BIOPETROL SYNTHESIZED FROM RUBBER SEED OIL THROUGH HETEROGENEOUS CATALYTIC CRACKING USING ZEOLITE CATALYST: EFFECT OF ACETONE IN SOLVENT EXTRACTION OF RUBBER SEED

DINESH RAJ THANIMALAY

UNIVERSITI MALAYSIA PAHANG

UNIVERSITI MALAYSIA PAHANG

BORANG PENGESAHAN STATUS TESIS

JUDUL: **BIOPETROL SYNTHESIZED RUBBER SEED OIL FROM THROUGH HETEROGENEOUS CATALYTIC CRACKING USING KAOLINITE AS CATALYST**

SESI PENGAJIAN: **2011/2012**

Saya **DINESH RAJ A/L THANIMALAY**

mengaku membenarkan kertas projek ini disimpan di Perpustakaan Universiti Malaysia Pahang dengan syarat-syarat kegunaan seperti berikut :

1. Hakmilik kertas projek adalah di bawah nama penulis melainkan penulisan sebagai projek bersama dan dibiayai oleh UMP, hakmiliknya adalah kepunyaan UMP.
2. Naskah salinan di dalam bentuk kertas atau mikro hanya boleh dibuat dengan kebenaran bertulis daripada penulis.
3. Perpustakaan Universiti Malaysia Pahang dibenarkan membuat salinan untuk tujuan pengajian mereka.
4. Kertas projek hanya boleh diterbitkan dengan kebenaran penulis. Bayaran royalti adalah mengikut kadar yang dipersetujui kelak.
5. *Saya membenarkan/~~tidak membenarkan~~ Perpustakaan membuat salinan kertas projek ini sebagai bahan pertukaran di antara institusi pengajian tinggi.
6. ** Sila tandakan (✓)

☐	SULIT	(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia seperti yang termaktub di dalam AKTA RAHSIA RASMI 1972)
☐	TERHAD	(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan).
☑	TIDAK TERHAD	

Disahkan oleh

(TANDATANGAN PENULIS)
Alamat Tetap**: 20 TKT RUSA 5, T.SELAMAT, 14000 BKT MERTAJAM, PULAU-PINANG.**

Tarikh: 16 JANUARI 2012

(TANDATANGAN PENYELIA)
EN SYAIFUL NIZAM BIN HASSAN

Tarikh: 18 JANUARI 2012

Saya akui bahawa saya telah membaca karya ini dan pada pandangan saya karya ini adalah memadai dari segi skop dan kualiti untuk tujuan penganugerahan ijazah Sarjana Muda Kejuruteraan Kimia.

Tandatangan :

Nama Penyelia : **EN SYAIFUL NIZAM BIN HASSAN**

Tarikh : **18 JANUARI 2012**

BIOPETROL SYNTHESIZED FROM RUBBER SEED OIL THROUGH HETEROGENEOUS CATALYTIC CRACKING USING ZEOLITE CATALYST: EFFECT OF ACETONE IN SOLVENT EXTRACTION OF RUBBER SEED

DINESH RAJ THANIMALAY

A thesis submitted in fulfillment of the
requirements for the award of the degree of
Bachelor of Chemical Engineering

Faculty of Chemical and Natural Resources Engineering
Universiti Malaysia Pahang

JANUARY 2012

DECLARATION

I declare that this thesis entitled *"Biopetrol Synthesized from Rubber Seed Oil through Heterogeneous Catalytic Cracking using Zeolite Catalyst: Effect of Acetone in Solvent Extraction of Rubber Seed"* is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :
Name : Dinesh Raj S/O Thanimalay
Date : 18 January 2012

To My Beloved Parents and Loved Ones

ACKNOWLEDGEMENT

Glory be to God, I thank God for all those blessings as I tend to finish this research with a great condition. This research, did gave me big impact in my life as I able to managed and hold the responsible to strive to the fullest effort. In preparing this thesis, I was in contact with many people including academician and practitioner. They have been contributed toward my knowledge and understanding in my project progress.

I am grateful and would like to express my sincere gratitude to my supervisor Mr. Syaiful Nizam Hassan for his germinal ideas, invaluable guidance, continuous encouragement and constant support in making this research possible. He has always impressed with his strong conviction for science and outstanding professional conduct.

My sincere thanks go to all my lab mates and members of the staff of the Chemical Engineering Department, UMP, who helped me in many ways and made my research work in an uninterrupted flow.

I acknowledge my sincere indebtedness and gratitude to my parents for their love, dream and sacrifice throughout my life. Acknowledgements for my roommates and friends for their support and technical aid upon completing this research. Also, not forgetting my dearly who always shower upon me her care and love from the beginning of this project, and also comforting me in times of trouble and upheavals. Lastly, I would like to acknowledge their comments and suggestions, which was crucial for the successful completion of this study.

ABSTRACT

Today scenario imposes great importance on the alternative of fossil fuel. In line with that is the development of biofuel derived from biomass. Common feedstocks for such process nowadays are corn, soya oil, palm oil etc. However, the rubber seed oil (RSO) can be extracted from its kernel to be derived as biopetrol. This method is much favarouble as the high concentration of fatty acids in rubber seeds. Also, rubber seeds are abundant and easily available throughout Malaysia. It will also be a secondary income for rubber plantation workers via collecting rubber seeds and selling it. The objectives of this experiment are; to synthesize isooctane form rubber seed oil using Zeolite as catalyst, to study the effect of solvent (Acetone) in the extraction of fatty acids from rubber seeds, and to analyze isooctane concentration through gas chromatography. Rubber seeds' kernel are cleaned, shelled, ground, blended, and dried. Extraction of fatty acids from rubber seeds are done by Soxhlet extraction method using Acetone as extraction solvent. The efficiency of solvent is analyzed by using different mass ratio between the solvents and rubber seeds kernel starting from 1:2 to 1:5. Rotary evaporator was used to evaporate the solvent, leaving behind crude rubber seed oil. The catalytic cracking of the mixture of 25ml of RSO and 5g of Zeolite catalyst at 350^0C for 45 minutes is to boost up the rate of reaction of breaking the long chains of fatty acids. The final product is analyzed through Gas Chromatography. Then the results of chromatograms are compared with the standard isooctane calibration curve. Through the calibration curve using backward calculation, the yield of biopetrol is determined. From this experiment the actual Isooctane concentration is about 53% to 78%. The result shows higher than expected result because of the factor of random reaction in catalytic cracking, hydrocarbon isomerization, high quantity of fatty acids in rubber seed oil which have been converted to Isooctane, and the small volume of sample analysis. This research can be further improved by the use of Supercritical CO^2 in extraction process, filling of inert gases or nitrogen in catalytic cracking chamber, elimination of impurities and minimization of human errors.

ABSTRAK

Senario hari ini mengenakan amat penting pada alternatif bahan api fosil. Sejajar dengan itu adalah pembangunan biofuel yang diperolehi dari biomass. Bahan utama biasa untuk proses itu kini adalah jagung, minyak soya, minyak sawit dan sebagainya. Walau bagaimanapun, minyak getah benih (RSO) boleh diekstrak daripada isirongnya yang akan disintesis sebagai biopetrol. Kaedah ini adalah lebih digemari oleh hal kerana kepekatan asid lemak dalam biji getah yang tinggi. Juga, biji getah yang banyak dan mudah didapati di seluruh Malaysia. Ia juga akan menjadi pendapatan tambahan bagi pekerja-pekerja ladang getah melalui memungut benih getah dan menjualnya. Objektif eksperimen ini adalah untuk sintesis isooctane daripada minyak benih getah menggunakan zeolit sebagai pemangkin, untuk mengkaji kesan pelarut (aseton) dalam pengekstrakan asid lemak daripada benih getah, dan untuk menganalisis kepekatan isooctane melalui kromatografi gas. Isirong benih getah dibersihkan, dan dibiarkan kering semalaman. Untuk mengeluarkan asid lemak daripada benih getah, perlu dilakukan melalui kaedah pengekstrakan Soxhlet menggunakan aseton sebagai pengekstrakan pelarut. Kecekapan pelarut dianalisis dengan menggunakan nisbah jisim yang berbeza di antara pelarut dan getah kernel benih bermula 1:2 hingga1:5. Penyejat Rotary telah digunakan untuk menyejat pelarut, meninggalkan minyak benih getah mentah. Keretakan pemangkin dilakukan dengan campuran 25ml RSO dan 5g zeolit pemangkin pada suhu 350^0C selama 45 minit adalah untuk meningkatkan kadar tindak balas memecahkan rantaian panjang asid lemak. Produk akhir dianalisis melalui Kromatograpi gas. Kemudian keputusan kromatogram dibandingkan dengan keluk penentukuran isooctane standard. Melalui keluk penentukuran menggunakan pengiraan mundur, hasil kepekatan yang sebenar biopetrol ditentukan. Daripada eksperimen ini kepekatan Isooctane sebenar adalah kira-kira 53% kepada 78%. Hasilnya menunjukkan lebih tinggi daripada hasil yang dijangka kerana faktor reaksi rawak dalam retak sebagai pemangkin, pengisomeran hidrokarbon, kuantiti yang tinggi asid lemak dalam minyak biji getah yang telah ditukar kepada Isooctane, dan kelantangan kecil analisis sampel. Kajian ini boleh terus diperbaiki dengan penggunaan kaedah Supercritical CO_2 dalam proses pengekstrakan, mengisi gas lengai atau nitrogen di dalam kebuk pemangkin retak, penghapusan kekotoran, dan mengurangkan kesilapan manusia.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

P	-	Pressure
m	-	Mass
ΔH	-	Enthalpy change of reaction
ΔS	-	Entropy change of reaction
ΔG	-	Energy change of reaction
T	-	Temperature
ρ	-	Density
μ	-	Viscosity of liquid (Pa.s)
h	-	Heat transfer coefficient
^{o}C	-	Degree Celsius
g	-	Gram
kg		Kilogram
K	-	Degree Kelvin
m	-	Meter
ml	-	Mililiter
L	-	Liter

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 FUEL CONSUMPTION SCENARIO

The recent increases in crude oil prices have created unprecedented opportunities to displace petroleum-derived materials with biofuel. Biofuel, as an alternative fuel, has many merits and made from renewable biological sources such as vegetable oils and animal fats. It is biodegradable and nontoxic has low emission profiles and so it is environmentally beneficial. Apart from that, biofuel is a renewable fuel, helping to achieve the EU renewable energy target of 12% of total energy output to consist of renewable energy by 2010 (European Commission, 1997). Carbon dioxide produced by combustion of biofuel can be recycled by photosynthesis, thereby minimizes the impact of biofuel combustion on the greenhouse effect (KEorbitz, 1999; Agarwal and Das, 2001). Additionally biofuel has a relatively high flash point (150 °C), which makes it less volatile and safer to transport or handle than petroleum diesel (Krawczyk, 1996).In support of this increasing consumption there have been substantial increases in biofuel production in recent years, a trend that is expected to continue. The EIA (Energy Information Administration) foreseeable that demand for biofuel will be at least 6.5 million gallons in 2010 and 7.3 million gallons in 2020.

Based on biofuel's potential, demand could reach as much as 470 million gallons in 2010 and 630 million gallons in 2020. Now the major producer of biofuel in the world is Germany. It has produced 2539 million ton in 2009 whilst Malaysia nearly exported 76 million gallons of biofuel in 2009. This growth is the result of the construction of new production plants and the expansion of existing ones.

In early 80's a petroleum company from Brazil produced biopetrol with a mixture of 10% vegetable oil has been used in pre-combustion chamber engines to maintain total power without any alterations or adjustments to the engine. At that point, it was not practical to substitute 100% vegetable oil for diesel fuel, but a blend of 20% vegetable oil and 80% diesel fuel was successful. Some short-term experiments used up to a 50/50 ratio also provide good solution for well maintain engine (Fangrui et al., Ma, 1998). This advantage engine adaptability to vegetable oil mixture with diesel makes an enriching step towards the feasibility of engine combustion with nominal effects using biopetrol. The table below shows the comparison between biopetrol and fossil fuel.

Table 1.1: Comparison between Biopetrol and Fossil Fuel

Aspect	**Biopetrol**	**Fossil fuel**
Greenhouse gas emission and environmental issue	Sustainable and biodegradable Reduces greenhouse gas emission Environmental friendly	Not biodegradable Contains a large amount of sulphur and oxygen which causes incomplete combustion which emits greenhouse gases Pollutes the environment
Energy security	Renewable energy source which can be produced from biomass	Non- renewable energy source and its depleting at a faster rate
Engine performance	Increases engine life Improves performance and efficiency in the combustion process	Incomplete combustion in the engine Leads to accumulation of particles in engine

Economy	Creates new job opportunies Increases the agricultural demand for biomass for to produce biofuel	Extraction of fossil fuel has high production cost The reduce in fossil fuel sources causes fossil fuel price to raise

1.2 BACKGROUND OF STUDY

Alternative fuels for transportation are becoming increasingly important due to diminishing petroleum reserves and the environmental consequences of exhaust gases from petroleum-fuelled engines. The energy source, fossil fuel, upon which we have come to rely on so deeply, is in higher demand than ever before, which more energy is needed all around to fulfill this demand. Fossil fuel alone seems to be insufficient to cater to the needs of the global community. In light of this, it is in the world's best interest to devote a substantial amount of resources towards alternative forms of energy. Biofuel, as biodiesel in this context, is at the forefront of these alternatives due to its ability to fuel conventional gasoline engines with minimum or no modifications, as well as form blends with fossil diesel.

Since biodiesel synthesis and production has proven to be successful, so another kind of biofuel should be synthesized and developed. Biopetrol seems to be a better alternative where majority of vehicles used in Malaysia are petrol based vehicle. In most of the researches regarding biopetrol, the most preferable choice of synthesis is done by using fatty acid as starting material through heterogeneous catalytic cracking. In this study, the biopetrol is material fatty acid extracted from rubber seeds via Soxhlet method and the synthesis is carried out through catalytic cracking using Zeolite as catalyst.

Biopetrol is an environmentally friendly alternative liquid fuel. There has been renewed interest in the use of vegetable oils for making biopetrol due to its less polluting and renewable nature as against the conventional petroleum diesel fuel. The

biggest difference between biofuels and petroleum feedstock is oxygen content. Biofuels have oxygen levels from 10% to 45% while petroleum has essentially none making the chemical properties of biofuels very different from petroleum.

1.3 PROBLEM STATEMENT

Due to the fact that the supply of fossil fuel is limited while energy demand continues rise, biofuel has been introduced as alternative renewable energy. The International Energy Outlook, an annual forecast by the U.S. Energy Information Administration forecast due to the driven by population and economic growth in developing countries, the world in 2035 would be more dependent on fossil fuels than ever, it finds. Countries overall would be consuming 49 percent more energy and spewing 43 percent more carbon dioxide into the atmosphere in 2035 than in 2007. To get rid from this problem, biofuel plays essential role in manner to reduce the emissions of carbon dioxide and reduce the consumption fossil fuel.

By using Zeolite as heterogeneous catalyst in this study would make the separation process easier and it also recyclable. The employment of homogeneous catalyst in the production of biodiesel has brought several disadvantages cause the catalyst can be dissolved in the methanol; the separation of catalyst from the product would be difficult. Thus, the manufacturing cost increases. Thus, the most feasible catalyst can be used in this study is Zeolite. This is due to the fact that aside from giving relatively high yield, it would monumentally reduce the length of processing time needed for production, and this would go well to supply the ever increasing rate of demand for alternative liquid fuel. With catalytic cracking, the biopetrol production industry in Malaysia would be able to cater to the needs of Malaysians at a faster rate, thereby eliminating the need for any dependence on foreign alternative fuel that may arise in the future. Malaysia would be able to deal with its own fuel crisis, at an optimal rate using its abundant feedstock resources.

1.4 RESEARCH OBJECTIVES

a) To extract fatty acid from rubber seeds kernel using Acetone as extraction solvent.

b) To synthesize isooctane from rubber seed oil through heterogeneous catalytic cracking using Zeolite as catalyst.

1.5 SCOPE OF STUDY

This study carried out by using rubber seed oil with Zeolite as base heterogeneous catalyst for the catalytic cracking reaction in lab scale reactor. In this study the following criteria have been given focus:

a) The extraction of fatty acid from rubber seeds using acetone as solvent via Soxhlet extraction.
b) Application of catalytic cracking method to crack the fatty acid complex molecule into smaller hydrocarbon molecules i.e. isooctane which is the most preferable.
c) Determination of the amount and concentration of isooctane using via an analysis using gas chromatography method.

1.6 RATIONALE & SIGNIFICANCE

The rationale and significance for this study are as below:

a) Biopetrol source which is rubber seed can be obtain in vast number because Malaysia has more than 1.7 million hector of rubber plantation.
b) Higher conversion of rubber seed oil would be achieved by catalytic cracking than thermal cracking by lowering the activation energy of the reaction.
c) Biopetrol is sulfur-free fuel and able reduce the emission of green emission gas thus making it environment friendly.
d) The cost of manufacturing will be reduced due to the simple process which is the purification process.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Finite petroleum reserves and the increasing demands for energy in industrial countries have created international unease. For example, the dependence of the United States on foreign petroleum both undermines its economic strength and threatens its national security. As highly populated countries such as China and India become more industrialized, they too might face similar problems. It is also clear that no country in the world is untouched by the negative environmental effects of petroleum extraction, refining, transportation and use. For these reasons, governments around the world are increasingly turning their attention to biofuels as an alternative source of energy.

Apart from the energy withdrawal with hydroelectricity and nuclear energy, the majority of the worlds energy needs are supplied through petrochemical sources, coal and natural gas. As these sources are finite and non renewable, at current usage rates will be consumed by the end of the coming century (Aksoy, 1990). In recent development, interest has arisen in the alternative sources for petroleum-based fuels which are with due concern of the depletion of world petroleum reserves and increased environmental concerns.

The use of vegetable oils as alternative fuel has been around since last century when the inventor of the diesel engine Rudolph Diesel first tested them, in his compression engine (Foglia, Jones,Haas, & Scott, 2000). Alternatively name for biofuel is oxygenated fuel, meaning that it emit low amount of carbon to the environment because it's contain higher hydrogen and oxygen than carbon (Armas *et al*., 2008). Further supporting information, the sulphur contents of vegetable oils are close

to zero and consequently, the environmental damage caused by sulphuric acid effects is reduced (Vicente *et al.*, 1998)

2.2 Fossil Fuel and Biofuel as Promising Alternative

Energy is an important factor of production in the global economy, and 90% of the commercially produced energy is from fossil fuels such as crude oil, coal, and gas, which are non-renewable in nature. Much of the energy supply in the world comes from geo-politically volatile economies. In order to enhance energy security, many countries, including the developed countries, have been emphasizing production and use of renewable energy sources such as biofuels, which is emerging as a growth industry in the current economic environment.(Thiam Leng,2006)

Nations like Brazil, the US, the European Union as well as many other countries around the world has given high priority for biofuels, due to concerns of oil dependence and interest in reducing CO2 emissions. All these regions have come up with significant subsidies for renewable energy production from agricultural sources. The impacts of these subsidies reach far beyond the borders of these economies. The global and sectoral implications of biofuel programs on agricultural markets and land use across the world seems much positive and gained significant response form the mass consumers. The very nature of biofuels production as a global economic activity affecting the pattern of energy demand and resource has motivated this study to initiate in the first place. When biofuels are mass produced and their usage is much preferred by consumers, the resulting impact of biofuel drives on output, prices, trade, land-use change, commodity price index, and job market.(Subash,Pramila 2006)

2.3 OCTANE NUMBER RATING FOR PETROL QUALITY

The quality of a fuel is measured with its 'octane number'. A good quality fuel has a good octane number. Octane number measures whether petrol is likely to cause knock in an engine. Knocking is caused by self-ignition in the engine's cylinders, which happens when the petrol/air-vapour mixture in the cylinder ignites before the plug sparks. This premature ignition pushes against the crankshaft instead of being with it, and produces a knocking or pinging sound. Knocking causes the engine to overheat and lose power, and it can damage the engine in the long run. In a properly functioning engine, the charge burns with the flame front progressing smoothly from the ignition point across the combustion chamber. Sometimes, at high compression ratios, depending on the composition and quality of the fuel, some of the charge may spontaneously ignite ahead of the flame front and starts to burn in an uncontrolled pattern. This will result in intense high frequency pressure waves followed by sharp sound which is actually caused by the premature combustion. (Reza Sadeghbeigi, 2000).

Octane rating crucial cause it reflects the quality, purity, refinement, efficiency and heat bearing capacity of petrol. It plays a role where it measures the ability of a fuel to resist knocking when ignited in a mixture with air in the cylinder of an internal combustion engine. As set by the petroleum industry, the octane number is determined by comparing, under standard conditions, the knock intensity of the fuel with that of blends of two reference fuels; isooctane which resists knocking and heptanes which knocks readily. In practical wise, the octane number is the percentage by volume of the isooctane in the fuel mixture of isooctane-heptane that similar to the fuel being tested in a standard engine. Normally, high Octane fuels are expensive because of high levels of refinement.(Agustin et al,2008)

The significant amount of isooctane in the fuel plays a defining role in choosing a reliable and engine safe which in turn provides the smoothness of ride and longevity of engine. Isooctane or in the standard form 2,2,4-trimethylpentane, is an octane monomer which defines the octane rating. This organic compound is highly branched that burns well with very little or negligible knock. By comparison, heptane is a straight, unbranched molecule with an octane rating of zero due to its high knocking properties.

In this case of study, biopetrol contains isooctane as its main constituent which gives it the characteristic to prevent knocking in the internal combustion engines. Meanwhile, the high concentration of oxygen in the biopetrol allows the engine to have much lower temperature combustion as well as reducing the emission of greenhouse gases such as carbon monoxide, and nitrogen oxides.

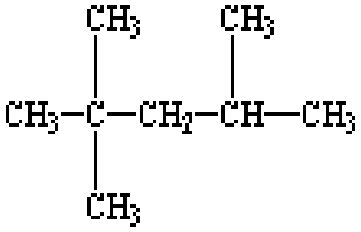


Figure 2.1: Chemical structure of Isooctane

Table 2.1: Physical & Chemical properties of Isooctane

Synonyms	Isobutyltrimethylpentane, 2,2,4-Trimethylpentane
Appearance	colorless liquid
Molecular formula	C_8H_{18} or $CH_3C(CH_3)_2CH_2CH(CH_3)CH_3$
Molecular weight	114.22 g/mol
Melting point ^{0}C	-107.38°C (166K, -161^{0}F)
Boiling point ^{0}C	99.3°C (372K, 211^{0}F)
Density	688 kg/ m^3
Specific gravity	0.692
Solubility in water	Immiscible
Auto ignition temperature:	396°C

Source: Safety Data for Isooctane,2005

2.4 INNOVATION OF BIODIESEL

Fossil fuel alone seems to be insufficient to cater to the needs of the global community. In light of this, it is in the world's best interest to devote a substantial amount of resources towards alternative forms of energy. Biofuel, as biodiesel in this context, is at the forefront of these alternatives due to its ability to fuel conventional diesel engines with minimum or no modifications, as well as form blends with fossil diesel.

Biodiesel is defined as fatty acid methyl esters prepared from any kind of feedstock including vegetable oils, animal fats, single cell oils, and waste material. Fatty acid ethyl esters can also be defined as and used to produce biodiesel. However, due to the relatively high price of ethanol compared to methanol, the use of ethyl esters has not so far been established. The preparation of fatty acid methyl esters can be achieved by a process called transesterification, which is the exchange of alcohol or acid moiety of an ester. (A.S.Ramadhas,2005)

Alcoholysis is the transesterification of an ester with an alcohol, in which methanolysis is the term used in the case of methanol. The reaction requires a catalyst, usually a strong base, such as sodium or potassium hydroxide, and produces new chemical compounds called methyl esters. It is these esters that have come to be known as biodiesel. Because its primary feedstock is a vegetable oil or animal fat, biodiesel is generally considered to be renewable. Since the carbon in the oil or fat originated mostly from carbon dioxide in the air, biodiesel is considered to contribute much less to global warming than fossil fuels. Diesel engines operated on biodiesel have lower emissions of carbon monoxide, unburned hydrocarbons, particulate matter, and air toxics than when operated on petroleum-based diesel fuel.

All feedstocks that contain fatty acids or glycerol can be used for biodiesel production including rubber seed oil, jatropha *curcas* oil, and papaya oil. In European countries, rapeseed oil is used due to its widespread availability. Soybean oil is used in the Unites States of America, while palm oil is used widely in tropical regions such as Malaysia. The use of methyl esters as fuel requires a low proportion of saturated fatty

acids in order to make the fuel function at low temperatures. In colder climates, rapeseed oil and olive oil have proven to be one of the best options. The usage of palm oil is ideal in Malaysia due its abundant availability as well as its suitability in warm climates. Palm oil can also be used as blends with other types of oil. Feedstock chosen is also influenced by national and international specifications of biodiesel that need to be fulfilled.(O.E.Ikwuagwu,2000)

2.5 BIOPETROL FROM FATTY ACIDS IN RUBBER SEEDS

Christopher Columbus is who the founder of rubber in tropical South America around 1500. Hevea brasiliensis, the common variety of rubber tree produces 99% of world's natural rubber. The seeds contain an oily endosperm. Generally, 37% by weight of the seed is only shell and the rest is kernel. The oil content of air dried is 47%.

Table 2.2: Fatty Acid Compositions in Rubber Seed Oil

Fatty acid composition (%)	Rubber seed oil
Palmitic acid	10.2
stearic	8.7
Oleic	24.6
Linoleic	39.6
Linolenic	13.2

Source: et al Shankaransh Srivastava, 2000

Fatty acid is a long unbranched aliphatic tail (chain) carboxylic acid, which is either saturated or unsaturated. Fatty acids can be saturated (acetic, butyric, palmitic acids), monosaturated (oleic acid), or polysaturated (linoleic, linolenic, arachidonic acids). Triglycerides from various vegetable oils give through transesterification a mixture of fatty acid esters which is now used increasingly as a substitute of diesel fuel and is named bio-diesel. Natural fatty acids are aliphatic monocarboxylic acids derived from, or contained in esterified form in an animal or vegetable fat, oil or wax. Natural fatty acids commonly have a chain of four to 28 carbons (usually unbranched and even

numbered), which may be saturated or unsaturated (Aigbodion A.I. and C.K.S., Pillai, 2000).

Figure 2.2: Fatty acids

Shortly, the production of bio-petrol from fatty acids obtained from rubber seeds provides energy security in terms of domestic targets, supply reliability, reducing the dependability of fossil fuels, domestic distribution, ready availability and renewability. Besides that, the bio-petrol has proven its advantages over the other traditional fossil fuels by its multi characteristics. There is no sulphur molecule in its molecular structure and it is derived from biomass and thus, brings about the reduction of greenhouse gasses emission. Also, it is biodegradable and non-toxic, therefore it possesses the potential of being environmental friendly. It has the potential to enhance the performance, efficiency and life of engines as it contains isooctane that helps to prevent knocking. Despite that, the production of bio-petrol using rubber seeds will help to generate new job opportunities and new businesses to alleviate property that leads to a better, more stable economy in the country. The production of bio-petrol from rubber seed oil also will not lead to the detriment of food supply because rubber seeds are non-food crops. It has lower impact on marine environment as the water pollution can be reduced by using bio-petrol in the boat engines since there will be more efficient burning of the fuel mixture, less carbon accumulation and particulate emissions. Faster starting and smoother operation of engines by using bio-petrol could reduce the discharge of unburned fuel. Finally, any accidental discharges of small amount of bio-petrol have

relatively low impact on the environment compared to petroleum, which contains more toxic and more water soluble aromatics.

2.6 CATALYTIC CRACKING

Breaks the complex hydrocarbons into simpler molecules in order to increase the quality and quantity of lighter more desirable products and decrease the amount of residuals is known as catalytic cracking. This process rearranges the molecular structure of hydrocarbon compounds to convert heavy hydrocarbon feedstock into lighter fractions such as kerosene, gasoline, liquified petroleum gas (LPG), heating oil, and petrochemical feedstock.

The catalysts used in refinery cracking units are typically solid materials such are zeolite, aluminum hydrosilicate, treated bentonite clay, fuller's earth, bauxite, and silicaalumina. Normally these catalysts are in the form of powders, beads, pellets or shaped materials called extrudites.(Jeanne Mager Stellman, 1998)

In the production of the bio-petrol using rubber seeds, the main component to be made into bio-petrol is actually the fatty acid. Like what have been discussed earlier, fatty acid is a long unbranched aliphatic chain carboxylic acid. The complex structure of the fatty acid is very hard to react with any other substances. Therefore, for any reactions to occur, the fatty acid has to be broken into simpler chain molecules.

For this study, heterogeneous catalytic cracking is applied. Catalytic cracking is a route to produce liquid fuels that contain linear and cyclic paraffins, olefins, aldehydes, ketones, and carboxylic acid. Catalytic cracking process has been widely used in oil industry. It is widely used to convert the high-boiling, high-molecular weight hydrocarbon fractions of petroleum crude oils to more valuable gasoline, olefinic gases and other products.(James H. Gary and Glenn E. Handwerk, 2001)(James. G. Speight, 2006) The petroleum refinery process in which heavy oil is passed through metal chambers (called catalytic crackers or cat crackers) under pressure and high temperature in the presence of catalysts. This boiling breaks up heavy, large and more complex long-chain oil molecules into lighter, smaller, and simpler short-chain molecules by the

breaking of carbon-carbon bonds in the precursors. The rate of cracking is dependent on the temperature and pressure applied as well as the presence of the catalyst. Temperature, heating rate, residence time and type of catalyst choice are important process control parameters (Ni et al., 2006).

Zeolite is used as an acid catalyst because it is very well known for its adsorbent qualities and has been used successfully in scientific trials of many chemical adsorptions. This characteristic of zeolite is very important because as it helps to boost up the rate of reaction for the production of bio-petrol and it provides more surfaces for reactions to occur.

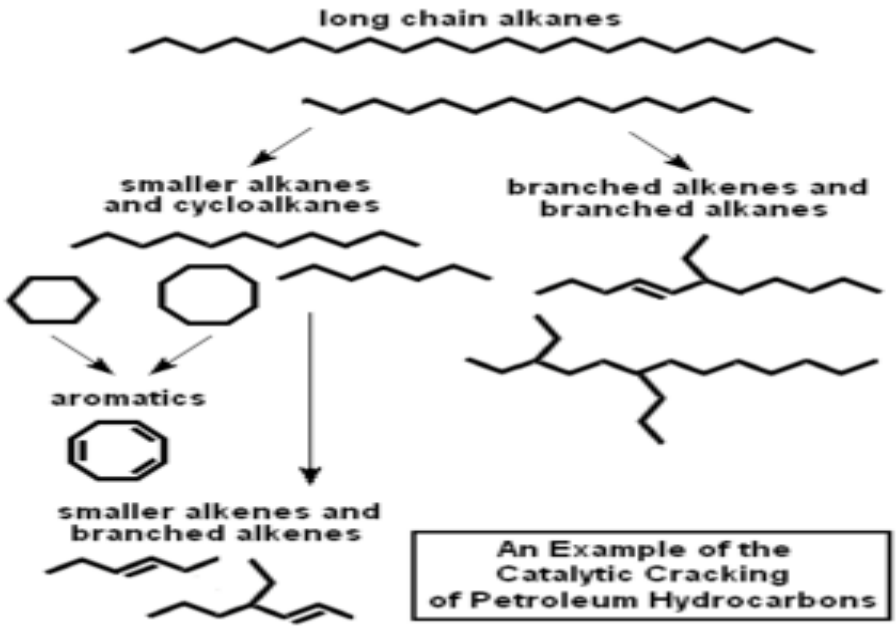


Figure 2.3: An example of catalytic cracking of Petroleum Hydrocarbons

2.7 ZEOLITE AS CATALYST

Zeolites have been an important material in catalysis and other applications such as adsorbents and detergents for the last 50 years. While new types of porous materials like ordered mesopores, metal organic frameworks are now attracting more R&D attention; zeolites are still the most important crystalline industrial catalysts.

Biofuel can produce from abundant biomass. To remove the abundant oxygen content from biomass- and convert it into a hydrophobic molecule with the appropriate combustion or chemical properties by using zeolites and mesoporous compounds to develop new biofuel generation processes. The development of new catalysts in the field of conversion of biomass to biofuels requires knowledge of the complex nature of the substrates to be converted. Starting from the main chemical aspects of the different biomass platforms, an overview of some of the zeolite and mesoporous materials technologies currently used commercially or tested at pilot and laboratory scale. (et al., Carlo Perego**,** 2010**)**

By using microporous HZSM-5 zeolite, mesoporous MCM-41, and composite micromesoporous zeolite as catalysts to produce biofuel from palm oil. The products obtained were gas, organic liquid product, water, and coke. The organic liquid product was composed of hydrocarbons corresponding to gasoline, kerosene, and diesel boiling point range. The maximum conversion of palm oil which was 99 wt% achieved when reaction temperature of 450°C, and a weight hourly space velocity of 2.5 h^{-1}. (OY Sang - Energy Sources)

Table 2.3: Properties of Zeolite

Product identification	
CAS No	1318-02-1 or 68989-22-0
Other names	Valfor (R) 100 Zeolite NaA; Sodium Aluminosilicate; Zeolite Type A
Chemical formula	$Na_2O.Al_2O_3.xSiO_2.yH_2$
Physical properties	
Appearance	White solid
Odor	odourless
Specific Gravity	>1 (Water = 1)
Chemical properties	
Solubility in water	Insoluble in cold water, hot water.
Hazard	
Potential Acute Health Effects:	Slightly hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation.

2.8 SOXHLET EXTRACTION

Extraction process is widely used in chemical engineering field of study as well as in the industries. The common use of this process is to extract some essentials form raw material and also to separate the desired product from a process. Liquid-liquid extraction, also known as solvent extraction and partitioning, is a method to separate compounds based on their relative solubility in two different immiscible liquids. It is an extraction of a substance from one liquid phase into another liquid phase. Liquid-liquid extraction is a basic technique in chemical laboratories, where in this study Soxhlet method is being used.

Soxhlet extractor is a piece of laboratory apparatus originally meant for the extraction of a lipid from a solid material. However, a Soxhlet extractor is not limited to the extraction of lipids. Typically, a Soxhlet extraction is required to extract substances with a low solubility in the extracting solvent, and the impurity is insoluble in that solvent. If the desired compound has a significant solubility in a solvent then a simple filtration can be used to separate the compound from the insoluble substance. The extraction process is carried out whereby; a solid material containing some of the desired compound (such as oil) is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the extractor. The Soxhlet extractor is connected onto a boiling flask containing the extraction solvent and a condenser is equipped above the extractor. The boiling flask should not be overfilled and the volume of solvent should be two to four times the volume of the Soxhlet chamber.

The solvent is heated or boiled to reflux. The standard extractor has a distillation arm where the solvent vapor travels up to, and floods into the chamber housing the thimble of solid. The solvent vapor then condenses as it is cooled by the help of a condenser, and drips back down into the chamber housing the solid material. The extractor chamber containing the thimble filled with solid material slowly fills with warm solvent. Some of the desired compound will then dissolve in the warm solvent. Once the solvent reaches the top of the siphon side arm, the chamber is automatically emptied with the solvent running back down to the distillation flask. The solvent is reheated; and the cycle may be allowed to repeat many times, over hours or days. During each cycle, there will be a portion of the non-volatile compound dissolves in the solvent. After many cycles the desired compound is concentrated in the distillation flask. . Furthermore, after the extraction, the apparatus is allowed to cool down before the solvent is removed, typically by means of a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble, and is usually discarded.

Traditionally Soxhlet method was originally used to determine fat in milk (F. Soxlet, 1879). The sample is placed in a thimble holder that is gradually filled with condensed fresh extractant (term used to refer to the solvent used for extraction) from a distillation flask (see figure 3). When the liquid reaches the overflow level, a siphon

aspirates the solute from the thimble-holder and unloads it back into the distillation flask, thus carrying the extracted analytes into the bulk liquid. This operation is repeated until extraction is complete. Operationally, Soxhlet extraction is thus a continuous–discrete technique. In fact, since the extractant acts stepwise, the assembly operated as a batch system; however, solvent is recirculated through the sample, so the system also operates in a continuous manner somehow.

Conventional Soxhlet extraction has some attractive advantages. The sample is repeatedly brought into contact with fresh portions of solvent, which facilitates displacement of the transfer equilibrium. Also, the system remains at a relatively high temperature by effect of the heat applied to the distillation flask reaching the extraction cavity to some extent. In addition, no filtration is required after leaching and sample throughput can be increased by performing several simultaneous extractions in parallel, which is facilitated by the low cost of the basic equipment. Moreover, Soxhlet extraction is a very simple methodology that requires little training and seemingly subject to no matrix effects – this assertion is not strictly true as seen when Soxhlet extraction is compared with supercritical fluid extraction of analytes strongly bound to their matrix (M.D. Luque de Castro *et al*, 1994).

This technique also poses some serious drawbacks as compared to other techniques for solid sample preparation are the long time required for extraction and the large amount of solvent wasted, which is not only expensive to dispose off, but also the source of additional, environmental problems (M.D. Luque de Castro and F. Priego-Capote, 2009). Samples are usually extracted at the solvent boiling point over long periods, which can result in thermal decomposition of thermolabile target species. Also, a conventional Soxhlet device provides no agitation, which would help to expedite the process. In addition, the large amounts of solvent used call for an evaporation–concentration step after extraction. Finally, the Soxhlet technique is limited by solvent and difficult to automate.

Conventional Soxhlet extraction, with its advantages and shortcomings, has been used as starting point for the development of a variety of modifications intended to alleviate or suppress the latter while keeping or even improving the former. Most of the

modifications reported over the last few decades have been aimed at bringing Soxhlet closer to that of the more recent techniques for solid sample preparation, by shortening leaching times with the use of auxiliary forms of energy and automating the extraction assembly.

The advantages of using this system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled. This technique is particularly useful of which the Soxhlet Extractor enabling the isolation of the desired compound where the compound has only a limited solubility in a solvent, and the impurity is insoluble in that solvent. In addition, the working principle of mechanism is so simple that one can obtain more desired compound without any difficulties.

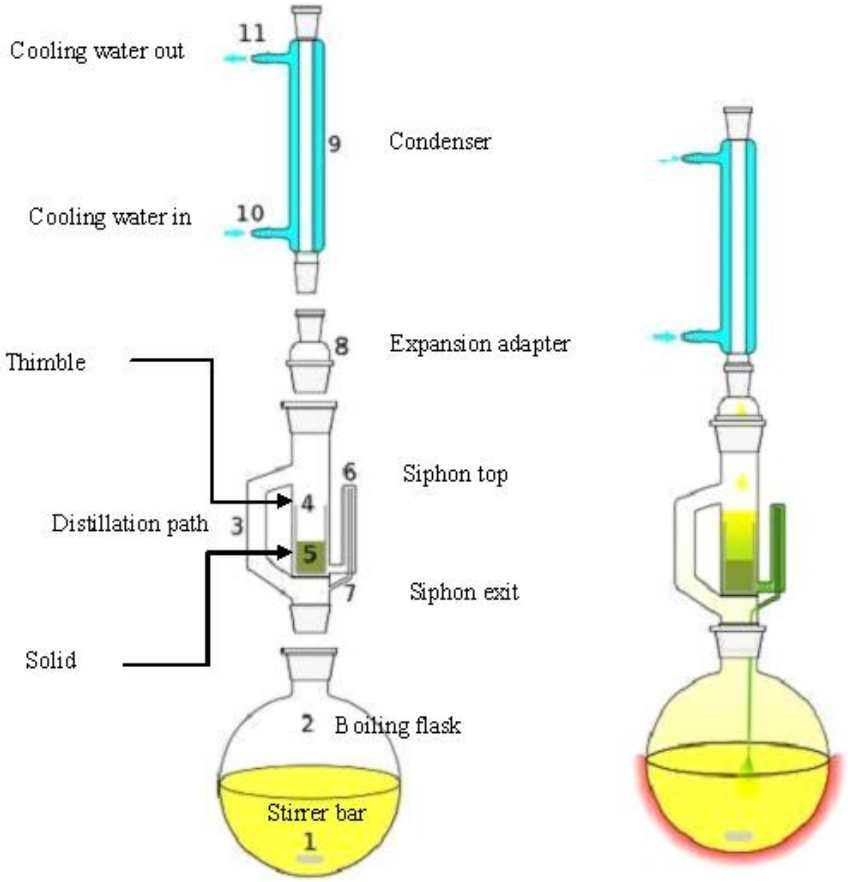


Figure 2.4: Soxhlet Extractor

2.9 ROTARY EVAPORATOR

Evaporation is a type of vaporization of a liquid that occurs only on the surface of a liquid. The other type of vaporization is boiling, which, instead, occurs on the entire mass of the liquid. Liquid commonly undergoes a natural process of evaporation in any given environment. In industry, evaporation is major part of separation process, where this also applies in the laboratory study as well. In this study, rotary evaporation technique is being used to remove the solvent form the essential oil for the further downstream process.(Silberberg Martin ,2006)

Rotary evaporator is widely used in chemical laboratories for remove the solvent from the reaction mixture efficiently by evaporation. Use of a rotary evaporator allows liquid solvents to be removed without excessive heating of what are often complex and sensitive solvent-solute combinations. Large quantities of solvent can be removed efficiently by using rotary evaporator. The table below shows the main components and function of a modern rotary evaporator.

Table 2.4: Main Components and Function of Rotary Evaporator

Components	Function
Motor	Helps to rotate the vessel or flask
Rotating vessel (evaporation flask)	Contain of sample
A vapor duct	Acts as the axis for sample rotation, and as vacuum tight conduit for the vapor being drawn off to the sample
Vacuum system	Constantly reduce the pressure within the evaporator system
Tube	Connected just below the condenser to release the pressure (in case if any
A heated fluid (can be water or oil) bath in either a metal container or crystallization dish	To heat the sample being evaporated to a temperature higher than their boiling point
A condensate (solvent) receiver flask at the bottom of the condenser	To collect the distilling solvent after it re-condenses for easy reuse or disposal

Figure 2.5: Rotary Evaporator

By partial emersion rotating flask is heated in a hot fluid bath. The flask's rotation provides improved heat transfer to the contained fluid mixture, as well as to reduce the occupancy of "bumps" caused by superheating of the liquid. In addition, since the flask is rotated during the evaporation process, the surface area is larger than normal which increases the evaporation rate significantly. To ensure that all the solvent to be evaporate, the temperature of the fluid bath is adjusted to a temperature higher than the boiling point of the solvent. Besides that, this method also avoids overheating of the target compound that is oxidation because lower temperatures are used. For solvent with boiling points < 100^0C, water bath is used and oil bath for solvent temperature >100^0C. Upon reaching the fixed temperature, the solvent will evaporates and the solvent vapors leave the flask by the connecting tube and are condensed in the condenser section. In the meanwhile, the cooling water is always flowed in and out of the system to ensure the cooling system is functioning to prevent overheating. The joints of the apparatus parts will be applied with a little amount of vacuum grease to allow air to flow in.(Kuk.M.S.1998)

In the laboratory, the vacuum line, a circulation bath or a membrane pump are used as source for the vacuum (40-50 mmHg). The fact that a vacuum is usually applied to the setup means that the boiling points of the solvents are going to be significantly lower than at ambient pressure (see table below).

Table 2.5: Boiling point of certain solvents at mentioned pressure

Solvent	**b.p. (760 mmHg)**	**b.p. (40 mmHg)**
acetonitrile	81.8 ^{o}C	7.7 ^{o}C
diethyl ether	34.6 ^{o}C	-27.7^{o}C
ethanol	78.4 ^{o}C	19 ^{o}C
ethyl acetate	77.1 ^{o}C	9.1 ^{o}C
hexane	68.7 ^{o}C	-2.3 ^{o}C
heptane	98.4 ^{o}C	22.3 ^{o}C
methanol	64.7 ^{o}C	5.0 ^{o}C
water	100 ^{o}C	34.0 ^{o}C

2.10 ACETONE AS SOLVENT IN SOXHLET EXTRACTION

Mostly acetone has been used as a solvent in laboratory and industries. Acetone is the organic compound with the formula $(CH_3)_2CO$. It also colourless and flammable liquid. Acetone is the organic compound with the formula $(CH_3)_2CO$. This colorless, mobile, flammable liquid is the simplest example of the ketones. Acetone is miscible with water and typically chosen as the solvent of choice for cleaning purposes in the laboratory. About 5.1 million tonnes were produced worldwide in 2009, mainly for use as a solvent and production of methyl methacrylate and bisphenol A. Common household uses of acetone are as the active ingredient in nail polish remover and as paint thinner. It is a common building block in organic chemistry.

Naturally, acetone is produced and disposed of in the human body as a result of normal metabolic processes. In fact, the body naturally increases the level of acetone in pregnant women, nursing mothers and children because their higher energy requirements lead to higher levels of acetone production. Ketogenic diets that increase acetone in the body are used to reduce epileptic attacks in infants and children who suffer from recalcitrant refractory epilepsy.

Taking a study where Acetone is being used in extraction it shows much advantage it as solvent or co-solvent with other compound. Using Cottonseed flakes were extracted with mixtures of *n*-hexane and acetone, with the concentration of acetone varying between 10 and 75%. Adding small amounts of acetone (25%) to *n*-hexane significantly increased the extraction of free and total gossypol from cottonseed flakes. Sensory testing detected no difference in the odour of cottonseed meals produced either by extraction with 100% *n*-hexane or by extraction with a 10:90 (vol/vol) mixture of acetone/hexane. More than 80% of the free gossypol was removed by the 10:90 mixture of acetone/hexane, whereas pure *n*-hexane extracted about 47% of the free gossypol from cottonseed flakes. A solvent mixture containing 25% acetone removed nearly 90% of the free gossypol that was removable by extraction with pure acetone; the residual meal had only a minimal increase in odor. In contrast, cottonseed meals produced by extraction with pure acetone had much higher odour intensity. The composition of the cottonseed crude oil was insignificantly affected by the acetone

concentration of the extraction solvent. The results indicate that mixtures of acetone and *n*-hexane can be used as extraction solvents to produce cottonseed crude oil without the concomitant development of odorous meals. (et al, M.S. Kuk, 2005)

Table 2.6: Properties of Acetone

Typical Properties	Value
Structural foranula	$CH_3 - \overset{\overset{O}{\|}}{C} - CH_3$
Molecular weight	58.079
Appearance	Colorless liquid
Odor	Pleasant, faintly aromatic, sweetish
Specific gravity at 25/25°	0.7880
Melting point, °C	-94.6
Boiling point at 760 mm Hg, °C	56.13
Vapor pressure at 20°C, mm Hg	181.7
Density at 20° C, g/ml	0.7898
lb/gal	6.59
Refractive index n20/D	1.359
Heat of vaporization, Kcal/mole at 760 mm and 56.1°C	7.092
Viscosity at 25°C, cps	0.3075
Flash point (closed cup), °C, (approx)	-20.0
Flash point (open cup), °C, (approx)	-9.0
Autoignition temperature, °C	465
Flammable limits at 25°C, vol. %	2.6 - 12.8
Electrical conductivity, 25°C, $ohm^{-1}cm^{-1}$	5.5×10^{-8}
Heat of combustion, Kcal/mole	427

Note: These are typical properties; not to be construed as specifications.

2.11 GAS CHROMATOGRAPHY

Gas chromatography (GC) is a type of chromatography in which the mobile phase is a carrier gas. This carrier gas is usually an inert gas such as helium or an unreactive gas such as nitrogen. The stationary phase is a microscopic layer of liquid or polymer on an inert solid support, inside glass or metal tubing, called a column. The instrument used to perform gas chromatographic separations is called a gas chromatograph. (Pavia,Donald,2006)

A gas chromatograph is a chemical analysis instrument for separating chemicals in a complex sample such as a sample of newly produced biodiesel. A gas chromatograph uses a column, through which different chemical constituents of a sample pass in a gas stream at different rates depending on their various chemical and physical properties and their interaction with the stationary phase. The chemicals are identified and detected electronically as they exit the end of the column. The stationary phase functions to separate different components causing each one to exit the column at different retention times. The carrier gas flow rate, as well as temperature can also be used to alter the order or time of retention.

Gas Chromatography is currently one of the most popular methods for separating and analyzing compounds. This is due to its high resolution, low limits of detection, high speed, high accuracy and high reproducibility. GC can be applied to the separation of any compound that is either naturally volatile (i.e. readily goes into the gas phase) or can be converted to a volatile derivative which is widely used in the separation of a number of organic and inorganic compounds. Examples of its applications are shown below:

a) Food testing and analysis
b) Drug testing and urine analysis
c) Characterization of natural and synthetic polymers
d) Petroleum based product testing
e) Environmental factor testing from sample analysis

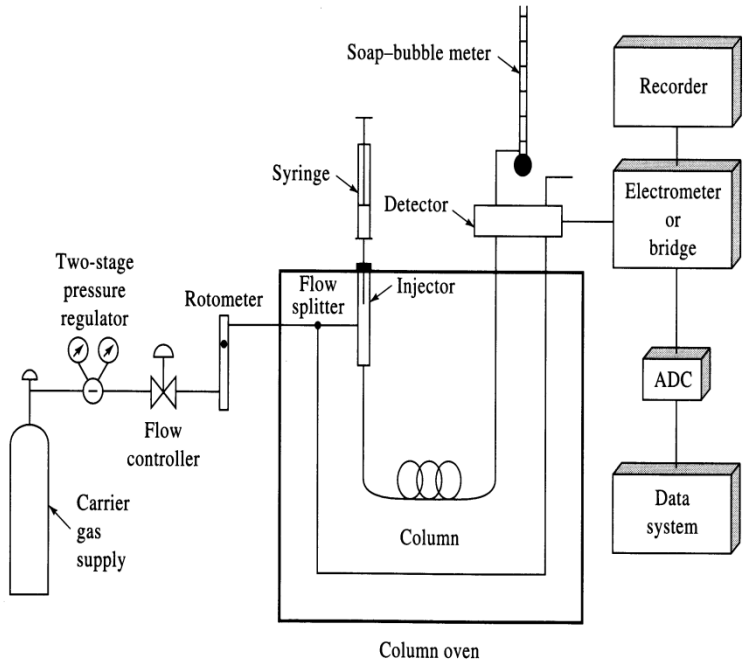


Figure 2.6: Diagram flow of a gas chromatography unit

The efficiency separation of the compounds by the Gas Chromatogram is dependent on the compounds travelling through the column at different rates. The rate at which a compound travels through a GC depends on the factors below:

a) Volatility of compound: Low boiling temperature (volatile) components will travel faster through the column than will high boiling components
b) Polarity of compounds: Polar compounds will move more slowly.
c) Column temperature: Raising the column temperature speeds up all the reaction of compounds in a mixture.
d) Column packing polarity: Usually, all compounds will move slower on polar columns, but polar compounds will show a larger effect.
e) Flow rate of the gas through the column: Speeding up the carrier gas flow increases the speed with which all compounds move through the column.
f) Length of the column: The longer the column, the longer it will take all compounds to elute. Longer columns are employed to obtain better separation.

After the sample is being injected into the gas chromatography unit, a chromatogram (peak area versus time) for the few diluted standards of isooctane, the calibration curve of the known peak area versus concentration can be obtained. Later then, the composition of the isooctane present in the rubber seed oil can be determined based on the calibration curve. From the final results obtained from the chromatogram and the calibration curve, it will be certified that bio-petrol can be produced from rubber seeds as long as the isooctane compound can be found in the rubber seed oil. (Harris,Daniel,1999)

Table 2.7: Commonly used Detectors

Detector	Type	Support gases	Selectivity	Detectability	Dynamic range
Flame ionization (FID)	Mass flow	Hydrogen and air	Most organic compounds.	100 pg	10^7
Thermal conductivity (TCD)	Concentration	Reference	Universal	1 ng	10^7
Electron capture (ECD)	Concentration	Make-up	Halides, nitrates, nitriles, peroxides, anhydrides, organometallics	50 fg	10^5
Nitrogen-phosphorus	Mass flow	Hydrogen and air	Nitrogen, phosphorus	10 pg	10^6
Flame photometric (FPD)	Mass flow	Hydrogen and air possibly oxygen	Sulphur, phosphorus, tin, boron, arsenic, germanium, selenium, chromium	100 pg	10^3
Photo-ionization (PID)	Concentration	Make-up	Aliphatics, aromatics, ketones, esters, aldehydes, amines, heterocyclics, organosulphurs, some organometallics	2 pg	10^7
Hall electrolytic conductivity	Mass flow	Hydrogen, oxygen	Halide, nitrogen, nitrosamine, sulphur		

CHAPTER 3

METHODOLOGY

3.1 MATERIAL

In this experimental works, rubber seeds will be needed as main material. This material will be much used in the Soxhlet extraction process whereby it will be inserted into thimble filter. Also, another material used will be anti-bumping granules to ensure that the liquid boils smoothly in the crucible during the catalytic cracking process. For the cracking process, Zeolite was used catalyst.

3.2 APPARATUS AND EQUIPMENT

In this project, the apparatus used can be grouped into four different categories according the processes in the experimental works. They are Soxhlet extraction, solvent evaporation, cracking, and sample dilution.

3.2.1 Soxhlet Extraction

During rubber seed oil extraction, Soxhlet Extraction Unit, round bottom flask of 500 mL, Schott Bottle of 500 mL, beaker of 100 mL, heating mantle, thimble filter, thermometer and filter funnel were used. This was including storing the extracted oil with solvent in the lab.

3.2.2 Solvent Evaporation

Rotary Evaporator unit was used to remove the solvent from the extracted oil. For this process, round bottom flask of 500 mL was used as well. The concentrated rubber seed oil was then transferred into falcon tube for storage purpose.

3.2.3 Catalytic Cracking

Crude rubber seed oil of 25 mL was injected into crucible with 5 mL syringe which was fitted with 0.2 µm filter. The cracked oil was then filtered into falcon tube using filter funnel with filter paper placed on it.

3.2.4 Sample Dilution

For dilution process Micro Pipette, vials of 1.5 mL, dropper, volumetric flask of 10 mL, and beaker of 50 mL were used. After labeling, the vials were then wrapped with Aluminium foil. All these equipment and apparatus were available in FKKSA's laboratory. Therefore, the sample will be fully analyzed at the laboratory.

3.3 CHEMICAL SUBSTANCES

Isooctane of purity 100% is needed in order to make a comparative analysis. As a solvent, standard Acetone of purity 100% used in the extraction process. In the dilution process of the cracked sample, Hexane of Gas Chromatography Purity level was used.

3.4 EXPERIMENT PROCEDURES

In order to achieve isooctane production from the samples, the experimental works are divided into five major sections. They were arranged in such a way that each part correlates with the other.

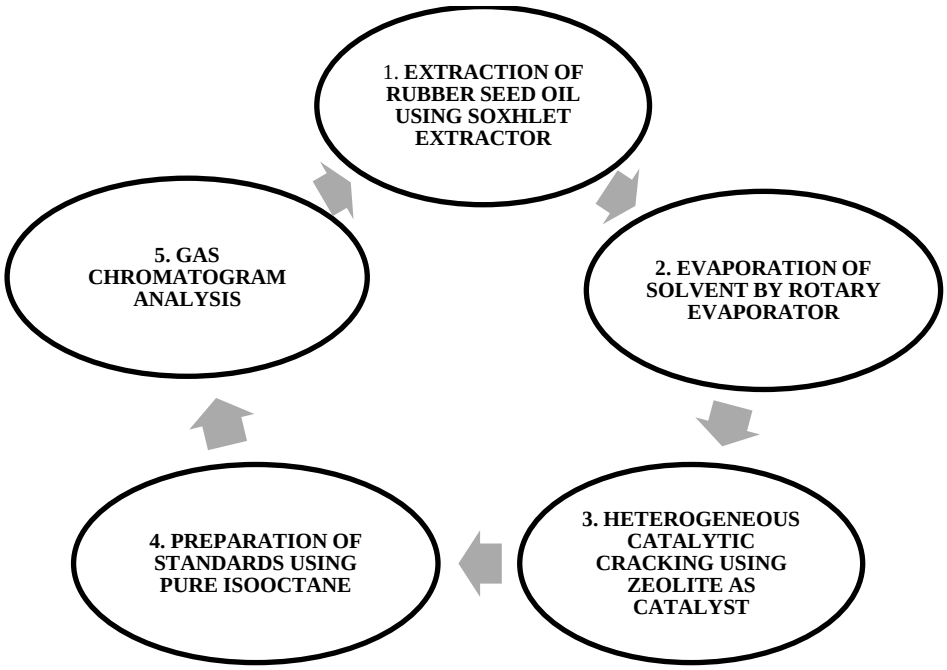


Figure 3.1: Overall Processes

3.4.1 Sample Preparation

Hevea brasiliensis seeds were cracked and the shells were carefully removed. Then the kernels were blended into small particles using a blender. The material was then dried in open area for two days. It is to ensure that the water content in the material is dried off and free of excess moisture.

3.4.2 Extraction of Rubber Seed Oil Using Soxhlet Extractor

The material of about 50 grams rubber seeds were measured. Then a thimble filter is filled until two third portions with the blended seeds and then inserted into the siphon exit of the Soxhlet extractor. Meanwhile, receiving flask is filled with about 190 mL of the Acetone solvent for the purpose of extraction of rubber seed oil. Then the mixture will be heated using a heater until the temperature of the solvent reaches about 75^0C or until the solvent is boiling. This process will be left to run for approximately eight hours. After the heating period is over, the unit was switched off and let to cool down to room temperature; where cooling time was approximately two hours. Then the rubber seed oil solvent mixture was transferred into Schott bottle for storage before

proceeding to solvent evaporation process. The bottle was wrapped with Aluminium foil to prevent from UV-light and other contaminants.

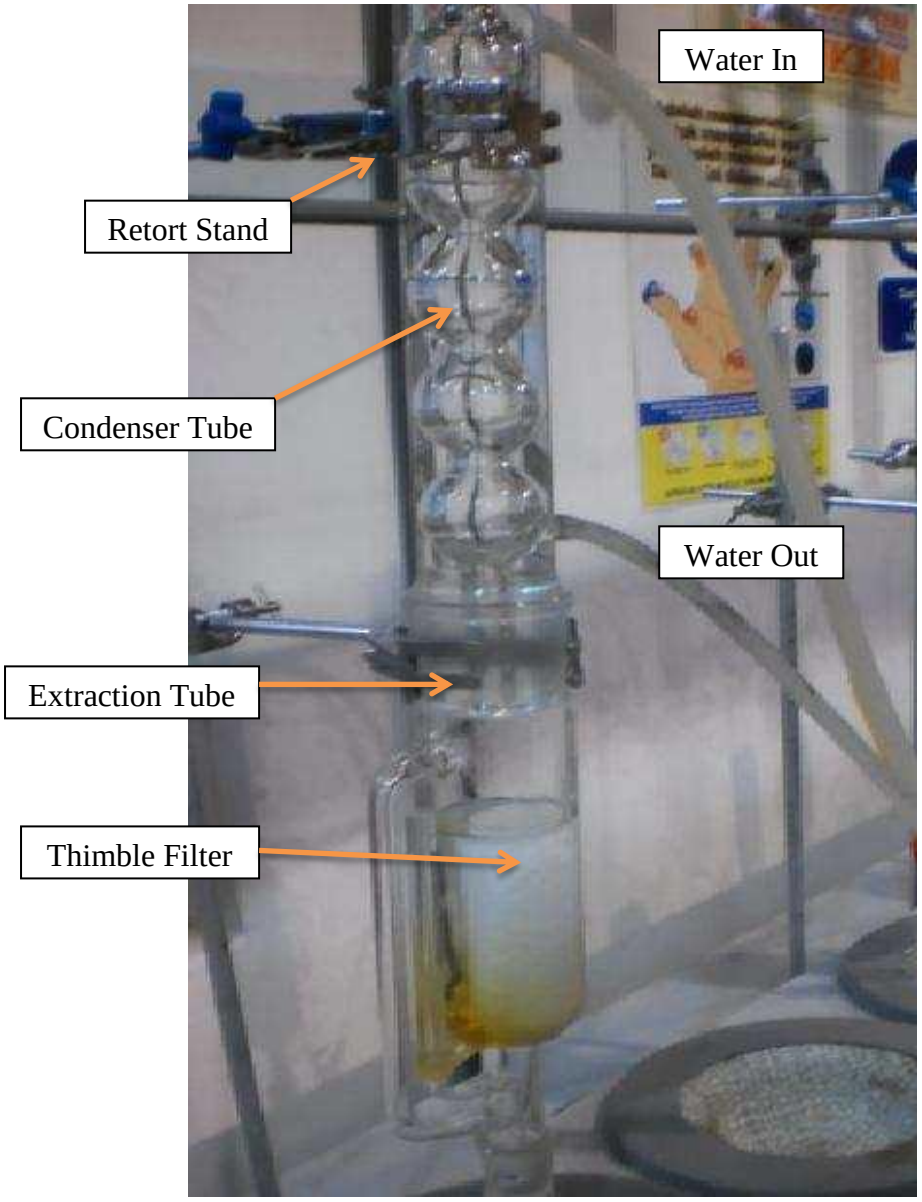


Figure 3.2: Set Up of Apparatus for Soxhlet Extraction

3.4.3 Evaporation of Solvent By Rotary Evaporator

The mixture was then brought to the rotary evaporator to evaporate the solvent at a temperature of around 60^0C which is a bit higher than the boiling point of Acetone. The liquid remaining in the flask was crude rubber seed oil.

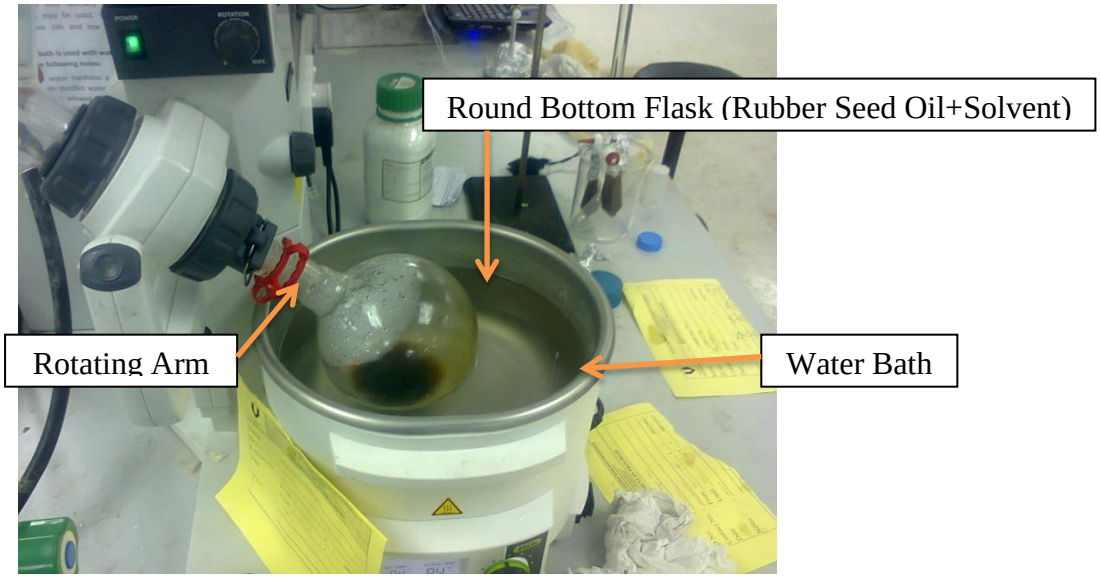


Figure 3.3: Set Up of Apparatus for Solvent Evaporation

3.4.4 Catalytic Cracking Using Zeolite Catalyst

The rubber seed oil is transferred into a flask. 5 grams of zeolite catalyst added into the crucible containing 25mL of rubber seed oil. The mixture of rubber seed oil and zeolite are placed in a furnace, and then heated until 300^0C and maintained it for 45 minutes. Five grams anti bumping granules are added into the mixture to ensure the heat energy transfers uniformly. Then, the samples will be filtered to remove the solid catalyst to obtain the distilled product oil.

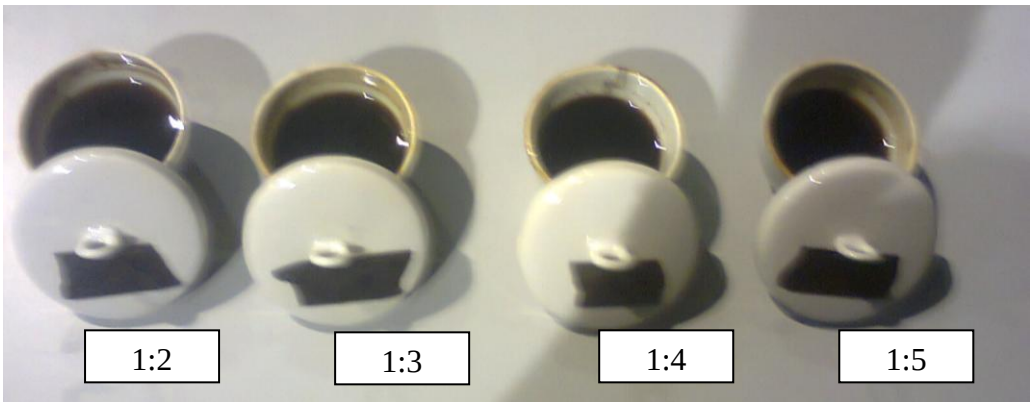


Figure 3.4: Oil Sample after cracking process

3.4.5 Preparation of Standards Using Calibration

Isooctane and hexane are prepared according to the portions in Table 3.1. The mixtures of the standards are injected into each vial through a 0.2μm syringe filter of about 1.5ml. Variable composition isooctane and hexane will give a standard for the outcome of the cracking process.

Table 3.1: Composition of the Isooctane-Hexane mixture

Vials	**Composition**	
	Isooctane	Hexane
1	0%	100%
2	80%	20%
3	60%	40%
4	40%	60%
5	20%	80%

3.4.6 Gas Chromatogram Sample Analysis

The filtered product oil for each ratio of catalyst used will be diluted with hexane as a solvent to give the four different mixture solutions – 1%, 5%, 10%, 15%, and 20% of filtered products. The samples are then injected into the 1.5mL vials using the 0.2μm syringe filter of about 1.5mL. The vials are labeled and must be arranged in sequence on the auto-injectors VS auto samplers at the gas chromatography's vial rack. Samples obtained (for every diluted mixture solutions) are analyzed using gas chromatography. The conditions of the gas chromatograph are set as in Table 3.2. The chromatogram (peak area versus time) of each standard are then obtained. Then, a calibration curve (peak area versus concentration) is plotted.

Biopetrol yield is to be measured by dividing the amount of biopetrol product with the amount of feedstock used.

$$\text{Percentage of Yield} = \frac{\text{Final Sample Product}}{\text{Oil}} \times 100\%$$

Table 3.2: Gas Chromatographer Conditions

Parts	**Conditions**
Temperature Column	Initial 50^{o}C, hold 3 minutes, program at 8^{o}C/min to 120^{o}C, hold 5 minutes
Detector Type	Flame Ionization Detector (FID)
Injector Temperature	250^{o}C
Detector Temperature	250^{o}C
Injection Size	1.0µL (10:1 split)
Hydrogen Flow	35mL/min
Air Flow	400mL/min
Helium Make Up	35mL/min
Carrier Gas	Helium, Compress Air, H_2, N_2
Carrier Gas Pressure	5 bar for every carrier gas

Standard Isooctane Dilution

Table 3.3: Standard Isooctane Preparation

Vial	Concentration (%)	
	Isooctane	Hexane
1	0	100
2	20	80
3	40	60
4	50	50
5	60	40

Sample Analysis

Table 3.4: Analysis Sample Preparation for the ratio 1:3

Vial	Concentration (%)	
	Sample	Hexane
1	1	99
2	5	95
3	10	90
4	15	85
5	20	80

Then the sample analysis was repeated again for other ratios which are 1:2, 1:4, and 1:5. Each ratio follows the standard which has been laid down in the above table.

3.5 OVERALL PROCESSES

Shortly, the whole processes and parameters or the conditions involved are briefly summarized in the chart below. This is including from the preparation of sample until the gas chromatography analysis.

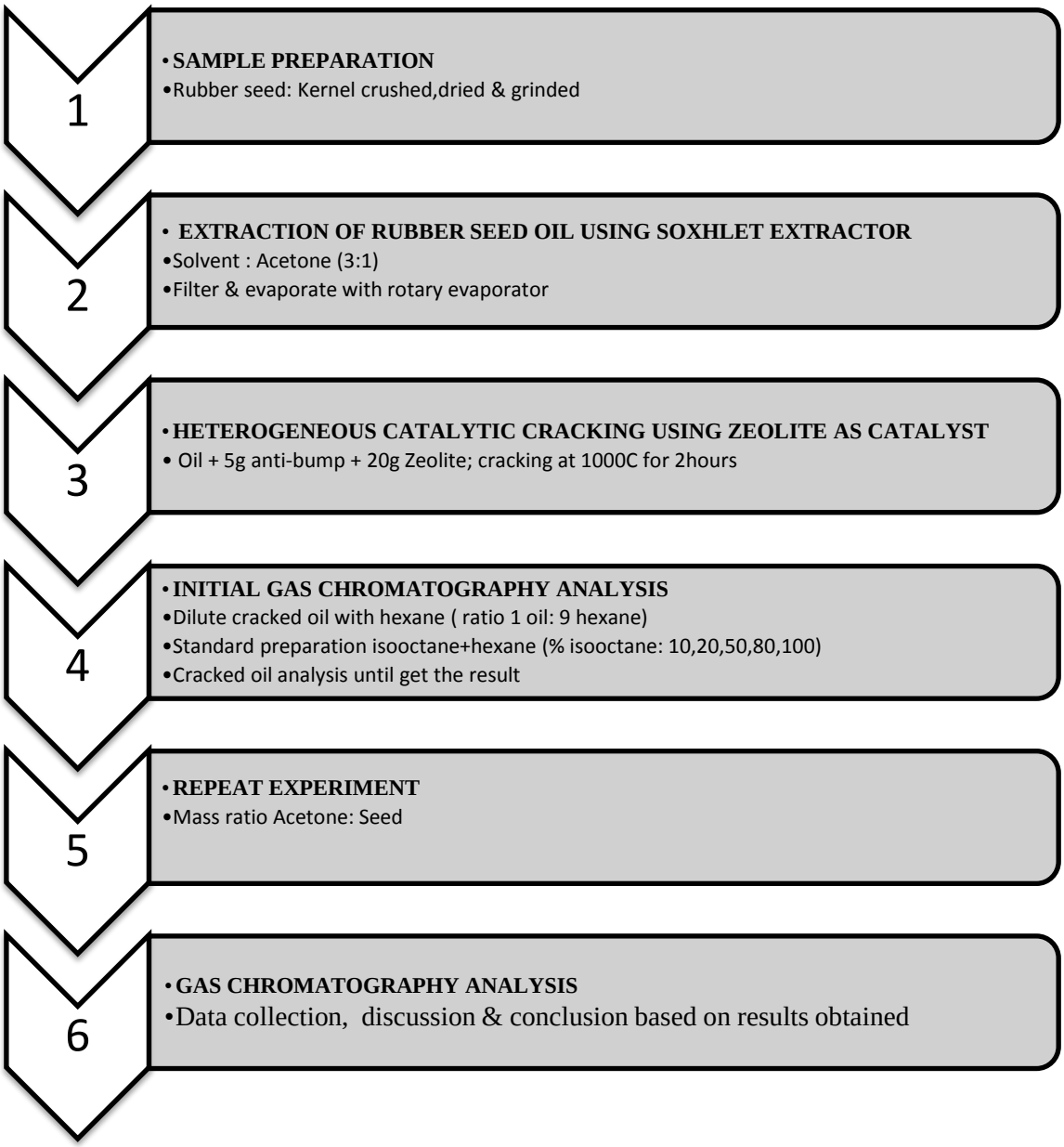


Figure 3.5: Process Summary

CHAPTER 4

RESULT AND DISCUSSION

4.1 OBSERVATION

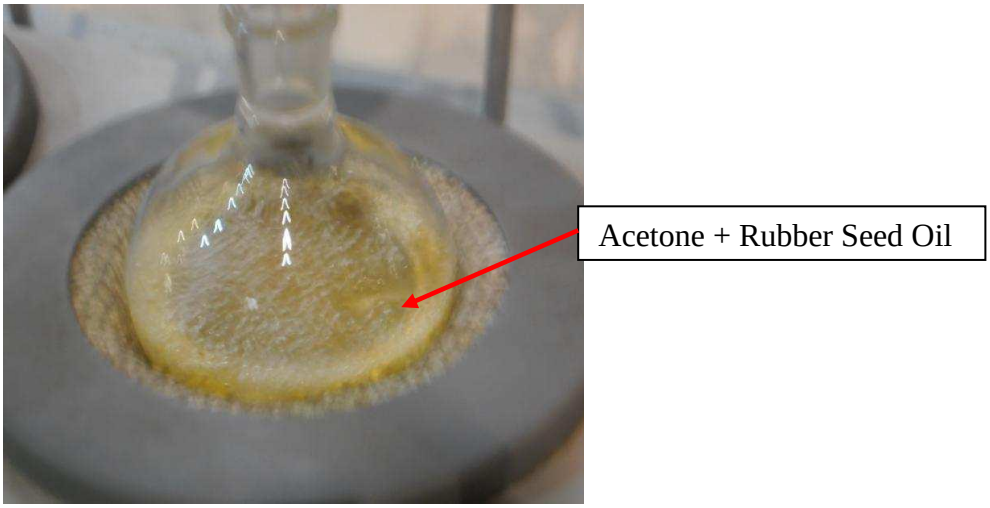


Figure 4.1 Solvent and Rubber Seed Oil Mixture during Soxhlet Extraction Process

From Figure 4.1 above, the solvent has changed from colourless to yellowish mixture after elapse of time during the extraction process. This observation is due to the presence of rubber seed oil in the round bottom flask along with the solvent. It has been observed that after eight hours of constant boiling and condensing in the process has made some of the solvent to escape as vapour through the leaks between joints and seals. But the amount of solvent loss in the process is negligible as it only contributes from just 4ml to 8ml. Additionally; the extraction mixture for each ratio was getting lighter in colour as the mass ratio increased. As for the concern of oil getting charred and darken, the mass ratio of 1:2 was in that situation, due to the limited amount of solvent used with the rubber seed.

Figure 4.2 Crude Rubber Seed Oil

The above figure shows crude rubber seed oil which has been extraction and the solvent been evaporated using rotary evaporator unit. The oil has formed two layers of differential contrast in colour. The top layer is being light while the bottom layer is being dark and susceptible for being rich in fatty acids as well as other heavy components which give the darker visibility to it. Besides that for the cracking process we shake well until it homogenizes, then form that 25ml taken for each ratio.

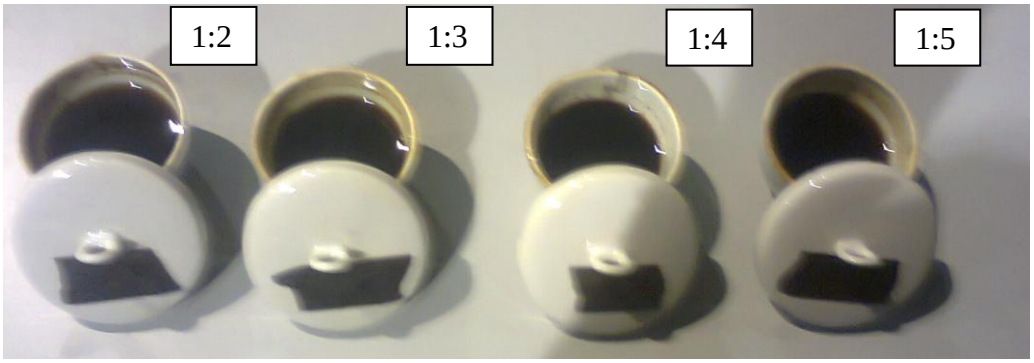


Figure 4.3 Cracked Rubber Seed Oil + 5gram of Zeolite + 5gram of Boiling Chips after Catalytic Cracking

Figure 4.4 Filtration of Cracked Oil

From the Figure 4.3 above, the mixture has changed its color from light brown (before cracking) into dark brown after cracking. The oil can be hardly seen as the mixture became very thick due to the burning of lighter components and some of it getting charred. Some of the lighter components were evaporated in the high temperature inside the furnace and the heavy elements were left behind in the crucible. It can be noticed also that the label on crucible cover getting burned, luckily it was arranged according to the ratio and things didn't get messed up. In Figure 4.4 the thick oil is being filtrated using filter paper via funnel. The thick colloids and particulates were left stuck in the paper and pure oil i.e. isooctane is flowing down into the flask. It can be observed that the oil is being light yellowish in colour thus adding to the fact that it could be rich in isooctane.

4.2 ANALYSIS OF STANDARD ISOOCTANE CALIBRATION CURVE

The standard mixtures contained of isooctane and hexane was analyzed using Gas Chromatography Flame Ionization Detector method. The standards prepared must be a variety of mixtures of different ratio of solvent and the favorable component of the chemical to be detected. Based on these standards, the presence of the specified chemical compound is identified through the chromatogram graph of peak area versus retention time; hence the results will be used as standard reference to calculate concentration of samples. Below are the Chromatogram Results for the Isooctane Standards:

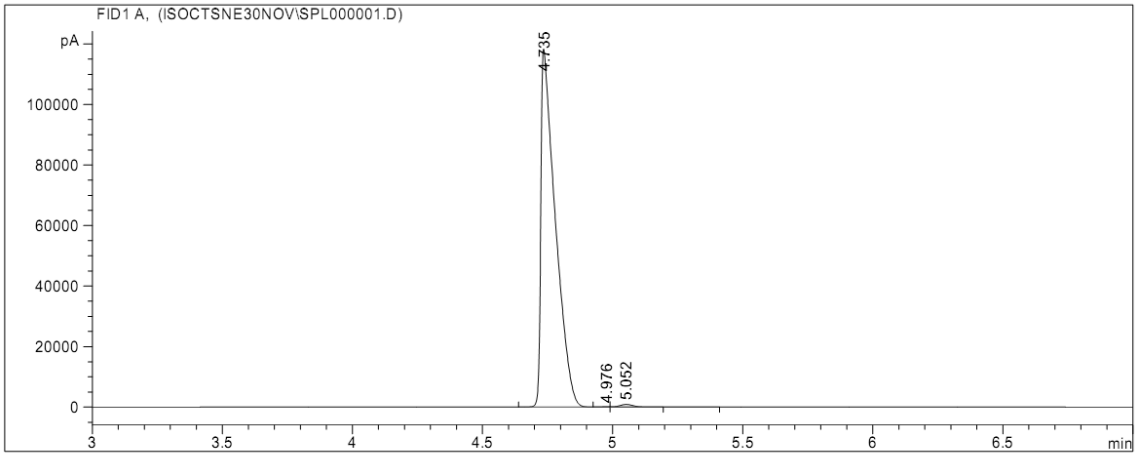


Figure 4.5 Chromatogram of 0% Standard Isooctane

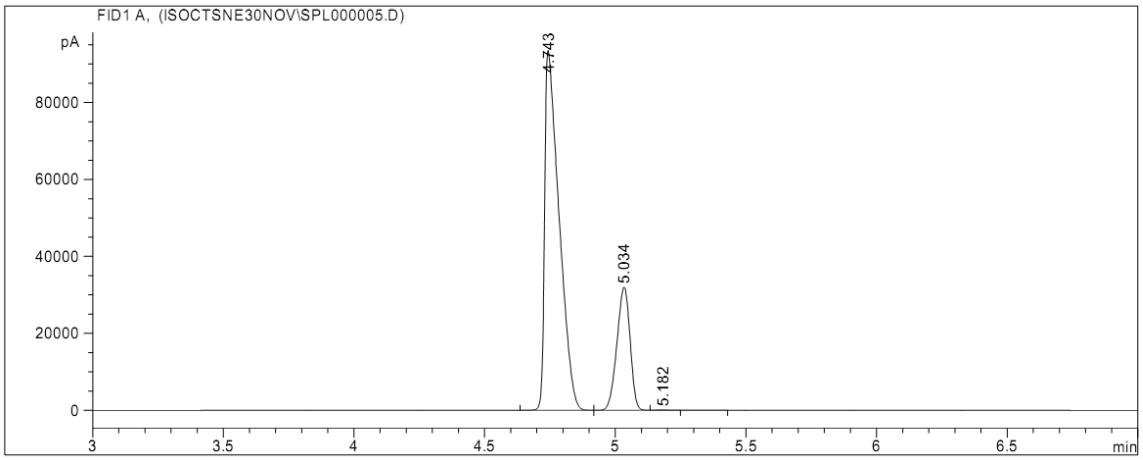


Figure 4.6 Chromatogram of 20% Standard Isooctane

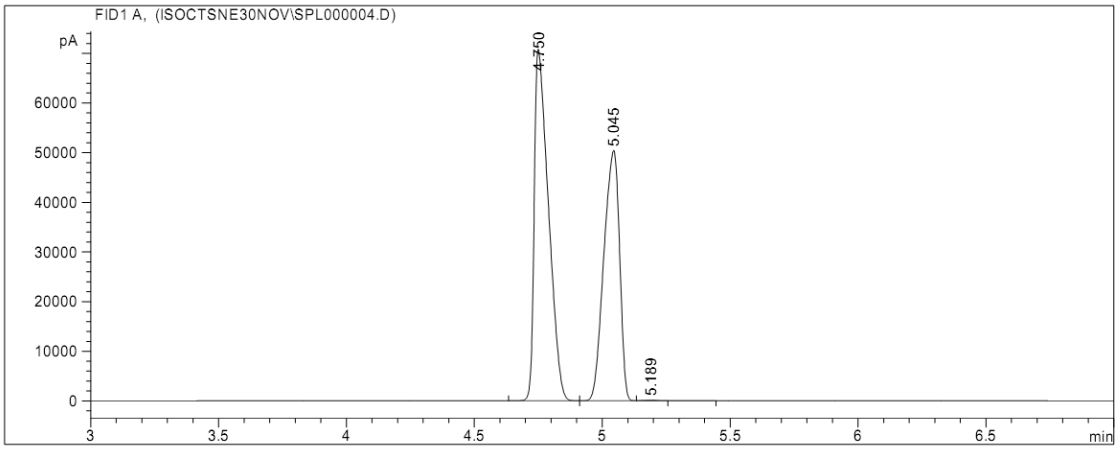


Figure 4.7 Chromatogram of 40% Standard Isooctane

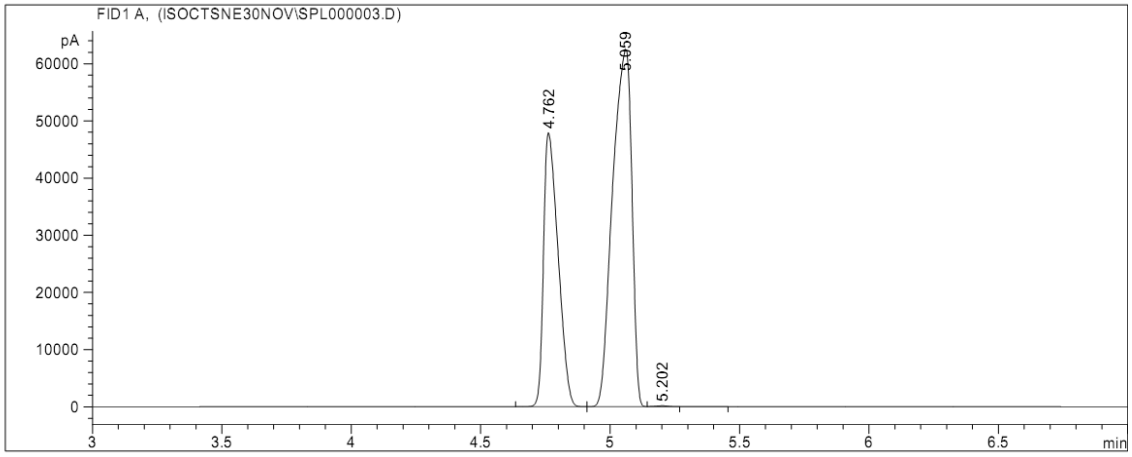


Figure 4.8 Chromatogram of 60% Standard Isooctane

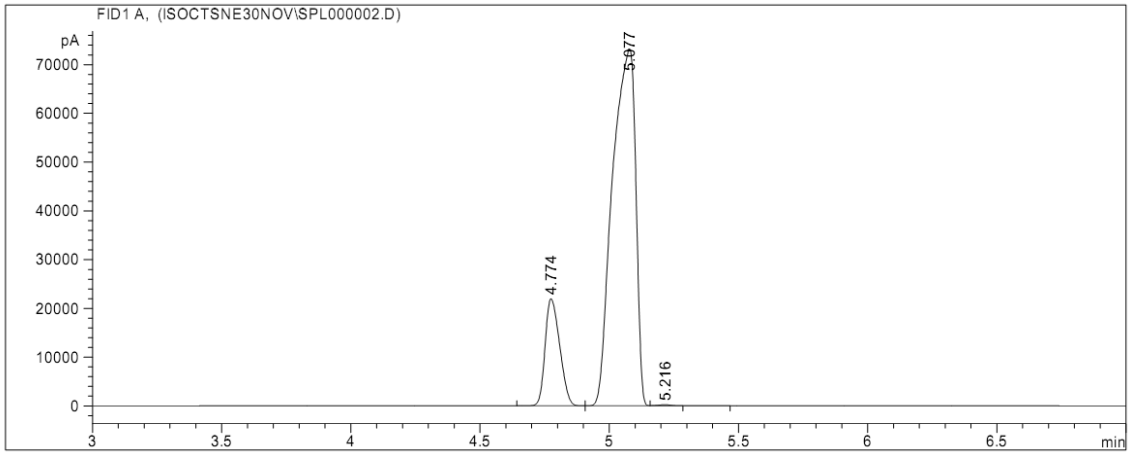


Figure 4.9 Chromatogram of 80% Standard Isooctane

Table 4.1 Chromatogram Analysis for Standard Isooctane *

CONCENTRATION OF ISOOCTANE (%)		HEXANE	ISOOCTANE
0	Retention time(min)	4.375	0
	Peak Area (pA*s)	4.56634E+05	0
	Peak Area (%)	99.96915	0
20	Retention time(min)	4.743	5.034
	Peak Area (pA*s)	3.57789E+05	1.12664E+05
	Peak Area (%)	76.05197	23.94803
40	Retention time(min)	4.750	5.045
	Peak Area (pA*s)	2.72851E+05	2.15759E+05
	Peak Area (%)	55.84228	44.15772
60	Retention time(min)	4.762	5.059
	Peak Area (pA*s)	1.86902E+05	3.22600E+05
	Peak Area (%)	36.68334	63.31666
80	Retention time(min)	4.774	5.077
	Peak Area (pA*s)	8.61127E+05	4.56298E+05
	Peak Area (%)	15.85654	84.02133

NOTE: * Please refer to Appendix A for the Chromatogram Analysis for Standard Isooctane

The standard sample analysis were all recorded and tabulated in Table 4.2. In order to obtain the standard calibration curve for the determination of the actual percentage of isooctane present in each of the samples, a graph of peak area (pA*s) versus concentration of the isooctane (%) are plotted

Table 4.2 Data of Concentration of Isooctane and Peak Area

Concentration of Isooctane (%)	Peak Area (pA*s)
0	0
20	1.12664E+05
40	2.15759E+05
60	3.22600E+05
80	4.56298E+05

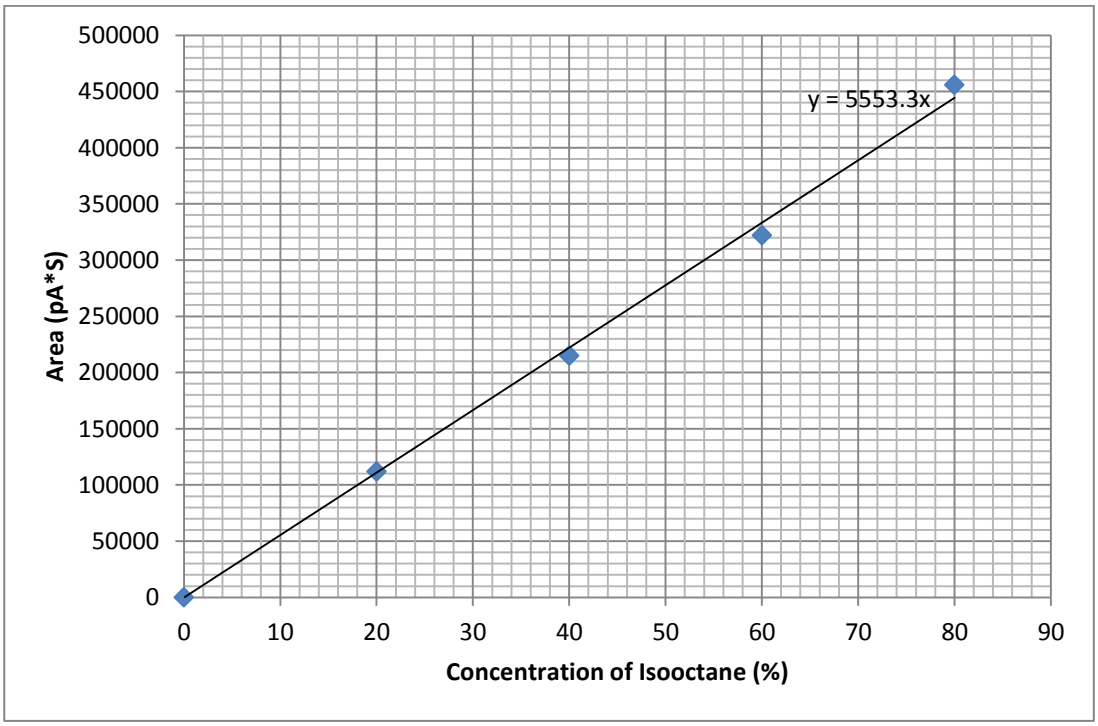


Figure 4.10 Standard Calibration Curve

From Figure 4.6, a straight line starting from origin along with other points was obtained. The line indicates that as the concentration of isooctane increase, the peak area increases as well. In simple words, the peak area of isooctane is directly proportional to its concentration. This calibration curve will used to determine the actual concentration of isooctane of each sample by calibrating the isooctane's peak area of each sample in the calibration curve to get the concentration.

Using graphical method, the straight line is taken from the best symmetrical degree among the plotted data. Therefore the standard calibration curve equation for isooctane is:

y = 5553.3x ----------(equation 1)

This equation can be used to determine the actual concentration of isooctane present in each sample via backward calculation.

4.3 ANALYSIS OF ACTUAL ISOOCTANE CONCENTRATION IN SAMPLES

The chromatogram analysis of the samples is obtained and the peak areas are identified of which chemical compound based on the retention time of Hexane and Isooctane based on the Chromatogram of the standard analysis. It is decided that the retention time of Isooctane and Hexane is around 5.05 and 4.75 minute respectively. Other elements which are present in the chromatogram can be discarded form the calculation as their retention varies with Isooctane and Hexane. The choosing of time was made decisively with analyzing all the chromatogram of different dilution factors, which thus gives the same retention time for both of the analyzing components. The peak area of hexane is the largest in comparison with isooctane as because it is the carrier agent. The peak areas of Isooctane and Hexane area identified and the table below shows the details from the data of the analysis:

Table 4.3 Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio (1:2)

Dilution Samples (%)	Isooctane			Hexane		
	Retention Time (min)	Peak Area (pA*s)	Area (%)	Retention Time (min)	Peak Area (pA*s)	Area (%)
1	5.057	3191.3467	0.73406	4.738	4.31561E+5	99.26594
5	5.066	2371.1450	0.66905	4.755	3.52007E+5	99.32369
10	5.066	2112.2595	0.64081	4.757	3.27489E+5	99.35252
15	5.066	2458.8784	0.69672	4.756	3.50397E+5	99.28519
20	5.073	1979.7994	0.63340	4.766	3.10548E+5	99.35395

Table 4.4 Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio (1:3)

Dilution Samples (%)	Isooctane			Hexane		
	Retention Time (min)	Peak Area (pA*s)	Area (%)	Retention Time (min)	Peak Area (pA*s)	Area (%)
1	5.058	2496.7898	0.68522	4.746	3.61879E+5	99.31478
5	5.079	2203.3213	0.61686	4.764	3.54983E+5	99.38314
10	5.079	1994.6862	0.60352	4.768	3.28497E+5	99.39095
15	5.078	2224.1150	0.59344	4.763	3.72509E+5	99.39355
20	5.079	1836.3671	0.58843	4.770	3.10185E+5	99.39255

Table 4.5 Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio (1:4)

Dilution Samples (%)	Isooctane			Hexane		
	Retention Time (min)	Peak Area (pA*s)	Area (%)	Retention Time (min)	Peak Area (pA*s)	Area (%)
1	5.055	2742.0903	0.68777	4.740	3.95950E+5	99.31223
5	5.081	2225.9150	0.62315	4.765	3.54968E+5	99.37354
10	5.080	2067.6350	0.60368	4.767	3.40420E+5	99.39049
15	5.079	2063.0759	0.58675	4.766	3.49512E+5	99.40354
20	5.079	1740.9628	0.56847	4.772	3.04470E+5	99.41754

Table 4.6 Chromatogram Analysis of Samples for Rubber Seed Mass to Solvent Mass Ratio (1:5)

Dilution Samples (%)	Isooctane			Hexane		
	Retention Time (min)	Peak Area (pA*s)	Area (%)	Retention Time (min)	Peak Area (pA*s)	Area (%)
1	5.058	2535.6504	0.68821	4.746	3.65907E+5	99.31179
5	5.082	2225.8081	0.60656	4.766	3.64730E+5	99.39344
10	5.084	2611.3660	0.60592	4.762	4.28328E+5	99.38606
15	5.082	2158.6335	0.58570	4.768	3.66342E+5	99.39905
20	5.081	1721.9626	0.55329	4.774	3.09429E+5	99.42389

In order to calculate the actual concentration of isooctane present in each of the samples, a backward calculation is carried out manually knowing that the samples must be diluted with the Hexane solvent before entering into the Gas Chromatogram. This is to make it easier for the samples to be injected into the column for analysis. Below are the equations used for the determination of isooctane concentration in each sample:

- Actual Peak area of Isooctane (%)

$$Actual\ Peak\ Area\ of\ Isooctane\ (\%) = \frac{Peak\ Area\ of\ Isooctane\ (\%)}{[(100 - Peak\ Area\ of\ Hexane)\%]}\ X\ 100\%$$

- Actual Peak Area of Isooctane (pA*s)

$$Actual\ Peak\ Area\ of\ Isooctane\ (pA * s) = \frac{Peak\ Area\ of\ Isooctane\ (pA * s)}{Peak\ Area\ of\ Isooctane\ (\%)}\ X\ [Actual\ Peak\ Area\ of\ Isooctane\ (\%)]$$

- Actual Concentration of Isooctane (%)

1. Based on equation 1 where the;

y = $Actual\ Peak\ Area\ of\ Isooctane\ (pA * s)$ and

x = $Actual\ Concentration\ of\ Isooctane\ (\%)$

- Actual Concentration of Isooctane (%) = y X [5553.3^{-1}]

Calculation for the ratio1:2 (1% Sample Dilution)

- Actual Peak area of Isooctane (%) = $\frac{0.73406}{[100-99.26594]} X\ 100\%$

= 100.0000%

- Actual Peak Area of Isooctane (pA*s) = $\frac{3191.3467}{0.73406} X\ 100.0000\%$

= 4.3475E+5

- Actual Concentration of Isooctane (%)

Based on equation 1 where the;

y = $Actual\ Peak\ Area\ of\ Isooctane\ (pA * s)$ and

x = $Actual\ Concentration\ of\ Isooctane\ (\%)$

Actual Concentration of Isooctane (%) = [4.3475E+5] X [5553.3^{-1}]

= 78.29%

*The results of the calculations for each ratio are shown in the Table below:

Table 4.7 Experimental Matrix and Results of each Sample for Rubber Seed Mass to Solvent Mass Ratio (1:2)

Dilution (%)	**Area Isooctane (pA*s)**	**Isooctane Area (%)**	**Hexane Area (%)**	**Actual Peak Area Isooctane (%)**	**Actual Peak Area (pA*s)**	**Actual Isooctane Concentration (%)**
1	3191.3467	0.73406	99.26594	100.0000	4.3475E+5	78.29
5	2371.1450	0.66905	99.32369	98.9265	3.5060E+5	63.13
10	2112.2595	0.64081	99.35252	98.9698	3.2623E+5	58.74
15	2458.8784	0.69672	99.28519	97.4693	3.4399E+5	61.94
20	1979.7994	0.63340	99.35395	98.0419	3.0645E+5	55.18

Table 4.8 Experimental Matrix and Results of each Sample for Rubber Seed Mass to Solvent Mass Ratio (1:3)

Dilution (%)	Area Isooctane (pA*s)	Isooctane Area (%)	Hexane Area (%)	Actual Peak Area Isooctane (%)	Actual Peak Area (pA*s)	Actual Isooctane Concentration (%)
1	2496.7898	0.68522	99.31478	100.0000	3.6438E+5	65.61
5	2203.3213	0.61686	99.38314	100.0000	3.5718E+5	64.32
10	1994.6862	0.60352	99.39095	99.0920	3.2751E+5	58.97
15	2224.1150	0.59344	99.39355	97.8547	3.6674E+5	66.04
20	1836.3671	0.58843	99.39255	96.8689	3.0231E+5	54.44

Table 4.9 Experimental Matrix and Results of each Sample for Rubber Seed Mass to Solvent Mass Ratio (1:4)

Dilution (%)	Area Isooctane (pA*s)	Isooctane Area (%)	Hexane Area (%)	Actual Peak Area Isooctane (%)	Actual Peak Area (pA*s)	Actual Isooctane Concentration (%)
1	2742.0903	0.68777	99.31223	100.0000	3.9869E+5	71.79
5	2225.9150	0.62315	99.37354	99.4716	3.5532E+5	63.98
10	2067.6350	0.60368	99.39049	99.0435	3.3923E+5	61.09
15	2063.0759	0.58675	99.40354	98.3721	3.4589E+5	62.28
20	1740.9628	0.56847	99.41754	97.5981	2.9890E+5	53.82

Table 4.10 Experimental Matrix and Results of each Sample for Rubber Seed Mass to Solvent Mass Ratio (1:5)

Dilution (%)	**Area Isooctane (pA*s)**	**Isooctane Area (%)**	**Hexane Area (%)**	**Actual Peak Area Isooctane (%)**	**Actual Peak Area (pA*s)**	**Actual Isooctane Concentration (%)**
1	2535.6504	0.68821	99.31179	100.0000	3.6844E+5	66.35
5	2225.8081	0.60656	99.39344	100.0000	3.6696E+5	66.08
10	2611.3660	0.60592	99.38606	98.6937	4.2535E+5	76.59
15	2158.6335	0.58570	99.39905	97.4623	3.5920E+5	64.68
20	1721.9626	0.55329	99.42389	96.0389	2.9889E+5	53.82

The value above in the table shows both experimental isooctane concentrations in samples and actual isooctane concentrations in samples. Using backward calculations, the actual concentrations of isooctane for ratios has been calculated. The actual concentrations corresponding to the peak area (pA*s) has been plotted in a linear graph of standard calibration curve. Besides that before the actual calculations has been done, the peak area of isooctane from chromatogram was used to determine the experimental value of concentration using the linear equation ‘y=5553.3x’. This data also have been plotted alongside with the standard calibration curve, but instead using a smaller scale to represent the points more clearly. All the graphs for the corresponding ratios have been shown in the figures below.

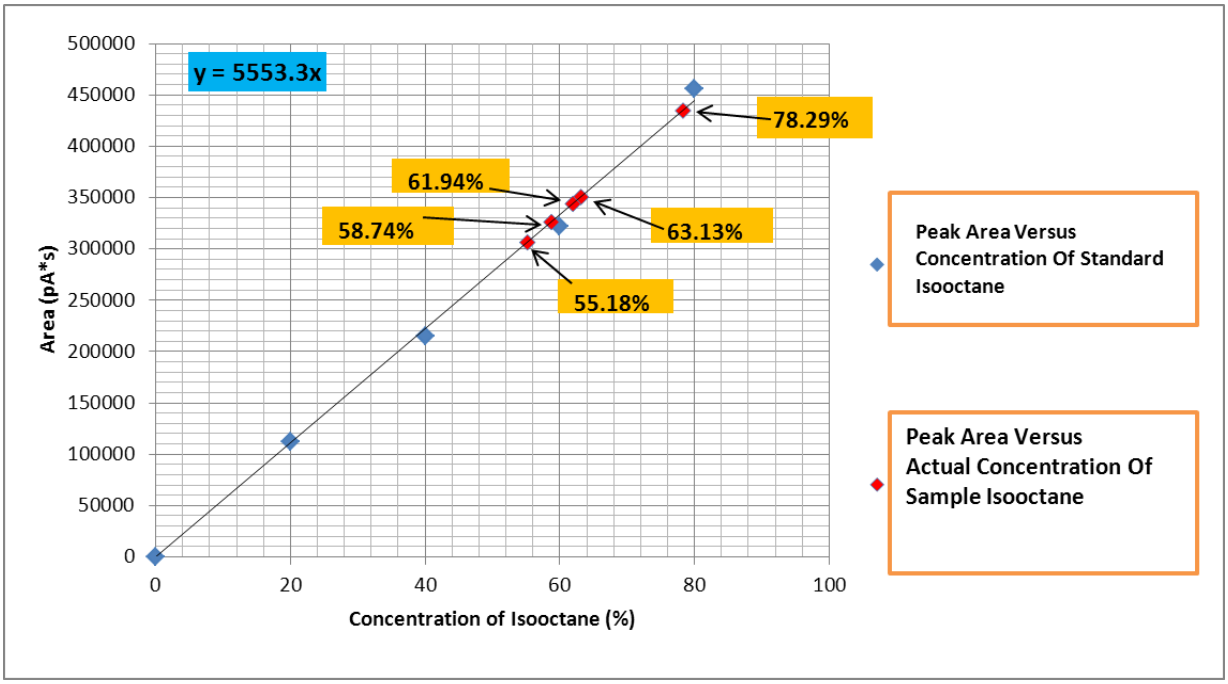


Figure 4.11 Actual Concentration of Isooctane Present In Samples (1:2)

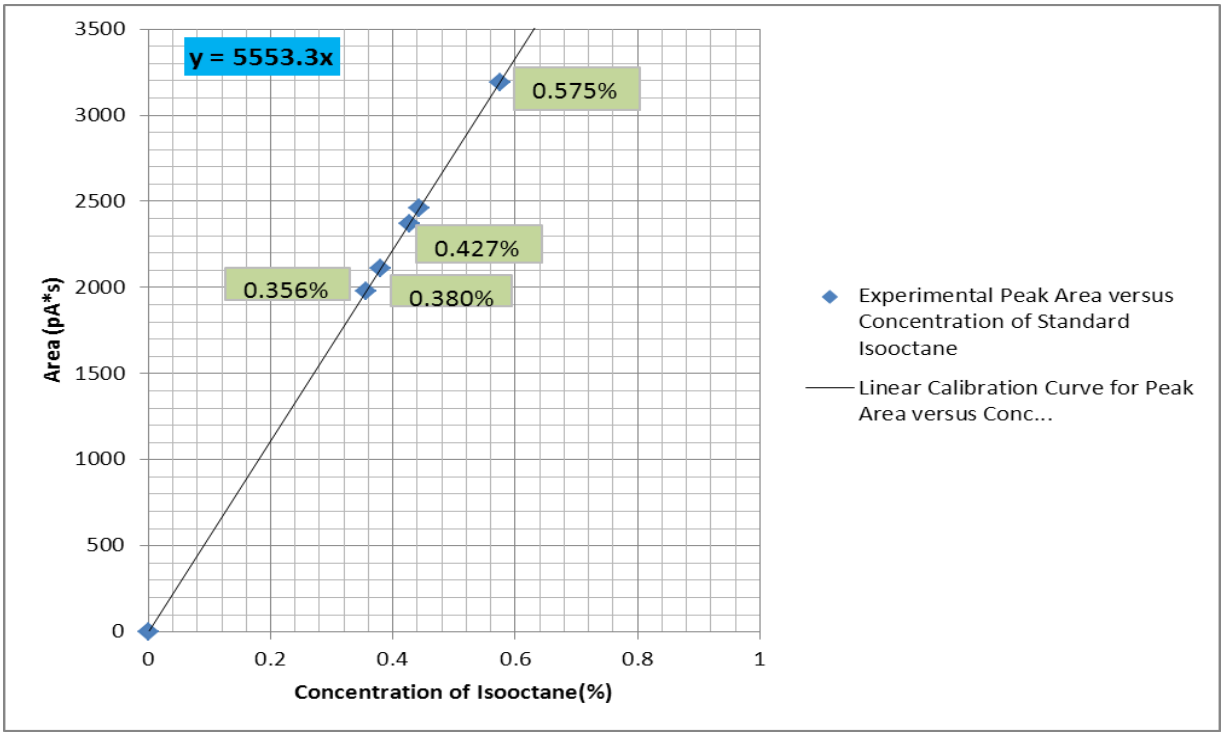


Figure 4.12 Experimental Concentration of Isooctane Present In Samples (1:2)

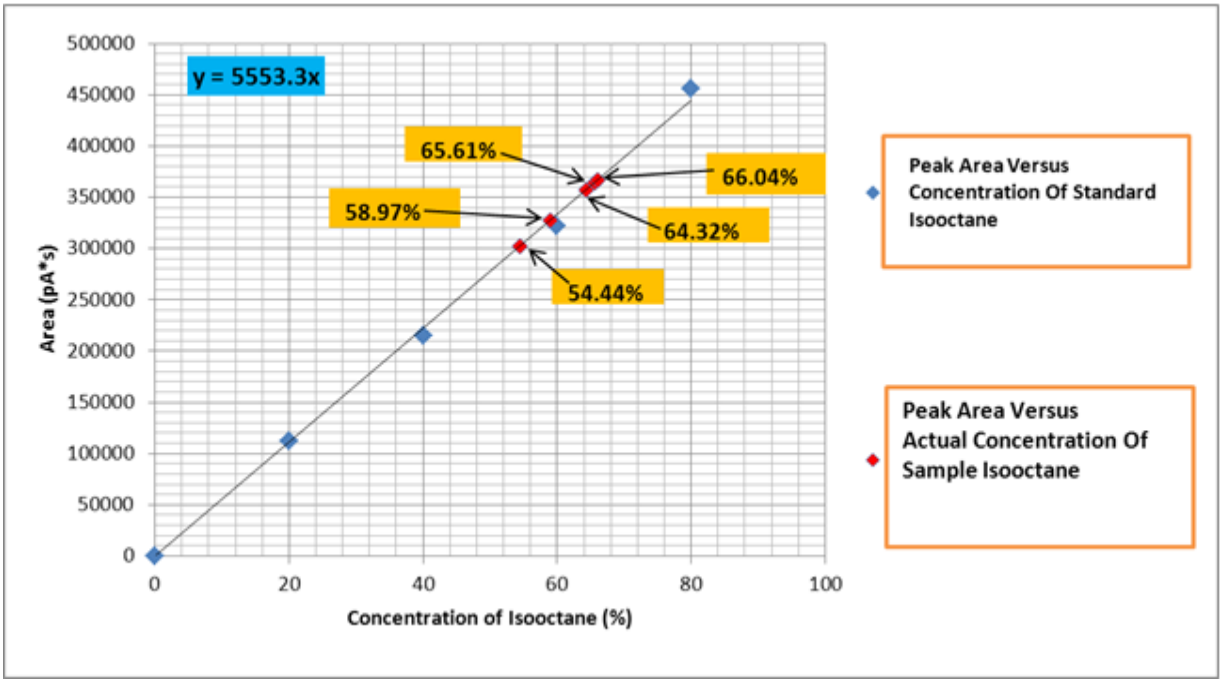


Figure 4.13 Actual Concentration of Isooctane Present In Samples (1:3)

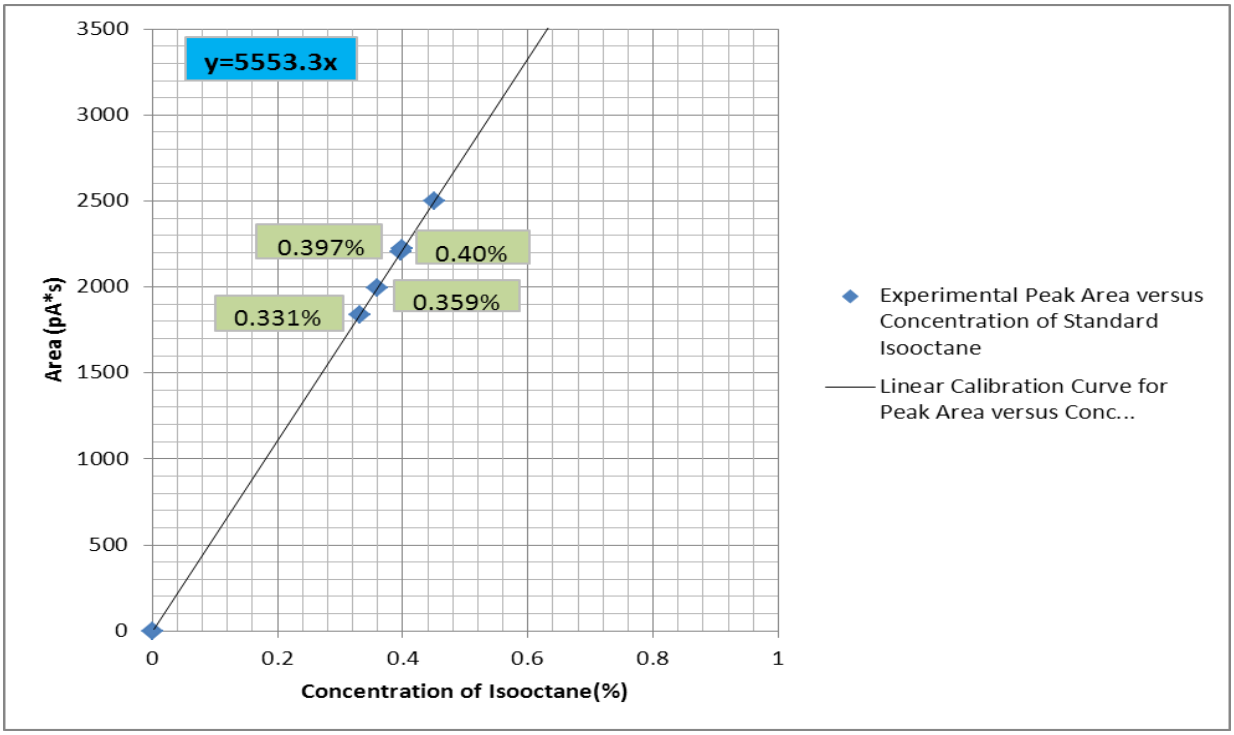


Figure 4.14 Experimental Concentration of Isooctane Present In Samples (1:3)

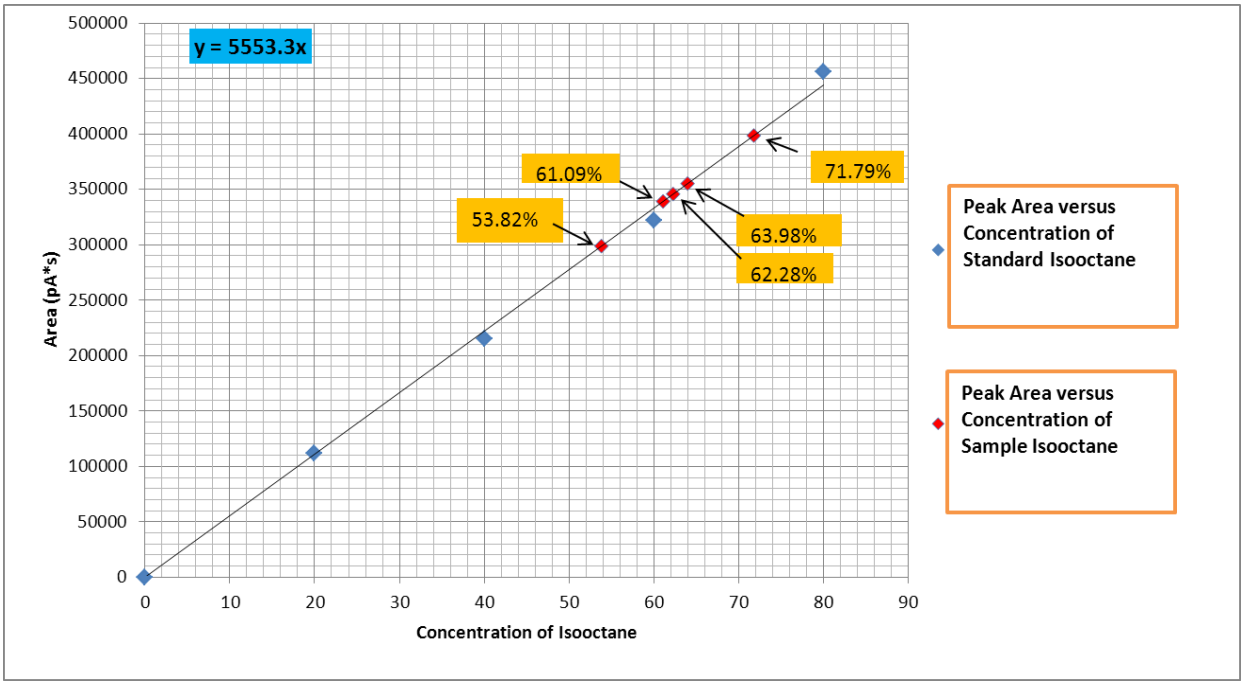


Figure 4.15 Actual Concentration of Isooctane Present In Samples (1:4)

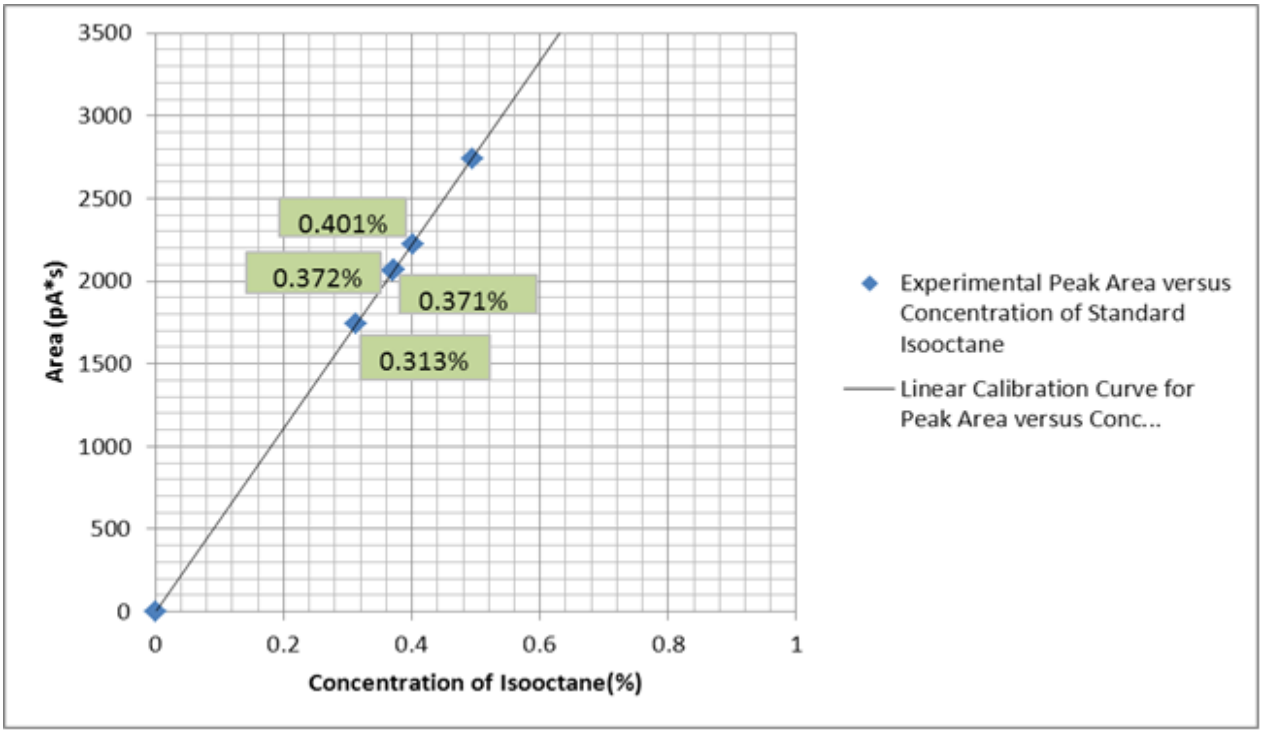


Figure 4.16 Experimental Concentration of Isooctane Present In Samples (1:4)

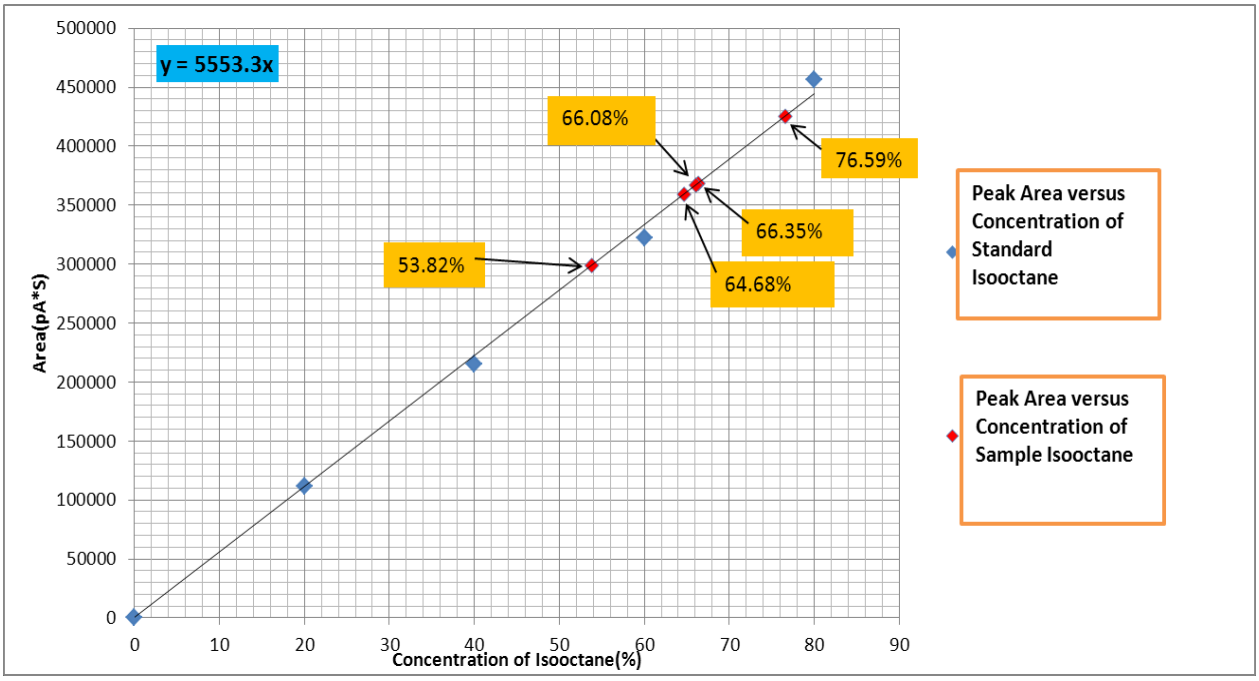


Figure 4.17 Actual Concentration of Isooctane Present In Samples (1:5)

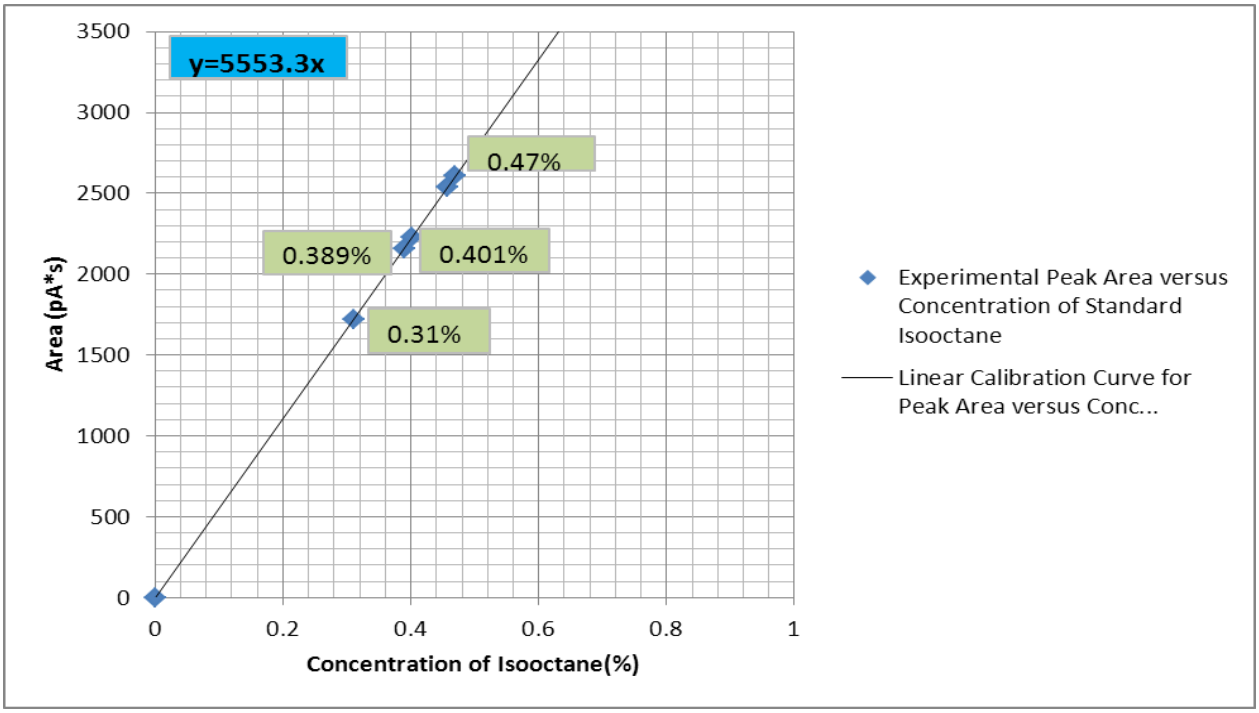


Figure 4.18 Experimental Concentration of Isooctane Present In Samples (1:5)

4.4 DISCUSSION

4.4.1 Catalyst Factor

In order to break the long hydrocarbon chain into smaller, simpler, and useful bit of compounds, sufficient amount of heat must be applied in order for the reactions to occur. The thermal cracking follows the path of higher activation energy which adds up to the production cost. The catalytic cracking is a short cut compared to the thermal cracking. It is because the presence of catalyst lowers the activation energy of the reaction, thus significantly reducing the heat duty. Overall, the catalyst also enables the reaction to be achieved in higher conversion. The long chain hydrocarbon molecules, i.e. fatty acids, are broken into various smaller hydrocarbon radicals in randomly manner. These radicals then will recombine in different arrangements through isomerization process. Zeolite plays an important role as catalyst via inhibiting active sites on its surface to enable the fatty acids to adsorb onto it. The adsorbed species will then be weakened at certain bonds along the hydrocarbon chain. The catalyst is very active in the exchange of ion for the extraction of fatty acids from rubber seed oil due to the presence of sodium as the predominant exchangeable cation. The desired Isooctane molecules are formed through this isomerization process. The presence of Isooctane produced in the rubber seed oil indicating that bio-petrol can be produced in this process.

Based on Table 4.7, the actual concentration of Isooctane for sample dilution of 1%, 5%, 10%, 15%, and 20% are 78.29%, 63.13%, 58.74%, 61.94% and 55.18% respectively. From this result, it can be discussed that the concentration of sample is inversely proportional to the actual concentration of Isooctane obtained. It can reason out that highly affect the yield of Isooctane is the catalyst factor. In this laboratory preparation, only 25ml of rubber seed oil is cracked using 5grams of Zeolite powder catalyst for the catalytic cracking process. Due to the small volume used of oil sample in the catalytic cracking process, the mixture of rubber seed oil, catalyst and 5grams of boiling chips is a very thick mixture. Besides that, after the addition of catalyst and boiling chips, the oil can hardly be seen. In this case, it can be assumed that since there is only very small volume with big portion of catalyst present, almost all of the fatty

acids present in rubber seed oil are broken into smaller molecules, creating high probability for the formation of Isooctane.

4.4.2 Various Types of Fatty Acids Present in the Rubber Seed Oil

Hevea brasiliensis, the common variety of rubber tree produces 99% of world's natural rubber. The seeds contain an oily endosperm. Generally, 37% by weight of the seed is only shell and the rest is kernel. The oil content of air dried is 47%. Rubber seed oil is a rich mixture of various fatty acids such as oleic acid and stearic acid. This factor of variety of fatty acids types in rubber seed oil gives an added advantage of multiple choices for the formation of isooctane during cracking process. Thus, the percentage yield for the desired product will be high, which is directly supported by the result of this results whereby the amount of isooctane concentration present in the sample is more than 50%.

Natural fatty acids commonly have a chain of four to 28 carbons (usually unbranched and even numbered), which may be saturated or unsaturated. Rubber seed oil consists of mainly saturated fatty acids that have long chains with no double bonds. The breaking of bond usually occurs in the middle section of the molecule thus giving higher probability of forming isooctane molecules which is very much likely in our case. Most of the fatty acids in the rubber seed oil are consist of straight chains hydrocarbons; palmitic acid, stearic, oleic, linoleic, linolenic, and erucic. Each and any of the C-C bonds can be broken up to form Isooctane. This is logical as the Zeolite catalyst can swell to several times of its original; more percentage of Isooctane concentration can be formed.

4.4.3 Contamination Factor

The result shows many other components present in the chromatogram during the analysis. Though isooctane and hexane are given importance in the readings, but the presence of other components does makes an alteration in the experimental results as it does differs minimally from the expected result. The probable cause for such occurrence could be the contamination factor. During the experiment, there might be impurities present in the apparatus and glassware used that will give an effect on the determination of Isooctane especially during the sample analysis using Gas Chromatogram.

Dilution process also is susceptible to contamination. In this process contamination is very much likely to occur especially when the dilution steps are done in a closed and air conditioned lab. It is because vegetable oils are very much likely to solidify below 25^{o}C. Isooctane is a liquid at room temperature but the unreacted fatty acid will affect the Isooctane formation as the temperature falls below the melting point of the fatty acids. The more the sample is dominated by the distillated product, the higher chances for the sample to be solidified.

One of the observations supports this fact, that is, the actual concentration of isooctane in the sample decreases as the dilution factor increases. This may also been caused by the sucking of other lighter and long chain hydrocarbon molecules along the micropipette.

4.4.4 Hydrocarbon Isomerization

Cracking is the process which breaks long chain hydrocarbon molecules into smaller chains. In this process the free radicals will combine to form new molecules in a random basis, in this case isooctane more likely to be formed. The high concentration of isooctane in the sample is due to the isomerization process. Long chain hydrocarbons are cracked into smaller branched molecules. Fatty acids are very much likely to be cracked to molecules smaller than decane. This is because the high heat applied in the process with the presence of catalyst influences much of the long chained molecules to

break down into smaller ones, and in this case isooctane is the much desired molecule as its' characteristic of stable molecular structure.

4.4.5 Mass Ratio of Rubber Seed to Solvent

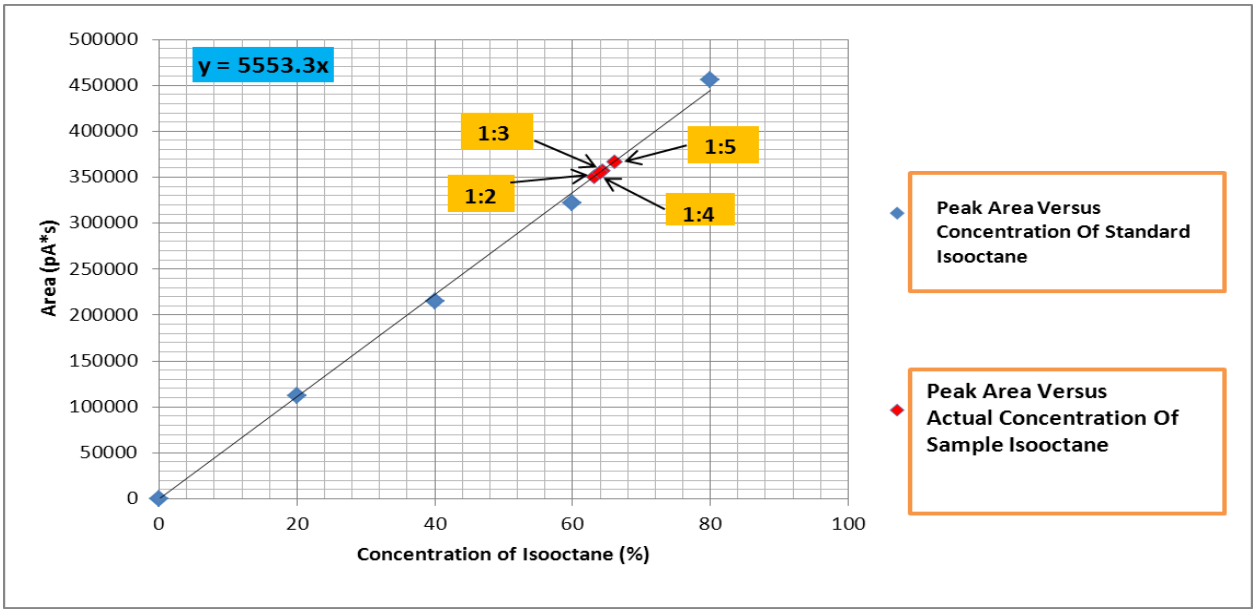


Figure 4.19 Actual Concentration of Isooctane Present In Each Ratio for 1% Dilution

The figure shows that the actual concentration of isooctane present in all mass ratios of solvent and rubber seed is in the range from 63% to 66%. The increasing of mass ratio shows an increasing trend in actual isooctane concentration, but the difference of concentration is not considered to be significant enough to support the idea that effect of solvent to rubber seed ratio could alter the yield of isooctane. Based from the experiment observations, the solvent-rubber seed ratio plays a significant role only in the yield of rubber seed oil from the solid kernel during the Soxhlet extraction process. It has been observed that, oil colour changes from darker brown to lighter brown as the mass ratio increased. Darker brown colour of oil indicates that the amount of saturated fatty acids present in the mixture is very much higher than unsaturated fatty acids. The higher ratios give lighter colour of evaporated oil can be explained by the fact the extra amount of solvents forms hydrogen bonds with the fatty acids thus preventing the fatty acids molecules from forming colloids which solidifies in room temperature.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

Based on this study using various external resources and experimental data as well, it has been confident enough that rubber seed possessed a sufficient amount of potential virgin oils needed to synthesize biopetrol. In this study, fatty acids have been successfully extracted form rubber seed kernel using Soxhlet extraction method whereby acetone is used as extraction solvent. For lab scale, Soxhlet extraction method is considered as an effective tool for extracting rubber seed oil. The role of acetone in this process for extracting oil is found convincing in yielding high volume of oil from rubber seed. The presence of oxygen molecule in acetone molecule exhibits polarity characteristic. This polar solvent attracts more fatty acids towards it compared to conventional alkane solvents. Experimental observation supports this claim, where 100grams of rubber seeds yields 60ml of crude rubber seed oil.

Furthermore, the amount of solvent used in the extraction process does not have a significant effect in the concentration of Isooctane in the cracking reaction. The mass ratio of solvent-rubber seed would not be a deciding factor for the whole study even as it has been set as a crucial parameter. In short, the synthesizing biopetrol from rubber seed through heterogeneous catalytic is possible, and the effect of solvent in extraction process does not significantly affect the yield of Isooctane. The concentration of Isooctane that obtained from catalytic cracking is from 53% to 78% as been proved in backward calculation. Also, sample analysis through gas Chromatography method proven to be reliable and effective in determining the concentration of Isooctane in sample. This experiment has successfully proved that isooctane can be synthesized from rubber seed by heterogeneous catalytic cracking using Zeolite as catalyst.

5.2 RECOMMENDATIONS

As the world faces energy crisis with the situation could be worsen in future as the fossil fuel resources running out quicker than expected. Catalytic cracking of rubber seed oil is beneficial to widen the renewable sources of energy in the future. However this alternative process should be upgraded with further technology in terms of technical, economic, environmental, and feasibility for large scale production or industrial process.

Soxhlet process makes the extraction of fatty acids form rubber seeds feasible, but this method poses serious drawbacks compared to other extraction processes. One of the important factors is extraction time. This process requires 6 to 8 hours to complete extraction and additionally 1 to 2 hours of cooling time needed to retrieve the mixture from the round bottom flask. Long hours of process does makes this research duration lengthy whereby other processes are in queue waiting for the completion of this part. Besides that, large amount of extracting solvent is wasted off in the thimble filter and some evaporating through the leaks of the apparatus thus posing environmental problems. Operating cost of a single unit very high due to the constant boiling of the solvent and also the continuous water flow in the condenser adds up to the cost where both energy and water resources are wasted very much. To overcome this problem it is suggested that Soxhlet extraction method should be replaced by Supercritical Carbon Dioxide (CO_2) extraction method. This method can be used to extract oil form rubber seed kernel where the use of supercritical CO_2 is done in a closed and sealed vessel thus eliminating the leakage problem. Also, the supercritical CO_2 does not pose health if inhaled or environmental problem if the gas is leaked through some faulty seals. This method is very much economical because it does not require heating or cooling factor; it only uses pure CO2 in extraction. The only drawback in this process is the risk of explosion of vessel or pipe during experiment due to the high pressure of CO_2 gas in the cylinder.

Apart from this method, Microwave Assisted Hydrodistillation (MAHD) can be used to extract rubber seed oil. MAHD is an advanced hydrodistillation method based on the use of microwave oven. In this process, energy is delivered rapidly to the whole

volume of solvent and rubber seed mixture thus leading to a rapid rise in temperature in a short period of time. The major benefit of this method is the shorter duration of process compared to Soxhlet method and it only takes from 45 minutes to 2 hours to complete the extraction.

Another recommendation can be considered in the experimental works is the filling of inert gases or nitrogen gas into the furnace vessel. Before commencing the experiment, the chamber should be evacuated to prevent oxygen from involving in the cracking process to avoid oxidation. This is to ensure that cracking of fatty acids effective and this corresponds to the increase of isooctane yield more than the current technique. Catalytic cracking done in the absence of oxygen is crucial to avoid burning of oil or product in the furnace due to the high temperature requirement of the process.

Human error and contamination could not be avoided in the experiment, which caused this study results to differ slightly from theoretical predictions. Maintaining clean condition and appropriate use of apparatus could minimize the contamination and interference in order to get more accurate results.

REFERENCES

E. Ikwuagwu *, I.C. Ononogbu, O.U. Njoku, 2000, Production of biodiesel using rubber[He6ea brasiliensis (Kunth. Muell.)] seed oil

A.S. Ramadhas*, S. Jayaraj, C. Muraleedharan, 2005, Biodiesel production from high FFA rubber seed oil

Pramila Tamunaidu, Subhash Bhatia, Journal on Catalytic Cracking of Palm Oil for the Production of Biofuels: Optimization Studies, School of Chemical Engineering, USM. 2006

Thiam Leng Chew, Subhash Bhatia, Journal on Catalytic Processes towards the Production of Biofuels in a Palm Oil and Oil Palm Biomass based biorefinery, School of Chemical Engineering, USM. 2006

Agustin Martinez, Susana Valencia, Raul Murciano, Henrique S. Cerqueira, Alexandre F. Costa, Eduardo Falabella S.-Aguir, Journal on Catalytic Behavior of Hybrid Co/SiO_2-(medium-pore) Zeolite catalysts during the One Stage Conversion of Syngas to Gasoline, PETROBAS, Brazil. 2008

M.S. Kuk*, R. Tetlow, and M.K. Dowd, Cottonseed Extraction with Mixtures of Acetone and Hexane, 2006

Kuk, M.S., Use of Hildebrand's Solubility Parameter in Oilseed Lipid Extraction, in Advances in Oils and Fats, Antioxidants,and Oilseed By-Products, Vol. II of Proceedings of the World Conference on Oilseed and Edible Oils Processing, edited by S.S. Koseoglu, K.C. Rhee, and R.F. Wilson, AOCS Press, Champaign, 1998, pp. 9–14.

Lusas, E.W., L.R. Watkin, S.S. Koseoglu, K.C. Rhee, and E.Hernandez, Replacing Hexane with Isopropyl Alcohol as an Oil Extraction Solvent, INFORM 5:525 (1994).

Kuk, M.S., and R.J. Hron, Jr., Cottonseed Extraction with a New Solvent System: Isohexane and Alcohol Mixtures, J. Am. OilChem. Soc. 75:927–930 (1998).

Abraham, G., K.M. Decossas, R.J. Hron, Jr., and M.S. Kuk, Process Engineering Economic Evaluation of Ethanol Extraction of Cottonseed: Preliminary Analysis, Ibid. 68:418–421 (1991).

Vaccarino, C., A New Process for Cottonseed, Ibid. 38:143–147 (1961).

Hron, R.J., Jr., and M.S. Kuk, Acetone Extracted Cottonseed Meals Without Catty Odor,J. Food Sci. 54:1088–1089 (1989).

Official Methods and Recommended Practices of the American Oil Chemists' Society, 4th edn., AOCS Press, Champaign, 1993.

Shankaransh Srivastava, Fatty acid composition of rubber seed oil , 2000

Aigbodion A.I. and C.K.S., Pillai, Processability characteristics and physico-mechanical properties of natural rubber modified with rubber seed oil and epoxidized rubber seed oil(2000)

James H. Gary and Glenn E. Handwerk, Petroleum Refining: Technology and Economics,(2001)

Fangrui Maa, Milford A. Hannab (1999). Biodiesel production: a review

Ma F, Hanna MA. Biodiesel production: a review. Bioresource Technol 1999;70:1–15

Carlo Perego, Extruded Catalyst Based On Silica/alumina Gel, 2010

M.S. Kuk ,Supercritical carbon dioxide extraction of cottonseed oil , 2005

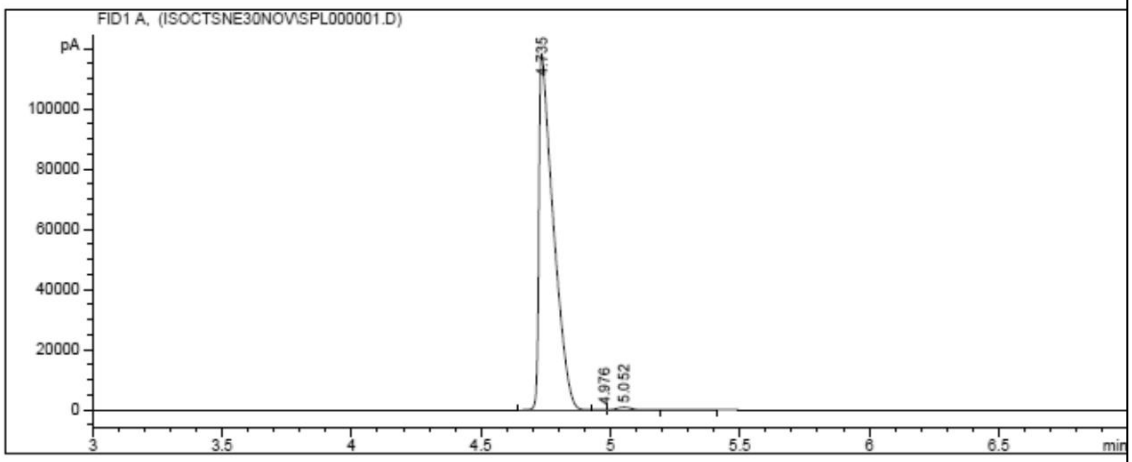
Appendix A

Chromatogram for Standard Isooctane

Chromatogram for Different Dilution Percentage for each Ratio

GC FID ANALYSIS: 0% STANDARD ISOOCTANE SOLUTION

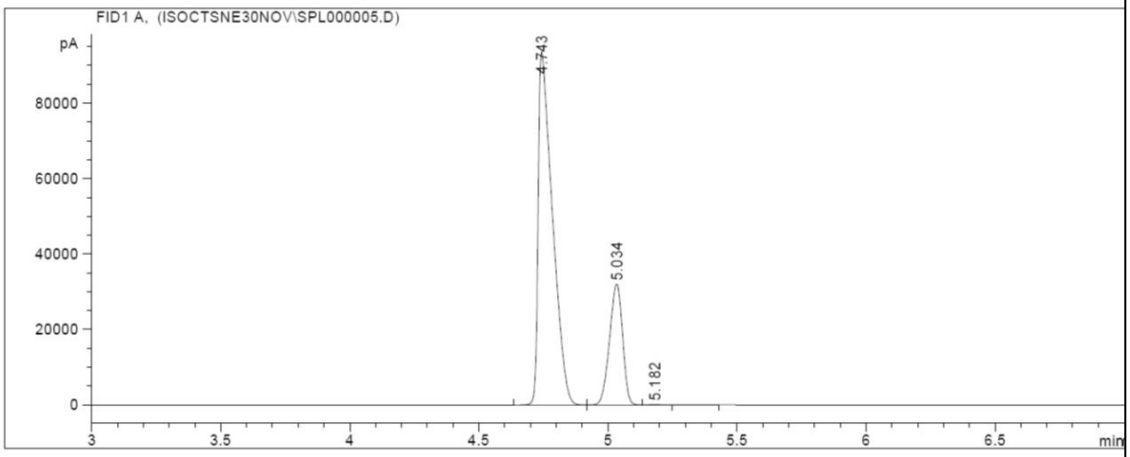
```
Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL000001.D
 Sample Name: s 1
    ======================================================================
    Acq. Operator    : fiza30nov                       Seq. Line :   1
    Acq. Instrument : Instrument 1                      Location : Vial 1
    Injection Date   : 30/11/2011 10:00:11                   Inj :   1
                                                      Inj Volume : 1 µl
    Acq. Method      : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed     : 30/11/2011 09:58:21  by fiza30nov
    Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed     : 13/12/2011 15:10:48  by fiza30nov
                       (modified after loading)
    Method Info      : isooctane/psm

    ======================================================================
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      27 August 2009 13:05:28
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime  Type   Width     Area      Area    Name
      #   [min]          [min]   [pA*s]       %
    ----|-------|------|-------|----------|--------|--------------------
       1   2.185         0.0000    0.00000  0.00000
       2   2.253         0.0000    0.00000  0.00000
       3   2.308         0.0000    0.00000  0.00000
       4   2.359         0.0000    0.00000  0.00000
       5   2.762         0.0000    0.00000  0.00000
       6   4.735 VB S    0.0525 4.56593e5  99.31207
       7   4.976 BV X    0.0349  140.91379  0.03065 ?
       8   5.052 VV X    0.0555 3021.87866  0.65728 ?

    Totals :                   4.59756e5
```

GC FID ANALYSIS: 20% STANDARD ISOOCTANE SOLUTION

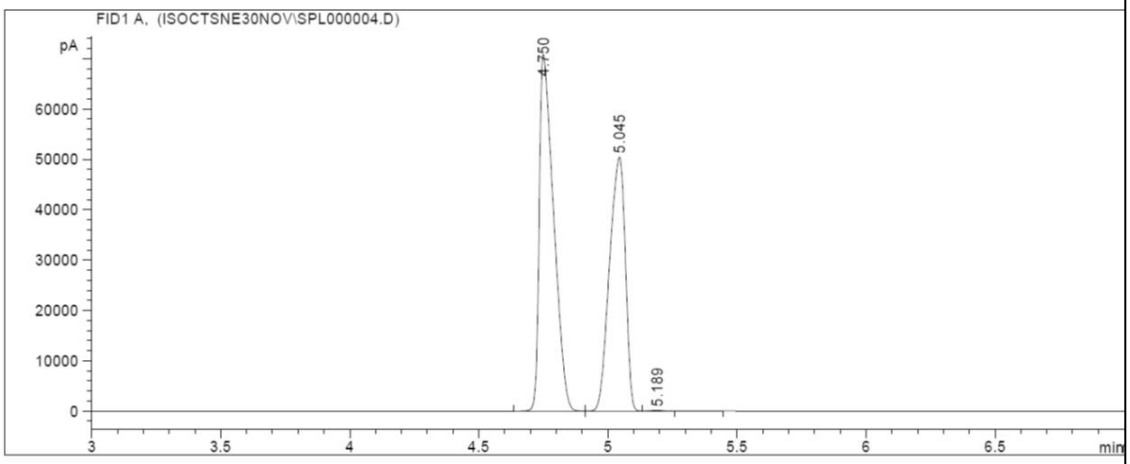
```
Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL000005.D
 Sample Name: s5
    ======================================================================
    Acq. Operator    : fiza30nov                       Seq. Line :   5
    Acq. Instrument : Instrument 1                      Location : Vial 5
    Injection Date  : 30/11/2011 11:30:10                    Inj :   1
                                                      Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 30/11/2011 09:58:21  by fiza30nov
    Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 13/12/2011 15:03:56  by fiza30nov
                      (modified after loading)
    Method Info     : isooctane/psm

    ======================================================================
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      27 August 2009 13:05:28
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime  Type   Width     Area      Area    Name
      #   [min]          [min]   [pA*s]       %
    ----|-------|------|-------|----------|--------|--------------------
       1   2.185         0.0000    0.00000  0.00000
       2   2.253         0.0000    0.00000  0.00000
       3   2.308         0.0000    0.00000  0.00000
       4   2.359         0.0000    0.00000  0.00000
       5   2.762         0.0000    0.00000  0.00000
       6   4.743 VV S    0.0541 3.57789e5  76.06407
       7   5.034 VB S    0.0545 1.12430e5  23.90208 ?
       8   5.182 BV T    0.0468  159.23813  0.03385 ?

    Totals :                   4.70378e5
```

GC FID ANALYSIS: 40% STANDARD ISOOCTANE SOLUTION

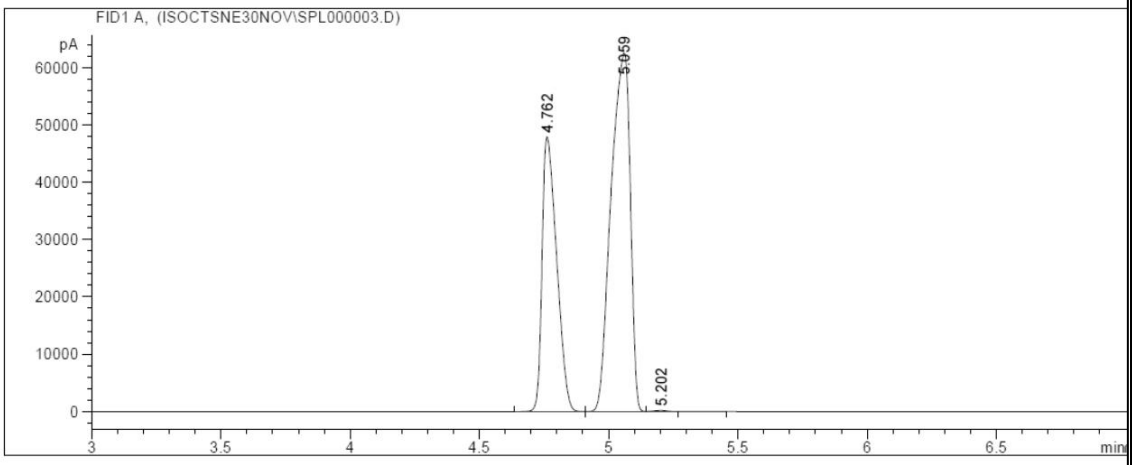
```
Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL000004.D
 Sample Name: s4
   ======================================================================
   Acq. Operator    : fiza30nov                       Seq. Line :   4
   Acq. Instrument : Instrument 1                      Location : Vial 4
   Injection Date  : 30/11/2011 11:07:43                    Inj :   1
                                                    Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 30/11/2011 09:58:21  by fiza30nov
   Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 13/12/2011 15:03:56  by fiza30nov
                     (modified after loading)
   Method Info     : isooctane/psm

   ======================================================================
```




```
   ======================================================================
                            Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Calib. Data Modified  :      27 August 2009 13:05:28
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime  Type   Width     Area      Area    Name
     #   [min]          [min]   [pA*s]        %
   ----|-------|------|-------|----------|--------|--------------------
      1   2.185         0.0000    0.00000  0.00000
      2   2.253         0.0000    0.00000  0.00000
      3   2.308         0.0000    0.00000  0.00000
      4   2.359         0.0000    0.00000  0.00000
      5   2.762         0.0000    0.00000  0.00000
      6   4.750 VV S    0.0524 2.72851e5  55.84981
      7   5.045 VB S    0.0708 2.15359e5  44.08180 ?
      8   5.189 BV T    0.0491  334.12787  0.06839 ?

   Totals :                   4.88544e5
```

GC FID ANALYSIS: 60% STANDARD ISOOCTANE SOLUTION

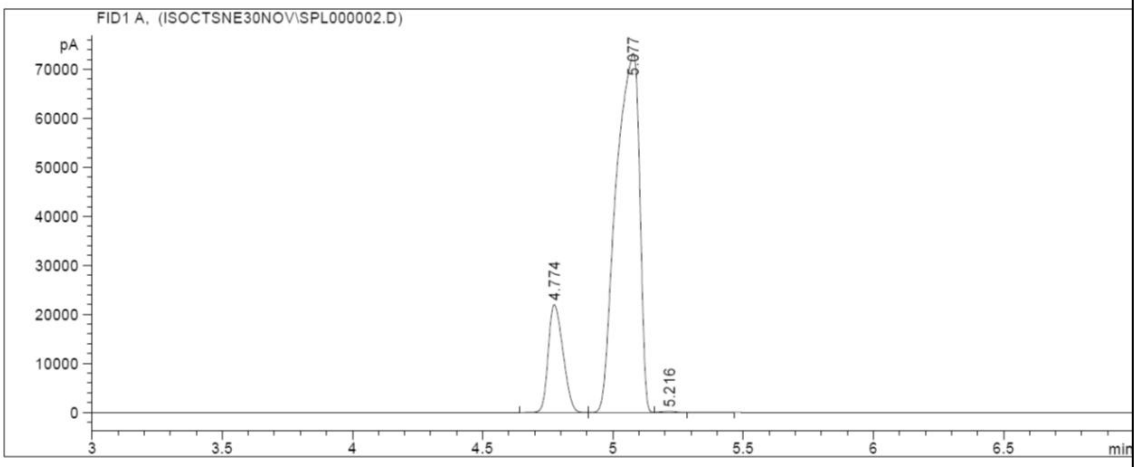
```
Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL000003.D
 Sample Name: s3
    ======================================================================
    Acq. Operator   : fiza30nov                      Seq. Line :   3
    Acq. Instrument : Instrument 1                    Location : Vial 3
    Injection Date  : 30/11/2011 10:45:10                  Inj :   1
                                                    Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 30/11/2011 09:58:21  by fiza30nov
    Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 13/12/2011 15:03:56  by fiza30nov
                      (modified after loading)
    Method Info     : isooctane/psm

    ======================================================================
```




```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      27 August 2009 13:05:28
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime  Type   Width     Area      Area    Name
      #   [min]          [min]   [pA*s]        %
    ----|-------|------|-------|----------|--------|--------------------
       1   2.185         0.0000    0.00000  0.00000
       2   2.253         0.0000    0.00000  0.00000
       3   2.308         0.0000    0.00000  0.00000
       4   2.359         0.0000    0.00000  0.00000
       5   2.762         0.0000    0.00000  0.00000
       6   4.762 VV S    0.0590 1.86902e5  36.68721
       7   5.059 VB S    0.0766 3.22048e5  63.21528 ?
       8   5.202 BV T    0.0502  496.78217  0.09751 ?

    Totals :                   5.09446e5
```

GC FID ANALYSIS: 80% STANDARD ISOOCTANE SOLUTION

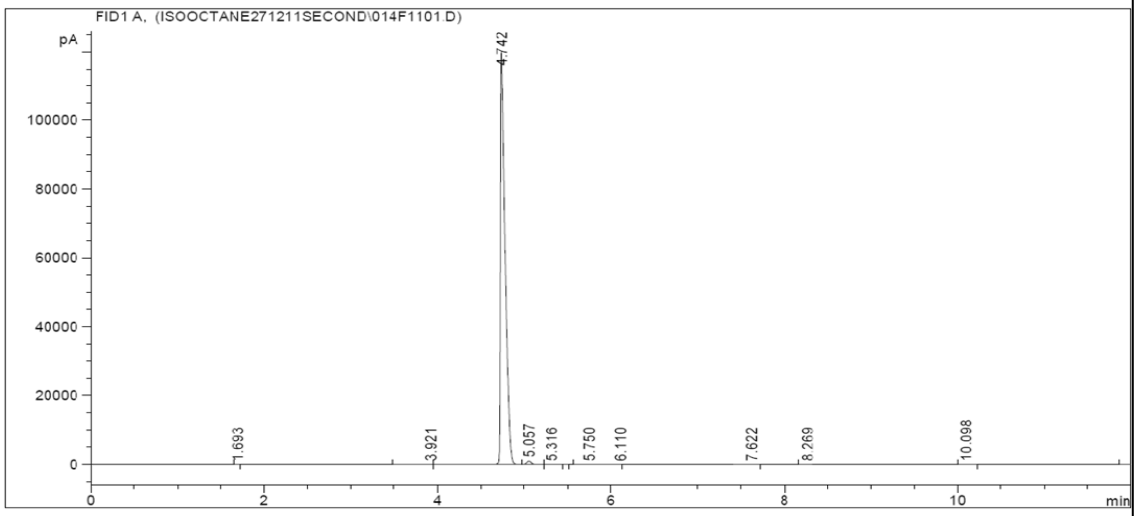
```
Data File C:\CHEM32\1\DATA\ISOCTSNE30NOV\SPL000002.D
 Sample Name: s2
   ======================================================================
   Acq. Operator    : fiza30nov                       Seq. Line :   2
   Acq. Instrument : Instrument 1                      Location : Vial 2
   Injection Date  : 30/11/2011 10:22:40                    Inj :   1
                                                    Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 30/11/2011 09:58:21  by fiza30nov
   Analysis Method : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 13/12/2011 15:09:52  by fiza30nov
                     (modified after loading)
   Method Info     : isooctane/psm

   ======================================================================
```




```
   ======================================================================
                              Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Calib. Data Modified  :      27 August 2009 13:05:28
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime  Type   Width     Area      Area    Name
     #   [min]          [min]   [pA*s]       %
   ----|-------|------|-------|----------|--------|--------------------
      1   2.185         0.0000    0.00000  0.00000
      2   2.253         0.0000    0.00000  0.00000
      3   2.308         0.0000    0.00000  0.00000
      4   2.359         0.0000    0.00000  0.00000
      5   2.762         0.0000    0.00000  0.00000
      6   4.774 BV S    0.0602 8.61105e4  15.85754
      7   5.077 VB S    0.0828 4.56253e5  84.02050 ?
      8   5.216 BV T    0.0503  662.29755  0.12196 ?

   Totals :                   5.43025e5
```

GC FID ANALYSIS: 1% SAMPLE DILUTION FOR THE RATIO 1:2

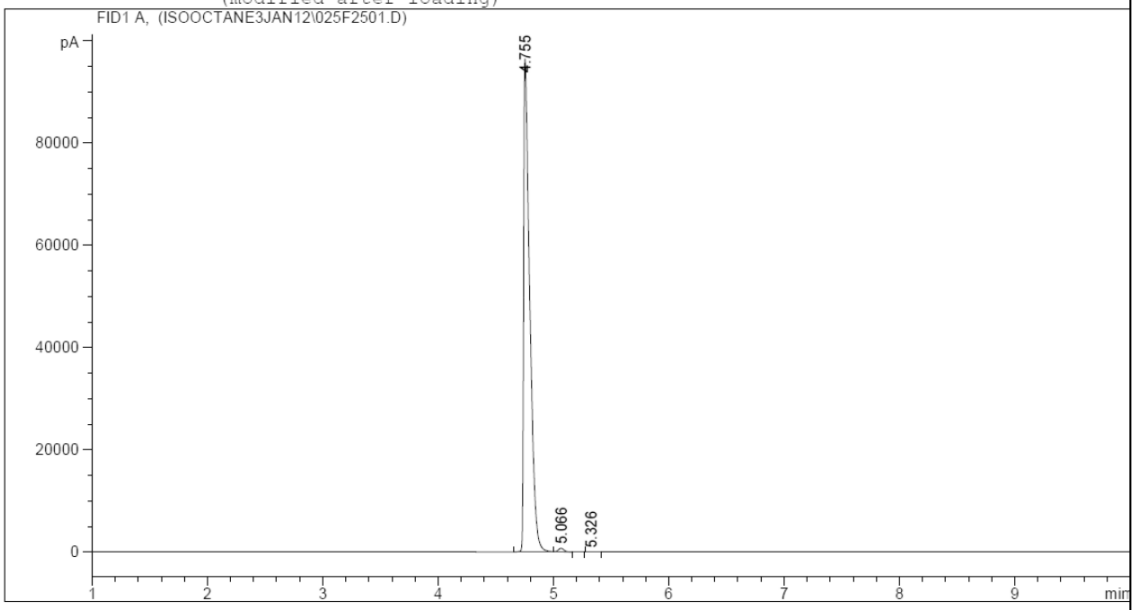
```
Data File C:\CHEM32\1\DATA\ISOOCTANE271211SECOND\014F1101.D
 Sample Name: D1N2sp2

    ======================================================================
    Acq. Operator    : wani27dec                       Seq. Line :  11
    Acq. Instrument : Instrument 1                      Location : Vial 14
    Injection Date  : 27/12/2011 15:32:57                    Inj :   1
                                                     Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 20/12/2011 09:21:16  by fiza20nov
    Analysis Method : C:\CHEM32\1\METHODS\B4ASHUTDOWN.M
    Last changed    : 28/12/2011 10:27:49  by wani27dec
                      (modified after loading)
    Method Info     : std testing
```




```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [pA*s]      [pA]        %
    ----|-------|----|-------|----------|----------|--------|
       1   1.693 BV    0.0475    2.68428 7.19730e-1  0.00062
       2   3.921 BV    0.1542    7.36331 5.72106e-1  0.00169
       3   4.742 VB S  0.0526 4.31591e5  1.18222e5  99.24625
       4   5.057 BV X  0.0537 3189.88867  938.43292  0.73353
       5   5.316 VB X  0.0554   62.74586   18.10252  0.01443
       6   5.750 BV    0.1480    1.91038 1.59217e-1  0.00044
       7   6.110 VV    0.0938    2.30371 3.20813e-1  0.00053
       8   7.622 BB    0.0593    1.08270 2.84988e-1  0.00025
       9   8.269 BV    0.0551    4.39290    1.02363  0.00101
      10  10.098 BB    0.0715    1.35743 3.01504e-1  0.00031
      11  12.083 BB    0.1904    2.81386 1.81005e-1  0.00065
      12  15.326 BB    0.0904    1.28769 2.04590e-1  0.00030


Instrument 1 28/12/2011 10:33:30  wani27dec                          Page   1 of 2

     Totals :                   4.34869e5  1.19182e5
```

GC FID ANALYSIS: 5% SAMPLE DILUTION FOR THE RATIO 1:2

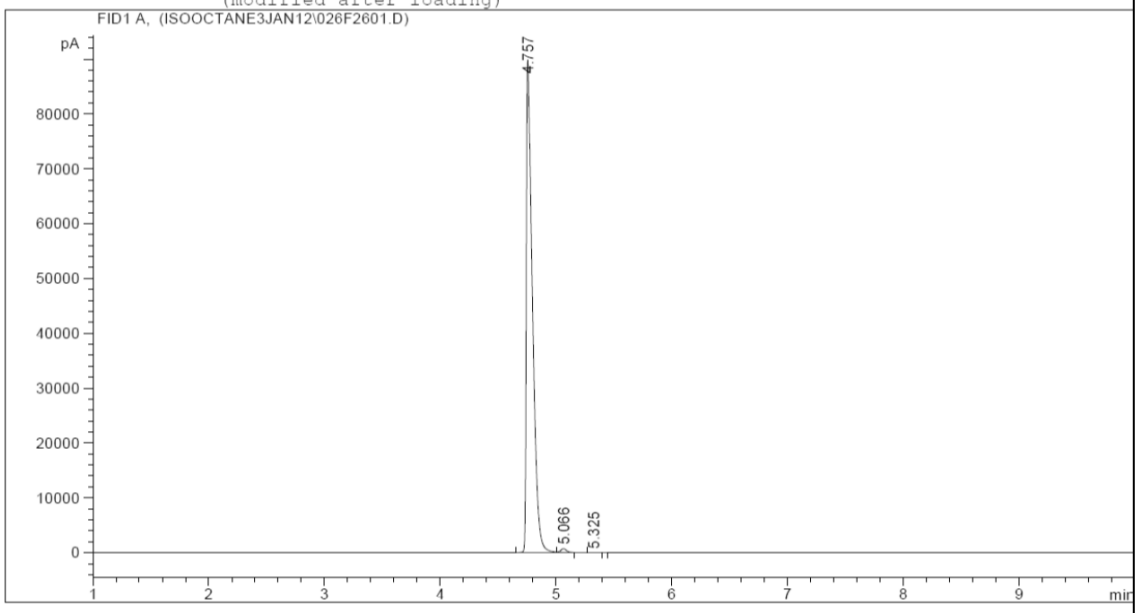
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\025F2501.D
 Sample Name: D1N2 5%

   ======================================================================
   Acq. Operator   : fiza3jan                       Seq. Line :  25
   Acq. Instrument : Instrument 1                   Location : Vial 25
   Injection Date  : 03/01/2012 18:26:29               Inj :   1
                                                   Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ======================================================================
                            Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]        %
   ----|-------|----|-------|----------|----------|--------|
      1   4.755 BB S  0.0537 3.52007e5  9.60973e4  99.32369
      2   5.066 BB X  0.0532 2371.14502  706.01501  0.66905
      3   5.326 BB    0.0497   25.72105    8.18742  0.00726

   Totals :                  3.54404e5  9.68115e4


   ======================================================================
                            *** End of Report ***
```

GC FID ANALYSIS: 10% SAMPLE DILUTION FOR THE RATIO 1:2

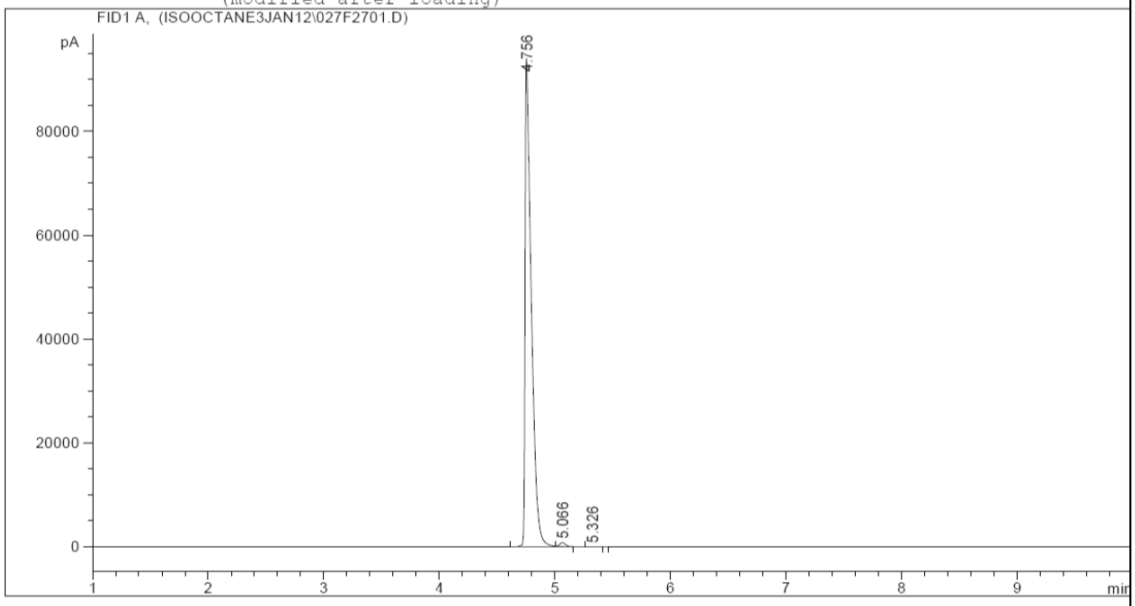
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\026F2601.D
 Sample Name: D1N2 10%

    ========================================================================
    Acq. Operator    : fiza3jan                        Seq. Line :  26
    Acq. Instrument : Instrument 1                      Location : Vial 26
    Injection Date  : 03/01/2012 18:48:58                    Inj :   1
                                                     Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 20/12/2011 09:21:16  by fiza20nov
    Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
    Last changed    : 05/01/2012 09:24:08  by fiza4jan
                      (modified after loading)
```




```
    ========================================================================
                              Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [pA*s]      [pA]        %
    ----|-------|----|-------|----------|----------|--------|
       1   4.757 BB S  0.0537 3.27489e5  8.94200e4  99.35252
       2   5.066 BB X  0.0517 2112.25952  636.57068  0.64081
       3   5.325 BB X  0.0526   21.97687    6.46669  0.00667

    Totals :                  3.29624e5  9.00630e4


    ========================================================================
                              *** End of Report ***
```

GC FID ANALYSIS: 15% SAMPLE DILUTION FOR THE RATIO 1:2

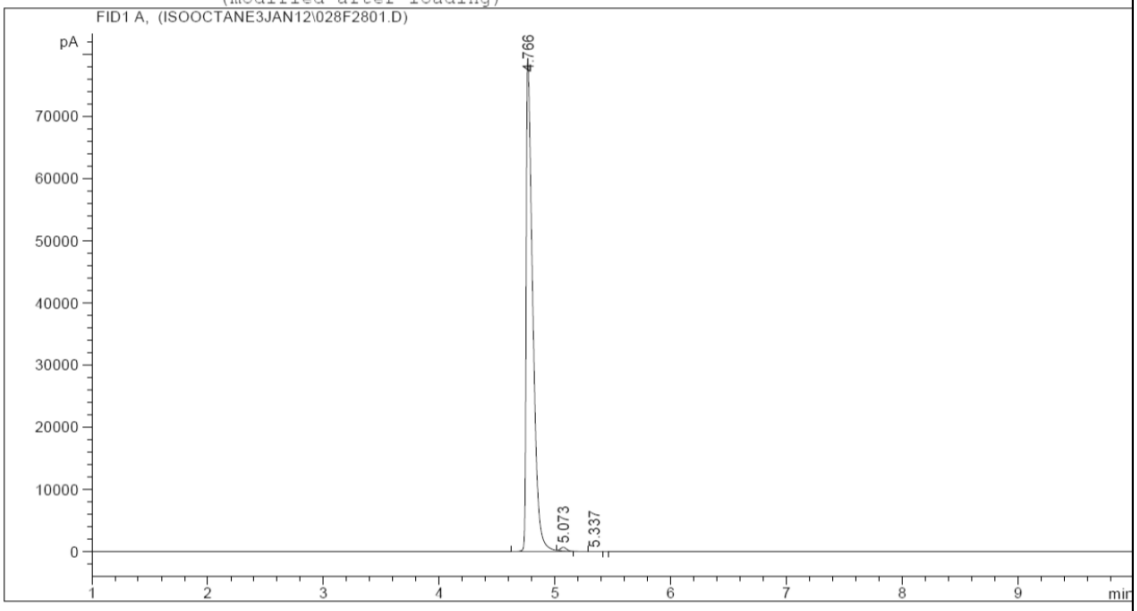
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\027F2701.D
 Sample Name: D1N2 15%

    ========================================================================
    Acq. Operator    : fiza3jan                        Seq. Line :  27
    Acq. Instrument : Instrument 1                      Location : Vial 27
    Injection Date  : 03/01/2012 19:11:30                    Inj :   1
                                                     Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 20/12/2011 09:21:16  by fiza20nov
    Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
    Last changed    : 05/01/2012 09:24:08  by fiza4jan
                      (modified after loading)
```




```
    ========================================================================
                               Area Percent Report
    ========================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [pA*s]      [pA]        %
    ----|-------|----|-------|----------|----------|--------|
       1   4.756 BB S  0.0505 3.50397e5  9.39951e4  99.28519
       2   5.066 BB X  0.0523 2458.87842  730.80341  0.69672
       3   5.326 BB X  0.0549   63.84619   18.21056  0.01809

    Totals :                  3.52920e5  9.47441e4


    ========================================================================
                               *** End of Report ***
```

GC FID ANALYSIS: 20% SAMPLE DILUTION FOR THE RATIO 1:2

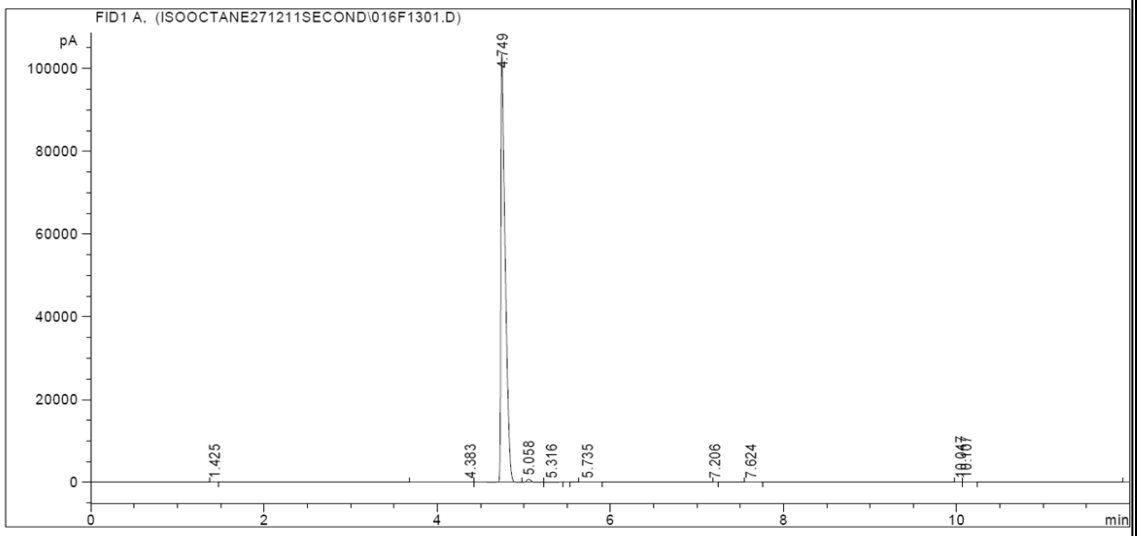
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\028F2801.D
 Sample Name: D1N2 20%

   ========================================================================
   Acq. Operator   : fiza3jan                        Seq. Line :  28
   Acq. Instrument : Instrument 1                    Location : Vial 28
   Injection Date  : 03/01/2012 19:34:06               Inj :   1
                                                    Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ========================================================================
                              Area Percent Report
   ========================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]       [min]    [pA*s]      [pA]       %
   ----|-------|----|-------|----------|----------|--------|
      1   4.766 BB S  0.0566 3.10548e5  7.94662e4  99.35395
      2   5.073 BB X  0.0549 1979.79944  564.09583  0.63340
      3   5.337 BB X  0.0561   39.54865   10.96636  0.01265

   Totals :                  3.12567e5  8.00413e4


   ========================================================================
                              *** End of Report ***
```

GC FID ANALYSIS: 1% SAMPLE DILUTION FOR THE RATIO 1:3

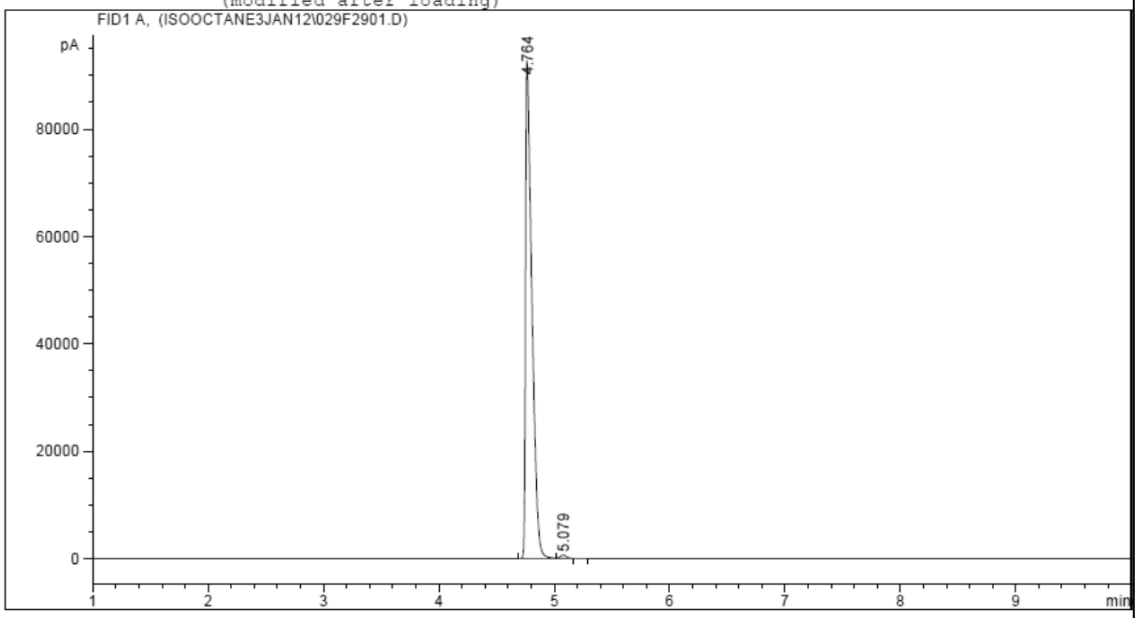
```
Data File C:\CHEM32\1\DATA\ISOOCTANE271211SECOND\016F1301.D
 Sample Name: D1N3sp2

   ======================================================================
   Acq. Operator   : wani27dec                       Seq. Line :  13
   Acq. Instrument : Instrument 1                     Location : Vial 16
   Injection Date  : 27/12/2011 16:17:58                  Inj :   1
                                                    Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\B4ASHUTDOWN.M
   Last changed    : 28/12/2011 10:27:49  by wani27dec
                     (modified after loading)
   Method Info     : std testing
```




```
   ======================================================================
                              Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]       %
   ----|-------|----|-------|----------|----------|--------|
      1   1.425 BV    0.0586    3.01587 6.44792e-1  0.00083
      2   4.383 BV    0.1773   13.83694 9.31429e-1  0.00380
      3   4.749 VB S  0.0511 3.61905e5  1.02745e5  99.30098
      4   5.058 BV X  0.0516 2496.32813  754.79321  0.68495
      5   5.316 VB X  0.0545   18.85948    5.42775  0.00517
      6   5.735 BB    0.0828    1.06018 1.61041e-1  0.00029
      7   7.206 BV    0.0249    1.14062 6.37786e-1  0.00031
      8   7.624 BB    0.0819    1.54242 2.73172e-1  0.00042
      9  10.047 BV    0.0567    2.47782 6.63985e-1  0.00068
     10  10.107 VB    0.0826    5.10769 9.37330e-1  0.00140
     11  12.082 BB    0.1690    2.47974 1.83123e-1  0.00068
     12  15.653 BV    0.0319    1.74317 7.28592e-1  0.00048

      Totals :                 3.64452e5  1.03511e5
```

GC FID ANALYSIS: 5% SAMPLE DILUTION FOR THE RATIO 1:3

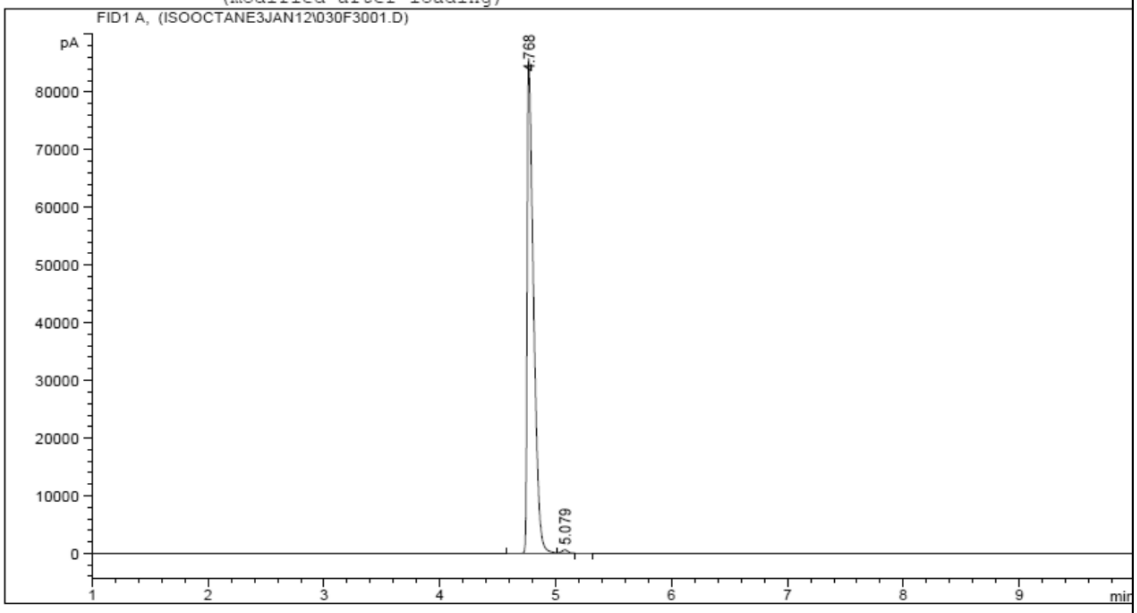
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\029F2901.D
 Sample Name: D1N3 5%

   ========================================================================
   Acq. Operator    : fiza3jan                        Seq. Line :  29
   Acq. Instrument : Instrument 1                     Location : Vial 29
   Injection Date  : 03/01/2012 19:56:39                   Inj :   1
                                                    Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ========================================================================
                              Area Percent Report
   ========================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]        %
   ----|-------|----|-------|----------|----------|--------|
      1   4.764 BB S  0.0558 3.54983e5  9.24159e4  99.38314
      2   5.079 BB T  0.0573 2203.32129  607.78876  0.61686

   Totals :                  3.57186e5  9.30237e4


   ========================================================================
                              *** End of Report ***
```

GC FID ANALYSIS: 10% SAMPLE DILUTION FOR THE RATIO 1:3

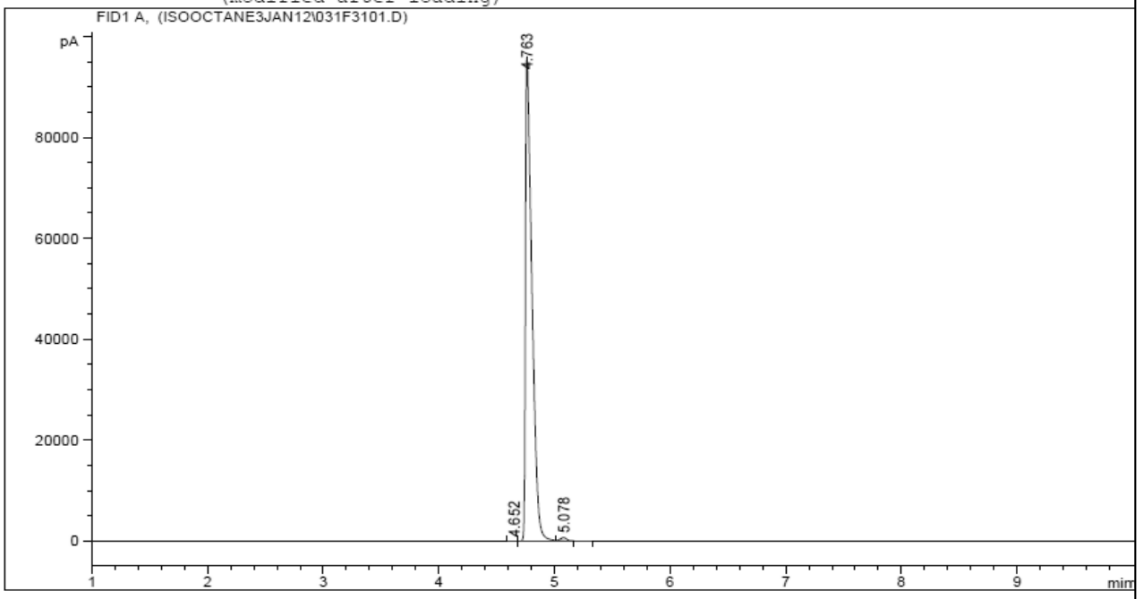
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\030F3001.D
 Sample Name: D1N3 10%

   ======================================================================
   Acq. Operator    : fiza3jan                       Seq. Line :  30
   Acq. Instrument : Instrument 1                     Location : Vial 30
   Injection Date  : 03/01/2012 20:19:10                 Inj :   1
                                                    Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ======================================================================
                              Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]       %
   ----|-------|----|-------|----------|----------|--------|
      1   4.768 BB S  0.0548 3.28497e5  8.54720e4  99.39095
      2   5.079 BB X  0.0561 1994.68616  566.21265  0.60352
      3  10.156 BB    0.0640   18.28007    4.35045  0.00553

   Totals :                  3.30510e5  8.60425e4


   ======================================================================
                              *** End of Report ***
```

GC FID ANALYSIS: 15% SAMPLE DILUTION FOR THE RATIO 1:3

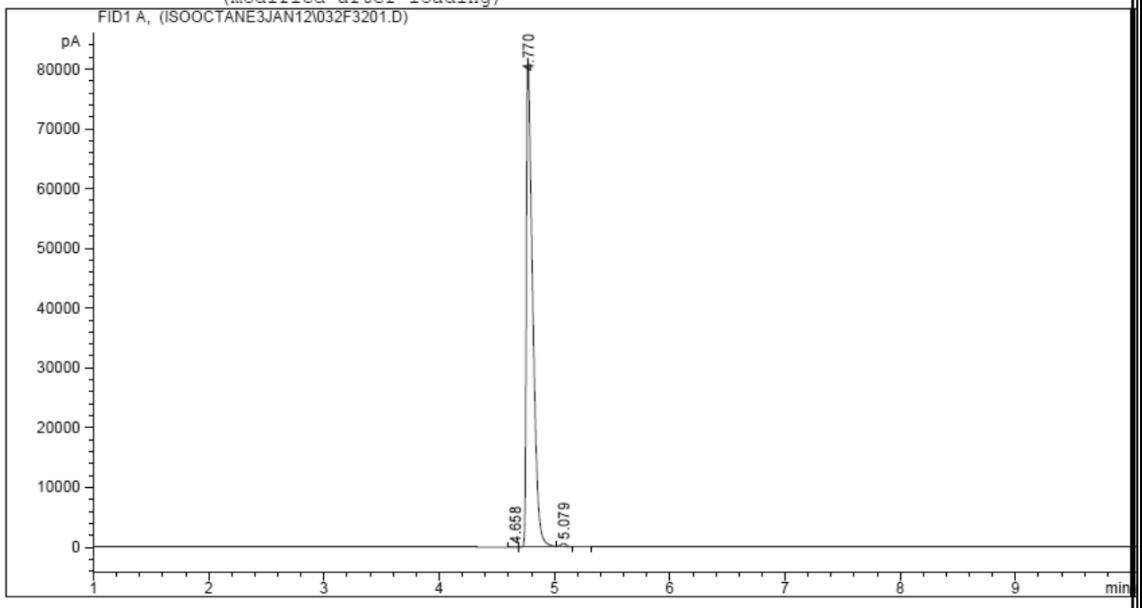
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\031F3101.D
 Sample Name: D1N3 15%

    ======================================================================
    Acq. Operator    : fiza3jan                        Seq. Line :  31
    Acq. Instrument : Instrument 1                      Location : Vial 31
    Injection Date  : 03/01/2012 20:41:49                    Inj :   1
                                                      Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 20/12/2011 09:21:16  by fiza20nov
    Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
    Last changed    : 05/01/2012 09:24:08  by fiza4jan
                      (modified after loading)
```




```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [pA*s]      [pA]       %
    ----|-------|----|-------|----------|----------|--------|
       1   4.652 BV    0.0481   18.20951    5.88600  0.00486
       2   4.763 VB S  0.0553 3.72509e5  9.60226e4  99.39355
       3   5.078 BB X  0.0554 2224.11499  626.96497  0.59344
       4  10.157 BB    0.0654   30.54182    7.06887  0.00815

    Totals :                  3.74782e5  9.66626e4


    ======================================================================
                               *** End of Report ***
```

GC FID ANALYSIS: 20% SAMPLE DILUTION FOR THE RATIO 1:3

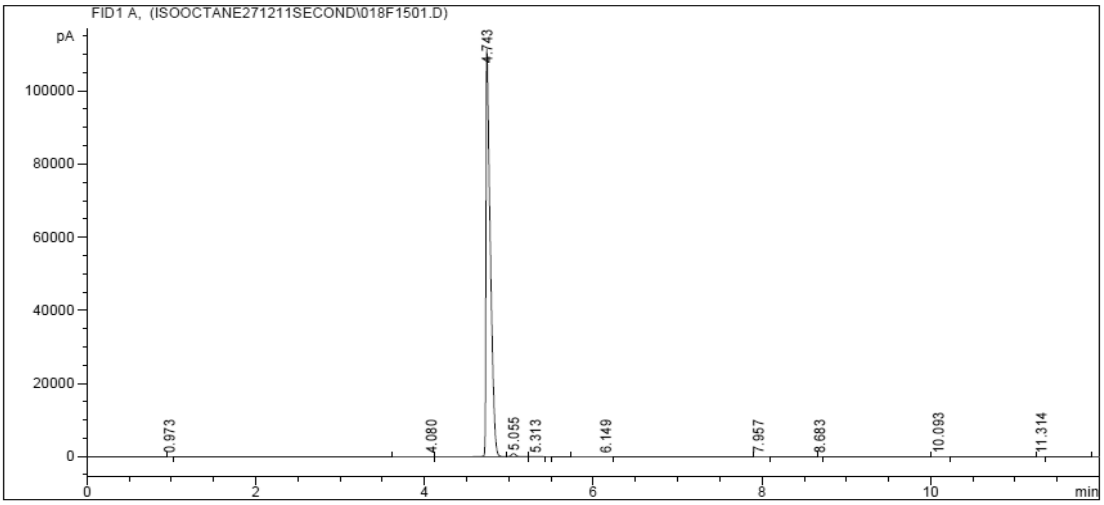
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\032F3201.D
 Sample Name: D1N3 20%

   ======================================================================
   Acq. Operator   : fiza3jan                       Seq. Line :  32
   Acq. Instrument : Instrument 1                   Location : Vial 32
   Injection Date  : 03/01/2012 21:04:23                 Inj :  1
                                                  Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ======================================================================
                            Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]        %
   ----|-------|----|-------|----------|----------|--------|
      1   4.658 BV    0.0494   22.96234    7.15339  0.00736
      2   4.770 VB S  0.0542 3.10185e5  8.19258e4  99.39255
      3   5.079 BB X  0.0540 1836.36707  535.61090  0.58843
      4  10.161 BB    0.0654   36.39363    8.42605  0.01166

   Totals :                  3.12081e5  8.24770e4

   ======================================================================
                            *** End of Report ***
```

GC FID ANALYSIS: 1% SAMPLE DILUTION FOR THE RATIO 1:4

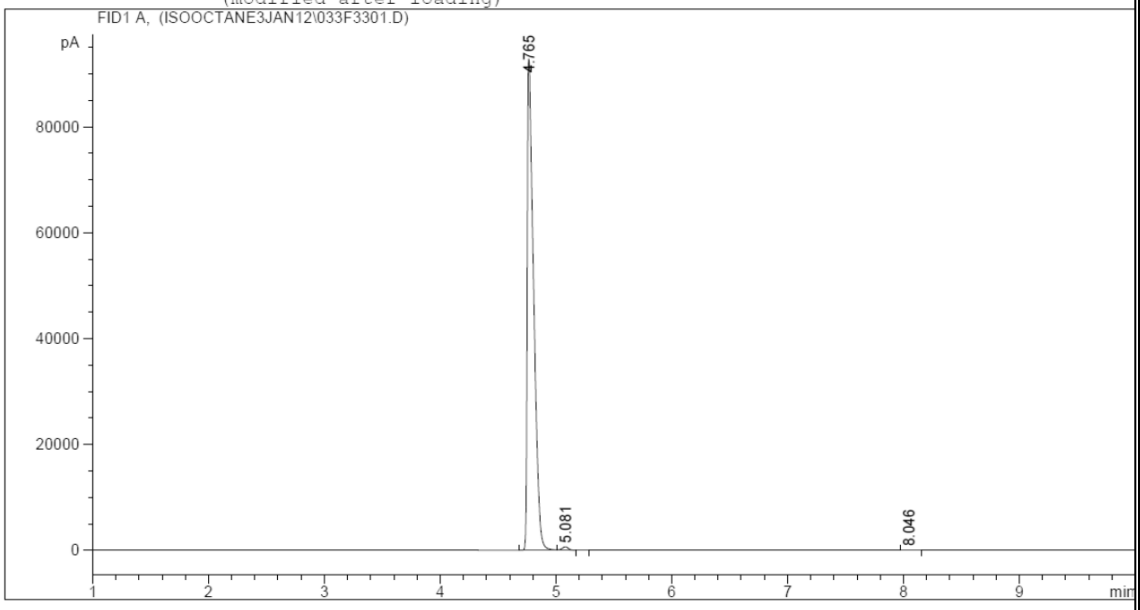
```
Data File C:\CHEM32\1\DATA\ISOOCTANE271211SECOND\018F1501.D
 Sample Name: D1N4sp2

    ======================================================================
    Acq. Operator   : wani27dec                      Seq. Line :  15
    Acq. Instrument : Instrument 1                    Location : Vial 18
    Injection Date  : 27/12/2011 17:03:00                 Inj :   1
                                                     Inj Volume : 1 µl
    Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
    Last changed    : 20/12/2011 09:21:16  by fiza20nov
    Analysis Method : C:\CHEM32\1\METHODS\B4ASHUTDOWN.M
    Last changed    : 28/12/2011 10:27:49  by wani27dec
                      (modified after loading)
    Method Info     : std testing
```




```
    ======================================================================
                               Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: FID1 A,

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [pA*s]      [pA]       %
    ----|-------|----|-------|----------|----------|--------|
       1   0.973 BV    0.0452    2.38132 6.74573e-1  0.00060
       2   4.080 BV    0.1247   10.18747 9.84432e-1  0.00255
       3   4.743 VB S  0.0517 3.95980e5  1.10739e5  99.29932
       4   5.055 BV X  0.0523 2741.67041  813.41901  0.68752
       5   5.313 VB X  0.0549   24.36732    6.95687  0.00611
       6   6.149 VB    0.0521    1.25823 3.57274e-1  0.00032
       7   7.957 BB    0.0437    3.91315    1.39444  0.00098
       8   8.683 BV    0.0358    2.35432 8.63105e-1  0.00059
       9  10.093 BB    0.0683 9.96176e-1 2.14131e-1  0.00025
      10  11.314 BV    0.0622    2.90134 5.81372e-1  0.00073
      11  12.168 BB    0.1842    2.76411 1.88345e-1  0.00069
      12  14.223 BV    0.0289    1.32383 7.63787e-1  0.00033

       Totals :                 3.98774e5  1.11566e5


                      *** End of Report ***
```

GC FID ANALYSIS: 5% SAMPLE DILUTION FOR THE RATIO 1:4

```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\033F3301.D
 Sample Name: D1N4 5%

   ========================================================================
   Acq. Operator   : fiza3jan                       Seq. Line :  33
   Acq. Instrument : Instrument 1                   Location : Vial 33
   Injection Date  : 03/01/2012 21:27:12                 Inj :  1
                                                  Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```

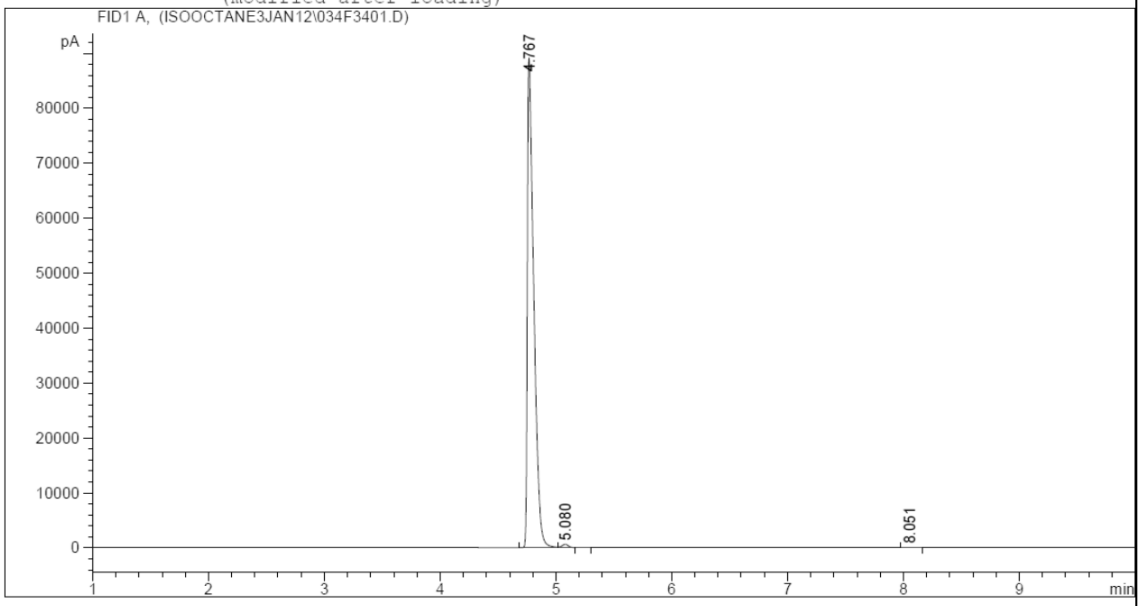



```
   ========================================================================
                            Area Percent Report
   ========================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]       %
   ----|-------|----|-------|----------|----------|--------|
      1   4.765 BB S  0.0536 3.54968e5  9.27780e4  99.37354
      2   5.081 BB T  0.0556 2225.91504  623.53308  0.62315
      3   8.046 BB    0.0634   11.81781    2.91015  0.00331

   Totals :                  3.57205e5  9.34044e4


   ========================================================================
                            *** End of Report ***
```

GC FID ANALYSIS: 10% SAMPLE DILUTION FOR THE RATIO 1:4

Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\034F3401.D
Sample Name: D1N4 10%

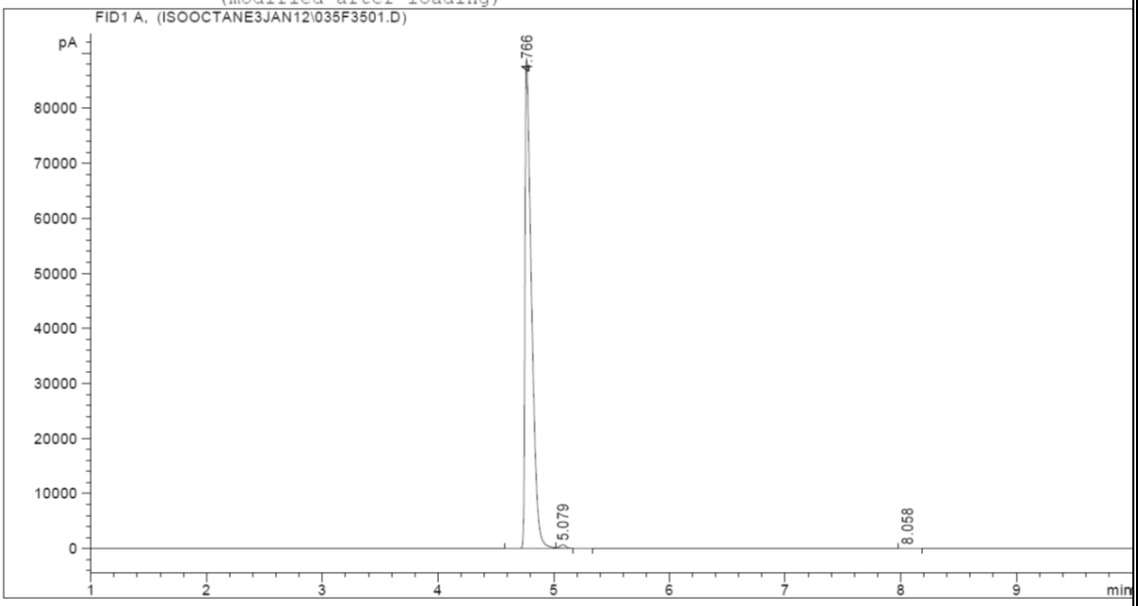
```
==========================================================================
Acq. Operator   : fiza3jan                        Seq. Line :  34
Acq. Instrument : Instrument 1                    Location : Vial 34
Injection Date  : 03/01/2012 21:49:51                  Inj :   1
                                                  Inj Volume : 1 µl
Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
Last changed    : 20/12/2011 09:21:16  by fiza20nov
Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
Last changed    : 05/01/2012 09:24:08  by fiza4jan
                  (modified after loading)
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: FID1 A,

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [pA*s]      [pA]        %
----|-------|----|-------|----------|----------|--------|
   1   4.767 BB S  0.0567 3.40420e5  8.88331e4  99.39049
   2   5.080 BB X  0.0551 2067.63501  587.59967  0.60368
   3   8.051 BB    0.0664   19.99839    4.53855  0.00584

Totals :                  3.42508e5  8.94253e4

==========================================================================
                          *** End of Report ***
```

GC FID ANALYSIS: 15% SAMPLE DILUTION FOR THE RATIO 1:4

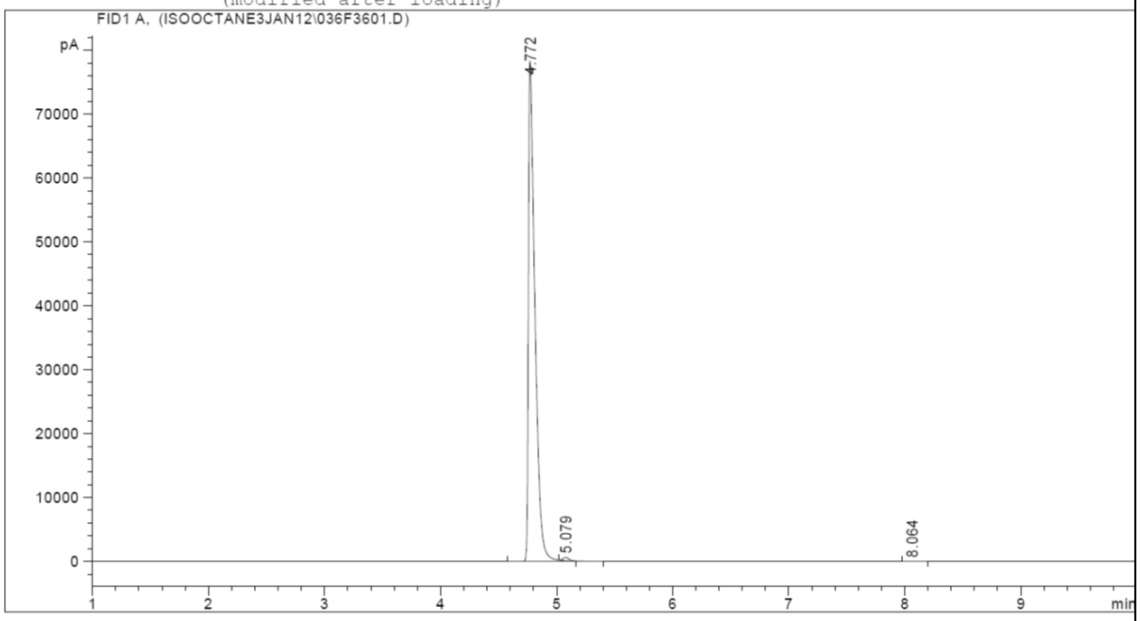
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\035F3501.D
 Sample Name: D1N4 15%

   ========================================================================
   Acq. Operator    : fiza3jan                        Seq. Line :  35
   Acq. Instrument : Instrument 1                     Location : Vial 35
   Injection Date  : 03/01/2012 22:12:23                   Inj :   1
                                                    Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ========================================================================
                              Area Percent Report
   ========================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]       %
   ----|-------|----|-------|----------|----------|--------|
      1   4.766 BB S  0.0588 3.49512e5  8.91276e4  99.40354
      2   5.079 BB X  0.0551 2063.07593  585.02948  0.58675
      3   8.058 BB    0.0706   34.13474    7.28966  0.00971

   Totals :                  3.51609e5  8.97199e4


   ========================================================================
                             *** End of Report ***
```

GC FID ANALYSIS: 20% SAMPLE DILUTION FOR THE RATIO 1:4

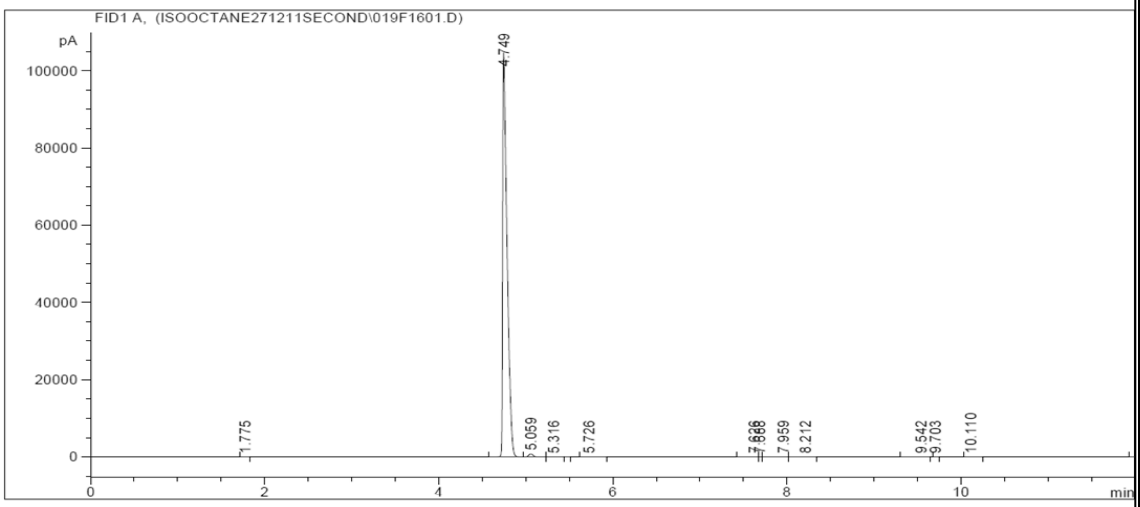
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\036F3601.D
 Sample Name: D1N4 20%

   ========================================================================
   Acq. Operator    : fiza3jan                        Seq. Line :  36
   Acq. Instrument : Instrument 1                      Location : Vial 36
   Injection Date  : 03/01/2012 22:34:56                    Inj :   1
                                                    Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ========================================================================
                             Area Percent Report
   ========================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]       [min]   [pA*s]      [pA]        %
   ----|-------|----|-------|---------|----------|--------|
      1   4.772 BB S  0.0555 3.04470e5  7.81451e4  99.41754
      2   5.079 BB X  0.0551 1740.96277  494.24863  0.56847
      3   8.064 BB    0.0749   42.84275    8.62643  0.01399

   Totals :                  3.06254e5  7.86480e4


   ========================================================================
                             *** End of Report ***
```

GC FID ANALYSIS: 1% SAMPLE DILUTION FOR THE RATIO 1:5

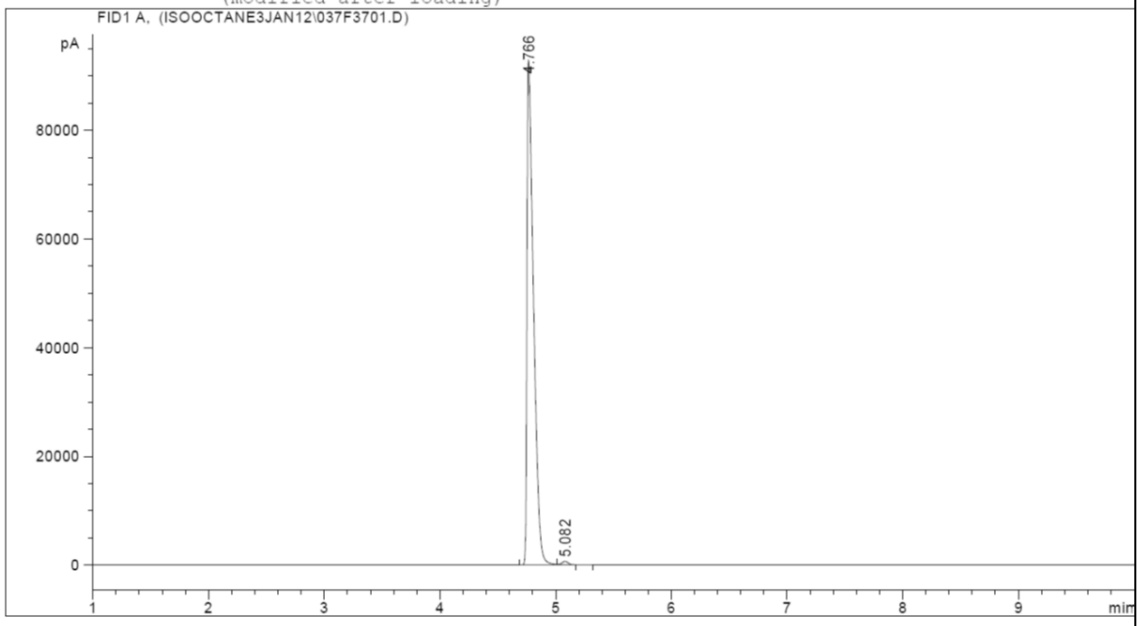
```
Data File C:\CHEM32\1\DATA\ISOOCTANE271211SECOND\019F1601.D
 Sample Name: D1N5sp1

   ======================================================================
   Acq. Operator   : wani27dec                      Seq. Line :  16
   Acq. Instrument : Instrument 1                   Location : Vial 19
   Injection Date  : 27/12/2011 17:25:28                 Inj :  1
                                                  Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\B4ASHUTDOWN.M
   Last changed    : 28/12/2011 10:27:49  by wani27dec
                     (modified after loading)
   Method Info     : std testing
```




```
   ======================================================================
                              Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]       %
   ----|-------|----|-------|----------|----------|--------|
      1   1.775 BV    0.0631    2.81453 5.55251e-1  0.00076
      2   4.749 VB S  0.0522 3.65915e5  1.03570e5  99.29378
      3   5.059 BV X  0.0517 2536.30005  763.68811  0.68824
      4   5.316 VB X  0.0543   20.89682    6.05499  0.00567
      5   5.726 BB    0.0897    2.12076 3.54974e-1  0.00058
      6   7.626 BV    0.1254    6.87029 7.26099e-1  0.00186
      7   7.688 VV    0.0307    2.01805 8.81539e-1  0.00055
      8   7.959 VV    0.2184    9.78061 5.52773e-1  0.00265
      9   8.212 VB    0.1739    4.35310 3.15714e-1  0.00118
     10   9.542 BB    0.1667    5.44978 4.13646e-1  0.00148
     11   9.703 BV    0.0295    1.99851 9.13699e-1  0.00054
     12  10.110 BB    0.0617    5.00693    1.25176  0.00136
     13  12.098 BV    0.0973    1.32361 1.78961e-1  0.00036
     14  14.811 BV    0.0461    2.58166 7.15854e-1  0.00070
     15  15.331 BB    0.0765    1.04275 2.11630e-1  0.00028

   Totals :                  3.68517e5  1.04347e5

              *** End of Report ***
```

GC FID ANALYSIS: 5% SAMPLE DILUTION FOR THE RATIO 1:5

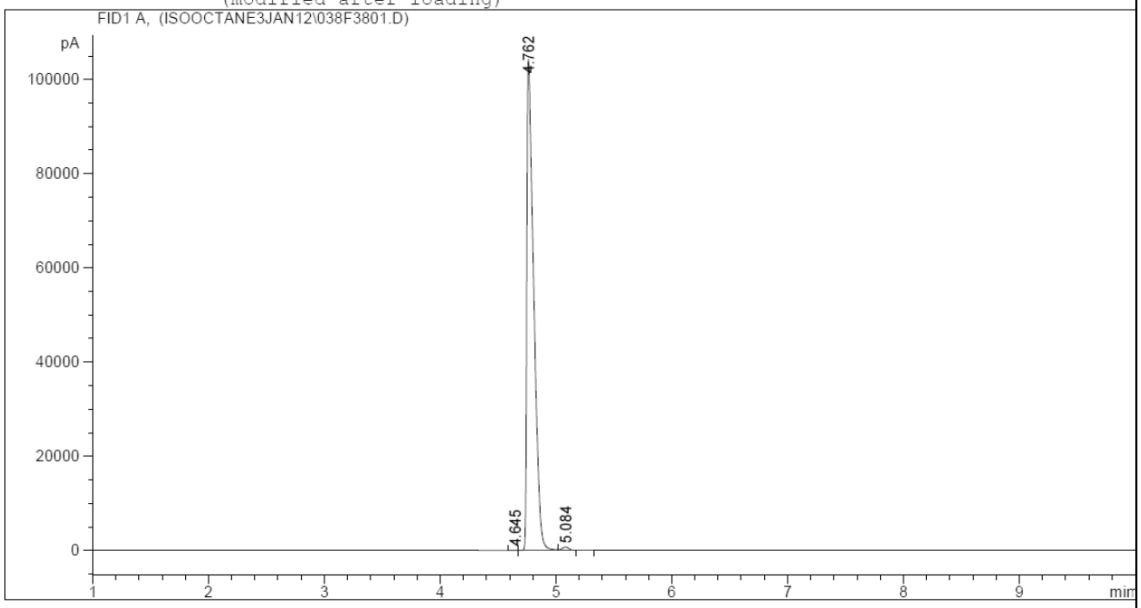
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\037F3701.D
 Sample Name: D1N5 5%

   ======================================================================
   Acq. Operator    : fiza3jan                      Seq. Line :  37
   Acq. Instrument : Instrument 1                   Location : Vial 37
   Injection Date  : 03/01/2012 22:57:37                 Inj :   1
                                                  Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ======================================================================
                            Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]       %
   ----|-------|----|-------|----------|----------|--------|
      1   4.766 BB S  0.0577 3.64730e5  9.30856e4  99.39344
      2   5.082 BB X  0.0574 2225.80811  612.07080  0.60656

   Totals :                  3.66956e5  9.36977e4


   ======================================================================
                            *** End of Report ***
```

GC FID ANALYSIS: 10% SAMPLE DILUTION FOR THE RATIO 1:5

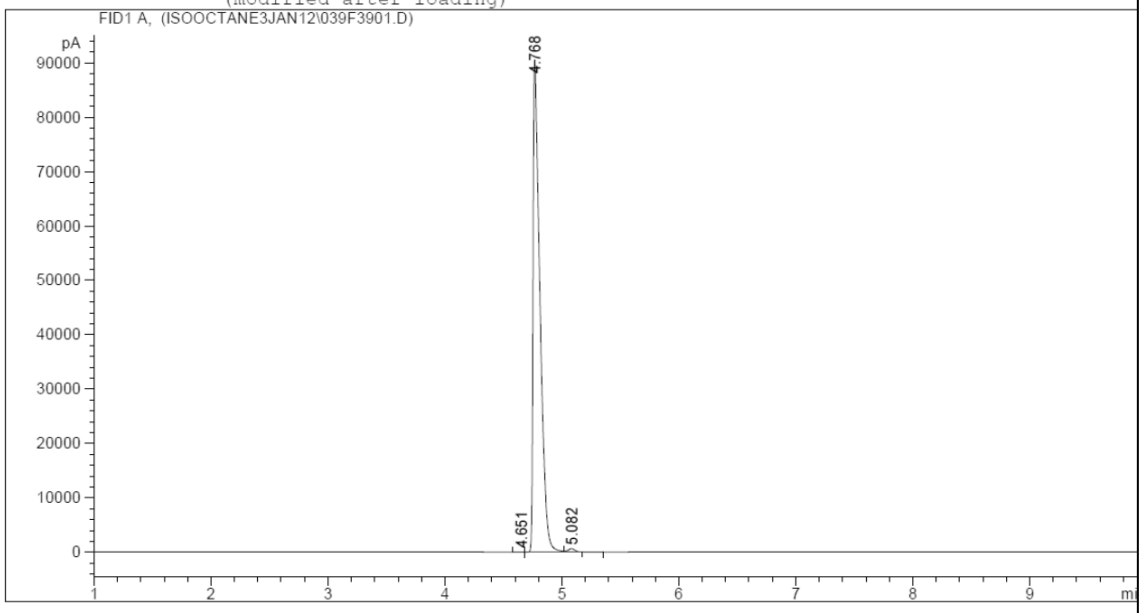
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\038F3801.D
 Sample Name: D1N5 10%

   ========================================================================
   Acq. Operator   : fiza3jan                       Seq. Line :  38
   Acq. Instrument : Instrument 1                   Location : Vial 38
   Injection Date  : 03/01/2012 23:20:10                 Inj :   1
                                                  Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ========================================================================
                            Area Percent Report
   ========================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]       [min]   [pA*s]      [pA]        %
   ----|-------|----|-------|----------|----------|--------|
      1   4.645 BV    0.0452   10.92875    3.72491  0.00254
      2   4.762 VB S  0.0600 4.28328e5  1.04113e5  99.38606
      3   5.084 BB X  0.0605 2611.36597  684.80133  0.60592
      4  10.172 BB    0.0665   23.62725    5.45714  0.00548

   Totals :                  4.30974e5  1.04807e5


   ========================================================================
                            *** End of Report ***
```

GC FID ANALYSIS: 15% SAMPLE DILUTION FOR THE RATIO 1:5

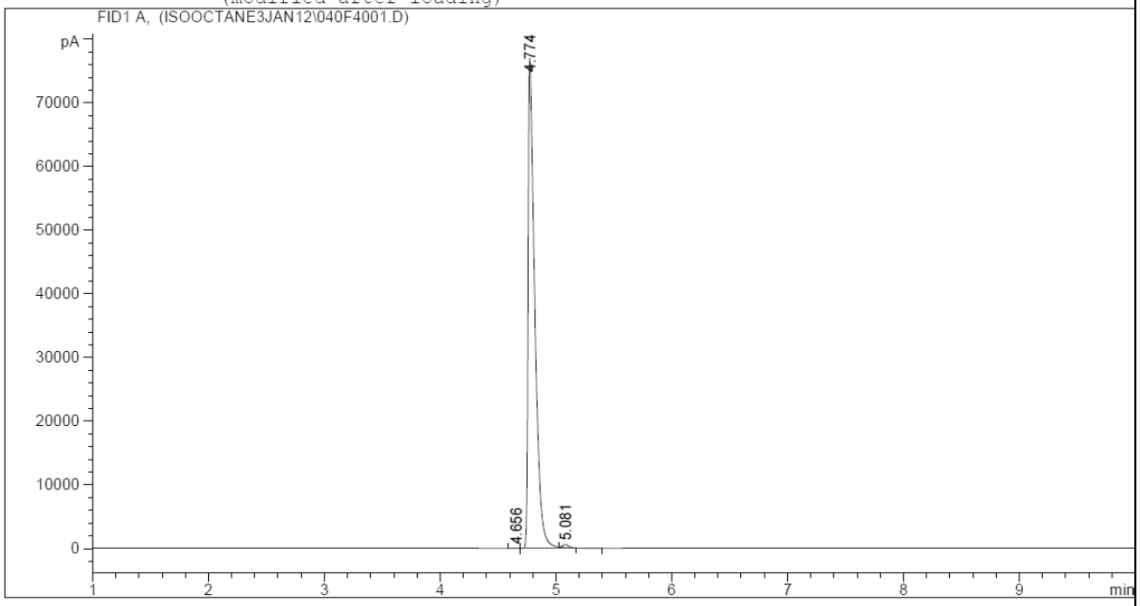
```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\039F3901.D
 Sample Name: D1N5 15%

   ======================================================================
   Acq. Operator   : fiza3jan                       Seq. Line :  39
   Acq. Instrument : Instrument 1                   Location : Vial 39
   Injection Date  : 03/01/2012 23:42:44                 Inj :   1
                                                  Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ======================================================================
                              Area Percent Report
   ======================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]        [min]   [pA*s]      [pA]        %
   ----|-------|----|-------|----------|----------|--------|
      1   4.651 BV    0.0496   19.29923    6.15546  0.00524
      2   4.768 VB S  0.0573 3.66342e5  9.03447e4  99.39905
      3   5.082 BB X  0.0580 2158.63354  585.47186  0.58570
      4  10.174 BB    0.0725   36.89540    8.04326  0.01001

   Totals :                  3.68556e5  9.09443e4


   ======================================================================
                              *** End of Report ***
```

GC FID ANALYSIS: 20% SAMPLE DILUTION FOR THE RATIO 1:5

```
Data File C:\CHEM32\1\DATA\ISOOCTANE3JAN12\040F4001.D
 Sample Name: D1N5 20%

   ========================================================================
   Acq. Operator   : fiza3jan                        Seq. Line :  40
   Acq. Instrument : Instrument 1                    Location : Vial 40
   Injection Date  : 04/01/2012 00:05:25                  Inj :  1
                                                  Inj Volume : 1 µl
   Acq. Method     : C:\CHEM32\1\METHODS\ISOOCTANE230108.M
   Last changed    : 20/12/2011 09:21:16  by fiza20nov
   Analysis Method : C:\CHEM32\1\METHODS\LEMON GRASS.M
   Last changed    : 05/01/2012 09:24:08  by fiza4jan
                     (modified after loading)
```




```
   ========================================================================
                            Area Percent Report
   ========================================================================

   Sorted By             :      Signal
   Multiplier            :      1.0000
   Dilution              :      1.0000
   Use Multiplier & Dilution Factor with ISTDs


   Signal 1: FID1 A,

   Peak RetTime Type  Width     Area      Height     Area
     #   [min]       [min]   [pA*s]      [pA]        %
   ----|-------|----|-------|----------|----------|--------|
      1   4.656 BV    0.0508   24.43244    7.35262  0.00785
      2   4.774 VB S  0.0590 3.09429e5  7.67755e4  99.42389
      3   5.081 BB X  0.0559 1721.96265  479.54697  0.55329
      4  10.176 BB    0.0722   46.57348    9.84657  0.01496

   Totals :                  3.11222e5  7.72722e4


   ========================================================================
                            *** End of Report ***
```

Appendix B

Material Safety Data Sheet: Acetone

Material Safety Data Sheet: Hexane

Material Safety Data Sheet: Isooctane

LIST OF MATERIAL SAFETY DATA SHEETS (MSDS)

B-1 ISOOCTANE MSDS

ISOOCTANE			
IUPAC name	2,2,4-Trimethylpentane		
Other names	isobutyltrimethylpentane		
PHYSICAL & CHEMICAL PROPERTIES			
Molecular formula:	$(CH_3)_3CCH_2CH(CH_3)_2$ C_8H_{18}	**Std enthalpy of formation** $\Delta_f H^{\ominus}_{298}$;	−259 kJ/mol
Molar mass:	114.22 g/mol	**Std enthalpy of combustion** $\Delta_c H^{\ominus}_{298}$;	−5461 kJ/mol
Appearance:	colorless liquid	**Standard molar entropy** $S^{\ominus}_{298}$:	328 $J \cdot K^{-1} \cdot mol^{-1}$
Density:	0.688 g/ml, liquid	**Auto ignition Temperature:**	417 °C
Melting point:	−107.38 °C (165.77K) 1.	**Vapor Pressure:**	41 mm Hg at 21 C
Boiling point:	99.3 °C (372.4 K)	**Vapor Density :**	3.9°C
Solubility in water:	Immiscible	**Explosion limits :**	1 - 6%

HANDLING AND STORAGE	
Precaution	i. (Always wear recommended personal protective equipment.) ii. Flammable liquid and vapors. Keep container closed. iii. Do not breathe vapors. Avoid contact with skin, eyes and mucous membranes. Keep away from heat, sparks and flame. iv. Electrically bond and ground all handling equipment. Protective neoprene or rubber gloves and apron are recommended.
Storage	i. Store in an area designed for storage of flammable liquids. (OSHA 29 CFR 1910.106) ii. Protect from temperature extremes and sunlight, and store away from incompatible substances and in accordance with 29 CFR 1910.106. iii. Avoid acids, bases, oxidizers, explosives, nitrogen-fluorine compounds, sulfites, perchlorates, reducing agents and plastics. iv. Flammable liquid and vapor. Once liquid solvent has been completely dispensed, containers which appear "empty" should be handled in the same manner as when they were "full" of liquid solvent.
FIRST AID MEASURES	
Eye Contact:	Rinse with plenty of water for at least 15 minutes. Get emergency medical assistance.
Skin Contact:	Rinse affected area with plenty of water until no evidence of chemical remains.
Inhalation:	Immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse, administer cardiopulmonary resuscitation (CPR). Contact physician immediately.
Ingestion:	Contact physician immediately. Aspiration hazard – do not induce vomiting.

B-2 HEXANE MSDS

HEXANE			
IUPAC name	Hexane, 2-methylpentane, 3-methylpentane, 2,2-dimethylbutane, 2,3-dimethylbutane		
Other names	n-hexane, normal hexane, Hexyl hydride		
PHYSICAL & CHEMICAL PROPERTIES			
Molecular formula:	C_6H_{14}	**Percent volatile**	100
Molar mass:	86 g/mol	**Appearance:**	Clear, colorless liquid & Light odor
Specific Gravity :	0.659	**Auto ignition Temperature:**	225°C (437°F)
Melting point:	-95 C	**Vapor Pressure:**	132 mm Hg at 20 C
Boiling point:	69 C	**Vapor Density :**	3 (air = 1)
Solubility in water:	Insoluble in water.	**Explosion limits :**	1.2% - 7.7%
Flash Point:	-10 F	**Hazard specification :**	i. Stable ii. Highly flammable iii. Irritant iv. Harmful by inhalation, ingestion or skin absorption
Molecular formula:	C_6H_{14}	**Percent volatile**	100

HANDLING AND STORAGE	
Precaution	Keep locked up. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Avoid contact with skin. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Keep away from incompatibles such as oxidizing agents.
Storage	Store in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame).

FIRST AID MEASURES	
Eye Contact:	Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.
Skin Contact:	Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists.
Inhalation:	Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:	Aspiration hazard. If swallowed, DO NOT INDUCES VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. Get medical attention immediately.

B-3 ACETONE MSDS

ACETONE			
IUPAC name	Acetone, Propan-2-one, 2-Propanone		
Other names	2-propanone,Dimethyl Ketone, Dimethylformaldehyde,Pyroacetic Acid		
PHYSICAL & CHEMICAL PROPERTIES			
Molecular formula:	C_3H_6O	**Percent volatile**	100
Molar mass:	58.08 g/mol	**Appearance:**	Clear, colorless liquid & Light odor
Specific Gravity :	0.786	**Auto ignition Temperature:**	465°C
Melting point:	-95.35°C	**Vapor Pressure:**	184 mm Hg @ 20° C
Boiling point:	56.2°C	**Vapor Density :**	2 (Air = 1)
Solubility in water:	Soluble in water	**Explosion limits :**	2.5% - 12.8%
Flash Point:	-20°C	**Hazard specification :**	i. Stable ii. Highly flammable iii. Irritant iv. Harmful by inhalation, ingestion or skin absorption
Molecular formula:	C_3H_6O	**Percent volatile**	100%

HANDLING AND STORAGE	
Precaution	Keep locked up. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Avoid contact with skin. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label.
Storage	Store in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame).
FIRST AID MEASURES	
Eye Contact:	Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.
Skin Contact:	Remove any contaminated clothing. Wipe off excess from skin. Wash skin with soap and water for at least 15 minutes. Get medical attention if irritation develops or persists.
Inhalation:	Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.
Ingestion:	Potential for aspiration if swallowed. Get medical aid immediately. Do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. If vomiting occurs naturally, have victim lean forward.