ASSESSMENT OF PUMPKIN AND BALSAM APPLE SEED OILS AS FEEDSTOCK FOR BIODIESEL PRODUCTION

BY

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MSC/SCI/22250/2012-2013

A DISSERTATION SUBMITTED TO THE SCHOOL OF POSTGRADUATE STUDIES, AHMADU BELLO UNIVERSITY ZARIA

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF
MASTERS DEGREE IN ANALYTICAL CHEMISTRY

DEPARTMENT OF CHEMISTRY,
FACULTY OF PHYSICAL SCIENCES
AHMADU BELLO UNIVERSITY,
ZARIA, NIGERIA

MAY, 2017

DECLARATION

I declare that the work in this dissertation entitled ASSESSMENT OF PUMPKIN AND BALSAM APPLE SEED OILS AS FEEDSTOCK FOR BIODIESEL PRODUCTION has been carried out by me in the Department of Chemistry. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this dissertation was previously presented for another degree or diploma at this or any other institution.

_____ _____

JAMES YUSUF **DATE**
MSC/SCI/22250/2012-2013

CERTIFICATION

This dissertation entitled **ASSESSMENT OF PUMPKIN AND BALSAM APPLE SEED OILS AS FEEDSTOCK FOR BIODIESEL PRODUCTION BY JAMES YUSUF** meets the regulations governing the award of the degree of Master of Science in Analytical chemistry of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

This dissertation is dedicated to Almighty God and to my late lovely mother Mrs. J. Y Vasty.

ACKNOWLEDGEMENT

My most profound gratitude goes to Almighty God, who in his infinite mercies, kindness and guidance has led me to the completion of this dissertation and my academic pursuit to this level.

My appreciation and gratitude also goes to my supervisors Prof. V.O Ajibola and Dr. S.E Abechi for sparing their valuable time, advice and needed materials for the success of this research work. I appreciate them a lot and may Almighty God reward their efforts.

I will also like to extend my appreciation to the Head of Department, the entire academic and non- academic staff of the Department of Chemistry Ahmadu Bello University, Zaria for their immense contributions. Special thanks to the laboratory staff. My appreciation also goes to the staff of the National Research Institute of Chemical Technology (NARICT) Zaria especially Mr. Salisu Ibrahim who found time to work with me in the laboratory despite his tight schedule. I would also like to appreciate Mallam Usman Musa from the Department of Chemical Engineering Ahmadu Bello University Zaria for his contribution. My gratitude also goes to my colleagues in the office especially Mr. Manasseh Y. Bidan, friends, coursemate and well-wishers for their contributions. May God continue to shower his blessings upon you all.

Finally, my profound appreciation goes to my father Mr. James Yakubu, my lovely wife Elizabeth and my brothers for their priceless efforts. May Almighty God shower them with mercies and unimaginable rewards for their contributions.

ABSTRACT

This research sought to assess the potential of oils obtained from pumpkin; *C. maxima* and balsam apple, *M. balsamina* for biodiesel production. Oils were extracted from the seeds using n-hexane in a soxhlet extractor, then transesterified to biodiesel using single step alkali hydrolysis. The biodiesel so produced were analyzed for their physicochemical and fuel properties using American Standards for Testing and Materials. Fatty acid methyl esters yield was measured using GC-MS. Percentage oil yields were 42.13 ± 0.11 % and 33.21 ± 0.18 % for *C. maxima* and *M. balsamina*, respectively. The physicochemical properties of the oils showed high acid values (*C. maxima* 8.415 ± 0.8 *and M. balsamina* 9.958 ± 0.5 mg/KOH/g*)*. The percentage oil degumming for *C. maxima* was 7.6 ± 0.1 % and *M. balsamina*, 10.12 ± 0.6 %. Some critical fuel parameters for both biodiesel produced from pumpkin and balsam apple showed compliance with American Standards for Testing and Materials and European standard specifications. The result of biodiesel produced from pumpkin showed; Colour 2.1 ± 0.0, cetane number 54.148 ± 0.52, flash point 127 ± 0.00 ^{o}C, cloud point 1.1 ± 0.00 ^{o}C, pour point -0.3 ± 0.0 ^{o}C, sulphur content 0.032 ± 0.01 ppm, kinematic viscosity 4.94 ± 0.0 mm^2/s and specific gravity 0.8713 ± 0.0 g/cm^3, and for the biodiesel produced from balsam apple; Colour 2.9 ± 0.0, cetane number 57.699 ± 0.23, flash point 137 ± 0.00 ^{o}C, cloud point 1.0 ± 0.00 ^{o}C, pour point -0.4 ± 0.0 ^{o}C, sulphur content 0.021 ± 0.01 ppm, kinematic viscosity 3.82 ± 0.0 mm^2/s and specific gravity 0.8615 ± 0.0 g/cm^3. The percentage yield of biodiesel from pumpkin and balsam apple oils were 96.78 ± 0.9 and 89.12 ± 0.04 % respectively. Stearic acid methyl ester was dominant with percentage of 14.03 % in BOMEs, which is saturated. The unsaturated methyl esters oleic acid was dominant in the POMEs with a percentage of 33.18 %. The results infer that oils from *C. maxima* and *M. balsamina* possess physicochemical properties that are suitable for biodiesel production. The yields of the oils are high enough and comparable with other oils that have been used for biodiesel production.

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LIST OF ABBREVIATIONS

BOMEs	Balsam Oil Methyl Esters
POMEs	Pumpkin Oil Methyl Esters
FAME	Fatty Acid Methyl Esters
FTIR	Fourier Transform Infrared Spectrophotometer
FAO	Food and Agricultural Organization
ASTM	American Standards for Testing and Materials
EN	European Standard

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CHAPTER ONE

INTRODUCTION

1.1 Background

There is a need for alternative energy sources to petroleum based fuels due to the depletion of the world's petroleum reserves, global warming and environmental concerns caused by exploration of oil. One possible alternative to fossil fuel is the use of fuels or diesel produced from oils of plant origin like vegetable oils and tree - borne seed oils. These have been found suitable for utilization in diesel engines (Michael and Briggs, 2004).

The concept of using vegetable oil as a fuel dates back to 1895 when Dr. Rudolf Diesel developed the first diesel engine to run on vegetable oil. Diesel demonstrated his engine at the World Exhibition in Paris in 1900 using peanut oil as fuel. Vegetable oils have long been promoted as possible alternatives for fossil fuel, but it is only in recent years that systematic efforts have been made to utilize vegetable oil based fuels in engines. Fuel from these sources is technically feasible, environmentally acceptable, and readily available. Different products from vegetable oil such as pure vegetable oil, mixtures of vegetable oil with petroleum diesel as well as biodiesel have been proposed as useful alternatives (Agarwal, 1998). Much of the world uses a system known as the "B" factor to state the amount of biodiesel in any fuel mixture. For example, fuel containing 20 % biodiesel is labeled B20. Pure biodiesel is referred to as B100.

The United State Standard Specification for Biodiesel (ASTM 6751), defines biodiesel as a fuel comprising mono-alkyl esters of long chain fatty acids derived

from vegetable oils or animal fats which can be used in diesel engines and heating systems (Mittelbach, 1983).

Biodiesel has clear benefits in comparison with diesel fuel; it is a renewable fuel, nontoxic, safer to handle, biodegradable, requires no engine modifications and reduces dependency on foreign oil imports (Gerpan, 2006; Carraretto *et al*., 2004). It also has favorable combustion and emission profiles, for instance emissions of carbon monoxide (CO) and particulate matter decrease by 45 %, hydrocarbon (HC) 70 % but NOx emissions increases by 10 % with 100 % biodiesel (B100) as a fuel (Anon, 2002). The carbon cycle, time for fixation of CO_2 from biodiesel is quite small compared to mineral diesel thus contributing more to the reduction of greenhouse gas emissions compared to fossil diesel (Gerpan, 2006; Carraretto *et al*., 2004; Agarwal *et al*., 2003). Agarwal *et al.* (2003), found that biodiesel provides good lubricating properties that can reduce component wear and enhance engine life. However, biodiesel production from edible oils has generated concern as it brings global in balance to the food supply and market demand (Butler, 2006).

1.2 Research Problem Statement

This research intends to solve the problem of biodiesel production from edible sources in order to avoid the in balance in food supply. The plant seeds of pumpkin and balsam apple will be studied as feedstocks for biodiesel production by ascertaining the physicochemical properties in order to have good quality and high yield in the course of producing the biodiesel. However modification in method of biodiesel production will be employed in terms of the following conditions; time, concentration of catalyst, temperature, methanol to oil ratio and biodiesel reactor. The biodiesel to be produce will be studied for fatty acid methyl esters, fuel properties and functional groups to

see the influence of the physicochemical properties and modifications employed in the biodiesel production.

1.3 Aim and objectives of the research

1.3.1 Aim

This research work investigates the potentials of pumpkin and balsam apple seed oils as alternative feedstock for biodiesel production.

1.3.2 Objectives

The aim of this research work will be achieved through the following objectives.

i. Extraction and determination of physicochemical properties of the crude oils from pumpkin and balsam apple
ii. Production of biodiesel (methyl esters) by transesterification of the oils and compare to standards
iii. Determination of acid methyl esters and some fuel properties of the biodiesel produced and compare to standards
iv. Characterize the acid methyl esters for different functional groups present and compare to other work.

1.4 Justification

The choice of pumpkin and balsam apple seed oils as the plants of interest in this research is based on its non-edibility. It is also available in large quantity in Nigeria. The oil will provide good lubricating properties that can reduce component wear and enhance engine life.

CHAPTER TWO

LITERATURE REVIEW

2.1 Historical Development of Biodiesel

The idea to use vegetable oils as fuels is more than hundred years old. Rudolf Diesel conducted engine tests on plant oils at the World's Exhibition in Paris in 1900 (Shay, 1993). The idea for this test had apparently stemmed from the French government, which was searching for a means of domestic fuel production in its African colonies. Several other European countries took up the idea after the Exhibition, which resulted in a number of articles in different European Countries (Ankapong, 2010). In the early 1970s, however, world-wide oil crises and growing ecological awareness led to the rediscovery of plant oils as possible alternatives to hydrocarbon based fuels but with some challenges (Ankapong, 2010). These problems can be solved by either adapting the engine to the fuel or by adapting the fuel to the engine. The former led to the development of plants oil engine, while the latter led to modification of plant oils by various technologies to produce fuels which approximate the eight (8) properties and performance of fossil fuel (Schwab *et al.,* 1987).

Vegetable oils are usually triglycerides, generally with a number of branched chains of different lengths and different degrees of saturation. These oils have about 10 % lower heating value than fossil diesel due to the oxygen present in their molecules.

Eka *et al*. (2010), investigated vegetable oil properties such as oil content, acid, iodine and saponification values, density and relative density, oil viscosity and heating value for a wide range of agricultural materials; beniseed, castor and rubber seed oils. The viscosity of vegetable oil is several times higher than that of mineral diesel due to its large molecular mass and chemical structure; this can lead to unfavorable pumping

and spray characteristics. The cloud point and pour point of vegetable oils are higher and the cetane number is comparable to that of fossil fuel (Saroj and Singh, 2011; Barnwal and Sharma, 2005; Peterson and Auld 1991).

The high flash point and lower volatility characteristics of vegetable oil result in increased carbon deposit formation, injector coking, lubricating oil dilution and degradation. With vegetable oil as a fuel, short term engine performance results are comparable to those with fossil fuel. However, long term engine performance with vegetable oil lead to severe engine deposits, piston ring sticking, and injector coking and thickening (Peterson *et al*., 1983).

Vegetable oils therefore need to be modified to bring their combustion related properties closer to those of mineral diesel (Ma and Hannah, 1999). The fuel modification is mainly aimed at reducing the viscosity and increasing the volatility.

Considerable efforts have been made to develop a vegetable oil derivative or biodiesel that approximates the properties and performance of the fossil fuels. The methods employed include preheating the oil, pyrolysis, micro emulsion, blending and transesterification (Krawczyk, 1996; Ma and Hannah, 1990).

2.2 Oil Seeds Processing

2.2.1 Extraction and purification of oils

Researchers have employed different methods in the extraction of oil from agricultural materials including (i) mechanical expression, (ii) solvent extraction (iii) a combination of mechanical expression and solvent extraction methods (Akinoso and Raji, 2010). Solvent extraction method was satisfactorily employed in processing oil from seeds of low oil content such as soybeans, rice bran, mango kernels (Shukla *et al.,* 1992). Oil extraction methods and factors affecting their yield are detailed for

plant and animal materials in Davis *et al.* (2008). The extracted oil can be purified by sedimentation process. This is the easiest way to get clear oil, but it takes about a week to reduce the sediment to 20 - 25 % of the raw oil volume. The purification process can be accelerated tremendously by boiling the oil with about 20 % of water (Janulis, 2004).

2.3 Methods of Biodiesel Processing

The alternative diesel fuels must be technically and environmentally acceptable, and economically competitive. From the viewpoint of these requirements, triglycerides (vegetable oils/animal fats) and their derivatives may be considered as viable alternatives for diesel fuels. The problems with substituting triglycerides for diesel fuels are mostly associated with their high viscosities, low volatilities and polyunsaturated character. The problems have been mitigated by developing vegetable oil derivatives that approximate the properties and performance and make them compatible with the hydrocarbon-based diesel fuels through:

2.3.1 Pyrolysis

Pyrolysis is strictly defined as the conversion of one substance into another by means of heat or with the aid of catalyst (Sonntag, 1979). It involves heating in the absence of air or oxygen (Sonntag, 1979) and cleavage of chemical bonds to yield small molecules (Weisz *et al.*, 1979). The pyrolysed material can be vegetable oils, animal fat, natural fatty acids and methyl esters of fatty acids. The pyrolysis of fats has been investigated for more than 100 years especially in those areas of the world that lack deposit of petroleum (Sonntag, 1979). The first pyrolysis of vegetable oil was conducted in an attempt to synthesize petroleum from vegetable oil. Since World War

I, many investigators have studied the pyrolysis of vegetable oil to obtain products suitable for fuel (Hanna and Raimo, 2012; Denaila *et al.,* 2004; Akgoz *et al.*, 2004). The cetane number of plant oils was found to increase by pyrolysis, and the concentration of sulphur, water and sediment for the resulting products were acceptable. In modern standards, viscosity of the pyrolysed oil is considered too high, ash and carbon residue far exceed the values for fossil fuel, and the cold flow properties are poor (Schwab *et al.*, 1987).

Moreover, removal of oxygen during thermal decomposition eliminates one of the main ecological benefits of oxygenated fuels, making complete combustion due to oxygen availability in the combustion chamber of an engine more difficulty (Ma and Hannah, 1999).

2.3.2 Micro-Emulsification

The formation of micro emulsions (co-solvency) is one of the potential solutions for solving the problem of vegetable oil viscosity. Micro-emulsions are defined as transparent, thermodynamically stable colloidal dispersions. The droplet diameters in micro-emulsions range from 100 to 1000 Å. A micro-emulsion can be made of vegetable oils with an ester and dispersant (co-solvent), or of vegetable oils, an alcohol and a surfactant and a cetane improver, with or without diesel fuels. Water (from aqueous ethanol) may also be present in order to use lower-proof ethanol, thus increasing water tolerance of the micro-emulsions (Ma and Hannah, 1999).

According to Schwab *et al.* (1987), micro-emulsion is a colloidal equilibrium dispersion of optically isotropic fluid which has micro structures with dimensions generally in the 1 – 50 μm range formed spontaneously from two normally immiscible liquids and one or more ionic or non ionic amphiphiles. It can improve spray

characteristics by explosive vaporization of low boiling constituents in the micelles (Pryde, 1984). Studies by Goering *et al.* (1982) on short term performance of both ionic and non ionic micro emulsions of aqueous ethanol in the soybean oil was nearly as good as that of No.2 diesel, in spite of the lower cetane number and energy content but their durabilities were not determined. Another study by Ziejewski *et al.*, (1984) using an emulsion of 53 % (vol) alkali – refined and winterized sunflower oil, 13.3 % (vol) 190 – proof ethanol and 33.4 % (vol) 1- butanol found that the non ionic emulsion had a viscosity of 6.31 cSt at 40 ^{o}C, a cetane number of 25 and an ash content of less than 0.01 %. These values indicated that micro emulsions of vegetable oils with alcohols could not be recommended for long term use in diesel engines. The fuels produced through micro-emulsion were found to lead to incomplete combustion, the formation of carbon deposits, and an increase in the viscosity of the lubricating oil. Also micro emulsions produce considerably lower volumetric heating values compared to hydrocarbon - based diesel fuel due to their high alcohol content (Saroj and Singh, 2011). They have also been assessed insufficient in terms of cetane number and cold temperature behaviours (Maurer, 1998).

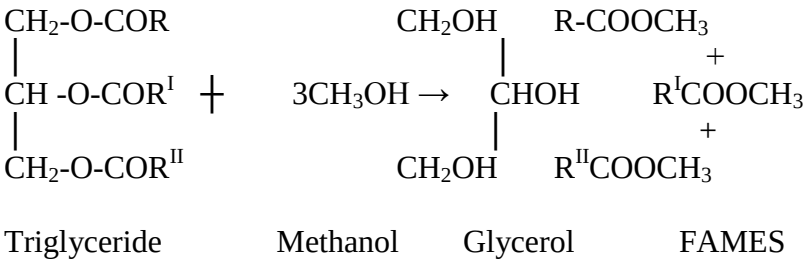
2.3.3 Dilution

Dilution of vegetable oils can be accomplished with such materials as diesel fuels, solvent or ethanol, and the diluted product used to run the engine. Caterpillar Brazil, in 1980 used pre-combustion chamber engines with a mixture of 10 % vegetable oil and 90 % petroleum diesel to maintain total power without any alteration or adjustment to the engine. Since it was not technically feasible to substitute 100 % vegetable oil for diesel fuel, more dilutions was tried and a blend of 20 % vegetable

oil and 80 % diesel fuel was found to be successful. Some short-term experiments were also tried using up to a 50/50 ratio (Singh and Singh, 2010).

2.3.4 Transesterification

Transesterification also called alcoholysis is the displacement of alkoxy group from an ester by another alcohol in a process similar to hydrolysis. This process has been widely used to reduce the viscosity of triglycerides. The reaction of triglyceride with methanol is presented in the general equation below.



Triglyceride Methanol Glycerol FAMES

Triglycerides are readily transesterified in the presence of alkaline catalyst at atmospheric pressure and at a temperature of approximately 60 to 70 °C with an excess of methanol. The mixture at the end of reaction is allowed to settle. The lower glycerol layer is drawn off while the upper methyl ester layer is washed to remove entrained glycerol and is then processed further. The excess methanol is recovered by distillation and sent to a rectifying column for purification and then recycled. Transesterification works well when the starting oil is of high quality which include low acid value, moisture content, viscosity, free fatty acid e.tc. In cases where the free fatty acid content of the oil is above 1 %, difficulties arise due to the formation of soap which promotes emulsification during the water washing stage and at FFA content above 2 % the process becomes unworkable (Atul *et al.*, 2012).

2.3.5 Storage and safety of biodiesel

As a general rule, blends of biodiesel and petroleum diesel should be treated like petroleum diesel. Biodiesel vegetable methyl esters contain no volatile organic compounds that can give rise to poisonous or noxious fumes (Khandelwal and Chauhan, 2012). There is no aromatic hydrocarbon (benzene, toluene, xylene) or chlorinated hydrocarbons. There is no lead or sulphur to react and release any harmful or corrosive gases (Khandelwal and Chauhan, 2012). However, in the case of biodiesel blends significant fumes released by benzene and other aromatics present in the base petroleum diesel fuel can persist. Biodiesel may cause eye irritation. Therefore safety glasses or face shields should be used to avoid mist or splash on face and eyes. Since it is a fire hazard, appropriate firefighting measures should be followed as per its fire hazard classification (Khandelwal and Chauhan, 2012). Hot biodiesel fuel may cause burn, soft skin and mild skin irritation so protective clothing including gloves is recommended. Even though adequate data for long term storage stability of biodiesel and its blends are not available, experience suggests a maximum of six (6) months. Some anti-oxidant additives like butylated hydroxytoluene and butylated hydroxyanisole can extend this period. Similar periods are applicable for storage of biodiesel and its blends in vehicle fuel tank. Because it is a mild solvent, biodiesel has a tendency to dissolve the sediments normally encountered in old tanks used for diesel fuel and cause filter blockage, injector failures and clogging of fuel lines (Khandelwal and Chauhan, 2012). Brass, copper, zinc e.t.c are oxidized by diesel and biodiesel fuels thus creating sediments. The fuel and fittings will start changing color as the sediments are formed. Storage tanks made of aluminum, steel and other metals that are not easily oxidized should be used (Khandelwal and Chauhan, 2012).

2.3.6 Biodegradability of biodiesel

Biodiesel is highly biodegradable in fresh water as well as soil environments and about 90–98 % of biodiesel is mineralized in 21–28 days under aerobic as well as anaerobic conditions (Saroj and Singh, 2011). Biodiesel has been reported to remove twice the amount of crude oil from sand compared to conventional shoreline cleaners (Saroj and Singh, 2011). This is because it increases the biodegradability of crude oil by means of co – metabolism. More than 98 % degradation of pure biodiesel after 28 days has been reported in comparison to 50 % and 56 % for petro diesel and gasoline respectively, in the same time. Also, the times taken to reach 50 % biodegradation reduced from 28 to 22 days in a 5 % biodiesel blend and from 28 to 16 days in case of a 20 % biodiesel blend at room temperature. The biodegradability of the blend was reported to increase with increase in biodiesel content. (Saroj and Singh, 2011).

2.4 Feed Stocks for Biodiesel Production

As shown in Fig. 2.2, four crops clearly dominate the oil feedstock used for global biodiesel production. With a share of nearly 85 %, rapeseed oil is by far leading the field, followed by sunflower seed, soybean and palm oils. The sector labeled “others” comprises of linseed oil; beef tallow and recycled frying oil (Korbitz, 1998).

It is also noteworthy (see table 2.1) that rapeseed, sunflower , soybean and palm oils constitute the four major oil crops cultivated for human consumption and various industrial applications (Gunstone, 2003). This could potentially laed to a comflict of interest between the biofuel and food industry. It is also an opportunity to expand the cultivation of these crops.

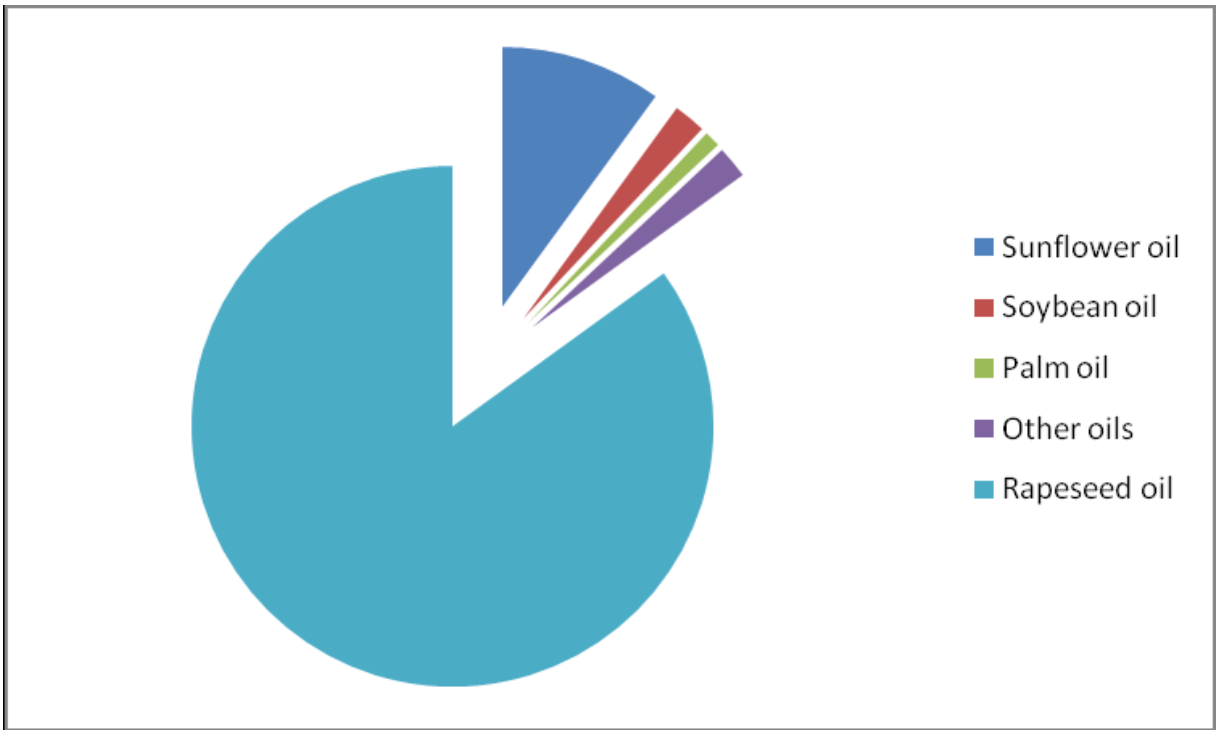


Fig.2.2: Dominant oil feedstock used for global biodiesel production

Source: (Gunstone, 2003).

Table 2.1:World-wide production of Eight major vegetable oils.

Vegetable oils	Estimated production (million metric tons)	Vegetable oils	Estimated production (million metric tons)
Soybean	31.83	Peanut	4.81
Palm	28.13	Cotton seed	3.90
Rapeseed	12.57	Palm kernel	3.50
Sunflower	9.45	Coconut	3.33

Source:(Korbitz, 1998).

2.4.1 Pumpkin

The plants of Cucurbitaceae family of which pumpkin (*C. maxima*) is a member, are generally grown in the tropical, subtropical, arid and temperate climates of the world (Fouko *et al*., 2009). A wide range of varieties are known including *C. eargysperma, C. filicifolia, C. foetidissima, C. maxima, C. mixta, C. moschata,* and *C. pepo* (Stevenson *et al*., 2007). In West Africa, pumpkins are planted in the months of April to June, the plant covers the soil surface within 4 – 6 weeks (Fouko *et al*., 2009), and yields up to 500 kg/m^2 or more depending on the variety, production system, weed competition, plant diseases and climatic circumstance (Ardabili *et al.*, 2011). Pumpkin seed oil has been extracted in Australia, Slovenia, and in Hungary (Murkovic *et al*., 1999) with variations in the range of the oil content. For example, oil content ranging from 34.5 - 43.6 % was reported in eight lines of *Cucurbita pepo* (Idouraine *et al*., 1996), 10.9 - 30.9 % for *Cucurbita maxima* (Stevenson *et al*., 2007), 27.83 % for cucurbitaceae maxima (Alfawaz, 2004), 50 % for gourds and 44 – 54 % for cucurbitaceae cultivated in Cameroun (Achu *et al.*, 2005).

2.4.2 Balsam Apple

The plants of Cucurbitaceae family of which balsam apple *M. balsamina* is a member are generally grown in the tropical regions of Africa. It is locally known as balsam apple (English), Garahuni (Hausa), Akbon-ndewe (Igbo) and Ejirin (Yoruba) (Gulab *et al.,* 2009). *M. balsamina* is a trailer plant with stems attaining 4-5 mm in length (Gulab *et al.,* 2009), which is fibrous to slightly hairy, tendril-bearing annual or perennial climber (10-12 metres), tuberous rootstock, whole plant bad-smelling (rather like the common thorn apple or Daturastramonium), more so, when bruised. It has simple tendrils leaves with lower surface paler than upper, deeply palmately 5-7-

lobed, to 12 cm long, margin toothed and stalked. It grows at an altitude of 0 to 1,293 mts (0 to 4,242 metre) with bright green leaves, pale yellow flowers round in shape and somewhat warty and solitary. Male and female flowers are monoceious. It bears striking orange to red spindle shaped fruit with 9 or 10 regular or irregular rows of cream or yellowish short blunt spines (Gulab *et al.*, 2009). When ripe, the fruits (25-60 mm) burst apart automatically into three valves that curl back (also opens when the tip is touched), revealing numerous seeds covered with a brilliant scarlet, extremely sticky coating. The seeds are embedded into a sweet edible red fleshy pulp test like water-melon (Welman, 2004).

2.4.3 Edible oils

Apart from the "great four" on biodiesel production- rapeseed, sunflower seed, soybean and palm oils, other edible plants oils have successfully been transesterified to produce biofuels. The choice of raw material cultivated in a specific region mainly depends on the respective climatic conditions. As the Mediterranean climate is not ideally suited to the cultivation of rapeseed, several alternative crops have been tested in Southern European countries, including sunflower, Ethiopian mustard and cardoon. Ethiopian mustard (*Brassica carinata*) is closely related to rapeseed. Native to the Ethiopian highlands, however, the crop is highly adaptive to low rainfall and high temperature and thus enables far higher seed yields than Brassica napus or Nigerian mustard (Cardone *et al*., 2003). Limitations to the use of *Brassica carinata* seed oil for biodiesel production have so far been its high contents of erucic and linoleic acid and the correspondingly high iodine value of about 130 $gI_2/100g$. Low erucic acid cultivars, which have recently been developed, may enhance the value of Ethiopian mustard seed meal as animal fodder, but do not display improved iodine value.

Cardoon (*Cynara cardunculus*) is a plant belonging to the Asteraceac family and is thus related to sunflower and safflower. It is native to the Mediterranean region, where it grows wild or is cultivated as fodder (Encinar *et al*., 1999). However, because its fatty acid composition is similar to that of soyabean, problems with meeting iodine value limits are to be expected.

Alternative edible oils utilized in South Asian countries include palm kernel (*Elaeis guineensis*) oil and coconut (*Cocos nucifera*)oil. Palm fruit kernels yield oil which significantly differs from palm oil (palm fruit oil) in its chemical composition, displaying a higher content of saturated short- chain fatty- acid, such as lauric and myristic acid. Palm kernel oil typically contains 600 to 1000 ppm of tocopherol and tocotienol, but only traces of carotenoids, so that it is yellowish in colour (Dembars, 2003). Both coconut oil and palm kernel oil are used in a wide arrange of dietary and 18 industrial applications (Ankapong Edward, 2010). They serve as cooking fats and starting materials for the production of margarines, shortenings and edible fats and coatings. Palm kernel oil is additionally utilized as an inexpensive substitute for cocoa butter, whereas coconut oil is used for production of detergents and cosmetics. Recently these two lauric oils have also entered the focus of attention as raw materials for biodiesel production (Abigor *et al.,* 2000). Eventhoughs their nearly 90 % content of saturated acids in their fatty acid pattern has positive effects on cetane number, it proves detrimental for cold- temperature properties such as pure biodiesel fuels based on lauric acid oils cannot be used in temperate climates.

2.4.4 Non-edible oils

One way of reducing the production cost for biodiesel fuels is the use of non-edible oil, which tend to be considerably cheaper than edible vegetable oils (Saroj and Singh,

2011). A number of plant oils contain substances which make them unsuitable for consumption. Among non-edible oils already used for biodiesel production, castor oil, pea nut oil and various oils which are produced in India deserve special attention.Castor (*Ricinus communis*) is known as a house plant, although the seed contains ricin, a highly toxic lectin. During oil recovery, ricin remains within the pressed cake. Nevertheless, the recovered oil is not fit for nutritional purposes due to its laxative effect (Roth and Kormann, 2000). This property is ascribed to the fact that castor oil contains up to 90 % of linoleic acid. Lower-cost feedstock is needed since biodiesel from food-grade oils is not economically competitive with petroleum-based diesel fuel. Non-edible plant oils have been found to be promising crude oils for the production of biodiesel. World annual petroleum consumption and vegetable oil production is about 4.018 and 0.107 billion tons, respectively (Demirbas, 2009). The cost of biodiesel and demand of vegetable oils can be reduced by non-edible oils, instead of vegetable oil.

2.5 Fatty Acid Methyl Esters (FAME)

These are fatty acid esters derived by transesterification of fats with methanol. The molecules in biodiesel are primarily FAME, usually obtained from vegetable oils by transesterification. They are used to produce detergents and biodiesel (Anneken *et al.*, 2006). FAME is typically produced by an alkali-catalyzed reaction between fats and methanol in the presence of base such as sodium hydroxide or sodium methoxide (Vyas *et al.*, 2006).

Table 2.2: Fatty acid composition of some traditional oil sources used for biodiesel production.

	12:0 (%wt)	Myristic 14:0 (%wt)	Palmatic 16:0 (%wt)	Stearic 18:0 (%wt)	Oleic 18:1 (%wt)	Linoleic 18:2 (%wt)	α-Linoleic 18:3 (%wt)	Arachidic 20:0 (%wt)	Eicosanoic 20:1 (%wt)	Behenic 22:0 (%wt)	References
Palm oil	0.5	1-2	40-48	4-5	37-46	9-11	0.3	-	-	-	Crabbe *et al.*, (2001)
Rapeseed oil	-	-	3-5	1-2	55-65	20-26	8-10	-	-	-	Ma *et al.*, (1999)
Soybean oil	-	-	11-12	3-5	23-25	52-56	6-8	-	-	-	Canakci *et al.*, (2001)
Sunflower oil	-	-	4	4	78	13	-	-	-	-	DaSilva *et al*,.(2003)
Mahua oil	-	-	16-28	20-25	41-51	8-13	-	0-3	-	-	Altin *et al*.,(2001)
Jatropha oil	-	0.5-1.4	12-17	5-9	37-63	19-41	-	-	-	-	Katwal and Soni (2003)
Karanja oil	-	-	3-7	2-8	44-71	10-18	-	-	-	-	Katwal and Soni (2003)
Neem oil	-	0.33	17	14.4	51.3	14.7	-	1.6	-	-	Radha and Manikanda (2011)
Soapnut oil	-	0.37	4.67	1.45	52.64	4.73	1.94	7.02	23.85	1.09	Chhetri*et al*.,2007

2.6 The Influence of Operating Variables of Transesterification on Biodiesel yield

2.6.1 Effect of reaction time and temperature on transesterification

The conversion rate of vegetable oils to methyl esters increases with reaction time for transesterified peanut, cotton-seed, sunflower and soybean oil under the condition of methanol–oil molar ratio 6:1, 0.5 % sodium methoxide catalyst and 60 ^{0}C (Freedman *et al.,* 1984). An approximate yield of 80 % was observed in this work after 1 min for soybean and sunflower oils. After 1 h, the conversion was almost the same for all four oils (93–98 %). Ma *et al.,* (1999), studied the effect of reaction time on transesterification of beef tallow using methanol. The reaction was very slow during the first minute due to mixing and dispersion of methanol into beef tallow. From one to 5 min, the reaction accelerated greatly with the production of beef tallow methyl esters reaching its maximum value at about 15 min. Transesterification is known to occur at different temperatures, depending on the oil used (Khandelwal and Chauhan, 2012). For the transesterification of refined oil with methanol to oil ratio of 6:1 and 1 % NaOH, the reaction was studied at three different temperatures (Freedman *et al.*, 1984). After 0.1 h, ester yields were 94, 87 and 64 % for 60 ^{o}C, 45 ^{o}C and 32 ^{o}C, respectively. After 1 h, ester formation was identical for 60 and 45^{o}C runs and only slightly lower for the 32 ^{o}C run. Temperature clearly influenced the reaction rate and yield of esters (Ma and Hannah, 1999).

Transesterification can occur at different temperatures, depending on the oil used. For the transesterification of refined oil using methanol to oil ratio (6:1) and 1 % NaOH. The rate of reaction was strongly influenced by temperature. However, given enough time, the reaction proceeded to near completion even at room temperature. Generally, the reaction is conducted close to the boiling point of alcohol at atmospheric pressure.

These mild reaction conditions require the removal of free fatty acids from the oil by refining or pre-esterification. Further increase in temperature is reported to have a negative effect on the conversion. Other studies have indicated that given enough time, transesterification can proceed satisfactorily at ambient temperatures in the case of the alkaline catalyst and it was observed that biodiesel recovery was affected at very low temperatures but conversion was almost unaffected due to low percentage of free fatty acid of starting material that's the oil (Freeman *et al.,* 1984). The conversion of oil to biodiesel approaches equilibrium as reaction times are increased, reaching a maximum conversion to ester at 2 hour reaction time. Similar reaction time had been reported with Madhuca Indica and Karanja oils, with the reaction proceeding very slow during the first minute due to mixing and dispersion of methanol into the catalyst. The reaction then accelerated greatly from 1-5min. In the production of beef tallow methyl esters, the maximum yield was reached at about 15 min (Nakpong and Wootthikanokkhan, 2010).

2.6.2 Molar ratio of alcohol to oil and type of alcohol used in biodiesel production

One of the most important variables affecting transeseterification yield is the molar ratio of alcohol to triglyceride. Stoichiometrically transesterification requires three moles of alcohol and one mole of triglyceride to yield three moles of fatty acid alkyl esters and one mole of glycerol. However, transesterification is a reversible reaction in which a large excess of alcohol is required to drive the reaction to the right. For maximum conversion to the ester, a molar ratio of 6:1 of methanol/oil should be used. The molar ratio has no effect on acid, peroxide, saponification and iodine value of methyl esters (Tomasevic and Marinkovic, 1999). However, the high molar ratio of

alcohol to vegetable oil interferes with the separation of glycerin because there is an increase in solubility. When glycerol remains in solution, it helps drive the equilibrium back to the left, lowering the yield of esters.

The transesterification of Cynara oil with ethanol was studied at molar ratios between 3:1 and 15:1. The ester yield increased as the molar ratio increased up to a value of 12:1. The best results were for molar ratios between 9:1 and 12:1. For molar ratios less than 6:1, the reaction was incomplete. For a molar ratio of 15:1 the separation of glycerin was difficult and the apparent yield of esters decreased because part of the glycerol remains in the biodiesel phase. Therefore, molar ratio 9:1 seemed to be the most desirable from a yield and separation perspective (Enciner *et al*., 2002). The base catalyzed formation of ethyl ester is difficult compared to the formation of methyl esters. Specifically the formation of stable emulsion during ethanolysis is a problem. Methanol and ethanol are not miscible with triglycerides at ambient temperature, and the reaction mixtures are usually mechanically stirred to enhance mass transfer and during reaction emulsions are usually formed. In the case of methanolysis, these emulsions quickly and easily break down to form a lower glycerol rich layer and upper methyl ester rich layer. In ethanolysis however, the emulsions are more stable and severely complicate the separation and purification of esters (Zhou *et al.*, 2003). The emulsions are caused in part by formation of the intermediates, monoglycerides and diglycerides, which have both polar hydroxyl groups and 23 non-polar hydrocarbon chains. These intermediates are strong surface active agents.

In the process of alcoholysis, the catalyst, either sodium hydroxide or potassium hydroxide is dissolved in polar alcohol phase, in which triglycerides must transfer in order to react. Methanol therefore gave the best biodiesel yield, followed by butanol and with the least yield being from ethanol (Hitesh *et al.,* 2012). There are many

reasons for this. Firstly, methanol is simpler in terms of chemical structure, thus the transesterification reaction is more likely to occur. Butanol and ethanol are more complex in terms of chemical structures; therefore giving more difficult transesterification. Secondly, the base - catalyzed formation of ethyl ester is difficult compared to that of methyl esters (Canakci and VanGerpen, 1999).

2.6.3 Catalyst type and concentration

Catalysts used for the transesterification of triglycerides are classified as alkali, acid, enzyme or heterogeneous catalysts, among which alkali catalysts like sodium hydroxide, sodium methoxide, potassium hydroxide, potassium methoxide are more effective (Ma *et al*.,1998). If the oil has high free fatty acid content and more water, acid catalyzed transesterification is suitable. The acids to be used can be sulphuric acid, phosphoric acid, hydrochloric acid or organic sulphonic acid.

Alkali metal alkoxides are the more effective transesterification catalysts compared to the acidic catalysts. Sodium alkoxides are among the most efficient catalysts used for this purpose (Khandelwal and Chauhan, 2012). Transmethylations occur approximately 4000 times faster in the presence of an alkaline catalyst than those catalyzed by the same amount of acidic catalyst (Khandelwal and Chauhan, 2012). Partly for this reason and partly because alkaline catalysts are less corrosive to industrial equipment than acidic catalysts, most commercial transesterification are conducted with alkaline catalysts. Further, increase in catalyst above a certain value concentration does not increase the conversion and it adds to processing costs because it is necessary to remove it from the mixture at the end of the reaction because some studies have shown that higher amounts of NaOH catalyst were required for higher FFA oils (Khandelwal and Chauhan, 2012).

Methanolysis of beef tallow was studied with the catalysts NaOH and NaOMe. Comparing the two catalysts, NaOH was significantly better than NaOMe (Ma *et al.*, 1998). The catalysts NaOH and NaOMe reached their maximum activity at 0.3 and 0.5 % w/w of the beef tallow, respectively. Sodium methoxide causes formation of several by-products mainly sodium salts, which are to be treated as waste. Therefore, high quality oil is required with this type of catalyst (Ahn *et al.*, 1995). The catalyst use in the process of alkaline methanolysis, mostly is sodium hydroxide or potassium hydroxide both in concentration from 0.4 to 2 % w/w of oil. Refined and crude oils with 1 % of either sodium hydroxide or potassium hydroxide catalyst resulted in successful conversion. Attempts have been made to use basic alkaline-earth metal compounds in the transesterification of rapeseed oil for production of fatty acid methyl esters. The reaction was found to proceed if methoxide ions are present in the reaction medium (Gryglewicz, 1999).

Although chemical transesterification using an alkaline catalysis process gives high conversion levelsof triglycerides to their corresponding methyl esters in short reaction times, the reaction has several drawbacks (Khandelwal and Chauhan, 2012). It is energy intensive, recovery of glycerol is low, the acidic or alkaline catalyst has to be removed from the product, alkaline waste water requires treatment, and free fatty acid and water interfere with the reaction. Enzymatic catalysts like lipases, on the other hand are able to effectively catalyze the transesterification of triglycerides in either aqueous or non-aqueous systems, and can overcome the problems mentioned above (Fuduka *et al.*, 2001). In particular, the by-products, glycerol can be easily removed without any complex process, and the free fatty acids contained in the waste oils and

fats can be completely converted to alkyl esters. In general, the production cost of a lipase catalyst is significantly greater than that of an alkaline one.

2.6.4 Purity of reactants

Impurities present in oils also affect conversion levels, which result in 67 to 84 % conversion to ester when compared with 94 to 97 % when using refined oils (Khandelwal and Chauhan, 2012). The free fatty acids in the original crude oils interfere with the catalyst. However, under conditions of high temperature and pressure this problem can be overcome. In another study it was observed that crude oils were equally good when refined for production of biodiesel, because oil quality is very important in this regard (Hitesh *et al.,* 2012).

2.7 Effect of Physicochemical Parameters on Biodiesel Production and Quality

2.7.1 Mixing intensity

Mixing the two phases is most significant during the slow rate period of the transesterification reaction. As the single phase is established, mixing becomes insignificant. The understanding of the mixing effects on the kinetics of the transesterification process is a valuable tool in the process scale-up and design. It has reported that after adding alcohol and catalyst to the oil, 5-10 minutes stirring helps in higher rate of conversion and recovery (Hitesh *et al.,* 2012).

2.7.2 Moisture content

Water could pose a greater negative effect than the presence of free fatty acids and hence the feedstock should be water free (Khandelwal and Chauhan, 2012). Even a

small amount of water (5 %) in the transesterification reaction would decrease the ester conversion from vegetable oil by 6 % (Kusdiana and Saka, 2004). The yield of the alkyl ester decreases due to presence of water and FFA as these two impurities cause soap formation, consume catalyst and reduce the effectiveness of catalyst. Moisture content from the vegetable oil is usually removed by heating in oven for 1 h at 383 K (Khandelwal and Chauhan, 2012). Meher *et al,* (2006) reported a precautionary step to prevent moisture absorbance and maintenance of catalytic activity by preparing the fresh solution of potassium hydroxide and methanol. It was found that even a small amount of water in the feedstock or from esterification reaction producing water from FFA might cause reduction in conversion to fatty acid methyl ester and formation of soap instead. Surprisingly, it was found that the presence of water had a positive effect on the yield of methyl esters when room-temperature methanol was replaced by supercritical methanol (Khandelwal and Chauhan, 2012). However, no explanation for this has been provided. It has also been reported that the presence of water had negligible effect on the conversion while using lipase as a catalyst (Saroj and Singh, 2011).

2.7.3 Free fatty acids and moisture content

Free fatty acids (FFAs) content after acid esterification should be minimal or otherwise less than 2 % FFAs. These FFAs react with the alkaline catalyst to produce soaps instead of esters (Khandelwal and Chauhan, 2012). There is a significant drop in the ester conversion when the free fatty acids are beyond 2 % (Saroj and Singh, 2011). Free fatty acids and moisture content are key parameters for determining the viability of the vegetable oil in the transesterification process. To carry the base catalyzed reaction to completion; a free fatty acid (FFA) value lower than 3 % is

needed. The higher the acidity of the oil, the smaller is the conversion efficiency and vice - versa (Dorado *et al.,* 2002). Ma *et al,* (1998), studied transesterification of beef tallow catalyzed by the NaOH in the presence of free fatty acids and water. Without adding FFA and water, the apparent yield of beef tallow methyl esters (BTME) was highest. When 0.6 % of FFA was added, the apparent yield of BTME reached its lowest, less than 5 %, with any level of water added. When 0.9 % of water was added, without addition of FFA, the apparent yield was about 17 %. If low quality beef tallow or vegetable oil, with high FFA is used to make biodiesel fuel, they must be refined by saponification using NaOH solution to remove the free fatty acids. Conversely, the acid catalyzed process can also be used for esterification of these free fatty acids but with lower yields.

The starting materials used for base catalyzed alcoholysis should meet certain specifications. The triglycerides should have lower acid value and all materials should be substantially anhydrous. The addition of more sodium hydroxide catalyst compensates for higher acidity, but the resulting soap causes an increase in viscosity or formation of gels that interfere in the reaction as well as with separation of glycerol (Freedman *et al.,* 1984). When the reaction conditions do not meet the above requirements, ester yields are significantly reduced. The methoxide and hydroxide of sodium or potassium should be maintained in anhydrous state. Prolonged contact with air will diminish the effectiveness of these catalysts through interaction with moisture and carbon dioxide. Most biodiesels are currently made from edible oils by using methanol and an alkaline catalyst. The problem with processing these low cost oils and fats is that they often contain large amounts of free fatty acids that cannot be converted to biodiesel using alkaline catalyst. Therefore, two-step esterification process is required for these types of feed stocks. Initially the FFA of these can be

converted to fatty acid methyl esters by an acid catalyzed pretreatment and in the second step transesterification is completed by using alkaline catalyst (Canakci and VanGerpen, 2001).

2.7.4 Stirring

Stirring can play an important role in the yield of biodiesel production. Transesterification reaction was carried out with 180, 360 and 600 revolutions per minute (rpm) and incomplete reaction was reported (Khandelwal and Chauhan, 2012), with 180 rpm. The yield of methyl ester was same with 360 and 600 rpm. Then mode of stirring also play vital role in the transesterification reaction. The yield of biodiesel increased from 85 % to 89.5 % when magnetic stirrer (1000 rpm) was replaced with mechanical stirrer (1100 rpm). A plausible explanation may be a thorough mixing of the reactants by mechanical stirrer (Meher *et al.,* 2006).

2.7.5 Specific gravity

Lower value of the specific gravity of the final product is an indication of completion of reaction and removal of heavy glycerine. The influence of molar ratio, temperature and catalyst quantity on the specific gravity of the biodiesel was studied by Miao and Wu (2006). The specific gravity of the product decreased sharply up to 2 h of reaction time using 30:1 molar ratio and up to 4 h of reaction time using 45:1 and 56:1 molar ratio after which it was almost constant. The best process combination of reaction time and methanol to oil ratio reduced the product specific gravity from 0.912 to 0.864 with 100 % catalyst, 56:1 molar ratio at 303 K in 4 hrs of reaction time (Saroj and Singh, 2011).

2.8 Stability of Biodiesel

Biodiesel ages more quickly than fossil diesel fuel due to the chemical structure of fatty acid esters present in biodiesel (Khandelwal and Chauhan, 2012). There are three types of stability criteria, which need to be studied:(a) oxidation stability (b) thermal stability and (c) storage stability poor oxidation and thermal stability can cause fuel thickening, formation of gum and sediments and may also affect engine performance (Khandelwal and Chauhan, 2012). Current knowledge and database is still sparse. It is desirable to carry out tests on biodiesel from different feedstock available and generate data in relation to fuel composition. Very little data is available on the long-term storage stability of bio-diesel.

2.9 Fuel Properties and Specification of Biodiesel

Since biodiesel is produced in quite differently scaled plants from vegetable oils of varying origin and quality, it was necessary to specify a standardization of fuel quality to guarantee engine performance without any difficulties. Austria was the first country in the world to define and approve the standards for rapeseed oil methyl esters as diesel fuel. As standardization is a prerequisite for successful market introduction and penetration of biodiesel, standards or guidelines for the quality of biodiesel have also been defined in other countries like Germany, Italy, France, the Czech Republic and in the United States.

The parameters, which define the quality of biodiesel, can be divided into two groups. One group contains general parameters, which are also used for mineral oil based fuel, and the other group especially describes the chemical composition and purity of fatty acid alkyl esters. Table 2.3 summarizes some of the important quality criteria.

Table: 2.3 International quality standards on pure biodiesel fuels. Source: (Meher *et al.*, 2006)**.**

Parameters	Austria (ON)	Czech (CSN)	France	Germany (DIN)	Italy (UNI)	USA (ASTM)
Density at 15 ^{o}C g/cm3	0.85–0.89	0.87–0.89	0.87–0.89	0.875–0.89	0.86–0.90	-
Viscosity at 37.8 mm^{2}/s	3.5–5.0	3.5–5.0	3.5–5.0	3.5–5.0	3.5–5.0	1.9-6.0
Flash point (^{o}C)	100	110	100	110	100	130
Pour point (^{o}C)	-	-5	-10	-	0-5	-
Cetane	≥49	≥48	≥49	≥49	-	≥47
Iodine number	≤120	-	≤115	≤115	-	-
Acid value (mgKOH/g)	≤120	≤0.5	≤0.5	≤0.5	≤0.5	≤0.5

2.9.1 Viscosity

Among the general parameters for biodiesel, viscosity controls the characteristics of the injection from the diesel injector. The viscosity of fatty acid methyl esters can go to very high levels and hence it is important to control it within an acceptable level to avoid negative impacts on fuel injector system performance (Saroj and Singh, 2011).

2.9.2 Flash point

Flash point of a fuel is the temperature at which it will ignite when exposed to a flame or spark. The flash point of biodiesel is higher than that of petrodiesel, and hence safer for transport purpose (Saroj and Singh, 2011).

2.9.3 Melt point or pour point

Melt or pour point refers to the temperature at which the oil in solid form starts to melt or pour. In cases where the temperatures fall below the melt point, the entire fuel system including all fuel lines and fuel tank will need to be heated. (Saroj and Singh, 2011).

2.9.4 Cloud point

The temperature at which oil starts to solidify is known as the cloud point. While operating an engine at temperatures below oil's cloud point, heating will be necessary in order to avoid waxing of the fuel (Saroj and Singh, 2011).

2.9.5 Cetane number /Aniline point

Cetane number is indicative of its ignition characteristics. The cetane number measures how easily ignition occurs and the smoothness of combustion. The higher

the cetane numbers the better the ignition properties. Cetane number affects a number of engine performance parameters like combustion, stability, driveability, white smoke, noise and emissions of CO and HC. Biodiesel has higher cetane number than conventional diesel fuel, and therefore results in higher combustion efficiency (Saroj and Singh, 2011).

2.9.6 Sulphur Percentage

This is the percentage by weight, of sulfur in the fuel Sulfur content is limited by law to very small percentages value for diesel fuel used in on road applications (Saroj and Singh, 2011).

2.10 Process Theory of Degumming

Theoretically, phospholipids, proteins and carbohydrates, vegetable gums and colloidal components have negative influence towards the storage of oil. They are considered as undesirable substances in refining because they increase the oil loss and hamper other operations. Oils that have certain amount of these substances therefore should be degummed in order to remove all those substances. Two kinds of phospholipids exist; those that are hydratable and those that are not (non-hydratable phosphatides -NHP). Hydratable phospholipids can be removed easily by the addition of water. The process can be conducted rapidly at elevated temperature or slowly at low temperature. However the temperature should stay below the temperature at which the phospholipids hydrate starts to become liquid crystals (usually ~ 40 ^{o}C). Phospholipids lose their lipophilic character by taking up water and become lipophobic and thus precipitate from oil (Bockish, 1998). The non-hydratable phospholipids can be converted to hydratables. The conversion of non-hydratable

phospholipids to hydratable is done usually through acidulation followed by neutralization. Traditionally, acids that are being used are usually sufficiently strong to hydrate phospholipids without hydrolyzing the triglycerides. At present, citric or phosphoric acid is normally being used for any type of vegetable oil. However, phosphoric acid is more preferred by the palm oil refiners in Malaysia because of lower unit cost and easier handling (Thiagarajan and Tang, 1991). Phospholipids are present in relatively small quantities of about 5 – 130 ppm in palm oil as compared with other vegetables oils. Sambanthamurthi *et al.* (2000) reported that the solvent extracted mesocarp oil usually contained 1000-2000 ppm phospholipids. However it is only present at level of 20-80 ppm in commercial crude palm oil. Phospholipids have been reported to show antioxidant effects. Their antioxidant-synergistic effects can be attributed to the sequestering of soluble prooxidant metal ions to form inactive species. Hydratable insoluble metal ions could also be dispersed by phospholipids through miscellar action. Since phospholipids and glycolipids cause reverse micelle, vesicle or emulsion droplet formation, phospholipids can remove metal ions and their hydrophilic salts from the lipid phase to reduce oxidation.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials

3.1.1 Chemicals used

Methanol (99.5 %), (Analytical Grade BDH)

Ethanol (99.5 %), (Analytical Grade BDH)

Normal hexane (99.5 %), (Analytical Grade BDH)

Wij's reagent

Carbon tetrachloride, (Analytical Grade BDH)

Diethyl ether (95 %), (Analytical Grade BDH)

Isopropyl alcohol (99.5 %), (Analytical Grade BDH)

Hydrochloric acid (95 %), (Analytical Grade BDH)

Potassium iodide (Analytical Grade BDH)

Anhydrous sodium thiosulphate (Analytical Grade M&B)

Potassium hydroxide (Analytical Grade M&B)

3.1.2 Equipment used

Gas Chromatography – Mass Spectrophotometer (QP2010)

Fourier Transform Infrared Spectrophotometer (8400S)

Viscometer

Hydrometer

Lovibond Tintometer

Flash point machine (Seta Multiflash pensky- martens)

3.2 Sample Collection and Preparation

Samples were collected from ripped seeds of pumpkin; *C. maxima* and balsam apple; *M. balsamina* in Kawo Area within Kaduna metropolis. The leaves and seeds were identified in the Herbarium section of Biological Science Department, Ahmadu Bello University Zaria. Voucher numbers 1139 and 2238 for balsam apple and pumpkin, respectively were kept for future reference. The seeds were peeled to obtain the kernels, which was air dried and pulverized to a fine powdered form and stored in air tight plastic containers.

3.3 Extraction Procedure

Two hundred gram (200 g) of air dried plant seeds of pumpkin; *C .maxima* and balsam apple*; M. balsamina* were weighed out and packed into a thimble, which were in turn placed in two separate Soxhlets extractors. Extracting solvent n-hexane (500 cm^3) and anti-bumping chips were put into a 1000 cm^3 round bottomed flask and heated on heating mantle at 60 ^{0}C. The extraction was allowed to continue until the solvent was clear. The solvent in the round bottomed flask was collected and concentrated in vacuo using a rotatory evaporator at 40 ^{0}C. The process was repeated to obtain the mean of the percentage extraction and enough oils for further analysis.

$$\% \text{ Extraction} = \frac{\text{Weight of oil extracted}}{\text{Weight of sample}} \times 100 \quad \text{---------- (i)}$$

3.4 Determination of Physicochemical Properties of the Vegetable Oils

3.4.1 Determination of the saponification value of vegetable oils

The American Standard for Testing and Material (ASTM) method- (D 5558-95) was used for the determination of the saponification values of the vegetable oils. The oil (5 g) was weighed into Erlenmeyer flask and 0.5 M ethanolic KOH was prepared by dissolving 7 g of KOH in 250 cm^3 ethanol and 25 cm^3 of the prepared 0.5 M ethanolic KOH was added and the resulting mixture was refluxed for 60 minutes. The resulting solution was subsequently titrated against 0.5 M HCl prepared by diluting 10.7 cm^3 HCl in 250 cm^3 of distil water using phenolphthalein as indicator. The resulting end point was obtained when the pink colour changed into colourless. The same procedure was used for the blank. The Saponification value (SV) was then calculated using the expression;

$$\text{Saponification value (S.V.)} = \frac{56.1\,(B-S) \times M \text{ of HCl}}{\text{Weight of sample}} \qquad \text{---------- (ii)}$$

(*Source*: **ASTM-D 5558 (95)**)

where,

B – Vol. of HCl required by blank

S – Vol. of HCl required by sample

M – Molarity of HCl

56.1– Molar mass of KOH

3.4.2 Determination of acid value of vegetable oils

Acid values of the oils were determined by ASTM method (ASTM – D 974(00). The oil (0.5 g) was weighed into 250 cm^3 conical flask and 50 cm^3 of neutralized ethyl

alcohol was added, prepared by neutralizing a solvent mixture of 25 cm^3 ethanol and 25 cm^3 diethyl ether with 0.1 M ethanolic KOH prepared by dissolving 1.4 g KOH in 250 cm^3 of ethanol using phenolphthalein as indicator. The mixture was added to the oil and heated on a water bath to dissolve the oil. The solution was then titrated against 0.1 M KOH prepared by dissolving 1.4 g KOH in 250 $cm^3$of distill water using phenolphthalein as indicator. The acid value was determined after which the free fatty acid was calculated respectively as follows;

$$\text{Acid Value} = \frac{A \times M \times 56.10}{W} \qquad \text{---------- (iii)}$$

(*Source*: **ASTM-D 974 (00)**)

where,

A = Vol. of 0.1 M KOH consumed by sample

M = Molarity of KOH

W = weight in grams of the sample

Then,

$$\text{Free fatty acid} = \frac{\text{Acid Value}}{2} \qquad \text{---------- (iv)}$$

3.4.3 Determination of iodine value of the vegetable oils

The oil (0.5 g) was weighed into conical flask and 20 cm^3 of carbon tetrachloride was added to dissolve the oil. 25 cm^3 of Wijs reagent was added into the flask using a measuring cylinder in a fume chamber and a stopper was inserted, the content of the flask was vigorously swirled and kept in the dark for 35 minutes. 20 cm^3 of 10 % aqueous potassium iodide prepared by diluting 10 cm^3 of potassium iodide in 90 cm^3 of distill water was added into the content of the flask using a measuring cylinder. The

content was titrated with 0.1 M sodium thiosulphate solution prepared by dissolving 3.95 g of anhydrous $Na_2S_2O_3$ in 250 cm^3 of distilled water. Few drops of 1 % starch indicator were added and the titration continued by adding the sodium thiosulphate drop wise until coloration disappeared after vigorously shaking. The same procedure was used for the blank test. The Iodine Value (I.V) is given by the expression;

$$\text{Iodine Value (I.V)} = \frac{126.9\ C\ (V1 - V2)}{M} \qquad \text{---------- (v)}$$

where,

C = concentration of sodium thiosulphate

V1 = volume of sodium thiosulphate used for blank

V2 = volume of sodium thiosulphate used for determination

M = mass of sample

12.69 = Constant.

3.4.4 Determination of refractive index of vegetable oils

Abbey refractometer was used in this determination. A drop of the sample was transferred into a glass slide of the refractometer. Water at 30 ^oC was circulated round the glass slide to keep its temperature uniform. Through the eye piece of the refractometer, the dark portion viewed was adjusted to be in line with the intersection of the cross. At no parallax error, the pointer on the scale pointed to the refractive index. This was repeated and the mean value noted and recorded as the refractive index.

3.4.5 Determination of density of vegetable oils

The densities of the oils were determined by ASTM - D 1298 (99) .The samples were brought to a specified temperature of 70 ^{0}C and a test portion was transferred to a hydrometer cylinder that had been brought to approximately the same temperature. The appropriate hydrometer, also at a similar temperature, was lowered into the test portion and allowed to settle. After temperature equilibrium has been reached, the hydrometer scale reading and the temperature of the test portion were taken. The observed hydrometer reading was reduced to the reference temperature by means of a petroleum measurement table. Any hydrometer correction was applied to the observed reading and the corrected hydrometer scale reading recorded to the nearest 0.1 kg/m^3 as density.

3.5 Pre-treatment of the Oils (Acid Degumming)

Two hundred (200 gram) of *Cucurbita maxima* oil was weighed and heated to 70 ^{0}C on a heating mantle. 25 % citric acid was prepared by dissolving 250 g citric acid in one liter of distil water and was added in the ratio of 3:1 citric acid to oil then mixed for 5 min. This was centrifuge for 30 min for separation of gums and other impurities which settled at the bottom. Oil at the top of the centrifuge test tubes, was decanted and reheated. The process was repeated for *Momordica balsamina* oil sample.

3.6 Transesterification

A modified method of biodiesel reaction was employed in terms of timing, catalyst, temperature, methanol to oil ratio and a biodiesel reactor, which differs from the convectional biodiesel reaction (Bello *et al.,* 2013). 100 g of the pretreated oil was heated to 60 ^{0}C and transferred into the jurk of a master chep blender. 20 g methanol

containing potassium hydroxide 1 % w/w (1g) of oil to form potassium methoxide was added. The stirring speed was kept constant at 10000 rpm using a time interval of 2, 4 and 6 mins to avoid over heating the blender. 16 g potassium methoxide solution was initially added representing 80 % and stirred for 4 min and the remaining 4 g was added at the 4 th min representing 20 % to complete the 6 th min, because the reaction was very slow at the beginning due to mixing and dispersion of alcohol into the feedstock.

3.7 Settling and Washing

After the reaction was complete, the mixture was poured into the separating funnel and allowed to settle overnight. The mixture was separated into two liquid layers, crude ester layer on top and glycerol layer at the bottom. (The density of glycerol is much higher than that of ester, and it is insoluble in the ester phase.) Glycerol was flowed out from the separatory funnel, and the remaining ester layer was washed three times with warm distilled water to remove soap, residual alcohol, catalyst, and unreacted tri, di-, and monoglycerides. The number of washing processes was not increased because increasing the washing beyond three times does not have further benefit. The volume of each of the purified biodiesel produced was measured and the biodiesel yield (vol. %), was calculated.

3.8 Determination of Fuel Properties of Biodiesel

3.8.1 Determination of pour point

The pour point was determined by ASTM method (ASTM D – 97).

The oil sample was poured into the test jar to the level mark. The test jar was closed with the cork carrying the high – pour thermometer. The position of the cork and the

thermometer were adjusted for the cork to fit tightly, the thermometer and the jar were coaxial and the thermometer bulb was immersed 3 mm below the surface of the sample. After this, the test jar was placed into the cooling medium. The sample was cooled at a specified rate and examined at interval of 3 ^{o}C for flow characteristics until a point was reached at which the sample showed no movement when the test jar was held in a horizontal position for 5 seconds. The observed readings of the thermometer were recorded. 3 ^{o}C was added to the recorded temperature and the result was recorded as the pour point

3.8.2 Determination of kinematic viscosity

The temperature of the viscometer bath was adjusted to 38.9 ^{o}C. A calibrated thermometer was held in upright position and inserted into the bath by a holder. A clean dry calibrated viscometer was selected and carefully flushed with a dry nitrogen gas to remove the moist room air. A sample of the biodiesel was drawn up into the working capillary of the viscometer and the timing bulb was then allowed to drain back as an additional safeguard against moisture condensing or freezing on the walls. The charged viscometer was inserted into the bath at a depth such that at no time during the measurement of the flow time was any portion of the sample in the viscometer less than 20 mm below the surface of the bath. The viscometer together with its content was allowed to remain in the bath for 30 minutes to reach the test temperature (38.9 ^{o}C). A suction bulb was used to adjust the head level of the biodiesel to a position in the capillary arm of the viscometer about 7 mm above the first timing mark. The biodiesel was then allowed to freely flow and the time required for the meniscus to pass from the first to the second timing marks was noted with a stop watch. The procedure was repeated to make a second measurement of flow time

and the average of these determinations was used to calculate the kinematic viscosity. The viscometer was thoroughly cleaned with sample solvent and dried by vacuum. The procedure was repeated for the other samples of the biodiesel (ASTM D 445-97)

Calculation:

$$v = C \times t \qquad \text{---------- (vi)}$$

Where,

v = kinematic viscosity, (mm^2/s)

C = calibration constant of the viscosity, (mm^2/s)

t = mean flow time

3.8.3 Determination of acid value of the biodiesel

The biodiesel (25.0 g) was weighed into a clean titration beaker. A magnetic stirrer was carefully placed into the beaker to prevent splashing of sample. 100 cm^3 of isopropyl alcohol was poured into the beaker and 0.1 cm^3 of p-naphtholbenzene indicator solution was added. The titration beaker was assembled with purging stopper previously connected with a rotameter and nitrogen purge gas. The burette tip was inserted into the beaker 10 mm above the surface of the solution through the hole in the stopper.

The purge gas was opened and allowed to flow through the beaker at a rate of 10 L/h for 30 seconds. The stirring rate to mix the solution efficiently was adjusted to prevent splashing or mixing the purge gas into the solution. The sample was titrated with the standardized 0.01 M KOH titrant at 28 ^oC. The end point was noted when the orange colour of the indicator changed to a stable green colour. The titration was conducted using the same procedure but this time omitting the sample (blank titration). (ASTM D-3339-95)

Calculation:

Calculate the acid number of the sample as follows:

$$\text{Acid number} = \frac{\text{mg KOH/g } 56.10\ M\ (A - B)}{W} \quad \text{---------- (vii)}$$

where,

A = millilitres of titrant required for titration of the sample

B = millilitres of titrant required for titration of the blank

M = molarity of the titrant

W = grams of sample used

3.8.4 Determination of density of biodiesel

The densities of the oils were determined by ASTM - D 1298 (99) method. The sample was brought to a specified temperature of 70 ^{o}C and a test portion was transferred to a hydrometer cylinder that had been brought to approximately the same temperature. The appropriate hydrometer, also at a similar temperature, was lowered into the test portion and allowed to settle. After temperature equilibrium has been reached, the hydrometer scale reading and the temperature of the test portion were taken. The observed hydrometer reading was reduced to the reference temperature by means of a petroleum measurement table. Any hydrometer correction was applied to the observed reading and the corrected hydrometer scale reading recorded to the nearest 0.1 kg/m^3 as density.

3.8.5 Determination of colour

The visual determination of the colour of the biodiesel was done using ASTM D – 1500 (98). The readings of the colour were made with Lovibond Tintometer. Prior to this, a sample container filled to a depth of 50 mm with distilled water was placed in the compartment of the colorimeter with the standard glasses to facilitate color adjustment. The biodiesel sample was then placed in its container in the middle of the compartment. The containers were covered to exclude all exterior light. The light source was switched on and the colour of the sample compared with the standard glasses ranging from 0.5 to 4.5. When an exact colour match was not found and the sample colour was between two standard colours, the higher of the two colours was reported.

3.8.6 Determination of cloud point

The cloud point was determined using ASTM D2500. A cylindrical test tube was filled with the biodiesel to a specific level (5 cm^3) and clamped with a wooden clamp bearing thermometer. The test tube was placed on the ice/salt bathe and the set up inspected at intervals for cloud formation. The temperature at which a distinct cloudiness appeared at the bottom of the test tube was observed and recorded as the cloud point of the biodiesel.

3.8.7 Determination of flash point

The flash point was determined using ASTM D93. Seta MultiflashPensky-Martens Flash Point Module Part Number 34100-2 was used to determine the flash point. The automatic PMCC module conforms precisely to national and international Pensky-Martens Closed Cup flash point test methods. It comprises a heated cup and lid, and a

DIPS pod containing the dipping mechanism, gas and electric ignitors, fire detection system and a stirrer. The Pensky- Martens module was used with the Multiflash Universal Base unit (p/n 34000-0). The base unit recognises the Pensky-Martens module is connected and instantaneously sets up standard test parameters and calibration data

3.8.8 Determination of cetane number

The cetane number was determined based on the formula proposed by Demirbas (2009) as there was no enough biodiesel to carry out the test. The formula is given below.

$$CN = \frac{46.3 + 5458}{S.V - 0.225 \times I.V} \quad \text{---------- (viii)}$$

Where,

S.V = Saponification of the biodiesel

I.V = Iodine Value of biodiesel

3.8.9 Determination of specific gravity

Specific gravity bottle was washed, rinsed with acetone and dried at room temperature in a dessicator and the weight of the empty bottle determined using an electronic weighing balance. The weight of the bottle filled with water was recorded. The same procedure was repeated with the oil and the specific gravity computed as follows;

$$\text{Specific gravity} = \frac{W2 - W1}{W3 - W1} \quad \text{---------- (ix)}$$

where,

W1 = weight of empty bottle

W2 = Weight of bottle + oil

W3 = weight of bottle + water

CHAPTER FOUR

RESULTS

4.1 Physicochemical Properties

The result of physicochemical properties of the studied oils are presented as mean ± standard deviation, which reveal that the oil content of both pumpkin and balsam apple are promising for biodiesel production with pumpkin (42.13 ± 0.1 %) having higher oil content with balsam apple (33.21 ± 0.18 %). The saponification value, iodine value, peroxide value, relative density and unsaponifiable value of pumpkin is higher compared to balsam apple, while the acid value, refractive index, free fatty acid of pumpkin is lower than that of balsam apple as shown in Table 4.1.

4.2 Percentage Degumming

The result of percentage degumming is presented as mean ± standard deviation, which reveals that *Cucurbita maxima* 7.6 ± 0.1 % is lower compared to *Momordica balsamina* 10.12 ± 0.6 % as shown in Table 4.2

4.3 Fatty Acid Methyl Esters Analysis

The analysis revealed the presence of myristic, nonenoic, palmitic, linoleic, capric, olei, stearic, arachidic, behenic, azelaic, alaidic, cerotic, eicosanoic, eicosatrienoic and linolenic acids as shown in Table 4.3 and 4.4

4.4 Fuel Properties

The result of fuel properties are presented as mean ± standard deviation, which shows that the colour, specific gravity, kinematic viscosity, pour point, cloud point, flash

point, cetane number and sulphur content for both balsam oil methyl esters and pumpkin oil methyl esters are within the limits of ASTM and EN as shown in Table 4.5. The FTIR spectral of POMEs and BOMEs are shown in Fig. 4.1 and 4.2.

Table 4.1: Result of physicochemical properties of the seed oils (Mean ± SD)

Oil property	Unit	*C. maxima* (Pumpkin)	*M. balsamina* (Balsam Apple)
Oil content	Percent	42.13 ± 0.1	3 3.21 ± 0.18
Saponification value	mgKOH/g	10.38 ± 0.1	2 07.57 ± 0.02
Iodine value	gI_2/100g	82.74 ± 0.4	171.31 ± 0.59
Peroxide value	Meq/kg	2.5 ± 0.06	1 ± 0.2
Acid value	mgKOH/g	8.415 ± 0.8	9.958 ± 0.5
Relative density	Dimensionless	0.883 ± 0.0	0.874 ± 0.0
Refractive index	Dimensionless	1.463 ± 0.0	1.481 ± 0.0
Free fatty acid	Percent	4.207 ± 0.0	4.979 ± 0.0
Unsaponifiable value	mgKOH/g	3.20 ± 0.4	2.98 ± 0.6
Viscosity	mm^2/sec	25.0 ± 0.0	27.4 ± 0.0

Table 4.2.: Percentage degumming of the oils (Mean ± SD)

Oil Type	% Gums layer after Centrifugation
C. maxima	7.6 ± 0.1 %
M. balsamina	10.12 ± 0.6 %

Table 4.3: GC-MS result for biodiesel produced from pumpkin

FATTY ACID	CARBON NUMBER	MOLECULAR FORMULAR	% COMPOSITION
Myristic Acid	14:0	$C_{15}H_{30}O_2$	0.85
Nonenoic Acid	9:0	$C_{10}H_{18}O_2$	0.66
Palmitic Acid	16:0	$C_{17}H_{34}O_2$	17.64
Linoleic Acid	18:2	$C_{19}H_{34}O_2$	17.73
Capric Acid	10:0	$C_{11}H_{22}O_2$	0.85
Oleic Acid	18:1	$C_{18}H_{34}O_2$	33.18
Stearic Acid	18:0	$C_{19}H_{38}O_2$	13.28
Arachidic Acid	20:0	$C_{21}H_{42}O_2$	3.40
Behenic Acid	22:0	$C_{23}H_{46}O_2$	1.78
Non-methyl esters	----------	----------	10.63
Total	----------	----------	100

Table 4.4: GC-MS result for biodiesel produced from balsam apple

FATTY ACID	CARBON NUMBER	MOLECULAR FORMULAR	% COMPOSITION
Azelaic Acid	C10:2	$C_{11}H_{20}O_2$	0.72
Linoleic Acid	C18:2	$C_{19}H_{34}O_2$	11.48
Palmitic Acid	C16:0	$C_{17}H_{34}O_2$	7.94
Stearic Acid	C18:0	$C_{19}H_{38}O_2$	14.03
Alaidic Acid	C18:1	$C_{19}H_{36}O_2$	8.43
Cerotic Acid	C26:1	$C_{27}H_{54}O_2$	10.18
Behenic Acid	C22:0	$C_{23}H_{46}O_2$	10.18
Eicosanoic Acid	C20:1	$C_{21}H_{42}O_2$	4.68
Eicosatrienoic Acid	C20:2	$C_{21}H_{54}O_2$	8.09
Linolenic Acid	C18:2	$C_{19}H_{32}O_2$	9.67
Non-methyl esters	-------	----------	14.6
Total	-------	----------	100

Table 4.5 Fuel properties of POMEs and BOMEs (Mean ± SD)

Fuel parameter	POMEs	BOMEs	ASTM Limits	European Standard
(%)Yield of Biodiesel	96.78 ± 0.9	89.12 ± 0.06	-	-
Colour	2.1 ± 0.0	2.9 ± 0.0	3.5	-
Specific gravity g/cm^3	0.8713 ± 0.0	0.8615 ± 0.0	-	-
Kinematic viscosity (mm^2/s)	4.94 ± 0.0	3.82 ± 0.0	6.0 Max	3.5-5.0
Pour point (^{o}C)	-0.3 ± 0.0	-0.4 ± 0.0	NS	NS
Cloud Point (^{o}C)	1.1 ± 0.0	1.0 ± 0.0	NS	NS
Flash point (^{o}C)	127 ± 0.0	137 ± 0.0	130 min	<120
Sulphur content (ppm)	0.03 2 ± 0.01	0.021 ± 0.01	0.050	-
Cetane number	54.148 ±0.52	57.699 ± 0.23	Min47	-

NS= Not specified

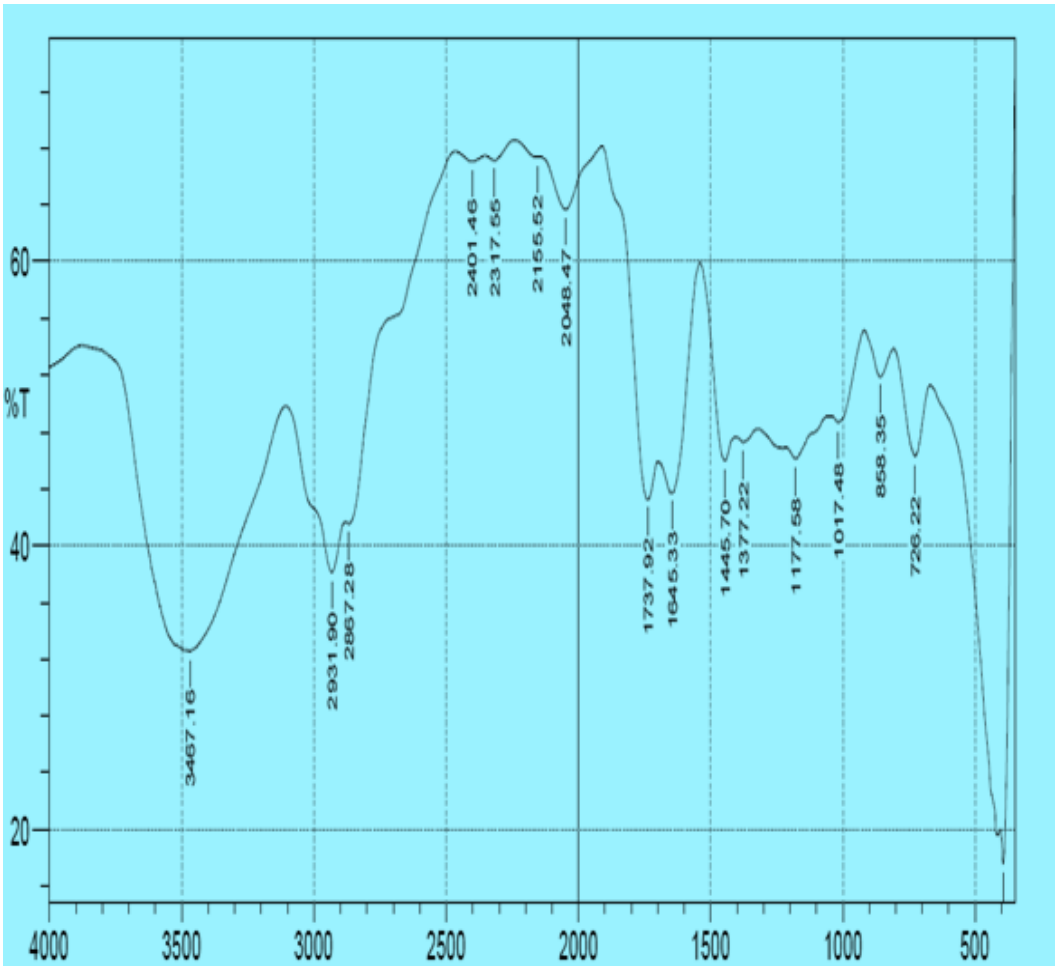


Fig.4. 1: FTIR Result for biodiesel produced from balsam apple

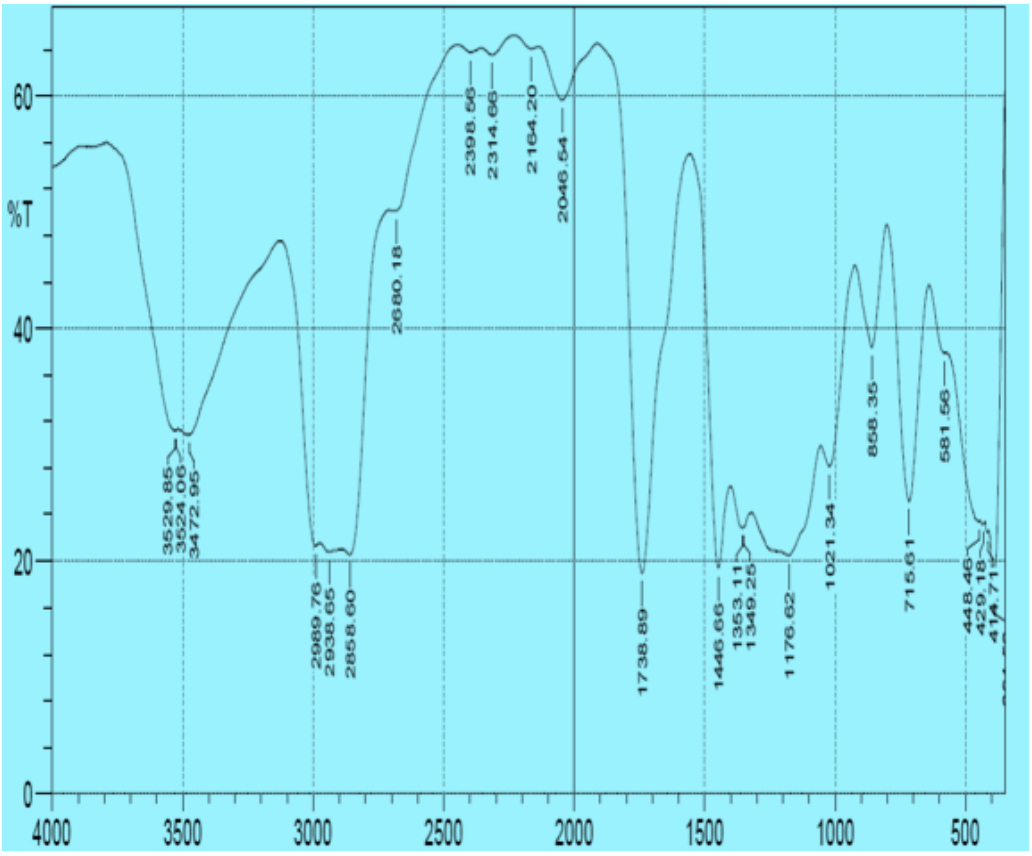


Fig. 4.2: FTIR Result for biodiesel produced from pumpkin

CHAPTER FIVE

DISCUSSION

5.1 Physicochemical Properties of the produced oils

Results of the physical and chemical properties of *Momordica balsamina* and *Cucurbita maxima* oil seeds studied are presented in Table 4.1. The percentage oil content of *Momordica balsamina* and *Cucurbita maxima* are 33.21 ± 0.18 % and 42.13 ± 0.11 % respectively. All the oils are liquids at room temperature (30 ^{o}C). The oil contents in this study were higher than the oil content of soybeans (19 – 21 %), mustard seed (33 – 41.5 %), cottonseed (15 – 20 %) reported by Shukla *et al.,* (1992) but lower than the oil content of *C. maxima* (55.8 %) and *C. pepo* (52.8 %) reported by Bwade *et al*., (2013) which may be likely due to difference in solvent use in the oil extraction. According to Food and Agricultural Organization (FAO) as reported by Akinoso and Raji (2010), any seed containing greater than 17 % of oil is considered to be an oil seed and can be utilsed as feedstock for biodiesel production, as such *Cucurbita maxima* and *M*omordica *balsamina* seeds are good feedstock for biodiesel.

The analysis revealed differences in the densities, specific gravities, refractive indexes, viscosities, saponification values, iodine values, peroxide values, unsaponifiable matter, acid values and free fatty acid of the studied oils.

Pumpkin (*Cucurbita maxima*) oil was found to be having high relative density 0.883 ± 0.0 than balsam apple (*Momordica balsamina*) with 0.874 ± 0.0 due to their difference in fatty acid composition, minor components and temperature (Fakhri and Qadir, 2011).

Pumpkin (*Cucurbita maxima*) oil is having a lower refractive indices of 1.463 ± 0.0 compared to balsam apple (*Momordica balsamina*) with a refractive indices of 1.473 ± 0.0. However, both the oils have refractive index lower than that of beniseed oil (1.474) (Akpan *et al.,* 2006) which may be due to variation in wavelength and temperature, degree of unsaturation and substitutions of component fatty acids. Since refractive index of an oil is the ratio of speed of light at a defined wavelength to its speed in the oil/fat itself (Aremu *et al.,* 2015).

The acid value of *Cucurbita maxima* was 8.415 ± 0.8 mgKOH/g and lower than the acid value of *Momordica balsamina* (9.958 ± 0.5 mgKOH/g). The lower the acid value, the lower the free fatty acid, the more suitable, the oil for transesterification process. The high acid value observed in *Momordica balsamina* may have resulted from the effect of the surface to volume area ratio on the drying time of the seed. Similar effect was reported by Fokou *et al.,* 2009. The acid value of oil from *Momordica balsamina* and *Cucurbita maxima* were higher in comparison to the acid value of Agusi 4.0 mgKOH/g (Mabaleha *et al.,* 2007) and rubber seed oil 8.17 mgKOH/g (Eka *et al.,* 2010) which indicates that the oil will be unstable over a long period of time and will not be protected against rancidity and peroxidation. This could be attributed to lack of natural antioxidants in the seeds such as vitamins C and A as well as other possible phytochemicals like flavonoids (Aremu *et al.,* 2015).

The saponification value for *Cucurbita maxima* and *Momordica balsamina* were 207.57 ± 0.1 mgKOH/g and 210.38 ± 0.02 mgKOH/g respectively, which are lower when compared with *cocos nucifera* or coconut oil 246 mgKOH/g (Eka *et al.,* 2010).

However biodiesel derived from oil with high saponification value causes exhaust emissions during burning in the engine (ASAE, 1999).

The iodine value of *Cucurbita maxima* and *Momordica balsamina* were found to be 182.74 ± 0.4 gI_2/100g and 171.31 ± 0.59 gI_2/100g respectively, which are higher than the iodine value of refined 87.72 gI_2/100g and unrefined castor oil 84.8 gI_2/100g (Akpan *et al.*, 2006). This indicates that both the oils studied are suitable for biodiesel production since low iodine value of vegetable oil produces biodiesel with high cloud and pour points; higher cloud and pour points means poor engine performance in cold temperatures (Mabaleha *et al*., 2007).

The viscosity for *Cucurbita maxima* and *Momordica balsamina* were found to be 25.0 ± 0.0 mm^2/sec and 27.4 ± 0.0 mm^2/sec respectively, which are lower compared to the viscosity of *Cucurbita pepo* oil 93.65 mm^2/sec (Younis *et al*., 2000) and *black benised* seed oil 33.2 mm^2/sec (Furiku *et al*., 2007). According to Nouredini *et al*., 1992 viscosity increases with molecular weight, as such *Momordica balsamina* is having high molecular weight since its viscosity is higher than *Cucurbita maxima* which in turn will affect injection lubrication, increase engine deposits and fuel atomization (Edward and Peggy, 2013)

The peroxide value of *Cucurbita maxima* was found to be 2.5 ± 0.06 Meq/kg. This is higher than that of *Momordica balsamina* 1 ± 0.2 Meq/kg and both are considered lower to Melon seed oil 5.63 Meq/kg (Olaofe *et al*., 2012) and higher than groundnut seed oil 0.74 Meq/kg (Ayoola and Adeyeye, 2010).This shows that *Momordica balsamina* will have less level of deterioration when attacked or exposed to oxygen,

since peroxide value indicates the level at which deterioration will take place as a result of oxidation owing to the availability of oxygen during storage (Abayeh *et al*., 2013).

The unsaponifiable value for *Cucurbita maxima* and *Momordica balsamina* were found to be 3.20 ± 0.4 mgKOH/g and 2.98 ± 0.6 mgKOH/g respectively, which is higher compared to African pear seed oil 2.62 mgKOH/g and Soy bean seed oil 0.837 mgKOH/g (Akanni *et al*., 2005). The high level of the unsaponifiable matter in the oils indicates the presence of good lubricating properties (Ihekoronye and Ngoddy, 1985).

5.2 Percentage Degumming

The percentage degumming after centrifugation shown in Table 4.2 reveal that *Cucurbita maxima* is having 7.6 ± 0.1 % and *Momordica balsamina* 10.12 ± 0.6 %. These are above the percentage degumming of crude Canola oil 4.50 ± 0.0 % and crude Sunflower oil 3.20 ± 0.0 % (Nick and Greg, 2008). This indicates that there is high level removal of hydratable and non-hydratable phosphatides, and other impurities especially metals contamination (P and Ca specifically) and also improvement in the yield of biodiesel production (Van Gerpen and Dvorak, 2002).

5.3 Fatty acid methyl esters

The profile of fatty acid methyl esters in POMEs shown in Table 4.3 indicates oleic acid ester as the dominant ester with one level of unsaturation. Oleic acid ester is dominant in Jatropha, mahua, neem, soapnut and karaja seeds oil as reported by Khandelwal and Chauhan (2012). However, linoleic acid ester with two level of

unsaturation is the dominant methyl esters in soybean, sunflower, rape and grape seeds oil (Ramos *et al.,* 2009). Biodiesel produced from this feedstock could deteriorate on long storage due to its susceptibility to oxidation. The higher the level of unsaturation the more the instability of the biodiesel produced (Bello *et al.,* 2013). Conversely, the presence of palmitic, myristic, stearic, and archidic acids as methyl esters in the biodiesel will help to enhancing the stability of the biodiesel, been saturated compounds and less susceptibility to peroxidation (Sokoto *et al.,* 2013). It is also observed that POMEs have less long chain of the acid methyl esters which in turn will have less effect on the fuel properties of the biodiesel produced (Bello *et al.,* 2013).

The result of fatty acid methyl esters in BOMEs shown in Table 4.4 indicate that stearic acid methyl esters been the dominant ester will help in enhancing the stability of the biodiesel, it is also a saturated compound and will have less susceptibility to peroxidation (Sokoto *et al.,* 2013). It is also observed that balsam oil methyl ester is have more long chain of the acid methyl esters compared to pumpkin oil methyl ester, and as such the long chain of balsam oil methyl ester will have effect on the fuel properties of the biodiesel produced (Bello *et al.,* 2013).

5.4 Fuel properties

The fuel properties of the POMEs and BOMEs produced is shown in Table 4.5. The percentage yield were recorded to be 96.7827.4 ± 0.9 and 89.1227.4 ± 0.04 % respectively, which are higher than the percentage yield of biodiesel produced from the oil seeds of *Gul Mohr* 87 % (Edward and Peggy, 2013). However, *Lagenaria vulgaris* with 96.52 % yield is higher than balsam oil methyl esters and lower than

pumpkin oil methyl esters (Sokoto *et al*., 2013). The kinematic viscosity of the fatty acid methyl esters corresponded to biodiesel specification for both ASTM and European Standard limits, which indicate the presence of short chain unsaturated methyl fatty acid esters and is likely to produce less deposit when burnt in combustion engines (Sokoto *et al*., 2013)**.**

The flash point of POMEs and BOMEs conforms to the ASTM and (EN 2003) for biodiesel limit. This shows that the biodiesel produced has fewer tendencies to ignite accidentally, since flash point is the lowest fuel temperature at which application of ignition source causes the vapor of the fuel sample to ignite under the prescribed test conditions and also a parameter used to assess the overall flammability hazard of fuel (Knothe *et al.,* 2005).

The cetane number of the BOMEs and POMEs were found to correspond to the biodiesel specification for ASTM Standard limits. The higher the cetane number the better it is in its ignition properties. Biodiesel has higher cetane number than conventional diesel fuel, which results in higher combustion efficiency (Saroj and Singh, 2011).

The pour point of the BOMEs and POMEs were found to be -0.327.4 ± 0.0 ^{o}C and -0.427.4 ± 0.0 ^{o}C respectively. There is no standard specification for pour point due to variation in atmospheric condition.

The cloud points of both BOMEs and POMEs were found to be 1.1 ± 0.0 ^{o}C and 1.0 ± 0.0 ^{o}C respectively. Cloud point test characterizes the low temperature operability of

diesel fuel. There is no standard specification for cloud point due to variation in atmospheric condition.

The specific gravity of BOMEs and POMEs were found to be 0.8713 ± 0.0 g/cm^3 and 0.8615 ± 0.0 g/cm^3 respectively; these are lower than the specific gravity of biodiesel produced from *Lagenaria Vulgaris* with specific gravity of 0.8879 g/cm^3 (Sokoto *et al*., 2013). This indicates that the biodiesel produced can be handled and stored safely. It is an important physical property in handling and storing of fuels (Abayeh *et al.,* 2013).

The sulphur content of both the BOMEs and POMEs where 0.032 ± 0.01 ppm and0.021 ± 0.01 ppm respectively, and were found to correspond to the biodiesel specification for ASTM Standard limit of 0.050 ppm. This indicates that the biodiesel produced could emit less SOx upon combustion (Edward and Peggy, 2013).

5.5 IR spectra

Figures 4.1 and 4.2 showed the IR spectra of POMEs and BOMEs respectively. Primarily, the biodiesel composed of mono-alkyl esters of fatty acids. This is confirmed by the presence of an intense band of C=O stretching of methyl ester and O-CH_3 group in the spectra. The absorption band attributed to O-CH_3 group confirms that the transesterification process has taken place (Darwin *et al.,* 2010). The FTIR spectra of the biodiesels show several major absorption bands at 1738 cm^{-1}, 1176 cm^{-1}, 2938 cm^{-1} and 1446 cm^{-1} which agrees with the findings by Sanford *et al.* (2009). This is also confirmed by the absorption spectra having strong ester peaks at 1750 cm^{-1} (C=O vibration) and around 1170 – 1200 cm^{-1} (C-O vibrations) and similar

transmittance pattern within the range 4000 cm^{-1} to 500 $cm^{-1}$of the biodiesels produced as reported in literature (Sanford *et al,*. 2009).

CHAPTER SIX

SUMMARY CONCLUSIONS AND RECOMMENDATIONS

6.1 Summary

The analysis of the oils, revealed variations in the oil contents, densities, specific gravities, refractive indices, viscosities, saponification values, iodine values, peroxide values, unsaponifiable matter, acid values and free fatty acid of the oils. The profile of POMEs and BOMEs reveal the presence of both saturated and unsaturated methyl esters and fuel properties determined showed compliance with ASTM and EN specifications. The IR spectra showed the presence of an intense band of C=O stretching of methyl ester and O-CH_3 groups

6.2 Conclusions

The oil contents of both the plants seed shows economic viability of feedstock for biodiesel production and the physicochemical properties of the oils are promising with the exception of saponification and free fatty acid values which were above their limits for biodiesel production. The percentage of biodiesel production from the oils were high, with *Cucurbita maxima* having the highest percentage yield of biodiesel production and less long chain methyl esters. The FTIR showed the intense band of C=O stretching of methyl ester and O-CH_3 group for both the biodiesels and the fuel properties agree with the ASTM and EN Standard limits.

6.3 Recommendations

6.3.1 Recommendation on the use of the oils produced

The percentage oil extraction yielded 42.13 ± 0.11 % and 33.21 ± 0.18 % for pumpkin and balsam apple plant seeds respectively, which is good for utilization in biodiesel production since it is greater than 17 % according to Food and Agricultural Organization

6.3.2 Recommendation for feature work

i. Optimal condition on the yield and quality of biodiesel produced from pumpkin and balsam apple oils should be investigated for higher yield and quality

ii. The high level of unsaturated fatty acid methyl esters of biodiesel produced from balsam apple and long chains of methyl esters should be investigated which usually affect fuel properties.

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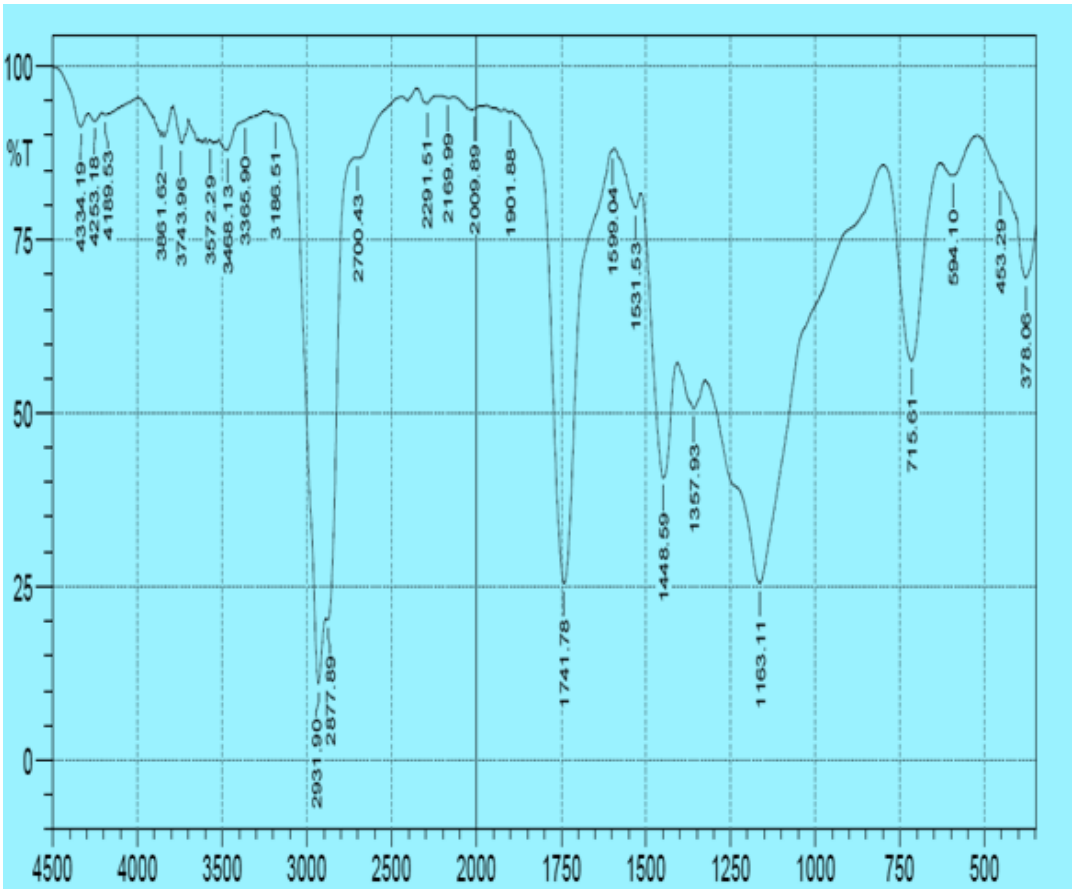
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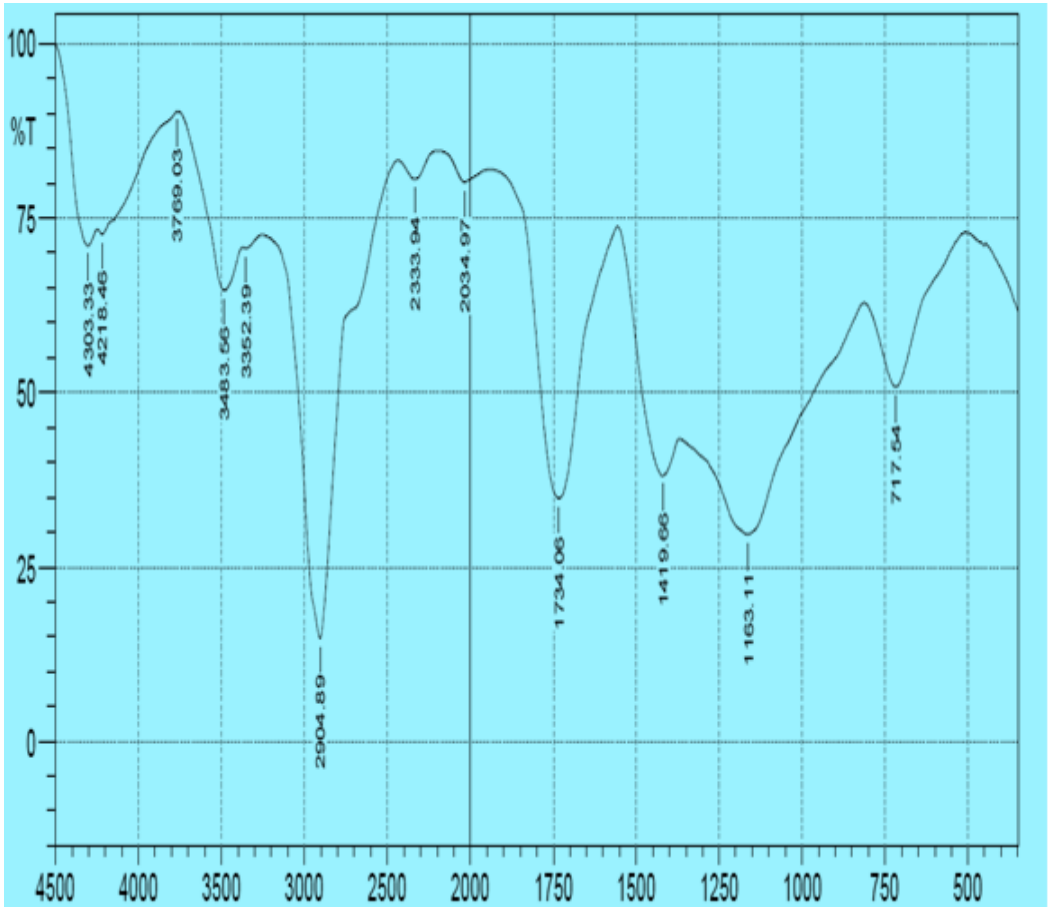
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APPENDIX I



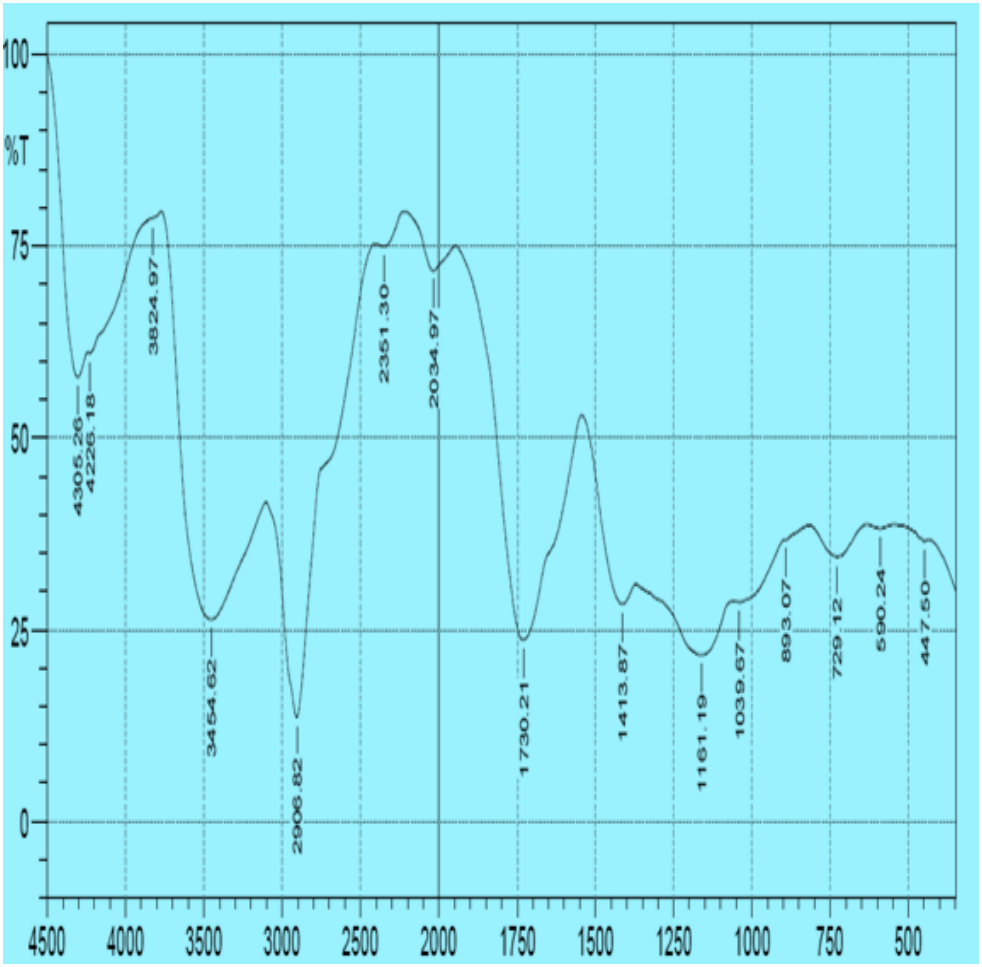
FTIR Result of Degummed Pumpkin

APPENDIX II



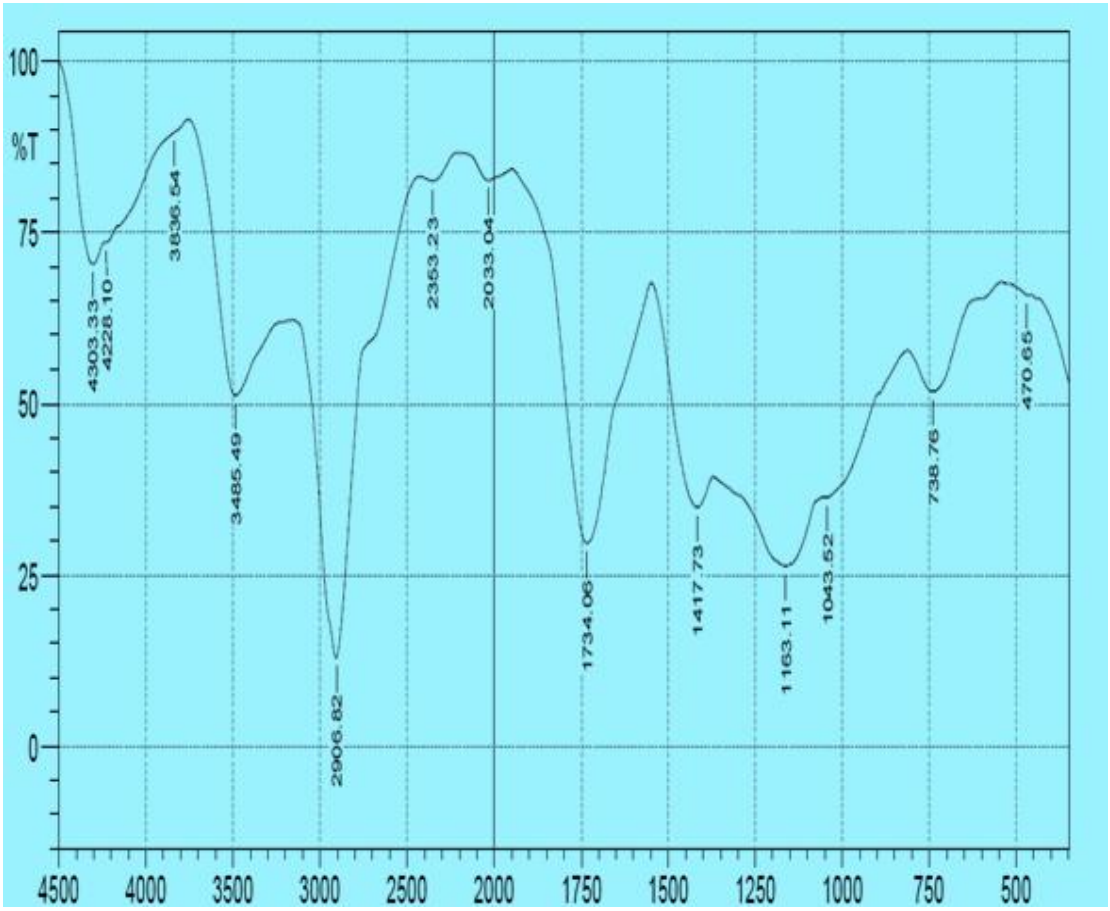
FTIR Result of Undegummed Pumpkin

APPENDIX III



FTI Result of Degummed Balsam apple

APPENDIX IV



FTIR Result of Undegummed Balsam apple

APPENDIX V

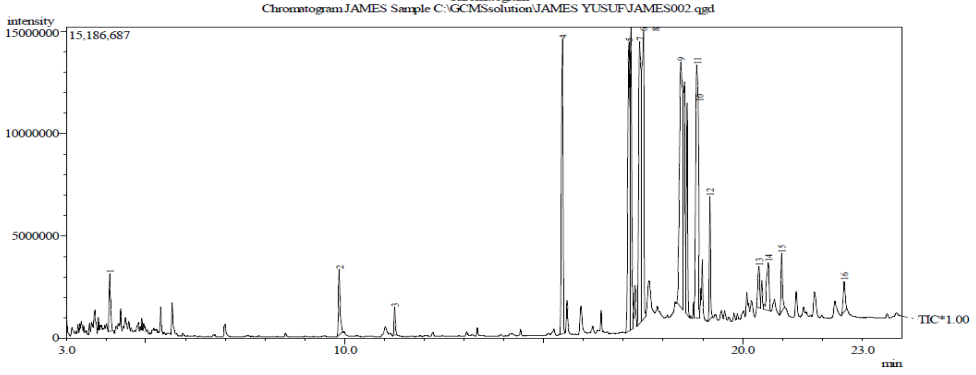
GC-MS Result of biodiesel produced from balsam apple

GCMS-QP2010 PLUS
SHIMADZU,JAPAN

NARICT, ZARIA
GCMS ANALYSIS

JAMES YUSUF (SAMPLE -BALSAM APPLE)

Chromatogram
Chromatogram JAMES Sample C:\GCMSsolution\JAMES YUSUF\JAMES002.qgd



Method

[Comment]

═══ Analytical Line 1 ═══

[AOC-20i]

# of Rinses with Presolvent	:4
# of Rinses with Solvent(post)	:4
# of Rinses with Sample	:3
Plunger Speed(Suction)	:High
Viscosity Comp. Time	:0.2 sec
Plunger Speed(Injection)	:High
Syringe Insertion Speed	:High
Injection Mode	:Normal
Pumping Times	:5
Inj. Port Dwell Time	:0.3 sec
Terminal Air Gap	:No
Plunger Washing Speed	:High
Washing Volume	:8uL
Syringe Suction Position	:0.0 mm
Syringe Injection Position	:0.0 mm
Use 3 Solvent Vial	:1 vial

[GC-2010]

Column Oven Temp.	:70.0 °C
Injection Temp.	:250.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:116.9 kPa
Total Flow	:40.8 mL/min
Column Flow	:1.80 mL/min
Linear Velocity	:49.2 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0
High Pressure Injection	:OFF
Carrier Gas Saver	:OFF
Splitter Hold	:OFF

Oven Temp. Program:

Rate	Temperature(°C)	Hold Time(min)
-	70.0	0.00
10.00	280.0	5.00

< Ready Check Heat Unit >
Column Oven : Yes
SPL2 : Yes
MS : Yes
< Ready Check Detector(FID) >
< Ready Check Baseline Drift >
< Ready Check Injection Flow >
SPL2 Carrier : Yes
SPL2 Purge : Yes
< Ready Check APC Flow >
< Ready Check Detector APC Flow >
External Wait :No
Equilibrium Time :3.0 min

[GC Program]

[GCMS-QP2010 Plus]
IonSourceTemp :200.00 °C
Interface Temp. :250.00 °C
Solvent Cut Time :2.50 min
Detector Gain Mode :Relative
Detector Gain :0.00 kV
Threshold :2000

[MS Table]
--Group 1 - Event 1--
Start Time :3.00min
End Time :24.00min
ACQ Mode :Scan
Event Time :0.50sec
Scan Speed : 666
Start m/z :30.00
End m/z :350.00

Sample Inlet Unit :GC

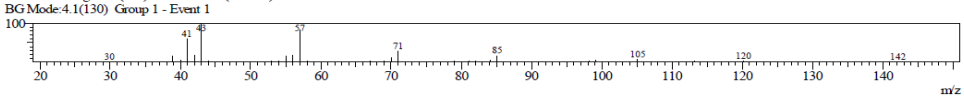
[MS Program]
Use MS Program :OFF

Peak Report TIC

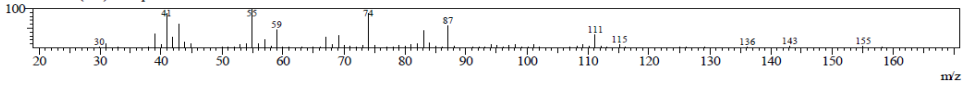
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	4.091	4.058	4.167	6798238	1.46	2810664	2.20	2.42		
2	9.863	9.817	9.950	9526590	2.05	3186527	2.49	2.99		
3	11.255	11.217	11.317	3332900	0.72	1357875	1.06	2.45		
4	15.467	15.400	15.517	36896553	7.94	14390885	11.26	2.56		
5	17.139	17.058	17.167	53379664	11.48	14110212	11.04	3.78		
6	17.191	17.167	17.258	39190706	8.43	14768353	11.56	2.65	V	
7	17.410	17.342	17.458	65236394	14.03	13747286	10.76	4.75		
8	17.505	17.458	17.558	47322277	10.18	14053763	11.00	3.37	V	
9	18.446	18.350	18.508	75652374	16.27	11981200	9.38	6.31		
10	18.601	18.567	18.667	23302811	5.01	10339804	8.09	2.25	V	
11	18.846	18.783	18.917	59704309	12.84	12352903	9.67	4.83	V	
12	19.175	19.117	19.242	13006093	2.80	5985592	4.68	2.17		
13	20.404	20.342	20.450	7355752	1.58	2027860	1.59	3.63		
14	20.642	20.517	20.708	11202157	2.41	2344669	1.83	4.78	V	
15	20.978	20.925	21.050	7777515	1.67	2828478	2.21	2.75		
16	22.544	22.492	22.617	5255497	1.13	1491699	1.17	3.52		
				464939830	100.00	127777770	100.00			

Spectrum

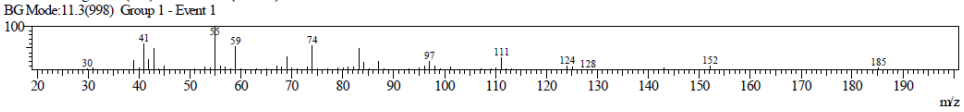
Line#:1 R.Time:4.1(Scan#:132)
MassPeaks:51
RawMode:Single 4.1(132) BasePeak:43(443352)
BG Mode:4.1(130) Group 1 - Event 1



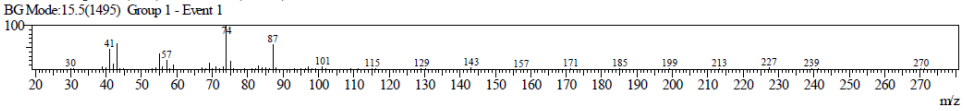
Line#:2 R.Time:9.9(Scan#:825)
MassPeaks:75
RawMode:Single 9.9(825) BasePeak:55(133552)
BG Mode:9.9(827) Group 1 - Event 1



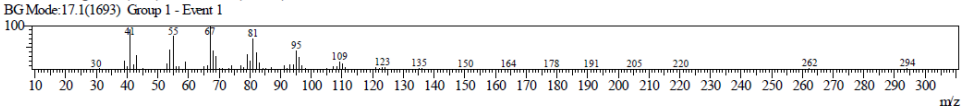
Line#:3 R.Time:11.3(Scan#:992)
MassPeaks:67
RawMode:Single 11.3(992) BasePeak:55(172798)
BG Mode:11.3(998) Group 1 - Event 1



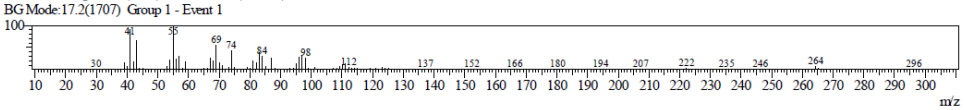
Line#:4 R.Time:15.5(Scan#:1497)
MassPeaks:103
RawMode:Single 15.5(1497) BasePeak:74(823377)
BG Mode:15.5(1495) Group 1 - Event 1



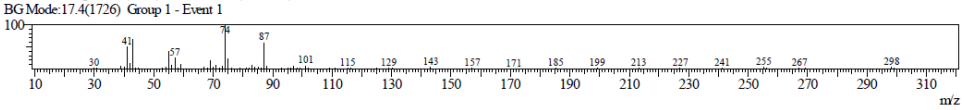
Line#:5 R.Time:17.1(Scan#:1698)
MassPeaks:147
RawMode:Single 17.1(1698) BasePeak:67(850352)
BG Mode:17.1(1693) Group 1 - Event 1



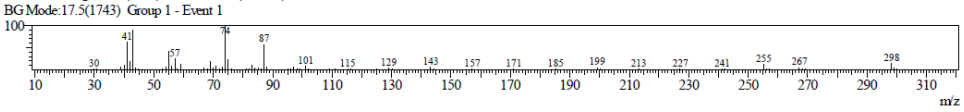
Line#:6 R.Time:17.2(Scan#:1704)
MassPeaks:148
RawMode:Single 17.2(1704) BasePeak:55(971301)
BG Mode:17.2(1707) Group 1 - Event 1



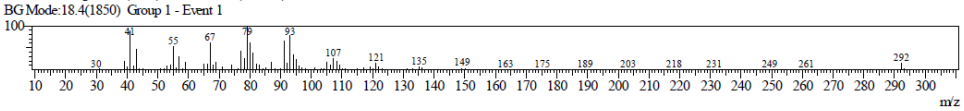
Line#:7 R.Time:17.4(Scan#:1730)
MassPeaks:111
RawMode:Single 17.4(1730) BasePeak:74(1672379)
BG Mode:17.4(1726) Group 1 - Event 1



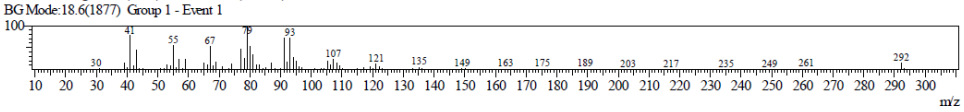
Line#:8 R.Time:17.5(Scan#:1742)
MassPeaks:108
RawMode:Single 17.5(1742) BasePeak:74(486512)
BG Mode:17.5(1743) Group 1 - Event 1



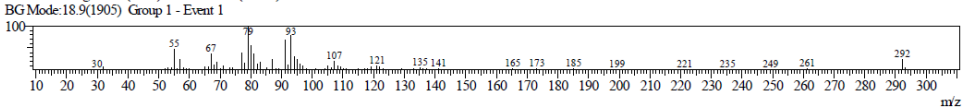
Line#:9 R.Time:18.4(Scan#:1854)
MassPeaks:136
RawMode:Single 18.4(1854) BasePeak:79(425426)
BG Mode:18.4(1850) Group 1 - Event 1



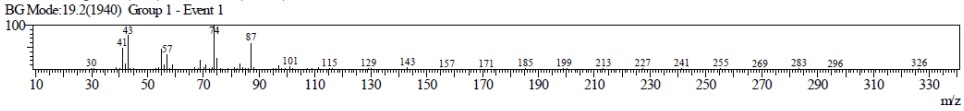
Line#:10 R.Time:18.6(Scan#:1873)
MassPeaks:131
RawMode:Single 18.6(1873) BasePeak:79(870078)
BG Mode:18.6(1877) Group 1 - Event 1



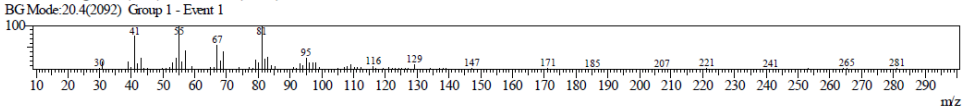
Line#:11 R.Time:18.9(Scan#:1903)
MassPeaks:124
RawMode:Single 18.9(1903) BasePeak:79(96357)
BG Mode:18.9(1905) Group 1 - Event 1



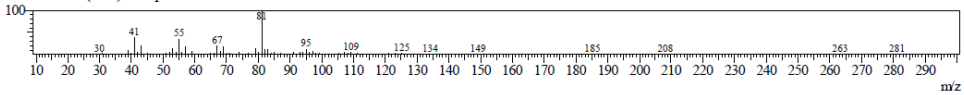
Line#:12 R.Time:19.2(Scan#:1942)
MassPeaks:103
RawMode:Single 19.2(1942) BasePeak:74(503346)
BG Mode:19.2(1940) Group 1 - Event 1



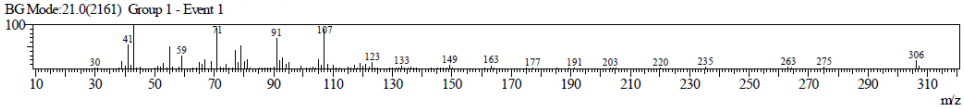
Line#:13 R.Time:20.4(Scan#:2090)
MassPeaks:95
RawMode:Single 20.4(2090) BasePeak:81(97597)
BG Mode:20.4(2092) Group 1 - Event 1



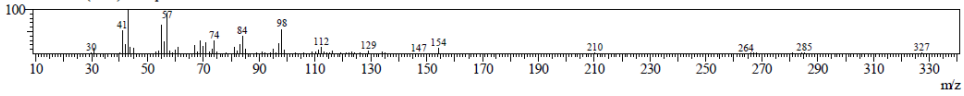
Line#:14 R.Time:20.6(Scan#:2118)
MassPeaks:97
RawMode:Single 20.6(2118) BasePeak:81(209676)
BG Mode:20.7(2121) Group 1 - Event 1



Line#:15 R.Time:21.0(Scan#:2158)
MassPeaks:111
RawMode:Single 21.0(2158) BasePeak:43(146850)
BG Mode:21.0(2161) Group 1 - Event 1



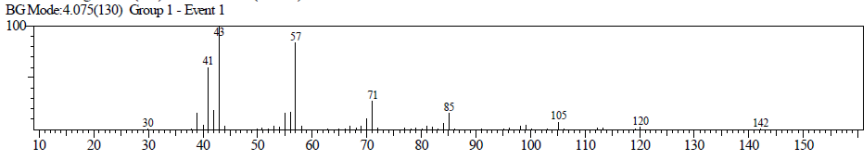
Line#:16 R.Time:22.5(Scan#:2346)
MassPeaks:76
RawMode:Single 22.5(2346) BasePeak:43(19595)
BG Mode:22.5(2345) Group 1 - Event 1



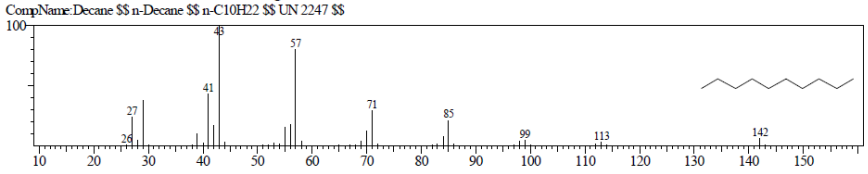
Spectrum Comparison

Library

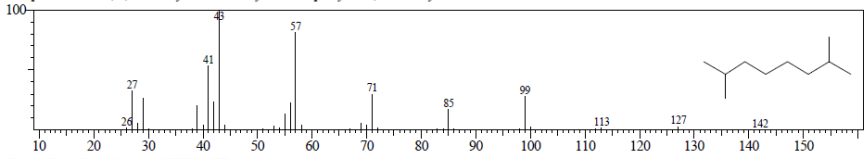
<< Target >>
Line#:1 R.Time:4.092(Scan#:132) MassPeaks:51
RawMode:Single 4.092(132) BasePeak:43.00(443352)
BG Mode:4.075(130) Group 1 - Event 1



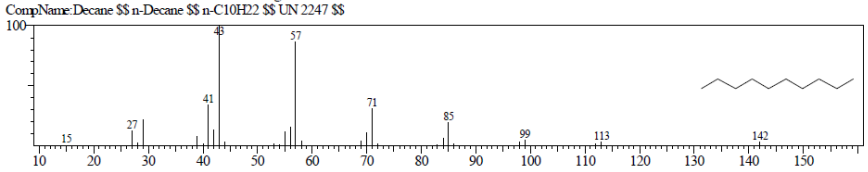
Hit#:1 Entry:11996 Library:NIST05.LIB
SI:93 Formula:C10H22 CAS:124-18-5 MolWeight:142 RetIndex:1015
CompName:Decane $$ n-Decane $$ n-C10H22 $$ UN 2247 $$



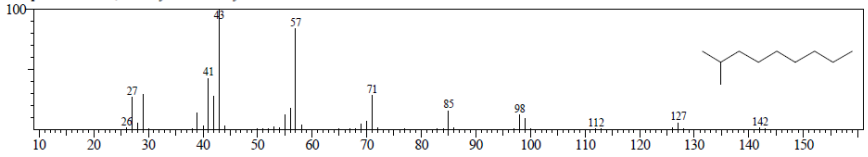
Hit#:2 Entry:7267 Library:NIST05s.LIB
SI:93 Formula:C10H22 CAS:1072-16-8 MolWeight:142 RetIndex:887
CompName:Octane, 2,7-dimethyl- $$ Diisoamyl $$ Diisopentyl $$ 2,7-Dimethyloctane $$



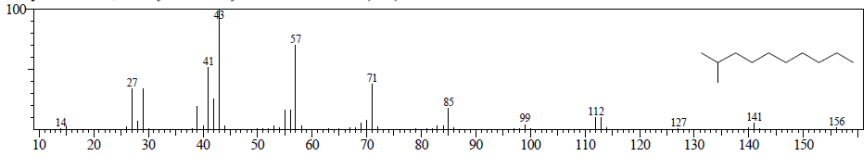
Hit#:3 Entry:7266 Library:NIST05s.LIB
SI:92 Formula:C10H22 CAS:124-18-5 MolWeight:142 RetIndex:1015
CompName:Decane $$ n-Decane $$ n-C10H22 $$ UN 2247 $$



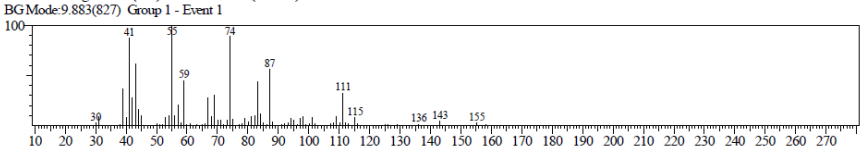
Hit#:4 Entry:11998 Library:NIST05.LIB
SI:92 Formula:C10H22 CAS:871-83-0 MolWeight:142 RetIndex:951
CompName:Nonane, 2-methyl- $$ 2-Methylnonane $$



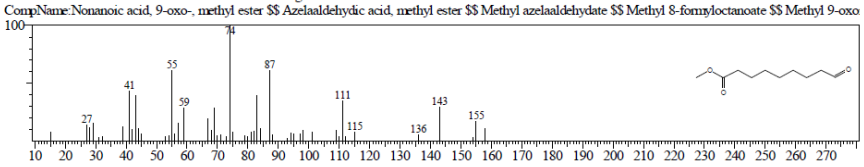
Hit#:5 Entry:18071 Library:NIST05.LIB
SI:92 Formula:C11H24 CAS:6975-98-0 MolWeight:156 RetIndex:1051
CompName:Decane, 2-methyl- $$ 2-Methyldecane $$ n-C8H17CH(CH3)2 $$



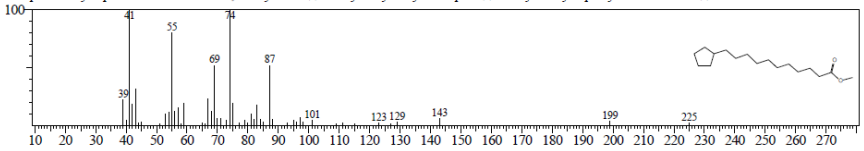
<< Target >>
Line#:2 R.Time:9.867(Scan#:825) MassPeaks:75
RawMode:Single 9.867(825) BasePeak:54.95(133552)
BG Mode:9.883(827) Group 1 - Event 1



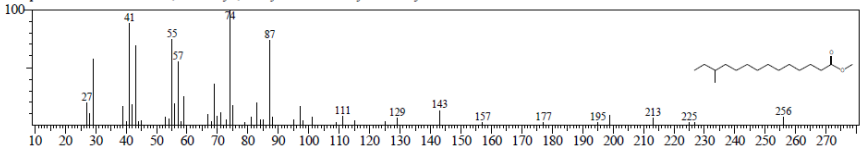
Hit#:1 Entry:33190 Library:NIST05.LIB
SI:88 Formula:C10H18O3 CAS:1931-63-1 MolWeight:186 RetIndex:1371
CompName:Nonanoic acid, 9-oxo-, methyl ester $$ Azelaaldehydic acid, methyl ester $$ Methyl azelaaldehydate $$ Methyl 8-formyloctanoate $$ Methyl 9-oxo



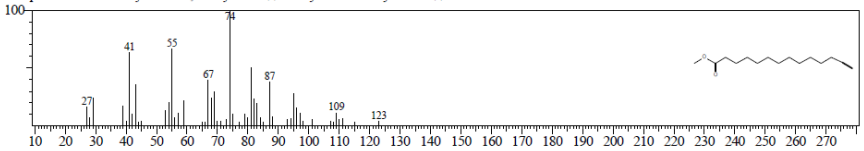
Hit#:2 Entry:82144 Library:NIST05.LIB
SI:87 Formula:C17H32O2 CAS:25779-85-5 MolWeight:268 RetIndex:1921
CompName:Cyclopentaneundecanoic acid, methyl ester $$ Methyl dihydrohydnocarpate $$ Methyl 11-cyclopentylundecanoate # $$



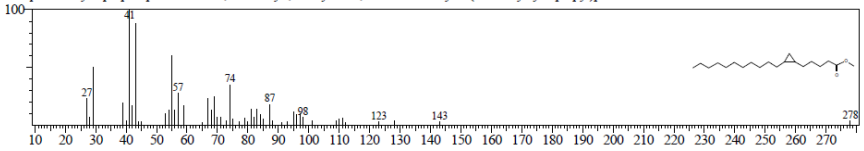
Hit#:3 Entry:75023 Library:NIST05.LIB
SI:84 Formula:C16H32O2 CAS:5129-66-8 MolWeight:256 RetIndex:1715
CompName:Tetradecanoic acid, 12-methyl-, methyl ester $$ Methyl 12-methyltetradecanoate $$



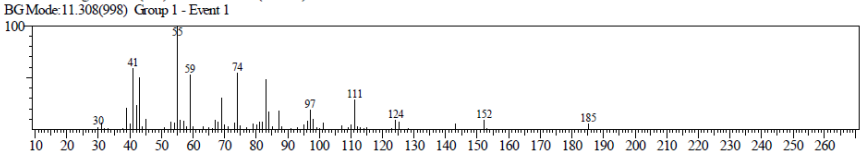
Hit#:4 Entry:64005 Library:NIST05.LIB
SI:84 Formula:C15H26O2 CAS:56909-03-6 MolWeight:238 RetIndex:1677
CompName:13-Tetradecynoic acid, methyl ester $$ Methyl 13-tetradecynoate # $$



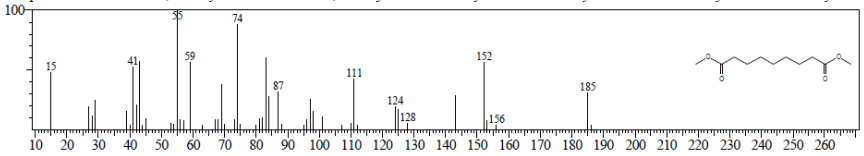
Hit#:5 Entry:106906 Library:NIST05.LIB
SI:83 Formula:C20H38O2 CAS:42199-20-2 MolWeight:310 RetIndex:2140
CompName:Cyclopropanepentanoic acid, 2-undecyl-, methyl ester, trans- $$ Methyl 5-(2-undecylcyclopropyl)pentanoate # $$



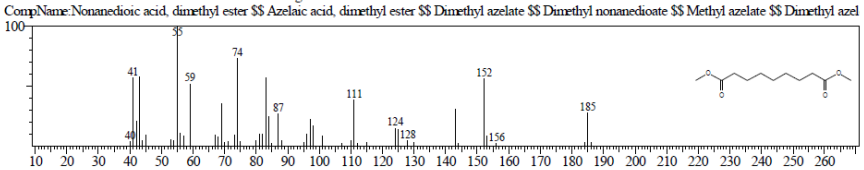
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Line#:3 R.Time:11.258(Scan#:992) MassPeaks:67
RawMode:Single 11.258(992) BasePeak:54.95(172798)
BG Mode:11.308(998) Group 1 - Event 1



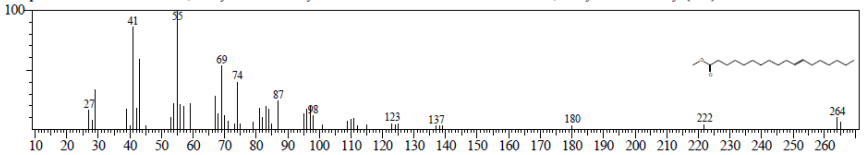
Hit#:1 Entry:18030 Library:NIST05s.LIB
SI:89 Formula:C11H20O4 CAS:1732-10-1 MolWeight:216 RetIndex:1449
CompName:Nonanedioic acid, dimethyl ester $$ Azelaic acid, dimethyl ester $$ Dimethyl azelate $$ Dimethyl nonanedioate $$ Methyl azelate $$ Dimethyl azel



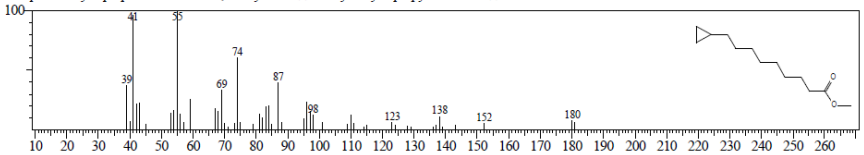
Hit#:2 Entry:18029 Library:NIST05s.LIB
SI:89 Formula:C11H20O4 CAS:1732-10-1 MolWeight:216 RetIndex:1449
CompName:Nonanedioic acid, dimethyl ester $$ Azelaic acid, dimethyl ester $$ Dimethyl azelate $$ Dimethyl nonanedioate $$ Methyl azelate $$ Dimethyl azel



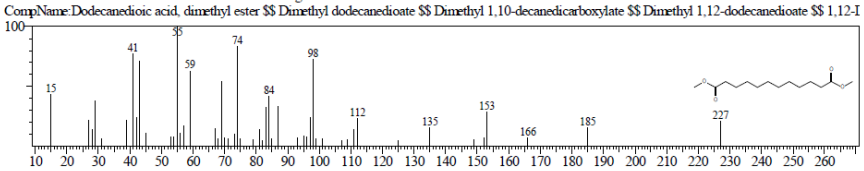
Hit#:3 Entry:98778 Library:NIST05.LIB
SI:83 Formula:C19H36O2 CAS:52380-33-3 MolWeight:296 RetIndex:2085
CompName:11-Octadecenoic acid, methyl ester $$ Methyl 11-octadecenoate $$ Octadec-11-enoic acid, methyl ester $$ Methyl (11E)-11-octadecenoate # $$



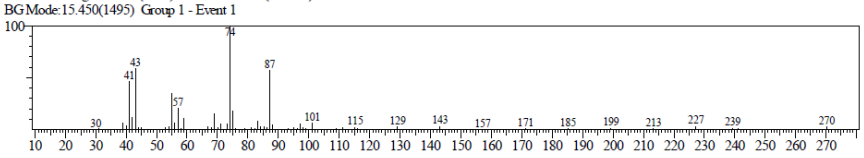
Hit#:4 Entry:48389 Library:NIST05.LIB
SI:83 Formula:C13H24O2 CAS:10152-60-0 MolWeight:212 RetIndex:1483
CompName:Cyclopropanenonanoic acid, methyl ester $$ Methyl 9-cyclopropylnonanoate $$



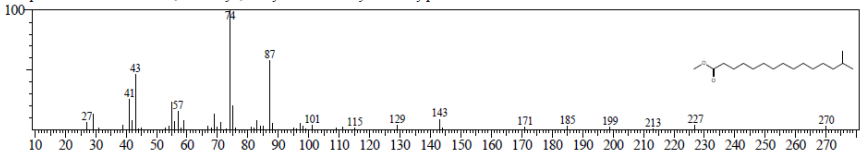
Hit#:5 Entry:75942 Library:NIST05.LIB
SI:83 Formula:C14H26O4 CAS:1731-79-9 MolWeight:258 RetIndex:1747
CompName:Dodecanedioic acid, dimethyl ester $$ Dimethyl dodecanedioate $$ Dimethyl 1,10-decanedicarboxylate $$ Dimethyl 1,12-dodecanedioate $$ 1,12-I



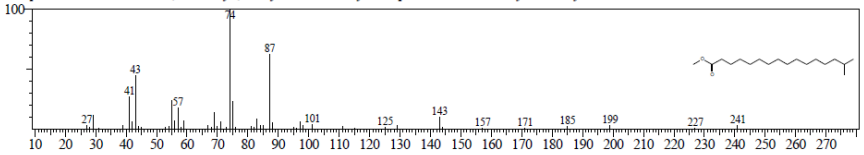
<< Target >>
Line#:4 R.Time:15.467(Scan#:1497) MassPeaks:103
RawMode:Single 15.467(1497) BasePeak:74.00(823377)
BG Mode:15.450(1495) Group 1 - Event 1



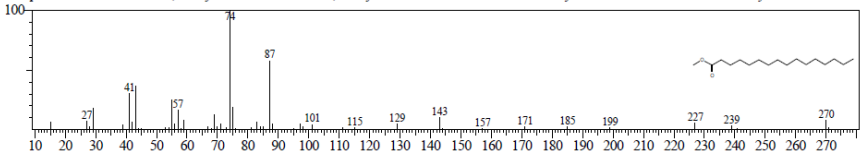
Hit#:1 Entry:22223 Library:NIST05s.LIB
SI:94 Formula:C17H34O2 CAS:5129-60-2 MolWeight:270 RetIndex:1814
CompName:Pentadecanoic acid, 14-methyl-, methyl ester $$ Methyl 14-methylpentadecanoate # $$



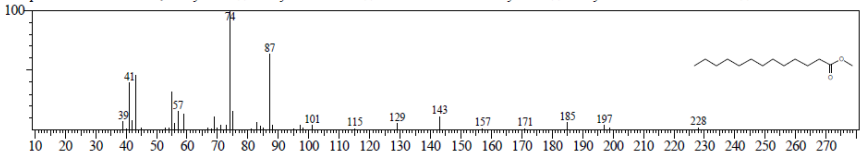
Hit#:2 Entry:22987 Library:NIST05s.LIB
SI:93 Formula:C18H36O2 CAS:6929-04-0 MolWeight:284 RetIndex:1914
CompName:Hexadecanoic acid, 15-methyl-, methyl ester $$ Methyl isoheptadecanoate $$ Methyl 15-methylhexadecanoate $$



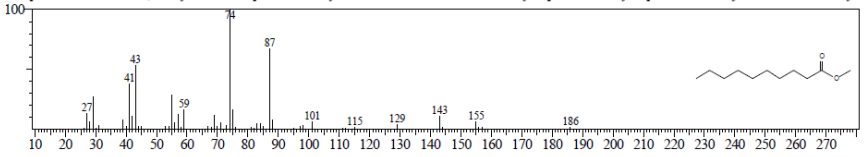
Hit#:3 Entry:22219 Library:NIST05s.LIB
SI:93 Formula:C17H34O2 CAS:112-39-0 MolWeight:270 RetIndex:1878
CompName:Hexadecanoic acid, methyl ester $$ Palmitic acid, methyl ester $$ n-Hexadecanoic acid methyl ester $$ Metholene 2216 $$ Methyl hexadecanoate $



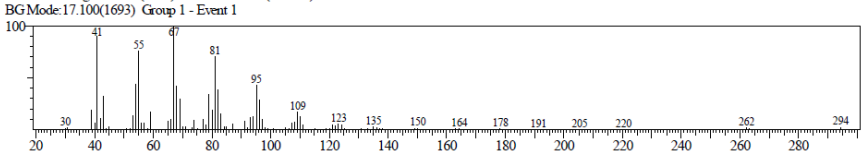
Hit#:4 Entry:19255 Library:NIST05s.LIB
SI:93 Formula:C14H28O2 CAS:1731-88-0 MolWeight:228 RetIndex:1580
CompName:Tridecanoic acid, methyl ester $$ Methyl tridecanoate $$ n-Tridecanoic acid methyl ester $$ Methyl ester of tridecanoic acid $$



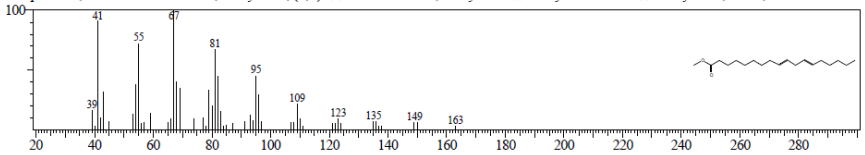
Hit#:5 Entry:14361 Library:NIST05s.LIB
SI:92 Formula:C11H22O2 CAS:110-42-9 MolWeight:186 RetIndex:1282
CompName:Decanoic acid, methyl ester $$ Capric acid methyl ester $$ Metholene 2095 $$ Methyl caprate $$ Methyl caprinate $$ Methyl decanoate $$ Methyl-



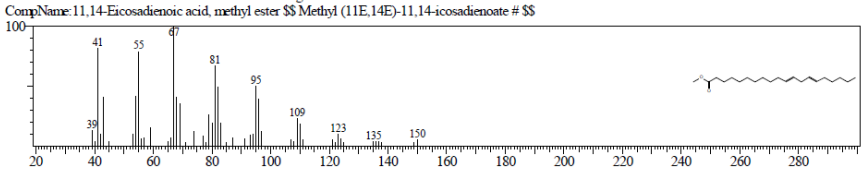
<< Target >>
Line#:5 R.Time:17.142(Scan#:1698) MassPeaks:147
RawMode:Single 17.142(1698) BasePeak:67.00(850352)
BG Mode:17.100(1693) Group 1 - Event 1



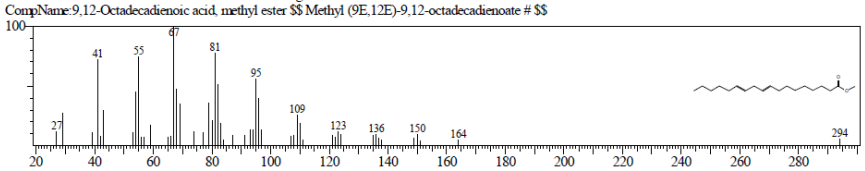
Hit#:1 Entry:23474 Library:NIST05s.LIB
SI:96 Formula:C19H34O2 CAS:2566-97-4 MolWeight:294 RetIndex:2093
CompName:9,12-Octadecadienoic acid, methyl ester, (E,E)- $$ Linolelaidic acid, methyl ester $$ Methyl linolelaidate $$ Methyl trans,trans-9,12-octadecadieno



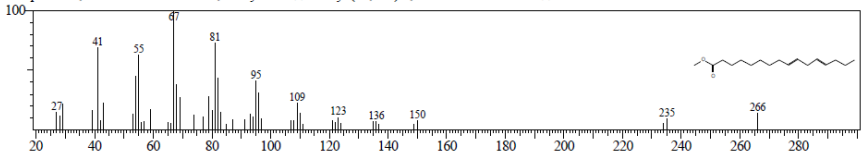
Hit#:2 Entry:113456 Library:NIST05.LIB
SI:95 Formula:C21H38O2 CAS:2463-02-7 MolWeight:322 RetIndex:2292
CompName:11,14-Eicosadienoic acid, methyl ester $$ Methyl (11E,14E)-11,14-icosadienoate # $$



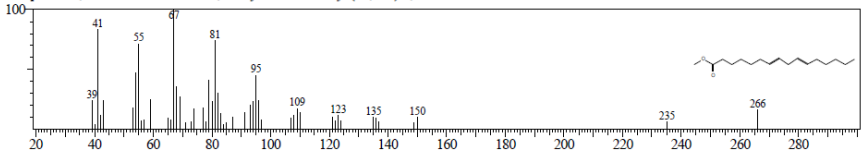
Hit#:3 Entry:97662 Library:NIST05.LIB
SI:93 Formula:C19H34O2 CAS:2462-85-3 MolWeight:294 RetIndex:2093
CompName:9,12-Octadecadienoic acid, methyl ester $$ Methyl (9E,12E)-9,12-octadecadienoate # $$



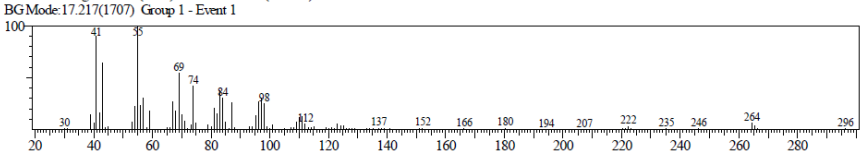
Hit#:4 Entry:80869 Library:NIST05.LIB
SI:92 Formula:C17H30O2 CAS:2462-80-8 MolWeight:266 RetIndex:1894
CompName:9,12-Hexadecadienoic acid, methyl ester $$ Methyl (9E,12E)-9,12-hexadecadienoate # $$



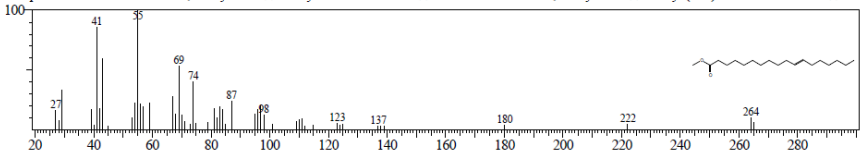
Hit#:5 Entry:80867 Library:NIST05.LIB
SI:92 Formula:C17H30O2 CAS:16106-03-9 MolWeight:266 RetIndex:1894
CompName:7,10-Hexadecadienoic acid, methyl ester $$ Methyl (7E,10E)-7,10-hexadecadienoate # $$



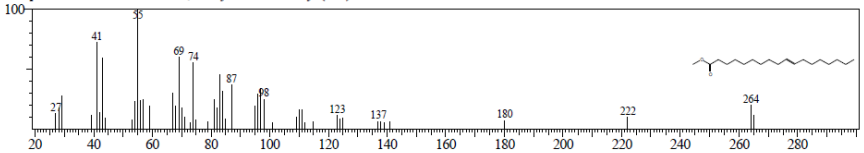
<< Target >>
Line#:6 R.Time:17.192(Scan#:1704) MassPeaks:148
RawMode:Single 17.192(1704) BasePeak:55.00(971301)
BG Mode:17.217(1707) Group 1 - Event 1



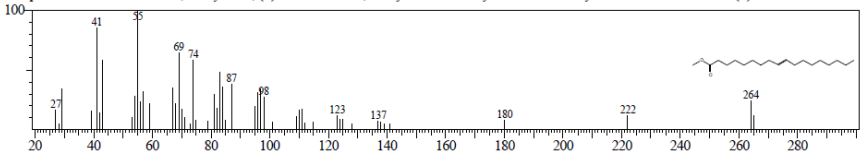
Hit#:1 Entry:98778 Library:NIST05.LIB
SI:94 Formula:C19H36O2 CAS:52380-33-3 MolWeight:296 RetIndex:2085
CompName:11-Octadecenoic acid, methyl ester $$ Methyl 11-octadecenoate $$ Octadec-11-enoic acid, methyl ester $$ Methyl (11E)-11-octadecenoate # $$



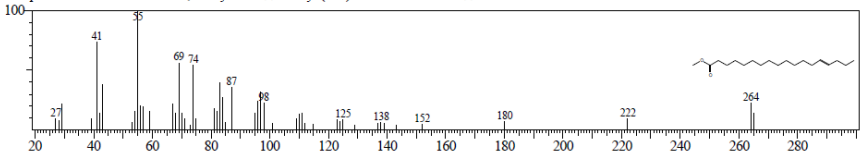
Hit#:2 Entry:98784 Library:NIST05.LIB
SI:92 Formula:C19H36O2 CAS:13481-95-3 MolWeight:296 RetIndex:2085
CompName:10-Octadecenoic acid, methyl ester $$ Methyl (10E)-10-octadecenoate # $$



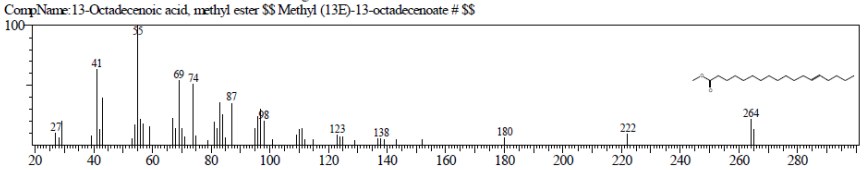
Hit#:3 Entry:23570 Library:NIST05s.LIB
SI:92 Formula:C19H36O2 CAS:1937-62-8 MolWeight:296 RetIndex:2085
CompName:9-Octadecenoic acid, methyl ester, (E)- $$ Elaidic acid, methyl ester $$ Methyl elaidate $$ Methyl trans-9-octadecenoate $$ (E)-9-Octadecenoic aci



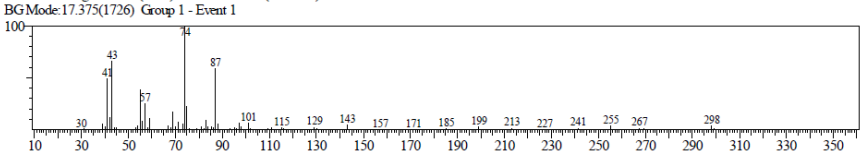
Hit#:4 Entry:98787 Library:NIST05.LIB
SI:91 Formula:C19H36O2 CAS:56554-48-4 MolWeight:296 RetIndex:2085
CompName:14-Octadecenoic acid, methyl ester $$ Methyl (14E)-14-octadecenoate # $$



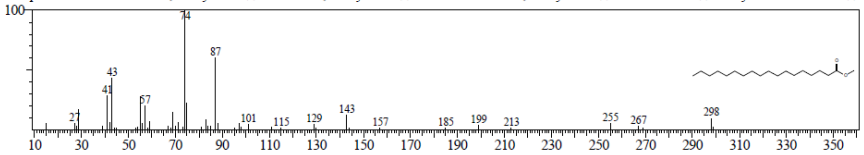
Hit#:5 Entry:98786 Library:NIST05.LIB
SI:91 Formula:C19H36O2 CAS:56554-47-3 MolWeight:296 RetIndex:2085
CompName:13-Octadecenoic acid, methyl ester $$ Methyl (13E)-13-octadecenoate # $$



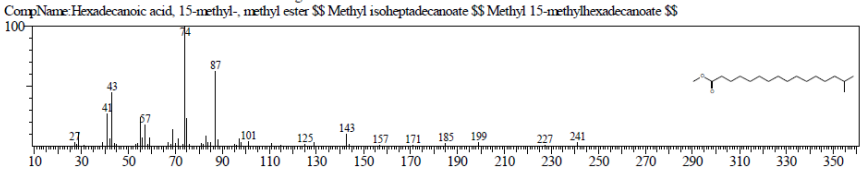
<< Target >>
Line#:7 R.Time:17.408(Scan#:1730) MassPeaks:111
RawMode:Single 17.408(1730) BasePeak:74.00(1672379)
BG Mode:17.375(1726) Group 1 - Event 1



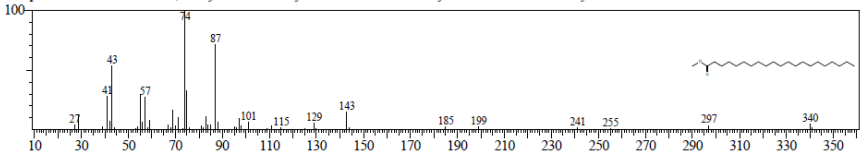
Hit#:1 Entry:100069 Library:NIST05.LIB
SI:92 Formula:C19H38O2 CAS:112-61-8 MolWeight:298 RetIndex:2077
CompName:Octadecanoic acid, methyl ester $$ Stearic acid, methyl ester $$ n-Octadecanoic acid, methyl ester $$ Kemester 9718 $$ Methyl n-octadecanoate $$



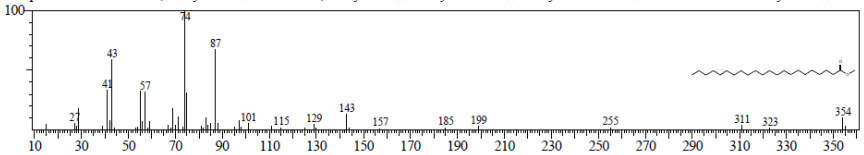
Hit#:2 Entry:22987 Library:NIST05s.LIB
SI:92 Formula:C18H36O2 CAS:6929-04-0 MolWeight:284 RetIndex:1914
CompName:Hexadecanoic acid, 15-methyl-, methyl ester $$ Methyl isoheptadecanoate $$ Methyl 15-methylhexadecanoate $$



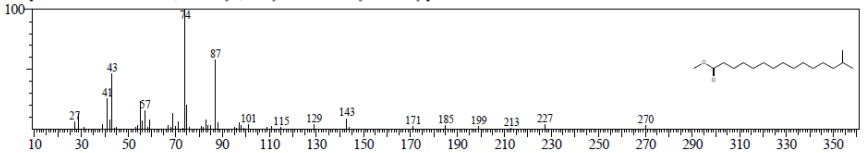
Hit#:3 Entry:25264 Library:NIST05s.LIB
SI:91 Formula:C22H44O2 CAS:6064-90-0 MolWeight:340 RetIndex:2375
CompName:Heneicosanoic acid, methyl ester $$ Methyl heneicosanoate $$ Methyl henelcosanoate $$ Methyl henicosanoate # $$



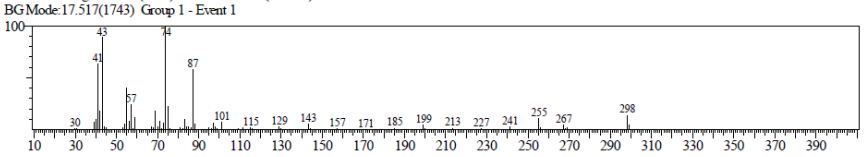
Hit#:4 Entry:25584 Library:NIST05s.LIB
SI:91 Formula:C23H46O2 CAS:929-77-1 MolWeight:354 RetIndex:2475
CompName:Docosanoic acid, methyl ester $$ Behenic acid, methyl ester $$ Methyl behenate $$ Methyl docosanoate $$ n-Docosanoic acid methyl ester $$ Kem



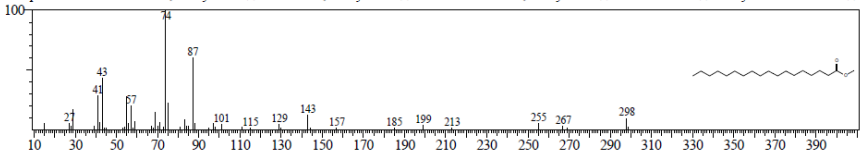
Hit#:5 Entry:22223 Library:NIST05s.LIB
SI:91 Formula:C17H34O2 CAS:5129-60-2 MolWeight:270 RetIndex:1814
CompName:Pentadecanoic acid, 14-methyl-, methyl ester $$ Methyl 14-methylpentadecanoate # $$



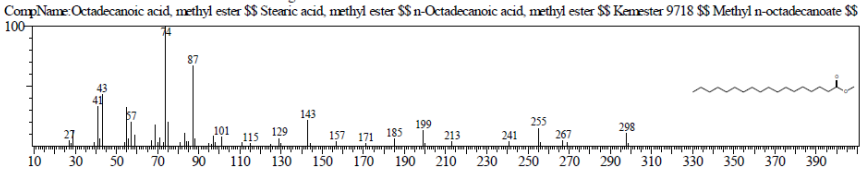
<< Target >>
Line#:8 R.Time:17.508(Scan#:1742) MassPeaks:108
RawMode:Single 17.508(1742) BasePeak:74.00(486512)
BG Mode:17.517(1743) Group 1 - Event 1



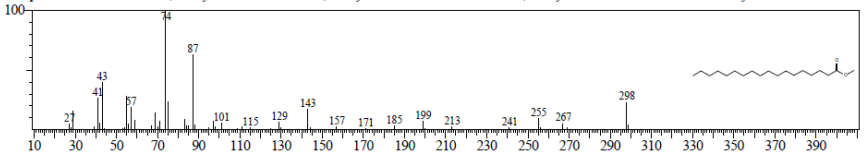
Hit#:1 Entry:100069 Library:NIST05.LIB
SI:90 Formula:C19H38O2 CAS:112-61-8 MolWeight:298 RetIndex:2077
CompName:Octadecanoic acid, methyl ester $$ Stearic acid, methyl ester $$ n-Octadecanoic acid, methyl ester $$ Kemester 9718 $$ Methyl n-octadecanoate $$



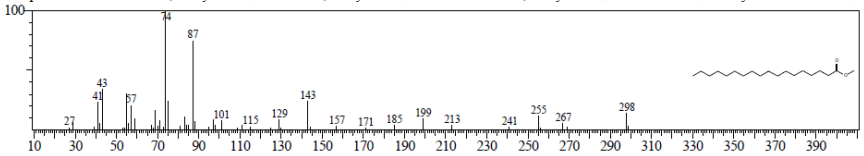
Hit#:2 Entry:23657 Library:NIST05s.LIB
SI:89 Formula:C19H38O2 CAS:112-61-8 MolWeight:298 RetIndex:2077
CompName:Octadecanoic acid, methyl ester $$ Stearic acid, methyl ester $$ n-Octadecanoic acid, methyl ester $$ Kemester 9718 $$ Methyl n-octadecanoate $$



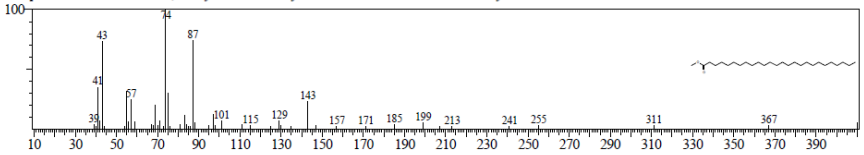
Hit#:3 Entry:23658 Library:NIST05s.LIB
SI:88 Formula:C19H38O2 CAS:112-61-8 MolWeight:298 RetIndex:2077
CompName:Octadecanoic acid, methyl ester $$ Stearic acid, methyl ester $$ n-Octadecanoic acid, methyl ester $$ Kemester 9718 $$ Methyl n-octadecanoate $$



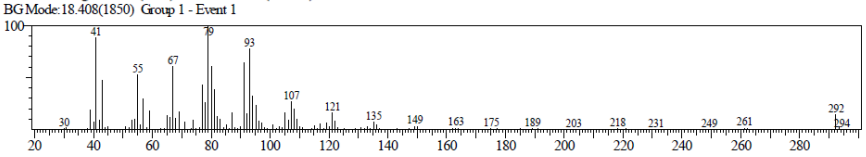
Hit#:4 Entry:23659 Library:NIST05s.LIB
SI:88 Formula:C19H38O2 CAS:112-61-8 MolWeight:298 RetIndex:2077
CompName:Octadecanoic acid, methyl ester $$ Stearic acid, methyl ester $$ n-Octadecanoic acid, methyl ester $$ Kemester 9718 $$ Methyl n-octadecanoate $$



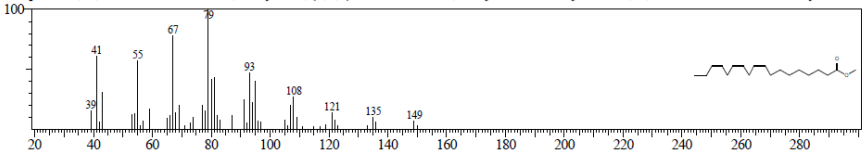
Hit#:5 Entry:26664 Library:NIST05s.LIB
SI:88 Formula:C27H54O2 CAS:5802-82-4 MolWeight:410 RetIndex:2872
CompName:Hexacosanoic acid, methyl ester $$ Methyl hexacosanoate $$ Cerotic acid methyl ester $$



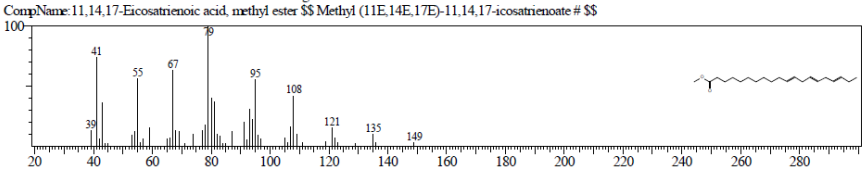
<< Target >>
Line#:9 R.Time:18.442(Scan#:1854) MassPeaks:136
RawMode:Single 18.442(1854) BasePeak:79.05(425426)
BG Mode:18.408(1850) Group 1 - Event 1



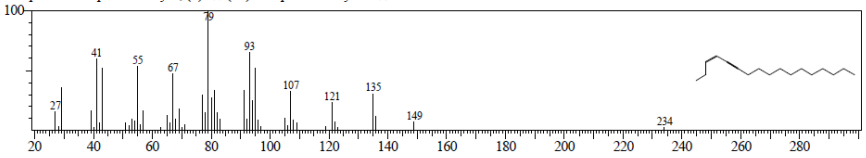
Hit#:1 Entry:23400 Library:NIST05s.LIB
SI:89 Formula:C19H32O2 CAS:301-00-8 MolWeight:292 RetIndex:2101
CompName:9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)- $$ Linolenic acid, methyl ester $$ Methyl all-cis-9,12,15-octadecatrienoate $$ Methyl linolena



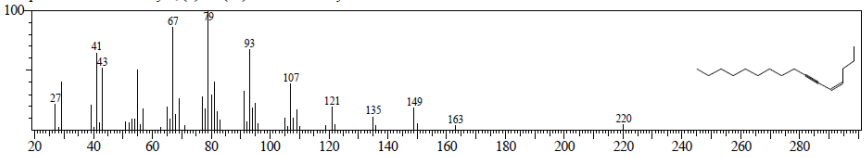
Hit#:2 Entry:112484 Library:NIST05.LIB
SI:88 Formula:C21H36O2 CAS:55682-88-7 MolWeight:320 RetIndex:2300
CompName:11,14,17-Eicosatrienoic acid, methyl ester $$ Methyl (11E,14E,17E)-11,14,17-icosatrienoate # $$



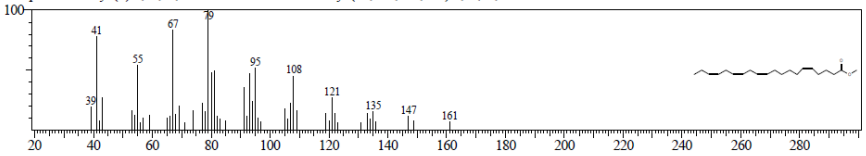
Hit#:3 Entry:61688 Library:NIST05.LIB
SI:87 Formula:C17H30 CAS:74744-55-1 MolWeight:234 RetIndex:1736
CompName:3-Heptadecen-5-yne, (Z)- $$ (3Z)-3-Heptadecen-5-yne # $$



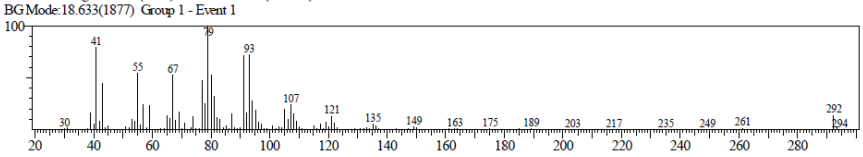
Hit#:4 Entry:53202 Library:NIST05.LIB
SI:87 Formula:C16H28 CAS:74744-54-0 MolWeight:220 RetIndex:1637
CompName:4-Hexadecen-6-yne, (Z)- $$ (4Z)-4-Hexadecen-6-yne # $$



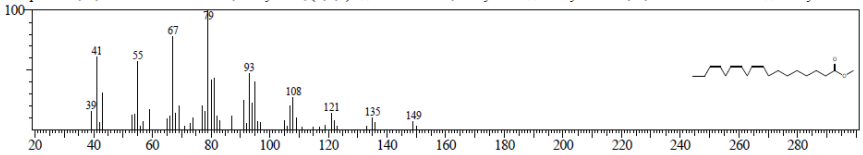
Hit#:5 Entry:111404 Library:NIST05.LIB
SI:87 Formula:C21H34O2 CAS:59149-01-8 MolWeight:318 RetIndex:2308
CompName:Methyl (Z)-5,11,14,17-eicosatetraenoate $$ Methyl (5Z,11Z,14Z,17Z)-5,11,14,17-icosatetraenoate # $$



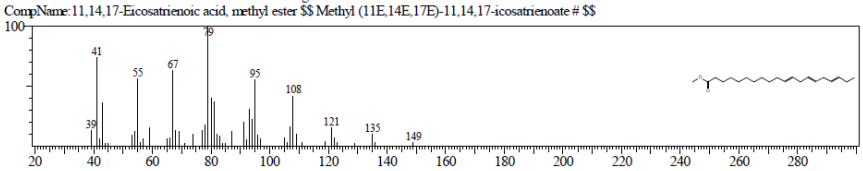
<< Target >>
Line#:10 R.Time:18.600(Scan#:1873) MassPeaks:131
RawMode:Single 18.600(1873) BasePeak:79.05(870078)
BG Mode:18.633(1877) Group 1 - Event 1



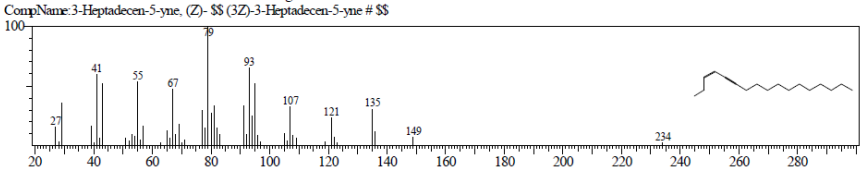
Hit#:1 Entry:23400 Library:NIST05s.LIB
SI:88 Formula:C19H32O2 CAS:301-00-8 MolWeight:292 RetIndex:2101
CompName:9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)- $$ Linolenic acid, methyl ester $$ Methyl all-cis-9,12,15-octadecatrienoate $$ Methyl linolen



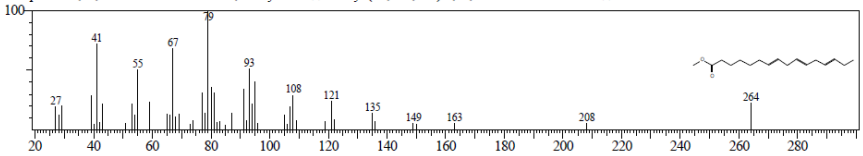
Hit#:2 Entry:112484 Library:NIST05.LIB
SI:88 Formula:C21H36O2 CAS:55682-88-7 MolWeight:320 RetIndex:2300
CompName:11,14,17-Eicosatrienoic acid, methyl ester $$ Methyl (11E,14E,17E)-11,14,17-icosatrienoate # $$



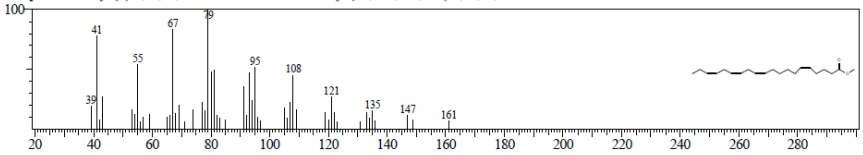
Hit#:3 Entry:61688 Library:NIST05.LIB
SI:87 Formula:C17H30 CAS:74744-55-1 MolWeight:234 RetIndex:1736
CompName:3-Heptadecen-5-yne, (Z)- $$ (3Z)-3-Heptadecen-5-yne # $$



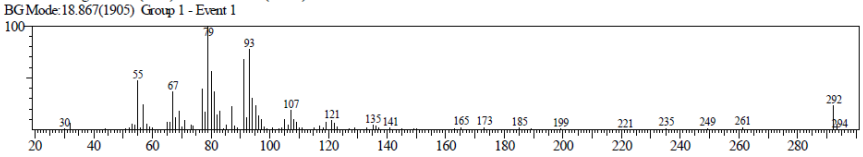
Hit#:4 Entry:79688 Library:NIST05.LIB
SI:86 Formula:C17H28O2 CAS:56554-30-4 MolWeight:264 RetIndex:1902
CompName:7,10,13-Hexadecatrienoic acid, methyl ester $$ Methyl (7E,10E,13E)-7,10,13-hexadecatrienoate # $$



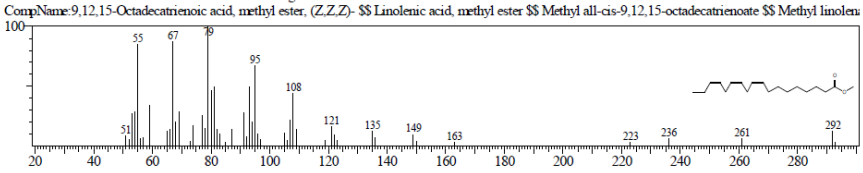
Hit#:5 Entry:111404 Library:NIST05.LIB
SI:86 Formula:C21H34O2 CAS:59149-01-8 MolWeight:318 RetIndex:2308
CompName:Methyl (Z)-5,11,14,17-eicosatetraenoate $$ Methyl (5Z,11Z,14Z,17Z)-5,11,14,17-icosatetraenoate # $$



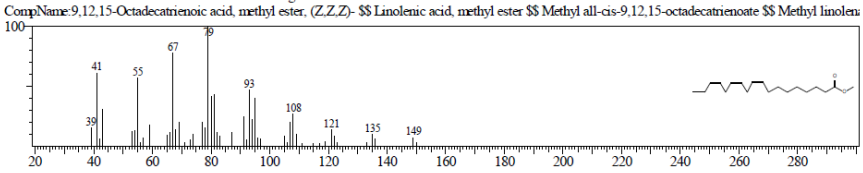
<< Target >>
Line#:11 R.Time:18.850(Scan#:1903) MassPeaks:124
RawMode:Single 18.850(1903) BasePeak:79.05(96357)
BG Mode:18.867(1905) Group 1 - Event 1



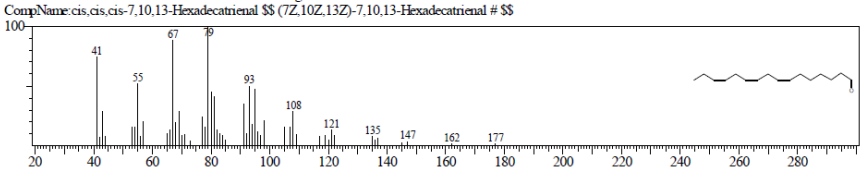
Hit#:1 Entry:23401 Library:NIST05s.LIB
SI:82 Formula:C19H32O2 CAS:301-00-8 MolWeight:292 RetIndex:2101
CompName:9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)- $$ Linolenic acid, methyl ester $$ Methyl all-cis-9,12,15-octadecatrienoate $$ Methyl linolen



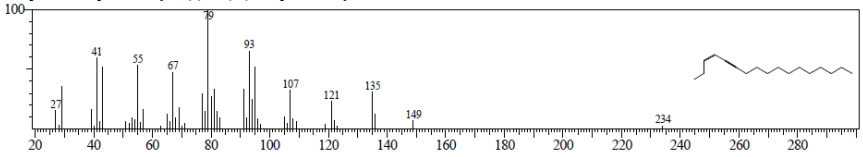
Hit#:2 Entry:23400 Library:NIST05s.LIB
SI:80 Formula:C19H32O2 CAS:301-00-8 MolWeight:292 RetIndex:2101
CompName:9,12,15-Octadecatrienoic acid, methyl ester, (Z,Z,Z)- $$ Linolenic acid, methyl ester $$ Methyl all-cis-9,12,15-octadecatrienoate $$ Methyl linolen



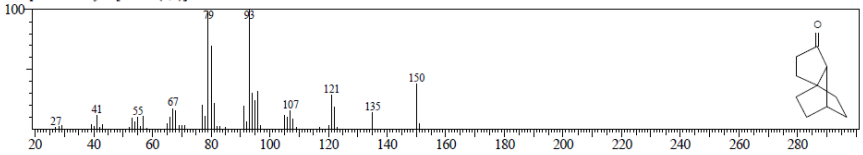
Hit#:3 Entry:61652 Library:NIST05.LIB
SI:79 Formula:C16H26O CAS:56797-43-4 MolWeight:234 RetIndex:1824
CompName:cis,cis,cis-7,10,13-Hexadecatrienal $$ (7Z,10Z,13Z)-7,10,13-Hexadecatrienal # $$



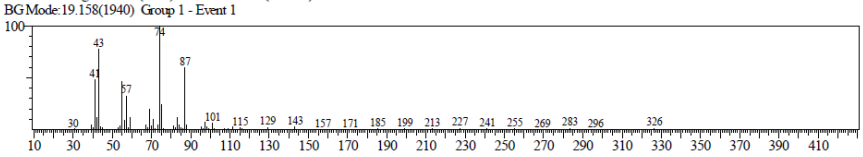
Hit#:4 Entry:61688 Library:NIST05.LIB
SI:78 Formula:C17H30 CAS:74744-55-1 MolWeight:234 RetIndex:1736
CompName:3-Heptadecen-5-yne, (Z)- $$ (3Z)-3-Heptadecen-5-yne # $$



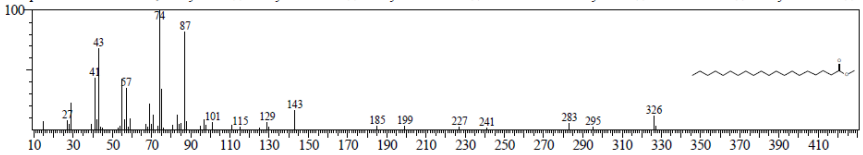
Hit#:5 Entry:14900 Library:NIST05.LIB
SI:78 Formula:C10H14O CAS:92144-38-2 MolWeight:150 RetIndex:1064
CompName:Tricyclo[4.2.2.0(1,5)]decan-4-one



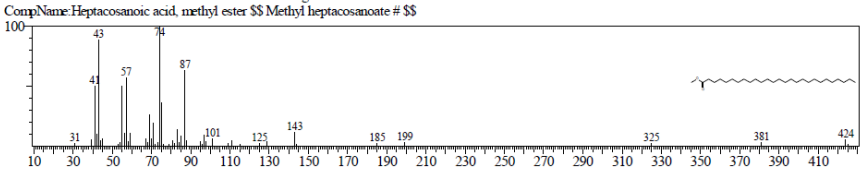
<< Target >>
Line#:12 R.Time:19.175(Scan#:1942) MassPeaks:103
RawMode:Single 19.175(1942) BasePeak:74.00(503346)
BG Mode:19.158(1940) Group 1 - Event 1



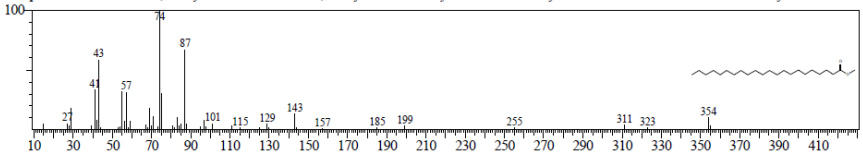
Hit#:1 Entry:24788 Library:NIST05s.LIB
SI:94 Formula:C21H42O2 CAS:1120-28-1 MolWeight:326 RetIndex:2276
CompName:Eicosanoic acid, methyl ester $$ Methyl arachisate $$ Methyl eicosanoate $$ Arachidic acid methyl ester $$ Kemester 2050 $$ Methyl aracidate $$



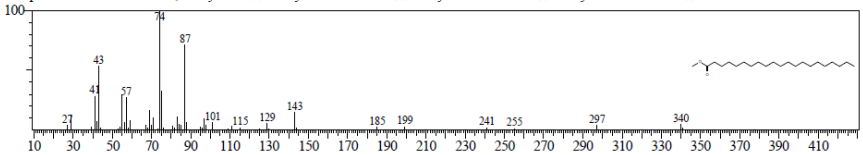
Hit#:2 Entry:26833 Library:NIST05s.LIB
SI:93 Formula:C28H56O2 CAS:55682-91-2 MolWeight:424 RetIndex:2972
CompName:Heptacosanoic acid, methyl ester $$ Methyl heptacosanoate # $$



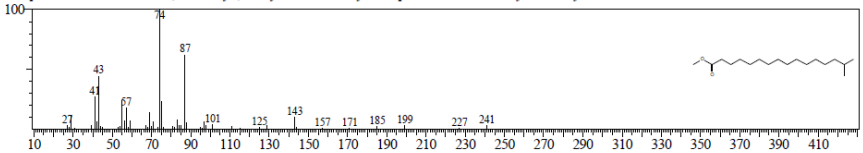
Hit#:3 Entry:25584 Library:NIST05s.LIB
SI:91 Formula:C23H46O2 CAS:929-77-1 MolWeight:354 RetIndex:2475
CompName:Docosanoic acid, methyl ester $$ Behenic acid, methyl ester $$ Methyl behenate $$ Methyl docosanoate $$ n-Docosanoic acid methyl ester $$ Kem



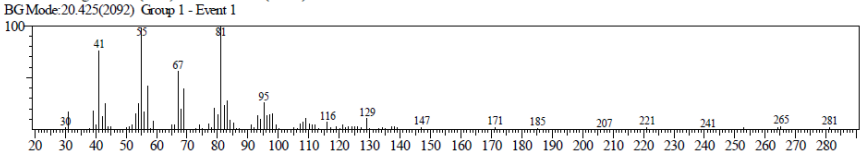
Hit#:4 Entry:25264 Library:NIST05s.LIB
SI:91 Formula:C22H44O2 CAS:6064-90-0 MolWeight:340 RetIndex:2375
CompName:Heneicosanoic acid, methyl ester $$ Methyl heneicosanoate $$ Methyl henelcosanoate $$ Methyl henicosanoate # $$



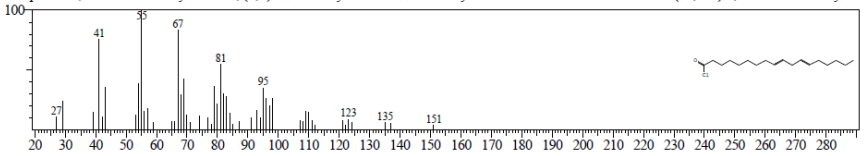
Hit#:5 Entry:22987 Library:NIST05s.LIB
SI:91 Formula:C18H36O2 CAS:6929-04-0 MolWeight:284 RetIndex:1914
CompName:Hexadecanoic acid, 15-methyl-, methyl ester $$ Methyl isoheptadecanoate $$ Methyl 15-methylhexadecanoate $$



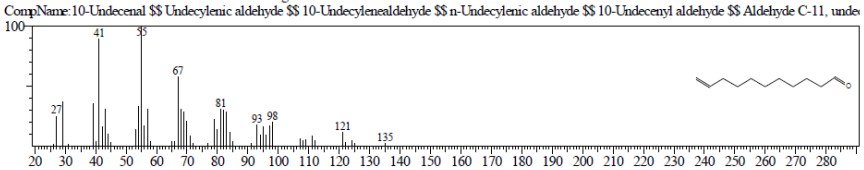
<< Target >>
Line#:13 R.Time:20.408(Scan#:2090) MassPeaks:95
RawMode:Single 20.408(2090) BasePeak:81.05(97597)
BG Mode:20.425(2092) Group 1 - Event 1



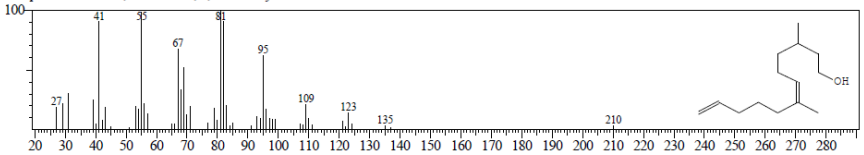
Hit#:1 Entry:23649 Library:NIST05s.LIB
SI:87 Formula:C18H31ClO CAS:7459-33-8 MolWeight:298 RetIndex:2139
CompName:9,12-Octadecadienoyl chloride, (Z,Z)- $$ Linoleoyl chloride $$ Lineoleoyl chloride $$ Linoleic acid chloride $$ (9E,12E)-9,12-Octadecadienoyl ch



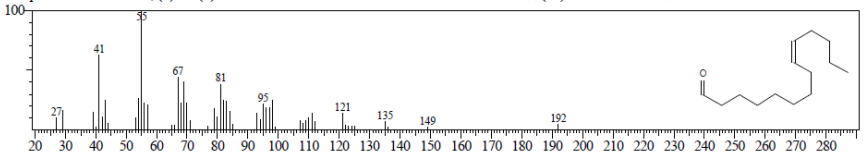
Hit#:2 Entry:11659 Library:NIST05s.LIB
SI:87 Formula:C11H20O CAS:112-45-8 MolWeight:168 RetIndex:1293
CompName:10-Undecenal $$ Undecylenic aldehyde $$ 10-Undecylenealdehyde $$ n-Undecylenic aldehyde $$ 10-Undecenyl aldehyde $$ Aldehyde C-11, unde



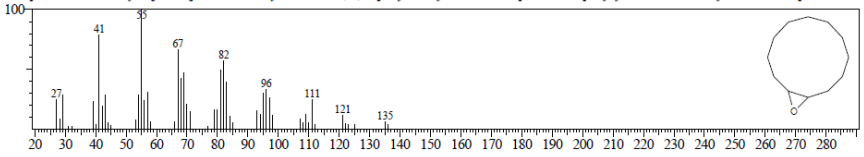
Hit#:3 Entry:47288 Library:NIST05.LIB
SI:86 Formula:C14H26O CAS:0-00-0 MolWeight:210 RetIndex:1567
CompName:Dodeca-1,6-dien-12-ol, 6,10-dimethyl-



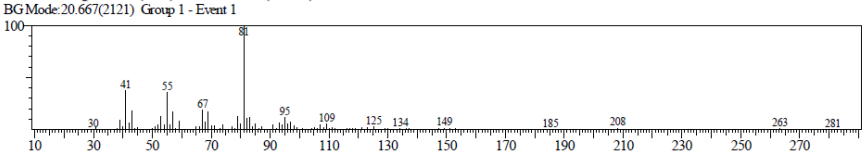
Hit#:4 Entry:47276 Library:NIST05.LIB
SI:86 Formula:C14H26O CAS:53939-27-8 MolWeight:210 RetIndex:1609
CompName:9-Tetradecenal, (Z)- $$ (Z)-9-Tetradecenal $$ Z-9-Tetradecenal $$ Z-9-Tetradecenol $$ (9Z)-9-Tetradecenal # $$ Z-9-Tetradecenal $$



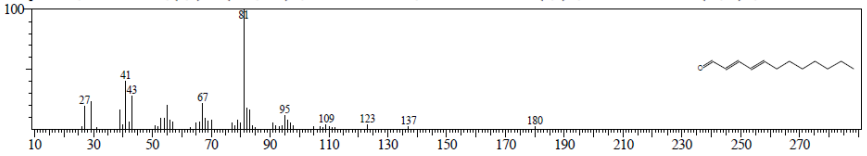
Hit#:5 Entry:31249 Library:NIST05.LIB
SI:85 Formula:C12H22O CAS:286-99-7 MolWeight:182 RetIndex:1450
CompName:13-Oxabicyclo[10.1.0]tridecane $$ Cyclododecane, 1,2-epoxy- $$ Cyclododecene epoxide $$ Epoxycyclododecane $$ Cyclododecane epoxide $$ C



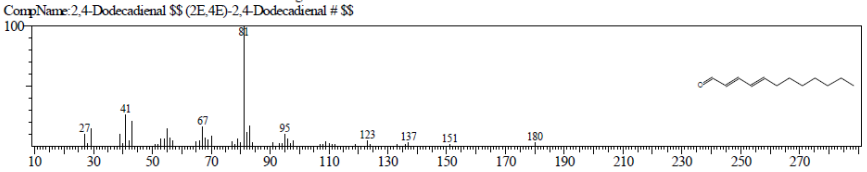
<< Target >>
Line#:14 R.Time:20.642(Scan#:2118) MassPeaks:97
RawMode:Single 20.642(2118) BasePeak:81.05(209676)
BG Mode:20.667(2121) Group 1 - Event 1



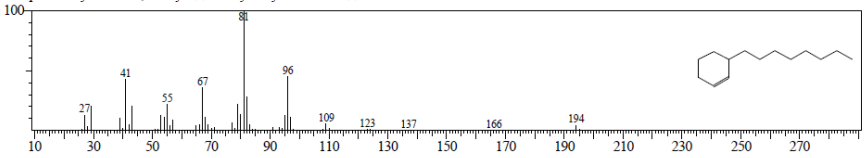
Hit#:1 Entry:30096 Library:NIST05.LIB
SI:88 Formula:C12H20O CAS:21662-16-8 MolWeight:180 RetIndex:1418
CompName:2,4-Dodecadienal, (E,E)- $$ (trans,trans)-2,4-Dodecadienal $$ 2,4-Dodecadien-1-al $$ (E,E)-2,4-Dodecadien-1-al $$ (2E,4E)-2,4-Dodecadienal # $



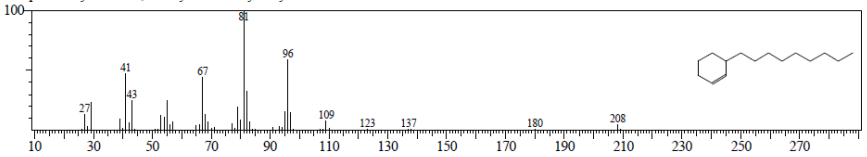
Hit#:2 Entry:30097 Library:NIST05.LIB
SI:85 Formula:C12H20O CAS:13162-47-5 MolWeight:180 RetIndex:1418
CompName:2,4-Dodecadienal $$ (2E,4E)-2,4-Dodecadienal # $$



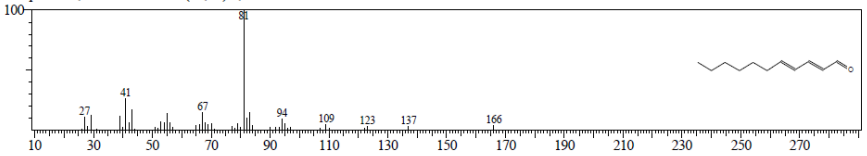
Hit#:3 Entry:37893 Library:NIST05.LIB
SI:85 Formula:C14H26 CAS:15232-94-7 MolWeight:194 RetIndex:1459
CompName:Cyclohexene, 3-octyl- $$ 3-Octyl-1-cyclohexene # $$



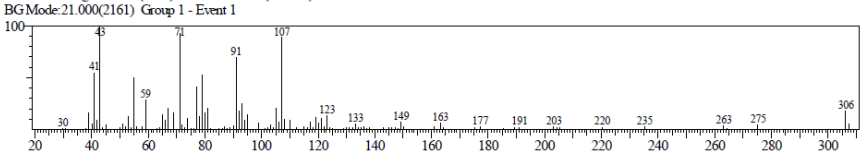
Hit#:4 Entry:46112 Library:NIST05.LIB
SI:84 Formula:C15H28 CAS:15232-79-8 MolWeight:208 RetIndex:1558
CompName:Cyclohexene, 3-nonyl- $$ 3-Nonyl-1-cyclohexene # $$



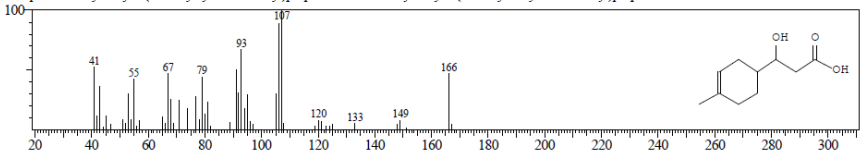
Hit#:5 Entry:22644 Library:NIST05.LIB
SI:84 Formula:C11H18O CAS:13162-46-4 MolWeight:166 RetIndex:1319
CompName:2,4-Undecadienal $$ (2E,4E)-2,4-Undecadienal # $$



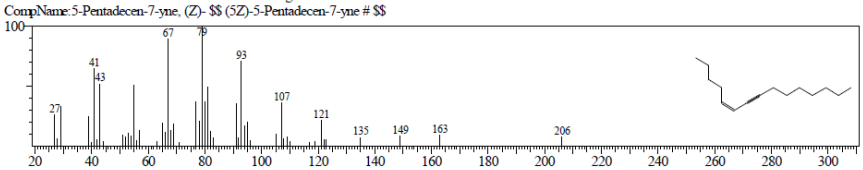
<< Target >>
Line#:15 R.Time:20.975(Scan#:2158) MassPeaks:111
RawMode:Single 20.975(2158) BasePeak:43.00(146850)
BG Mode:21.000(2161) Group 1 - Event 1



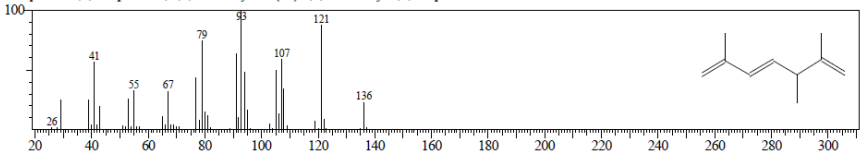
Hit#:1 Entry:32059 Library:NIST05.LIB
SI:75 Formula:C10H16O3 CAS:85426-39-7 MolWeight:184 RetIndex:1570
CompName:3-Hydroxy-3-(4-methylcyclohex-3-enyl)propionic acid $$ 3-Hydroxy-3-(4-methyl-3-cyclohexen-1-yl)propanoic acid # $$



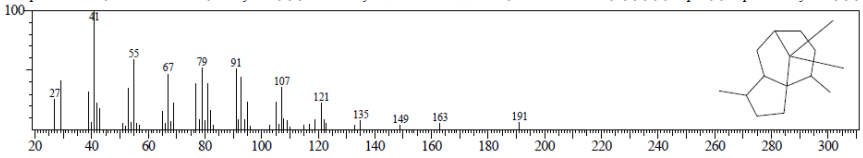
Hit#:2 Entry:44766 Library:NIST05.LIB
SI:75 Formula:C15H26 CAS:74744-50-6 MolWeight:206 RetIndex:1538
CompName:5-Pentadecen-7-yne, (Z)- $$ (5Z)-5-Pentadecen-7-yne # $$



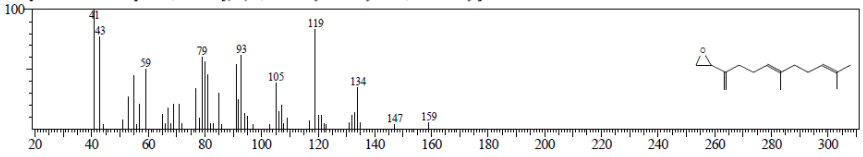
Hit#:3 Entry:9607 Library:NIST05.LIB
SI:74 Formula:C10H16 CAS:42123-66-0 MolWeight:136 RetIndex:894
CompName:1,3,6-Heptatriene, 2,5,6-trimethyl- $$ (3E)-2,5,6-Trimethyl-1,3,6-heptatriene # $$



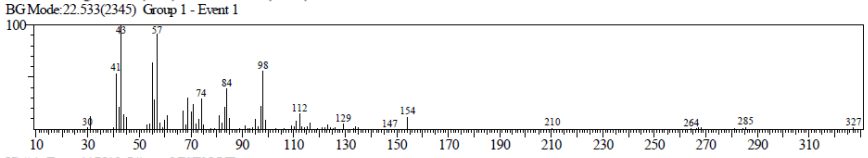
Hit#:4 Entry:44755 Library:NIST05.LIB
SI:74 Formula:C15H26 CAS:19078-35-4 MolWeight:206 RetIndex:1393
CompName:1H-3a,7-Methanoazulene, octahydro-1,4,9,9-tetramethyl- $$ Patchulane $$ 1H-3a,7-Methanoazulene, 2,3,4,5,6,7.alpha.,8,8.alpha.-octahydro-1,4,9,



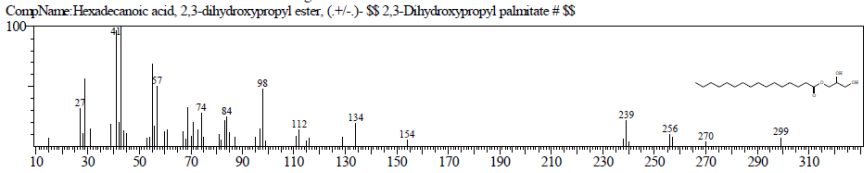
Hit#:5 Entry:53064 Library:NIST05.LIB
SI:74 Formula:C15H24O CAS:83637-40-5 MolWeight:220 RetIndex:1540
CompName:Farnesene epoxide, E- $$ 2-[(4E)-5,9-Dimethyl-1-methylene-4,8-decadienyl]oxirane # $$



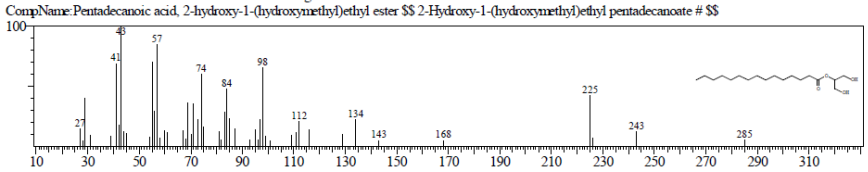
<< Target >>
Line#:16 R.Time:22.542(Scan#:2346) MassPeaks:76
RawMode:Single 22.542(2346) BasePeak:43.00(19595)
BG Mode:22.533(2345) Group 1 - Event 1



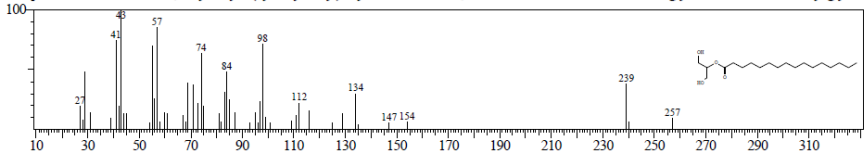
Hit#:1 Entry:117518 Library:NIST05.LIB
SI:86 Formula:C19H38O4 CAS:19670-51-0 MolWeight:330 RetIndex:2482
CompName:Hexadecanoic acid, 2,3-dihydroxypropyl ester, (+/-)- $$ 2,3-Dihydroxypropyl palmitate # $$



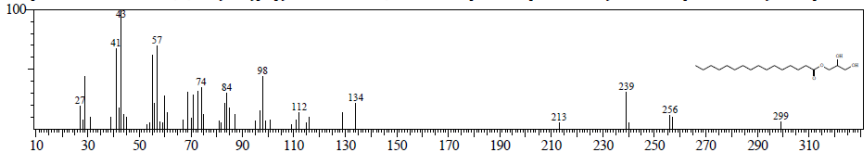
Hit#:2 Entry:110136 Library:NIST05.LIB
SI:86 Formula:C18H36O4 CAS:98863-01-5 MolWeight:316 RetIndex:2399
CompName:Pentadecanoic acid, 2-hydroxy-1-(hydroxymethyl)ethyl ester $$ 2-Hydroxy-1-(hydroxymethyl)ethyl pentadecanoate # $$



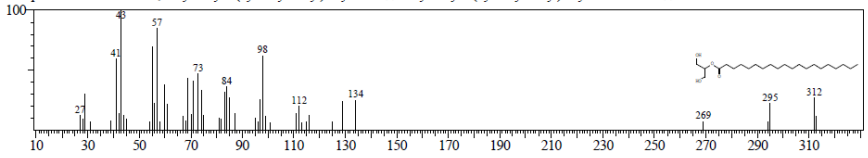
Hit#:3 Entry:117519 Library:NIST05.LIB
SI:86 Formula:C19H38O4 CAS:23470-00-0 MolWeight:330 RetIndex:2498
CompName:Hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl)ethyl ester $$ Palmitin, 2-mono- $$ Palmitic acid .beta.-monoglyceride $$ 2-Hexadecanoyl glycerc



Hit#:4 Entry:117520 Library:NIST05.LIB
SI:86 Formula:C19H38O4 CAS:542-44-9 MolWeight:330 RetIndex:2482
CompName:Hexadecanoic acid, 2,3-dihydroxypropyl ester $$ Palmitin, 1-mono- $$ .alpha.-Monopalmitin $$ Glycerol 1-monopalmitate $$ Glycerol 1-palmitate



Hit#:5 Entry:140926 Library:NIST05.LIB
SI:85 Formula:C23H46O4 CAS:55334-78-6 MolWeight:386 RetIndex:2896
CompName:Eicosanoic acid, 2-hydroxy-1-(hydroxymethyl)ethyl ester $$ 2-Hydroxy-1-(hydroxymethyl)ethyl icosanoate # $$



APPENDIX VI

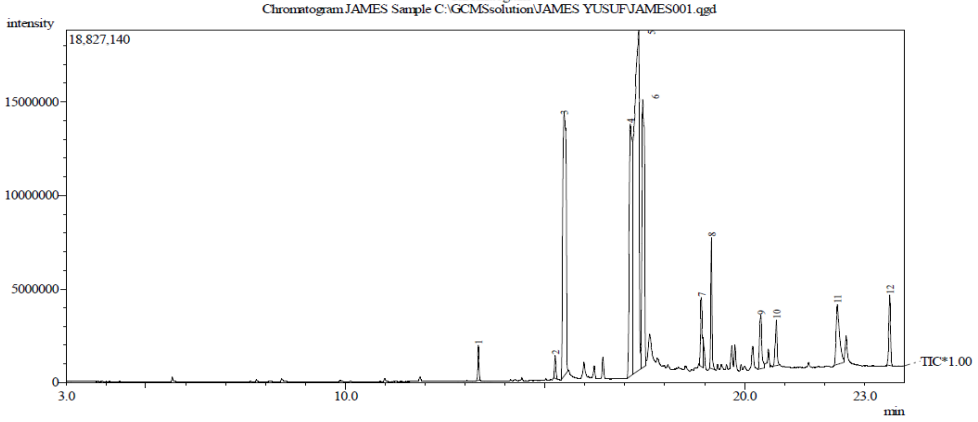
GC-MS Result of biodesiel produced from pumpkin

GCMS-QP2010 PLUS
SHIMADZU,JAPAN

NARICT, ZARIA
GCMS ANALYSIS

JAMES YUSUF (SAMPLE -PUMPKIN)

Chromatogram
Chromatogram JAMES Sample C:\GCMSsolution\JAMES YUSUF\JAMES001.qgd



Method

[Comment]

Analytical Line 1

[AOC-20i]

# of Rinses with Presolvent	:4
# of Rinses with Solvent(post)	:4
# of Rinses with Sample	:3
Plunger Speed(Suction)	:High
Viscosity Comp. Time	:0.2 sec
Plunger Speed(Injection)	:High
Syringe Insertion Speed	:High
Injection Mode	:Normal
Pumping Times	:5
Inj. Port Dwell Time	:0.3 sec
Terminal Air Gap	:No
Plunger Washing Speed	:High
Washing Volume	:8uL
Syringe Suction Position	:0.0 mm
Syringe Injection Position	:0.0 mm
Use 3 Solvent Vial	:1 vial

[GC-2010]

Column Oven Temp.	:70.0 °C
Injection Temp.	:250.00 °C
Injection Mode	:Split
Flow Control Mode	:Linear Velocity
Pressure	:116.9 kPa
Total Flow	:40.8 mL/min
Column Flow	:1.80 mL/min
Linear Velocity	:49.2 cm/sec
Purge Flow	:3.0 mL/min
Split Ratio	:20.0
High Pressure Injection	:OFF
Carrier Gas Saver	:OFF
Splitter Hold	:OFF

Oven Temp. Program:

Rate	Temperature(°C)	Hold Time(min)
-	70.0	0.00
10.00	280.0	5.00

< Ready Check Heat Unit >
Column Oven : Yes
SPL2 : Yes
MS : Yes
< Ready Check Detector(FID) >
< Ready Check Baseline Drift >
< Ready Check Injection Flow >
SPL2 Carrier : Yes
SPL2 Purge : Yes
< Ready Check APC Flow >
< Ready Check Detector APC Flow >
External Wait :No
Equilibrium Time :3.0 min

[GC Program]

[GCMS-QP2010 Plus]
IonSourceTemp :200.00 °C
Interface Temp. :250.00 °C
Solvent Cut Time :2.50 min
Detector Gain Mode :Relative
Detector Gain :0.00 kV
Threshold :2000

[MS Table]
--Group 1 - Event 1--
Start Time :3.00min
End Time :24.00min
ACQ Mode :Scan
Event Time :0.50sec
Scan Speed : 666
Start m/z :30.00
End m/z :350.00

Sample Inlet Unit :GC

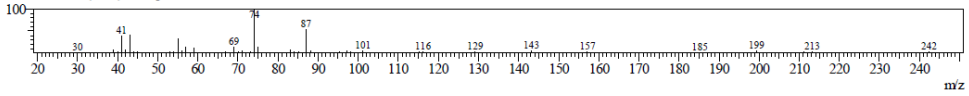
[MS Program]
Use MS Program :OFF

Peak Report TIC

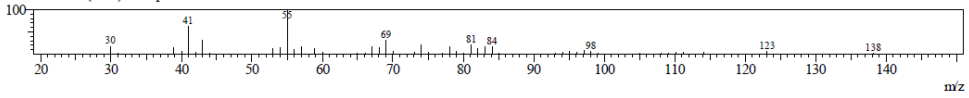
Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	13.328	13.283	13.375	3873605	0.85	1881826	2.18	2.06		
2	15.256	15.217	15.325	3028388	0.66	1232747	1.43	2.46		
3	15.474	15.400	15.583	80526430	17.64	14112208	16.38	5.71		
4	17.135	17.058	17.200	80947833	17.73	13468225	15.63	6.01		
5	17.346	17.200	17.400	151506645	33.18	18168506	21.09	8.34	V	
6	17.451	17.400	17.533	60633076	13.28	14322976	16.63	4.23	V	
7	18.914	18.867	18.950	9877750	2.16	3686311	4.28	2.68		
8	19.166	19.117	19.258	15530985	3.40	7010538	8.14	2.22		
9	20.404	20.333	20.492	12001813	2.63	2837334	3.29	4.23		
10	20.794	20.642	20.875	8125079	1.78	2453188	2.85	3.31	V	
11	22.326	22.242	22.483	18633131	4.08	3204751	3.72	5.81		
12	23.630	23.567	23.700	11934068	2.61	3774269	4.38	3.16		
				456618803	100.00	86152879	100.00			

Spectrum

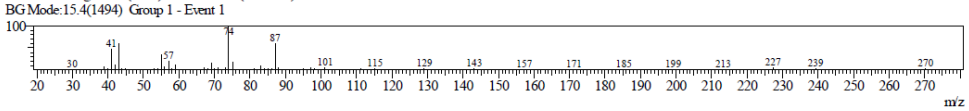
Line#:1 R.Time:13.3(Scan#:1240)
MassPeaks:62
RawMode:Single 13.3(1240) BasePeak:74(161265)
BG Mode:13.3(1242) Group 1 - Event 1



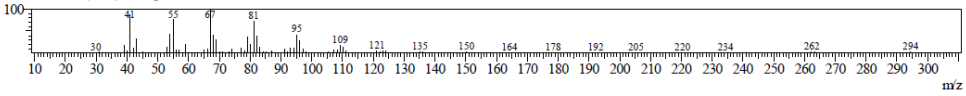
Line#:2 R.Time:15.3(Scan#:1472)
MassPeaks:55
RawMode:Single 15.3(1472) BasePeak:55(11454)
BG Mode:15.3(1471) Group 1 - Event 1



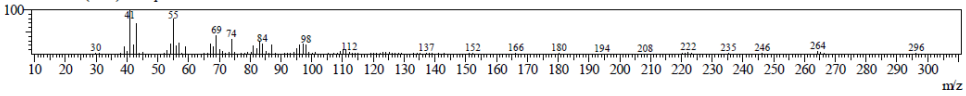
Line#:3 R.Time:15.5(Scan#:1497)
MassPeaks:105
RawMode:Single 15.5(1497) BasePeak:74(1273162)
BG Mode:15.4(1494) Group 1 - Event 1



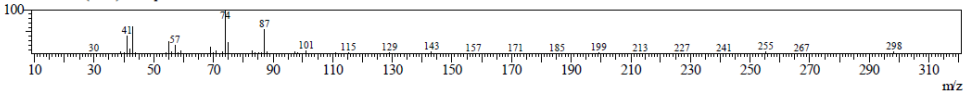
Line#:4 R.Time:17.1(Scan#:1697)
MassPeaks:144
RawMode:Single 17.1(1697) BasePeak:67(1235957)
BG Mode:17.1(1691) Group 1 - Event 1



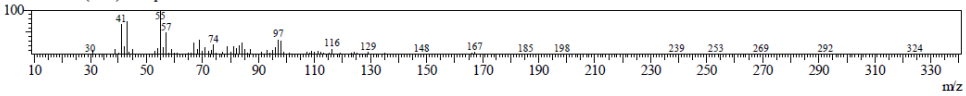
Line#:5 R.Time:17.4(Scan#:1723)
MassPeaks:159
RawMode:Single 17.4(1723) BasePeak:41(1379490)
BG Mode:17.4(1725) Group 1 - Event 1



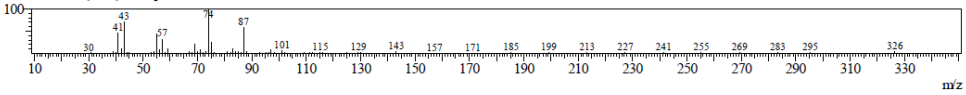
Line#:6 R.Time:17.4(Scan#:1735)
MassPeaks:105
RawMode:Single 17.4(1735) BasePeak:74(734049)
BG Mode:17.4(1733) Group 1 - Event 1



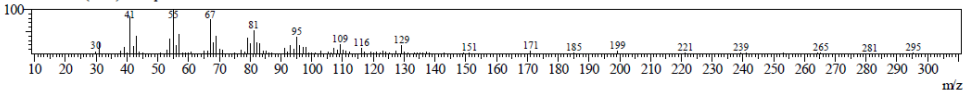
Line#:7 R.Time:18.9(Scan#:1911)
MassPeaks:102
RawMode:Single 18.9(1911) BasePeak:55(362474)
BG Mode:18.9(1907) Group 1 - Event 1



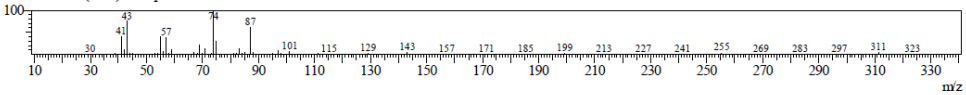
Line#:8 R.Time:19.2(Scan#:1941)
MassPeaks:106
RawMode:Single 19.2(1941) BasePeak:74(947978)
BG Mode:19.1(1938) Group 1 - Event 1



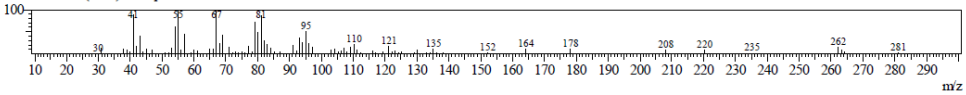
Line#:9 R.Time:20.4(Scan#:2090)
MassPeaks:101
RawMode:Single 20.4(2090) BasePeak:55(41168)
BG Mode:20.4(2091) Group 1 - Event 1



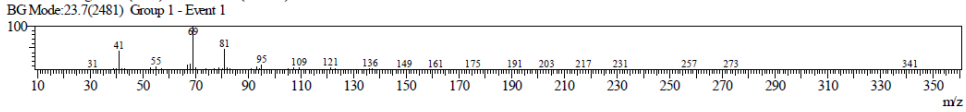
Line#:10 R.Time:20.8(Scan#:2136)
MassPeaks:89
RawMode:Single 20.8(2136) BasePeak:74(344626)
BG Mode:20.7(2130) Group 1 - Event 1



Line#:11 R.Time:22.3(Scan#:2320)
MassPeaks:100
RawMode:Single 22.3(2320) BasePeak:67(26728)
BG Mode:22.3(2322) Group 1 - Event 1

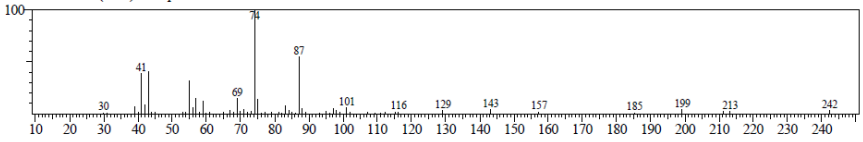


Line#:12 R.Time:23.6(Scan#:2477)
MassPeaks:85
RawMode:Single 23.6(2477) BasePeak:69(928079)
BG Mode:23.7(2481) Group 1 - Event 1

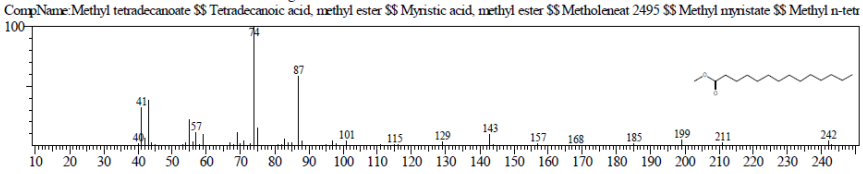


Spectrum Comparison

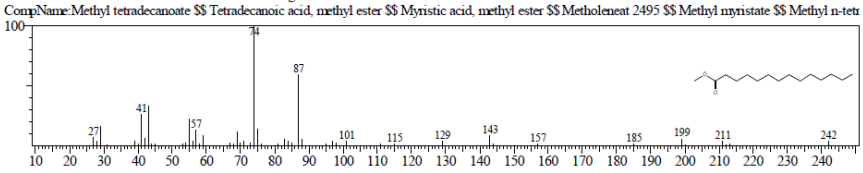
Library
<< Target >>
Line#:1 R.Time:13.325(Scan#:1240) MassPeaks:62
RawMode:Single 13.325(1240) BasePeak:74.05(161265)
BG Mode:13.342(1242) Group 1 - Event 1



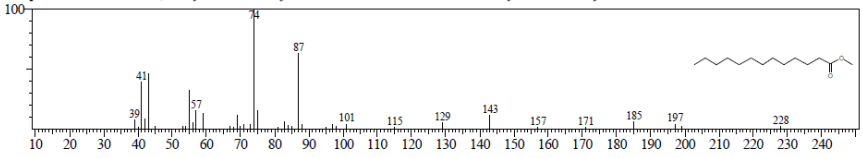
Hit#:1 Entry:20373 Library:NIST05s.LIB
SI:93 Formula:C15H30O2 CAS:124-10-7 MolWeight:242 RetIndex:1680
CompName:Methyl tetradecanoate $$ Tetradecanoic acid, methyl ester $$ Myristic acid, methyl ester $$ Metholeneat 2495 $$ Methyl myristate $$ Methyl n-tetr



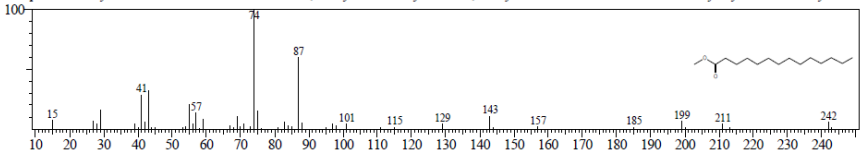
Hit#:2 Entry:20372 Library:NIST05s.LIB
SI:93 Formula:C15H30O2 CAS:124-10-7 MolWeight:242 RetIndex:1680
CompName:Methyl tetradecanoate $$ Tetradecanoic acid, methyl ester $$ Myristic acid, methyl ester $$ Metholeneat 2495 $$ Methyl myristate $$ Methyl n-tetr



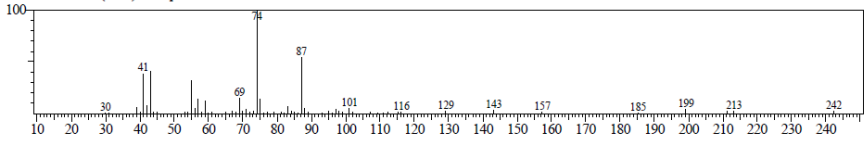
Hit#:3 Entry:19255 Library:NIST05s.LIB
SI:93 Formula:C14H28O2 CAS:1731-88-0 MolWeight:228 RetIndex:1580
CompName:Tridecanoic acid, methyl ester $$ Methyl tridecanoate $$ n-Tridecanoic acid methyl ester $$ Methyl ester of tridecanoic acid $$



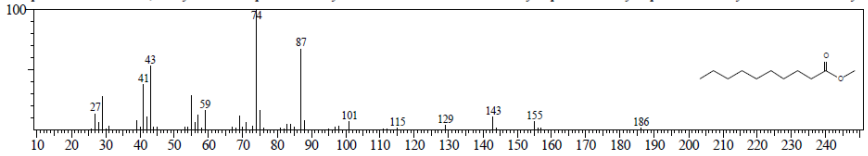
Hit#:4 Entry:20374 Library:NIST05s.LIB
SI:93 Formula:C15H30O2 CAS:124-10-7 MolWeight:242 RetIndex:1680
CompName:Methyl tetradecanoate $$ Tetradecanoic acid, methyl ester $$ Myristic acid, methyl ester $$ Metholeneat 2495 $$ Methyl myristate $$ Methyl n-tetr



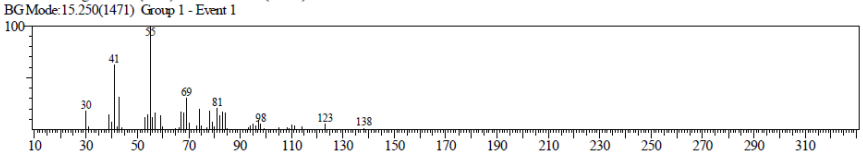
<< Target >>
Line#:1 R.Time:13.325(Scan#:1240) MassPeaks:62
RawMode:Single 13.325(1240) BasePeak:74.05(161265)
BG Mode:13.342(1242) Group 1 - Event 1



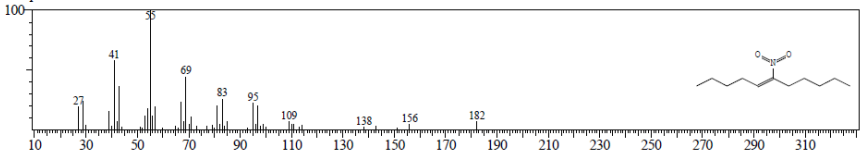
Hit#:5 Entry:14361 Library:NIST05s.LIB
SI:92 Formula:C11H22O2 CAS:110-42-9 MolWeight:186 RetIndex:1282
CompName:Decanoic acid, methyl ester $$ Capric acid methyl ester $$ Metholene 2095 $$ Methyl caprate $$ Methyl caprinate $$ Methyl decanoate $$ Methyl-



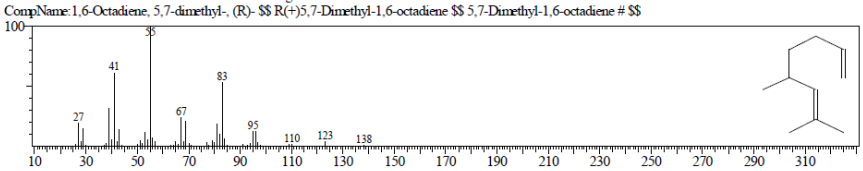
<< Target >>
Line#:2 R.Time:15.258(Scan#:1472) MassPeaks:55
RawMode:Single 15.258(1472) BasePeak:55.00(11454)
BG Mode:15.250(1471) Group 1 - Event 1



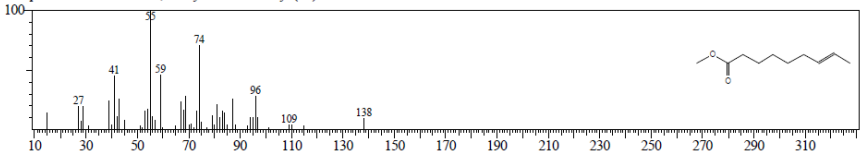
Hit#:1 Entry:40499 Library:NIST05.LIB
SI:84 Formula:C11H21NO2 CAS:0-00-0 MolWeight:199 RetIndex:1481
CompName:6-Nitroundec-5-ene



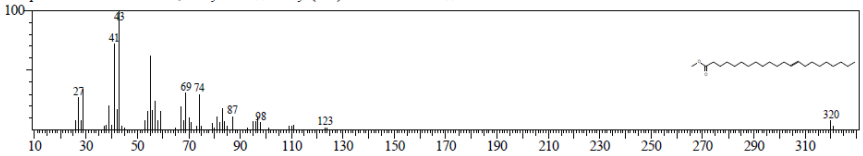
Hit#:2 Entry:10258 Library:NIST05.LIB
SI:82 Formula:C10H18 CAS:85006-04-8 MolWeight:138 RetIndex:926
CompName:1,6-Octadiene, 5,7-dimethyl-, (R)- $$ R(+)5,7-Dimethyl-1,6-octadiene $$ 5,7-Dimethyl-1,6-octadiene # $$



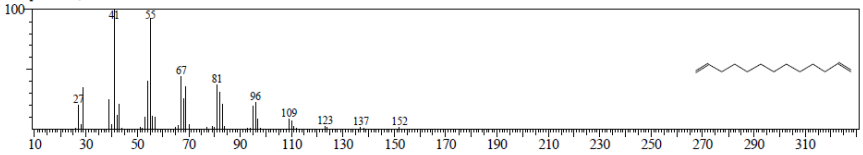
Hit#:3 Entry:24729 Library:NIST05.LIB
SI:81 Formula:C10H18O2 CAS:20731-22-0 MolWeight:170 RetIndex:1191
CompName:7-Nonenoic acid, methyl ester $$ Methyl (7E)-7-nonenoate # $$



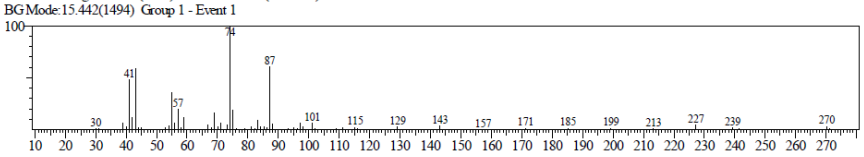
Hit#:4 Entry:128191 Library:NIST05.LIB
SI:81 Formula:C23H44O2 CAS:56630-69-4 MolWeight:352 RetIndex:2483
CompName:13-Docosenoic acid, methyl ester $$ Methyl (13E)-13-docosenoate # $$



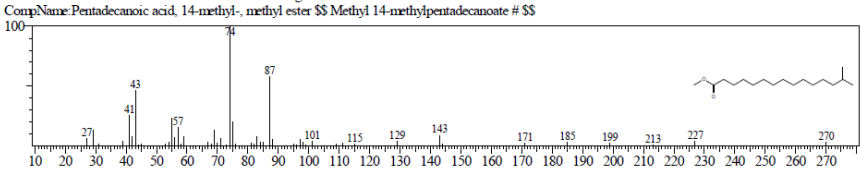
Hit#:5 Entry:13485 Library:NIST05s.LIB
SI:81 Formula:C13H24 CAS:21964-48-7 MolWeight:180 RetIndex:1294
CompName:1,12-Tridecadiene



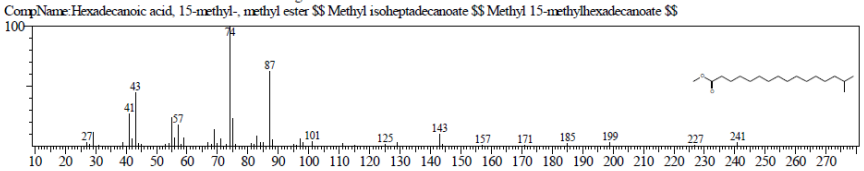
<< Target >>
Line#:3 R.Time:15.467(Scan#:1497) MassPeaks:105
RawMode:Single 15.467(1497) BasePeak:74.00(1273162)
BG Mode:15.442(1494) Group 1 - Event 1



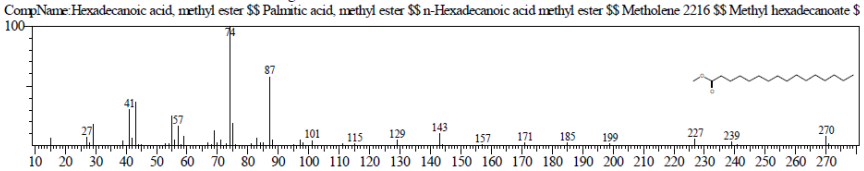
Hit#:1 Entry:22223 Library:NIST05s.LIB
SI:93 Formula:C17H34O2 CAS:5129-60-2 MolWeight:270 RetIndex:1814
CompName:Pentadecanoic acid, 14-methyl-, methyl ester $$ Methyl 14-methylpentadecanoate # $$



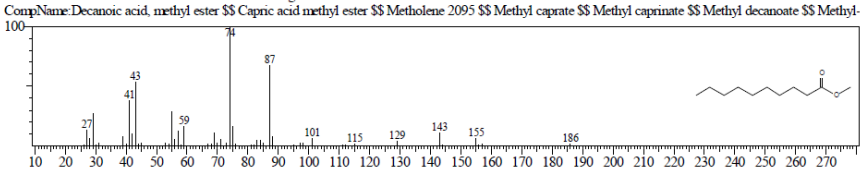
Hit#:2 Entry:22987 Library:NIST05s.LIB
SI:93 Formula:C18H36O2 CAS:6929-04-0 MolWeight:284 RetIndex:1914
CompName:Hexadecanoic acid, 15-methyl-, methyl ester $$ Methyl isoheptadecanoate $$ Methyl 15-methylhexadecanoate $$



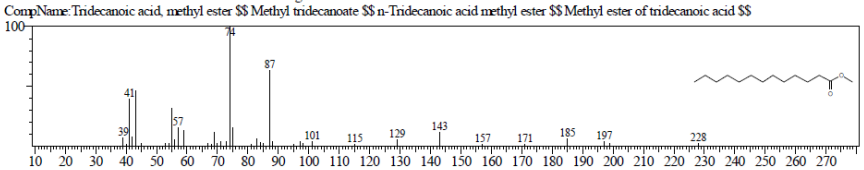
Hit#:3 Entry:22219 Library:NIST05s.LIB
SI:93 Formula:C17H34O2 CAS:112-39-0 MolWeight:270 RetIndex:1878
CompName:Hexadecanoic acid, methyl ester $$ Palmitic acid, methyl ester $$ n-Hexadecanoic acid methyl ester $$ Metholene 2216 $$ Methyl hexadecanoate $



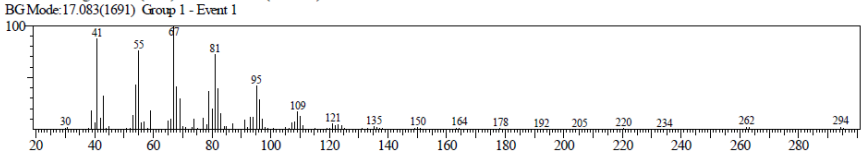
Hit#:4 Entry:14361 Library:NIST05s.LIB
SI:92 Formula:C11H22O2 CAS:110-42-9 MolWeight:186 RetIndex:1282
CompName:Decanoic acid, methyl ester $$ Capric acid methyl ester $$ Metholene 2095 $$ Methyl caprate $$ Methyl caprinate $$ Methyl decanoate $$ Methyl-



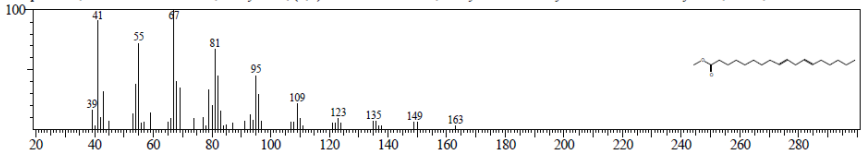
Hit#:5 Entry:19255 Library:NIST05s.LIB
SI:92 Formula:C14H28O2 CAS:1731-88-0 MolWeight:228 RetIndex:1580
CompName:Tridecanoic acid, methyl ester $$ Methyl tridecanoate $$ n-Tridecanoic acid methyl ester $$ Methyl ester of tridecanoic acid $$



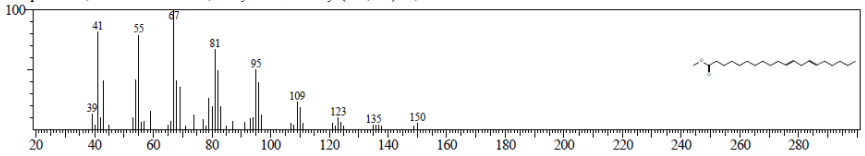
<< Target >>
Line#:4 R.Time:17.133(Scan#:1697) MassPeaks:144
RawMode:Single 17.133(1697) BasePeak:67.00(1235957)
BG Mode:17.083(1691) Group 1 - Event 1



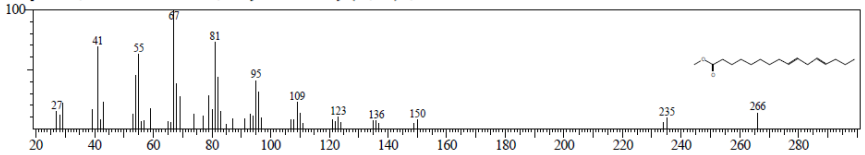
Hit#:1 Entry:23474 Library:NIST05s.LIB
SI:96 Formula:C19H34O2 CAS:2566-97-4 MolWeight:294 RetIndex:2093
CompName:9,12-Octadecadienoic acid, methyl ester, (E,E)- $$ Linolelaidic acid, methyl ester $$ Methyl linolelaidate $$ Methyl trans,trans-9,12-octadecadieno



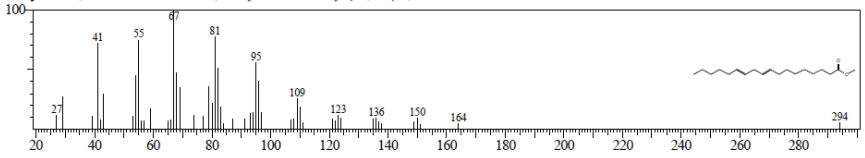
Hit#:2 Entry:113456 Library:NIST05.LIB
SI:95 Formula:C21H38O2 CAS:2463-02-7 MolWeight:322 RetIndex:2292
CompName:11,14-Eicosadienoic acid, methyl ester $$ Methyl (11E,14E)-11,14-icosadienoate # $$



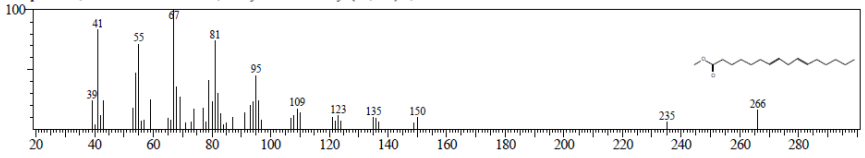
Hit#:3 Entry:80869 Library:NIST05.LIB
SI:93 Formula:C17H30O2 CAS:2462-80-8 MolWeight:266 RetIndex:1894
CompName:9,12-Hexadecadienoic acid, methyl ester $$ Methyl (9E,12E)-9,12-hexadecadienoate # $$



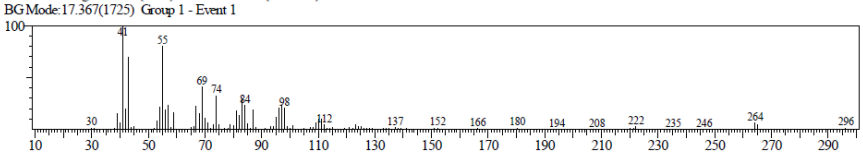
Hit#:4 Entry:97662 Library:NIST05.LIB
SI:93 Formula:C19H34O2 CAS:2462-85-3 MolWeight:294 RetIndex:2093
CompName:9,12-Octadecadienoic acid, methyl ester $$ Methyl (9E,12E)-9,12-octadecadienoate # $$



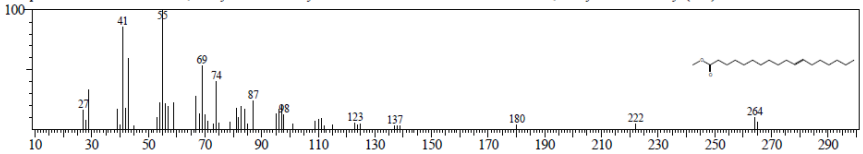
Hit#:5 Entry:80867 Library:NIST05.LIB
SI:93 Formula:C17H30O2 CAS:16106-03-9 MolWeight:266 RetIndex:1894
CompName:7,10-Hexadecadienoic acid, methyl ester $$ Methyl (7E,10E)-7,10-hexadecadienoate # $$



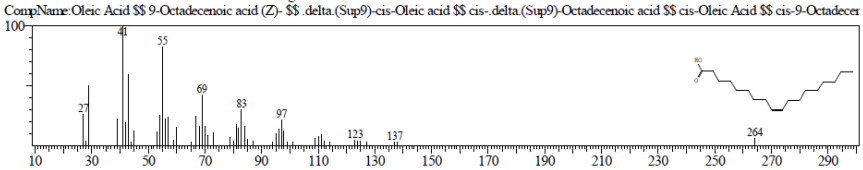
<< Target >>
Line#:5 R.Time:17.350(Scan#:1723) MassPeaks:159
RawMode:Single 17.350(1723) BasePeak:40.95(1379490)
BG Mode:17.367(1725) Group 1 - Event 1



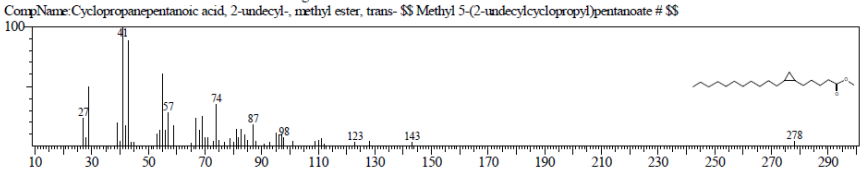
Hit#:1 Entry:98778 Library:NIST05.LIB
SI:93 Formula:C19H36O2 CAS:52380-33-3 MolWeight:296 RetIndex:2085
CompName:11-Octadecenoic acid, methyl ester $$ Methyl 11-octadecenoate $$ Octadec-11-enoic acid, methyl ester $$ Methyl (11E)-11-octadecenoate # $$



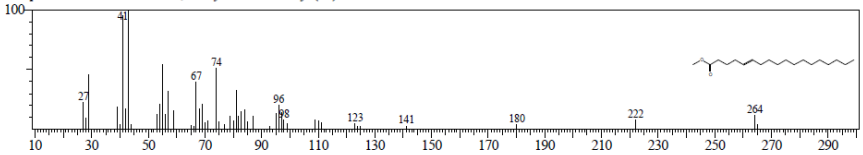
Hit#:2 Entry:22869 Library:NIST05s.LIB
SI:90 Formula:C18H34O2 CAS:112-80-1 MolWeight:282 RetIndex:2175
CompName:Oleic Acid $$ 9-Octadecenoic acid (Z)- $$ .delta.(Sup9)-cis-Oleic acid $$ cis-.delta.(Sup9)-Octadecenoic acid $$ cis-Oleic Acid $$ cis-9-Octadecer



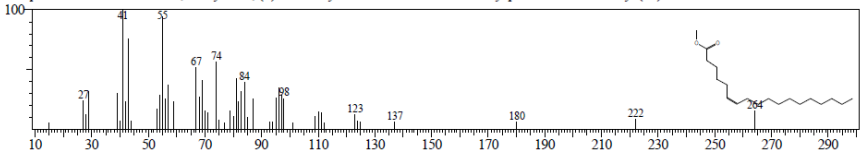
Hit#:3 Entry:106906 Library:NIST05.LIB
SI:90 Formula:C20H38O2 CAS:42199-20-2 MolWeight:310 RetIndex:2140
CompName:Cyclopropanepentanoic acid, 2-undecyl-, methyl ester, trans- $$ Methyl 5-(2-undecylcyclopropyl)pentanoate # $$



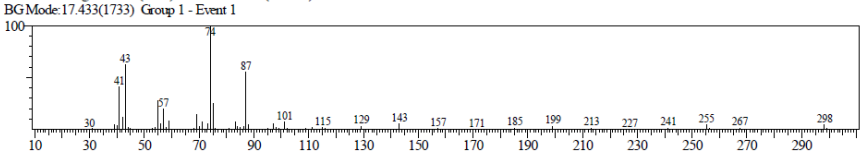
Hit#:4 Entry:23568 Library:NIST05s.LIB
SI:89 Formula:C19H36O2 CAS:56554-45-1 MolWeight:296 RetIndex:2085
CompName:5-Octadecenoic acid, methyl ester $$ Methyl (5E)-5-octadecenoate # $$



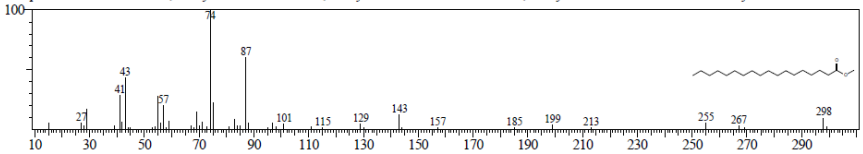
Hit#:5 Entry:23567 Library:NIST05s.LIB
SI:89 Formula:C19H36O2 CAS:2777-58-4 MolWeight:296 RetIndex:2085
CompName:6-Octadecenoic acid, methyl ester, (Z)- $$ Methyl cis-6-octadecenoate $$ Methyl petroselinate $$ Methyl (6Z)-6-octadecenoate # $$



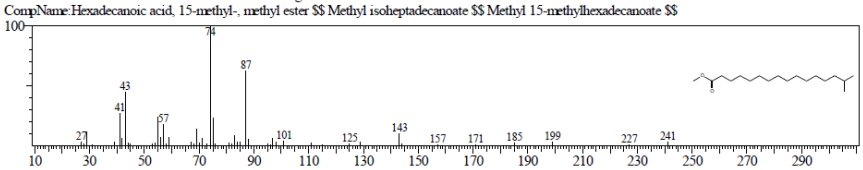
<< Target >>
Line#:6 R.Time:17.450(Scan#:1735) MassPeaks:105
RawMode:Single 17.450(1735) BasePeak:74.00(734049)
BG Mode:17.433(1733) Group 1 - Event 1



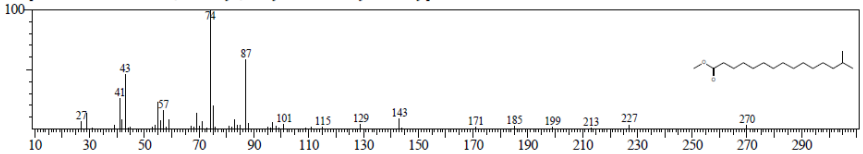
Hit#:1 Entry:100069 Library:NIST05.LIB
SI:93 Formula:C19H38O2 CAS:112-61-8 MolWeight:298 RetIndex:2077
CompName:Octadecanoic acid, methyl ester $$ Stearic acid, methyl ester $$ n-Octadecanoic acid, methyl ester $$ Kemester 9718 $$ Methyl n-octadecanoate $$



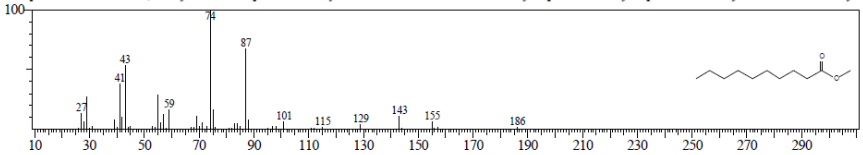
Hit#:2 Entry:22987 Library:NIST05s.LIB
SI:92 Formula:C18H36O2 CAS:6929-04-0 MolWeight:284 RetIndex:1914
CompName:Hexadecanoic acid, 15-methyl-, methyl ester $$ Methyl isoheptadecanoate $$ Methyl 15-methylhexadecanoate $$



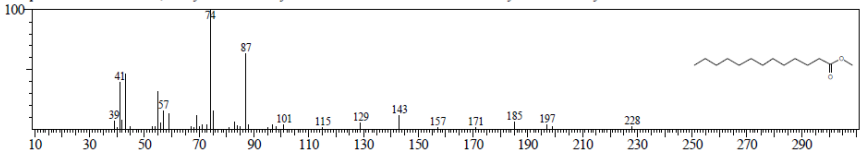
Hit#:3 Entry:22223 Library:NIST05s.LIB
SI:91 Formula:C17H34O2 CAS:5129-60-2 MolWeight:270 RetIndex:1814
CompName:Pentadecanoic acid, 14-methyl-, methyl ester $$ Methyl 14-methylpentadecanoate # $$



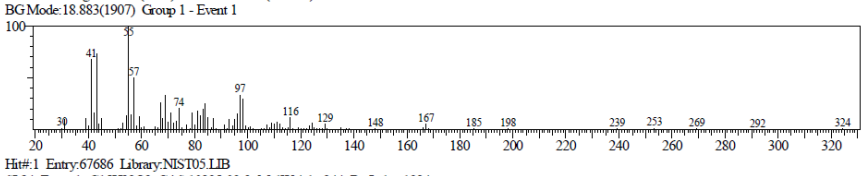
Hit#:4 Entry:14361 Library:NIST05s.LIB
SI:91 Formula:C11H22O2 CAS:110-42-9 MolWeight:186 RetIndex:1282
CompName:Decanoic acid, methyl ester $$ Capric acid methyl ester $$ Metholene 2095 $$ Methyl caprate $$ Methyl caprinate $$ Methyl decanoate $$ Methyl-



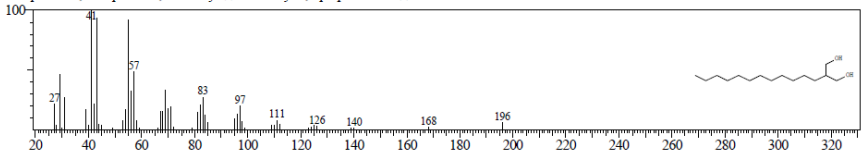
Hit#:5 Entry:19255 Library:NIST05s.LIB
SI:91 Formula:C14H28O2 CAS:1731-88-0 MolWeight:228 RetIndex:1580
CompName:Tridecanoic acid, methyl ester $$ Methyl tridecanoate $$ n-Tridecanoic acid methyl ester $$ Methyl ester of tridecanoic acid $$



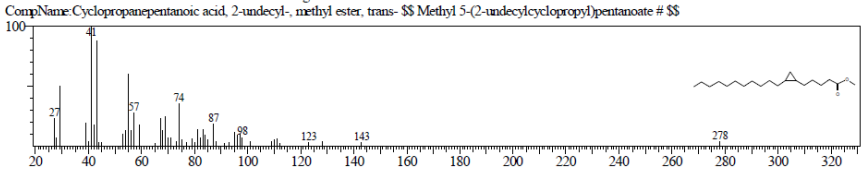
<< Target >>
Line#:7 R.Time:18.917(Scan#:1911) MassPeaks:102
RawMode:Single 18.917(1911) BasePeak:55.00(362474)
BG Mode:18.883(1907) Group 1 - Event 1



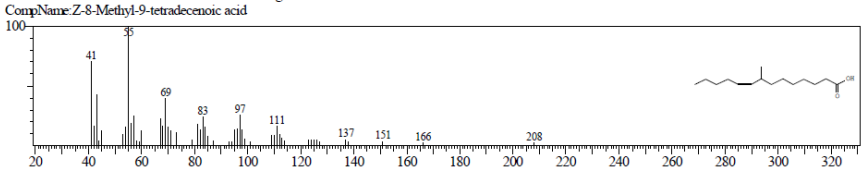
Hit#:1 Entry:67686 Library:NIST05.LIB
SI:84 Formula:C15H32O2 CAS:10395-09-2 MolWeight:244 RetIndex:1934
CompName:1,3-Propanediol, 2-dodecyl $$ 2-Dodecyl-1,3-propanediol # $$



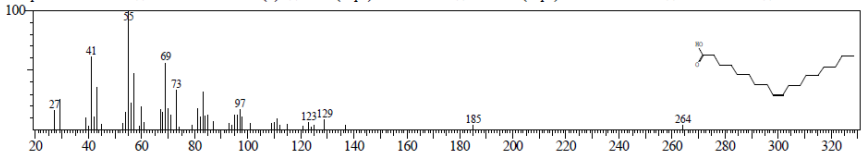
Hit#:2 Entry:106906 Library:NIST05.LIB
SI:84 Formula:C20H38O2 CAS:42199-20-2 MolWeight:310 RetIndex:2140
CompName:Cyclopropanepentanoic acid, 2-undecyl-, methyl ester, trans- $$ Methyl 5-(2-undecylcyclopropyl)pentanoate # $$



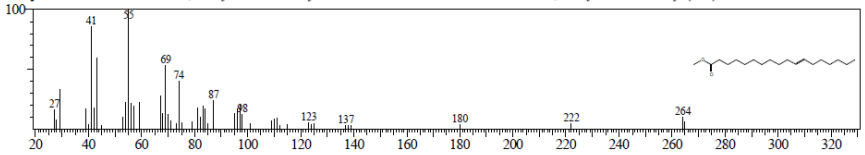
Hit#:3 Entry:65289 Library:NIST05.LIB
SI:84 Formula:C15H28O2 CAS:0-00-0 MolWeight:240 RetIndex:1813
CompName:Z-8-Methyl-9-tetradecenoic acid



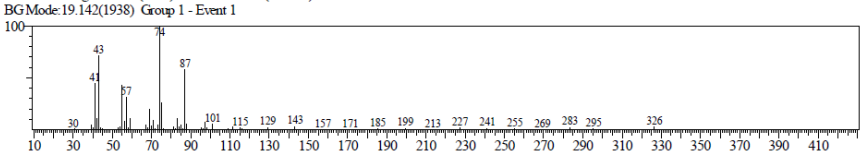
Hit#:4 Entry:22870 Library:NIST05s.LIB
SI:84 Formula:C18H34O2 CAS:112-80-1 MolWeight:282 RetIndex:2175
CompName:Oleic Acid $$ 9-Octadecenoic acid (Z)- $$ .delta.(Sup9)-cis-Oleic acid $$ cis-.delta.(Sup9)-Octadecenoic acid $$ cis-Oleic Acid $$ cis-9-Octadecer



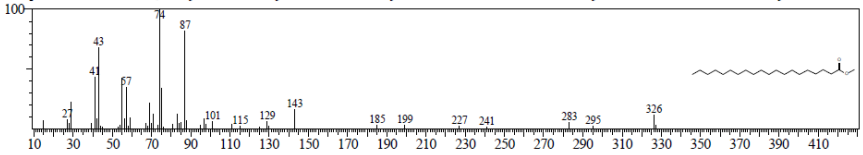
Hit#:5 Entry:98778 Library:NIST05.LIB
SI:84 Formula:C19H36O2 CAS:52380-33-3 MolWeight:296 RetIndex:2085
CompName:11-Octadecenoic acid, methyl ester $$ Methyl 11-octadecenoate $$ Octadec-11-enoic acid, methyl ester $$ Methyl (11E)-11-octadecenoate # $$



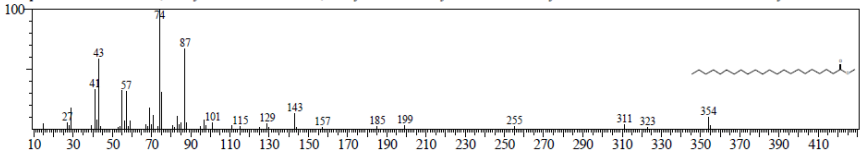
<< Target >>
Line#:8 R.Time:19.167(Scan#:1941) MassPeaks:106
RawMode:Single 19.167(1941) BasePeak:74.05(947978)
BG Mode:19.142(1938) Group 1 - Event 1



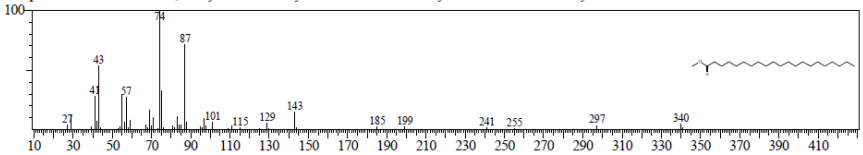
Hit#:1 Entry:24788 Library:NIST05s.LIB
SI:94 Formula:C21H42O2 CAS:1120-28-1 MolWeight:326 RetIndex:2276
CompName:Eicosanoic acid, methyl ester $$ Methyl arachisate $$ Methyl eicosanoate $$ Arachidic acid methyl ester $$ Kemester 2050 $$ Methyl aracidate $$



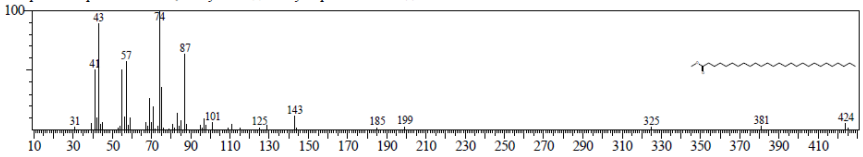
Hit#:2 Entry:25584 Library:NIST05s.LIB
SI:92 Formula:C23H46O2 CAS:929-77-1 MolWeight:354 RetIndex:2475
CompName:Docosanoic acid, methyl ester $$ Behenic acid, methyl ester $$ Methyl behenate $$ Methyl docosanoate $$ n-Docosanoic acid methyl ester $$ Kem



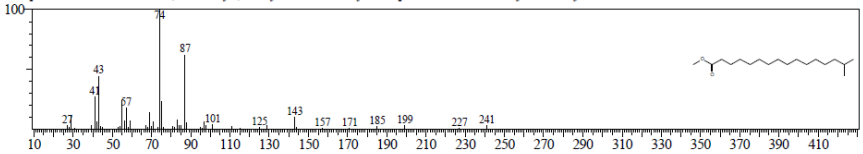
Hit#:3 Entry:25264 Library:NIST05s.LIB
SI:92 Formula:C22H44O2 CAS:6064-90-0 MolWeight:340 RetIndex:2375
CompName:Heneicosanoic acid, methyl ester $$ Methyl heneicosanoate $$ Methyl henelcosanoate $$ Methyl henicosanoate # $$



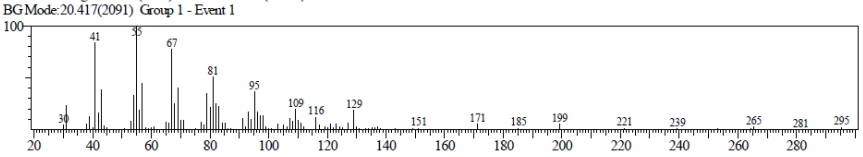
Hit#:4 Entry:26833 Library:NIST05s.LIB
SI:92 Formula:C28H56O2 CAS:55682-91-2 MolWeight:424 RetIndex:2972
CompName:Heptacosanoic acid, methyl ester $$ Methyl heptacosanoate # $$



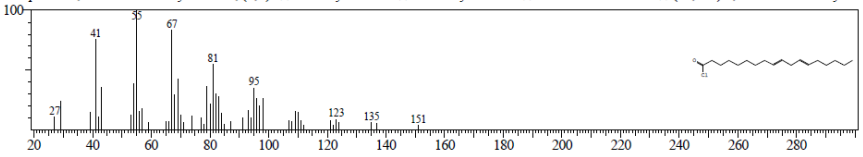
Hit#:5 Entry:22987 Library:NIST05s.LIB
SI:91 Formula:C18H36O2 CAS:6929-04-0 MolWeight:284 RetIndex:1914
CompName:Hexadecanoic acid, 15-methyl-, methyl ester $$ Methyl isoheptadecanoate $$ Methyl 15-methylhexadecanoate $$



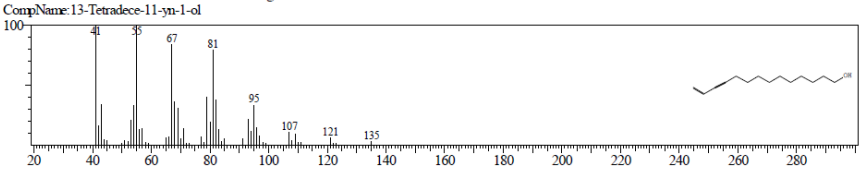
<< Target >>
Line#:9 R.Time:20.408(Scan#:2090) MassPeaks:101
RawMode:Single 20.408(2090) BasePeak:55.00(41168)
BG Mode:20.417(2091) Group 1 - Event 1



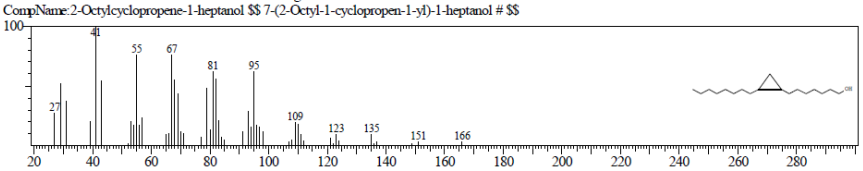
Hit#:1 Entry:23649 Library:NIST05s.LIB
SI:88 Formula:C18H31ClO CAS:7459-33-8 MolWeight:298 RetIndex:2139
CompName:9,12-Octadecadienoyl chloride, (Z,Z)- $$ Linoleoyl chloride $$ Lineoleoyl chloride $$ Linoleic acid chloride $$ (9E,12E)-9,12-Octadecadienoyl ch



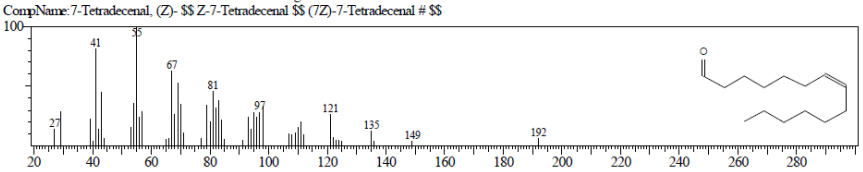
Hit#:2 Entry:46036 Library:NIST05.LIB
SI:86 Formula:C14H24O CAS:0-00-0 MolWeight:208 RetIndex:1663
CompName:13-Tetradece-11-yn-1-ol



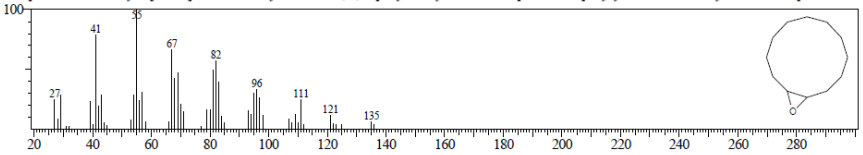
Hit#:3 Entry:80929 Library:NIST05.LIB
SI:86 Formula:C18H34O CAS:54467-85-5 MolWeight:266 RetIndex:2056
CompName:2-Octylcyclopropene-1-heptanol $$ 7-(2-Octyl-1-cyclopropen-1-yl)-1-heptanol # $$



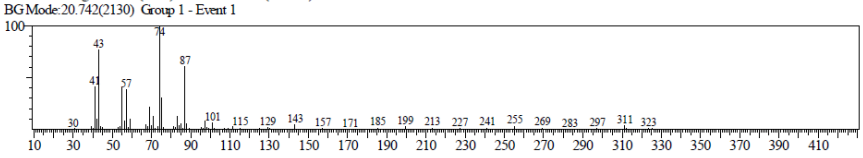
Hit#:4 Entry:47275 Library:NIST05.LIB
SI:86 Formula:C14H26O CAS:65128-96-3 MolWeight:210 RetIndex:1609
CompName:7-Tetradecenal, (Z)- $$ Z-7-Tetradecenal $$ (7Z)-7-Tetradecenal # $$



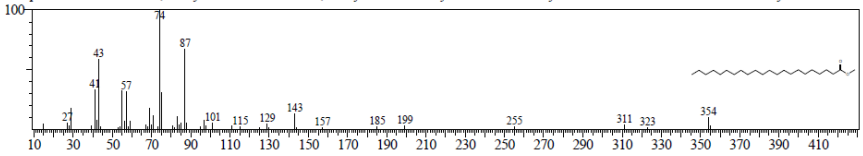
Hit#:5 Entry:31249 Library:NIST05.LIB
SI:86 Formula:C12H22O CAS:286-99-7 MolWeight:182 RetIndex:1450
CompName:13-Oxabicyclo[10.1.0]tridecane $$ Cyclododecane, 1,2-epoxy- $$ Cyclododecene epoxide $$ Epoxycyclododecane $$ Cyclododecane epoxide $$ C



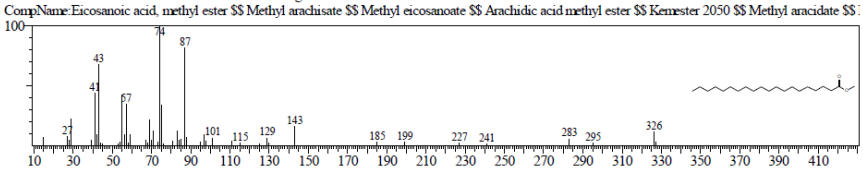
<< Target >>
Line#:10 R.Time:20.792(Scan#:2136) MassPeaks:89
RawMode:Single 20.792(2136) BasePeak:74.05(344626)
BG Mode:20.742(2130) Group 1 - Event 1



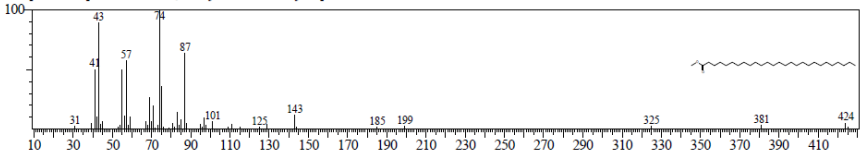
Hit#:1 Entry:25584 Library:NIST05s.LIB
SI:93 Formula:C23H46O2 CAS:929-77-1 MolWeight:354 RetIndex:2475
CompName:Docosanoic acid, methyl ester $$ Behenic acid, methyl ester $$ Methyl behenate $$ Methyl docosanoate $$ n-Docosanoic acid methyl ester $$ Kem



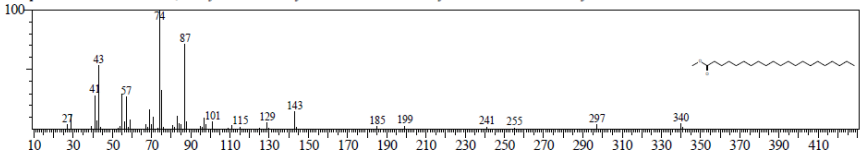
Hit#:2 Entry:24788 Library:NIST05s.LIB
SI:93 Formula:C21H42O2 CAS:1120-28-1 MolWeight:326 RetIndex:2276
CompName:Eicosanoic acid, methyl ester $$ Methyl arachisate $$ Methyl eicosanoate $$ Arachidic acid methyl ester $$ Kemester 2050 $$ Methyl aracidate $$



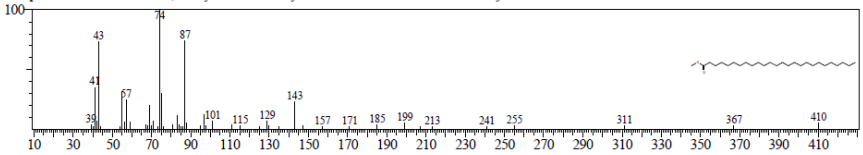
Hit#:3 Entry:26833 Library:NIST05s.LIB
SI:93 Formula:C28H56O2 CAS:55682-91-2 MolWeight:424 RetIndex:2972
CompName:Heptacosanoic acid, methyl ester $$ Methyl heptacosanoate # $$



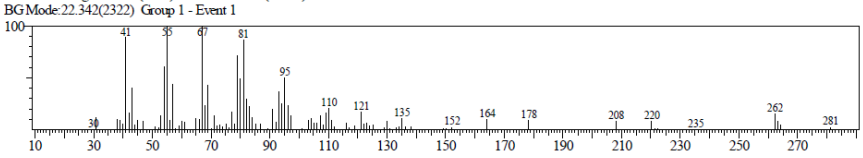
Hit#:4 Entry:25264 Library:NIST05s.LIB
SI:92 Formula:C22H44O2 CAS:6064-90-0 MolWeight:340 RetIndex:2375
CompName:Heneicosanoic acid, methyl ester $$ Methyl heneicosanoate $$ Methyl henelcosanoate $$ Methyl henicosanoate # $$



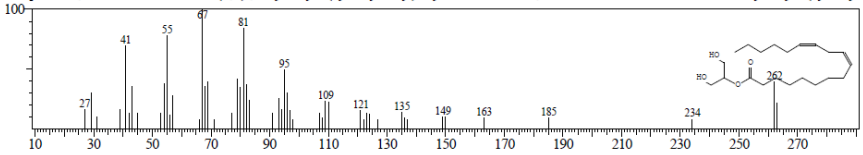
Hit#:5 Entry:26664 Library:NIST05s.LIB
SI:92 Formula:C27H54O2 CAS:5802-82-4 MolWeight:410 RetIndex:2872
CompName:Hexacosanoic acid, methyl ester $$ Methyl hexacosanoate $$ Cerotic acid methyl ester $$



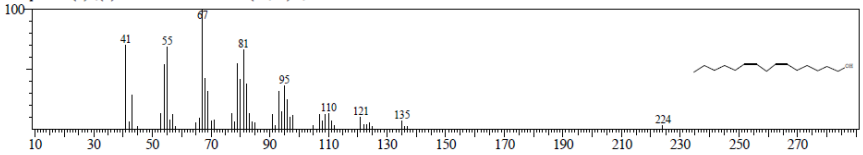
<< Target >>
Line#:11 R.Time:22.325(Scan#:2320) MassPeaks:100
RawMode:Single 22.325(2320) BasePeak:67.05(26728)
BG Mode:22.342(2322) Group 1 - Event 1



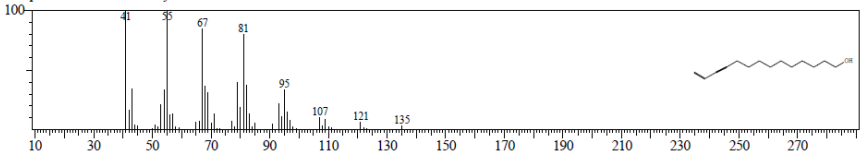
Hit#:1 Entry:128977 Library:NIST05.LIB
SI:84 Formula:C21H38O4 CAS:3443-82-1 MolWeight:354 RetIndex:2713
CompName:9,12-Octadecadienoic acid (Z,Z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester $$ Linolein, 2-mono- $$ .beta.-Monolinolein $$ 2-Hydroxy-1-(hydroxyr



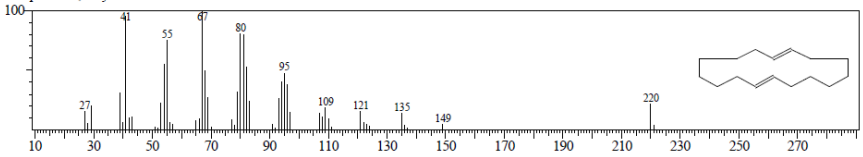
Hit#:2 Entry:55812 Library:NIST05.LIB
SI:84 Formula:C15H28O CAS:77899-11-7 MolWeight:224 RetIndex:1771
CompName:(Z)6,(Z)9-Pentadecadien-1-ol $$ (6Z,9Z)-6,9-Pentadecadien-1-ol # $$



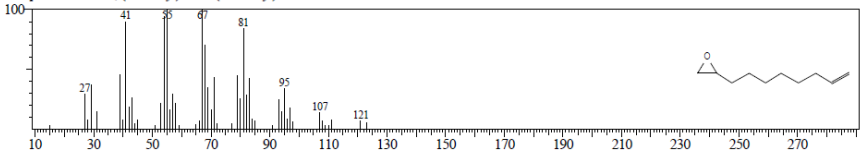
Hit#:3 Entry:46036 Library:NIST05.LIB
SI:83 Formula:C14H24O CAS:0-00-0 MolWeight:208 RetIndex:1663
CompName:13-Tetradece-11-yn-1-ol



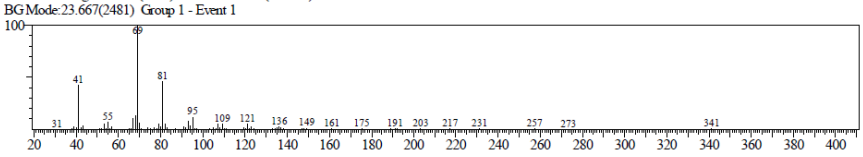
Hit#:4 Entry:53196 Library:NIST05.LIB
SI:83 Formula:C16H28 CAS:4277-06-9 MolWeight:220 RetIndex:1883
CompName:1,9-Cyclohexadecadiene



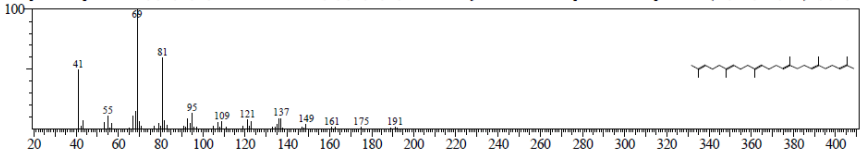
Hit#:5 Entry:16918 Library:NIST05.LIB
SI:82 Formula:C10H18O CAS:85721-25-1 MolWeight:154 RetIndex:1096
CompName:Oxirane, (7-octenyl)- $$ 2-(7-Octenyl)oxirane # $$



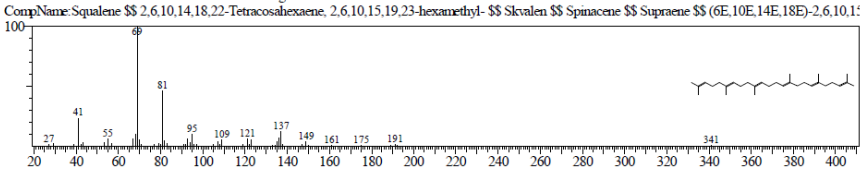
<< Target >>
Line#:12 R.Time:23.633(Scan#:2477) MassPeaks:85
RawMode:Single 23.633(2477) BasePeak:69.05(928079)
BG Mode:23.667(2481) Group 1 - Event 1



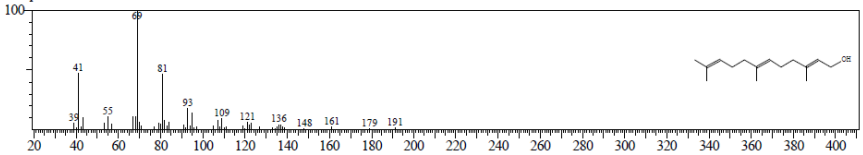
Hit#:1 Entry:26669 Library:NIST05s.LIB
SI:92 Formula:C30H50 CAS:7683-64-9 MolWeight:410 RetIndex:2914
CompName:Squalene $$ 2,6,10,14,18,22-Tetracosahexaene, 2,6,10,15,19,23-hexamethyl- $$ Skvalen $$ Spinacene $$ Supraene $$ (6E,10E,14E,18E)-2,6,10,1



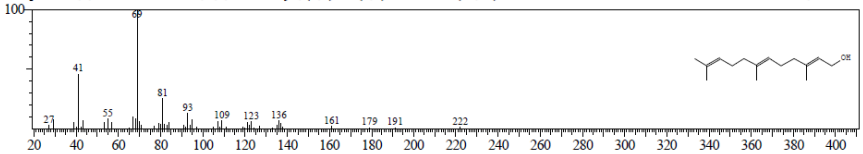
Hit#:2 Entry:26668 Library:NIST05s.LIB
SI:92 Formula:C30H50 CAS:7683-64-9 MolWeight:410 RetIndex:2914
CompName:Squalene $$ 2,6,10,14,18,22-Tetracosahexaene, 2,6,10,15,19,23-hexamethyl- $$ Skvalen $$ Spinacene $$ Supraene $$ (6E,10E,14E,18E)-2,6,10,1



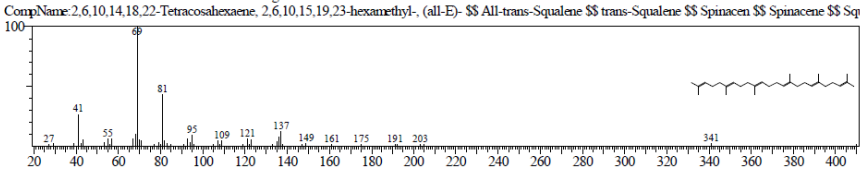
Hit#:3 Entry:54489 Library:NIST05.LIB
SI:91 Formula:C15H26O CAS:0-00-0 MolWeight:222 RetIndex:1710
CompName:Farnesol isomer a



Hit#:4 Entry:18658 Library:NIST05s.LIB
SI:90 Formula:C15H26O CAS:106-28-5 MolWeight:222 RetIndex:1710
CompName:2,6,10-Dodecatrien-1-ol, 3,7,11-trimethyl-, (E,E)- $$ (E,E)-Farnesol $$ (2E,6E)-Farnesol $$ All-trans-Farnesol $$ trans-Farnesol $$ trans,trans-Far



Hit#:5 Entry:26671 Library:NIST05s.LIB
SI:90 Formula:C30H50 CAS:111-02-4 MolWeight:410 RetIndex:2914
CompName:2,6,10,14,18,22-Tetracosahexaene, 2,6,10,15,19,23-hexamethyl-, (all-E)- $$ All-trans-Squalene $$ trans-Squalene $$ Spinacen $$ Spinacene $$ Sq



APPENDIX VII



FIG. 3.2 SETTLING AND WASHING