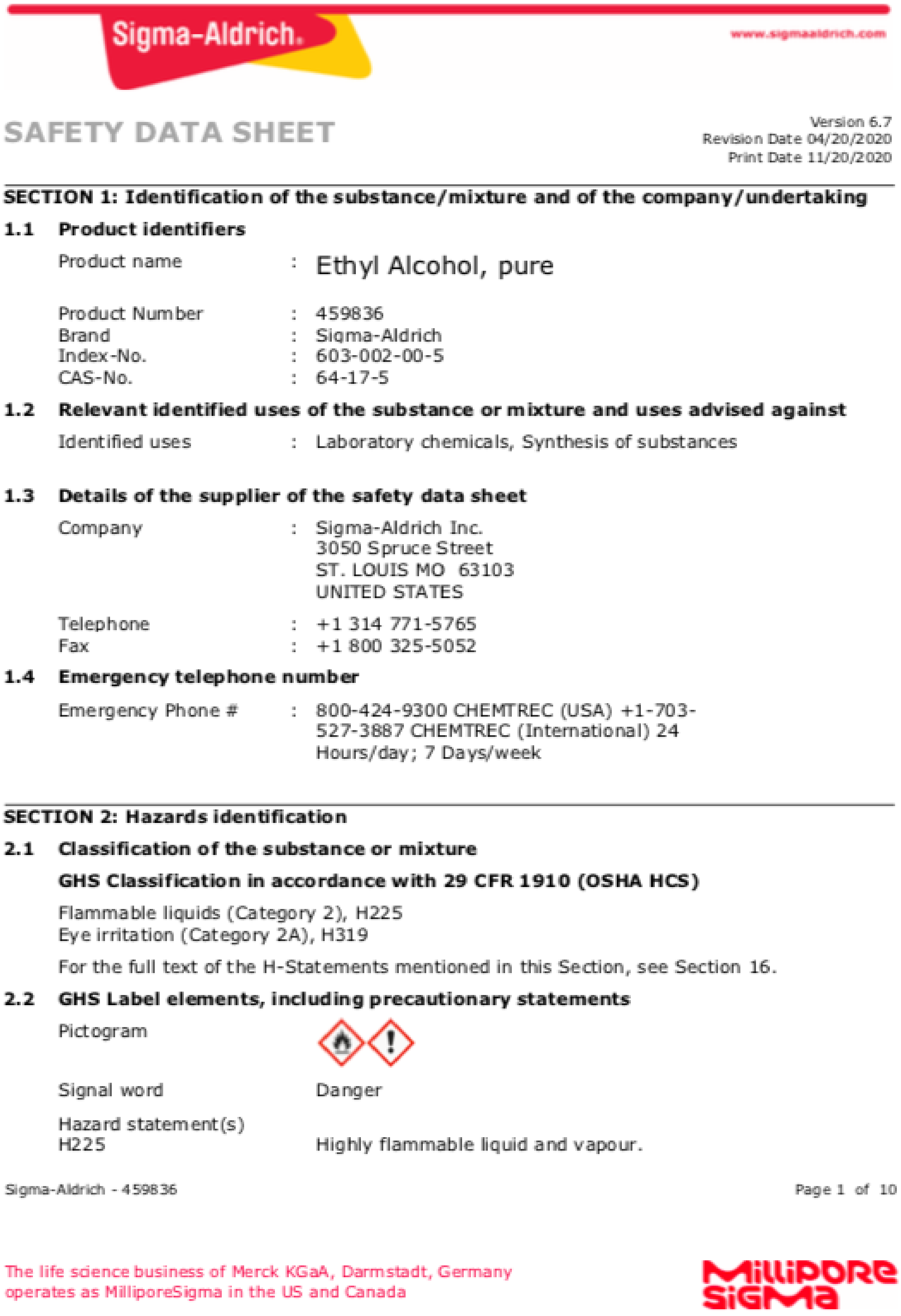
1 stage → R

2 stages → 99%

✓

Figure F-3: Stepping off – Semi-batch run 2

Appendix G: Material Safety Data Sheets



Sigma-Aldrich.

www.sigmaaldrich.com

SAFETY DATA SHEET

Version 6.7
Revision Date 04/20/2020
Print Date 11/20/2020

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifiers

Product name : Ethyl Alcohol, pure

Product Number : 459836
Brand : Sigma-Aldrich
Index-No. : 603-002-00-5
CAS-No. : 64-17-5

1.2 Relevant identified uses of the substance or mixture and uses advised against

Identified uses : Laboratory chemicals, Synthesis of substances

1.3 Details of the supplier of the safety data sheet

Company : Sigma-Aldrich Inc.
3050 Spruce Street
ST. LOUIS MO 63103
UNITED STATES

Telephone : +1 314 771-5765
Fax : +1 800 325-5052

1.4 Emergency telephone number

Emergency Phone # : 800-424-9300 CHEMTREC (USA) +1-703-527-3887 CHEMTREC (International) 24 Hours/day; 7 Days/week

SECTION 2: Hazards identification

2.1 Classification of the substance or mixture

GHS Classification in accordance with 29 CFR 1910 (OSHA HCS)

Flammable liquids (Category 2), H225
Eye irritation (Category 2A), H319

For the full text of the H-Statements mentioned in this Section, see Section 16.

2.2 GHS Label elements, including precautionary statements

Pictogram

Signal word Danger

Hazard statement(s)
H225 Highly flammable liquid and vapour.

H319	Causes serious eye irritation.

Precautionary statement(s)

P210	Keep away from heat/sparks/open flames/hot surfaces. No smoking.
P233	Keep container tightly closed.
P240	Ground/bond container and receiving equipment.
P241	Use explosion-proof electrical/ ventilating/ lighting equipment.
P242	Use only non-sparking tools.
P243	Take precautionary measures against static discharge.
P264	Wash skin thoroughly after handling.
P280	Wear protective gloves/ eye protection/ face protection.
P303 + P361 + P353	IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower.
P305 + P351 + P338	IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
P337 + P313	If eye irritation persists: Get medical advice/ attention.
P370 + P378	In case of fire: Use dry sand, dry chemical or alcohol-resistant foam to extinguish.
P403 + P235	Store in a well-ventilated place. Keep cool.
P501	Dispose of contents/ container to an approved waste disposal plant.

2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

---

SECTION 3: Composition/information on ingredients

3.1 Substances

Synonyms : Absolute alcohol

Formula : C_2H_6O
Molecular weight : 46.07 g/mol
CAS-No. : 64-17-5
EC-No. : 200-578-6
Index-No. : 603-002-00-5

Component	Classification	Concentration
ethanol		
	Flam. Liq. 2; Eye Irrit. 2A; H225, H319	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

SECTION 4: First aid measures

4.1 Description of first aid measures

General advice

Consult a physician. Show this safety data sheet to the doctor in attendance.Move out of dangerous area.

If inhaled
If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact
Wash off with soap and plenty of water. Consult a physician.

In case of eye contact
Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician.

If swallowed
Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

4.2 Most important symptoms and effects, both acute and delayed

The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11

4.3 Indication of any immediate medical attention and special treatment needed

No data available

SECTION 5: Firefighting measures

5.1 Extinguishing media

Suitable extinguishing media
Dry powder Dry sand

Unsuitable extinguishing media
Do NOT use water jet.

5.2 Special hazards arising from the substance or mixture

Carbon oxides
Combustible.

5.3 Advice for firefighters

Wear self-contained breathing apparatus for firefighting if necessary.

5.4 Further information

Use water spray to cool unopened containers.

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures

Use personal protective equipment. Avoid breathing vapours, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapours accumulating to form explosive concentrations. Vapours can accumulate in low areas.
For personal protection see section 8.

6.2 Environmental precautions

Prevent further leakage or spillage if safe to do so. Do not let product enter drains.

6.3 Methods and materials for containment and cleaning up

Contain spillage, and then collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container for disposal according to local / national regulations (see section 13).

6.4 Reference to other sections

For disposal see section 13.

SECTION 7: Handling and storage

7.1 Precautions for safe handling

Avoid contact with skin and eyes. Avoid inhalation of vapour or mist.
Use explosion-proof equipment.Keep away from sources of ignition - No smoking.Take measures to prevent the build up of electrostatic charge.
For precautions see section 2.2.

7.2 Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place. Containers which are opened must be carefully resealed and kept upright to prevent leakage.

Hygroscopic.
Storage class (TRGS 510): 3: Flammable liquids

7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Components with workplace control parameters

Component	CAS-No.	Value	Control parameters	Basis
ethanol	64-17-5	TWA	1,000 ppm 1,900 mg/m3	USA. OSHA - TABLE Z-1 Limits for Air Contaminants - 1910.1000
		TWA	1,000 ppm 1,900 mg/m3	USA. Occupational Exposure Limits (OSHA) - Table Z-1 Limits for Air Contaminants
	Remarks	The value in mg/m3 is approximate.		
		STEL	1,000 ppm	USA. ACGIH Threshold Limit Values (TLV)
		Upper Respiratory Tract irritation Confirmed animal carcinogen with unknown relevance to humans		
		TWA	1,000 ppm 1,900 mg/m3	USA. NIOSH Recommended Exposure Limits
		PEL	1,000 ppm 1,900 mg/m3	California permissible exposure limits for chemical contaminants (Title 8, Article 107)

8.2 Exposure controls

Appropriate engineering controls

Handle in accordance with good industrial hygiene and safety practice. Wash hands before breaks and at the end of workday.

Personal protective equipment

Eye/face protection
Face shield and safety glasses Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU).

Skin protection
Handle with gloves. Gloves must be inspected prior to use. Use proper glove removal technique (without touching glove's outer surface) to avoid skin contact with this product. Dispose of contaminated gloves after use in accordance with applicable laws and good laboratory practices. Wash and dry hands.

Full contact
Material: butyl-rubber
Minimum layer thickness: 0.3 mm
Break through time: 480 min
Material tested:Butoject® (KCL 897 / Aldrich Z677647, Size M)

Splash contact
Material: Nitrile rubber
Minimum layer thickness: 0.2 mm
Break through time: 38 min
Material tested:Dermatril® P (KCL 743 / Aldrich Z677388, Size M)

data source: KCL GmbH, D-36124 Eichenzell, phone +49 (0)6659 87300, e-mail sales@kcl.de, test method: EN374
If used in solution, or mixed with other substances, and under conditions which differ from EN 374, contact the supplier of the CE approved gloves. This recommendation is advisory only and must be evaluated by an industrial hygienist and safety officer familiar with the specific situation of anticipated use by our customers. It should not be construed as offering an approval for any specific use scenario.

Body Protection
Impervious clothing, Flame retardant antistatic protective clothing., The type of protective equipment must be selected according to the concentration and amount of the dangerous substance at the specific workplace.

Respiratory protection
Where risk assessment shows air-purifying respirators are appropriate use a full-face respirator with multi-purpose combination (US) or type ABEK (EN 14387) respirator cartridges as a backup to engineering controls. If the respirator is the sole means of protection, use a full-face supplied air respirator. Use respirators and components tested and approved under appropriate government standards such as NIOSH (US) or CEN (EU).

Control of environmental exposure
Prevent further leakage or spillage if safe to do so. Do not let product enter drains.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

a)	Appearance	Form: liquid Colour: colourless
b)	Odour	pungent
c)	Odour Threshold	No data available

d)	pH	7.0 at 10 g/l at 20 °C (68 °F)
e)	Melting point/freezing point	Melting point/range: -114 °C (-173 °F)
f)	Initial boiling point and boiling range	78 °C 172 °F
g)	Flash point	13 °C (55 °F) - closed cup
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	Upper explosion limit: 13.5 %(V) Lower explosion limit: 2.5 %(V)
k)	Vapour pressure	0.57 hPa at 19.6 °C (67.3 °F)
l)	Vapour density	1.6
m)	Relative density	0.789 g/mL at 25 °C (77 °F)
n)	Water solubility	1,000 g/l at 20 °C (68 °F) - completely miscible
o)	Partition coefficient: n-octanol/water	log Pow: -0.35 at 24 °C (75 °F) - Bioaccumulation is not expected.
p)	Auto-ignition temperature	455 °C (851 °F) at 1,013 hPa - DIN 51794
q)	Decomposition temperature	Distillable in an undecomposed state at normal pressure.
r)	Viscosity	No data available
s)	Explosive properties	No data available
t)	Oxidizing properties	No data available

9.2 Other safety information

Conductivity	< 1 µS/cm
Surface tension	72.75 mN/m at 20 °C (68 °F)
Relative vapour density	1.6

SECTION 10: Stability and reactivity

10.1 Reactivity

No data available

10.2 Chemical stability

Stable under recommended storage conditions.

10.3 Possibility of hazardous reactions

Vapours may form explosive mixture with air.

10.4 Conditions to avoid

Heat, flames and sparks.

10.5 Incompatible materials

rubber, various plastics

10.6 Hazardous decomposition products

Other decomposition products - No data available
Hazardous decomposition products formed under fire conditions. - Carbon oxides
In the event of fire: see section 5

SECTION 11: Toxicological information

11.1 Information on toxicological effects

Acute toxicity

LD50 Oral - Rat - male and female - 10,470 mg/kg
(OECD Test Guideline 401)
LC50 Inhalation - Rat - male and female - 4 h - 124.7 mg/l
(OECD Test Guideline 403)
Dermal: No data available
No data available

Skin corrosion/irritation

Skin - Rabbit
Result: No skin irritation - 24 h
(OECD Test Guideline 404)

Serious eye damage/eye irritation

Eyes - Rabbit
Result: Causes serious eye irritation.
(OECD Test Guideline 405)

Respiratory or skin sensitisation

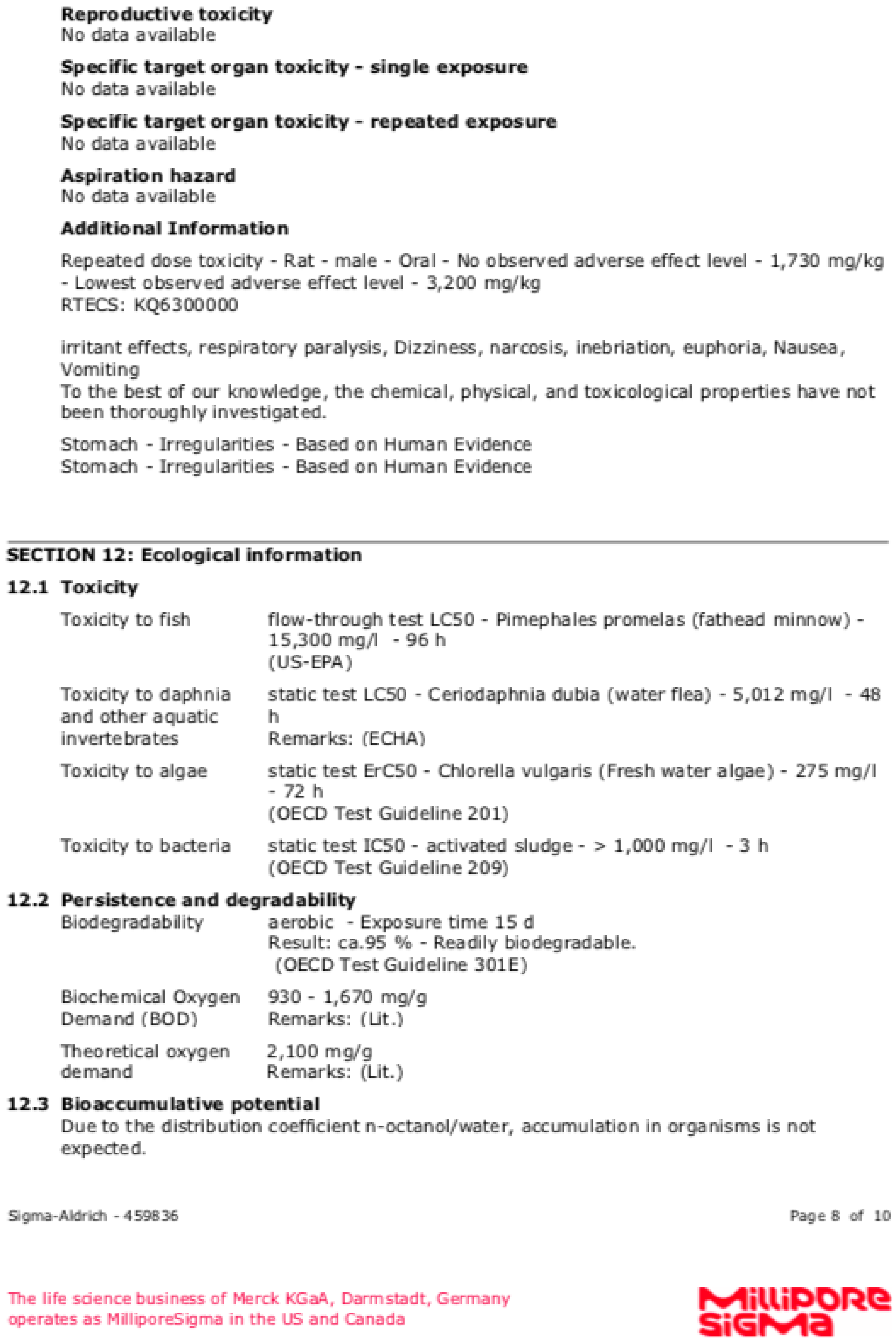
Maximisation Test - Guinea pig
Result: negative
(OECD Test Guideline 406)
Remarks: (in analogy to similar products)

Germ cell mutagenicity

Ames test
Salmonella typhimurium
Result: negative
In vitro mammalian cell gene mutation test
mouse lymphoma cells
Result: negative
OECD Test Guideline 478
Mouse - male
Result: Positive results were obtained in some in vivo tests.

Carcinogenicity

IARC:	No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.
NTP:	No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.
OSHA:	No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.



Reproductive toxicity
No data available

Specific target organ toxicity - single exposure
No data available

Specific target organ toxicity - repeated exposure
No data available

Aspiration hazard
No data available

Additional Information

Repeated dose toxicity - Rat - male - Oral - No observed adverse effect level - 1,730 mg/kg - Lowest observed adverse effect level - 3,200 mg/kg
RTECS: KQ6300000

irritant effects, respiratory paralysis, Dizziness, narcosis, inebriation, euphoria, Nausea, Vomiting
To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

Stomach - Irregularities - Based on Human Evidence
Stomach - Irregularities - Based on Human Evidence

SECTION 12: Ecological information

12.1 Toxicity

Toxicity to fish	flow-through test LC50 - Pimephales promelas (fathead minnow) - 15,300 mg/l - 96 h (US-EPA)
Toxicity to daphnia and other aquatic invertebrates	static test LC50 - Ceriodaphnia dubia (water flea) - 5,012 mg/l - 48 h Remarks: (ECHA)
Toxicity to algae	static test ErC50 - Chlorella vulgaris (Fresh water algae) - 275 mg/l - 72 h (OECD Test Guideline 201)
Toxicity to bacteria	static test IC50 - activated sludge - > 1,000 mg/l - 3 h (OECD Test Guideline 209)

12.2 Persistence and degradability

Biodegradability	aerobic - Exposure time 15 d Result: ca.95 % - Readily biodegradable. (OECD Test Guideline 301E)
Biochemical Oxygen Demand (BOD)	930 - 1,670 mg/g Remarks: (Lit.)
Theoretical oxygen demand	2,100 mg/g Remarks: (Lit.)

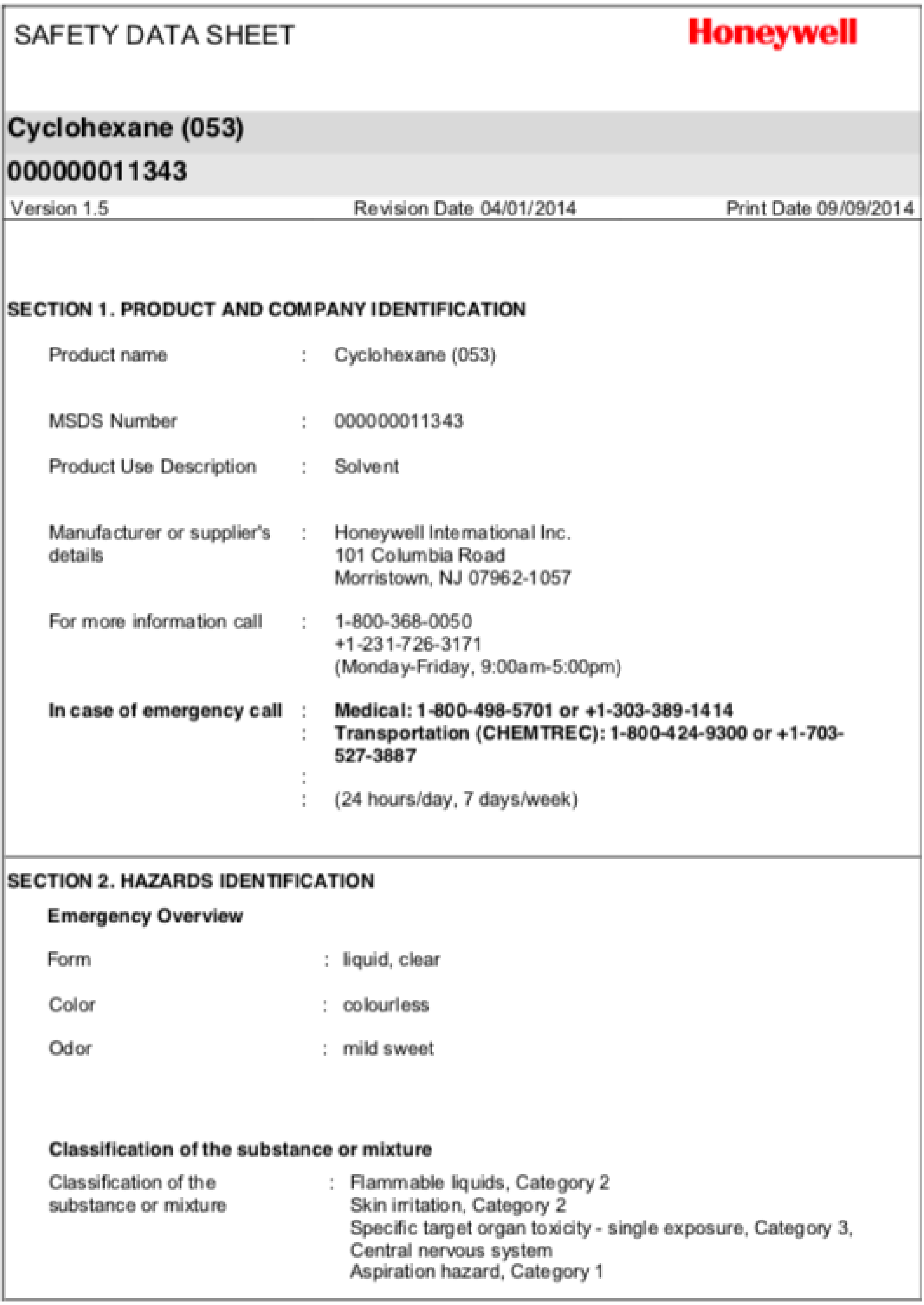
12.3 Bioaccumulative potential

Due to the distribution coefficient n-octanol/water, accumulation in organisms is not expected.

Sigma-Aldrich - 459836 Page 8 of 10

The life science business of Merck KGaA, Darmstadt, Germany operates as MilliporeSigma in the US and Canada

Figure G-1: Material Safety Data Sheet: Ethanol (Sigma-Aldrich, 2020)



SAFETY DATA SHEET **Honeywell**

Cyclohexane (053)

000000011343

Version 1.5 Revision Date 04/01/2014 Print Date 09/09/2014

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name : Cyclohexane (053)

MSDS Number : 000000011343

Product Use Description : Solvent

Manufacturer or supplier's details : Honeywell International Inc.
101 Columbia Road
Morristown, NJ 07962-1057

For more information call : 1-800-368-0050
+1-231-726-3171
(Monday-Friday, 9:00am-5:00pm)

In case of emergency call : **Medical: 1-800-498-5701 or +1-303-389-1414**
: **Transportation (CHEMTREC): 1-800-424-9300 or +1-703-527-3887**
:
: (24 hours/day, 7 days/week)

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

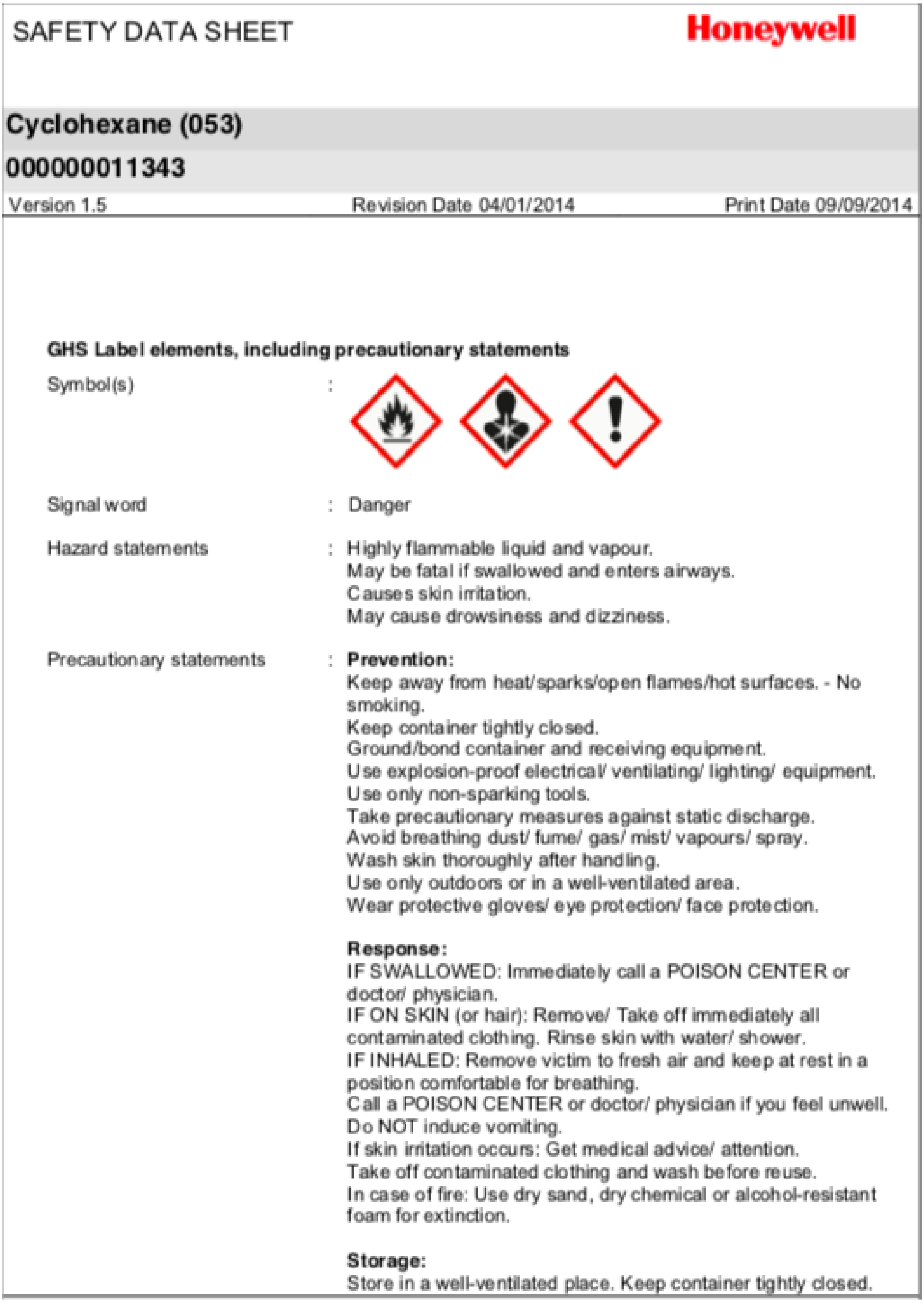
Form : liquid, clear

Color : colourless

Odor : mild sweet

Classification of the substance or mixture

Classification of the substance or mixture : Flammable liquids, Category 2
Skin irritation, Category 2
Specific target organ toxicity - single exposure, Category 3, Central nervous system
Aspiration hazard, Category 1



SAFETY DATA SHEET **Honeywell**

Cyclohexane (053)

000000011343

Version 1.5 Revision Date 04/01/2014 Print Date 09/09/2014

GHS Label elements, including precautionary statements

Symbol(s) :

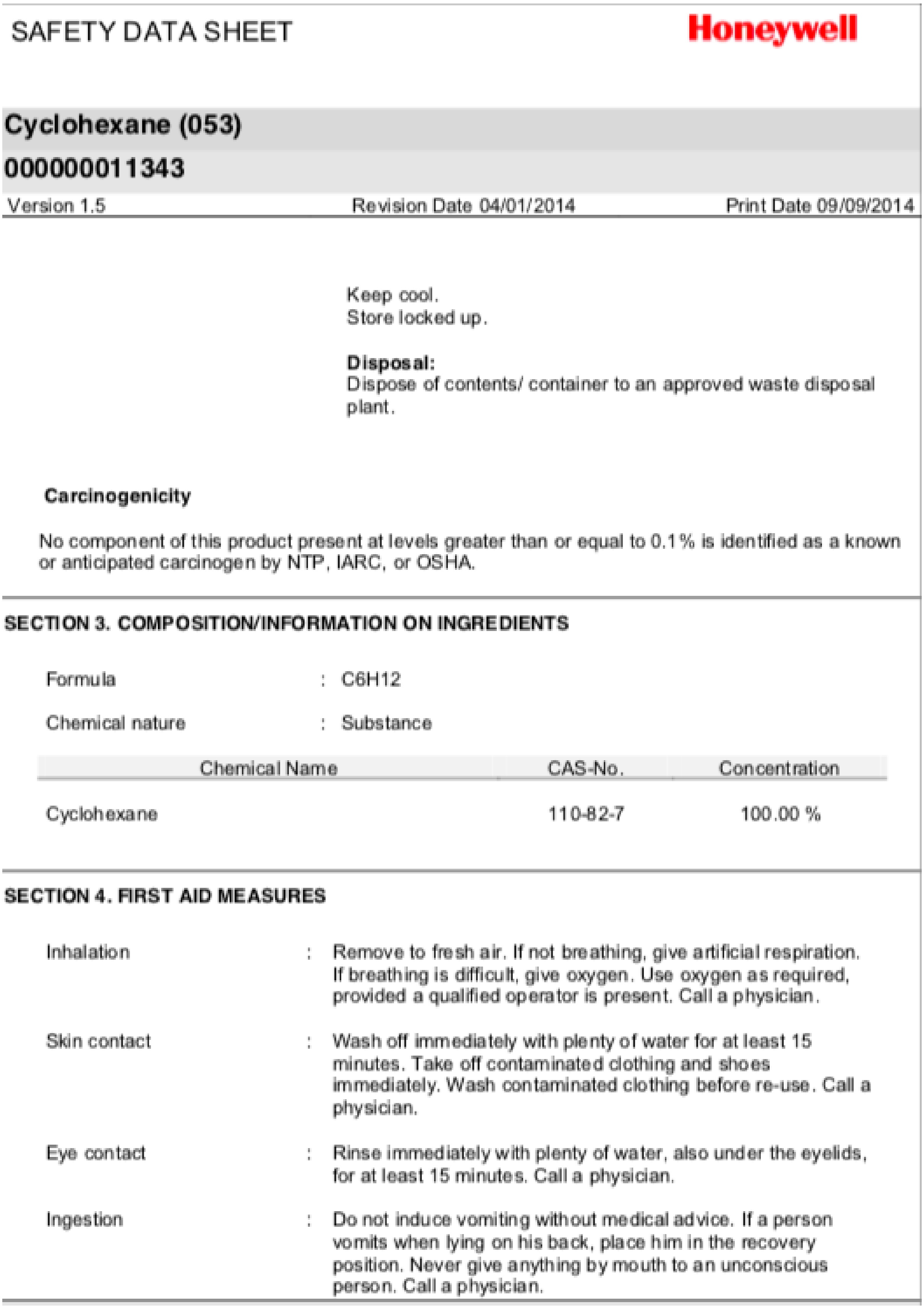
Signal word : Danger

Hazard statements : Highly flammable liquid and vapour.
May be fatal if swallowed and enters airways.
Causes skin irritation.
May cause drowsiness and dizziness.

Precautionary statements : **Prevention:**
Keep away from heat/sparks/open flames/hot surfaces. - No smoking.
Keep container tightly closed.
Ground/bond container and receiving equipment.
Use explosion-proof electrical/ ventilating/ lighting/ equipment.
Use only non-sparking tools.
Take precautionary measures against static discharge.
Avoid breathing dust/ fume/ gas/ mist/ vapours/ spray.
Wash skin thoroughly after handling.
Use only outdoors or in a well-ventilated area.
Wear protective gloves/ eye protection/ face protection.

Response:
IF SWALLOWED: Immediately call a POISON CENTER or doctor/ physician.
IF ON SKIN (or hair): Remove/ Take off immediately all contaminated clothing. Rinse skin with water/ shower.
IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
Call a POISON CENTER or doctor/ physician if you feel unwell.
Do NOT induce vomiting.
If skin irritation occurs: Get medical advice/ attention.
Take off contaminated clothing and wash before reuse.
In case of fire: Use dry sand, dry chemical or alcohol-resistant foam for extinction.

Storage:
Store in a well-ventilated place. Keep container tightly closed.



Keep cool.
Store locked up.

Disposal:
Dispose of contents/ container to an approved waste disposal plant.

Carcinogenicity

No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP, IARC, or OSHA.

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

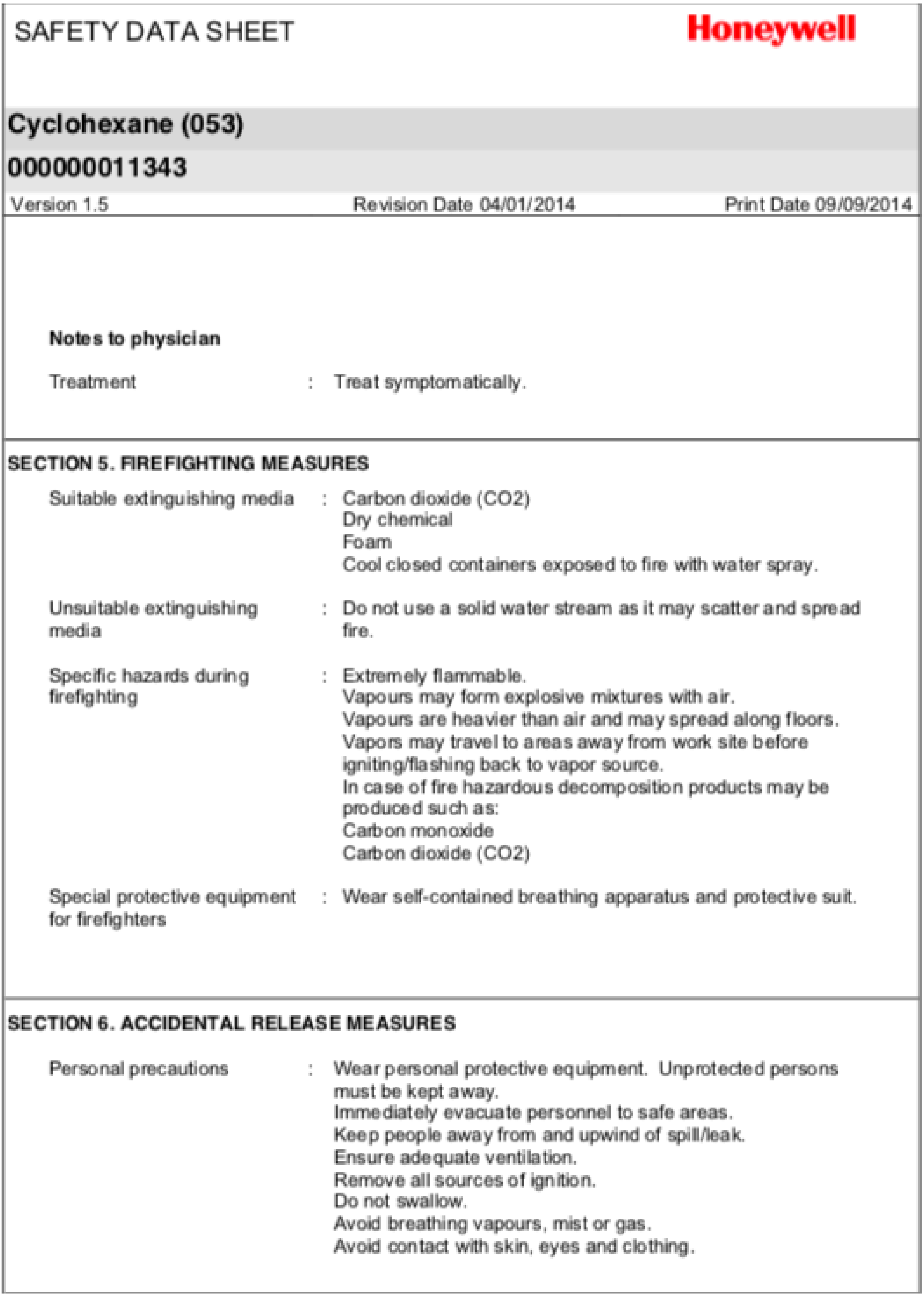
Formula : $C6H12$

Chemical nature : Substance

Chemical Name	CAS-No.	Concentration
Cyclohexane	110-82-7	100.00 %

SECTION 4. FIRST AID MEASURES

Inhalation	: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Use oxygen as required, provided a qualified operator is present. Call a physician.
Skin contact	: Wash off immediately with plenty of water for at least 15 minutes. Take off contaminated clothing and shoes immediately. Wash contaminated clothing before re-use. Call a physician.
Eye contact	: Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. Call a physician.
Ingestion	: Do not induce vomiting without medical advice. If a person vomits when lying on his back, place him in the recovery position. Never give anything by mouth to an unconscious person. Call a physician.



Notes to physician

Treatment : Treat symptomatically.

SECTION 5. FIREFIGHTING MEASURES

Suitable extinguishing media	:	Carbon dioxide (CO2) Dry chemical Foam Cool closed containers exposed to fire with water spray.
Unsuitable extinguishing media	:	Do not use a solid water stream as it may scatter and spread fire.
Specific hazards during firefighting	:	Extremely flammable. Vapours may form explosive mixtures with air. Vapours are heavier than air and may spread along floors. Vapors may travel to areas away from work site before igniting/flashing back to vapor source. In case of fire hazardous decomposition products may be produced such as: Carbon monoxide Carbon dioxide (CO2)
Special protective equipment for firefighters	:	Wear self-contained breathing apparatus and protective suit.

SECTION 6. ACCIDENTAL RELEASE MEASURES

Personal precautions	:	Wear personal protective equipment. Unprotected persons must be kept away. Immediately evacuate personnel to safe areas. Keep people away from and upwind of spill/leak. Ensure adequate ventilation. Remove all sources of ignition. Do not swallow. Avoid breathing vapours, mist or gas. Avoid contact with skin, eyes and clothing.

Environmental precautions	Prevent further leakage or spillage if safe to do so. Prevent product from entering drains. Discharge into the environment must be avoided. Do not flush into surface water or sanitary sewer system. Do not allow run-off from fire fighting to enter drains or water courses.
Methods for cleaning up	Ventilate the area. No sparking tools should be used. Use explosion-proof equipment. Contain spillage, and then collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container for disposal according to local / national regulations (see section 13).

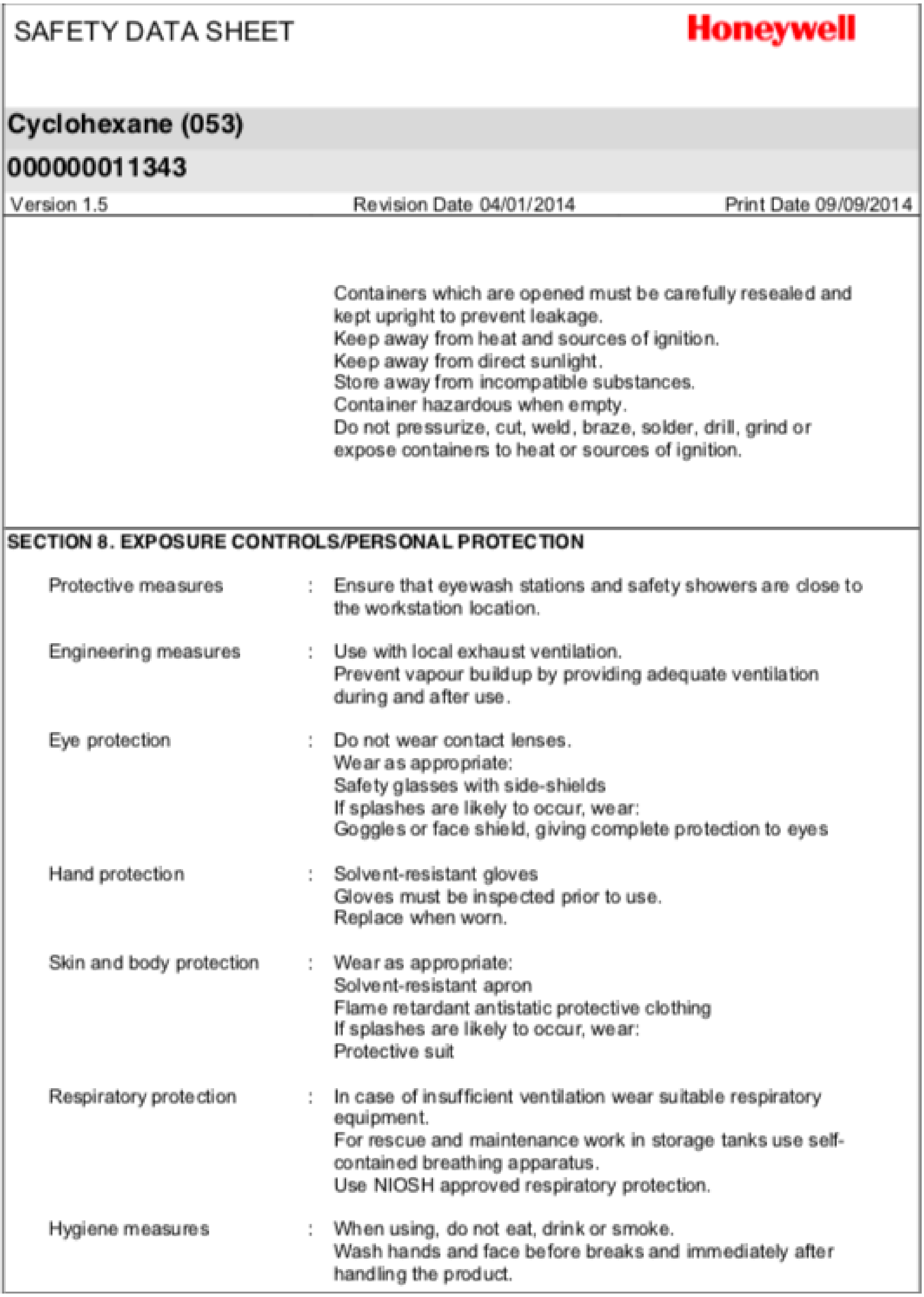
SECTION 7. HANDLING AND STORAGE

Handling

Handling	Wear personal protective equipment. Use only in well-ventilated areas. Keep container tightly closed. Do not smoke. Do not swallow. Avoid breathing vapours, mist or gas. Avoid contact with skin, eyes and clothing.
Advice on protection against fire and explosion	Keep away from fire, sparks and heated surfaces. Take precautionary measures against static discharges. Ensure all equipment is electrically grounded before beginning transfer operations. Use explosion-proof equipment. Keep product and empty container away from heat and sources of ignition. No sparking tools should be used. No smoking.

Storage

Requirements for storage areas and containers	Store in area designed for storage of flammable liquids. Protect from physical damage. Keep containers tightly closed in a dry, cool and well-ventilated place.



SAFETY DATA SHEET **Honeywell**

Cyclohexane (053)

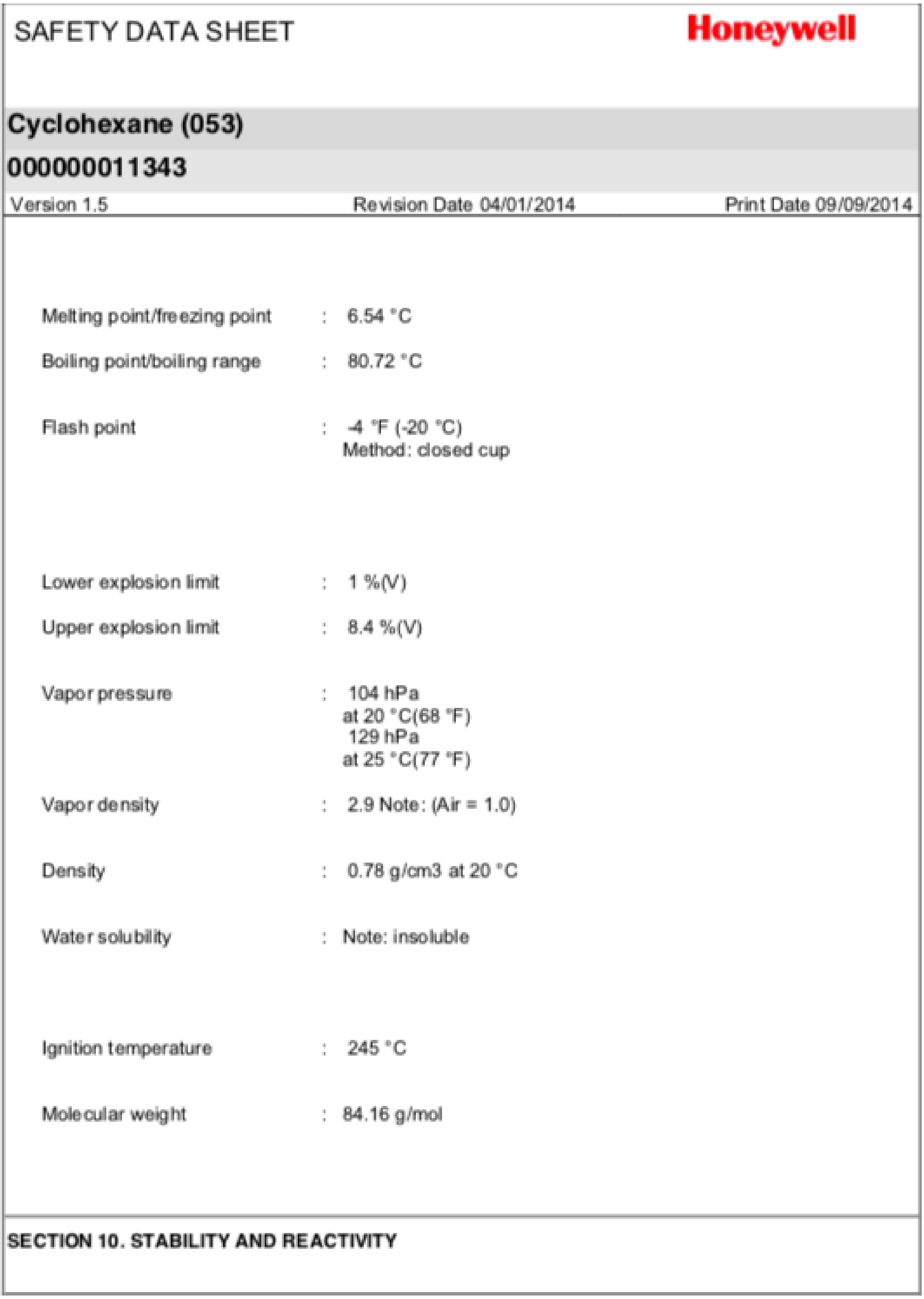
000000011343

Version 1.5 Revision Date 04/01/2014 Print Date 09/09/2014

Containers which are opened must be carefully resealed and kept upright to prevent leakage.
Keep away from heat and sources of ignition.
Keep away from direct sunlight.
Store away from incompatible substances.
Container hazardous when empty.
Do not pressurize, cut, weld, braze, solder, drill, grind or expose containers to heat or sources of ignition.

SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Protective measures	:	Ensure that eyewash stations and safety showers are close to the workstation location.
Engineering measures	:	Use with local exhaust ventilation. Prevent vapour buildup by providing adequate ventilation during and after use.
Eye protection	:	Do not wear contact lenses. Wear as appropriate: Safety glasses with side-shields If splashes are likely to occur, wear: Goggles or face shield, giving complete protection to eyes
Hand protection	:	Solvent-resistant gloves Gloves must be inspected prior to use. Replace when worn.
Skin and body protection	:	Wear as appropriate: Solvent-resistant apron Flame retardant antistatic protective clothing If splashes are likely to occur, wear: Protective suit
Respiratory protection	:	In case of insufficient ventilation wear suitable respiratory equipment. For rescue and maintenance work in storage tanks use self-contained breathing apparatus. Use NIOSH approved respiratory protection.
Hygiene measures	:	When using, do not eat, drink or smoke. Wash hands and face before breaks and immediately after handling the product.



SAFETY DATA SHEET **Honeywell**

Cyclohexane (053)

000000011343

Version 1.5 Revision Date 04/01/2014 Print Date 09/09/2014

Melting point/freezing point : 6.54 °C

Boiling point/boiling range : 80.72 °C

Flash point : -4 °F (-20 °C)
Method: closed cup

Lower explosion limit : 1 %(V)

Upper explosion limit : 8.4 %(V)

Vapor pressure : 104 hPa
at 20 °C(68 °F)
129 hPa
at 25 °C(77 °F)

Vapor density : 2.9 Note: (Air = 1.0)

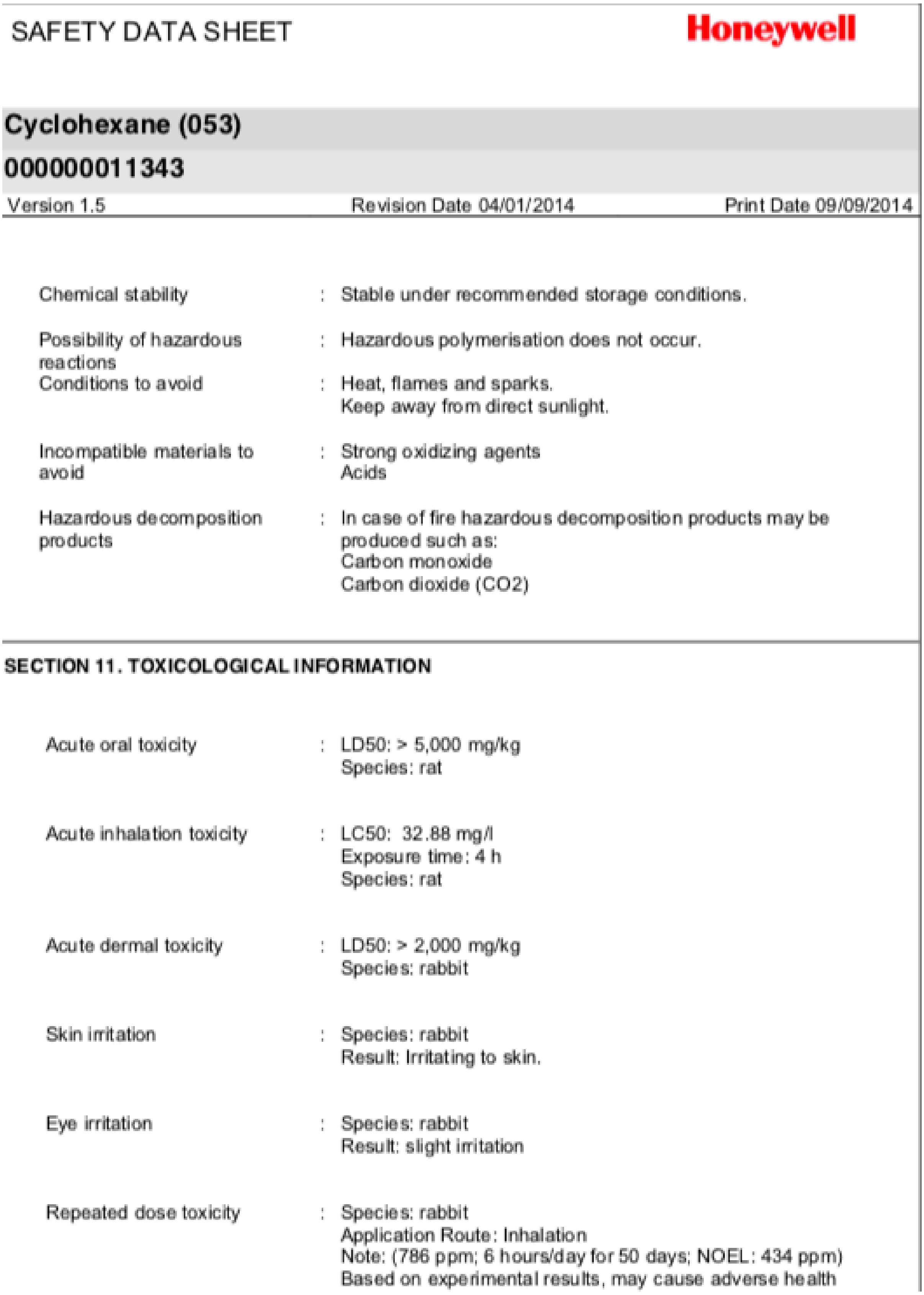
Density : 0.78 g/cm3 at 20 °C

Water solubility : Note: insoluble

Ignition temperature : 245 °C

Molecular weight : 84.16 g/mol

SECTION 10. STABILITY AND REACTIVITY



Chemical stability : Stable under recommended storage conditions.

Possibility of hazardous reactions : Hazardous polymerisation does not occur.

Conditions to avoid : Heat, flames and sparks.
Keep away from direct sunlight.

Incompatible materials to avoid : Strong oxidizing agents
Acids

Hazardous decomposition products : In case of fire hazardous decomposition products may be produced such as:
Carbon monoxide
Carbon dioxide (CO_2)

SECTION 11. TOXICOLOGICAL INFORMATION

Acute oral toxicity : LD50: > 5,000 mg/kg
Species: rat

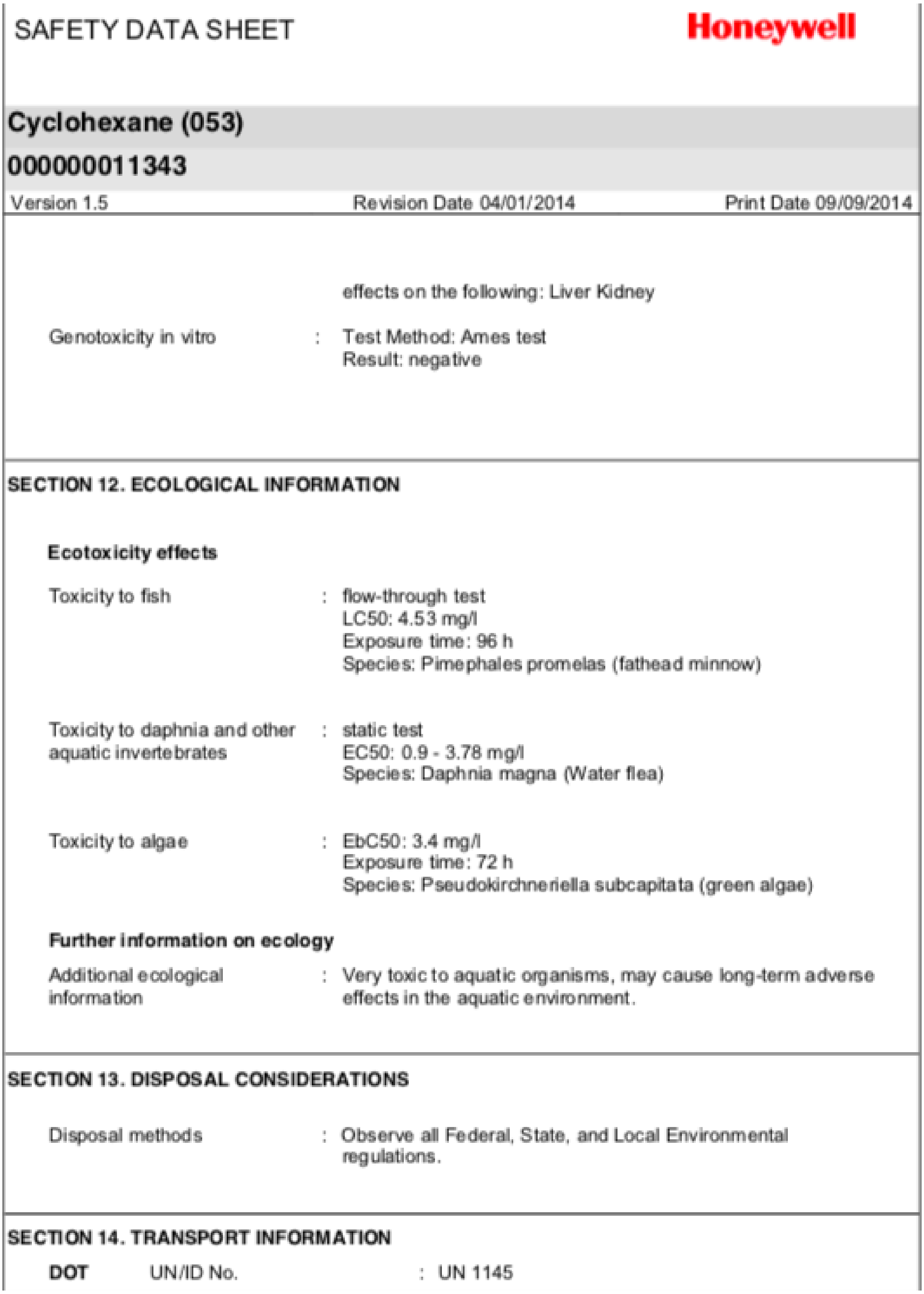
Acute inhalation toxicity : LC50: 32.88 mg/l
Exposure time: 4 h
Species: rat

Acute dermal toxicity : LD50: > 2,000 mg/kg
Species: rabbit

Skin irritation : Species: rabbit
Result: Irritating to skin.

Eye irritation : Species: rabbit
Result: slight irritation

Repeated dose toxicity : Species: rabbit
Application Route: Inhalation
Note: (786 ppm; 6 hours/day for 50 days; NOEL: 434 ppm)
Based on experimental results, may cause adverse health



SAFETY DATA SHEET

Honeywell

Cyclohexane (053)

000000011343

Version 1.5　Revision Date 04/01/2014　Print Date 09/09/2014

effects on the following: Liver Kidney

Genotoxicity in vitro	Test Method: Ames test Result: negative

SECTION 12. ECOLOGICAL INFORMATION

Ecotoxicity effects

Toxicity to fish	flow-through test LC50: 4.53 mg/l Exposure time: 96 h Species: Pimephales promelas (fathead minnow)
Toxicity to daphnia and other aquatic invertebrates	static test EC50: 0.9 - 3.78 mg/l Species: Daphnia magna (Water flea)
Toxicity to algae	EbC50: 3.4 mg/l Exposure time: 72 h Species: Pseudokirchneriella subcapitata (green algae)

Further information on ecology

Additional ecological information	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

SECTION 13. DISPOSAL CONSIDERATIONS

Disposal methods	Observe all Federal, State, and Local Environmental regulations.

SECTION 14. TRANSPORT INFORMATION

DOT	UN/ID No.	UN 1145

Figure G-2: Material Safety Data Sheet: Cyclohexane (Honeywell, 2014)

ISO9001:2000 Certified

Material Safety Data Sheet
Water, Deionized

Section 1 - Chemical Product and Company Identification

MSDS Name:
Water, Deionized
Catalog Numbers:
LC26750
Synonyms:
None
Company Identification:
LabChem Inc
200 William Pitt Way
Pittsburgh, PA 15238
Company Phone Number:
(412) 826-5230
Emergency Phone Number:
(800) 424-9300
CHEMTREC Phone Number:
(800) 424-9300

Section 2 – Composition, Information on Ingredients

CAS#	Chemical Name:	Percent
7732-18-5	Water	100

Section 3 - Hazards Identification

Emergency Overview

Appearance: *Clear, colorless liquid*
Water is expected to be a low hazard for usual industrial handling.
Target Organs: *None*

Potential Health Effects

Eye:
Low hazard for usual industrial handling.
Skin:
Low hazard for usual industrial handling.
Ingestion:
Low hazard for usual industrial handling.
Inhalation:
Low hazard for usual industrial handling.
Chronic:
No chronic hazards are associated with exposure to water.

Material Safety Data Sheet
Water, Deionized

Section 4 - First Aid Measures

Eyes:
No special first aid measures are necessary.

Skin:
No special first aid measures are necessary.

Ingestion:
No special first aid measures are necessary.

Inhalation:
No special first aid measures are necessary.

Notes to Physician:
Treat symptomatically and supportively.

Section 5 - Fire Fighting Measures

General Information:
As in any fire, wear a self-contained breathing apparatus in pressure-demand, MSHA/NIOSH (approved or equivalent), and full protective gear.

Extinguishing Media:
For small fires, use dry chemical, carbon dioxide, water spray or alcohol-resistant foam.

Autoignition Temperature:
Not applicable.

Flash Point:
Not applicable.

NFPA Rating:
CAS# 7732-18-5: Health- 0, Flammability- 0, Instability- 0.

Explosion Limits:
Lower: n/a Upper: n/a

Section 6 - Accidental Release Measures

General Information:
Use proper personal protective equipment as indicated in Section 8.

Spills/Leaks:
No special cleaning procedures are necessary. Wipe up spill with a mop or other absorbent material.

Section 7 - Handling and Storage

Handling:
No special precautions are necessary.

Storage:
No special precautions are necessary.

Section 8 - Exposure Controls, Personal Protection

Engineering Controls:

There are no special ventilation requirements.

Exposure Limits:

Chemical Name:	ACGIH	NIOSH	OSHA
Water	none listed	none listed	none listed

OSHA Vacated PELs:

None listed.

Personal Protective Equipment

Eyes:

Wear appropriate protective eyeglasses or chemical safety goggles as described by OSHA's eye and face protection regulations in 29 CFR 1910.133 or European Standard EN166.

Skin:

Wear appropriate gloves to prevent skin exposure.

Clothing:

Wear appropriate protective clothing to prevent skin exposure.

Respirators:

Follow the OSHA respirator regulations found in 29 CFR 1910.134 or European Standard EN 149. Use a NIOSH/MSHA or European Standard EN 149 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.

Section 9 - Physical and Chemical Properties

Physical State:	Clear liquid
Color:	Colorless
Odor:	Odorless
pH:	7
Vapor Pressure:	Not available
Vapor Density:	17.5 mm Hg @ 20℃
Evaporation Rate:	Not available
Viscosity:	1 cP @ 20℃
Boiling Point:	100℃
Freezing/Melting Point:	0℃
Decomposition Temperature:	Not available
Solubility in water:	Soluble
Specific Gravity/Density:	1.0
Molecular Formula:	H_2O
Molecular Weight:	18.0134

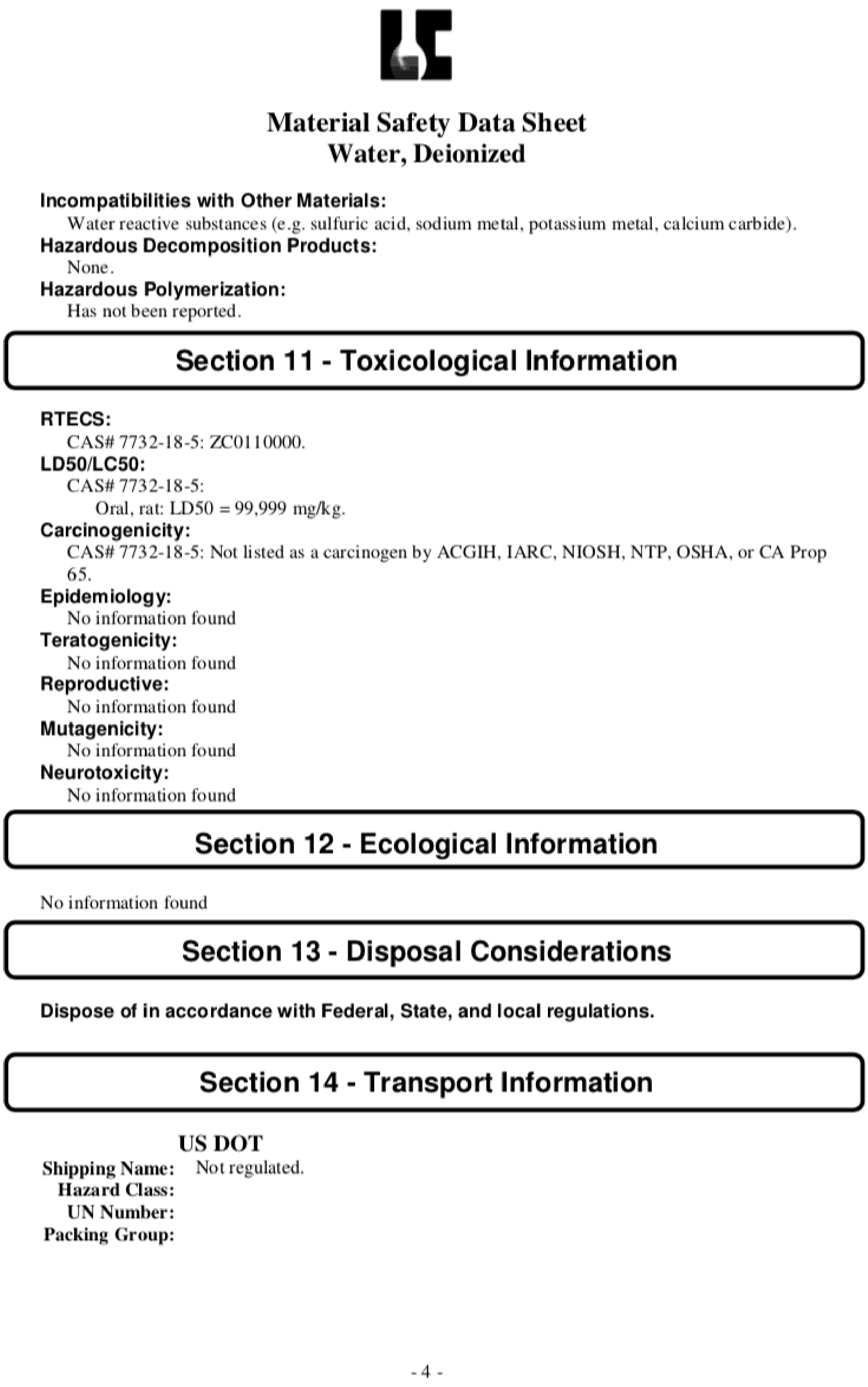
Section 10 - Stability and Reactivity

Chemical Stability:

Stable under normal temperatures and pressures.

Conditions to Avoid:

Incompatible materials.



Material Safety Data Sheet
Water, Deionized

Incompatibilities with Other Materials:
Water reactive substances (e.g. sulfuric acid, sodium metal, potassium metal, calcium carbide).

Hazardous Decomposition Products:
None.

Hazardous Polymerization:
Has not been reported.

Section 11 - Toxicological Information

RTECS:
CAS# 7732-18-5: ZC0110000.

LD50/LC50:
CAS# 7732-18-5:
Oral, rat: LD50 = 99,999 mg/kg.

Carcinogenicity:
CAS# 7732-18-5: Not listed as a carcinogen by ACGIH, IARC, NIOSH, NTP, OSHA, or CA Prop 65.

Epidemiology:
No information found

Teratogenicity:
No information found

Reproductive:
No information found

Mutagenicity:
No information found

Neurotoxicity:
No information found

Section 12 - Ecological Information

No information found

Section 13 - Disposal Considerations

Dispose of in accordance with Federal, State, and local regulations.

Section 14 - Transport Information

	US DOT
Shipping Name:	Not regulated.
Hazard Class:	
UN Number:	
Packing Group:	

Figure G-3: Material Safety Data Sheet: Deionised Water (LabChem, 2009)

LabChem
performance through chemistry

Acetone

Safety Data Sheet

according to Federal Register / Vol. 77, No. 58 / Monday, March 26, 2012 / Rules and Regulations
Date of issue: 11/12/1998 Revision date: 04/24/2018 Supersedes: 04/24/2018 Version: 1.3

SECTION 1: Identification

1.1. Identification

Product form : Substance
Substance name : Acetone
Chemical name : 2-Propanone
CAS-No. : 67-64-1
Product code : LC10420, LC10425
Formula : C3H6O
Synonyms : 2-propanone / beta-ketopropane / dimethyl formaldehyde / dimethyl ketone / dimethylketal / DMK (=dimethyl ketone) / keto propane / methyl ketone / pyroacetic acid / pyroacetic ether / pyroacetic spirit

1.2. Recommended use and restrictions on use

Use of the substance/mixture : Solvent
Cleaning product
Chemical raw material
Recommended use : Laboratory chemicals
Restrictions on use : Not for food, drug or household use

1.3. Supplier

LabChem, Inc.
Jackson's Pointe Commerce Park Building 1000, 1010 Jackson's Pointe Court
Zelienople, PA 16063 - USA
T 412-826-5230 - F 724-473-0647

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

Flammable liquids Category 2	H225	Highly flammable liquid and vapour
Serious eye damage/eye irritation Category 2A	H319	Causes serious eye irritation
Specific target organ toxicity (single exposure) Category 3	H336	May cause drowsiness or dizziness

Full text of H statements : see section 16

2.2. GHS Label elements, including precautionary statements

GHS US labeling

Hazard pictograms (GHS US) :

GHS02

GHS07

Signal word (GHS US) : Danger
Hazard statements (GHS US) : H225 - Highly flammable liquid and vapour
H319 - Causes serious eye irritation
H336 - May cause drowsiness or dizziness
Precautionary statements (GHS US) : P210 - Keep away from heat, hot surfaces, open flames, sparks. - No smoking.
P233 - Keep container tightly closed.
P240 - Ground/bond container and receiving equipment.
P241 - Use explosion-proof electrical, lighting, ventilating equipment
P242 - Use only non-sparking tools.
P243 - Take precautionary measures against static discharge.
P261 - Avoid breathing mist, spray, vapors.

P264 - Wash exposed skin thoroughly after handling.
P271 - Use only outdoors or in a well-ventilated area.
P280 - Wear eye protection, face protection, protective clothing, protective gloves.
P303+P361+P353 - IF ON SKIN (or hair): Remove/Take off immediately all contaminated clothing. Rinse skin with water/shower.
P304+P340 - IF INHALED: Remove person to fresh air and keep comfortable for breathing.
P305+P351+P338 - If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing
P312 - Call a POISON CENTER or doctor/physician if you feel unwell.
P337+P313 - If eye irritation persists: Get medical advice/attention.
P370+P378 - In case of fire: Use dry chemical powder, alcohol-resistant foam, carbon dioxide (CO2) to extinguish
P403+P233 - Store in a well-ventilated place. Keep container tightly closed.
P405 - Store locked up.
P501 - Dispose of contents/container to comply with local, state and federal regulations
P235 - Keep cool.

2.3. Other hazards which do not result in classification

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
Acetone (Main constituent)	(CAS-No.) 67-64-1	100	Flam. Liq. 2, H225 Eye Irrit. 2A, H319 STOT SE 3, H336

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 : Check the vital functions. Unconscious: maintain adequate airway and respiration. Respiratory arrest: artificial respiration or oxygen. Cardiac arrest: perform resuscitation. Victim conscious with labored breathing: half-seated. Victim in shock: on his back with legs slightly raised. Vomiting: prevent asphyxia/aspiration pneumonia. Prevent cooling by covering the victim (no warming up). Keep watching the victim. Give psychological aid. Keep the victim calm, avoid physical strain. Depending on the victim's condition: doctor/hospital.

First-aid measures after inhalation : Remove the victim into fresh air. Respiratory problems: consult a doctor/medical service.

First-aid measures after skin contact : Wash immediately with lots of water. Soap may be used. Do not apply (chemical) neutralizing agents. Remove clothing before washing. Take victim to a doctor if irritation persists.

First-aid measures after eye contact : Rinse immediately with plenty of water. Remove contact lenses, if present and easy to do. Continue rinsing. Do not apply neutralizing agents. Take victim to an ophthalmologist if irritation persists.

First-aid measures after ingestion : Rinse mouth with water. Immediately after ingestion: give lots of water to drink. Do not give milk/oil to drink. Do not induce vomiting. Give activated charcoal. Call Poison Information Centre (www.big.be/antigif.htm). Consult a doctor/medical service if you feel unwell. Ingestion of large quantities: immediately to hospital. Doctor: gastric lavage.

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

Symptoms/effects : Not expected to present a significant hazard under anticipated conditions of normal use.

Symptoms/effects after inhalation : EXPOSURE TO HIGH CONCENTRATIONS: Feeling of weakness. Irritation of the respiratory tract. Nausea. Vomiting. Headache. Central nervous system depression. Dizziness. Narcosis. Excited/restless. Drunkenness. Disturbed motor response. Respiratory difficulties. Disturbances of consciousness.

Symptoms/effects after skin contact : ON CONTINUOUS EXPOSURE/CONTACT: Dry skin. Cracking of the skin.

Symptoms/effects after eye contact : Irritation of the eye tissue.

Symptoms/effects after ingestion	: Dry/sore throat. Risk of aspiration pneumonia. Symptoms similar to those listed under inhalation. AFTER ABSORPTION OF LARGE QUANTITIES: Irritation of the gastric/intestinal mucosa. Change in the blood composition. Change in urine output. Renal disease. Enlargement/disease of the liver.
Symptoms/effects upon intravenous administration	: Not available.
Chronic symptoms	: ON CONTINUOUS/REPEATED EXPOSURE/CONTACT: Red skin. Skin rash/inflammation. Dry/sore throat. Headache. Nausea. Feeling of weakness. Loss of weight. Possible inflammation of the respiratory tract.

4.3. Immediate medical attention and special treatment, if necessary

Obtain medical assistance.

SECTION 5: Fire-fighting measures

5.1. Suitable (and unsuitable) extinguishing media

Suitable extinguishing media	: Quick-acting ABC powder extinguisher. Quick-acting BC powder extinguisher. Quick-acting class B foam extinguisher. Quick-acting CO2 extinguisher. Class B foam (alcohol-resistant). Water spray if puddle cannot expand.
Unsuitable extinguishing media	: Water (quick-acting extinguisher, reel); risk of puddle expansion. Water; risk of puddle expansion.

5.2. Specific hazards arising from the chemical

Fire hazard	: DIRECT FIRE HAZARD. Highly flammable liquid and vapour. Gas/vapor flammable with air within explosion limits. INDIRECT FIRE HAZARD. May be ignited by sparks. Gas/vapor spreads at floor level: ignition hazard. Reactions involving a fire hazard: see "Reactivity Hazard".
Explosion hazard	: DIRECT EXPLOSION HAZARD. Gas/vapour explosive with air within explosion limits. INDIRECT EXPLOSION HAZARD. Heat may cause pressure rise in tanks/drums: explosion risk. may be ignited by sparks. Reactions with explosion hazards: see "Reactivity Hazard".
Reactivity	: Violent to explosive reaction with many compounds. Prolonged storage: on exposure to light: release of harmful gases/vapours.

5.3. Special protective equipment and precautions for fire-fighters

Firefighting instructions	: Cool tanks/drums with water spray/remove them into safety. Physical explosion risk: extinguish/cool from behind cover. Do not move the load if exposed to heat. After cooling: persistant risk of physical explosion.
Protection during firefighting	: Heat/fire exposure: compressed air/oxygen apparatus.

SECTION 6: Accidental release measures

6.1. Personal precautions, protective equipment and emergency procedures

6.1.1. For non-emergency personnel

Protective equipment	: Gloves. Protective goggles. Protective clothing. Large spills/in enclosed spaces: compressed air apparatus.
Emergency procedures	: Keep upwind. Mark the danger area. Consider evacuation. Seal off low-lying areas. Close doors and windows of adjacent premises. Stop engines and no smoking. No naked flames or sparks. Spark- and explosion-proof appliances and lighting equipment. Keep containers closed. Wash contaminated clothes.

6.1.2. For emergency responders

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

6.2. Environmental precautions

Prevent spreading in sewers.

6.3. Methods and material for containment and cleaning up

For containment	: Contain released substance, pump into suitable containers. Plug the leak, cut off the supply. Dam up the liquid spill. Try to reduce evaporation. Measure the concentration of the explosive gas-air mixture. Dilute/disperse combustible gas/vapour with water curtain. Provide equipment/receptacles with earthing. Do not use compressed air for pumping over spills.
Methods for cleaning up	: Take up liquid spill into inert absorbent material, e.g.: sand, earth, vermiculite. Scoop absorbed substance into closing containers. Spill must not return in its original container. Carefully collect the spill/leftovers. Damaged/cooled tanks must be emptied. Do not use compressed air for pumping over spills. Clean contaminated surfaces with an excess of water. Take collected spill to manufacturer/competent authority. Wash clothing and equipment after handling.

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	: Use spark-/explosionproof appliances and lighting system. Take precautions against electrostatic charges. Keep away from naked flames/heat. Keep away from ignition sources/sparks. Measure the concentration in the air regularly. Work under local exhaust/ventilation. Comply with the legal requirements. Remove contaminated clothing immediately. Clean contaminated clothing. Handle uncleaned empty containers as full ones. Thoroughly clean/dry the installation before use. Do not discharge the waste into the drain. Do not use compressed air for pumping over. Keep container tightly closed.
Hygiene measures	: Do not eat, drink or smoke when using this product. Wash contaminated clothing before reuse. 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 only in the original container in a cool, well ventilated place away from : Heat sources, Direct sunlight, incompatible materials. Keep container closed when not in use.
Incompatible products	: Strong bases. Strong acids.
Incompatible materials	: Sources of ignition. Direct sunlight.
Storage temperature	: 15 - 20 °C
Heat-ignition	: KEEP SUBSTANCE AWAY FROM: heat sources. ignition sources.
Prohibitions on mixed storage	: KEEP SUBSTANCE AWAY FROM: oxidizing agents. reducing agents. strong acids. (strong) bases. halogens. amines.
Storage area	: Store in a cool area. Keep out of direct sunlight. Store in a dry area. Store in a dark area. Ventilation at floor level. Fireproof storeroom. Provide for an automatic sprinkler system. Provide for a tub to collect spills. Provide the tank with earthing. Meet the legal requirements.
Special rules on packaging	: SPECIAL REQUIREMENTS: closing. with pressure relief valve. clean. opaque. correctly labelled. meet the legal requirements. Secure fragile packagings in solid containers.
Packaging materials	: SUITABLE MATERIAL: steel. stainless steel. carbon steel. aluminium. iron. copper. nickel. bronze. glass. MATERIAL TO AVOID: synthetic material.

SECTION 8: Exposure controls/personal protection

8.1. Control parameters

Acetone (67-64-1)		
ACGIH	ACGIH TWA (ppm)	250 ppm
ACGIH	ACGIH STEL (ppm)	500 ppm
NIOSH	NIOSH REL (TWA) (mg/m³)	590 mg/m³
NIOSH	NIOSH REL (TWA) (ppm)	250 ppm

8.2. Appropriate engineering controls

Appropriate engineering controls	: Emergency eye wash fountains should be available in the immediate vicinity of any potential exposure.

8.3. Individual protection measures/Personal protective equipment

Personal protective equipment:

Safety glasses. Gloves. Protective clothing. Face shield. High gas/vapor concentration: gas mask with filter type A.

Materials for protective clothing:

GIVE GOOD RESISTANCE: butyl rubber. tetrafluoroethylene. GIVE LESS RESISTANCE: chlorosulfonated polyethylene. natural rubber. neoprene. polyurethane. PVA. styrene-butadiene rubber. GIVE POOR RESISTANCE: nitrile rubber. polyethylene. PVC. viton. nitrile rubber/PVC

Hand protection:

Gloves

Eye protection:

Safety glasses

Skin and body protection:

Head/neck protection. Protective clothing

Respiratory protection:

Full face mask with filter type AX at conc. in air > exposure limit

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
Appearance	: Liquid.
	: Colourless
	: Aromatic odour Sweet odour Fruity odour
Odor threshold	: No data available
pH	: 7 (10 g/l)
Melting point	: -95 °C
Freezing point	: No data available
Boiling point	: 56 °C
Critical temperature	: 235 °C
Critical pressure	: 47010 hPa
Flash point	: -17 °C (Closed cup)
Relative evaporation rate (butyl acetate=1)	: 6
Relative evaporation rate (ether=1)	: 2
Flammability (solid, gas)	: Non flammable.
Vapor pressure	: 247 hPa (20 °C)
Vapor pressure at 50 °C	: 828 hPa
Relative vapor density at 20 °C	: 2
Relative density	: 0.79
Relative density of saturated gas/air mixture	: 1.2
Specific gravity / density	: 786 kg/m^3
Molecular mass	: 58.08 g/mol
Solubility	: Soluble in water. Soluble in ethanol. Soluble in ether. Soluble in dimethyl ether. Soluble in petroleum spirit. Soluble in chloroform. Soluble in dimethylformamide. Soluble in oils/fats. Water: complete Ethanol: complete Ether: complete
Log Pow	: -0.24 (Test data)
Auto-ignition temperature	: 465 °C
Decomposition temperature	: No data available
Viscosity, kinematic	: 0.417 mm^2/s
Viscosity, dynamic	: 0.32 mPa·s (20 °C)
Explosion limits	: 2 - 12.8 vol % 60 - 310 g/m^3 Lower explosive limit (LEL): 2 vol % UEL: 12.8 vol %
Explosive properties	: No data available.
Oxidizing properties	: None.

9.2. Other information

Minimum ignition energy : 1.15 mJ
Specific conductivity : 6000000 pS/m (25 °C)
Saturation concentration : 589 g/m³
VOC content : 100 %
Other properties : Gas/vapour heavier than air at 20°C. Clear. Highly volatile. Neutral reaction.

SECTION 10: Stability and reactivity

10.1. Reactivity

Violent to explosive reaction with many compounds. Prolonged storage: on exposure to light: release of harmful gases/vapours.

10.2. Chemical stability

Unstable on exposure to light.

10.3. Possibility of hazardous reactions

Reacts with (strong) oxidizers.

10.4. Conditions to avoid

Direct sunlight. Extremely high or low temperatures.

10.5. Incompatible materials

Strong acids. Strong bases. Strong oxidizers.

10.6. Hazardous decomposition products

fume. Carbon monoxide. Carbon dioxide.

SECTION 11: Toxicological information

11.1. Information on toxicological effects

Likely routes of exposure : Inhalation; Skin and eye contact
Acute toxicity : Not classified

Acetone (67-64-1)	
LD50 oral rat	5800 mg/kg (Equivalent or similar to OECD 401, Rat, Female, Experimental value, Oral)
LD50 dermal rabbit	20000 mg/kg (Equivalent or similar to OECD 402, Rabbit, Male, Experimental value, Dermal)
LC50 inhalation rat (mg/l)	76 mg/l (Other, 4 h, Rat, Female, Experimental value, Inhalation (vapours))
ATE US (oral)	5800 mg/kg body weight
ATE US (dermal)	20000 mg/kg body weight
ATE US (gases)	30000 ppmV/4h
ATE US (vapors)	71 mg/l/4h
ATE US (dust, mist)	71 mg/l/4h

Skin corrosion/irritation : Not classified
pH: 7 (10 g/l)
Serious eye damage/irritation : Causes serious eye irritation.
pH: 7 (10 g/l)
Respiratory or skin sensitization : Not classified
Germ cell mutagenicity : Not classified
Based on available data, the classification criteria are not met
Carcinogenicity : Not classified
Reproductive toxicity : Not classified
Based on available data, the classification criteria are not met
Specific target organ toxicity – single exposure : May cause drowsiness or dizziness.
Specific target organ toxicity – repeated exposure : Not classified
Aspiration hazard : Not classified
Potential Adverse human health effects and symptoms : Based on available data, the classification criteria are not met.

Symptoms/effects after inhalation : EXPOSURE TO HIGH CONCENTRATIONS: Feeling of weakness. Irritation of the respiratory tract. Nausea. Vomiting. Headache. Central nervous system depression. Dizziness. Narcosis. Excited/restless. Drunkenness. Disturbed motor response. Respiratory difficulties. Disturbances of consciousness.

Symptoms/effects after skin contact : ON CONTINUOUS EXPOSURE/CONTACT: Dry skin. Cracking of the skin.

Symptoms/effects after eye contact : Irritation of the eye tissue.

Symptoms/effects after ingestion : Dry/sore throat. Risk of aspiration pneumonia. Symptoms similar to those listed under inhalation. AFTER ABSORPTION OF LARGE QUANTITIES: Irritation of the gastric/intestinal mucosa. Change in the blood composition. Change in urine output. Renal disease. Enlargement/disease of the liver.

Symptoms/effects upon intravenous administration : Not available.

Chronic symptoms : ON CONTINUOUS/REPEATED EXPOSURE/CONTACT: Red skin. Skin rash/inflammation. Dry/sore throat. Headache. Nausea. Feeling of weakness. Loss of weight. Possible inflammation of the respiratory tract.

SECTION 12: Ecological information

12.1. Toxicity

Ecology - general : Not classified as dangerous for the environment according to the criteria of Regulation (EC) No 1272/2008.

Ecology - air : Not included in the list of substances which may contribute to the greenhouse effect (IPCC). Not included in the list of fluorinated greenhouse gases (Regulation (EU) No 517/2014). Not classified as dangerous for the ozone layer (Regulation (EC) No 1005/2009).

Ecology - water : Not harmful to crustacea. Not harmful to fishes. Inhibition of activated sludge. Not harmful to algae. Not harmful to plankton.

Acetone (67-64-1)	
LC50 fish 1	5540 mg/l (EU Method C.1, 96 h, Salmo gairdneri, Static system, Fresh water, Experimental value, Nominal concentration)

12.2. Persistence and degradability

Acetone (67-64-1)	
Persistence and degradability	Biodegradable in the soil. Biodegradable in the soil under anaerobic conditions. Readily biodegradable in water.
Biochemical oxygen demand (BOD)	1.43 g O_2/g substance
Chemical oxygen demand (COD)	1.92 g O_2/g substance
ThOD	2.2 g O_2/g substance
BOD (% of ThOD)	0.872 (20 day(s), Literature study)

12.3. Bioaccumulative potential

Acetone (67-64-1)	
BCF fish 1	0.69 (Pisces)
BCF other aquatic organisms 1	3 (BCFWIN, Calculated value)
Log Pow	-0.24 (Test data)
Bioaccumulative potential	Not bioaccumulative.

12.4. Mobility in soil

Acetone (67-64-1)	
Surface tension	0.0237 N/m
Ecology - soil	No (test)data on mobility of the substance available.

12.5. Other adverse effects

Other information : Avoid release to the environment.

SECTION 13: Disposal considerations

13.1. Disposal methods

Waste disposal recommendations : Do not discharge into drains or the environment. Remove waste in accordance with local and/or national regulations. Hazardous waste shall not be mixed together with other waste. Different types of hazardous waste shall not be mixed together if this may entail a risk of pollution or create problems for the further management of the waste. Hazardous waste shall be managed responsibly. All entities that store, transport or handle hazardous waste shall take the necessary measures to prevent risks of pollution or damage to people or animals. Recycle by distillation. Incinerate under surveillance with energy recovery.

Additional information : Hazardous waste according to Directive 2008/98/EC, as amended by Regulation (EU) No 1357/2014 and Regulation (EU) No 2017/997.

Ecology - waste materials : Avoid release to the environment.

SECTION 14: Transport information

Department of Transportation (DOT)

In accordance with DOT

Transport document description : UN1090 Acetone, 3, II

UN-No.(DOT) : UN1090
Proper Shipping Name (DOT) : Acetone
Transport hazard class(es) (DOT) : 3 - Class 3 - Flammable and combustible liquid 49 CFR 173.120
Packing group (DOT) : II - Medium Danger
Hazard labels (DOT) : 3 - Flammable liquid

DOT Packaging Non Bulk (49 CFR 173.xxx) : 202
DOT Packaging Bulk (49 CFR 173.xxx) : 242
DOT Special Provisions (49 CFR 172.102) : IB2 - Authorized IBCs: Metal (31A, 31B and 31N); Rigid plastics (31H1 and 31H2); Composite (31HZ1). Additional Requirement: Only liquids with a vapor pressure less than or equal to 110 kPa at 50 C (1.1 bar at 122 F), or 130 kPa at 55 C (1.3 bar at 131 F) are authorized.
T4 - 2.65 178.274(d)(2) Normal............ 178.275(d)(3)
TP1 - The maximum degree of filling must not exceed the degree of filling determined by the following: Degree of filling = 97 / 1 + a (tr - tf) Where: tr is the maximum mean bulk temperature during transport, and tf is the temperature in degrees celsius of the liquid during filling.
DOT Packaging Exceptions (49 CFR 173.xxx) : 150
DOT Quantity Limitations Passenger aircraft/rail (49 CFR 173.27) : 5 L
DOT Quantity Limitations Cargo aircraft only (49 CFR 175.75) : 60 L
DOT Vessel Stowage Location : B - (i) The material may be stowed "on deck" or "under deck" on a cargo vessel and on a passenger vessel carrying a number of passengers limited to not more than the larger of 25 passengers, or one passenger per each 3 m of overall vessel length; and (ii) "On deck only" on passenger vessels in which the number of passengers specified in paragraph (k)(2)(i) of this section is exceeded.
Other information : No supplementary information available.

Transportation of Dangerous Goods

Transport document description : UN1090 ACETONE, 3, II
UN-No. (TDG) : UN1090
Proper Shipping Name (Transportation of Dangerous Goods) : ACETONE
TDG Primary Hazard Classes : 3 - Class 3 - Flammable Liquids
Packing group : II - Medium Danger
Explosive Limit and Limited Quantity Index : 1 L
Passenger Carrying Road Vehicle or Passenger Carrying Railway Vehicle Index : 5 L
Passenger Carrying Ship Index : Forbidden

Transport by sea

Transport document description (IMDG)	: UN 1090 acetone, 3, II
UN-No. (IMDG)	: 1090
Proper Shipping Name (IMDG)	: acetone
Class (IMDG)	: 3 - Flammable liquids
Packing group (IMDG)	: II - substances presenting medium danger
EmS-No. (1)	: F-E
EmS-No. (2)	: S-D

Air transport

Transport document description (IATA)	: UN 1090 Acetone, 3, II
UN-No. (IATA)	: 1090
Proper Shipping Name (IATA)	: Acetone
Class (IATA)	: 3 - Flammable Liquids
Packing group (IATA)	: II - Medium Danger

SECTION 15: Regulatory information

15.1. US Federal regulations

Acetone (67-64-1)	
Listed on the United States TSCA (Toxic Substances Control Act) inventory	
RQ (Reportable quantity, section 304 of EPA's List of Lists)	5000 lb
SARA Section 311/312 Hazard Classes	Immediate (acute) health hazard Fire hazard

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

Acetone (67-64-1)
Listed on the Canadian DSL (Domestic Substances List)

EU-Regulations

No additional information available

National regulations

Acetone (67-64-1)
Listed on the Canadian IDL (Ingredient Disclosure List)

15.3. US State regulations

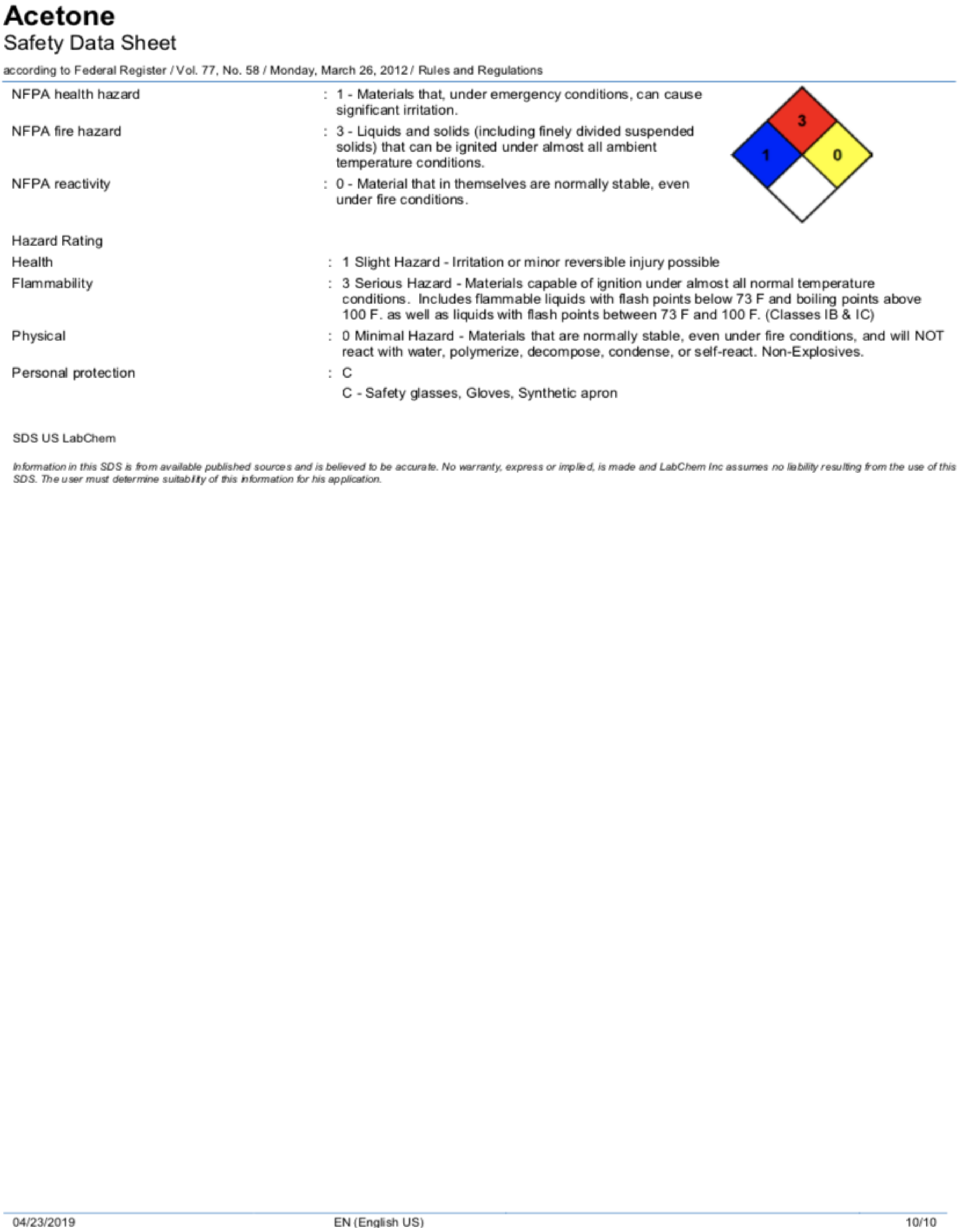
California Proposition 65 - This product does not contain any substances known to the state of California to cause cancer, developmental and/or reproductive harm

SECTION 16: Other information

Revision date	: 04/24/2018
Other information	: None.

Full text of H-phrases: see section 16:

H225	Highly flammable liquid and vapour
H319	Causes serious eye irritation
H336	May cause drowsiness or dizziness



Acetone

Safety Data Sheet

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

NFPA health hazard	: 1 - Materials that, under emergency conditions, can cause significant irritation.
NFPA fire hazard	: 3 - Liquids and solids (including finely divided suspended solids) that can be ignited under almost all ambient temperature conditions.
NFPA reactivity	: 0 - Material that in themselves are normally stable, even under fire conditions.

Hazard Rating

Health	: 1 Slight Hazard - Irritation or minor reversible injury possible
Flammability	: 3 Serious Hazard - Materials capable of ignition under almost all normal temperature conditions. Includes flammable liquids with flash points below 73 F and boiling points above 100 F. as well as liquids with flash points between 73 F and 100 F. (Classes IB & IC)
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	: C C - Safety glasses, Gloves, Synthetic apron

SDS US LabChem

04/23/2019 EN (English US) 10/10

Figure G-4: Material Safety Data Sheet: Acetone (LabChem, 2018)