

**UNIVERSITI TEKNOLOGI MARA**

**CHARACTERIZATION OF AGARWOOD DISTIL  
WATER BASED ON TIME INTERVAL**

**NURUL NADIA BINTI ABDULLAH**

**B.Eng (Hons) Chemical and Bioprocess**

**July 2018**

**UNIVERSITI TEKNOLOGI MARA**

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WATER BASED ON TIME INTERVAL**

**NURUL NADIA BINTI ABDULLAH**

Thesis submitted in fulfillment  
of the requirements for the degree of  
**Bachelor of Engineering**  
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**Universiti Teknologi Mara  
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## ABSTRACT

Agarwood distil water or known as hydrosol is obtained by using hydrodistillation method. Agarwood distil water is widely used in medicine since it contains high anti-oxidant and antibacterial properties that will improve the human health. The aim of this study is to analyze the characterization of Agarwood distil water from *Aquilaria malaccensis* at various time by using Fourier Transforms Infrared (FTIR) spectroscopy, Gas Chromatography-Mass Spectrometer (GC-MS) and also by using Inductive Couple Plasma (ICP). The functional groups has been successful identified which there were presence of same functional groups for each hydrosols samples at different production temperature such as alcohol group, alkene group, alkyne group and also aromatic ring alkane group. It showed that the different production time interval did not affect the functional groups. Then, there were about 48 chemical compounds of hydrosol have been successful identified and the different chemical compounds were found for the different production temperature or at various time. In this work, 1,2,4,5-Tetrazine, 1,4-diethylhexahydro was the main compound found in hydrosol. Lastly, all the samples of these hydrosols did not contain any metals and safe to be used as medical treatment. In conclusion, the hydrosol from hydrodistillation process contains 1,2,4,5-Tetrazine, 1,4-diethylhexahydro which is potentially used is the treatment of diarrhoea and worm infections, and also used to treat the involuntary movements (chorea) of Huntington's disease or progressive brain disorder that causes uncontrolled movements, emotional problems, and loss of thinking ability.

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## LIST OF ABBREVIATIONS

### Abbreviations

GC-MS	Gas Chromatography-Mass Spectrometer
FTIR	Fourier Transforms Infrared
ICP	Inductive Couple Plasma
UiTM	Universiti Teknologi MARA
SFE	Supercritical Fluid Carbon Dioxide Extraction
APT	Agarwood-Producing Trees
BMW	Black Magic Wood
EI	Electron Impact Ionization
GC	Gas Chromatograph
MS	Mass Spectrometer
DO	Dissolve Oxygen

# CHAPTER ONE

## INTRODUCTION

### 1.1 Background Study

Agarwood or commonly known as gaharu, aloeswood, jinkoh, oud, agallocha and eaglewood is found in *Aquilaria* species which comes from family of *Thymelaeaceae*. *Aquilaria* is basically a woody plant which is inherent in Southeast Asia (Tetsuro Ito *et al.*, 2012). Agarwood has about 15 species and there are four species are found in Thailand which their specific names are *Aquilaria Crassna*, *Aquilaria Baillonil*, *Aquilaria Subintegra* and *Aquilaria Malaccens* (Penpun Wetwitayaklung *et al.*, 2009). The species of *Aquilaria Crassna* is usually used in order to extract the oil because it contained excellence quality Agarwood oil. Table below shows the classification of Agarwood:

Table 1.1: Classification of Agarwood (Khalil *et al.*, 2013)

<b>Kingdom</b>	<b>Plantae</b>
Division	Magnoliophyta
Class	Magnoliopsida
Order	Malvales
Family	Thymelaecca
Genus	<i>Aquilaria</i>

Agarwood is actually a resinous heartwood substance that comes from the tree as a non-specific host response to insects, wounding or bacterial infections (Rozi Mohamed *et al.*, 2014). The heartwood is basically in brown or black color and fragrant. After infection of Agarwood tree with parasitic mold, the aromatic resin will start to produce. The resin contained the tree extractives that has good aroma that comes from the compound namely sesquiterpene. The major sesquiterpenes compounds that contained in

the essential oil are including 11-dien-9one, gamma-selinene, selina-3, 11-dien-14-al and derivate chromonomes (Yoswathana *et al.*, 2013).

The main organs of the Agarwood tree are the stem and branches because those organs are the doposition of 'gaharu' occur (Mohamed.R *et al.*, 2013). Then, there is also the existence of included phloem by the cambium and the concentrated resin is contained in the included phloem. The included phloem is characterized by the phloem tissue's layers that embedded in the xylem. Generally, Agarwood has a few grades namely, A, B, C and D that is classified according to its physical properties, formation and unique scent.

Nowadays, Agarwood is very highly valuable fragrant woods because it contained economically essential oil and hydrosols with its aromatic unique product (Nuttawan Yoswathana *et al.*, 2013). It is widely used in many industries including medicine, cosmetics, perfume and aromatherapy industries. However, it is also has been used for the religious ritual. For the medicine area in Malaysia, the Agarwood was used by mixing the Agarwood with the coconut oil to use it as a liniment. In addition, the function of Gaharu is also to treat the smallpox, diarrhea, abdominal cramp, elderly fatigue, heart palpitations, as carminative, as a stimulant, for the pregnancy and also for the female that has disease of genital organs.

There are three forms of Agarwood that have sold which are in form of pieces of heartwood, heartwood oil and heartwood powder. The heartwood is burned to produce the aromatic vapor in houses and also in shrines. The powder form of wood is used as incense and medicine. The Gaharu oil and resinous are very expensive because the production yield during extraction are low. Furthermore, the formation of resinous wood is very rare to occur in young and wild trees. Basically, the good quality Gaharu's price can achieve to RM10,000 per kg based on the resinous wood's grade.

Besides, there are a lot of extraction methods that can be applied to produce essential oils. The common methods that have been used to extract the oils from the plants are including hydrodistillation, supercritical fluid carbon dioxide extraction (SFE), steam distillation and solvent extraction. The classical method that used to extract the Agarwood oil is by using hydrodistillation method. This method takes about 7-10 days



and is guarantee safe to operate. Then, supercritical fluid carbon dioxide extraction (SFE) method is better than hydrodistillation method as it has high diffusivity, low viscosity, non-toxic, nonflammable, consume less energy, good transport properties and extraction, chemically stable and produce high yields.

In a nutshell, Gaharu distil water is an unique product and is widely used as tonic drink, for external skin, act as anti-depressant, remove anxiety and easing insomnia. It also has many benefits especially to improve human health. The taste of Gaharu distil water is quite similar to normal distilled water because it comes from the condensation process of boiled Gaharu. The hydrosol is quite expensive due to the high demand but low production yield of hydrosol. The different extraction methods of Gaharu distil water have their advantages and disadvantages themselves.

## **1.1 Objectives**

The objectives of this study is to analyze the characterization of Agarwood distil water from *Aquilaria malaccensis* at different time interval by using Fourier Transforms Infrared (FTIR) spectroscopy and Gas Chromatography-Mass Spectrometer (GC-MS). Next, another objective of this research is to make a test on the water quality of Agarwood distil water or hydrosols which is heavy metal test by using Inductive Couple Plasma (ICP) in order to obtain the high purity of hydrosols.

## **1.2 Problem Statement**

Basically, the producing of Gaharu is from the tree producing the resin that contained the infection from spreading and wound covering. Agarwood is tropical tree that allocates in rain forest southeast which is Laos, Cambodia, Thailand and Vietnam. The main uses of Gaharu are for medicine, perfumes and incense. Year by year, the technology is developed so that there are more equipment and process were found for the extraction of Agarwood distil water from Gaharu. However, that technology still has problem in the extraction of Agarwood distil water from Gaharu and leads to the low production yield of Agarwood distil water. In order to achieve high yield Agarwood distil water with low time extraction, there are many equipment that have been developed. This research has conducted the extraction to obtain Agarwood distil water by using

hydrodistillation method which is classical method because this method is believed can produce high amount of Gaharu distil water with safe operation and in shorter time.

Then, there are several grade of Agarwood which are grade A, B, C and D that have been classified based on their physical properties. The physical properties are always changing from time to time and different from one country to the other country. This condition will cause the grade of the wood cannot be divided into right grade. The grade of the wood supposedly divided according to the chemical properties to give the accurate results. So in this research, gas chromatography-mass spectrometry (GCMS) and Fourier Transform Infrared (FTIR) were used to obtain the chemical properties of hydrosols.

Besides that, some of the people are not preferred to use this hydrosol because they said this hydrosol was contained mold contamination and heavy metal. This will cause the people in suffer from diarrhea, cancer, and many other diseases. This condition is so dangerous because it involved human's lives. So in this study, the hydrosols that obtained from hydrodistillation process was undergo water quality test. The test was done by using Inductively Couple Plasma (ICP) system.

### **1.3 Scope of Research**

In this research, the extraction time of Agarwood distil water is the parameter that can be manipulated in studying the chemical profiling of Agarwood distil water and the quality of Agarwood distil water. However, there are limited values for the parameter. During extraction of Agarwood distil water, the time interval plays important role. The longer the time of extraction process, the higher the temperature of Agarwood distil water. The hydrodistillation process cannot be exceeding the temperature of 120<sup>0</sup>C to avoid the plant material from burning (Jutarut *et al.*, 2011). The various time that have been conducted during extraction are 12 hours, 18 hours, 24 hours, 30 hours, 36 hours, 42 hours and 48 hours.

## CHAPTER TWO

### LITERATURE REVIEW

#### 2.1 Gaharu

Basically, Gaharu or known as Agarwood is derived from its very important source that is *Aquilaria* species which is wounded trees and it is known as aromatic resin-infused wood (Yangyang Liu *et al.*, 2013). *Aquilaria crassna* which is an angiosperm within the *Thymelaeaceae* family is an evergreen tree. In the genus *Aquilaria*, it contained fifteen species which are eight of them can be produce agarwood (Moitreyee Saikia *et al.*, 2012). The ‘Agarwood’ term is actually referring to the resinous heartwood that can turn to aromatic. Agarwood usually consist of therapeutic and tonic properties (N.Ismail *et al.*, 2016).

Then, there are a lot of different names of Agarwood in this world which are according to the content of oleoresin such as eaglewood, aloeswood, calambac, garoo wood and oud. In Malaysia, the agarwood tree is usually called as “karas” (Nor Atikah *et al.*, 2015). The numerous Agarwood different names are depending on the country language and also the philosophy which it is located. Agarwood usually grows at 700-1400 m of altitude. The figure below shows the whole plant of Agarwood, and the cutting of stem of one week, 6 months and 20 months old Agarwood.



Figure 2.1: (A) Agarwood's Whole Plant, (B) One Week Agarwood, (C) 6 Months Agarwood, (D) 20 months Agarwood (Janey Alam *et al.*, 2015)

In Asia, the agarwood-producing trees (APT) species with high diversity was found in Indonesia compared to other countries (Hidayat *et al.*, 2017). Gaharu is not tapped like other products of resin, but it is accumulated inside the tree and the wood tissue was impregnated in order to produce aromatic nodules known as Gaharu wood (Gary *et al.*, 2001). It is not tapped like the extraction of sap and latexes from numerous tree species in the tropical forests because the tree was not secreted the Gaharu after making the incision (Obidzinski *et al.*, 1997).

Due to longer time of incubation, the quality of inoculated resin is high and it has a purer fragrance. As a result, *Aquilaria* trees are usually has been cut down broadly in order to find the agarwood that contained resins (Boon *et al.*, 2016). The *Aquilaria* wood that can be classified as healthy is those that have soft, white and without smelly resins. The formation of Agarwood is happened when it is affected by exterior factors including the graze of animal, the lightning strike or the attack of insect.

The harvesting of Agarwood normally will have one cycle after planting of 7 years. The resin of agarwood will be produced by only 60% of trees at 1000 ha. There are about 833 trees will be plant in one hectare hence about 500 trees are assumed to be produce agarwood resin after seven years. The formation of Agarwood normally happened slowly and irregularly in old trees. This condition cause the market demand is higher than Agarwood's supply from wild sources. Furthermore, it is assumed that about 1000 gram of Agarwood will be produced for each tree hence about 500 kg of Agarwood resin will be produced in one hectare. Besides that, the leaves of Agarwood is actually have the probable for antidiabetic by action mechanism as inhibitor of alpha-glucosidase and also can increase the resistance of liver insulin by activation of AMP-activated protein kinase (AMK) (Fauzia *et al.*, 2016).

Nowadays, there are many researchers and scientists that are doing the research and development about *Aquilaria* trees. In Malaysia, Gaharu becomes popular because it is very useful and has a lot of benefits to improve human health. There is also the Research Institutions that conducting the research about this Gaharu. For an example, the Forest research Centre has started the project's collaborative to induce the Agarwood's formation by inoculating the tree of Gaharu.

## 2.2 Type of Extraction Method

First of all, there are many types of extraction method in order to obtain the hydrosol from Agarwood. This extraction method will produce both Agarwood distilled water and also Agarwood essential oil. The various methods of extraction are including water distillation (hydrodistillation), solvent extraction, supercritical fluid extraction (SFE), steam distillation, cold pressing and spinning band distillation. Every method has their specific advantages and disadvantages. For an example, there is disadvantage of solvent extraction which is usually toxic, flammable and required high cost.

In this work, the extraction method that is applied is hydrodistillation method which is classical method because it is safe to operate compared to other method and also environmentally friendly. In addition, hydrodistillation method is the famous extraction process among the local seller (Nor Atikah *et al.*, 2015). Another advantage of this method is the volatile elements will be condensing into water and it is much cheaper compared to other extraction method. Basically, the time taken for hydrodistillation method is about 7-10 days. Figure below shows the apparatus of extractor that usually used in any industrial that produce hydrosol and agarwood oil.

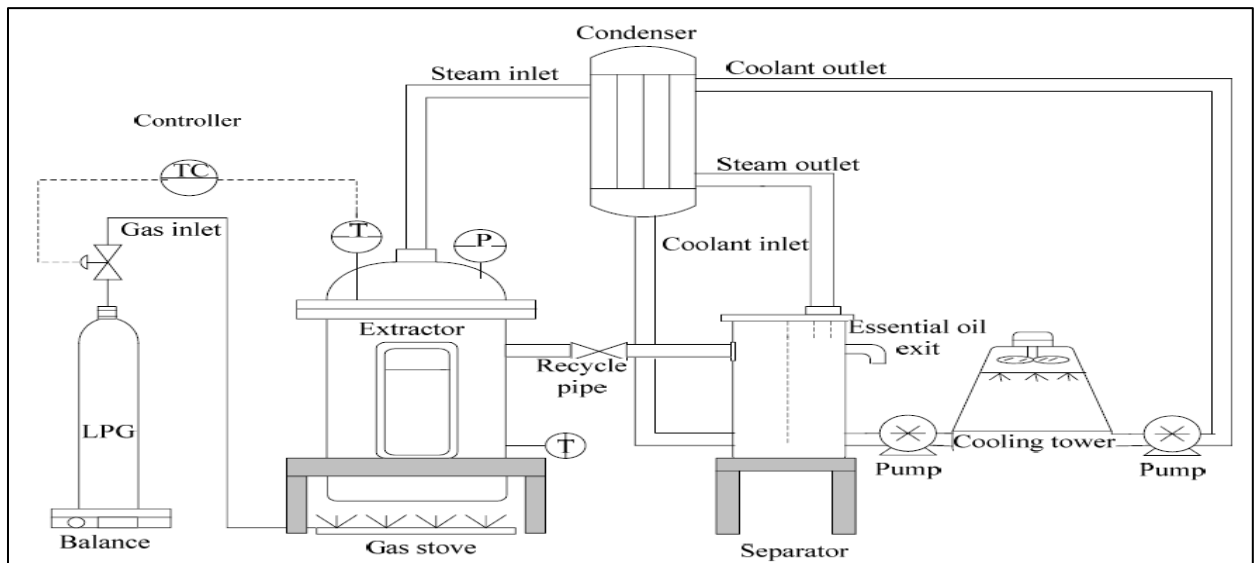


Figure 2.2: The Extractor Facilities (Jutarut *et.al*, 2011)

According to the previous figure, Jutarut *et.al* reported that the unit of distillation comprises of four main components which are a stainless steel extractor that has 0.4 m of

inner diameter and 0.5 m of height, a stainless steel condenser that has 0.4m of inner diameter and 1.2 m of height, a stainless steel separator that has 0.3 m of inner diameter and 1.2 m of height, and lastly is a cooling tower that has motor of 1.5kW. The gas stove is used in order to heat the extractor that is connected with the controller to control the usage of gas and temperature of extraction. The temperature of extraction process cannot be exceeded 120<sup>0</sup> because it can burn the material of plant.

By using hydrodistillation process, the material of plant which is Gaharu will be immersed in the water which is in heated still. This process normally conducted under atmospheric pressure and a reduced pressure. The ratio of solid-to-water is usually 1:50 g/mL is applied during this process. Then, the steam of water and essential oil will be produce and will leave the hot suspension. It will then condense, collected and is separated by using decantation. It will obtain two products which are essential oil and the hydrosol or floral water. This hydrosol is actually contained the constituents of the essential oil.

During hydrodistillation process, the temperature will increase and lead to the increasing of pressure inside the organ's plant that consist of essential oil (Svetomir *et al.*, 2013). When the pressure is rise above certain level, it will break the cell wall and then will release the essential oil. During distillation, component of essential oil and steam are in intimate interaction (Akwasi *et al.*, 2015).

### **2.3 Agarwood Distilled Water / Hydrosol**

Generally, Agarwood distilled water is commonly known as hydrosol, floral waters or hydroflorates. During aromatic plants distillation, the distillation water with dissolved components of essential oil is called as hydrosol (Rao *et al.*, 2012). Hydrosol is actually quite similar to essential oil but hydrosol has less concentration compared to the essential oil. The word 'hydro' gives the meaning of water meanwhile the word of 'sol' gives the meaning of solution. Hence, the word hydrosol can be defined as the watery solution that obtained during process of distillation that comprises of both the distilled-water plant components and essential oil.

Hydrosol is actually the by-product of the production of essential oil and it consists of plant essence in every drop. It is usually made by distilling the material of plant which allow the breakdown of the cell walls and then will produce the oil of the plant. The process of cooling was done to the steam and oil and it causes the steam becomes water and the oil to move at the top. This water is known as hydrosols and the oil is known as essential oil. In this study, the Gaharu wood was undergo the extraction process which is hydrodistillation in order to obtain the gaharu distilled water or hydrosol and Gaharu essential oil. Gaharu distilled water usually has soft aroma compared to essential oil of Gaharu. However, the scent is almost same with the Gaharu oil. Hydrosol consists of several carbohydrate and complex sugars in the solution.

There are many human said that the hydrosols are not really recommended because they contained mold contamination or bacterial. However, when the process of distillation and the process packaging of hydrosol are conducted carefully, there will be no any spoilage occur easily. For example, the hydrosols must be placed in the aseptic container with seal and then kept in a low temperature place to overcome the contamination. Every hydrosol obtained will be different because it depends on the technique of distillation used. Basically, the high quality of hydrosol is obtained from the initial part of distillation process instead of the final part of distillation process. Furthermore, the high quality hydrosols are those that not having any visible droplets of oil. The pH of hydrosol usually low which is about 4.5 to 5.5 and it produce pleasantly aroma with the smell bright and normally looks like water which is clear. The pH value

gives the effects to the therapeutic of the hydrosol. There are some inhibitory effects on various bacteria that have by hydrosol.

## 2.4 Essential Oil

Essential oil which is usually known as fly oil or volatile oil is actually a fluid compound that can be derived from some parts of the plant such as stems, fruits, leaves, seeds, flowers or roots by technique of distillation (Mahfud *et al.*, 2017). They are contained in secretory cells, canals, cavities, glandular trichomes or epidermic cells. There are around 3000 essential oils are presented. However, only 10% of the essential oil that presented is significant to be commercial. The percent consumption of essential oil is about 8-10% in every year.

The essential oil can be produced from various plant species which is about 150-200 species such as family of *Lauraceae*, *Labiatae*, *Pinaceae*, *Compositae*, *Umbelliferaceae* and *Myrtaceae*. The main producer country of the essential oil in the world is Indonesia which is having 45 species of plant that can produce essential oil. However, only 15 plants have been exported including Agarwood oil, ginger oil, cajeput oil, blackpepper oil, kaffir lime oil, massoil oil, turpentine oil, cananga oil, patchouli oil, vetiver oil, sandalwood oil, citronella oil, bud oil, cullilawan oil and sassafras oil. Then, about 90 types of essential oil are traded in international market.

Essential oils are actually consists of the isomers that have complex mixtures like sesquiterpenes, monoterpenes, aliphatic compounds and aromatic compounds. Generally, the pleasant odor and aroma of agarwood oil will cause the chemical components to present. Basically, any pure essential oils can be classified into two different chemical components groups which are hydrocarbons and the oxygenated compounds. For the hydrocarbons, they are made up of terpenes which are monoterpenes, sesquiterpenes and diterpenes. Terpenes functions as to prevent the toxins accumulation and help to release the existing toxins from the kidneys and liver. Meanwhile for the oxygenated compounds, they are consisting of esters, ethers, acids, ketones, lactones, acetals, aldehydes, phenols, alcohols and oxides. These hydrocarbons and oxygenated compounds are affected the characteristics of odor and aroma of the oils.



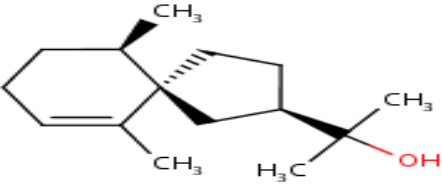
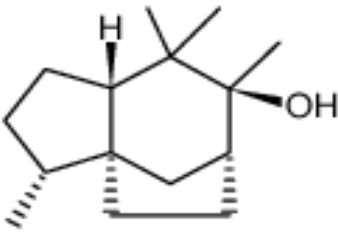
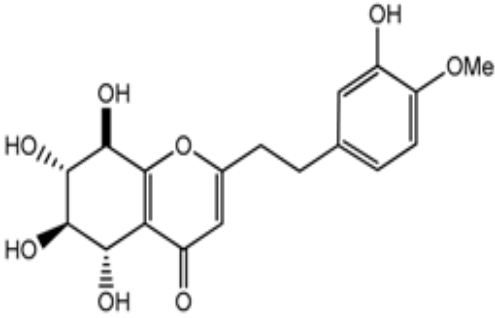
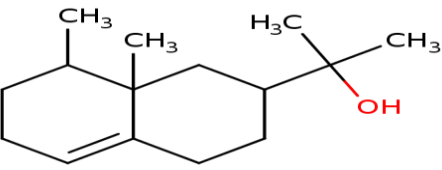
## 2.5 Chemical Component in Agarwood

Generally, the fragrance substances or aromatic of resin of Agarwood essence is belonging to the sesquiterpene and has particular chemical structure (Adi *et al.*, 2016). Sesquiterpenes can be classified as the main active constituents that have important function which is giving the scent, pleasant odor and unique aroma of agarwood (Yumi *et al.*, 2014). Sesquiterpene usually has properties of anti-allergy and anti-inflammatory. There are about 15 carbon atoms that contained in the sesquiterpenes and also have multifaceted pharmacological actions. This sesquiterpene not only found in Gaharu but also can be found in floral oils like rose and chamomile.

It has been reported that the components that usually contained in the Agarwoods are sesquiterpene-noids of eremophilane-, 5,6 nor-guaiane-, 5-7 spirovetivane-, 6guaiane-, 5,7 eudesmane-, 8 and prezizaane-type, 5 2-(2-phenylethyl) chromone derivatives (Jun-ya *et al.*, 2006). There is three new sesquiterpenic furanoids that derived from the fungus infected plant. The fungus that is dominant to infect the *Aquilaria*'s stem is known as *Aspergillus niger*. Other than that, there is also other pathogenic fungus that contained in the Agarwood which are *Penicillium*, *Fusarium* and *Trichoderma sp.*

In addition, there is component of monoterpenes in Gaharu that contained about 10 carbon atoms structure that resulting from units of two isoprenes which are mostly found in the essential oils. Monoterpenes are minimally having one double bond and they will willingly react to the sources of heat and air. For Gaharu essential oil, it has been reported that the major compounds that usually contained in them are limonene (16–18%),  $\alpha$ -pinene (38–54%) and myrcene (9–19%) (Svetomir *et al.*, 2013). The character, content and composition of essential oils that extracted from plant of different species, same species of plant or different parts of species plant will be not same to each other because of the different climate, the age of plant, soil factor and also different locations geography. The important chemical compounds that contained in Gaharu which contribute to the scent of Gaharu are agarospirol, jinkohol, jinkohol-eremol and khusenol.

Table 2.1: Chemical Compounds in Agarwood (from google image)

Name of Chemical Compound	Chemical Structure	Molecular Weight (g/mol)
Agarospirol		222.366
Jinkohol		222.366
Jinkohol-eremol		222.366
Kusenol		472.527

## 2.6 Uses of Agarwood

Agarwood has been used for various purposes for many thousands year throughout the world. Agarwood is a non-timber forest product that is very expensive which can be used in perfumes, incense, medications, aromatherapy and spiritual ceremonies (Yangyang *et al.*, 2013). It is very important in traditional medicinal plant that used to treat numerous infectious ailments including inflammatory diseases and also has been widely used to treat digestive and sedative disorders in Arabs and Japanese (Saad Sabbar *et al.*, 2016).

Incense is actually comes from Latin words which is *incendere* that giving the meaning of ‘to burn’. It contained the aromatic material that can produce the good aroma after burning. Basically, the smoke of incense is functioning as mask odor and also used in aromatherapy. In Chinese, the people there describe the smell of the incense is sweet. In Japanese, Indians and Arabians, this incense is used during the party of festal and also used in the religious. For example in Japan, the agarwood is used in ancient *koh doh* or “incense’s listening” ceremony.

The quality of the incense agarwood can be classified according to the physical characteristics which are including the density, solubility, odour and colour. In addition, incense smoke consists of two types which are Eastern incense and Western incense. The production of Eastern incense is from the natural plants like agarwood. Meanwhile for Western incense, it is processing from the resin in the flowers and oils. In order to produce pleasant aromatic in the house or anywhere, the incense is burned. The Agarwood that is in powder form is to given in appetite’s loss functioning as increases the circulation of blood, lessens the cough and reliefs in asthma and bronchitis. Then, Agarwood also used to treat the fevers because Agarwood has the cooling effect. It also can treat colic pain, toothache, pains and headache during the pregnancy.

Besides that, the Gaharu distilled water is very useful to the people that have sensitive skin or sensitive body such as elderly and infants. Besides, hydrosol also can be used for the patients who undergo eye surgery or whose have irritated eyes. They can directly apply the hydrosol to their eyes to compress it. However, all the things including

utensils and containers must be sterilize before treating the eyes. In addition, hydrosol is very suitable to apply for external skin which is can act as anti-depressant, can get rid of the anxiety and easing insomnia. This hydrosol also can be used as a tonic drink in order to balance the woman hormone and help during menopause. In India, there are a few farmers that applied the hydrosols on the agricultural crops in order to avoid insect pests and also disease. Besides, both hydrosols and essential oils are antiseptic and anti-inflammatory. However, many patients more preferred to use hydrosol because it is the component of water so that it more tolerable and refreshing. Next, hydrosols can be used by the young children and also babies safely.

In addition, Gaharu oil is actually an essential oil that comes from steam and water distillation of Gaharu. Gaharu essential oil has various uses such as to treat cancer, rheumatism, hepatitis, kidney disorders, malaria and also to relieve stress. For an example, the Gaharu in Tibet functions as a drug like painkillers, stomachache, nerve stimulant, anti-asthmatic and lung tumors (Adi Parwata *et al.*, 2016). In addition, a drink that contained one drop of Gaharu oil can help to warm up the cold body of human during cool seasons. Gaharu oil also contained high value in manufacturing of luxury perfume, soap and fragrance (M.A. Islam *et al.*, 2014). Table below shows the summaries of Agarwood's uses:

Table 2.2: Uses of Agarwood (M.Farid *et al.*, 2010)

Agarwood's type	Uses
Incense production	Ceremonies of religious, homes, meditation and rituals.
Gaharu oil	For perfume, products of toiletry like shampoo and soap.
Wood	Chinese, Ayurvedic and other Asian traditional Asian medicine.

## 2.7 Grading and Prizing of Agarwood

Generally, there are many forms of Agarwood which are including chips form, blocks, powder, logs and flakes. Agarwood can be classified into some grades which are Grade Super A, Grade A, Grade B, Grade C, and Grade D. The most expensive is Grade Super A compared to others Grade. The classification of Grade and the value of Agarwood are based on the quality of the Agarwood. The indicators of Agarwood's quality are including the density and color of the wood.

The wood that has greater quantity of oleoresin will be in dark color compared to the less amount of oleoresin contained in wood will have less-darkened color. Agarwood that originates from the older trees will have deep in color and usually have the best quality. So, there are several sellers that soaking the chips of wood in a petroleum-based synthetic oil mixture in order to make it becomes dark in color. This type of wood is called as black magic wood (BMW). Figure below shows the Grade A Agarwood, Grade B Agarwood and “Black Magic Wood” (BMW).

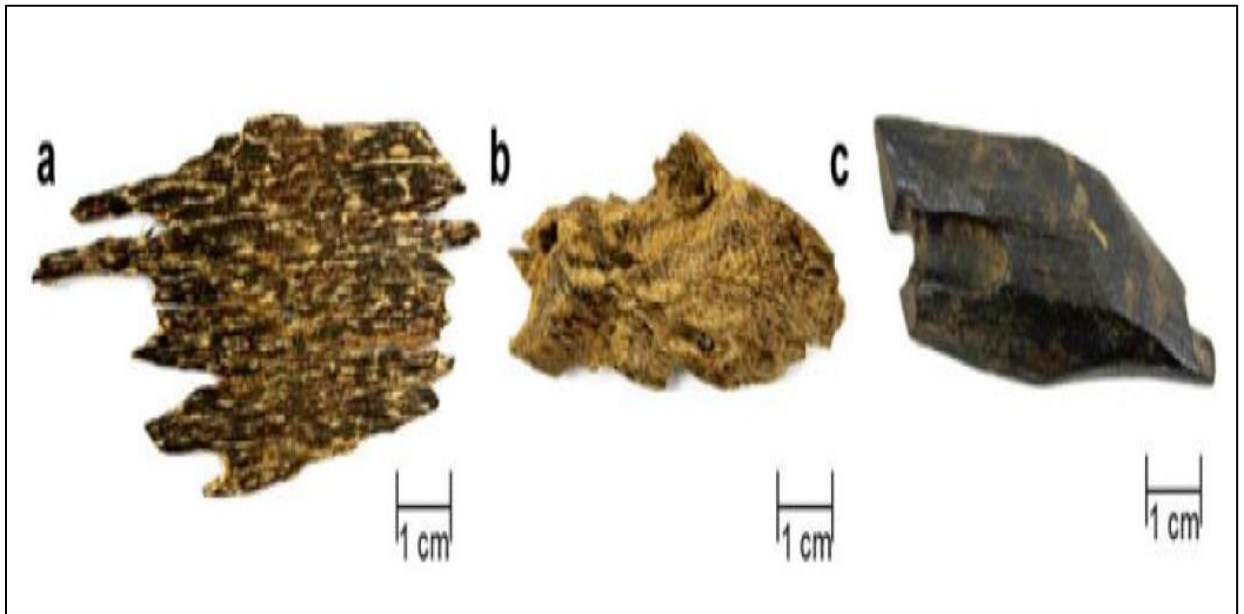


Figure 2.3: (a) Grade A Agarwood, (b) Grade B Agarwood, and (c) “Black Magic Wood” (BMW) (Lee et al., 2016)

Besides, the grade of Agarwood also has the grading system itself in according to the country. However, there is no efficient explanation about this system. Then, from the information that has been supplied by many traders, there are some other characteristics in order to differentiate the grades of Agarwood. First, the higher quantity of resin in Agarwood will cause the higher price. It is because the high amount of resin will lead to high pure level of scent will come from the piece of woods. The next characteristic is the age and tree's location. The portion of the tree from where the removing of Agarwood is can be the indicator of the Agarwood's quality. For instance, the more valuable Agarwood is derived from the root's part compared to other tree's part. In addition, more quantity of resin is contained in thick pieces of Agarwood compared to the thin pieces.

The price of Agarwood oil is quite expensive because the oil has unique aroma (Yumi *et al.*, 2014). In this world, the most expensive of Agarwood is the first-grade Agarwood which is about ranging of a few dollars for 1 kg for low quality, meanwhile for the top quality Agarwood, the price is more than USD 30,000 for 1 kg (K Jayachandran *et al.*, 2014). Besides that, it has been also reported that the price of Grade A Agarwood is about RM1,000 for 1 kilogramme in 1985 and then increased to RM14,000 for 1 kilogramme in 2008 (M.Farid *et al.*, 2010). The price is increased of numerous Agarwood's Grades because of the highly demand of Agarwood and also the less supplier of Agarwood.

However in the Middle East, the price of Malaysian Agarwood that has the lowest grade is USD19 per kg meanwhile for the top grade it cost about USD9589 per kg. In every year, it is estimated that the Agarwood's value shipped out of Singapore is exceeded \$1.2 billion. Next, the demand of Agarwood in industrial of perfume has increase year by year. As a result, the price of Agarwood will be higher because of the less supply of Agarwood in this world. So, the price of raw material of perfume which is Agarwood becomes the most expensive in the world.

## 2.8 Gas Chromatography-Mass Spectrometry (GC-MS)

Gas Chromatography Mass Spectrometry (GC-MS) is actually the two techniques which are gas chromatography and mass spectroscopy that are combined together in order to appraise the quantitative and qualitative of the mixture compounds (N.Ismail *et al.*, 2016). Basically, the goal of GC-MS is to identify, to separate, and to quantify the mixture compounds like essential oils and also to define the unknown compounds arrangement (Isabel *et al.*, 2009). There are two criteria that must be fulfill in order to accomplish this goal which are the identification of the individual mass spectrum must be determine correctly and the plenty of chromatographic peaks must be calculate correctly corresponded to each sample compounds (Lea G. Johnsen *et al.*, 2017).

In mass analysis, the ions or particles of charged are necessary in order to develop with the electron impact ionization (EI). In gas chromatography, the mass spectrometer is used as detector and it was developed by Fred McLafferty and Roland Gohlke in 1950s (Kataria Sahil *et al.*, 2011). These bulky devices are sensitive with their fragile materials. The computers was developed in order to help in simplification of the instrument usage and to shorten the time taken in sample analyzing. The figure below shows the GC-MS looks like.



Figure 2.4: Insides of GC-MS looks like, with the gas chromatography column (Kataria Sahil *et al.*, 2011)

To be clearer, figure below shows the schematic diagram of GC-MS.

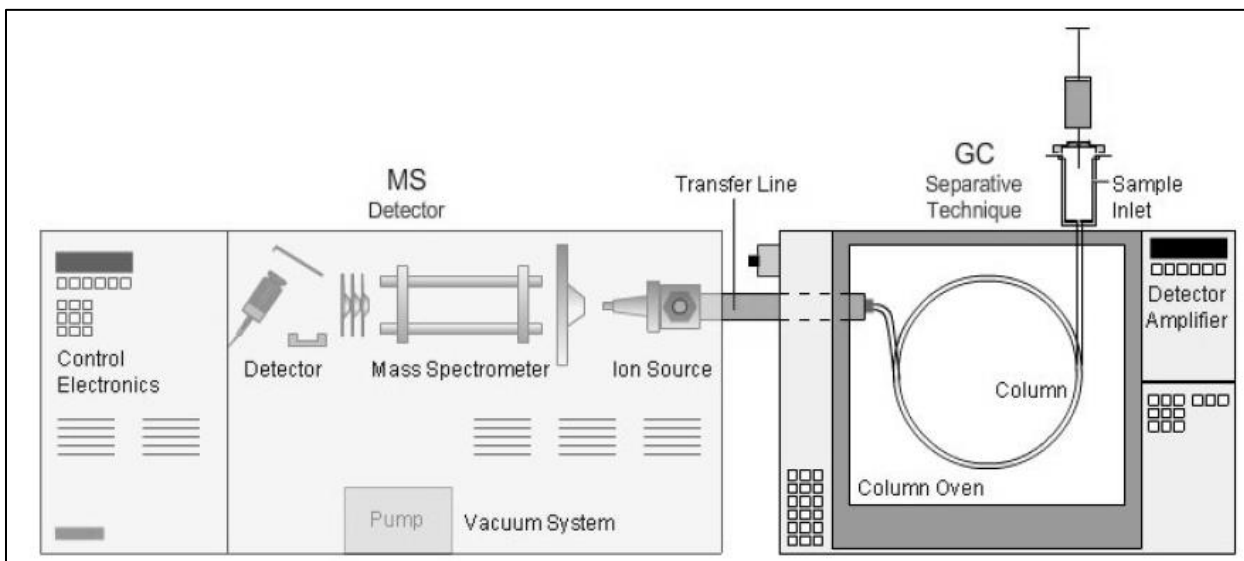


Figure 2.5: GC-MS Schematic Diagram (Maqbool *et al.*, 2014)

Based on the figure above, GC-MS has two major building blocks which are gas chromatograph (GC) separative technique that consist of injector, column and oven and the second building block is mass spectrometer (MS) detector that consist of mass spectrometer, control electronics, detector, ion source and oven. Gas chromatography equipment usually comprises of injection port which located at the end of metal column that is packed with material of substrate and the detector which is located at the other end of the metal column. Injector functions as to volatilize the sample and then entered the gas chromatograph column. Column is used as the transportation medium of sample components by using gaseous mobile phase. The size of columns capillary gas chromatograph is normally 10-120 m long, 0.10-0.50 mm internal diameter and length of 1-5 meters. Then, in any gas chromatography, there is an oven that normally has range temperature about 5<sup>0</sup>C to 400<sup>0</sup>C but sometimes the temperature can stand at low which is about -25<sup>0</sup>C with the cooling of cryogenic.

GC-MS are widely used in many industries including pharmaceutical industry, food, flavor, beverage and fragrance industry. In this research, GC-MS was used to analyze the chemical profiles in Gaharu distilled water. A sample of hydrosol is introduced into the GC in order to be heated to become vapor and then carried by inert



gas which is helium along the column. When it is vaporized through the column, it will separate into constituent of individual molecular after it interacts with the column stationary phase. Next, the constituent that has been separated is passed into the MS. This constituent will become ionized in the MS module. The amplification and detection as current of the charged constituent is done by the MS. The peak will be presented in a chromatograph for each of the constituent. In order to determine the substance, all the peaks will be compared to a molecule's library.

The chemical profiles analysis is really required to classify the Agarwood oil based on their respective classes where we can measure the accurate results (Nurlaila *et al.*, 2013). In a nutshell, GC-MS can be classified as an advanced technique compared to other analytical equipment because it gives very effective result, more efficient and reproducible (Ashish chauhan *et al.*, 2014).

## **2.9 Inductively Coupled Plasma (ICP)**

Generally, inductively coupled (ICP) system has been widely used in industry in order to determine directly the trace elements in geological things and directly can be obtained a lot of information about the sample (Qi Liang *et al.*, 2000). The determinations of trace metal in samples are depending on the atomic emission spectrometry (IES). ICP actually comes from source of plasma where the energy is provided by the electrical currents which is made by electromagnetic induction. It consists of sufficient ions and electrons concentration in order to make the conductive gas electrical. This technique is great and the equipment is quite sensitive in order to define the trace metal accurately. The positive ions in these plasmas are mostly all singly-charged and just a few negative ions there. So the electrons and ions are having quite equal amounts in each plasma unit. ICP is basically comprises of three concentric tubes that made from silica. Coupling is obtained when the magnetic field is generated by passing the electric current with high frequency through a cooled coil's induction.

Then, the components that include in ICP are ICP torch, introduction system sample (nebulizer), generator with high frequency, transfer optics and spectrometer, and lastly the computer interface. The type of solution that is preferred is aqueous solution

instead of organic solution because organic solutions need special manipulation prior to ICP injection. Then, sample of solids are not preferred because it can cause the instrumentation to be clogged. The function of nebulizer is to convert the aqueous solution into an aerosol. Some advantages of ICP are including its capability to determine all type of elements except Argon. It is also can perform the analysis of multi elemental rapidly.

In this study, inductively coupled plasma (ICP) system was used in order to determine the total heavy metal content that presence in the hydrosol such as Arsenic (As), Magnesium (Mg), Plumbum (Pb), Ferum (Fe), Zinc (Zn), Lead (Pb) and Cadmium (Cd). This heavy metal test was done in order to make sure there are no heavy metal present in the Gaharu distil water. It is because heavy metal can cause risks for the healthiness of human and also give bad effect to the environment (Monisha *et al.*, 2014). In this work, we must make sure that there is zero heavy metal in Gaharu distil water in order to save human's lives and also to keep our environment from the pollution.

Figure below shows the ICP cross-sectional view.

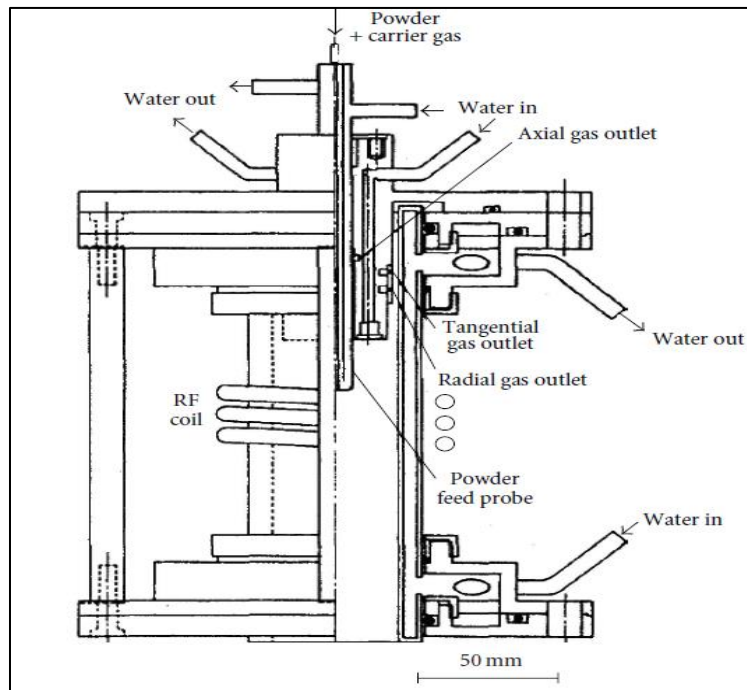


Figure 2.6: ICP cross-sectional view (Okumura, 2010)

## 2.10 Fourier Transforms Infrared (FTIR)

FTIR measures the frequencies at which the sample absorbs, and also the intensities of these absorptions. The frequencies are helpful for the identification of the sample's chemical make-up due to the fact that chemical functional groups are responsible for the absorption of radiation at different frequencies. FTIR is very helpful for the predictive assignment of chemical compounds.



*Figure 2.7: Fourier Transforms Infrared (FTIR)(from google image)*

## CHAPTER 3

### RESEARCH METHODOLOGY

#### 3.1 Materials and instruments

The materials and instruments require in this research are including: Agarwood of *Aquilaria species* that used in this study were obtained from natural population of Kuala Krai, Kelantan in September 2017, distilled water, hexane, Fourier Transforms Infrared (FTIR) spectroscopy (Perkin Elmer 2000 Model), gas chromatographic-mass spectrometer (GC-MS) model Varian 240-MS completes with the 450-GC with a Combi PAL autosampler from CTC Analytics, Inductive Couple Plasma (ICP) Spectrometer (ThermoFisher Scientific) with brand or model is iCAP 6000 series, 1 Liter of separation funnel, hydrodistillation (extraction facilities), grinder, 50 ml of beakers, 50 ml of measuring cylinder, dropper, 20 ml of pipette, seven unit of 500 ml plastic bottles, volumetric flask and vial cronus clear 12 x 32mm.

#### 3.2 Methodology

##### 3.2.1 Grinding

About 25 kg of dried Agarwood were ground by using grinder machine. The large trunk of Agarwood was chopped into the smaller size. This is done in order to obtain the maximum surface area for the process of extraction and also to give maximum contact time between the particle of Agarwood and the solvent. Figures below show the Agarwood collection.



Figure 3.1: Agarwood were cut into smaller size



Figure 3.2: The smaller size of Agarwood

### 3.2.2 Extraction Method

In this study, the type of extraction method that has been used is hydrodistillation process or known as condensation process of boiled Agarwood. Traditionally, the hydrosol was extracted from *Aquilaria* sp has been done by using hydrodistillation method (Norul *et al.*, 2015). About 25 kg dried Agarwood was extracted by using extraction facilities. Before the time of extraction process was taken, the system was left for 15 minutes to equilibrium state. It is to ensure the process of extraction was conducted in stable condition. Hydrodistillation was conducted at various time starting with 12 hours, 18 hours, 24 hours, 30 hours, 36 hours, 42 hours and 48 hours to collect the gaharu distil water in a bottle. The extraction process was started at 6pm on 14<sup>th</sup> March 2018. About 40ml of gaharu distil water was collected in a bottle every 6 hours which is until 6am of 16<sup>th</sup> March 2018. The hydrodistillation process cannot be exceeding the temperature of 120<sup>0</sup>C to avoid the plant material from burning. The temperature of this extraction process is constant about 100<sup>0</sup>C.

During the distillation process, the vapor that consist of volatile compounds and steam were rise to the condenser from the extractor, where two phases of immiscible liquid are formed which are known as Agarwood oil and aromatic water (hydrosol). The oil of Agarwood which is lighter than hydrosol was separated from the hydrosol and located at the top of the separation funnel, meanwhile the hydrosol is formed below the Gaharu oil. 500ml of hydrosol was

collected for every 6 hours in the 500ml of plastic bottle in order to analyze their chemical profiling and evaluate the quality of hydrosol. Figure below shows the figure of extraction facilities that has been use during this research.



Figure 3.3: Extraction Facilities



Figure 3.4: Separation funnel that separate oil and hydrosol

### 3.2.3 Physiochemical Studies of Hydrosol

The hydrosol that obtained from extraction of Gaharu (hydrodistillation) were tested in order to obtain the chemical profiling and also to study the water quality. For the chemical profiling, it was analyzed by using gas chromatographic mass spectrometer (GC-MS). Meanwhile for water quality, it is to define dissolve oxygen (DO) value, pH value and also test for the heavy metal like Arsenic (Ar), Mercury (Hg) and Plumbum (Pb) at various extraction time.



### 3.2.4 Preparation of Sample Hydrosol in Hexane

First of all, about 1ml of hydrosol of each seven samples of hydrosols was filled up into the seven different beakers by using pipette. Then, 10 ml of hexane was pipetted into each beaker that contained hydrosol. The solution was shaking for 2 minutes to make sure the mixture was well mixed. After that, visually observe the solution if there was any layer formation. The two layers were formed which were hexane layer and hydrosol. Then, about 1 ml of the hexane solution's layer was transferred into the vial cronus clear 12 x 32mm by using dropper. Then, the solution was analyzed using GC-MS to analyze the chemical constituents in hydrosol. Figure below shows the preparation of sample hydrosol in hexane:



Figure 3.5: Seven beakers that contained two layers of hydrosol and hexane solution after shaking



Figure 3.6: The solution kept in the vial cronus clear to undergo GC-MS analysis

### 3.2.5 Characterization Method by Gas Chromatographic-Mass Spectrometer (GC-MS)

The characterization method or chemical profiling of Gaharu distil water is obtained by chromatographic technique which is by using gas chromatographic mass spectrometer (GC-MS) model Varian 240-MS completes with the 450-GC, with a Combi PAL autosampler from CTC Analytics. It is to analyze the chemical components that contained in Gaharu hydrosol. The sample of hydrosol was injected in GC-MS that equipped with a split injector and the carrier gas that used is Helium. Then, electron ionization (EI) mass spectra were noted in the full scan mode at certain electron energy. At the end, the system will display the results obtained. We can detect the peaks from GCMS depends on the total ion chromatography (TIC) and mass chromatography. Figure below shows the figure of GC-MS that has been use during this research.



Figure 3.7: Gas Chromatographic-Mass Spectrometer (GC-MS) model Varian 240-MS completes with the 450-GC, with a Combi PAL autosampler from CTC Analytics



### 3.2.6 Water Quality Test for Heavy Metal

The Gaharu distil water is undergo heavy metal test which are analyzed by using Inductive Couple Plasma (ICP) system (ThermoFisher Scientific) with brand or model is iCAP 6000 series. This test is conducted to make sure whether there is present of heavy metal or not in the Gaharu distil water. We must make sure that the results are not containing any heavy metals in this hydrosol because those elements can give a risk to human health if they use this hydrosol. Figure below shows the figure of ICP that has been use during this research. In this study, test for the heavy metal is conducted which were Arsenic (Ar), Magnesium (Mg), Ferum (Fe), Zinc (Zn), Lead (Pb) and Cadmium (Cd) at various extraction time.

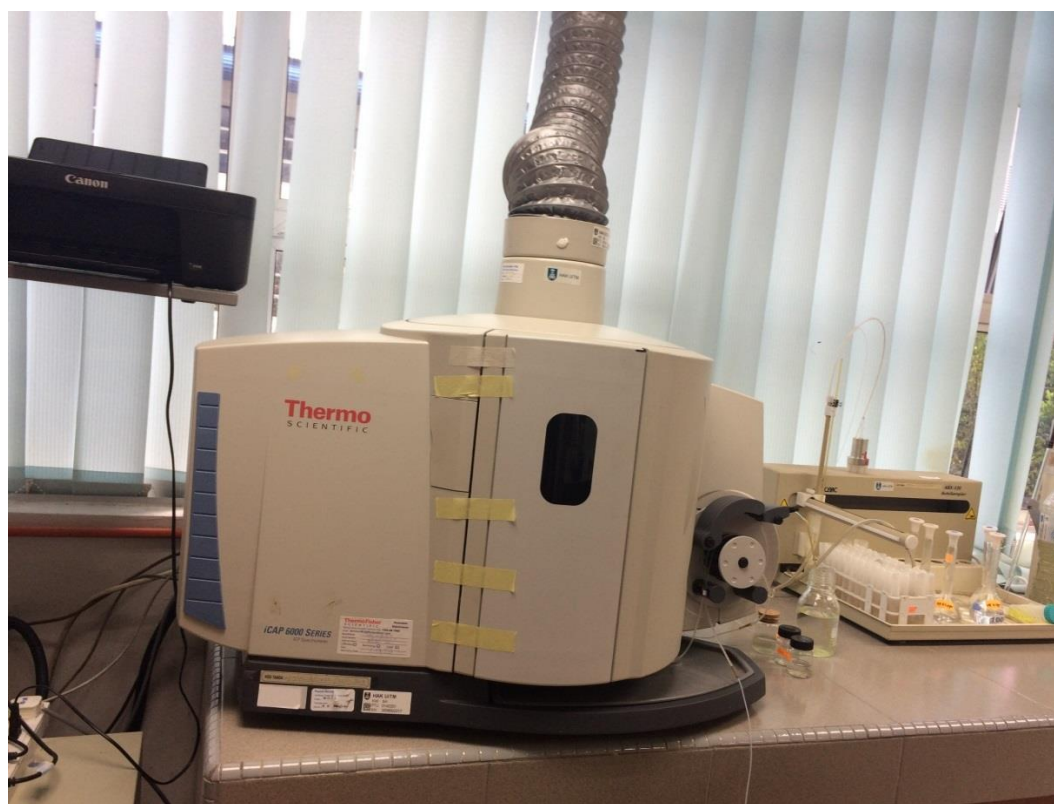


Figure 3.8: Inductive Couple Plasma (ICP) system system (ThermoFisher Scientific) with brand or model is iCAP 6000 series

### 3.2.7 Characterization Method by Fourier Transform Infrared (FTIR)

All hydrosols were characterized by FTIR spectroscopy (Perkin Elmer 2000 Model) to identify the active functional groups. The FTIR study was carried out by using the Perkin Elmer System 2000 FTIR instrument. First, the transparent Pellets (thin disc) were formed by mixing 5mg of the sample with 100 mg of potassium bromide (KBr) (1:20) using a mould and press, and compressed under a pressure of 7 ton. The investigation was performed within the wavelength ranging from 4000 to 400  $\text{cm}^{-1}$  and the spectrum takes about three minutes to be recorded. The acquisition of the spectra and peaks assignment was performed using FTIR software Spectrum 3.02.01 (Perkin Elmer, Inc., Waltham, MA).



Figure 3.9: Fourier Transform Infrared (FTIR)

## CHAPTER 4

### RESULT AND DISCUSSION

#### 4.1 Introduction

By using hydrodistillation method, the 25 kg dried Agarwood could gave about 1 litre of Agarwood distil water or known as hydrosol. This extraction process of hydrosol was run continuously about 48 hours. However, in this study, the hydrosols were collected at various time of extraction process starting with 12 hours, 18 hours, 24 hours, 30 hours, 36 hours, 42 hours and 48 hours by collecting hydrosols in the bottles. The extraction process was started at 6pm on 14<sup>th</sup> March 2018. About 40ml of hydrosol was collected in each bottle every 6 hours which is until 6am of 16<sup>th</sup> March 2018. The hydrosols were successful collected in seven bottles for every 6 hours as shown below:



Figure 4.1: Collection of hydrosols every 6 hours



Figure 4.2: Seven bottles of hydrosol collection

The samples of hydrosols have been collected about 7 bottles with different time of extraction process. All samples were clear in color like normal water. All these samples were analyzed by using Fourier Transform Infrared (FTIR), Gas Chromatography-Mass Spectrophotometer (GC-MS) and Inductive Couple Plasma (ICP).

## 4.2 Characterization by Fourier Transform Infrared (FTIR) analysis

All the seven samples of hydrosols were characterized by using FTIR. By using FTIR analysis, the characteristic of the absorption peak such as scope and location of hydrosols chemical contents or the functional groups could be identified. About a few drops of each sample of hydrosols were used during FTIR analysis. The infrared spectra of all the samples of hydrosols were taken and evaluated. All the spectrums did not present major changes of peaks for Agarwood distil water at various extraction time. Figures 4.3, 4.4, 4.5, 4.6, 4.7, 4.8 and 4.9 show the IR spectrum of all the samples hydrosols that were collected respectively.

Base on the result, O-H bond of hydrogen bonded alcohol or phenol ( $3600\text{--}3200\text{ cm}^{-1}$ ) group frequency was present in all sample of hydrosols which at different time extraction process with frequency of  $3309.49\text{ cm}^{-1}$ ,  $3309.67\text{ cm}^{-1}$ ,  $3309.02\text{ cm}^{-1}$ ,  $3309.68\text{ cm}^{-1}$ ,  $3309.74\text{ cm}^{-1}$ ,  $3308.97\text{ cm}^{-1}$  and  $3308.68\text{ cm}^{-1}$  respectively. A broad spectrum can be detected from the figures below that denote the availability O-H bond. This is the very important part because the presence of O-H bond indicates the existence of phenolic compound in the hydrosols. This finding was considered successful because it was same as proposed by Khalil *et al.*, (2013) where they had identified the presence of alcohol or phenol functional group in Agarwood or hydrosol.

Then, the band of most prominent in alkynes looks like to the carbon-carbon triple bond. The alkyne  $\text{C}\equiv\text{C-C}$  stretch ( $2260\text{--}2100\text{ cm}^{-1}$ ) group frequency was identified in all samples of hydrosols with frequency  $2101.49\text{ cm}^{-1}$ ,  $2155.24\text{ cm}^{-1}$ ,  $2116.73\text{ cm}^{-1}$ ,  $2134.91\text{ cm}^{-1}$ ,  $2116.25\text{ cm}^{-1}$ ,  $2067.66\text{ cm}^{-1}$  and  $2115.13\text{ cm}^{-1}$  respectively. There was very little organic compounds show an absorption in this region. All these frequencies show as a sharp and weak band at around  $2100\text{ cm}^{-1}$ . The band was weak because of the triple bond that was not very polar. In certain cases, such as for the highly symmetrical alkynes, it might not represent at all because of the polarity of the triple bond was low linked with those alkynes.

Next, H-O-H bond ( $1640\text{--}1630\text{ cm}^{-1}$ ) group frequency was also present in all samples of hydrosols at frequency of  $1636.49\text{ cm}^{-1}$ ,  $1636.36\text{ cm}^{-1}$ ,  $1636.44\text{ cm}^{-1}$ ,  $1636.36\text{ cm}^{-1}$

$\text{cm}^{-1}$ ,  $1636.40 \text{ cm}^{-1}$ ,  $1636.50 \text{ cm}^{-1}$  and  $1636.45 \text{ cm}^{-1}$  respectively. All the samples contained H-O-H bond from water which the Agarwood hydrosol itself is a water homogenous mixture and sesquiterpenoids of Agarwood. From this indication, it is estimated that all these samples of hydrosols have majority of Agarwood sesquiterpenoids.

Last but not least, C-C bond of aromatic ring ( $1500\text{-}1400 \text{ cm}^{-1}$ ) group frequency was identified in all samples of hydrosols with frequency at  $1497.69 \text{ cm}^{-1}$ ,  $1497.90 \text{ cm}^{-1}$ ,  $1497.95 \text{ cm}^{-1}$ ,  $1497.88 \text{ cm}^{-1}$ ,  $1497.90 \text{ cm}^{-1}$ ,  $1497.98 \text{ cm}^{-1}$  and  $1498.07 \text{ cm}^{-1}$  respectively. This aromatic ring was synonym with the characteristic of hydrosol that gave out the distinctive Agarwood aroma. Table below shows the functional groups of hydrosols that obtained from this study.

Table 4.1: Functional groups of hydrosols from *Aquilaria malaccensis*

Band assignment	Functional Group	Group band ( $\text{cm}^{-1}$ )
O-H	Alcohol/Phenol	3600-3200
In ring C-C stretches	Aromatic Ring Alkane	1500-1400
$\text{C}\equiv\text{C}$ -C Stretch	Alkyne	2260-2100
H-O-H	Alkene	1640-1630



All the seven figures below show the plots of FTIR for Agarwood distil water that has been extracted at various time or temperature.

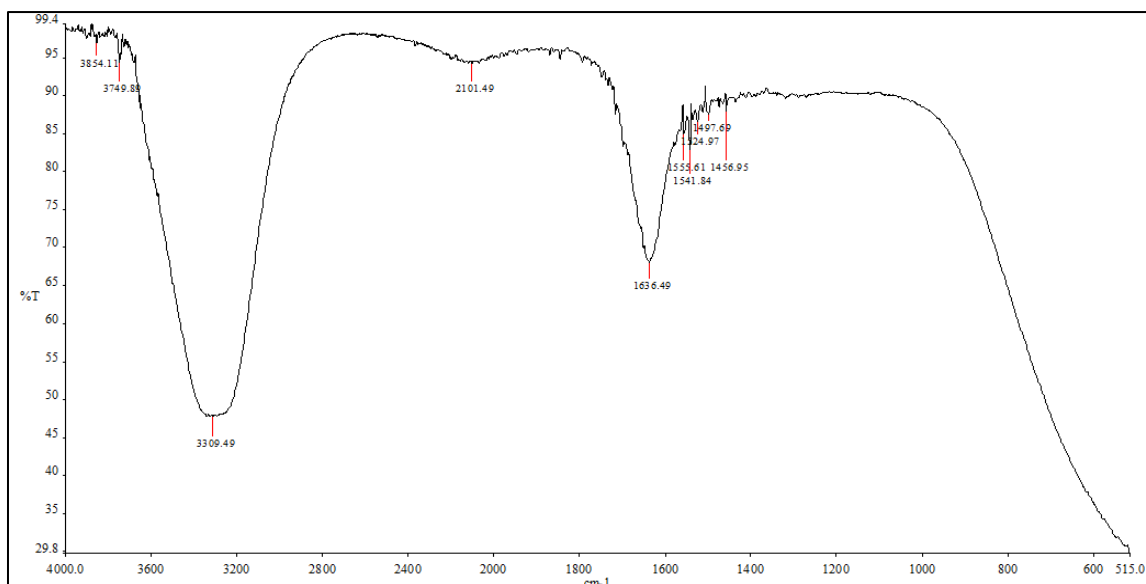


Figure 4.3: FTIR Spectrum for Agarwood distil water after 12 hours extraction process

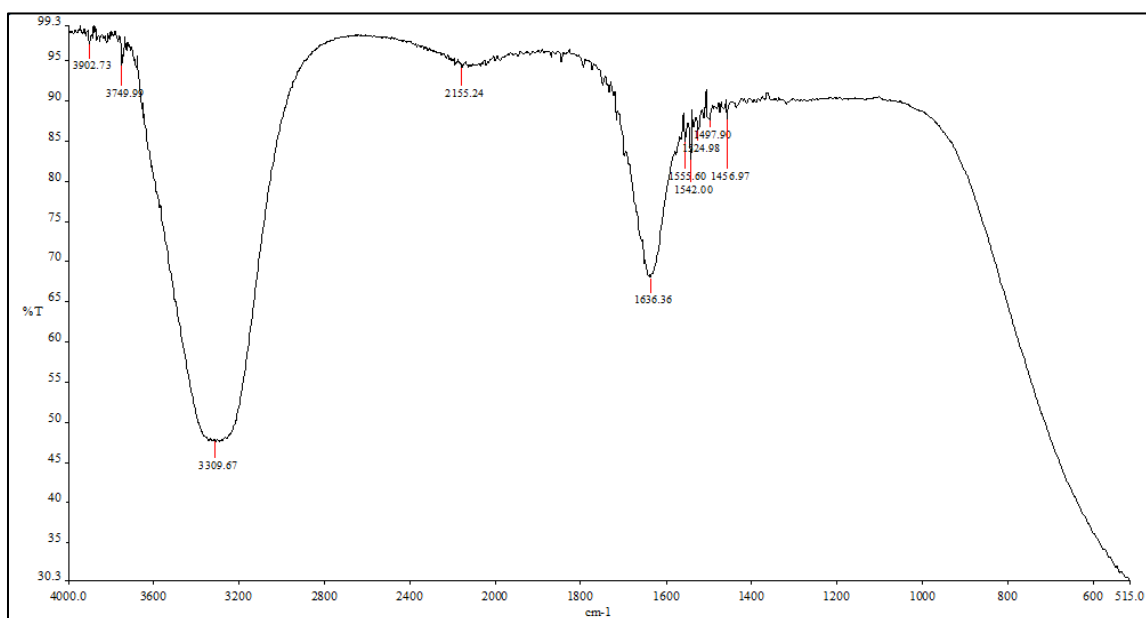


Figure 4.4: FTIR Spectrum for Agarwood distil water after 18 hours extraction process

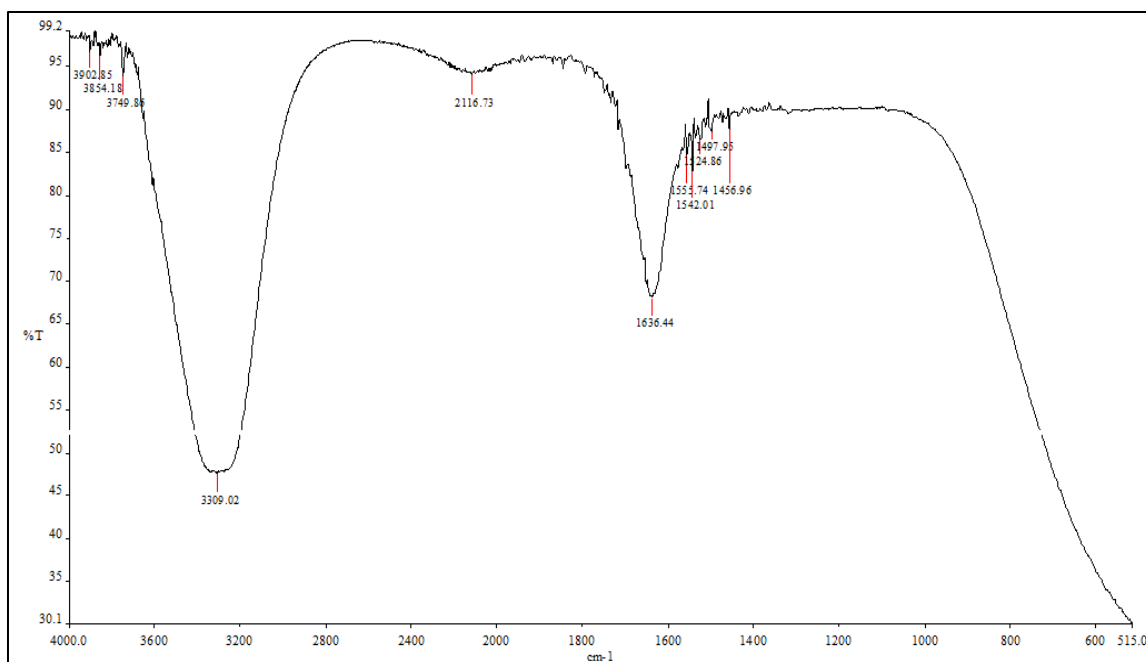


Figure 4.5: FTIR Spectrum for Agarwood distil water after 24 hours extraction process

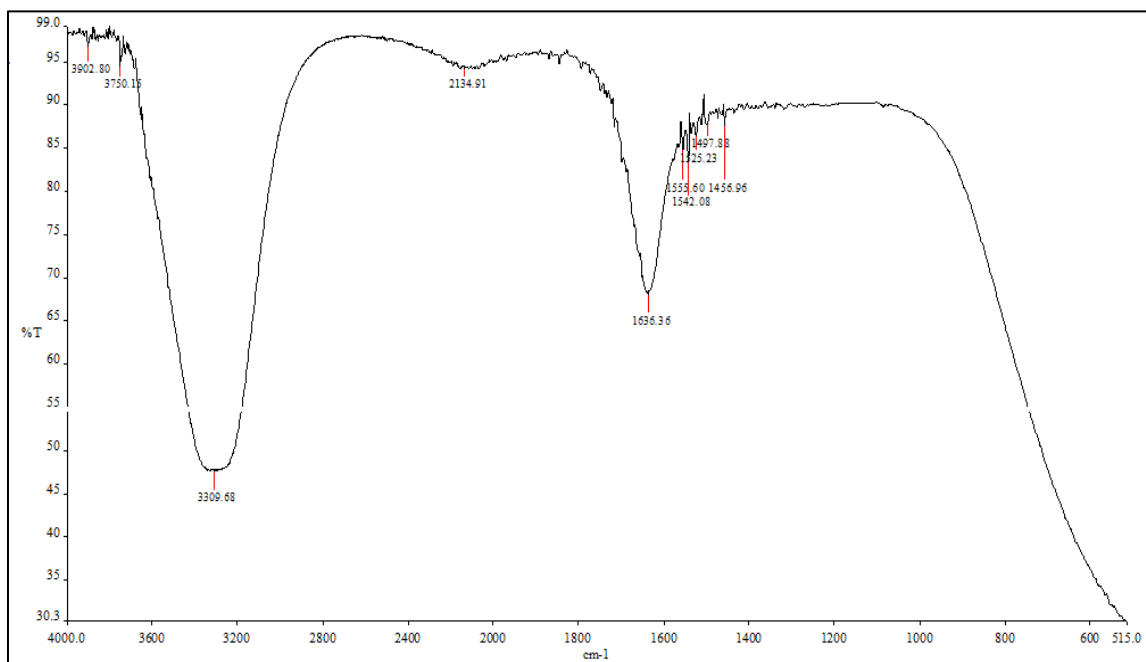


Figure 4.6: FTIR Spectrum for Agarwood distil water after 30 hours extraction process



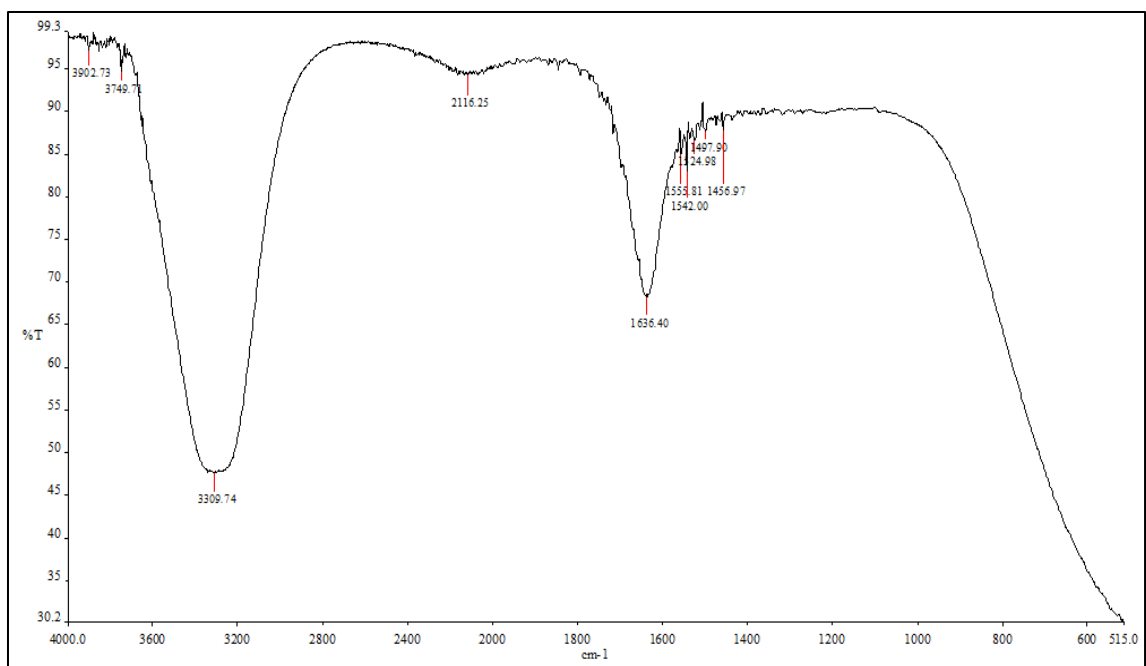


Figure 4.7: FTIR Spectrum for Agarwood distil water after 36 hours extraction process

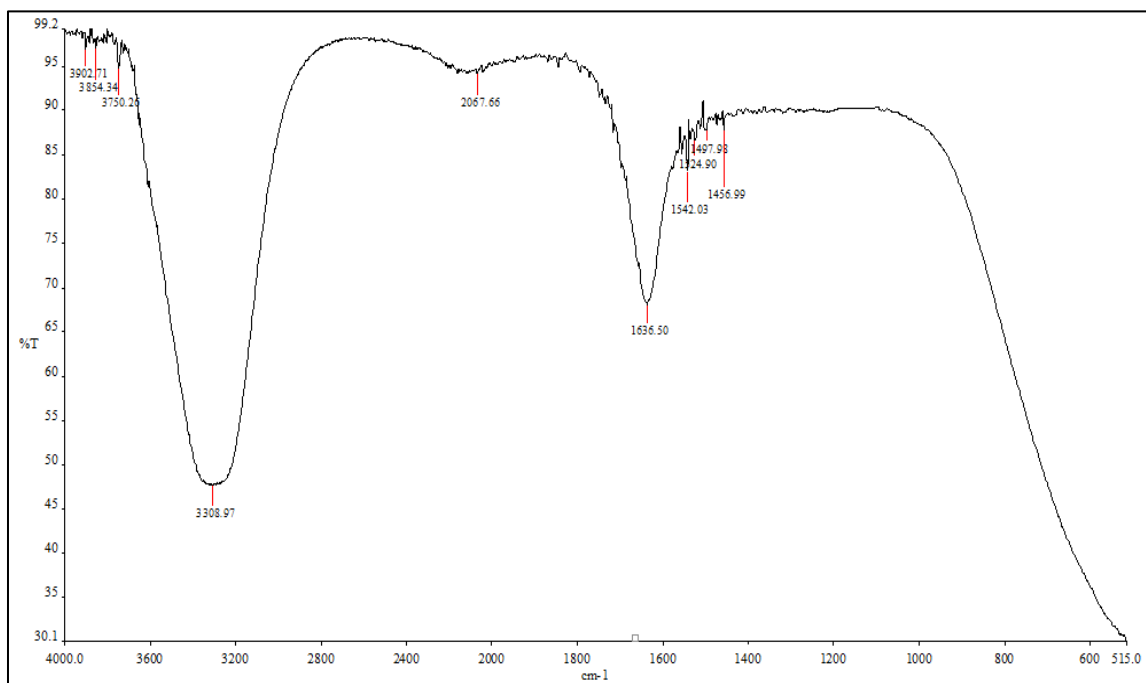


Figure 4.8: FTIR Spectrum for Agarwood distil water after 42 hours extraction process

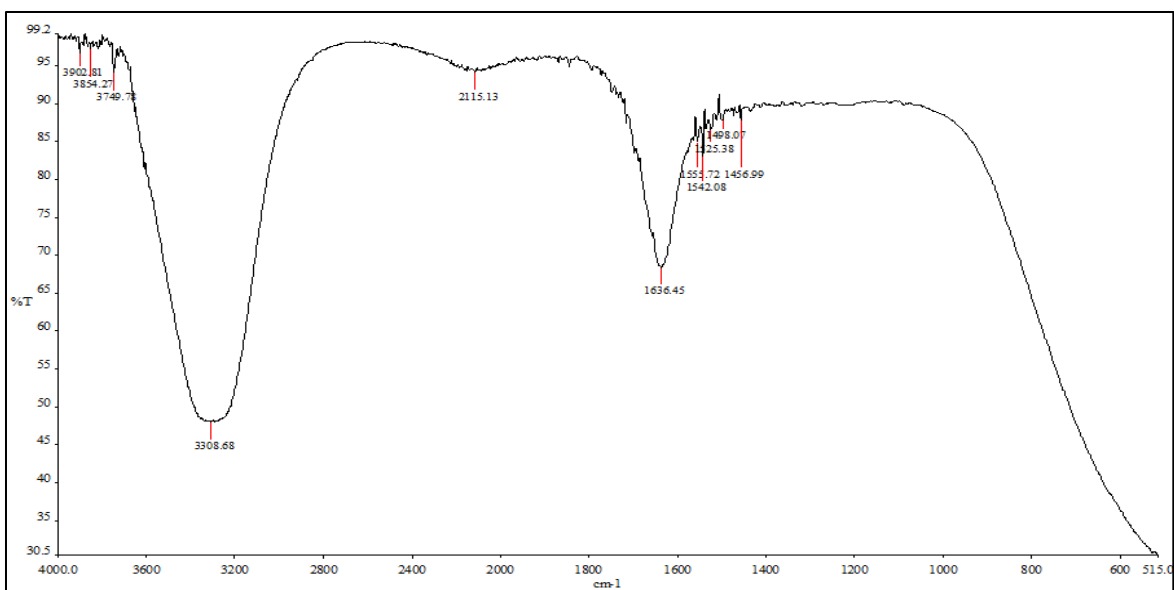


Figure 4.9: FTIR Spectrum for Agarwood distil water after 48 hours extraction process

Table 4.2: Summary of characteristic peaks bands on FTIR spectra

Functional groups (bands)	Wavenumber (cm <sup>-1</sup> )						
Time of extraction process	12 hours	18 hours	24 hours	30 hours	36 hours	42 hours	48 hours
-OH stretch	3309.49	3309.67	3309.02	3309.68	3309.74	3308.97	3308.68
C≡C-C stretch	2101.49	2155.24	2116.73	2134.91	2116.25	2067.66	2115.13
H-O-H water	1636.49	1636.36	1636.44	1636.36	1636.40	1636.50	1636.45
C-C aromatic stretching	1497.69	1497.90	1497.95	1497.88	1497.90	1497.98	1498.07

As a conclusion, it was found that all samples of hydrosols from various extraction time consists the alcohol or phenols functional groups that were very useful in order to prove the presence of phenolic compounds inside the hydrosols. Then, the

presence of H-O-H bond from water since the Agarwood hydrosol itself was a homogenous mixture of water and Agarwood sesquiterpenoids. Next, the presence of C-C bond of aromatic ring was very important because that aromatic ring gave the pleasant smell of hydrosol. Besides, the patterns of FTIR spectrum for all the samples were quite similar in which the entire spectrum did not show significance change of peaks. So, it means that the different time of extraction process did not affect the pattern of FTIR spectrum because all of the samples hydrosols had almost same functional groups. The spectrum of all samples of Agarwood hydrosols were recorded at range of 4000 – 400 cm<sup>-1</sup> (mid infrared spectroscopy) at 4 cm<sup>-1</sup> resolution (FTIR model: Nicolet Avatar 370 DTGS).

#### **4.3 Characterization by using Gas Chromatography-Mass Spectrophotometer (GC-MS)**

GCMS was used to detect the chemical contents in hydrosol. Basically, all of the Agarwood were complex mixtures of sesquiterpene hydrocarbons, sesquiterpene alcohols, aromatic compounds, and aliphatic hydrocarbons that very difficult to be identified based on MS alone where the chemical compounds of Agarwood should be identified by comparing the mass spectral data with the existing Wiley library and reference library spectral data (Nor Azah M.A *et al.*, 2008).

In this study, seven hydrosols samples of different time process extraction were identified by GC-MS analysis. The chemical compounds that were detected in this hydrosol by GC-MS instruments were 48 in total as listed in table 4.3. According to the result, it was found that the chemical component that was present in almost samples was 1,2,4,5-Tetrazine, 1,4-diethylhexahydro. This showed that 1,2,4,5-Tetrazine, 1,4-diethylhexahydro was the main compounds found in hydrosol at different time interval. 1,2,4,5-Tetrazine, 1,4-diethylhexahydro formula was C<sub>6</sub>H<sub>16</sub>N<sub>4</sub> with molecular weight of 144.21804 g/mol. This compound is used in the treatment of diarrhea and worm infections, and also used to treat the involuntary movements (chorea) of Huntington's disease or progressive brain disorder that causes uncontrolled movements, emotional problems, and loss of thinking ability.

From the results, it indicates that there were some differences and variations in the chemical composition of hydrosol at different time. According to Jutarut Pornpunyapat *et al.*, (2011), the different extraction process time will effect on the chemical compounds of hydrosol. The higher number of chemical components should be result at longer extraction time. So, this study was similar to the findings by Jutarut Pornpunyapat *et al.*, (2011) because the results showed the differences of chemical contents in all samples at different production time. Meanwhile, for the number of chemical components, the result showed that the number of chemical component in hydrosol was increased from hour 12 until hour 36 which were from 8 to 15 of chemical components. Then it was decreased at hour 42 and hour 48. The decreasing in number of chemical components means that there was something wrong because it should be the higher number of chemical compounds when the production time was longer.

This failure may cause by the incorrect preparation of sample hydrosol in hexane. In this study, the solvent extraction had been conducted before the analysis of GC-MS was done. The reason of conducting this solvent extraction was because of the sample of this hydrosol was water based solution, so that it should undergo the solvent extraction which was by using the hexane as a solvent in order to give a better sensitivity for the GC-MS test. It is because GC-MS instrument could not handle the sample from the water base solution and it also had a potential to cause the damage of column. It should inject the extracted sample in the GC-MS because it could not detect the chemical component from the water based. However, this step of solvent extraction also did not give the best result. It might cause of the ratio of sample to hexane were not suitable. The ratio of sample to hexane that was used in this study was 1:10. It might be the solvent could not extract the ion in sample properly because the content of sample was too little. Hence, another step should be done in order to get the most accurate results. Table 4.3 shows the chemical compounds of the all samples.

Table 4.3: Compounds Identified from GC-MS Analysis of Hydrosol

No	Compounds	Chromatogram Area (%)						
	Time of extraction process	12 hours	18 hours	24 hours	30 hours	36 hours	42 hours	48 hours
1	Pentane, 3-ethyl-2,2-dimethyl	0.2120	-	-	0.3690	-	-	-
2	2-Octanol, 8,8-dimethoxy-2,6-dimethyl	0.0466	-	-	-	-	-	-
3	1,2,4,5-Tetrazine, 1,4-diethylhexahydro	0.0459	-	0.0612	0.0967	0.0665	-	0.1140
4	Allyldimethyl(vinyl)silane	0.0057	0.0352	0.0284	0.0077	-	0.0050	0.0146
5	2,2-Dimethylpropanoic anhydride	0.0046	-	-	-	-	-	-
6	3H-1,2,4-Triazol-3-one, 1,2-dihydro	0.0056	0.0058	-	0.0102	-	0.0050	0.0098
7	Oxalic acid, cyclohexyl propyl ester	0.1760	-	-	-	-	-	-
8	Oxalic acid, butyl cyclobutyl ester	0.0213	-	-	-	-	-	-
9	2-Isopropyl-3-vinyloxirane	-	0.6600	0.2570	-	-	-	0.6870
10	4-Methoxypyrrrolo[2,3-d]pyrimidine	-	0.0056	-	-	-	-	-
11	Thiazole	-	0.0040	-	-	-	-	-
12	1H-Tetrazole, 1-	-	0.0094	-	-	-	-	-

	methyl							
13	2-Methyl-1,5-hexadiene-3-ol	-	0.0029	-	-	-	-	-
14	Acetamide, N-(2-hydroxyethyl	-	0.0490	-	-	-	-	-
15	Silicon tetrafluoride	-	0.0019	-	-	-	-	-
16	Oxalic acid, cyclohexyl hexyl ester	-	-	-	-	-	-	0.2520
17	Nonane, 2,2,3-trimethyl	-	-	1.160 0	-	-	-	-
18	2-Propynal	-	-	0.015 9	-	-	-	-
19	1-Pentanol, 4-methyl	-	-	0.016 1	-	-	-	-
20	Cyclobutanecarboxylic acid, 2-propenyl ester	-	-	0.348 0	0.158 0	-	0.390 0	-
21	Butane, 1-chloro	-	-	0.018 7	-	-	-	-
22	(Trimethylsilyl)acetylene	-	-	0.013 8	-	-	-	-
23	1-Hexene, 4-methyl	-	-	-	0.606 0	0.624 0	0.225 0	-
24	Trimethylaluminum	-	-	-	0.000 3	-	-	-
25	Thiopivalic acid	-	-	-	0.052 7	-	-	0.0640
26	6-Heptene-2,4-diol	-	-	-	0.036	-	-	-

					1			
27	Hexanoic acid, 2-oxo-, methyl ester	-	-	-	0.0085	0.0182	-	0.0090
28	2-Pentanone, 5,5'-oxybis	-	-	-	0.0606	-	-	-
29	Cyclopropane, 2-bromo-1,1,3-trimethyl	-	-	-	0.3430	0.3860	-	-
30	5-Nonanone	-	-	-	0.0181	-	-	-
31	Decane, 2,2,3-trimethyl	-	-	-	-	0.3130	-	-
32	4,8-Dioxatricyclo[5.1.0.0(3,5)]octane, 1-methyl-5-(1-methylethyl)	-	-	-	-	0.0008	-	-
33	Cyclopropanecarboxylic acid, but-3-yn-2-yl ester	-	-	-	-	0.0048	-	-
34	2-Pentanol, 5-methoxy-2-methyl	-	-	-	-	0.0805	-	-
35	S-Methyl 3-methylbutanethioate	-	-	-	-	0.0408	-	-
36	Hexane, 1-(3-butenyloxy)	-	-	-	-	0.0461	-	-
37	1-Penten-3-ol	-	-	-	-	0.0107	-	-
38	Butyric acid, 2,2-	-	-	-	-	0.024	-	-

	dimethyl-, vinyl ester					7		
39	2,6-Octadiene, 2,4-dimethyl	-	-	-	-	0.6360	-	-
40	6,6-Dimethyl-1,3-heptadien-5-ol	-	-	-	-	0.0943	-	-
41	Ethanone, 1-cyclobutyl	-	-	-	-	0.0283	-	-
42	Vinyldimethyl(hydroxymethyl)silane	-	-	-	-	-	0.0172	-
43	Pentanal, 2,2-dimethyl	-	-	-	-	-	0.0215	-
44	1-Propen-2-ol, formate	-	-	-	-	-	0.0059	-
45	2-Methylheptanoic acid	-	-	-	-	-	0.0035	-
46	Propane, 2-methyl-2-nitro-	-	-	-	-	-	-	0.0404
47	Azetidine	-	-	-	-	-	-	0.0458
48	1-Heptene, 3-methoxy	-	-	-	-	-	-	0.0042



#### 4.4 Water quality test by using Inductive Couple Plasma (ICP)

Table 4.4: Concentration of heavy metals in hydrosols

<b>Elements</b>	<b>Concentration (ppm)</b>						
<b>Time of extraction process</b>	<b>12 hours</b>	<b>18 hours</b>	<b>24 hours</b>	<b>30 hours</b>	<b>36 hours</b>	<b>42 hours</b>	<b>48 hours</b>
<b>Arsenic (As)</b>	-0.0070	-0.0080	-0.0090	-0.0080	-0.0080	-0.3500	0.0005
<b>Magnesium (Mg)</b>	-0.0030	0.0030	-0.0001	0.0004	0.0060	0.1150	0.0070
<b>Ferum (Fe)</b>	0.0020	0.0020	0.0020	0.0010	0.0022	0.0020	0.0020
<b>Zinc (zn)</b>	0.0010	-0.0004	-0.0005	-0.0006	-0.0007	0.1200	0.0042
<b>Lead (Pb)</b>	0.0010	0.0020	0.0020	0.0005	0.0004	0.0340	0.0001
<b>Cadmium (Cd)</b>	-0.0006	-0.0006	-0.0005	-0.0006	-0.0005	-0.0100	-0.0006

Base on the results in table above, the concentration values of the heavy metals for all samples were very small value including negative values. Hence, it can be concluded that there were no presence of heavy metals in the hydrosols. So, the hydrosols were safe to be use in daily life and also as medical treatment.

## CHAPTER 5

### CONCLUSION AND RECOMMENDATIONS

As a conclusion, the aim of this work which was the characterization of Agarwood distil water had been successful achieved at different time interval by using three analysis which were Fourier Transforms Infrared (FTIR) analysis, gas chromatographic-mass spectrometer (GC-MS) and Inductive Couple Plasma (ICP). From FTIR analysis, the active functional groups of hydrosol that were found in this study were including O-H bond of hydrogen bonded alcohol or phenol, alkyne  $C\equiv C-C$  stretch, C-C bond of aromatic ring and also the presence of H-O-H bond from water. This work has revealed that the hydrosols from *Aquilaria malaccensis* consist of phenolic compounds. This was according to the presence of alcohol or phenols group in the Fourier Transforms Infrared (FTIR) analysis. The different extraction process time did not affect the pattern of FTIR spectrum. Next, by using gas chromatographic-mass spectrometer (GC-MS), we had successful identified about 48 total of chemical components in all samples of Agarwood distil water and the main compound that was found was 1,2,4,5-Tetrazine, 1,4-diethylhexahydro because it contained in almost samples. This compound is used in the treatment of diarrhoea and worm infections, and also used to treat the involuntary movements (chorea) of Huntington's disease or progressive brain disorder that causes uncontrolled movements, emotional problems, and loss of thinking ability. Lastly, we could observed that there were very small amount of concentrations of metals in the hydrosols and some were in negative values after testing them using Inductive Couple Plasma (ICP). It was considered that there were no any presence of metals in the Agarwood distils water. Hence, the Agarwood distil water were safe to be drink and could be used as the treatment. The recommendations should be conducted was to handle the solvent extraction carefully and use the suitable ratio of water to hexane.

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## **APPENDICES**



## APPENDIX A

### Manuscript of Characterization of Agarwood Distil Water Based on Time Interval

Nurul Nadia Binti Abdullah (Bachelor of Engineering (Hons.) Chemical and Bioprocess)

1

## Characterization of Agarwood Distil Water Based on Different Time Interval

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**Abstract**— Agarwood distil water or known as hydrosol is obtained by using hydrodistillation method. Agarwood distil water is widely used in medicine since it contains high antioxidant and antibacterial properties that will improve the human health. The aim of this study is to analyze the characterization of Agarwood distil water from *Aquilaria malaccensis* at different time interval by using Fourier Transforms Infrared (FTIR) spectroscopy and Gas Chromatography-Mass Spectrometer (GC-MS) and also make water quality test by using Inductive Couple Plasma (ICP). The functional groups has been successful identified which there were presence of same functional groups for each hydrosol samples at different production temperature such as alcohol group, alkene group, alkyne group and also aromatic ring alkane group. It showed that the different production temperature did not affect the functional groups. Then, there were about 48 chemical compounds of hydrosol have been successful identified and the different chemical compounds were found for the different time interval. In this work, 1,2,4,5-Tetrazine, 1,4-diethylhexahydro was the main compound found in hydrosol. Lastly, all the samples of these hydrosols did not contain any heavy metals and safe to be used as medical treatment. In conclusion, the hydrosol from hydrodistillation process contains 1,2,4,5-Tetrazine, 1,4-diethylhexahydro which is potentially used is the treatment of diarrhoea and worm infections, and also used to treat the involuntary movements (chorea) of Huntington's disease or progressive brain disorder that causes uncontrolled movements, emotional problems, and loss of thinking ability.

**Keywords**— Agarwood, hydrodistillation, *Aquilaria malaccensis*, agarospirol, jinkohol, jinkoh creosol, khusenol

### I. INTRODUCTION

Agarwood or commonly known as gaharu, aloeswood, jinkoh, oud, spaliocha and eaglewood is found in *Aquilaria* species which comes from family of *Thymelaeaceae*. *Aquilaria* is basically a woody plant which is inherent in Southeast Asia (Tetsuro Ito *et al.*, 2012). Agarwood has about 15 species and there are four species are found in Thailand which their specific names are *Aquilaria Crassna*, *Aquilaria Batilomil*, *Aquilaria Subintegra* and *Aquilaria Malaccense* (Ponpan Wetsitayaklung *et al.*, 2009). Generally, Agarwood has a few grades namely, A, B, C and D that is classified according to its physical properties, formation and unique scent. Agarwood is very highly valuable fragrant woods because it contained economically essential oil and hydrosols with its aromatic unique product (Nattawan Yosothana *et al.*, 2013). It is widely used in many industries including medicine, cosmetics, perfume and aromatherapy industries (Mohamed *et al.*, 2013). It also plays an important role in Chinese Traditional Medicine for obvious medicinal effects as a sedative and coemimative, and to relieve gastric problems, coughs, rheumatism and high fever (

Yangyang Liu *et al.*, 2013). There are three forms of Agarwood that have sold which are in form of pieces of heartwood, heartwood oil and heartwood powder. The heartwood is burned to produce the aromatic vapor in houses and also in shrines (N. Ismail *et al.*, 2016). The powder form of wood is used as incense and medicine. The figure below shows the whole plant of Agarwood, and the cutting of stem of one week, 6 months and 20 months old Agarwood.



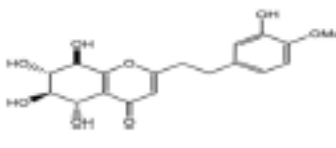
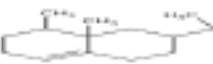


Fig.1: (A) Agarwood's Whole Plant, (B) One Week Agarwood, (C) 6 Months Agarwood, (D) 20 months Agarwood (Jersey Alam *et al.*, 2015)

The Agarwood distil water are very expensive because the production yield during extraction are low and increasing in international demand (Mohd Farid *et al.*, 2010). Basically, the good quality Agarwood's price can achieve to RM10,000 per kg based on the resinous wood's grade. Meanwhile for other qualities of Agarwood oil are sold about RM50 to RM200 per 12 g. The common methods that have been used to extract the oils and distil water from the plants are including hydrodistillation, supercritical fluid carbon dioxide extraction (SFE), steam distillation and solvent extraction (Muhammad Harewan *et al.*, 2013). The classical method that used to extract the Agarwood oil is by using hydrodistillation method. This method takes about 7-10 days and is guarantee safe to operate. Then, supercritical fluid carbon dioxide extraction (SFE) method is better than hydrodistillation method as it has high diffusivity, low viscosity, non-toxic, nonflammable, consume less energy, good transport properties and extraction, chemically stable and produce high yields.

Generally, the fragrance substances or aromatic of resin of Agarwood essence is belonging to the sesquiterpene and has particular chemical structure (Adi *et al.*, 2016). Sesquiterpenes can be classified as the main active constituents that have important function which is giving the scent, pleasant odor and unique aroma of agarwood (Yumi *et al.*, 2014). Sesquiterpene usually has properties of anti-allergy and anti-inflammatory. There are about 15 carbon atoms that contained in the sesquiterpenes and also have multifaceted pharmacological actions. This sesquiterpene not only found in Gaharu but also can be found in floral oils like rose and chamomile. The important chemical compounds that contained in Gaharu which contribute to the scent of Gaharu are agarospirol, jinkohol, jinkohol-creosol and khusenol.

**Table 1:** Chemical Compounds in Agarwood (Seri Chempaka Bt. Mohd. Yusof *et al.*, 2012)

Name of Chemical Compound	Chemical Structure
Agaropsinol	
Jinkohol	
Jinkohol-crenol	
Kusenol	

The objective of the current research was to identify the characterization of hydrosols at different time interval by using Fourier Transform Infrared (FTIR) spectroscopy, gas chromatographic-mass spectrometer (GC-MS) and Inductive Couple Plasma (ICP).

## II. METHODOLOGY

### A. Materials and Instruments

Agarwood of *Aquilaria malaccensis* species that used in this study were obtained from natural population of Kuala Krai, Kelantan in September 2017, distilled water, hexane, Fourier Transform Infrared (FTIR) spectroscopy (Perkin Elmer 2000 Model), gas chromatographic-mass spectrometer (GC-MS) model Varian 240-MS completes with the 450-GC with a Combi PAL autosampler from CTC Analytics, Inductive Couple Plasma (ICP) Spectrometer (ThermoFisher Scientific) with brand or model is iCAP 6000 series, 1 Liter of separation funnel, hydrodistillation (extraction facilities), grinder, 50 ml of beakers, 50 ml of measuring cylinder, dropper, 20ml of pipette, seven unit of 500 ml plastic bottles, volumetric flask and vial cronus clear 12 x 32mm.

### B. Preparation of Plant Materials

About 25 kg of dried Agarwood were ground by using grinder machine. The large trunk of Agarwood was chopped into the smaller size. This is done in order to obtain the maximum surface area for the process of extraction and also to give maximum contact time between the particle of Agarwood and the solvent.

### C. Hydrodistillation Method

In this study, the type of extraction method that has been used is hydrodistillation process. The hydrosol was extracted from *Aquilaria* sp of family Thymelaeaceae. About 25 kg dried Agarwood was extracted by using extraction facilities. The experiment was run continuously for 48 hours. Before the time of

extraction process was taken, the system was left for 15 minutes to equilibrium state. It is to ensure the process of extraction was conducted in stable condition.

By using hydrodistillation process, the material of plant which is Gaharu will be immersed in the water which is in heated still. This process was conducted under atmospheric pressure and a reduced pressure. The ratio of solid-to-water is usually 1:50 g/ml. is applied during this process. Then, the steam of water and essential oil will be produce and will leave the hot suspension. It will then condense, collected and is separated by using decantation. It will obtain two products which are essential oil and the hydrosol or floral water. This hydrosol is actually contained the constituents of the essential oil.

Hydrodistillation was conducted at various time starting with 12 hours, 18 hours, 24 hours, 30 hours, 36 hours, 42 hours and 48 hours to collect the gaharu distil water in a bottle. The extraction process was started at 6pm on 14<sup>th</sup> March 2018. About 40ml of gaharu distil water was collected in a bottle every 6 hours which is until 6am of 16<sup>th</sup> March 2018. The hydrodistillation process cannot be exceeding the temperature of 120°C to avoid the plant material from burning. The temperature of this extraction process is constant about 100°C.

During the distillation process, the vapor that consist of volatile compounds and steam were rise to the condenser from the extractor, where two phases of immiscible liquid are formed which are known as Agarwood oil and aromatic water (hydrosol). The oil of Agarwood which is lighter than hydrosol was separated from the hydrosol and located at the top of the separation funnel, meanwhile the hydrosol is formed below the Gaharu oil. 40ml of hydrosol was collected for every 6 hours in the 250ml of plastic bottle in order to analyze their chemical profiling and evaluate the quality of hydrosol.

### D. Phytochemical studies of hydrosol

The hydrosol that obtained from extraction of Agarwood (hydrodistillation) were tested in order to obtain the characterization or chemical profiling, functional groups and also to study the water quality. For the chemical profiling, it was analyzed by using gas chromatographic mass spectrometer (GC-MS), then for functional groups identification was using Fourier Transform Infrared (FTIR) spectroscopy. Meanwhile for water quality, Inductive Couple Plasma (ICP) was used to test whether there were the presence of heavy metals or not such as Arsenic (Ar), Magnesium (Mg), Ferum (Fe), Zinc (Zn), Lead (Pb) and Cadmium (Cd) at various production temperatures.

### E. Characterization of hydrosol by Fourier Transform Infrared (FTIR)

All hydrosols were characterized by FTIR spectroscopy (Perkin Elmer 2000 Model) to identify the active functional groups that presence in hydrosol. The FTIR study was carried out by using the Perkin Elmer System 2000 FTIR instrument. First, the transparent Pellets (thin disc) were formed by mixing 5mg of the sample with 100 mg of potassium bromide (KBr) (1:20) using a mould and press, and compressed under a pressure of 7 ton. The investigation was performed within the wavelength ranging from 4000 to 400 cm<sup>-1</sup> and the spectrum takes about three minutes to be recorded. The acquisition of the spectra and peaks assignment was performed using FTIR software Spectrum 3.02.01 (Perkin Elmer, Inc., Waltham, MA). Lastly, comparison between the resultant spectrum with the standard for entirely functional groups was conducted.

### F. Preparation of Sample Hydrosol in Hexane

About 1ml of hydrosol of each seven samples of hydrosols was filled up into the seven different beakers by using pipette. Then, 10



ml of hexane was pipetted into each beaker that contained hydrosol. The solution was shaking for 2 minutes to make sure the mixture was well mixed. After that, visually observe the solution if there was any layer formation. The two layers were formed which were hexane layer and hydrosol. Then, about 1 ml of the hexane solution's layer was transferred into the vial cronus clear 12 x 32mm by using dropper. Then, the solution was analyzed using GC-MS to analyze the chemical constituents in hydrosol.

#### G. Study of chemical compounds by Gas Chromatography Mass Transfer (GC-MS)

Hydrosol composition was studied by GC-MS analysis using a Hewlett Packard gas chromatograph (GC 5890) coupled with a mass selective detector (5972) (Hewlett Packard, Palo Alto, USA). Separation of the analytes by gas chromatography was carried out using a silica capillary column (30 m length, 0.25 mm diameter, 0.25 mm film thickness) of HP-5MS (Hewlett Packard). Separation of the compounds involved injection of 1.0 µL of the hydrosol into the front inlet of the gas chromatograph operating at 250°C in the splitless mode. The flow rate of the carrier gas, helium, was 2.0 mL/min with a 1:50 split ratio. The oven program commenced at 80°C, where it was held for 2 min and then increased at a rate of 10°C/min to 250°C, where it was held for 10 min. The interface temperature was 250°C. Ionization of the analytes by electron impact (EI) was obtained using an emission current of 70 eV. The ion source temperature was set at 250°C and the scan scope was set from 32 to 500 amu. The compounds were characterized by database matching and comparison of their MS spectra with existing data in the Wiley and Adams library search data.

#### H. Water quality test for heavy metal by using Inductive Couple Plasma (ICP)

The Agarwood distil water was undergo heavy metal test which were analyzed by using Inductive Couple Plasma (ICP) system (ThermoFisher Scientific) with brand or model is iCAP 6000 series. This test was conducted to make sure whether there was present of heavy metal or not in the Agarwood distil water. We must make sure that the results are not containing any heavy metals in this hydrosol because those elements can give a risk to human health if they use this hydrosol.

### III. RESULTS AND DISCUSSION

#### A. Hydrodistillation Extraction

By using hydrodistillation method, the 25 kg dried Agarwood could gave about 1 litre of Agarwood distil water or known as hydrosol. This extraction process of hydrosol was run continuously about 48 hours. However, in this study, the hydrosols were collected at various time of extraction process starting with 12 hours, 18 hours, 24 hours, 30 hours, 36 hours, 42 hours and 48 hours by collecting hydrosols in the bottles. The extraction process was started at 6pm on 14<sup>th</sup> March 2018. About 40ml of hydrosol was collected in each bottle every 6 hours which is until 6am of 16<sup>th</sup> March 2018. The samples of hydrosols have been collected about 7 bottles with different time of extraction process. All samples were clear in color like normal water. All these samples were analyzed by using Fourier Transform Infrared (FTIR), Gas Chromatography-Mass Spectrophotometer (GC-MS) and Inductive Couple Plasma (ICP).

#### B. Characterization of hydrosol by Fourier Transform Infrared (FTIR)

All the seven samples of hydrosols were characterized by using FTIR. By using FTIR analysis, the characteristic of the absorption peak such as scope and location of hydrosols chemical contents or the functional groups could be identified. About a few drops of

each sample of hydrosols were used during FTIR analysis. The infrared spectra of all the samples of hydrosols were taken and evaluated. All the spectrums did not present major changes of peaks for Agarwood distil water at various extraction process. Figures 2 (a-g) show the IR spectrum of all the samples hydrosols that were collected respectively.

Base on the result, O-H bond of hydrogen bonded alcohol or phenol (3600-3200 cm<sup>-1</sup>) group frequency was present in all sample of hydrosols which at different time extraction process with frequency of 3309.49 cm<sup>-1</sup>, 3309.67 cm<sup>-1</sup>, 3309.02 cm<sup>-1</sup>, 3309.68 cm<sup>-1</sup>, 3309.74 cm<sup>-1</sup>, 3308.97 cm<sup>-1</sup> and 3308.68 cm<sup>-1</sup> respectively. A broad spectrum can be detected from the figures below that denote the availability O-H bond. This is the very important part because the presence of O-H bond indicates the existence of phenolic compound in the hydrosols. This finding was considered successful because it was same as proposed by Khalil et al., (2013) where they had identified the presence of alcohol or phenol functional group in Agarwood or hydrosol.

Then, the band of most prominent in alkynes resembles to the carbon-carbon triple bond. The alkyne C≡C stretch (2260-2100 cm<sup>-1</sup>) group frequency was identified in all samples of hydrosols with frequency 2101.49 cm<sup>-1</sup>, 2155.24cm<sup>-1</sup>, 2116.73cm<sup>-1</sup>, 2134.91 cm<sup>-1</sup>, 2116.25cm<sup>-1</sup>, 2067.66cm<sup>-1</sup> and 2115.13cm<sup>-1</sup> respectively. There was very little organic compounds show an absorption in this region. All these frequencies illustrate as a sharp and weak band at around 2100 cm<sup>-1</sup>. The band was weak because of the triple bond that was not very polar. In certain cases, such as for the highly symmetrical alkynes, it might not represent at all because of the polarity of the triple bond was low linked with those alkynes.

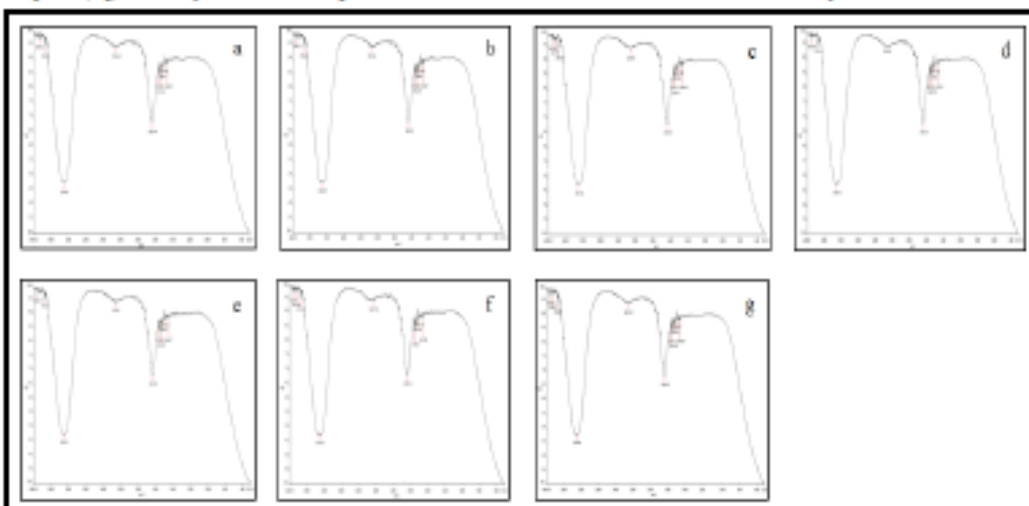
Next, H-O-H bond (1640-1630cm<sup>-1</sup>) group frequency was also present in all samples of hydrosols at frequency of 1636.49 cm<sup>-1</sup>, 1636.36 cm<sup>-1</sup>, 1636.44 cm<sup>-1</sup>, 1636.36 cm<sup>-1</sup>, 1636.40 cm<sup>-1</sup>, 1636.50 cm<sup>-1</sup> and 1636.45 cm<sup>-1</sup> respectively. All the samples contained H-O-H bond from water since the Agarwood hydrosol itself is a homogenous mixture of water and Agarwood sesquiterpenoids. From this evidence, it is expected that all these samples of hydrosols have majority of Agarwood sesquiterpenoids.

Last but not least, C-C bond of aromatic ring (1500-1400 cm<sup>-1</sup>) group frequency was identified in all samples of hydrosols with frequency at 1497.69 cm<sup>-1</sup>, 1497.90 cm<sup>-1</sup>, 1497.95 cm<sup>-1</sup>, 1497.88 cm<sup>-1</sup>, 1497.90 cm<sup>-1</sup>, 1497.98 cm<sup>-1</sup> and 1498.07 cm<sup>-1</sup> respectively. This aromatic ring was synonym with the characteristic of hydrosol that gave out the distinctive Agarwood aroma. Table 2 shows the functional groups of hydrosols that obtained from this study.

**Table 2:** Functional groups of hydrosols from *Agarwood malaccensis*

Band assignment	Functional Group	Group band (cm <sup>-1</sup> )
O-H	Alcohol/Phenol	3600-3200
In ring C-C stretches	Aromatic Ring Alkene	1500-1400
C≡C-C Stretch	Alkyne	2260-2110
H-O-H	Alkene	1640-1630

Figure 2 (a-g) show the plots of FTIR for Agarwood distil water that has been extracted at various time or temperature.



**Fig.2:** FTIR Spectrum for Agarwood distil water (a) For 12 hours extraction process, (b) For 18 hours extraction process, (c) For 24 hours extraction process, (d) For 30 hours extraction process, (e) For 36 hours extraction process, (f) For 42 hours extraction process, (g) For 48 hours extraction process

Table3 shows the summary of characteristic peaks bands on FTIR spectra for all the sample hydrosols at various time of extraction process.

Table 3: Summary of characteristic peaks bands on FTIR spectra

Functional groups (bands)	Wavenumber (cm <sup>-1</sup> )						
Time of extraction process	12 hours	18 hours	24 hours	30 hours	36 hours	42 hours	48 hours
-OH stretch	3309.49	3309.67	3309.02	3309.68	3309.74	3308.97	3308.68
C≡C-C stretch	2101.49	2155.24	2116.73	2134.91	2116.25	2067.66	2115.13
H-O-H water	1636.49	1636.36	1636.44	1636.36	1636.40	1636.50	1636.45
C-C aromatic stretching	1497.69	1497.90	1497.95	1497.88	1497.90	1497.98	1498.07

As a conclusion, it was found that all samples of hydrosols from various extraction time or temperature consists the alcohol or phenols functional groups that were very useful in order to prove the presence of phenolic compounds inside the hydrosols. Then, the presence of H-O-H bond from water since the Agarwood hydrosol itself was a homogenous mixture of water and Agarwood sesquiterpenoids. Next, the presence of C-C bond of aromatic ring was very important because that aromatic ring gave the pleasant smell of hydrosol. Besides, the patterns of FTIR spectrum for all the samples were quite similar in which the entire spectrum did not show significance change of peaks. So, it means that the different time of extraction process or different production temperature did not affect the pattern of FTIR spectrum because all of the samples hydrosols had almost same functional groups. The spectrum of all samples of Agarwood hydrosols were recorded at range of 4000 – 400 cm<sup>-1</sup> (mid infrared spectroscopy) at 4 cm<sup>-1</sup> resolution (FTIR model: Nicolet Avatar 370 DTGS).

### C. Characterization of hydrosol by Gas Chromatography Mass Transfer (GC-MS)

GCMS was used to detect the chemical contents in hydrosol. Basically, all of the Agarwood were complex mixtures of sesquiterpene hydrocarbons, sesquiterpene alcohols, aromatic compounds, and aliphatic hydrocarbons that very difficult to be identified based on MS alone where the chemical compounds of Agarwood should be identified by comparing the mass spectral

data with the existing Wiley library and reference library spectral data (Nor Anah M.A et al., 2008).

In this study, seven hydrosols samples of different time process extraction were identified by GC-MS analysis. The chemical compounds that were detected in this hydrosol by GC-MS instruments were 48 in total as listed in table 4.3. According to the result, it was found that the chemical component that was present in almost samples was 1,2,4,5-Tetrazone, 1,4-diethylhexahydro. This showed that 1,2,4,5-Tetrazone, 1,4-diethylhexahydro was the main compounds found in hydrosol at different time interval. 1,2,4,5-Tetrazone, 1,4-diethylhexahydro formula was C<sub>10</sub>H<sub>16</sub>N<sub>4</sub> with molecular weight of 144.21804 g/mol. This compound is used in the treatment of *dianthos* and worm infections, and also used to treat the involuntary movements (chorea) of Huntington's disease or progressive brain disorder that causes uncontrolled movements, emotional problems, and loss of thinking ability.

From the results, it indicates that there were some differences and variations in the chemical composition of hydrosol at different time. According to Jutarut Pornpunyapatt et al., (2011), the different extraction process time will effect on the chemical compounds of hydrosol. The higher number of chemical components should be result at longer extraction time. So, this study was similar to the findings by Jutarut Pornpunyapatt et al., (2011) because the results showed the differences of chemical contents in all samples at different production time. Meanwhile, for the number of chemical components, the result showed that the



number of chemical component in hydrosol was increased from hour 12 until hour 36 which were from 8 to 15 of chemical components. Then it was decreased at hour 42 and hour 48. The decreasing in number of chemical components means that there was something wrong because it should be the higher number of chemical compounds when the production time was longer.

This failure may cause by the incorrect preparation of sample hydrosol in hexane. In this study, the solvent extraction had been conducted before the analysis of GC-MS was done. The reason of conducting this solvent extraction was because of the sample of this hydrosol was water based solution, so that it should undergo the solvent extraction which was by using the hexane as a solvent

in order to give a better sensitivity for the GC-MS test. It is because GC-MS instrument could not handle the sample from the water base solution and it also had a potential to cause the damage of column. It should inject the extracted sample in the GC-MS because it could not detect the chemical component from the water based. However, this step of solvent extraction also did not give the best result. It might cause of the ratio of sample to hexane were not suitable. The ratio of sample to hexane that was used in this study was 1:10. It might be the solvent could not extract the ion in sample properly because the content of sample was too little. Hence, another step should be done in order to get the most accurate results. Table 4 shows the chemical compounds of the all samples.

Table 4: Compounds Identified From GC-MS Analysis of Hydrosol

No	Compounds	Chromatogram Area (%)							
		Time of extraction process	12 hours	18 hours	24 hours	30 hours	36 hours	42 hours	48 hours
1	Pentane, 3-ethyl-2,2-dimethyl		0.2120	-	-	0.3690	-	-	-
2	2-Octanol, 8,8-dimethoxy-2,6-dimethyl		0.0466	-	-	-	-	-	-
3	1,2,4,5-Tetrazine, 1,4-diethylhexahydro		0.0459	-	0.0612	0.0967	0.0665	-	0.1140
4	Allyldimethyl(vinyl)silane		0.0057	0.0352	0.0284	0.0077	-	0.0050	0.0146
5	2,2-Dimethylpropanoic anhydride		0.0046	-	-	-	-	-	-
6	3H-1,2,4-Triazol-3-one, 1,2-dihydro		0.0056	0.0058	-	0.0102	-	0.0050	0.0098
7	Oxalic acid, cyclohexyl propyl ester		0.1760	-	-	-	-	-	-
8	Oxalic acid, butyl cyclobutyl ester		0.0213	-	-	-	-	-	-
9	2-Isopropyl-3-vinylloxane		-	0.6600	0.2570	-	-	-	0.6870
10	4-Methoxypyrimido[2,3-d]pyrimidine		-	0.0056	-	-	-	-	-
11	Thiazole		-	0.0040	-	-	-	-	-
12	1H-Tetrazole, 1-methyl		-	0.0094	-	-	-	-	-
13	2-Methyl-1,5-hexadiene-3-ol		-	0.0029	-	-	-	-	-
14	Acetamide, N-(2-hydroxyethyl)		-	0.0490	-	-	-	-	-
15	Silicon tetrafluoride		-	0.0019	-	-	-	-	-
16	Oxalic acid, cyclohexyl hexyl ester		-	-	-	-	-	-	0.2520
17	Nonane, 2,2,3-trimethyl		-	-	1.1600	-	-	-	-
18	2-Propenal		-	-	0.0159	-	-	-	-
19	1-Pentanol, 4-methyl		-	-	0.0161	-	-	-	-
20	Cyclobutanecarboxylic acid, 2-propenyl ester		-	-	0.3480	0.1580	-	0.3900	-
21	Butane, 1-chloro		-	-	0.0187	-	-	-	-
22	(Trimethylsilyl)acetylene		-	-	0.0138	-	-	-	-
23	1-Hexene, 4-methyl		-	-	-	0.6060	0.6240	0.2250	-
24	Trimethylaluminum		-	-	-	0.0003	-	-	-
25	Thiopivalic acid		-	-	-	0.0527	-	-	0.0640
26	6-Heptene-2,4-diol		-	-	-	0.0361	-	-	-
27	Hexanoic acid, 2-oxo-, methyl ester		-	-	-	0.0085	0.0182	-	0.0090
28	2-Pentanone, 5,5'-oxybis		-	-	-	0.0606	-	-	-
29	Cyclopropane, 2-bromo-1,1,3-trimethyl		-	-	-	0.3430	0.3860	-	-
30	5-Nonanone		-	-	-	0.0181	-	-	-
31	Decane, 2,2,3-trimethyl		-	-	-	-	0.3130	-	-
32	4,8-Dioxatricyclo[5.1.0.0(3,5)]octane, 1-methyl-5-(1-methylethyl)		-	-	-	-	0.0008	-	-
33	Cyclopropanecarboxylic acid, but-3-yn-2-yl ester		-	-	-	-	0.0048	-	-
34	2-Pentanol, 5-methoxy-2-methyl		-	-	-	-	0.0805	-	-
35	8-Methyl 3-methylbutanethioate		-	-	-	-	0.0408	-	-
36	Hexane, 1-(3-butenyloxy)		-	-	-	-	0.0461	-	-
37	1-Penten-3-ol		-	-	-	-	0.0107	-	-
38	Butyric acid, 2,2-dimethyl-, vinyl ester		-	-	-	-	0.0247	-	-
39	2,6-Octadiene, 2,4-dimethyl		-	-	-	-	0.6360	-	-
40	6,6-Dimethyl-1,3-heptadien-5-ol		-	-	-	-	0.0943	-	-
41	Ethanoic acid, 1-cyclobutyl		-	-	-	-	0.0283	-	-
42	Vinyl dimethyl(hydroxymethyl)silane		-	-	-	-	-	0.0172	-
43	Pentanal, 2,2-dimethyl		-	-	-	-	-	0.0215	-
44	1-Propen-2-ol, formate		-	-	-	-	-	0.0059	-
45	2-Methylheptanoic acid		-	-	-	-	-	0.0035	-
46	Propane, 2-methyl-2-nitro-		-	-	-	-	-	-	0.0404
47	Azetidine		-	-	-	-	-	-	0.0458
48	1-Hexene, 3-methoxy		-	-	-	-	-	-	0.0042

#### D. Water quality test by using Inductive Couple Plasma (ICP)

By using Inductive Couple Plasma (ICP), the result was obtained as shown in table 5.

Table 5: Concentration Values of The Metals

Elements	Amount (mg/L)
Arsenic (As)	ND
Magnesium (Mg)	ND
Iron (Fe)	ND
Zinc (zn)	ND
Lead (Pb)	ND
Cadmium (Cd)	ND

Base on the results in table above, the concentration values of the metals (As, Mg, Fe, Zn, Pb and Cd) for all samples could not be detected. Hence, it can be concluded that there were no presence of heavy metals in the hydrosols. So, the hydrosols were safe to be use in daily life and also as medical treatment.

#### IV. CONCLUSION

As a conclusion, the aim of this work which was the characterization of Agarwood distill water had been successful achieved at different time interval by using three analysis which were Fourier Transform Infrared (FTIR) analysis, gas chromatographic-mass spectrometer (GC-MS) and Inductive Couple Plasma (ICP). From FTIR analysis, the active functional groups of hydrosol that were found in this study were including O-H bond of hydrogen bonded alcohol or phenol, alkyne C≡C stretch, C-C bond of aromatic ring and also the presence of H-O-H bond from water. This work has revealed that the hydrosols from *Aquilaria malaccensis* consist of phenolic compounds. This was according to the presence of alcohol or phenols group in the Fourier Transform Infrared (FTIR) analysis. The different extraction process time did not affect the pattern of FTIR spectrum. Next, by using gas chromatographic-mass spectrometer (GC-MS), we had successful identified about 48 total of chemical components in all samples of Agarwood distill water and the main compound that was found was 1,2,4,5-Tetrazine, 1,4-dihydropyridine because it contained in almost samples. This compound is used in the treatment of diabetes and worm infections, and also used to treat the involuntary movements (chorea) of Huntington's disease or progressive brain disorder that causes uncontrolled movements, emotional problems, and loss of thinking ability. Lastly, by using Inductive Couple Plasma (ICP), the concentration values of the metals (As, Mg, Fe, Zn, Pb and Cd) for all samples could not be detected. Hence, it can be concluded that there were no presence of heavy metals in the hydrosols. So, the hydrosols were safe to be use in daily life and also as medical treatment.

#### ACKNOWLEDGMENT

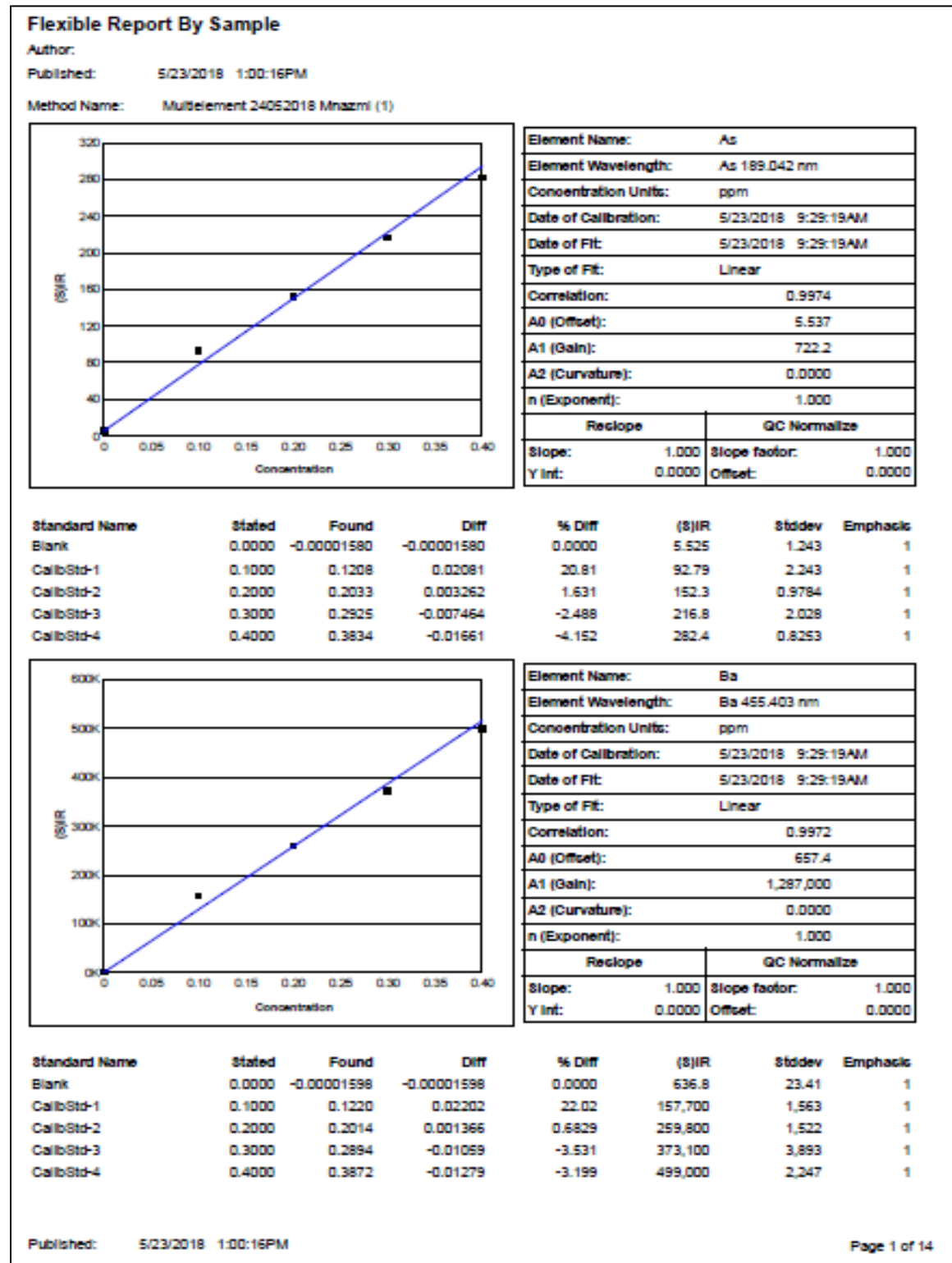
Thank you to my supervisor, Prof Dr Ku Halim Ku Hamid, special gratitude to Universiti Teknologi Mara, UTM Shah Alam, specifically to Faculty of Chemical Engineering for allowing the usage of facilities. Then, many thanks to all who have been directly and indirectly help through the work.

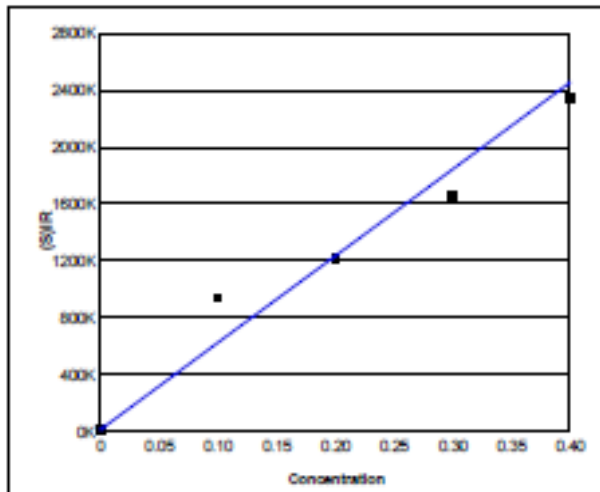
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## APPENDIX B

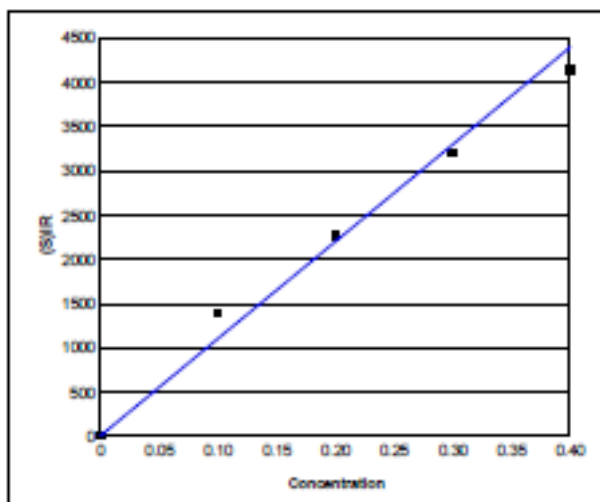
### Result by Using ICP Analysis





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Concentration Units:	ppm		
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Date of Fit:	5/23/2018 9:29:19AM		
Type of Fit:	Linear		
Correlation:	0.9853		
A0 (Offset):	14,520		
A1 (Gain):	6,105,000		
A2 (Curvature):	0.0000		
n (Exponent):	1.000		
	Slope	QC Normalize	
Slope:	1.000	Slope factor:	1.000
Y Int:	0.0000	Offset:	0.0000

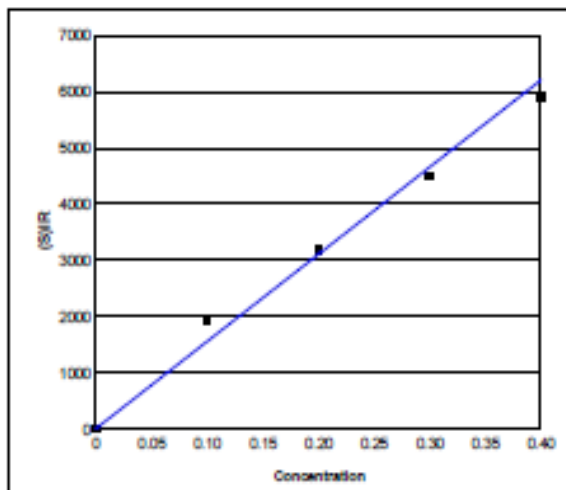
Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphacis
Blank	0.0000	-0.00003518	-0.00003518	0.0000	14,300	188.7	1
CalibStd-1	0.1000	0.1512	0.05116	51.16	937,300	1,814	1
CalibStd-2	0.2000	0.1973	-0.002672	-1.336	1,219,000	11,290	1
CalibStd-3	0.3000	0.2697	-0.03029	-10.10	1,661,000	7,116	1
CalibStd-4	0.4000	0.3818	-0.01820	-4.549	2,345,000	28,880	1



Element Name:	Cd		
Element Wavelength:	Cd 228.802 nm		
Concentration Units:	ppm		
Date of Calibration:	5/23/2018 9:29:19AM		
Date of Fit:	5/23/2018 9:29:19AM		
Type of Fit:	Linear		
Correlation:	0.9958		
A0 (Offset):	7.314		
A1 (Gain):	10,980		
A2 (Curvature):	0.0000		
n (Exponent):	1.000		
Reclipse		QC Normalize	
Slope:	1.000	Slope factor:	1.000
Y Int:	0.0000	Offset:	0.0000

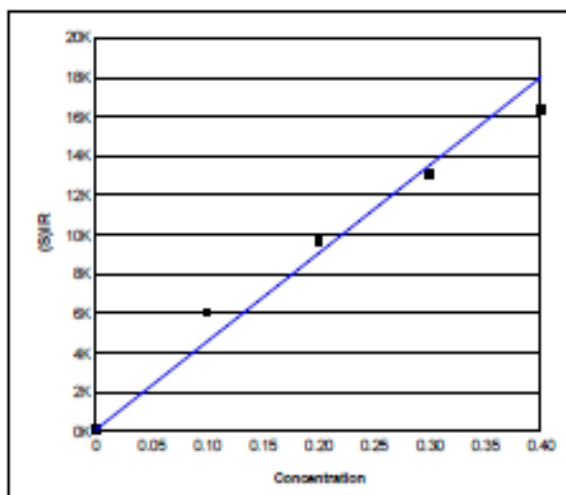
Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphack
Blank	0.0000	-0.00002012	-0.00002012	0.0000	7.093	0.4166	1
CalibStd-1	0.1000	0.1258	0.02576	25.76	1.388	3.837	1
CalibStd-2	0.2000	0.2061	0.006091	3.045	2.270	6.570	1
CalibStd-3	0.3000	0.2913	-0.008678	-2.893	3.206	10.57	1
CalibStd-4	0.4000	0.3768	-0.02317	-5.792	4.145	10.42	1





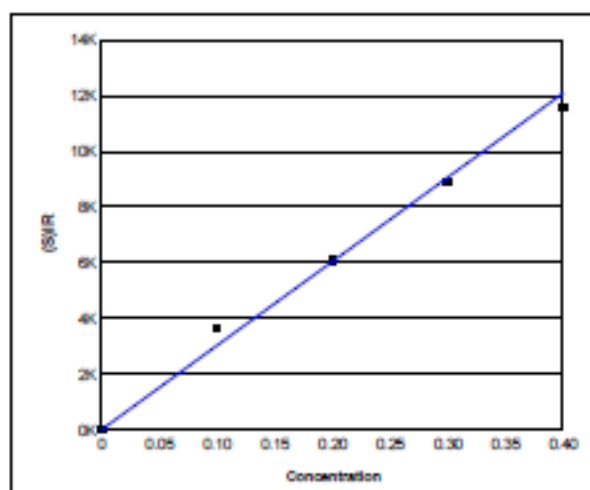
Element Name:		Co
Element Wavelength:		Co 228.616 nm
Concentration Units:		ppm
Date of Calibration:		5/23/2018 9:29:19AM
Date of Fit:		5/23/2018 9:29:19AM
Type of Fit:		Linear
Correlation:		0.9963
A0 (Offset):		9.015
A1 (Gain):		15,500
A2 (Curvature):		0.0000
n (Exponent):		1.000
Reslope		QC Normalize
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Y Int:	0.0000	Offset: 0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphatic
Blank	0.0000	-0.00001879	-0.00001879	0.0000	8.723	0.4409	1
CalbStd-1	0.1000	0.1246	0.02464	24.64	1,941	4.147	1
CalbStd-2	0.2000	0.2044	0.004404	2.202	3,178	2.839	1
CalbStd-3	0.3000	0.2905	-0.009482	-3.161	4,513	20.10	1
CalbStd-4	0.4000	0.3804	-0.01957	-4.892	5,907	10.04	1



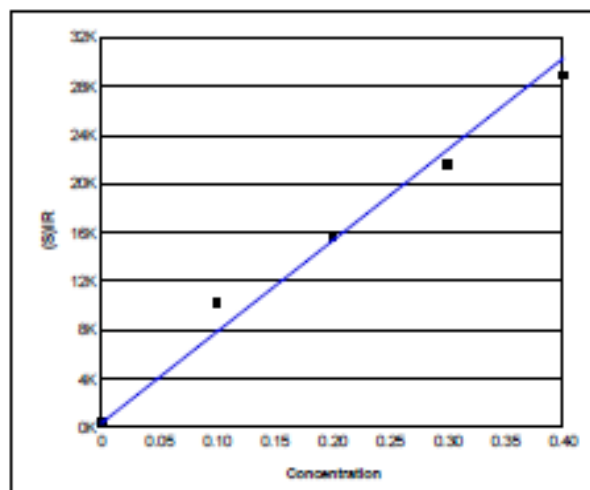
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Element Wavelength:		Cu 324.754 nm
Concentration Units:		ppm
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Date of Fit:		5/23/2018 9:29:19AM
Type of Fit:		Linear
Correlation:		0.9925
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A1 (Gain):		44,790
A2 (Curvature):		0.0000
n (Exponent):		1.000
Reslope		QC Normalize
Slope:	1.000	Slope factor: 1.000
Y Int:	0.0000	Offset: 0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphatic
Blank	0.0000	-0.00002706	-0.00002706	0.0000	100.4	2.083	1
CalbStd-1	0.1000	0.1327	0.03273	32.73	6,046	49.45	1
CalbStd-2	0.2000	0.2132	0.01322	6.608	9,651	106.8	1
CalbStd-3	0.3000	0.2905	-0.009464	-3.155	13,110	179.8	1
CalbStd-4	0.4000	0.3635	-0.03648	-9.119	16,380	273.3	1



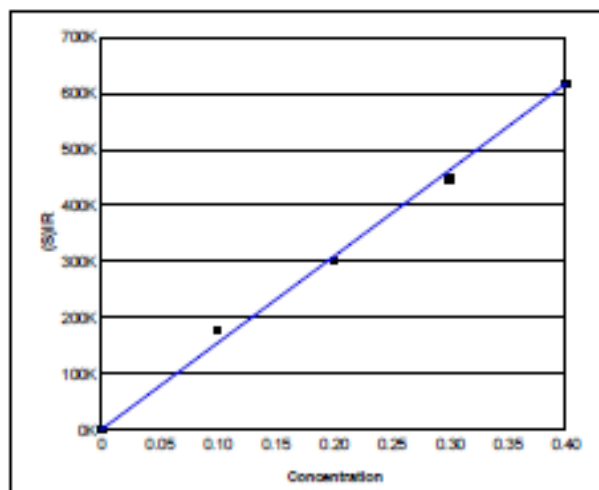
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Element Wavelength:	Fe 259.940 nm		
Concentration Units:	ppm		
Date of Calibration:	5/23/2018 9:29:19AM		
Date of Fit:	5/23/2018 9:29:19AM		
Type of Fit:	Linear		
Correlation:	0.9973		
A0 (Offset):	4.970		
A1 (Gain):	30,220		
A2 (Curvature):	0.0000		
n (Exponent):	1.000		
Reslope		QC Normalize	
Slope:	1.000	Slope factor:	1.000
Y Int:	0.0000	Offset:	0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.00001587	-0.00001587	0.0000	4.490	1.824	1
CalbStd-1	0.1000	0.1213	0.02135	21.35	3,673	8.061	1
CalbStd-2	0.2000	0.2015	0.001539	0.7695	6,096	10.83	1
CalbStd-3	0.3000	0.2937	-0.006299	-2.100	8,882	19.60	1
CalbStd-4	0.4000	0.3834	-0.01659	-4.147	11,590	21.95	1



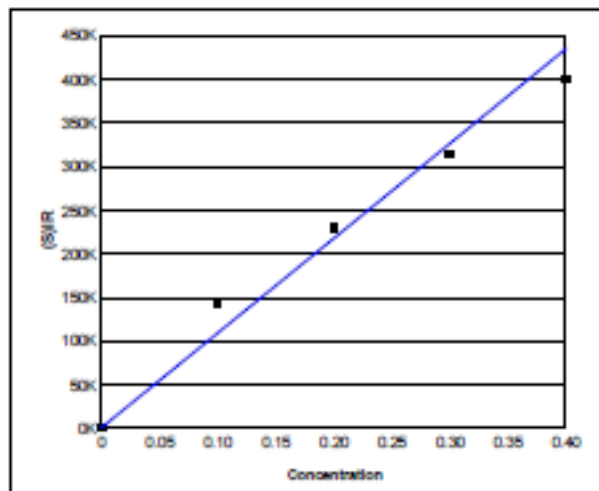
Element Name:	K
Element Wavelength:	K 766.490 nm
Concentration Units:	ppm
Date of Calibration:	5/23/2018 9:29:19AM
Date of Fit:	5/23/2018 9:29:19AM
Type of Fit:	Linear
Correlation:	0.9941
A0 (Offset):	370.5
A1 (Gain):	74,860
A2 (Curvature):	0.0000
n (Exponent):	1.000
Reslope	QC Normalize
Slope:	1.000
Y Int:	0.0000
Slope factor:	1.000
Offset:	0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.00002332	-0.00002332	0.0000	368.7	14.34	1
CalbStd-1	0.1000	0.1314	0.03142	31.42	10,210	52.03	1
CalbStd-2	0.2000	0.2047	0.004694	2.347	15,690	203.5	1
CalbStd-3	0.3000	0.2830	-0.01704	-5.681	21,550	163.1	1
CalbStd-4	0.4000	0.3809	-0.01907	-4.767	28,890	366.8	1



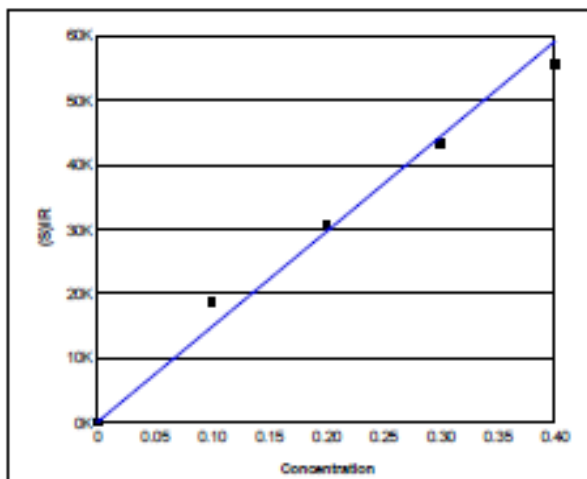
Element Name:	Li
Element Wavelength:	Li 670.784 nm
Concentration Units:	ppm
Date of Calibration:	5/23/2018 9:29:19AM
Date of Fit:	5/23/2018 9:29:19AM
Type of Fit:	Linear
Correlation:	0.9987
A0 (Offset):	781.0
A1 (Gain):	1,543,000
A2 (Curvature):	0.0000
n (Exponent):	1.000
Reslope	
QC Normalize	
Slope:	1.000
Y Int:	0.0000
Slope factor:	1.000
Offset:	0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.000009353	-0.000009353	0.0000	766.6	32.16	1
CalibStd-1	0.1000	0.1149	0.01493	14.93	178,200	1,571	1
CalibStd-2	0.2000	0.1959	-0.004057	-2.028	303,200	2,065	1
CalibStd-3	0.3000	0.2900	-0.009983	-3.328	448,400	534.3	1
CalibStd-4	0.4000	0.3991	-0.0008927	-0.2232	616,800	931.2	1



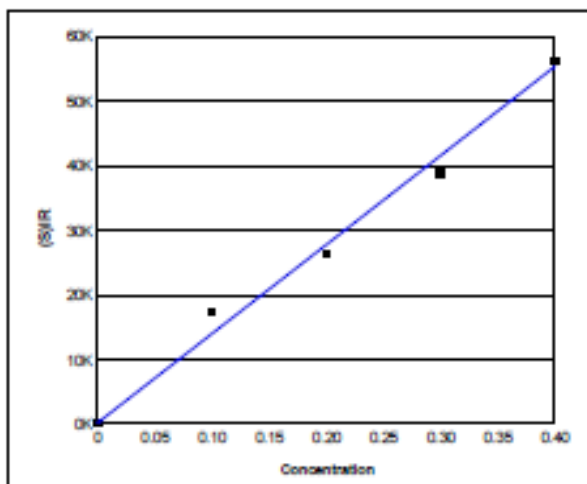
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Concentration Units:	ppm
Date of Calibration:	5/23/2018 9:29:19AM
Date of Fit:	5/23/2018 9:29:19AM
Type of Fit:	Linear
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A0 (Offset):	991.1
A1 (Gain):	1,086,000
A2 (Curvature):	0.0000
n (Exponent):	1.000
Reslope	
QC Normalize	
Slope:	1.000
Y Int:	0.0000
Slope factor:	1.000
Offset:	0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.00002532	-0.00002532	0.0000	963.6	21.97	1
CalibStd-1	0.1000	0.1312	0.03121	31.21	143,500	757.4	1
CalibStd-2	0.2000	0.2113	0.01125	5.625	230,400	1,098	1
CalibStd-3	0.3000	0.2892	-0.01075	-3.585	315,100	3,996	1
CalibStd-4	0.4000	0.3683	-0.03170	-7.926	400,900	5,132	1



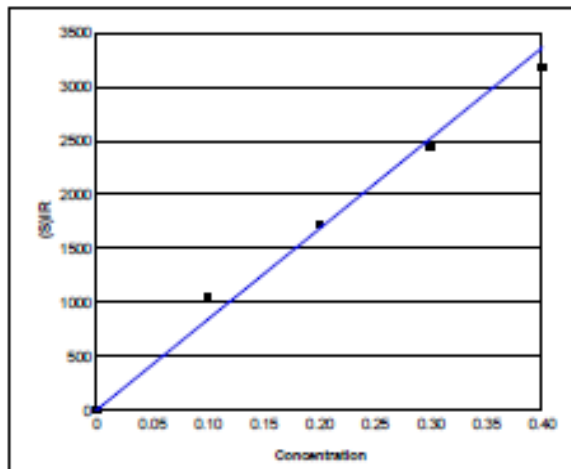
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Date of Fit:	5/23/2018 9:29:19AM
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A2 (Curvature):	0.0000
n (Exponent):	1.000
Reslope	QC Normalize
Slope:	1.000
Y Int:	0.0000
Slope factor:	1.000
Offset:	0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.00002022	-0.00002022	0.0000	88.42	0.6887	1
CalibStd-1	0.1000	0.1257	0.02568	25.68	18,650	50.99	1
CalibStd-2	0.2000	0.2065	0.006484	3.242	30,580	85.71	1
CalibStd-3	0.3000	0.2921	-0.007927	-2.642	43,220	216.2	1
CalibStd-4	0.4000	0.3758	-0.02424	-6.059	55,580	37.17	1



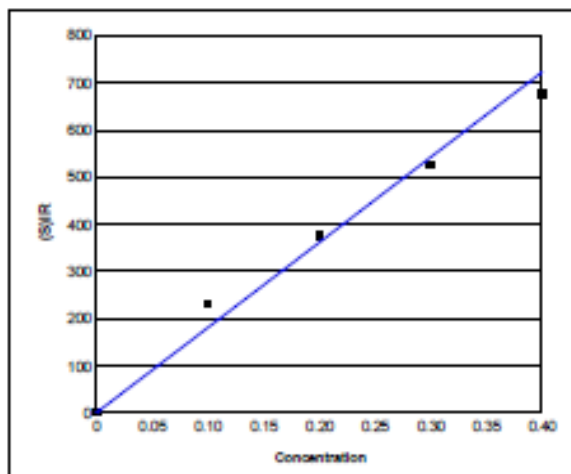
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Concentration Units:	ppm
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Date of Fit:	5/23/2018 9:29:19AM
Type of Fit:	Linear
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A1 (Gain):	138,200
A2 (Curvature):	0.0000
n (Exponent):	1.000
Reslope	QC Normalize
Slope:	1.000
Y Int:	0.0000
Slope factor:	1.000
Offset:	0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.00001364	-0.00001364	0.0000	172.8	2.215	1
CalibStd-1	0.1000	0.1237	0.02368	23.68	17,270	300.6	1
CalibStd-2	0.2000	0.1900	-0.009990	-4.995	26,430	259.5	1
CalibStd-3	0.3000	0.2805	-0.01948	-6.492	38,940	715.3	1
CalibStd-4	0.4000	0.4058	0.005791	1.448	56,250	520.0	1



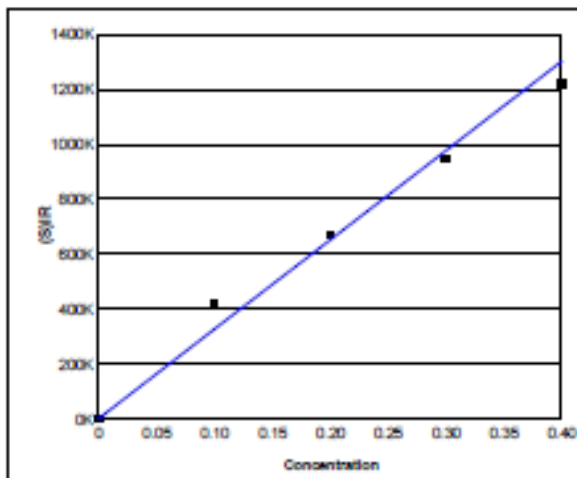
Element Name:		Ni
Element Wavelength:		Ni 221.647 nm
Concentration Units:		ppm
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Date of Fit:		5/23/2018 9:29:19AM
Type of Fit:		Linear
Correlation:		0.9961
A0 (Offset):		2.714
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A2 (Curvature):		0.0000
n (Exponent):		1.000
Reclope		QC Normalize
Slope:	1.000	Slope factor: 1.000
Y Int:	0.0000	Offset: 0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.00001928	-0.00001928	0.0000	2.552	0.8921	1
CalibStd-1	0.1000	0.1248	0.02480	24.80	1,051	1.818	1
CalibStd-2	0.2000	0.2055	0.005516	2.758	1,729	4.191	1
CalibStd-3	0.3000	0.2916	-0.008441	-2.814	2,452	4.911	1
CalibStd-4	0.4000	0.3781	-0.02188	-5.470	3,180	5.497	1



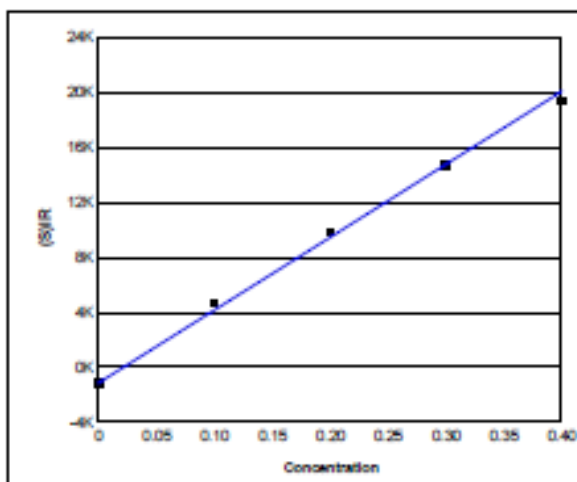
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Element Wavelength:		Pb 220.353 nm
Concentration Units:		ppm
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Date of Fit:		5/23/2018 9:29:19AM
Type of Fit:		Linear
Correlation:		0.9952
A0 (Offset):		0.7611
A1 (Gain):		1,806
A2 (Curvature):		0.0000
n (Exponent):		1.000
Reclope		QC Normalize
Slope:	1.000	Slope factor: 1.000
Y Int:	0.0000	Offset: 0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.00002160	-0.00002160	0.0000	0.7221	0.9130	1
CalibStd-1	0.1000	0.1272	0.02719	27.19	230.5	1.056	1
CalibStd-2	0.2000	0.2079	0.007851	3.925	376.2	1.957	1
CalibStd-3	0.3000	0.2909	-0.009090	-3.030	526.2	1.334	1
CalibStd-4	0.4000	0.3740	-0.02595	-6.488	676.3	1.015	1



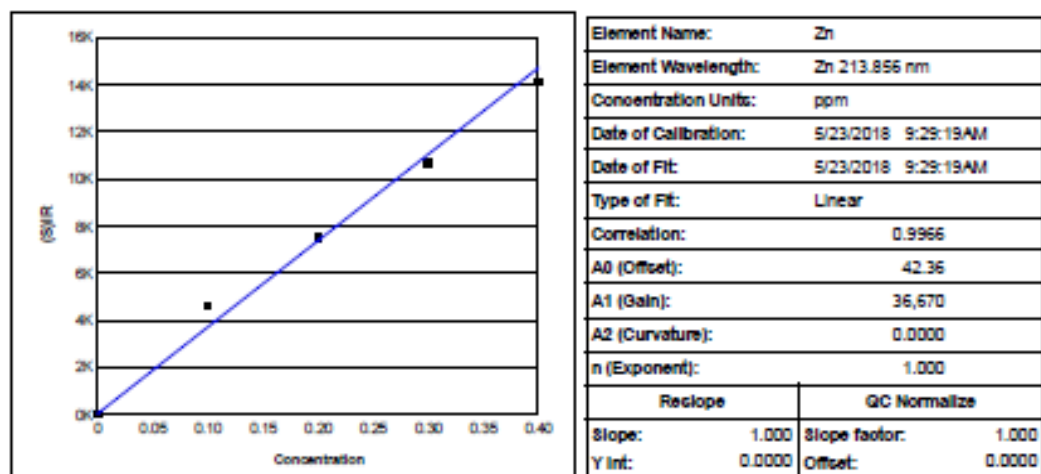
Element Name:	Sr
Element Wavelength:	Sr 407.771 nm
Concentration Units:	ppm
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Date of Fit:	5/23/2018 9:29:19AM
Type of Fit:	Linear
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A0 (Offset):	1,975
A1 (Gain):	3,252,000
A2 (Curvature):	0.0000
n (Exponent):	1.000
Reslope	
QC Normalize	
Slope:	1.000
Y Int:	0.0000
Slope factor:	1.000
Offset:	0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)/R	Stddev	Emphasis
Blank	0.0000	-0.00002153	-0.00002153	0.0000	1,905	88.80	1
CalibStd-1	0.1000	0.1275	0.02752	27.52	416,600	1,100	1
CalibStd-2	0.2000	0.2063	0.006337	3.168	672,900	1,860	1
CalibStd-3	0.3000	0.2916	-0.008373	-2.791	950,200	5,343	1
CalibStd-4	0.4000	0.3745	-0.02548	-6.371	1,220,000	7,064	1



Element Name:	V
Element Wavelength:	V 309.311 nm
Concentration Units:	ppm
Date of Calibration:	5/23/2018 9:29:19AM
Date of Fit:	5/23/2018 9:29:19AM
Type of Fit:	Linear
Correlation:	0.9992
A0 (Offset):	-1,127
A1 (Gain):	53,020
A2 (Curvature):	0.0000
n (Exponent):	1.000
Reslope	
QC Normalize	
Slope:	1.000
Y Int:	0.0000
Slope factor:	1.000
Offset:	0.0000

Standard Name	Stated	Found	Diff	% Diff	(S)/R	Stddev	Emphasis
Blank	0.0000	-0.000008687	-0.000008687	0.0000	-1,127	193.0	1
CalibStd-1	0.1000	0.1099	0.009859	9.859	4,698	479.7	1
CalibStd-2	0.2000	0.2058	0.005758	2.879	9,782	219.6	1
CalibStd-3	0.3000	0.2982	-0.001760	-0.5866	14,690	106.1	1
CalibStd-4	0.4000	0.3861	-0.01386	-3.464	19,350	90.58	1



Standard Name	Stated	Found	Diff	% Diff	(S)IR	Stddev	Emphasis
Blank	0.0000	-0.00001788	-0.00001788	0.0000	41.71	1.978	1
CalibStd-1	0.1000	0.1238	0.02384	23.84	4,584	19.34	1
CalibStd-2	0.2000	0.2034	0.003363	1.682	7,500	9.867	1
CalibStd-3	0.3000	0.2899	-0.01013	-3.377	10,670	39.25	1
CalibStd-4	0.4000	0.3829	-0.01707	-4.268	14,080	32.50	1

Sample Name	Acquisition Date	Correction Factor
Initial 12hrs	5/23/2018 9:57:12AM	1.00

#### Concentration

Element/Wavelength	As1880	Ba4554	Ca8833	Cd2288	Co2288
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.00725205	-0.000215441	0.060404	-0.000415058	0.00090156
Std Dev:	0.00119917	0.000189866	0.00893461	0.000311287	0.001111133
%RSD:	16.5356	88.1288	14.7914	74.9985	123.267
Repeat: 1	-0.00677183	3.10762e-006	0.0705597	-6.40298e-005	0.00216128
Repeat: 2	-0.00636741	-0.000309671	0.0568991	-0.000523608	0.000483532
Repeat: 3	-0.0086169	-0.00033976	0.0537533	-0.000657535	5.98626e-005
Element/Wavelength	Cu3247	Fe2588	K_7884	Li8707	Mg2785
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000240393	0.0031332	0.013023	-0.000484374	0.00273302
Std Dev:	0.000125185	0.000780871	0.000511925	5.09805e-006	0.000157535
%RSD:	52.0751	24.9225	3.93093	1.0525	5.76415
Repeat: 1	-9.64956e-005	0.00400786	0.0135294	-0.000479285	0.00290975
Repeat: 2	-0.000300451	0.00288558	0.0125058	-0.000489481	0.00268198
Repeat: 3	-0.000324233	0.00250616	0.0130338	-0.000484356	0.00260733
Element/Wavelength	Mn2678	Na5885	Ni2218	Pb2203	Sr4077
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000281675	0.0311675	-0.000740639	0.00139834	-0.000504432
Std Dev:	0.000101685	0.000629331	0.000287224	0.000289125	2.72644e-005
%RSD:	36.1002	2.01919	38.7806	20.6763	5.40497
Repeat: 1	-0.000164293	0.0308385	-0.000448979	0.0011203	-0.000473292
Repeat: 2	-0.000337916	0.0307709	-0.000749726	0.00137731	-0.000524011
Repeat: 3	-0.000342815	0.0318932	-0.00102321	0.00169741	-0.000515994
Element/Wavelength	V_3083	Zn2138			
Units:	ppm	ppm			

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Avg. of Repeats:	0.00327092	0.00162604			
Std Dev:	0.000124981	0.000898387			
%RSD:	3.82098	55.25			
Repeat: 1	0.00312777	0.00264916			
Repeat: 2	0.00332659	0.00126285			
Repeat: 3	0.00335838	0.000966104			
Sample Name	Acquisition Date		Correction Factor		
18hrs	5/23/2018 10:00:05AM		1.00		
<u>Concentration</u>					
Element/Wavelength	As1880	Ba4554	Ca3883	Cd2288	Co2288
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.00834785	-0.000372983	0.0530638	-0.000582861	-0.000446633
Std Dev:	0.00108102	2.2551e-005	0.000715551	3.0789e-005	4.2368e-006
%RSD:	12.9496	6.04613	1.34847	5.28239	0.948609
Repeat: 1	-0.00958433	-0.000347284	0.0538307	-0.000552506	-0.000443975
Repeat: 2	-0.0078777	-0.000382195	0.0529465	-0.000582011	-0.000451519
Repeat: 3	-0.00758152	-0.000389469	0.0524141	-0.000614067	-0.000444405
Element/Wavelength	Cu3247	Fe2588	K_7684	Li8707	Mg2795
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000362735	0.00205466	0.0115115	-0.00048127	0.00259136
Std Dev:	0.000193827	0.00022336	8.96508e-005	4.42786e-006	3.63619e-005
%RSD:	53.435	10.8709	0.778791	0.920036	1.4032
Repeat: 1	-0.000154274	0.00223246	0.0115528	-0.000483605	0.00263277
Repeat: 2	-0.000396416	0.00212755	0.0114087	-0.000484041	0.00256464
Repeat: 3	-0.000537514	0.00180396	0.0115731	-0.000476163	0.00257666
Element/Wavelength	Mn2678	Na5886	Ni2218	Pb2203	Sr4077
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000323515	0.0289372	-0.0010222	0.00152114	-0.000524819
Std Dev:	2.66642e-005	0.000432422	6.4249e-005	0.000516623	8.28145e-006
%RSD:	8.24201	1.49435	6.28535	33.9628	1.57796
Repeat: 1	-0.000293256	0.0292882	-0.000964505	0.00206061	-0.000515261
Repeat: 2	-0.000343569	0.0284541	-0.00109144	0.00147193	-0.000529832
Repeat: 3	-0.000333721	0.0290692	-0.00101066	0.00103089	-0.000529366
Element/Wavelength	V_3083	Zn2138			
Units:	ppm	ppm			
Avg. of Repeats:	0.000872989	-0.000321138			
Std Dev:	0.000410913	4.77506e-005			
%RSD:	47.0696	14.8692			
Repeat: 1	0.000399537	-0.00026706			
Repeat: 2	0.00108267	-0.000357493			
Repeat: 3	0.00113676	-0.000338861			
Sample Name	Acquisition Date		Correction Factor		
24hrs	5/23/2018 10:02:59AM		1.00		
<u>Concentration</u>					
Element/Wavelength	As1880	Ba4554	Ca3883	Cd2288	Co2288
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.00910579	-0.000454836	0.00668423	-0.00055283	-0.000497278
Std Dev:	0.000457818	3.95587e-005	0.000347569	5.52419e-005	0.000118458
%RSD:	5.02777	8.69736	5.19984	9.99256	23.8213
Repeat: 1	-0.00963383	-0.000409234	0.0070783	-0.000609453	-0.000364549
Repeat: 2	-0.00886359	-0.000479927	0.00655306	-0.000499081	-0.000592273
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Repeat: 3	-0.00881995	-0.000475347	0.00642134	-0.000549956	-0.00053501
Element/Wavelength	Cu3247	Fe2586	K_7884	Li8707	Mg2786
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.00045684	0.00174924	0.00232151	-0.000493271	-0.000131551
Std Dev:	6.17313e-005	0.000182291	0.000264057	5.28814e-006	2.67767e-005
%RSD:	13.5127	10.4212	11.3743	1.07206	20.3546
Repeat: 1	-0.000517174	0.00192408	0.00232054	-0.000491731	-0.000100644
Repeat: 2	-0.000393801	0.00176333	0.00258606	-0.000499158	-0.000146272
Repeat: 3	-0.000459544	0.00156031	0.00205794	-0.000488923	-0.000147738
Element/Wavelength	Mn2678	Na5886	NI2218	Pb2203	Sr4077
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000467066	0.00513405	-5.094e-005	0.0016577	-0.000585868
Std Dev:	2.01695e-005	4.24251e-005	5.84855e-005	0.000632459	1.39441e-005
%RSD:	4.31834	0.826347	114.813	38.1528	2.38008
Repeat: 1	-0.000444388	0.00514058	1.64938e-005	0.00227662	-0.000569867
Repeat: 2	-0.000473813	0.00517283	-8.14835e-005	0.00101243	-0.000592319
Repeat: 3	-0.000482998	0.00508874	-8.78302e-005	0.00168415	-0.000595419
Element/Wavelength	V_3083	Zn2138			
Units:	ppm	ppm			
Avg. of Repeats:	0.00162688	-0.000520485			
Std Dev:	0.000237964	6.85941e-005			
%RSD:	14.627	13.1789			
Repeat: 1	0.00136121	-0.00041383			
Repeat: 2	0.00169897	-0.000563536			
Repeat: 3	0.00182046	-0.00056537			
Sample Name	Acquisition Date		Correction Factor		
30hrs	5/23/2018 10:05:41AM		1.00		
Concentration					
Element/Wavelength	As1880	Ba4564	Ca3833	Cd2288	Co2288
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.00822283	-0.00040043	0.00364194	-0.000618721	-0.000490385
Std Dev:	0.000420579	1.95471e-005	0.000142373	1.73612e-005	2.80878e-005
%RSD:	5.11477	4.88152	3.90927	2.80598	5.72771
Repeat: 1	-0.00794483	-0.000378167	0.00380156	-0.000637137	-0.00047376
Repeat: 2	-0.00801698	-0.000408347	0.0035962	-0.000616374	-0.000474581
Repeat: 3	-0.00870668	-0.000414777	0.00352806	-0.000602653	-0.000522815
Element/Wavelength	Cu3247	Fe2586	K_7884	Li8707	Mg2786
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000679647	0.00145736	0.00186524	-0.000499378	4.38084e-005
Std Dev:	7.28651e-005	2.78467e-005	0.000232424	5.85043e-006	2.16741e-005
%RSD:	10.721	1.91076	12.4608	1.17154	49.4748
Repeat: 1	-0.000689659	0.0014348	0.00212092	-0.000500976	6.6922e-005
Repeat: 2	-0.000602294	0.00144879	0.00166676	-0.000492894	4.05634e-005
Repeat: 3	-0.000746989	0.00148848	0.00180803	-0.000504263	2.39397e-005
Element/Wavelength	Mn2678	Na5886	NI2218	Pb2203	Sr4077
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000422175	0.00439196	-0.000277258	0.0009786	-0.000585927
Std Dev:	1.8176e-005	7.02433e-005	7.7613e-005	0.000387449	7.41975e-006
%RSD:	4.30532	1.59936	27.9931	39.5921	1.26632
Repeat: 1	-0.000401564	0.0044324	-0.000187683	0.0010844	-0.000577376
Repeat: 2	-0.000429048	0.00443263	-0.000324497	0.00130216	-0.000589743
Repeat: 3	-0.000435912	0.00431085	-0.000319593	0.000549241	-0.000590663
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Element/Wavelength	V_3083	Zn2138			
Units:	ppm	ppm			
Avg. of Repeats:	0.00224138	-0.000590408			
Std Dev:	0.000323613	4.77533e-005			
%RSD:	14.4381	8.08819			
Repeat: 1	0.0021883	-0.00057826			
Repeat: 2	0.0019476	-0.000549903			
Repeat: 3	0.00258826	-0.000643062			
Sample Name	Acquisition Date		Correction Factor		
36hrs	5/23/2018 10:08:30AM		1.00		
<u>Concentration</u>					
Element/Wavelength	As1880	Ba4554	Ca3833	Cd2288	Co2288
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.00781729	-0.000314856	0.108633	-0.000597432	-0.000592703
Std Dev:	0.000312307	1.71424e-005	0.000645584	0.000114046	4.49357e-005
%RSD:	3.99508	5.44452	0.594279	19.0894	7.58148
Repeat: 1	-0.00816298	-0.000296092	0.10789	-0.00065705	-0.000630032
Repeat: 2	-0.00773338	-0.000318778	0.108952	-0.000669313	-0.000542827
Repeat: 3	-0.00755551	-0.000329697	0.109057	-0.000465933	-0.00060525
Element/Wavelength	Cu3247	Fe2586	K_7864	Li8707	Mg2795
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000608118	0.00231604	0.0213665	-0.00048903	0.0060382
Std Dev:	0.000163632	9.65051e-005	0.000300975	8.85289e-006	5.93424e-005
%RSD:	26.908	4.16681	1.40863	1.8103	0.982783
Repeat: 1	-0.000620709	0.0024005	0.021019	-0.000488397	0.00607383
Repeat: 2	-0.000438554	0.00221086	0.0215456	-0.000480511	0.00607107
Repeat: 3	-0.000765091	0.00233678	0.0215349	-0.000498183	0.0059697
Element/Wavelength	Mn2578	Na5886	Ni2218	Pb2203	Sr4077
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000241563	0.0559744	-0.00199866	0.000485267	-0.000449357
Std Dev:	1.28952e-005	0.000504403	0.000122839	0.000332304	1.00235e-005
%RSD:	5.33823	0.90113	6.1461	68.4786	2.23063
Repeat: 1	-0.000230479	0.0554015	-0.00189365	0.000527096	-0.000440254
Repeat: 2	-0.000255716	0.0561703	-0.00213374	0.000794677	-0.000447718
Repeat: 3	-0.000238494	0.0563515	-0.00196858	0.000134029	-0.000460099
Element/Wavelength	V_3083	Zn2138			
Units:	ppm	ppm			
Avg. of Repeats:	0.00233779	-0.000702473			
Std Dev:	0.000158523	3.42994e-005			
%RSD:	6.78089	4.88266			
Repeat: 1	0.00242124	-0.000664785			
Repeat: 2	0.00215497	-0.000710774			
Repeat: 3	0.00243715	-0.00073186			
Sample Name	Acquisition Date		Correction Factor		
42hrs	5/23/2018 10:15:06AM		50.00		
<u>Concentration</u>					
Element/Wavelength	As1880	Ba4554	Ca3833	Cd2288	Co2288
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.353653	-0.0140523	5.67064	0.00945866	-0.0112767
Std Dev:	0.0122486	0.00330272	0.290125	0.0650165	0.0155121
%RSD:	3.46346	23.5031	5.11626	687.375	137.559
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Repeat: 1	-0.35445	-0.0103766	5.34401	-0.0297866	-0.0180242
Repeat: 2	-0.365483	-0.01501	5.76947	-0.0263443	-0.0222722
Repeat: 3	-0.341025	-0.0167704	5.89843	0.0845069	0.00646644
Element/Wavelength	Cu3247	Fe2589	K_7864	Li8707	Mg2795
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.041257	-0.00235694	0.382674	-0.018497	0.115848
Std Dev:	0.00127498	0.0089993	0.037255	0.000734581	0.00114594
%RSD:	3.09035	381.821	9.73544	3.97135	0.989176
Repeat: 1	-0.0398263	0.00131134	0.425204	-0.0191588	0.114561
Repeat: 2	-0.0422731	-0.012611	0.367002	-0.0177066	0.11676
Repeat: 3	-0.0416715	0.00422884	0.355814	-0.0186256	0.116221
Element/Wavelength	Mn2678	Na5886	NI2218	Pb2203	Sr4077
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	0.0601506	6.14699	-0.254425	0.0346898	-0.00958574
Std Dev:	0.0273445	0.050439	0.0151217	0.0360082	0.00393433
%RSD:	45.46	0.820548	5.94349	103.801	41.0435
Repeat: 1	0.089712	6.2029	-0.264507	0.0289383	-0.00521357
Repeat: 2	0.0549778	6.13319	-0.26173	0.00190345	-0.0107032
Repeat: 3	0.035762	6.10489	-0.237038	0.0732276	-0.0128405
Element/Wavelength	V_3083	Zn2138			
Units:	ppm	ppm			
Avg. of Repeats:	0.316964	0.11811			
Std Dev:	0.0170451	0.119748			
%RSD:	5.37762	101.387			
Repeat: 1	0.298977	0.0562897			
Repeat: 2	0.319037	0.0419067			
Repeat: 3	0.332878	0.256133			
Sample Name	Acquisition Date		Correction Factor		
48hrs	5/23/2018 10:18:03AM		1.00		
Concentration					
Element/Wavelength	As1880	Ba4554	Ce3833	Cd2288	Co2288
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.00767185	0.000544749	0.289698	-0.000572743	-0.000434073
Std Dev:	0.000261118	0.000103188	0.0103336	4.61408e-005	0.000112345
%RSD:	3.40358	18.9423	3.56703	8.05611	25.8816
Repeat: 1	-0.00797335	0.000437145	0.277948	-0.000526765	-0.000327885
Repeat: 2	-0.00752362	0.000554237	0.293771	-0.000572419	-0.000422633
Repeat: 3	-0.00751859	0.000642865	0.297374	-0.000619045	-0.0005517
Element/Wavelength	Cu3247	Fe2589	K_7864	Li8707	Mg2795
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	-0.000178598	0.00223006	0.0366468	-6.31258e-005	0.00785061
Std Dev:	4.70641e-005	0.00127362	0.000606728	1.62901e-005	0.000108133
%RSD:	26.3519	57.1113	1.65561	25.8058	1.37738
Repeat: 1	-0.000232745	0.00155415	0.0360483	-6.52587e-005	0.00773488
Repeat: 2	-0.000155542	0.00143689	0.0366306	-7.82444e-005	0.00786788
Repeat: 3	-0.000147508	0.00369915	0.0372614	-4.58743e-005	0.00794907
Element/Wavelength	Mn2678	Na5886	NI2218	Pb2203	Sr4077
Units:	ppm	ppm	ppm	ppm	ppm
Avg. of Repeats:	7.65094e-005	2.10351	-0.016956	-0.00019814	-0.000142796
Std Dev:	0.000279323	0.0434817	0.000145914	0.000521761	3.36572e-005
%RSD:	365.083	2.06711	0.860543	263.329	23.5702
Repeat: 1	0.000393967	2.0768	-0.016792	-0.00067794	-0.000106377
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Repeat: 2	-3.28558e-005	2.08004	-0.017005	-0.000273801	-0.000172756
Repeat: 3	-0.000131583	2.15368	-0.0170712	0.000357321	-0.000149253
Element/Wavelength	V_3083	Zn2138			
Units:	ppm	ppm			
Avg. of Repeats:	0.0063292	0.0042646			
Std Dev:	0.000274146	8.73813e-005			
%RSD:	4.33144	2.04899			
Repeat: 1	0.00638231	0.00421561			
Repeat: 2	0.00657291	0.0042127			
Repeat: 3	0.00603239	0.00436548			

## APPENDIX C

### Results by Using GC-MS Analysis

AMDIS GC/MS Analysis Report - Data: C:\VARIANMS\DATA\1040.FIN		Page 001
<p style="text-align: center;">AMDIS GC/MS Analysis Report Organization: UTM Division: Chemical Engineering</p>		
<p>Data: 1040.SMS Library: NISTMS32.DLL Number of Identifications: 9</p>		
RT(min)	Chemical Name	
3.0470	>Pentano, 3-ethyl-2,2-dimethyl- (DF:16747323)	
	Width = 3.6	Weighted = 52
	Purity = 34%	Reverse = 52
	Model = 56 m/z	Corrections: n/a
	Min. Abund. = 0.066%	
	Scan = 185	
	Peak Tailored = 5.3	
	S/N (total) = 130	
	Base Peak = 67857	
	Amount = 0.212%	
3.1192	>2-Octanol, 8,8-dimethoxy-2,6-dimethyl- (DF:141924)	
	Width = 5.1	Weighted = 60
	Purity = 7.2%	Reverse = 62
	Model = 59 m/z (69)	Corrections: n/a
	Min. Abund. = 0.49%	
	Scan = 193	
	Peak Tailored = 1.5	
	S/N (total) = 56	
	Base Peak = 9042	
	Amount = 0.046%	
3.3563	>1,2,4,5-Tetrizine, 1,4-dioxythoxyhydro- (DF:35035699)	
	Width = 4.5	Weighted = 65
	Purity = 6.3%	Reverse = 72
	Model = 72 m/z	Corrections: n/a
	Min. Abund. = 0.32%	
	Scan = 217	
	Peak Tailored = 1.7	
	S/N (total) = 60	
	Base Peak = 13963	
	Amount = 0.045%	
3.5737	>Allyldimethylvinylsilane (DF:22146254)	
	Width = 4.6	Weighted = 78
	Purity = 1.8%	Reverse = 78
	Model = 85 m/z	Corrections: n/a
	Min. Abund. = 1.0%	
	Scan = 239	
	Peak Tailored = 2.1	
	S/N (total) = 21	
	Base Peak = 4117	
	Amount = 0.0057%	
3.5786	>2,2-Dimethylpropanoic anhydride (DF:1538756)	
	Width = 3.6	Weighted = 87
	Purity = 4.8%	Reverse = 94
	Model = TIC	Corrections: n/a
	Min. Abund. = 0.39%	
	Scan = 240	
	Peak Tailored = 1.8	
	S/N (total) = 35	
	Base Peak = 11405	
	Amount = 0.0029%	
3.7082	>2,2-Dimethylpropanoic anhydride (DF:1538756)	
	Width = 3.7	Weighted = 88
	Purity = 6.3%	Reverse = 93
	Model = TIC	Corrections: n/a
	Min. Abund. = 0.36%	
	Scan = 253	
	Peak Tailored = 1.9	
	S/N (total) = 37	
	Base Peak = 12987	
	Amount = 0.0046%	
Chemist:	Operator: yzid	
<p style="text-align: center;">-----</p>		
AMDIS GC/MS Analysis Report - Wed May 23 14:16:18 2018		Page 001

3.7122 >3H-1,2,4-Triazole-3-one, 1,2-dihydro- (ID# 930336)  
 Width = 5.1 Weighted = 81  
 Purity = 1.5% Reverse = 85  
 Model = 55 m/z Corrections: n/a  
 Min. Abund. = 0.94%  
 Scan = 323  
 Peak Tailings = 1.2  
 S/N (total) = 20  
 Base Peak = 4722  
 Amount = 0.00024

4.0027 >Oxalic acid, cyclohexyl propyl ester (ID# EPA-309303)  
 Width = 1.0 Weighted = 85  
 Purity = 37% Reverse = 88  
 Model = 55 m/z Corrections: n/a  
 Min. Abund. = 0.055%  
 Scan = 283  
 Peak Tailings = 1.6  
 S/N (total) = 122  
 Base Peak = 5118  
 Amount = 0.176%

4.3973 >Oxalic acid, butyl cyclobutyl ester (ID# EPA-309694)  
 Width = 1.0 Weighted = 72  
 Purity = 1.9% Reverse = 78  
 Model = 55 m/z (83) Corrections: n/a  
 Min. Abund. = 0.73%  
 Scan = 323  
 Peak Tailings = 1.2  
 S/N (total) = 24  
 Base Peak = 464  
 Amount = 0.0213%

This report consists of 2 pages

AMDIS GC/MS Analysis Report  
 Organization: UTM  
 Division: Chemical Engineering

Date: 2027.SMS  
 Library: NISTMS32.DLL  
 Number of identifications: 10

RT(min) Chemical Name

3.0470 >2-Isopropyl-3-vinylcyclohexane (ID# 7077230)  
 Width = 5.0 Weighted = 79  
 Purity = 50% Reverse = 81  
 Model = 57 m/z (52) Corrections: n/a  
 Min. Abund. = 0.038%  
 Scan = 185  
 Peak Tailings = 8.6  
 S/N (total) = 24  
 Base Peak = 120678  
 Amount = 0.660%

3.0628 >4-Methoxypyrimido[2,3-c]pyrimidine (ID# 4786769)  
 Width = 3.4 Weighted = 42  
 Purity = 1.1% Reverse = 43  
 Model = 52 m/z (57) Corrections: n/a  
 Min. Abund. = 2.8%  
 Scan = 186  
 Peak Tailings = 2.5  
 S/N (total) = 29  
 Base Peak = 1647  
 Amount = 0.00558%

3.0732 >Thiazole (ID# 288471)  
 Width = 3.3 Weighted = 48  
 Purity = 1.3% Reverse = 76  
 Model = 55 m/z (57) Corrections: n/a  
 Min. Abund. = 2.2%  
 Scan = 188  
 Peak Tailings = 0.0  
 S/N (total) = 29  
 Base Peak = 2037  
 Amount = 0.00399%

3.0926 >Allylidimethylvinylsilane (ID# 22146254)  
 Width = 4.4 Weighted = 63  
 Purity = 4.9% Reverse = 79  
 Model = 59 m/z Corrections: n/a  
 Min. Abund. = 0.35%  
 Scan = 190  
 Peak Tailings = 1.4  
 S/N (total) = 58  
 Base Peak = 13345  
 Amount = 0.0322%

3.1295 >1H-Tetrazole, 1-methyl- (ID# 16681779)  
 Width = 2.4 Weighted = 57  
 Purity = 1.3% Reverse = 86  
 Model = 55 m/z (57) Corrections: n/a  
 Min. Abund. = 1.1%  
 Scan = 194  
 Peak Tailings = 1.1  
 S/N (total) = 28  
 Base Peak = 4164  
 Amount = 0.00937%

3.2568 >2-Methyl-1,5-hexadecane-3-ol (ID# 17123603)  
 Width = 5.4 Weighted = 64  
 Purity = 1.3% Reverse = 64  
 Model = 71 m/z Corrections: n/a  
 Min. Abund. = 1.0%  
 Scan = 207  
 Peak Tailings = 1.9  
 S/N (total) = 26  
 Base Peak = 4684  
 Amount = 0.00291%

Chemist:

Operator:  
 yzaid

3.3337 >Acetamide, N-(2-hydroxyethyl)- (ID#142267)  
 Width = 4.0 Weighted = 65  
 Purity = 10% Reverse = 72  
 Model = 72 m/z Corrections: n/a  
 Min. Abund. = 0.24%  
 Scan = 215  
 Peak Tailings = 1.4  
 S/N (total) = 70  
 Base Peak = 18956  
 Amount = 0.0490%

3.5431 >Silicon tetrafluoride (ID#7783611)  
 Width = 3.8 Weighted = 86  
 Purity = 1.8% Reverse = 95  
 Model = TIC Corrections: n/a  
 Min. Abund. = 0.72%  
 Scan = 235  
 Peak Tailings = 1.1  
 S/N (total) = 27  
 Base Peak = 6429  
 Amount = 0.00185%

3.5490 >3H-1,2,4-Triazol-3-one, 1,2-dihydro- (ID#930336)  
 Width = 4.0 Weighted = 77  
 Purity = 1.6% Reverse = 96  
 Model = 85 m/z Corrections: n/a  
 Min. Abund. = 0.82%  
 Scan = 237  
 Peak Tailings = 1.6  
 S/N (total) = 25  
 Base Peak = 6687  
 Amount = 0.00577%

3.9742 >Oxalic acid, cyclohexyl hexyl ester (ID#EPA-309308)  
 Width = 5.1 Weighted = 85  
 Purity = 5.1% Reverse = 85  
 Model = TIC Corrections: n/a  
 Min. Abund. = 0.044%  
 Scan = 280  
 Peak Tailings = 1.8  
 S/N (total) = 153  
 Base Peak = 106236  
 Amount = 0.127%

This report consists of 2 pages

AMDIS GC/MS Analysis Report  
 Organization: UTM  
 Division: Chemical Engineering

Data: 3023.SMS  
 Library: NISTMS32.DLI  
 Number of identifications: 9

RT(min) Chemical Name

3.0460 >Nonane, 2,2,3-trimethyl- (ID#55499042)  
 Width = 3.9 Weighted = 79  
 Purity = 71% Reverse = 81  
 Model = 57 m/z (53) Corrections: n/a  
 Min. Abund. = 0.021%  
 Scan = 185  
 Peak Tailings = 8.5  
 S/N (total) = 298  
 Base Peak = 214073  
 Amount = 1.16%

3.0470 >2-Isopropyl-3-vinylcyclohexane (ID#7077230)  
 Width = 3.4 Weighted = 80  
 Purity = 44% Reverse = 82  
 Model = TIC Corrections: n/a  
 Min. Abund. = 0.038%  
 Scan = 185  
 Peak Tailings = 3.8  
 S/N (total) = 236  
 Base Peak = 118969  
 Amount = 0.257%

3.0548 >2-Propynal (ID#624679)  
 Width = 3.1 Weighted = 61  
 Purity = 3.1% Reverse = 74  
 Model = 53 m/z (57) Corrections: n/a  
 Min. Abund. = 0.46%  
 Scan = 186  
 Peak Tailings = 7.0  
 S/N (total) = 40  
 Base Peak = 9746  
 Amount = 0.0159%

3.0916 >Allyldimethylvinylsilane (ID#22146254)  
 Width = 5.2 Weighted = 71  
 Purity = 5.0% Reverse = 84  
 Model = 85 m/z Corrections: n/a  
 Min. Abund. = 0.34%  
 Scan = 190  
 Peak Tailings = 1.3  
 S/N (total) = 60  
 Base Peak = 13101  
 Amount = 0.0284%

3.1392 >1-Pentanol, 4-methyl- (ID#626891)  
 Width = 3.2 Weighted = 53  
 Purity = 4.3% Reverse = 62  
 Model = 69 m/z (85) Corrections: n/a  
 Min. Abund. = 0.55%  
 Scan = 190  
 Peak Tailings = 0.8  
 S/N (total) = 50  
 Base Peak = 8175  
 Amount = 0.0161%

3.3299 >1,2,4,5-Tetrazine, 1,4-diethylhexahydro- (ID#35035699)  
 Width = 4.5 Weighted = 61  
 Purity = 9.9% Reverse = 69  
 Model = 72 m/z Corrections: n/a  
 Min. Abund. = 0.22%  
 Scan = 214  
 Peak Tailings = 1.8  
 S/N (total) = 78  
 Base Peak = 30126  
 Amount = 0.0612%

Chemist:

Operator:  
 yzaid

3.9767 >Cyclobutanecarboxylic acid, 2-propenyl ester (ID# EPA-282603)

Width = 5.0	Weighted = 82
Purity = 45%	Reverse = 84
Model = 55 m/z (51)	Corrections: n/a
Min. Abund. = 0.038%	
Scan = 281	
Peak Tailings = 1.5	
S/N (total) = 179	
Base Peak = 118.278	
Amount = 0.348%	

3.9893 >Butane, 1-chloro- (ID# 109693)

Width = 4.7	Weighted = 72
Purity = 3.1%	Reverse = 72
Model = 51 m/z (55)	Corrections: n/a
Min. Abund. = 0.37%	
Scan = 282	
Peak Tailings = 1.6	
S/N (total) = 43	
Base Peak = 121.90	
Amount = 0.0187%	

4.3725 >(Trimethylsilyl)acetylene (ID# 1066542)

Width = 10.3	Weighted = 43
Purity = 3.9%	Reverse = 51
Model = 83 m/z (55)	Corrections: n/a
Min. Abund. = 0.89%	
Scan = 321	
Peak Tailings = 0.5	
S/N (total) = 26	
Base Peak = 46.68	
Amount = 0.0138%	

This report consists of 2 pages



AMD15 GC/MS Analysis Report  
Organization: UTM  
Division: Chemical Engineering

Data: 4020.SMS  
Library: NISTMS32.DLL  
Number of Identifications: 13

RT(min)	Chemical Name		
3.0460	>1-Hexano, 4-methyl- (ID#3769231)		
	Width = >11	Weighted = 81	
	Purity = 42%	Reverse = 81	
	Model = 57 m/z	Corrections:	n/a
	Min. Abund. = 0.037%		
	Scan = 185		
	Peak Tailor = 10.4		
	S/N (total) = 229		
	Base Peak = 120609		
	Amount = 0.606%		
3.0470	>Pentano, 3-ethyl-2,2-dimethyl- (ID#16747323)		
	Width = >6	Weighted = 81	
	Purity = 44%	Reverse = 83	
	Model = 57 m/z	Corrections:	n/a
	Min. Abund. = 0.036%		
	Scan = 185		
	Peak Tailor = 5.7		
	S/N (total) = 234		
	Base Peak = 124398		
	Amount = 0.369%		
3.0925	>Trimethylaluminum (ID#75241)		
	Width = >2	Weighted = 84	
	Purity = 1.8%	Reverse = 82	
	Model = 57 m/z	Corrections:	n/a
	Min. Abund. = 0.43%		
	Scan = 190		
	Peak Tailor = 0.0		
	S/N (total) = 36		
	Base Peak = 10299		
	Amount = 0.00291%		
3.1110	>Thiophene acid (ID#55561029)		
	Width = >8	Weighted = 61	
	Purity = 10%	Reverse = 65	
	Model = 57 m/z (56)	Corrections:	n/a
	Min. Abund. = 0.24%		
	Scan = 192		
	Peak Tailor = 5.0		
	S/N (total) = 83		
	Base Peak = 18601		
	Amount = 0.0527%		
3.3494	>1,2,4,5-tetrahydro, 1,4-diethylhexahydro- (ID#35035699)		
	Width = 4.5	Weighted = 76	
	Purity = 15%	Reverse = 81	
	Model = 85 m/z (89)	Corrections:	n/a
	Min. Abund. = 0.17%		
	Scan = 216		
	Peak Tailor = 1.7		
	S/N (total) = 96		
	Base Peak = 25589		
	Amount = 0.0967%		
3.3542	>6-Heptano-2,4-diol (ID#19781761)		
	Width = 4.9	Weighted = 60	
	Purity = 2.4%	Reverse = 73	
	Model = 85 m/z (85)	Corrections:	n/a
	Min. Abund. = 0.48%		
	Scan = 217		
	Peak Tailor = 1.2		
	S/N (total) = 62		
	Base Peak = 6178		
	Amount = 0.0361%		

Chemist:

Operator:  
yzaid

3.5745	>Hexanoic acid, 2-oxo-, methyl ester (ID#6375831)		
	Width = >7	Weighted = 80	
	Purity = 10%	Reverse = 87	
	Model = 73	Corrections:	n/a
	Min. Abund. = 0.25%		
	Scan = 185		
	Peak Tailor = 61.4		
	S/N (total) = 229		
	Base Peak = 0.03248%		
3.5775	>Allyldimethylvinylsilane (ID#22140254)		
	Width = 4.2	Weighted = 76	
	Purity = 1%	Reverse = 76	
	Model = 85 m/z	Corrections:	n/a
	Min. Abund. = 0.04%		
	Scan = 185		
	Peak Tailor = 1.0		
	S/N (total) = 23		
	Base Peak = 5088		
	Amount = 0.00773%		
3.7005	>3H-1,2,4-Triazolo[3-one, 1,2-dihydro- (ID#930334)		
	Width = 4.4%	Weighted = 80	
	Purity = 2.4%	Reverse = 80	
	Model = 85 m/z	Corrections:	n/a
	Min. Abund. = 0.57%		
	Scan = 202		
	Peak Tailor = 1.7		
	S/N (total) = 73		
	Base Peak = 0.0331%		
3.9916	>2-Pentanoic acid, 5,5-dimethyl- (ID#93677658)		
	Width = 4.3	Weighted = 57	
	Purity = 2.0%	Reverse = 61	
	Model = 85 m/z (83)	Corrections:	n/a
	Min. Abund. = 0.03%		
	Scan = 185		
	Peak Tailor = 5.0		
	S/N (total) = 229		
	Base Peak = 0.0302%		
3.9935	>Cyclobutanecarboxylic acid, 2-propenylester (ID#EPA-282603)		
	Width = 4.1%	Weighted = 83	
	Purity = 1%	Reverse = 83	
	Model = 85 m/z	Corrections:	n/a
	Min. Abund. = 0.036%		
	Scan = 185		
	Peak Tailor = 1.6		
	S/N (total) = 1216		
	Base Peak = 0.0311%		
	Amount = 0.108%		
3.9945	>Cyclopropane, 2-bromo-1,1,3-trimethyl- (ID#34617002)		
	Width = 4.1%	Weighted = 80	
	Purity = 1%	Reverse = 80	
	Model = 85 m/z (83)	Corrections:	n/a
	Min. Abund. = 0.03%		
	Scan = 202		
	Peak Tailor = 1.6		
	S/N (total) = 1216		
	Base Peak = 0.0311%		
4.0007	>5-Nonanone (ID#502567)		
	Width = >4	Weighted = 58	
	Purity = 1%	Reverse = 67	
	Model = 73 m/z (83)	Corrections:	n/a
	Min. Abund. = 1.0%		
	Scan = 185		
	Peak Tailor = 0.1		
	S/N (total) = 1216		
	Base Peak = 0.0322%		



AMDIS GC/MS Analysis Report  
 Organization: UTM  
 Division: Chemical Engineering

Data: 5019.SMS  
 Library: NISTMS32.DLL  
 Number of identifications: 15

RT(min)	Chemical Name
3.0460	>1-Hexano, 4-methyl- (ID#3769231)
	Width = >10
	Purity = 40%
	Model = 57 m/z
	Min. Abund. = 0.035%
	Scan = 185
	Peak Tailors = 9.5
	S/N (total) = 223
	Base Peak = 125843
	Amount = 0.624%
	Weighted = 83
	Reverse = 83
	Corrections: n/a
3.0470	>Decano, 2,2,3-trimethyl- (ID#62338094)
	Width = >5
	Purity = 43%
	Model = TIC
	Min. Abund. = 0.037%
	Scan = 185
	Peak Tailors = 4.8
	S/N (total) = 233
	Base Peak = 118519
	Amount = 0.313%
	Weighted = 78
	Reverse = 80
	Corrections: n/a
3.0829	>4,8-Dioxabicyclo[5.1.0.0(3,5)]octano, 1-methyl-5-(1-methylethyl)-, (1a,3a,5a,7a)- (ID#42669595)
	Width = >3
	Purity = 0 n/a
	Model = 79 m/z
	Min. Abund. = 2.9%
	Scan = 189
	Peak Tailors = 4.7
	S/N (total) = 25
	Base Peak = 1502
	Amount = 0.00798%
	Weighted = 56
	Reverse = 64
	Corrections: n/a
3.1004	>Cyclopropanecarboxylic acid, but-3-yn-2-yl ester (ID#EPA-299373)
	Width = >2
	Purity = 1.6%
	Model = 69 m/z (85)
	Min. Abund. = 0.66%
	Scan = 191
	Peak Tailors = 1.4
	S/N (total) = 36
	Base Peak = 6715
	Amount = 0.00481%
	Weighted = 77
	Reverse = 83
	Corrections: n/a
3.1091	>2-Pentanol, 5-methoxy-2-methyl- (ID#55724044)
	Width = 4.9
	Purity = 11%
	Model = 85 m/z (69)
	Min. Abund. = 0.15%
	Scan = 192
	Peak Tailors = 1.3
	S/N (total) = 94
	Base Peak = 29223
	Amount = 0.0805%
	Weighted = 69
	Reverse = 70
	Corrections: n/a
3.3472	>1,2,4,5-Tetrazine, 1,4-diethylhexahydro- (ID#35035699)
	Width = 5.1
	Purity = 29%
	Model = TIC
	Min. Abund. = 0.090%
	Scan = 216
	Peak Tailors = 1.3
	S/N (total) = 156
	Base Peak = 49965
	Amount = 0.0665%
	Weighted = 70
	Reverse = 78
	Corrections: n/a

Chemist:

Operator:  
 yzaid

3.5655 >Hexanoic acid, 2-oxo-, methyl ester (ID#6395831)  
 Width = 6.7 Weighted = 78  
 Purity = 15% Reverse = 83  
 Model = TIC Corrections: n/a  
 Min. Abund. = 0.16%  
 Scan = 239  
 Peak Tailino = 1.8  
 S/N (total) = 81  
 Base Peak = 27540  
 Amount = 0.0182%

3.5684 >5-Methyl 3-methylbutanethioate (ID#23747457)  
 Width = 4.7 Weighted = 77  
 Purity = 9.4% Reverse = 95  
 Model = 85 m/z Corrections: n/a  
 Min. Abund. = 0.21%  
 Scan = 239  
 Peak Tailino = 1.1  
 S/N (total) = 64  
 Base Peak = 20793  
 Amount = 0.0408%

3.6963 >Hexano, 1-(3-butenyloxy)- (ID#107995551)  
 Width = 4.3 Weighted = 63  
 Purity = 9.7% Reverse = 69  
 Model = 85 m/z (57) Corrections: n/a  
 Min. Abund. = 0.25%  
 Scan = 252  
 Peak Tailino = 1.4  
 S/N (total) = 62  
 Base Peak = 17873  
 Amount = 0.0461%

3.7227 >1-Pentanol-3-ol (ID#616251)  
 Width = >2 Weighted = 73  
 Purity = 4.5% Reverse = 80  
 Model = 57 m/z (85) Corrections: n/a  
 Min. Abund. = 0.31%  
 Scan = 255  
 Peak Tailino = 3.1  
 S/N (total) = 38  
 Base Peak = 14807  
 Amount = 0.0107%

3.9831 >Butyric acid, 2,2-dimethyl-, vinyl ester (ID#13170008)  
 Width = 4.3 Weighted = 51  
 Purity = 2.4% Reverse = 74  
 Model = 82 m/z (83) Corrections: n/a  
 Min. Abund. = 0.66%  
 Scan = 282  
 Peak Tailino = 1.9  
 S/N (total) = 60  
 Base Peak = 6767  
 Amount = 0.0247%

3.9908 >2,6-Octadiene, 2,4-dimethyl- (ID#63843038)  
 Width = 4.8 Weighted = 80  
 Purity = 6.2% Reverse = 81  
 Model = 83 m/z (79) Corrections: n/a  
 Min. Abund. = 0.020%  
 Scan = 282  
 Peak Tailino = 1.6  
 S/N (total) = 302  
 Base Peak = 77533  
 Amount = 0.636%

3.9985 >Cyclopropane, 2-bromo-1,1,3-trimethyl- (ID#36617002)  
 Width = 4.7 Weighted = 90  
 Purity = 65% Reverse = 90  
 Model = 79 m/z Corrections: n/a  
 Min. Abund. = 0.018%  
 Scan = 283  
 Peak Tailino = 1.1  
 S/N (total) = 289  
 Base Peak = 243872  
 Amount = 0.386%

4.3824 >6,6-Dimethyl-1,3-heptadien-5-ol (ID#81912030)  
 Width = 10.6 Weighted = 64  
 Purity = 17% Reverse = 65  
 Model = 83 m/z (55) Corrections: n/a  
 Min. Abund. = 0.14%  
 Scan = 322  
 Peak Tailino = 1.4  
 S/N (total) = 59  
 Base Peak = 30797  
 Amount = 0.0943%

Chemist:

Operator:  
yzaid

4.4316 >Ethanone, 1-cyclobutyl- (ID#3019258)  
 Width = >8 Weighted = 79  
 Purity = 9.2% Reverse = 84  
 Model = 55 m/z Corrections: n/a  
 Min. Abund. = 0.24%  
 Scan = 327  
 Peak Tailino = 999.0  
 S/N (total) = 42  
 Base Peak = 18442  
 Amount = 0.0283%

This report consists of 3 pages

AMDIS GC/MS Analysis Report  
Organization: UTM  
Division: Chemical Engineering

Data: 6015.SMS  
Library: NISTMS32.DLL  
Number of Identifications: 8

RT(min)	Chemical Name		
3.0470	>1-Hexano, 4-methyl- (ID#3769231)		
	Width = 3.8	Weighted = 80	
	Purity = 37%	Reverse = 82	
	Model = 55 m/z	Corrections:	n/a
	Min. Abund. = 0.11%		
	Scan = 185		
	Peak Tailings = 7.8		
	S/N (total) = 72		
	Base Peak = 37204		
	Amount = 0.225%		
3.1247	>Vinylidimethyl(hydroxymethyl)silane (ID#120491409)		
	Width = 4.5	Weighted = 59	
	Purity = 5.3%	Reverse = 59	
	Model = 85 m/z	Corrections:	n/a
	Min. Abund. = 0.95%		
	Scan = 193		
	Peak Tailings = 2.0		
	S/N (total) = 22		
	Base Peak = 4538		
	Amount = 0.0172%		
3.3649	>Pentano, 2,2-dimethyl- (ID#14250885)		
	Width = 4.0	Weighted = 69	
	Purity = 37%	Reverse = 87	
	Model = TIC	Corrections:	n/a
	Min. Abund. = 0.28%		
	Scan = 217		
	Peak Tailings = 7.1		
	S/N (total) = 45		
	Base Peak = 15294		
	Amount = 0.0215%		
3.6920	>Allyldimethylvinylsilane (ID#22146254)		
	Width = 3.7	Weighted = 79	
	Purity = 2.0%	Reverse = 79	
	Model = 85 m/z	Corrections:	n/a
	Min. Abund. = 2.0%		
	Scan = 240		
	Peak Tailings = 0.9		
	S/N (total) = 11		
	Base Peak = 2091		
	Amount = 0.0050%		
3.7180	>3H-1,2,4-Triazol-3-one, 1,2-dihydro- (ID#930336)		
	Width = 3.7	Weighted = 78	
	Purity = 2.3%	Reverse = 96	
	Model = 85 m/z	Corrections:	n/a
	Min. Abund. = 1.5%		
	Scan = 252		
	Peak Tailings = 1.5		
	S/N (total) = 11		
	Base Peak = 2800		
	Amount = 0.0050%		
4.0135	>Cyclobutanecarboxylic acid, 2-propenyl ester (ID#EPA-282603)		
	Width = 4.6	Weighted = 81	
	Purity = 62%	Reverse = 82	
	Model = 55 m/z	Corrections:	n/a
	Min. Abund. = 0.040%		
	Scan = 281		
	Peak Tailings = 1.9		
	S/N (total) = 129		
	Base Peak = 107789		
	Amount = 0.390%		

Chemist:

Operator:  
yzaid

4.1675	>1-Propen-2-ol, formate (ID#32978000)		
	Width = 3.5	Weighted = 86	
	Purity = 3.3%	Reverse = 86	
	Model = 55 m/z	Corrections:	n/a
	Min. Abund. = 1.3%		
	Scan = 297		
	Peak Tailings = 1.0		
	S/N (total) = 11		
	Base Peak = 3133		
	Amount = 0.00590%		
30.7374	>2-Methylheptanoic acid (ID#1188029)		
	Width = 14.2	Weighted = 64	
	Purity = 2.8%	Reverse = 64	
	Model = 74 m/z	Corrections:	n/a
	Min. Abund. = 6.0%		
	Scan = 2752		
	Peak Tailings = 1.2		
	S/N (total) = 17		
	Base Peak = 714		
	Amount = 0.00349%		

This report consists of 2 pages

AMDIS GC/MS Analysis Report  
 Organization: UTM  
 Division: Chemical Engineering

Data: 7012.SMS  
 Library: NISTMS32.DLL  
 Number of Identifications: 12

RT(min)	Chemical Name
3.0460	>2-Isopropyl-3-vinylcyclohexane (ID#7077230)
	Width = >11 Weighted = 80
	Purity = 40% Reverse = 81
	Model = 57 m/z Corrections: n/a
	Min. Abund. = 0.036%
	Scan = 185
	Peak Tailor = 10.5
	S/N (total) = 218
	Base Peak = 118089
	Amount = 0.687%
3.0470	>2-Isopropyl-3-vinylcyclohexane (ID#7077230)
	Width = >6 Weighted = 78
	Purity = 41% Reverse = 79
	Model = TIC Corrections: n/a
	Min. Abund. = 0.036%
	Scan = 185
	Peak Tailor = 5.8
	S/N (total) = 221
	Base Peak = 117684
	Amount = 0.356%
3.0936	>Propane, 2-methyl-2-nitro- (ID#594707)
	Width = >7 Weighted = 74
	Purity = 5 % Reverse = 87
	Model = 57 m/z (56) Corrections: n/a
	Min. Abund. = 0.20%
	Scan = 190
	Peak Tailor = 4.3
	S/N (total) = 62
	Base Peak = 21047
	Amount = 0.040%
3.0994	>Allyldimethylvinylsilane (ID#22146254)
	Width = >10 Weighted = 71
	Purity = 9.2% Reverse = 88
	Model = TIC Corrections: n/a
	Min. Abund. = 0.20%
	Scan = 191
	Peak Tailor = 2.2
	S/N (total) = 80
	Base Peak = 21304
	Amount = 0.014%
3.1082	>Thiophalic acid (ID#55561029)
	Width = 4.9 Weighted = 66
	Purity = 9.6% Reverse = 67
	Model = 85 m/z (57) Corrections: n/a
	Min. Abund. = 0.23%
	Scan = 192
	Peak Tailor = 1.3
	S/N (total) = 80
	Base Peak = 18110
	Amount = 0.064%
3.1247	>Azetidine (ID#503297)
	Width = >12 Weighted = 84
	Purity = 5.5% Reverse = 95
	Model = 57 m/z (69) Corrections: n/a
	Min. Abund. = 0.22%
	Scan = 193
	Peak Tailor = 5.2
	S/N (total) = 60
	Base Peak = 19102
	Amount = 0.045%

Chemist:

Operator:  
 yzid

3.1470	>1-Haptene, 3-methoxy- (ID#14093584)	
	Width = 2.2	Weighted = 45
	Purity = 1.4%	Reverse = 60
	Model = 58 m/z (57)	Corrections: n/a
	Min. Abund. = 2.2%	
	Scan = 195	
	Peak Tailoring = 1.1	
	S/N (total) = 29	
	Base Peak = 1882	
	Amount = 0.00419%	
3.3465	>1,2,4,5-Tetrazine, 1,4-diethylhexahydro- (ID#35035699)	
	Width = 4.3	Weighted = 70
	Purity = 18%	Reverse = 77
	Model = 85 m/z	Corrections: n/a
	Min. Abund. = 0.13%	
	Scan = 216	
	Peak Tailoring = 1.6	
	S/N (total) = 112	
	Base Peak = 30982	
	Amount = 0.114%	
3.5659	>Hexanoic acid, 2-oxo-, methyl ester (ID#6395831)	
	Width = 2.7	Weighted = 77
	Purity = 10%	Reverse = 82
	Model = TIC	Corrections: n/a
	Min. Abund. = 0.23%	
	Scan = 239	
	Peak Tailoring = 1.5	
	S/N (total) = 63	
	Base Peak = 18477	
	Amount = 0.00903%	
3.5688	>Allyldimethylvinylsilane (ID#22146254)	
	Width = 4.0	Weighted = 67
	Purity = 3.1%	Reverse = 79
	Model = 85 m/z	Corrections: n/a
	Min. Abund. = 0.57%	
	Scan = 239	
	Peak Tailoring = 1.0	
	S/N (total) = 35	
	Base Peak = 7506	
	Amount = 0.0108%	
3.6939	>3H-1,2,4-Triazol-3-one, 1,2-dihydro- (ID#930336)	
	Width = 4.6	Weighted = 78
	Purity = 2.7%	Reverse = 96
	Model = 85 m/z	Corrections: n/a
	Min. Abund. = 0.50%	
	Scan = 252	
	Peak Tailoring = 2.0	
	S/N (total) = 30	
	Base Peak = 8451	
	Amount = 0.00980%	
3.9906	>Oxalic acid, cyclohexyl hexyl ester (ID#EPA-309308)	
	Width = 5.1	Weighted = 83
	Purity = 50%	Reverse = 85
	Model = TIC	Corrections: n/a
	Min. Abund. = 0.027%	
	Scan = 282	
	Peak Tailoring = 1.6	
	S/N (total) = 217	
	Base Peak = 186607	
	Amount = 0.252%	

This report consists of 2 pages

## **AUTHOR'S PROFILE**



Nurul Nadia Binti Abdullah completed her Research Project in Bachelor Degree of Chemical and Bioprocess Engineering at Faculty of Chemical Engineering, Universiti Teknologi MARA, Shah Alam, Selangor.