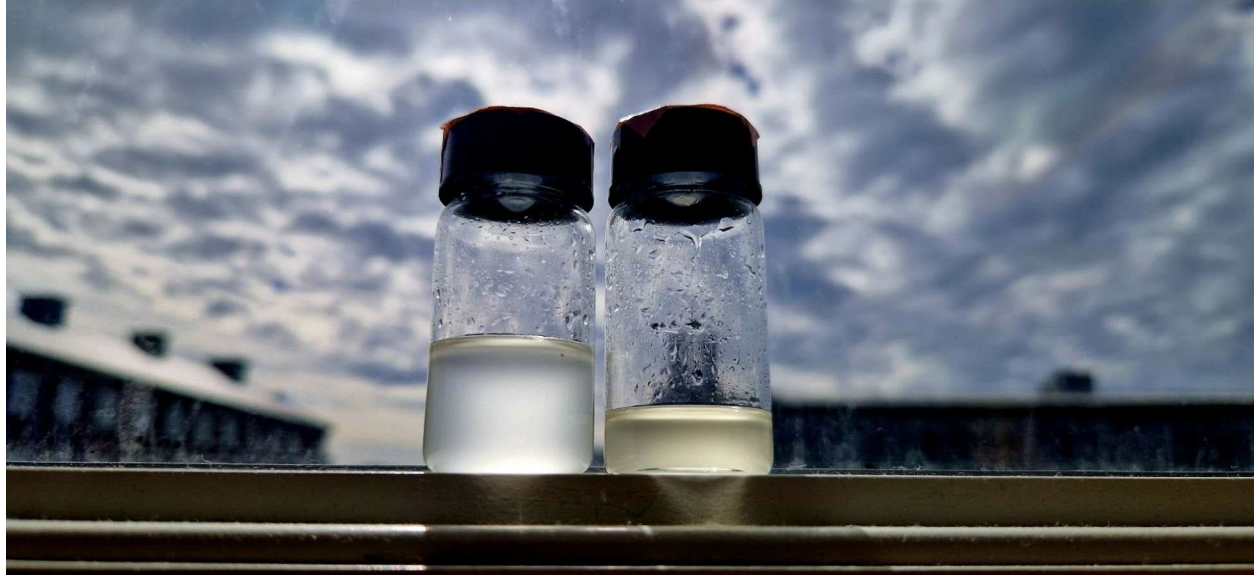




CHALMERS
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Synthesis and evaluation of bio-based esters from renewable alcohols and acids as potential lubricant base oils for industrial applications

Bachelor's thesis in chemical Engineering

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CHALMERS UNIVERSITY OF TECHNOLOGY
Gothenburg, Sweden 2026
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Abstract

Lubricants play a critical role in reducing friction and wear in industrial and mechanical systems, yet conventional lubricant base oils are predominantly derived from fossil resources. In response to increasing environmental concerns and the growing demand for sustainable materials, bio-based esters have emerged as promising alternatives for lubricant applications.

The aim of this study was to synthesize and evaluate bio-based esters derived from renewable alcohols and platform acids as potential lubricant base oils. Esterification reactions were performed using lactic acid, levulinic acid and itaconic acid together with various alcohols including 1-dodecanol, 1-undecanol, 2-ethyl-1-hexanol and oleyl alcohol through Fischer-Speier esterification. The synthesized products were characterized using FTIR and NMR spectroscopy, while rheological measurements were conducted to evaluate viscosity behavior and viscosity index characteristics.

The results confirmed successful ester formation for several synthesized products through characteristic ester signals observed in both FTIR and NMR analyses. Rheological evaluation demonstrated that the synthesized esters exhibited significantly different flow behaviors depending on molecular structure and ester composition. Oleyl lactate and oleyl levulinate were identified as the most promising candidates due to their oil-like characteristics, favorable viscosity behavior and comparatively high apparent viscosity index values. Oleyl lactate exhibited viscosity index values comparable to commercial synthetic lubricants, while oleyl levulinate showed particularly high apparent viscosity stability under defined shear conditions.

Overall, the study demonstrates the potential of renewable platform-acid-derived esters as sustainable lubricant base oil candidates. The results further highlight the importance of considering shear-dependent rheological behavior when evaluating bio-based ester systems using conventional viscosity index methods. Further work involving tribological performance, oxidation stability and long-term lubricant testing is required to fully assess their industrial applicability.

Keywords: Bio-based lubricants, Esterification, Lubricant base oils, Lactic acid, Levulinic acid, Itaconic acid, Rheology, Viscosity index, Renewable esters, Fischer-Speier esterification

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List of Acronyms

Below is the list of acronyms that have been used throughout this thesis listed in alphabetical order:

EHL	Elastohydrodynamic Lubrication
FTIR	Fourier-transform infrared
NMR	Nuclear Magnetic Resonance
PLA	Polylactic Acid
p-TSA	Para-Toluenesulfonic acid
TMS	Tetramethylsilane
VI	Viscosity Index

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1

Introduction

The continued reliance on fossil-based alternatives as building blocks in various industrial sectors represents today a major global challenge. This resource remains the dominant source of carbon dioxide emissions and a primary driver of climate change and pollution, highlighting the urgent need for more sustainable alternatives [1, 2]. This challenge is particularly relevant in industrial applications, where fossil-based raw materials are still widely used, including as base oils in diverse lubrication categories [3].

In this study, bio-based esters derived from renewable alcohols and organic acids are synthesized and evaluated to investigate their potential as lubricant base oils for various industrial applications, by analysing the structure and physicochemical properties of the synthesized esters as well as evaluating their lubrication performance.

1.1 Background

Lubrication is essential for the performance and lifetime of bearings because a lubricant film separates the contact surfaces and reduces friction and wear [4, 5]. By preventing direct metal-to-metal contact, the lubricant not only minimizes surface damage but also reduces energy loss during operation as well as heat generation. Insufficient or improper lubrication has been identified as one of the most common causes of bearing failure in rotating machinery [5, 6]. Conventional lubricants are largely based on petroleum-derived mineral oils [7]. However, increasing environmental concerns and the need for sustainable technologies have driven research toward bio-based lubricants derived from renewable resources [8]. Ester-based oils synthesized from alcohols and carboxylic acids are considered promising candidates for sustainable lubricants because they can exhibit high lubricity, favourable viscosity-temperature behaviour and potential biodegradability [8, 9].

1.2 Aim

The aim of this thesis is to investigate the potential of ester-based, bio derived lubricants as sustainable alternatives to conventional fossil based mineral oils in rolling bearing applications. The study specifically seeks to evaluate whether these bio-based lubricants can provide sufficient lubricant performance in terms of friction reduction, wear resistance, and thermal resistance, while also contributing to

improved environmental sustainability.

Additionally, the thesis aims to analyze and evaluate the key properties of ester-based oils, such as viscosity, temperature characteristics, and lubricity, and assess how these influence the formation and stability of the lubricant film in rolling contacts. By comparing their performance with that of traditional mineral oils, the study aims to determine the feasibility of implementing bio-based lubricants in real world engineering applications.

1.3 Limitations / Demarcations

Due to time limitations, NMR and viscosity analysis of product from Cetylalcohol could not be completed. Consequently, a comprehensive evaluation and conclusion regarding the properties and performance of the resulting lubricants could not be reached.

2

Theory

In the following sections, information on lubrication and tribology, lubricant synthesis, lubricant properties, comparison between conventional and bio-based lubricants, and selection of Renewable Alcohols and Carboxylic Acids and their properties are described.

2.1 Lubrication and Tribology

Tribology is the science of friction, wear, and lubrication interacting surfaces that are in relative motion. It encompasses the study of how interacting surfaces behave under mechanical load and how lubricants can be used to reduce friction and material degradation. Within, for example rolling bearings, tribology is essential as there is a lot of localized sliding and very high contact pressures that occur at the contact between rolling elements and bearing race [10]. These conditions make proper lubrication critical for efficient rolling operation and long service life. Tribology plays a crucial role in the design and operation of industrial systems, including gears, bearings, compressors, hydraulic equipment, turbines, and manufacturing machinery.

Lubrication is the application of a substance, typically a liquid or semi-solid lubricant, to separate contacting surfaces and thereby reduce friction and wear. When a sufficiently thick lubricant film is established, direct metal-to-metal contact is prevented, which minimizes surface damage, decreases energy losses, and reduces heat generation. In addition to reducing friction and wear, lubricants also contribute to cooling, corrosion protection, sealing, and the removal of contaminants from contact zones. These functions are essential for maintaining the efficiency, reliability, and service life of industrial equipment [10].

According to Nguyen-Schäfer, lubrication in many industrial machine elements is primarily governed by hydrodynamic and elastohydrodynamic lubrication (EHL) [10]. EHL conditions are common in highly loaded concentrated contacts such as gears, rolling bearings, and cam-follower systems, where very high pressures cause elastic deformation of the contacting surfaces and a significant increase in lubricant viscosity. This enables the formation of an extremely thin lubricant film, often only a few hundred nanometers thick, which is nevertheless sufficient to separate the

surfaces and support the applied load [4, 5, 10].

Lubricants are commonly supplied as oils or greases depending on the application and operating conditions. In grease-lubricated systems, the base oil is gradually released from the thickener and forms the lubricating film between contacting surfaces. The thickness and effectiveness of this film depend on several factors, including lubricant viscosity, operating speed, temperature, and applied load. If the lubricant film becomes too thin, the contact may enter mixed or boundary lubrication regimes, where partial surface contact occurs and wear increases significantly [4, 5, 7, 10]

In addition to reducing friction and wear, lubricants contribute to heat dissipation, corrosion protection, sealing, and contamination control. These functions are essential in a wide range of industrial applications, including bearings, gearboxes, hydraulic systems, compressors, and turbines. Therefore, the selection and performance of the lubricant are key factors in determining equipment reliability and service life.

2.2 Base oils and Esters

Base oils constitute the primary component of industrial lubricants and are responsible for the formation of a lubricant film that separates contacting surfaces under operating conditions. Normally, these base oils are derived from petroleum refining processes and are commonly known as mineral oils [11]. However, increasing environmental concerns and the need for sustainable alternatives have led to the development of synthetic and bio-based base oils, notably ester-based lubricants [12].

2.2.1 Mineral Base Oils

Mineral oils are produced through the distillation and refining of crude oil and are classified according to the level of processing and chemical composition [13]. Mineral oils are widely used in industrial applications due to their low cost, good availability, and good performance in many lubrication regimes. Mineral oils also exhibit good oxidative stability and compatibility with existing additive packages, making them reliable for long term usage [12].

Despite these advantages, mineral oils have several limitations. Their viscosity and temperature behavior is often inferior to those of synthetic alternatives, resulting in reduced performance under extreme temperature conditions [13]. Additionally, mineral oils have relatively low biodegradability and can contribute to environmental pollution in cases of leakage or poor disposal. These drawbacks have motivated the search for more sustainable and higher-performance alternatives [11, 12].

2.2.2 Ester-Based Oils

Esters are a class of synthetic lubricants formed through the reaction between alcohols and carboxylic acids, such as in Fischer-Speier esterification, which is also the method used in this study. Bio-based esters can be synthesized from renewable raw materials, including fatty acids and alcohols derived from biomass, making them attractive from a sustainability perspective [11, 12].

Ester-based oils display several advantageous properties compared to mineral oils. Due to their polar structure, esters have a natural affinity for metal surfaces, which enhances lubricity and promotes the formation of stable boundary films. This can lead to reduced friction and wear, particularly under mixed or boundary lubrication conditions. Furthermore, ester-based lubricants typically exhibit higher viscosity indices (VI) than mineral oils, meaning that their viscosity changes less with temperature [14, 15]. A study by Borrás et al. (2018) reported that synthetic ester-based and poly- α -olefin-based lubricants showed a lower change in viscosity with temperature than a reference lubricant based on mineral oil, which was attributed to their higher viscosity indices [15, 16]. This characteristic is beneficial in maintaining a more stable lubricant film over a broad operating temperature range.

Furthermore, the physicochemical properties of ester-based oils are strongly influenced by the molecular structure of the alcohol and acid components. Variations in alkyl chain length, branching and degree of unsaturation can affect important lubricant properties such as viscosity, polarity, fluidity and intermolecular interactions. Studies on alkyl levulinate esters have shown that increasing alkyl chain length improves miscibility, storage stability and low-temperature flow behavior in hydrophobic fuel systems, while longer-chain levulinates demonstrated superior physicochemical performance compared to shorter-chain analogues [17]. Linear long-chain esters may generally promote stronger van der Waals interactions and improved film-forming ability due to more efficient molecular packing, whereas branched structures may enhance molecular mobility and improve low-temperature fluidity. Such structure-property relationships are highly relevant in lubricant systems, where viscosity stability and lubricant film behavior strongly influence friction and wear performance [17].

Another important advantage of ester-based oils is their high biodegradability and low toxicity, making them more environmentally friendly. This is particularly relevant in applications where loss of lubricant to the environment is unavoidable [14].

However, ester oils also have certain limitations. They can be more susceptible to hydrolysis in the presence of water, leading to degradation and the formation of unwanted acidic byproducts. Additionally, their oxidative stability may be lower than that of highly refined mineral oils unless appropriate additives are used [15]. Cost is another factor, as the production of synthetic or bio-based esters is generally more expensive than conventional mineral oil refining.

2.2.3 Comparison of Usage, Quality, and Viability

Mineral oils are currently the most widely used base oils in industrial lubrication due to their low cost, high availability, and well-established performance in many applications [7]. They are compatible with existing lubricant formulations and additive systems, which makes them practical and economically attractive for large-scale use. For this reason, mineral oils remain dominant in applications such as bearings, gears, hydraulic systems, compressors, and general industrial machinery [7].

However, the quality and performance of mineral oils are limited by their petroleum origin and molecular composition [12]. Although refined mineral oils can provide good lubrication under normal operating conditions, they often show lower viscosity-temperature stability compared with synthetic ester-based oils. This means that their viscosity may change more significantly with temperature, which can reduce lubricant film stability at high temperatures or cause poor flow behaviour at low temperatures [8, 12]. In addition, mineral oils generally have lower biodegradability and may cause environmental problems if leakage, spillage, or improper disposal occurs [8].

Ester-based oils, especially those produced from renewable alcohols and carboxylic acids, offer several advantages from both a performance and sustainability perspective. Their polar ester groups improve interaction with metal surfaces, which can enhance lubricity and support the formation of protective lubricant films [8, 11, 12]. They also often show better viscosity-temperature behaviour and higher biodegradability than mineral oils. These properties make bio-based esters promising candidates for applications where environmental impact is important, such as agriculture, forestry and other areas where lubricant loss to the environment may occur [8, 11].

Despite these advantages, ester-based oils also face some challenges. They are generally more expensive to produce than mineral oils, and their long-term stability can be affected by hydrolysis and oxidation [11]. Therefore, additives or further chemical modification may be required to improve their durability in demanding industrial applications. Their performance also depends strongly on molecular structure, including alkyl chain length, branching, unsaturation, and degree of esterification [12].

In general, mineral oils remain the most viable option for many conventional industrial applications due to their low cost, availability, and proven reliability. However, bio-based ester oils present a promising alternative where improved biodegradability, renewable origin, and strong lubricating properties are desired. With further optimization of stability and formulation, ester-based oils could become increasingly viable replacements for mineral oils in selected industrial lubrication applications.

2.3 Esterification and selection of alcohols and acids

Fischer–Speier esterification is an acid-catalyzed reaction between a carboxylic acid and an alcohol to form an ester and water. It has been widely used for more than a century and is still regarded as a benchmark method for ester synthesis because of its simplicity and compatibility with many different acid-alcohol combinations [18]. Recent reviews also highlight that the reaction remains relevant due to continued development of both homogeneous and heterogeneous catalytic systems, including alternatives to traditional sulfuric acid catalysis [18].

Fischer–Speier esterification is especially relevant because the target lubricant candidates are ester-based oils synthesized from renewable alcohols and bio-based carboxylic acids. The reaction provides a direct route to levulinate, lactate, and itaconate esters, where the molecular structure of the selected acid and alcohol influences properties such as polarity, viscosity, fluidity, and potential lubricant film formation.

2.3.1 Efficiency of primary alcohols in esterification

Primary alcohols are generally considered highly effective substrates in esterification reactions due to their relatively low steric hindrance and favorable nucleophilicity. Compared to secondary and tertiary alcohols, primary alcohols can more readily attack activated carboxylic acid intermediates, often resulting in higher conversion and ester yields. Wang *et al.* demonstrated that a wide range of carboxylic acids could be efficiently esterified with primary alcohols under mild conditions, while more sterically hindered alcohols such as isopropanol and tert-butanol showed little or no ester formation under the same reaction conditions [19].

The authors further reported that esterification reactions involving primary alcohols consistently produced high yields, often exceeding 90%, including esterifications with allyl alcohols and various linear alcohol systems. This enhanced reactivity was attributed to the favorable nucleophilicity of primary alcohols and their lower steric hindrance compared to more substituted alcohols [19].

2.3.2 Bio-based platform acids for lubricant applications

Over the past decades, lactic acid, levulinic acid and itaconic acid have gained increasing attention as important bio-based building blocks, owing to their availability from renewable resources and their versatility in chemical synthesis [20] [21] [22]. Among these, lactic acid has achieved the greatest level of industrial maturity, particularly as a precursor in the production of polylactic acid (PLA) [20].

In contrast, levulinic acid and itaconic acid have emerged as strong candidates for future bio-based applications and have been widely recognized as promising platform chemicals due to their versatility and potential for the synthesis of a wide range of

renewable materials and functional derivatives.[21] [22]

The following subsections will discuss the structural features, physicochemical properties and potential relevance of these platform acids in the development of bio-based ester lubricants.

2.3.2.1 Lactic acid

Lactic acid is considered one of the most important biomass-derived platform chemicals due to its versatility and potential role in future biorefineries. It can be produced from renewable feedstocks such as sugars and polysaccharides and serves as a precursor for a broad range of value-added chemicals, green solvents and biodegradable materials [23] [20]. In particular, lactic acid has gained significant industrial importance through its use in the production of polylactic acid (PLA), a biodegradable polyester with the potential to replace fossil-derived plastics in selected applications. Owing to its renewable origin, broad chemical functionality and growing industrial demand, lactic acid has attracted considerable interest as a sustainable building block for the synthesis of bio-based chemicals[20].

Structurally, lactic acid (2-hydroxypropanoic acid) is an α -hydroxy carboxylic acid containing both a carboxylic acid group and a hydroxyl group within a three-carbon molecule [23]. This bifunctional structure gives lactic acid high chemical versatility and enables a wide range of reactions, including esterification and condensation processes. Due to its tendency to undergo intermolecular esterification, lactic acid can form oligomeric species in concentrated solutions, consisting of short lactic acid chains linked through ester bonds, reflecting its strong reactivity toward ester formation[23] [24]. In addition, hydroxyl functionality contributes to increased polarity and hydrogen-bonding capability in lactic acid-derived esters. Such characteristics are of particular interest in lubricant applications, especially under boundary lubrication conditions where the lubricant film is extremely thin and lubricant–surface interactions become increasingly important. In these regimes, the polarity and adsorption behavior of the lubricant strongly influence friction and wear performance. Consequently, lactic acid-derived esters may represent promising candidates for the development of sustainable lubricant base oils [25].

2.3.2.2 Itaconic acid

Itaconic acid, also known as 2-methylenesuccinic acid, is a biomass-derived platform chemical recognized among the top renewable building blocks identified by the U.S. Department of Energy [26]. Industrial interest in itaconic acid has increased considerably due to its multifunctional structure and broad applicability in polymer and material synthesis. Today, IA is primarily produced through microbial fermentation of carbohydrate-rich feedstocks such as glucose and other sugars, providing a more sustainable alternative to petrochemical-based production routes [27, 26]. Its structure, comprising two carboxylic acid groups and a conjugated double bond, provides high chemical versatility and enables a wide range of transformations including esterification, polymerization and copolymerization reactions [27].

Consequently, itaconic acid is considered a valuable precursor for the synthesis of bio-based monomers, polymers and functional materials.

The functional nature of itaconic acid enables the formation of both monoesters and diesters with different alcohols, allowing variations in molecular structure and physicochemical behavior. The properties of itaconate-derived esters are strongly influenced by the alcohol component, where linear alkyl chains generally increase viscosity and intermolecular interactions through more efficient molecular packing, while branched or unsaturated chains tend to enhance molecular flexibility and fluidity. Such structure–property relationships are highly relevant in lubricant applications, since viscosity, film formation and low-temperature behavior are important factors affecting lubrication performance [27, 26] [25].

2.3.2.3 Levulinic acid

Levulinic acid is considered one of the most promising biomass-derived platform chemicals due to its wide convertibility into a broad range of value-added chemicals and industrial products [22]. It can be produced from renewable carbohydrate-based feedstocks such as glucose, fructose, sucrose and lignocellulosic biomass through acid-catalyzed conversion processes [28]. Owing to its renewable origin, carbon-neutral potential and versatile downstream chemistry, levulinic acid has attracted significant industrial and scientific interest as a sustainable alternative to fossil-derived chemical feedstocks. Numerous levulinic acid derivatives, including alkyl levulinates, γ -valerolactone and valerate esters, have demonstrated potential applications in green solvents, fuel additives, polymers and bio-based lubricants [28].

Levulinic acid is a multifunctional molecule containing both a ketone group and a carboxylic acid group, providing reactive sites for esterification and the formation of alkyl levulinates [28]. The physicochemical properties of levulinate esters are strongly influenced by the structure and chain length of the alcohol used during esterification. Studies have shown that increasing alkyl chain length improves the miscibility and stability of levulinate esters in hydrophobic fuel systems, while also enhancing fluidity, low-temperature behavior and storage stability [28]. Long-chain levulinate esters have additionally demonstrated improved physicochemical performance compared to shorter-chain analogues, owing to stronger hydrophobic interactions and more favorable intermolecular behavior. Such structure–property relationships are highly relevant for lubricant applications, where viscosity, film formation and fluid characteristics are strongly dependent on molecular structure [28, 22].

3

Methods and Materials

3.1 Synthesis Procedure

The method used to synthesis the lubricants is the Fischer-Speier esterification. React 1 gram of acid with 1:1 mole ratio of alcohol to acid, together with 43mg p-TSA as the catalyst, were heated to 90°C for 2 to 4 hours. For reaction with Itaconic acid, a 2:1 mole ratio of alcohol to acid was also used since itaconic acid has two carboxylic funtional groups that can perform esterification. The mixture was cooled down and samples are sent for NMR analysis and rheology analysis.

3.2 Materials

The materials and corresponding quantities used in the synthesis of the different lubricants are summarized in Tables 3.1, 3.2, and 3.3.

Alcohol	Levulinic Acid	Alcohol
1-Dodecanol	1g	2.049g
1-Undecaol	1g	1.895g
2-Ethyl-1-Hexanol	1g	1.432g
Oleyl alcohol	1g	2.313g
Cetyl alcohol	1g	2.6914g

Table 3.1: List of materials and quantities used in the synthesis of levulinate lubricants. In this synthesis, an excess of alcohol was employed relative to the molar amount of acid.

Alcohol	Lactic Acid	Alcohol
1-Dodecanol	1g	2.0686g
1-Undecanol	1g	1.9128
2-Ethyl-1-Hexanol	1g	1.4457g
Oleylcohol	1g	2.98g
Cetylalcohol	1g	2.0878g

Table 3.2: List of materials and quantities used in the synthesis of lactate lubricants. In this synthesis, a 1:1 molar ratio of acid and alcohol was used.

Alcohol	Itaconic Acid	Alcohol 1:1	Alcohol 1:2
1-Dodecanol	1g	1.432g	2.864g
1-Undecanol	1g	1.324g	2.648g
2-Ethyl-1-hexanol	1g	1g	2g
Oleylcohol	1g	2.063g	4.126g
Cetylalcohol	1g	1.8635g	3.7269g

Table 3.3: List of materials and quantities used in the synthesis of itaconate lubricants. In this synthesis, acid-to-alcohol molar ratios of 1:1 and 1:2 were employed.

3.3 NMR Analysis

Nuclear Magnetic Resonance (NMR) spectroscopy is used to determine the molecular structure, dynamics, and chemical environment of atoms within a compound. It is based on the principle that certain atomic nuclei, such as hydrogen-1 (1H) and carbon-13 (^{13}C), possess a property called spin [29]. When placed in a strong external magnetic field, these nuclei absorb electromagnetic radiation at characteristic resonance frequencies, which depend on their chemical surroundings [29]. Chemical shifts (δ , ppm) are referenced relative to a standard compound, typically tetramethylsilane (TMS), and were used to identify functional groups and verify successful synthesis [29].

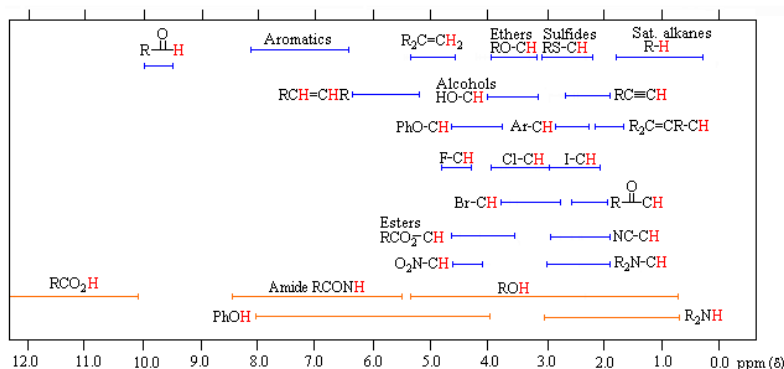


Figure 3.1: 1H NMR Chemical Shift for samples in $CDCl_3$ [30].

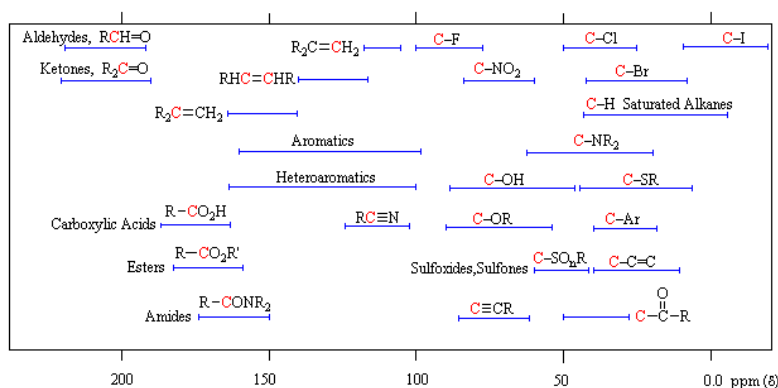


Figure 3.2: ^{13}C NMR Chemical Shift for samples in CDCl_3 [30].

In this study, NMR spectroscopy was employed to confirm the structure and purity of the synthesized compounds. Samples were dissolved in appropriate deuterated solvents, such as CDCl_3 , to avoid interference from hydrogen signals in the solvent [29]. The resulting spectra provided information on the number of chemically distinct environments, their relative positions, and interactions between neighboring nuclei through spin–spin coupling.

Overall, NMR serves as a key method for structural characterization, allowing for detailed insight into molecular composition and confirming the identity of the products obtained in this work.

3.4 FTIR Analysis

Fourier-transform infrared spectroscopy (FTIR) was used as a complementary characterization method for the synthesized ester-based lubricant samples. A small amount of selected samples which were deemed as promising was sent away for FTIR analysis. The obtained spectra were used to identify the main functional groups present in the samples and to support the structural characterization performed by NMR. The IR frequency ranges used to identify the main functional groups were determined using the IR Spectrum Table and Chart from Sigma-Aldrich [31].

3.5 Rheological Analysis

The rheological properties of the synthesized ester-based lubricants were analyzed using a rheometer. A rheometer measures the response of a fluid when it is subjected to controlled shear. During the measurement, a small amount of sample is placed between the measuring geometry and the rheometer plate. The instrument then applies either a controlled stress or strain while recording the resulting flow response of the sample [32, 33].

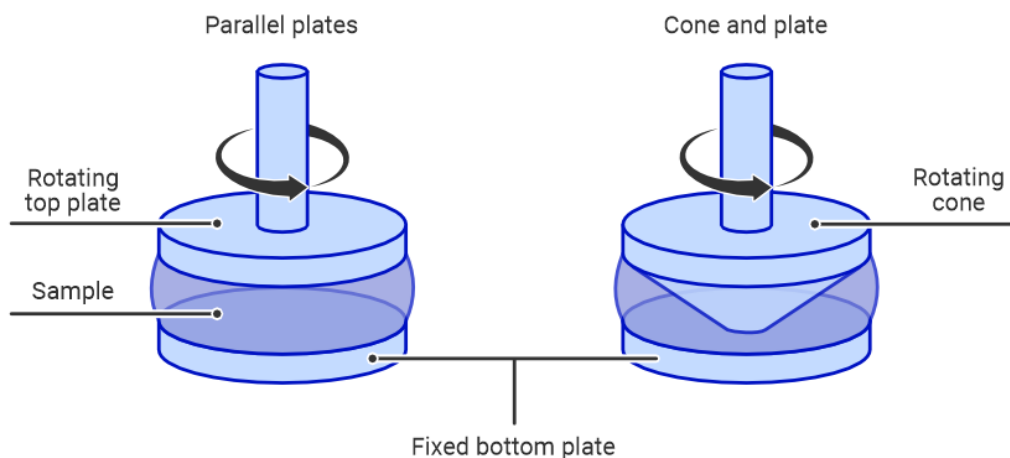


Figure 3.3: Parallel plates and cone and plate rotational rheometer setups [32].

The data obtained from the rheometer can be used to determine the dynamic viscosity of the synthesized lubricants as a function of the shear rate and shear stress. The dynamic viscosity, η , was calculated from the ratio between shear stress and shear rate according to Equation 3.1,

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (3.1)$$

where τ is the shear stress and $\dot{\gamma}$ is the shear rate. This makes it possible to evaluate whether the lubricants behave as Newtonian fluids, where viscosity remains relatively constant with changing shear rate, or whether they show non-Newtonian behavior. The kinematic viscosity, ν which is an important property for lubricant evaluation because it describes the flow behavior of the lubricant while accounting for its density is calculated according to Equation 3.2

$$\nu = \frac{\eta}{\rho} \quad (3.2)$$

where ν is the kinematic viscosity, η is the dynamic viscosity, and ρ is the density of the lubricant.

Temperature-dependent viscosity measurements were performed on three samples that were liquid at room temperature. These samples were measured at 40°C and 90°C to calculate their viscosity index. The remaining samples were not evaluated for viscosity index because many remained solid up to and beyond 40°C, making temperature-dependent viscosity measurements more difficult.

3.5.0.1 Viscosity index

Viscosity describes a fluid's resistance to flow and is one of the most important properties of lubricating oils, as it strongly influences film formation, friction and wear behavior. In general, liquid viscosity decreases with increasing temperature due to reduced intermolecular interactions and momentum transfer between fluid layers. Since lubricants operate under varying thermal conditions, it is important to characterize how viscosity changes with temperature. For this purpose, the viscosity index (VI) is commonly used as an empirical measure of the temperature dependence of lubricant viscosity. Oils with higher viscosity index values exhibit smaller viscosity changes with temperature and are therefore generally considered more suitable for applications requiring stable lubrication performance over a broad temperature range [34].

The viscosity index is traditionally determined from the kinematic viscosities measured at 40 °C and 100 °C according to ASTM D2270 and ISO 2909. For oils with higher viscosity index values, the viscosity remains more stable as temperature changes. In this work, ASTM D341 was additionally used to estimate the kinematic viscosity at 100 °C from experimentally measured viscosity values obtained at lower temperatures [35].

Oleyl lactate and oleyl levulinate were selected for viscosity index evaluation due to their pronounced oil-like consistency and promising NMR and rheological characteristics. Since both samples exhibited non-Newtonian and shear-thinning behavior, the viscosity index was evaluated at two defined shear rates: 7.95 s⁻¹ and 100 s⁻¹. The lower shear rate represents the transition region where the viscosity curves begin approaching a more stable flow regime, while the higher shear rate was considered more representative of practical lubrication and flow conditions as shown in figure

3. Methods and Materials

A.40. This approach enabled a more representative assessment of the temperature-dependent viscosity behavior of the synthesized ester systems under controlled shear conditions.

The viscosity index was calculated according to ASTM D2270 and ISO 2909 using the measured and estimated kinematic viscosities at different temperatures.

4

Results and Discussion

A total of twenty ester-based products were successfully synthesized through Fischer-Speier esterification using combinations of five renewable alcohols, 1-Dodecanol, 1-Undecanol, and 2-Ethyl-1-Hexanol, Oleyl alcohol and Cetyl alcohol, and three bio-based carboxylic acids, Levulinic acid, Lactic acid, and Itaconic acid. The synthesized products comprises of five levulinate esters, five lactate esters, and ten itaconate esters, of which includes both mono- and diester itaconate products depending on the alcohol to acid molar ratio employed. These products were selected to provide a broad range of molecular structures and chain lengths in order to evaluate how structural variations influence properties relevant to lubricant base oils. The cetyl alcohol ester samples were not analyzed by NMR because the products were physically very hard and did not dissolve sufficiently in the NMR solvent. For the same reason, viscosity measurements could not be performed on these samples. However, FTIR analysis was still possible and was therefore used to support the characterization of the Cetyl alcohol esters. The experimental synthesis and targeted compounds are described in **Chapter 3**, and representative NMR spectra are presented in **Appendix A**.

4.1 NMR Spectra Results

The NMR spectra indicate that the targeted ester products were formed in the synthesized samples. This is supported by the appearance of ester carbonyl resonances in the ^{13}C NMR spectra at approximately 170-176 ppm as well as typical O-CH₂ and O-CH proton signals in the ^1H NMR spectra at approximately 3.8-4.5 ppm (**see figures A.1 to A.24**). These signals are consistent with the formation of ester bonds and therefore confirm that esterification occurred during the reactions. For example, the itaconate products showed characteristic vinyl proton signals, while the levulinate products exhibited distinctive methylene signals. These peaks matched the expected structures and provided additional verification that the desired ester products had been successfully formed. The disappearance or substantial reduction of hydroxyl and carboxylic acid proton signals indicated that the starting materials had been largely consumed during the reaction.

However, while the spectra shows confirmation of ester formation, it does not pro-

vide proof of complete conversion or high product purity. In some samples, some residual starting materials may still be present, particularly unreacted alcohol. This is especially true for the levulinate reactions for 1-Undecanol, 1-Dodecanol and 2-Ethyl-1-Hexanol, where an excess of alcohol was used. Because the alkyl chain signals of the product and the alkyl chain signals of the starting alcohol overlap in the 1H NMR spectra, small amounts of residual alcohol are difficult to exclude based only on the spectra.

4.2 Rheology Results

The rheological results were used to evaluate the flow behaviour and viscosity characteristics of the synthesized ester-based lubricants. By measuring shear stress and dynamic viscosity as functions of shear rate, it was possible to assess whether the samples behaved approximately as newtonian fluids or showed shear-dependent behavior. For samples that showed newtonian behavior, the average dynamic viscosity values obtained from the rheological measurements are used. These were then combined with the measured densities of the samples to calculate their respective kinematic viscosities. For samples showing non-newtonian behaviour, the viscosity was treated as an apparent viscosity rather than a constant material property. The apparent dynamic viscosity was calculated at two chosen shear rates, those being 7.95s^{-1} and 100s^{-1} . Therefore, the viscosity values for these samples depend on the selected shear rate range. Density measurements were attempted in order to calculate the kinematic viscosities. However, due to several samples solidifying inside the pipettes, this made the density calculations difficult or impossible to determine accurately. Graphs representing Shear Rate/Shear Stress and Shear Rate/Viscosity are represented in **Appendix A**.

Among the synthesized products, oleyl lactate and oleyl levulinate were identified as the most promising candidates due to their clear NMR confirmation, oil-like appearance and measurable rheological behavior. The calculated viscosity index values indicated favorable temperature–viscosity characteristics for both ester systems. Oleyl lactate exhibited a viscosity index of 163.3 at 100 s^{-1} , which is comparable to commercially available synthetic lubricants such as Shell Helix Ultra 5W-30, reported to have a viscosity index of approximately 163 according to ASTM D2270. This suggests that oleyl lactate exhibits temperature-dependent viscosity behavior within the range typically associated with high-performance lubricant base oils [36] [35].

Oleyl levulinate exhibited an even higher apparent viscosity index of 206.3 at 100 s^{-1} , indicating relatively low viscosity sensitivity to temperature changes under higher shear conditions. Such values are within the upper range commonly associated with highly temperature-stable synthetic or bio-based lubricant systems. The results therefore suggest that long-chain levulinate esters may possess favorable rheological properties relevant for lubricant applications requiring stable film formation over varying temperatures.

In contrast, the apparent viscosity index calculated for oleyl levulinate at 7.95 s^{-1} reached 705.4, which is significantly above the range typically reported for conventional lubricating oils. Although ASTM D2270 is commonly used for multigrade and additive-containing lubricant systems, the method is fundamentally based on kinematic viscosity measurements at defined temperatures and does not explicitly account for pronounced shear-dependent rheological behavior. The unusually high apparent viscosity index is therefore likely influenced by the strong shear-thinning behavior observed for oleyl levulinate during rheological testing. Consequently, the value obtained at low shear conditions should be interpreted as a shear-dependent apparent viscosity index rather than a directly comparable conventional viscosity index. These findings further highlight the importance of evaluating bio-based ester systems under controlled rheological conditions, particularly when non-Newtonian flow behavior is present.

Product	Avg dynamic viscosity [mPa·s]	Density [g/cm ³]	Kinematic viscosity [mm ² /s]
1-Undecyl Lactate	14.401	0.885	16.272
1-Dodecyl Lactate	17.417	0.825	21.112
2-Eth-1-Hex Lactate	7.768	0.896	8.67
1-Undecyl Levulinate	7.58	0.900	8.422
1-Dodecyl Levulinate	9.123	-	-
2-Eth-1-Hex Levulinate	4.4016	0.905	4.864
1-Undecyl Itaconate 1:2 70°C	5.069	-	-
1-Undecyl Itaconate 1:2 90°C	4.226	-	-
2-Eth-1-Hex Itaconate 1:2 90°C	3.7163	0.960	3.871

Table 4.1: Dynamic and kinematic viscosity results for samples showing Newtonian behavior.

Product	Dynamic viscosity at 7.95 s^{-1} [mPa·s]	Dynamic viscosity at 100 s^{-1} [mPa·s]	Density [g/cm ³]	Kinematic viscosity at 7.95 s^{-1} [mm ² /s]	Kinematic viscosity at 100 s^{-1} [mm ² /s]
1-Dodecyl itaconate 1:1 90°C	23.227673	9.7215	-	-	-
1-Dodecyl itaconate 1:2 90°C	5.960252	4.735	0.847	7.037	5.59
2-Eth-1-Hex Itaconate 1:1 90°C	6340	142	0.828	7657.005	171.498
Oleyl Lactate 90°C	5.05434	3.6139	0.846	5.974	4.272
Oleyl Lactate 40°C	20.498113	11.374	0.846	24.229	13.444
Oleyl Levulinate 40°C	11.007421	9.3156	0.884	12.452	10.538
Oleyl Levulinate 90°C	7.112579	3.3748	0.884	8.046	3.818
Oleyl Itaconate 1:1 90°C	22.410063	9.9298	0.833	26.903	11.921
Oleyl Itaconate 1:2 40°C	152.377358	41.06	0.828	184.031	49.589
Oleyl Itaconate 1:2 90°C	10.424654	6.8801	0.828	12.59	8.309

Table 4.2: Dynamic and kinematic viscosity results for samples showing non-Newtonian behavior.

Product	Kinematic viscosity at 40°C [mm²/s]	Kinematic viscosity at 100°C [mm²/s]	Viscosity Index
Oleyl Lactate	24.229	4.903	128.7
Oleyl Levulinate	12.452	7.475	705.4

Table 4.3: Shear dependant viscosity index at 7.95s^{-1} calculated according to ASTM D2270 and ISO 2909 [37].

Product	Kinematic viscosity at 40°C [mm²/s]	Kinematic viscosity at 100°C [mm²/s]	Viscosity Index
Oleyl Lactate	13.444	3.618	163.3
Oleyl Levulinate	10.538	3.286	206.3

Table 4.4: Shear dependant viscosity index at 100s^{-1} calculated according to ASTM D2270 and ISO 2909 [37].

4.3 FTIR Results

Fourier-transform infrared spectroscopy (FTIR) analysis was carried out on selected ester samples that showed promising viscosity behaviour and good NMR results. These samples were chosen to further confirm successful ester formation and to support their suitability as potential lubricant base oils.

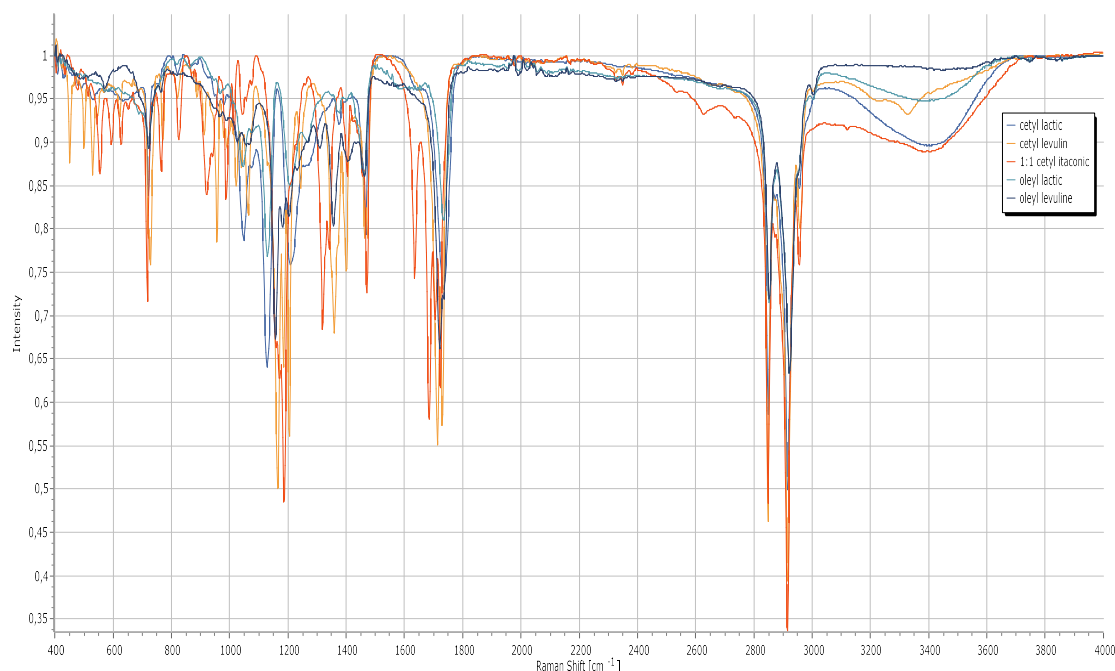


Figure 4.1: FTIR spectra of selected synthesised bio-based ester samples

The spectra showed a strong absorption band around $1730\text{-}1750\text{ cm}^{-1}$, corresponding to the ester carbonyl $\text{C}=\text{O}$ stretch, which indicates successful ester formation. Additional peaks around $1150\text{-}1250\text{ cm}^{-1}$ were assigned to $\text{C}-\text{O}$ stretching of the ester group, while peaks around $2850\text{-}2950\text{ cm}^{-1}$ correspond to aliphatic $\text{C}-\text{H}$ stretching from the hydrocarbon chains.

The weak or reduced broad $\text{O}-\text{H}$ absorption in the $3200\text{-}3600\text{ cm}^{-1}$ region suggests that most alcohol and acid starting materials were converted during esterification. Overall, the FTIR spectra support the successful synthesis of the selected ester products and confirm the presence of functional groups expected in bio-based lubricant base oils.

5

Conclusion

This thesis investigated the synthesis and preliminary evaluation of bio-based ester lubricants produced from renewable alcohols and carboxylic acids. The aim was to assess their potential as sustainable alternatives to conventional fossil-based mineral oils for industrial lubrication applications. A range of levulinate, lactate, and itaconate esters was synthesized using Fischer-Speier esterification and evaluated mainly through NMR, FTIR, and rheological analysis.

The results showed that several ester products were successfully formed. NMR analysis confirmed ester formation through characteristic ester carbonyl signals in the ^{13}C NMR spectra and O-CH/O-CH₂ signals in the ^1H NMR spectra. FTIR analysis further supported ester formation by showing strong carbonyl absorption bands around $1730\text{-}1750\text{ cm}^{-1}$ and C-O stretching bands around $1150\text{-}1250\text{ cm}^{-1}$. Together, these results indicate that renewable acids and alcohols can be used to synthesize ester compounds with potential relevance as lubricant base oils.

Rheological measurements showed that the synthesized esters had different flow behaviours depending on their molecular structure. Several lactate and levulinate esters showed approximately Newtonian behaviour and relatively low viscosities, indicating good fluidity. In contrast, some itaconate and Oleyl-based esters showed non-Newtonian behaviour, where viscosity decreased with increasing shear rate. This suggests that chain length, branching, unsaturation, and degree of esterification strongly influence viscosity and flow behaviour.

The most interesting samples were therefore the liquid lactate and levulinate esters, particularly those based on 1-Undecanol, 1-Dodecanol, 2-Ethyl-1-Hexanol, and Oleyl alcohol, as these showed confirmed ester formation and measurable viscosity values. Among these, oleyl lactate and oleyl levulinate were identified as especially promising due to their favorable rheological behavior and oil-like characteristics. Oleyl lactate exhibited viscosity index values comparable to commercial synthetic lubricants, while oleyl levulinate showed particularly high apparent viscosity stability under defined shear conditions. The rheological evaluation further demonstrated that shear-dependent flow behavior can strongly influence viscosity index calculations for bio-based ester systems and should therefore be carefully considered when applying conventional ASTM-based evaluation methods. Some oleyl and itaconate

5. Conclusion

esters may also be interesting for applications requiring higher viscosity or shear-thinning behaviour, although further testing is needed. The cetyl alcohol-derived esters could not be analyzed by NMR or viscosity measurements because the products were very hard and did not dissolve sufficiently in the NMR solvent, but FTIR analysis could still be performed.

This study shows that bio-based ester lubricants have potential as sustainable lubricant base oil candidates. However, further work is required before they can be considered practical replacements for mineral oils. Future studies should focus on the influence of additives, more in depth viscosity index measurements, oxidation and hydrolytic stability, and tribological testing under realistic operating conditions.

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Appendix 1

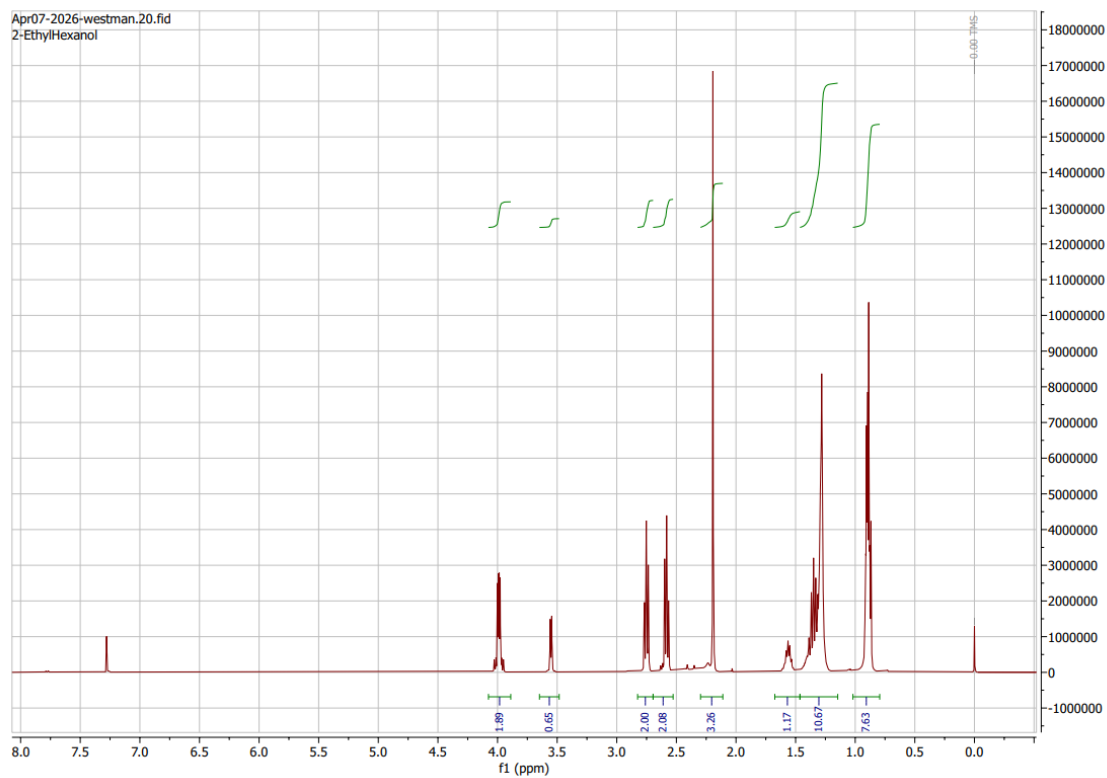


Figure A.1: 1H NMR From reaction to form 2-Ethyl-1-Hexyl Levulinate

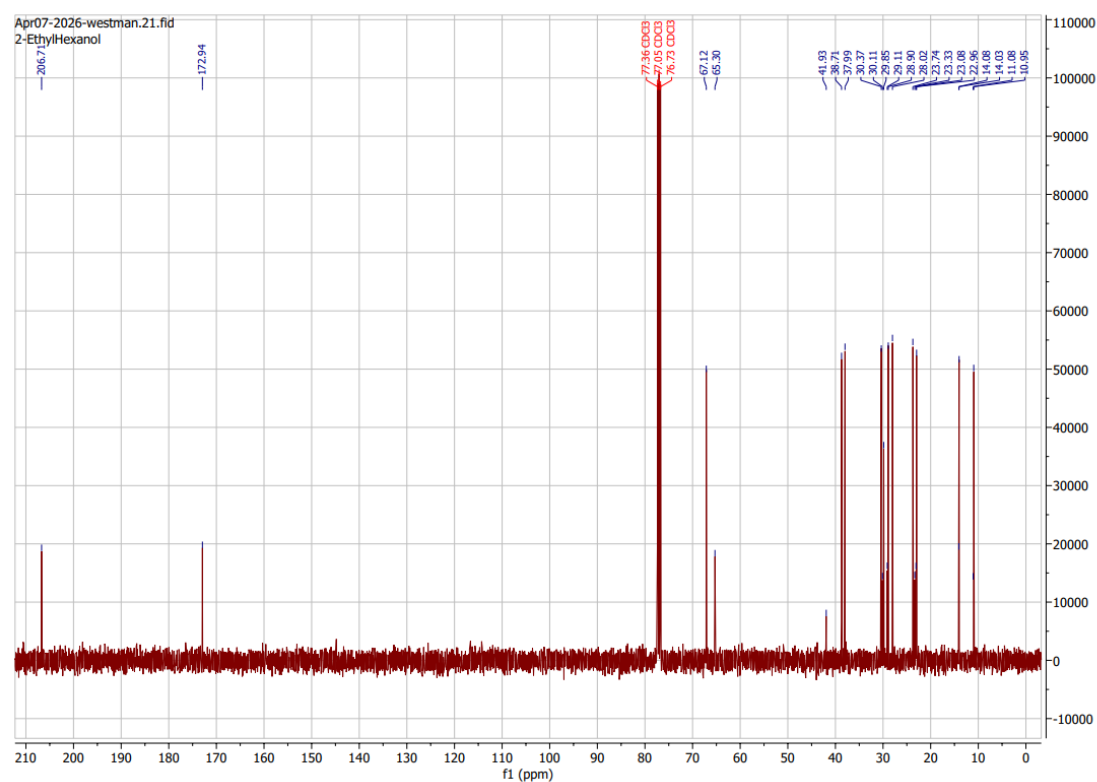


Figure A.2: ^{13}C NMR From reaction to form 2-Ethyl-1-Hexyl Levulinate

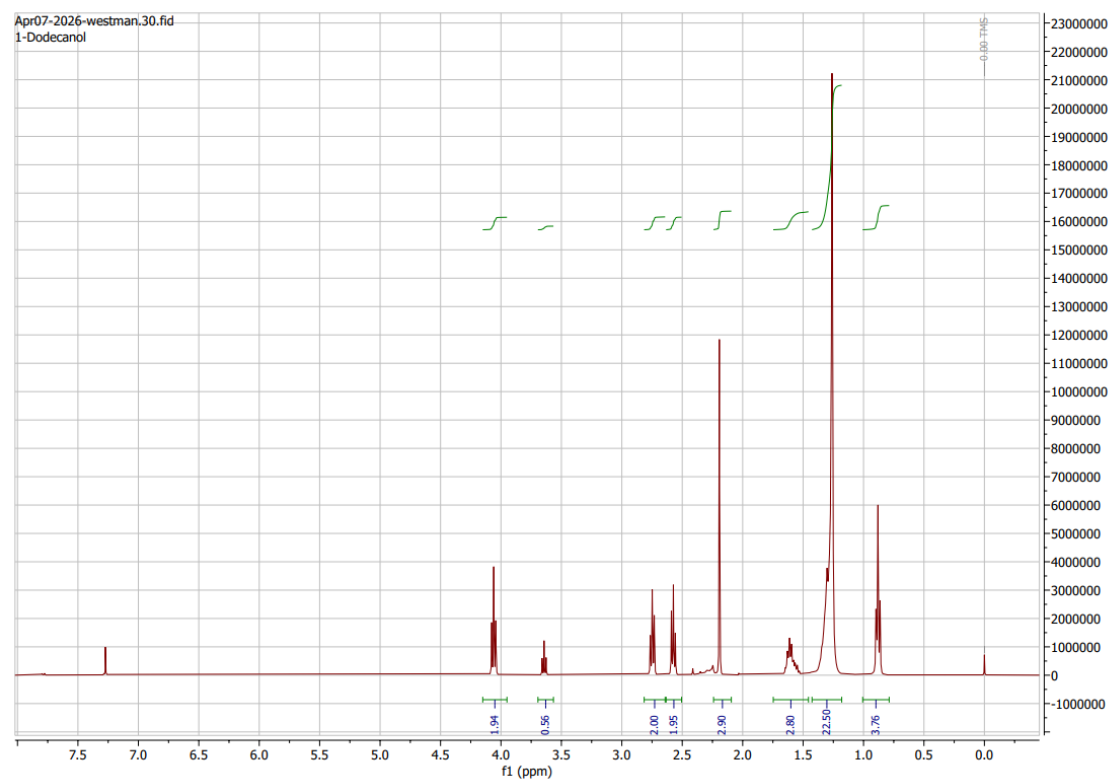


Figure A.3: ^1H NMR From reaction to form 1-Dodecyl Levulinate

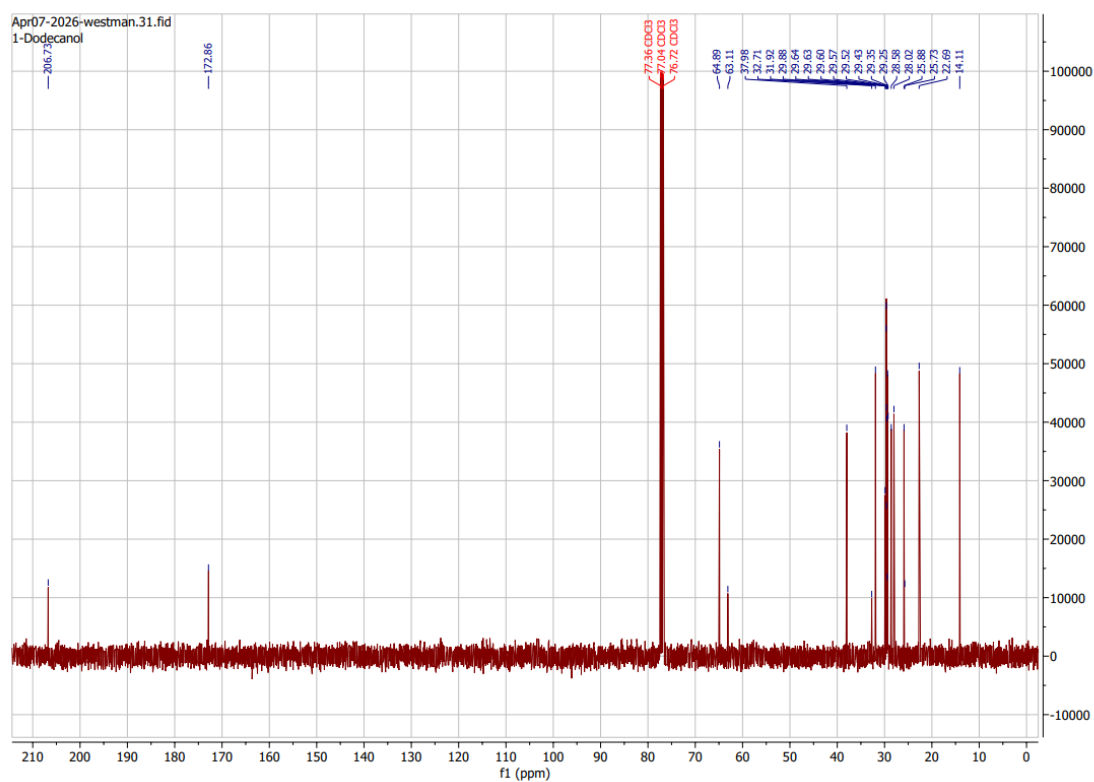


Figure A.4: ^{13}C NMR From reaction to form 1-Dodecyl Levulinate

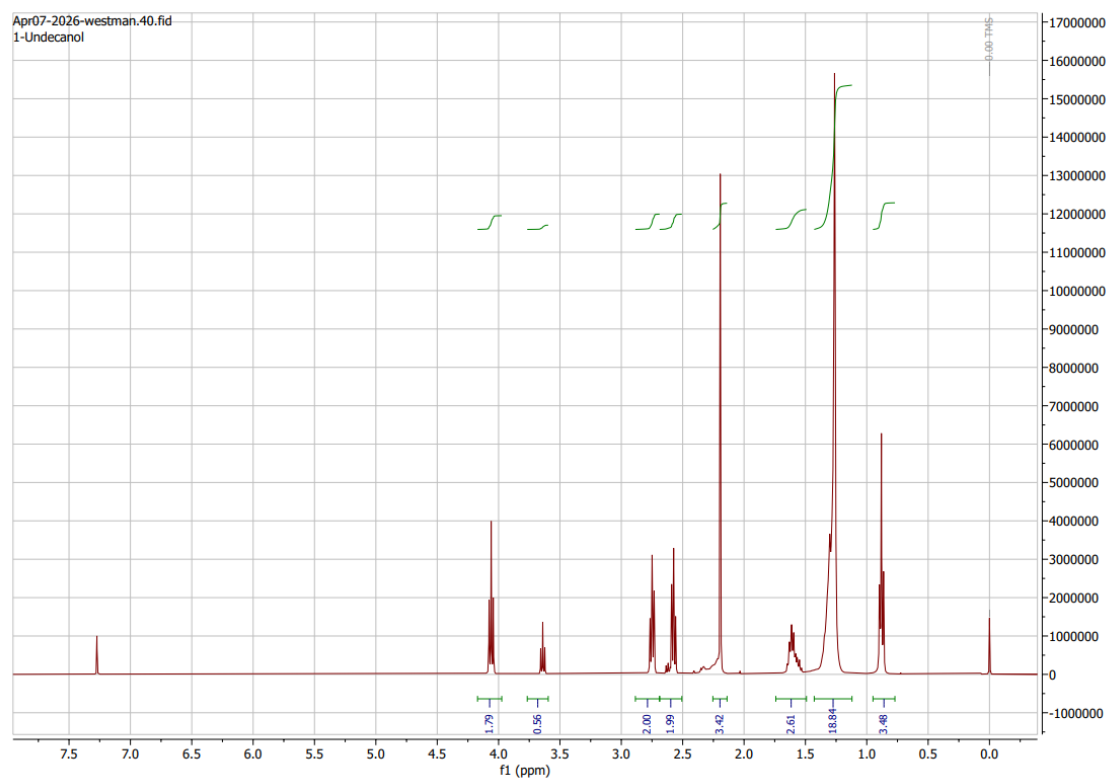


Figure A.5: ^1H NMR From reaction to form 1-Undecyl Levulinate

A. Appendix 1

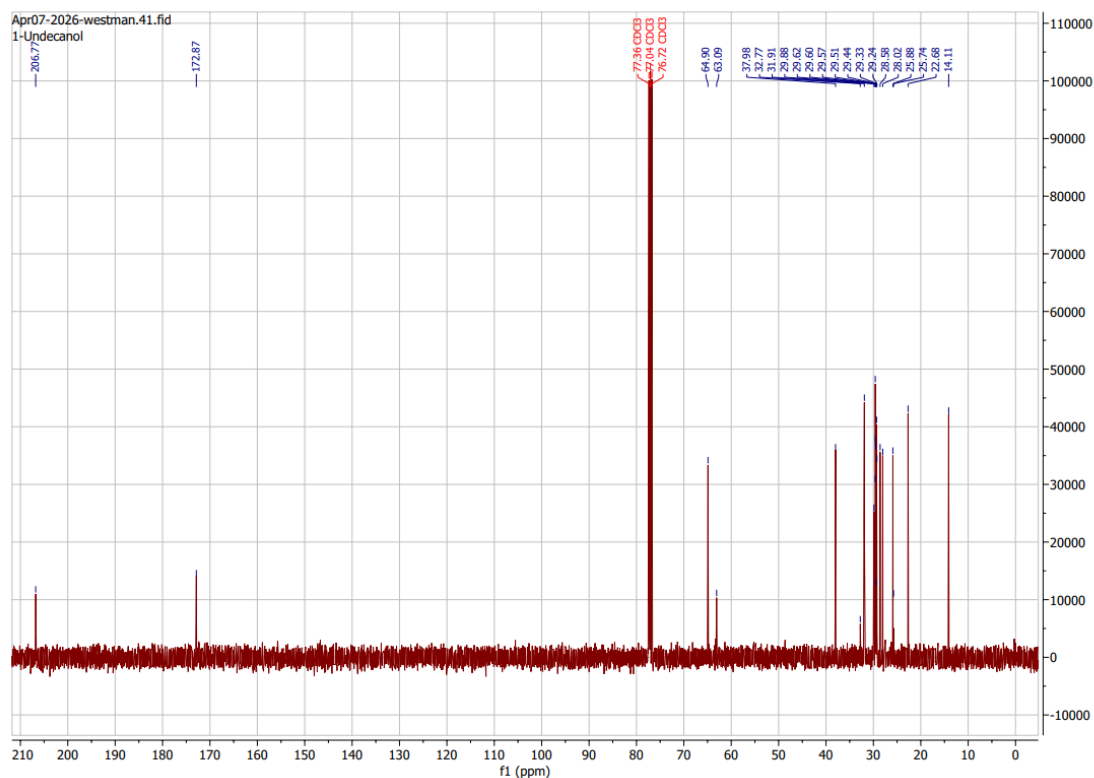


Figure A.6: ^{13}C NMR From reaction to form 1-Undecyl Levulinate

Lactic Acid - 2-ethyl-1-hexanol

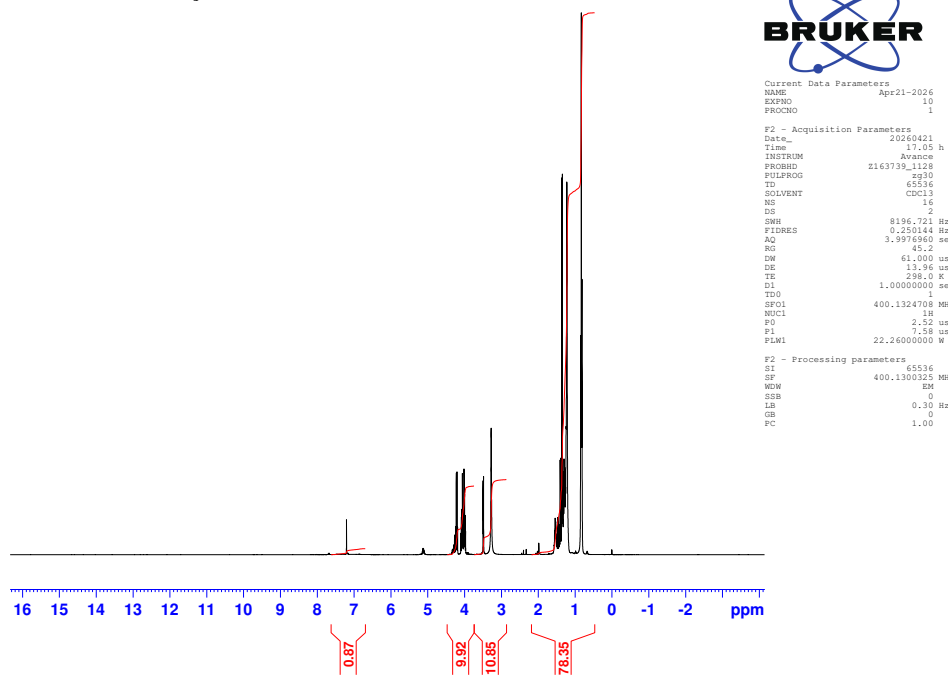
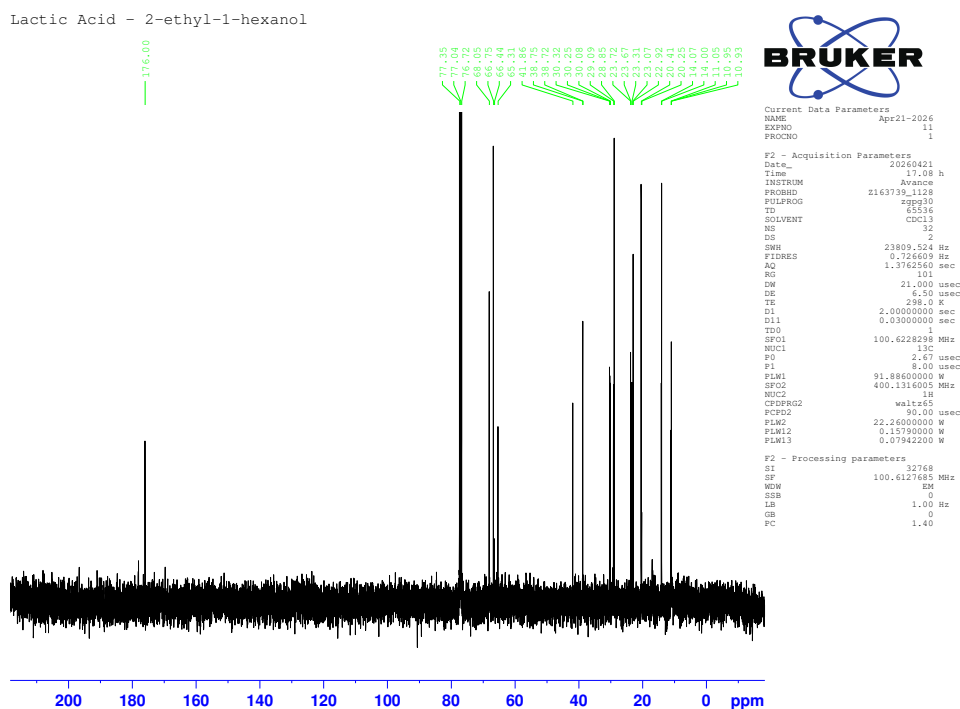
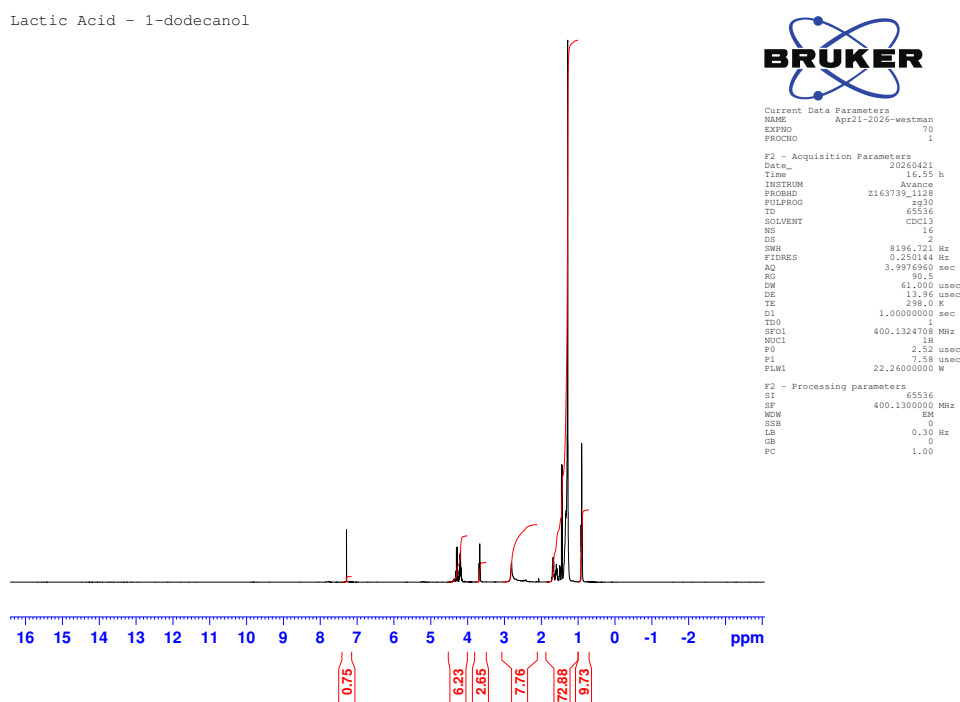


Figure A.7: ^1H NMR From reaction to form 2-Ethyl-1-Hexyl Lactate

Figure A.8: ^{13}C NMR From reaction to form 2-Ethyl-1-Hexyl LactateFigure A.9: ^1H NMR From reaction to form 1-Dodecyl Lactate

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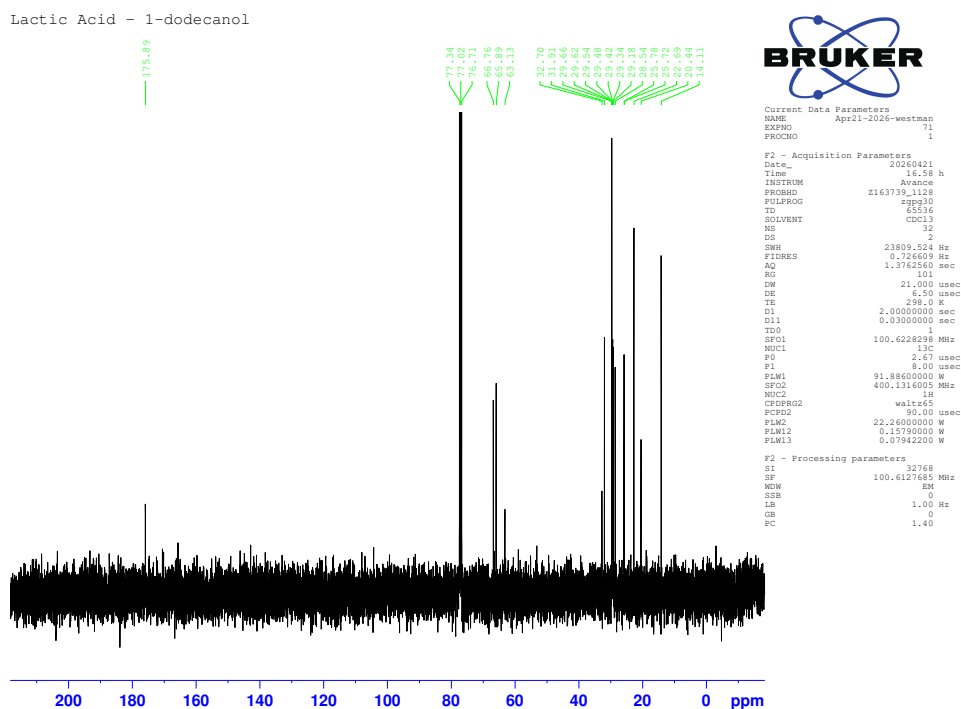


Figure A.10: ^{13}C NMR From reaction to form 1-Dodecyl Lactate

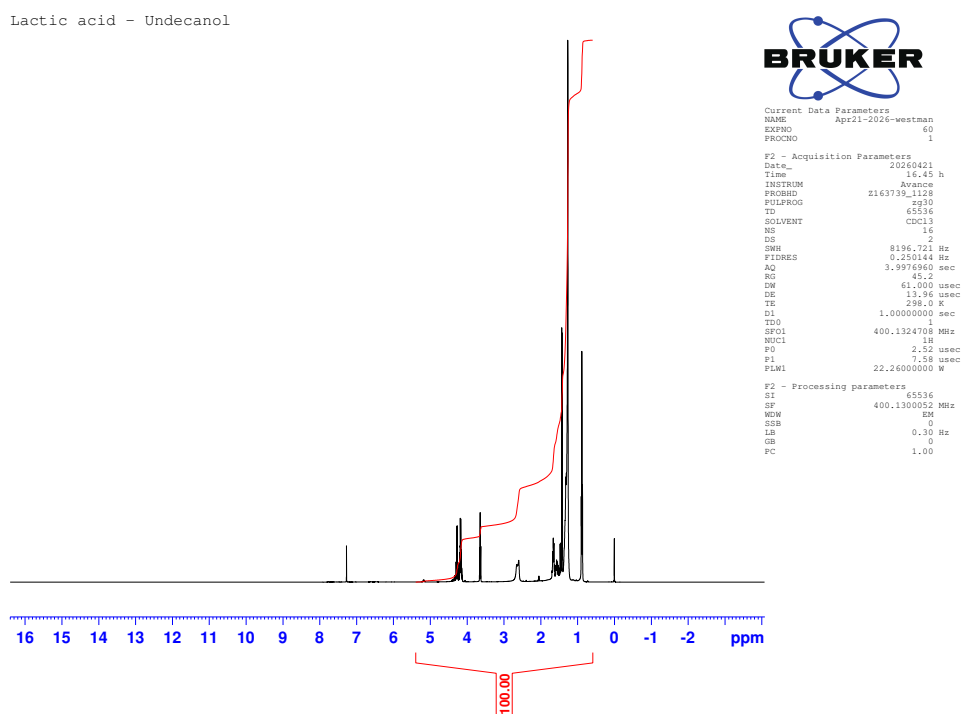
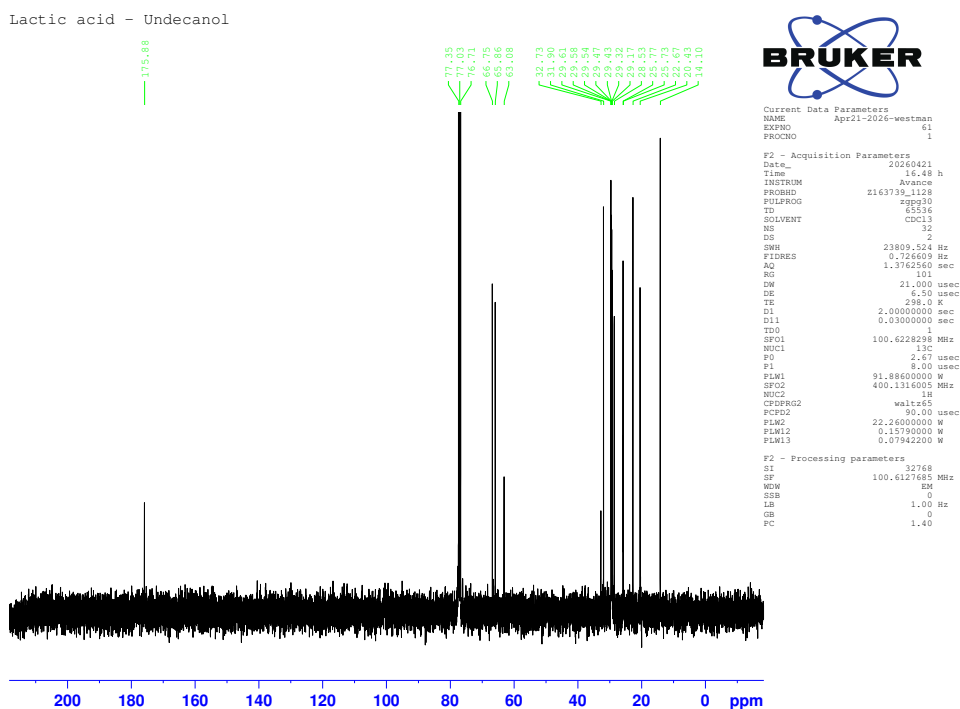
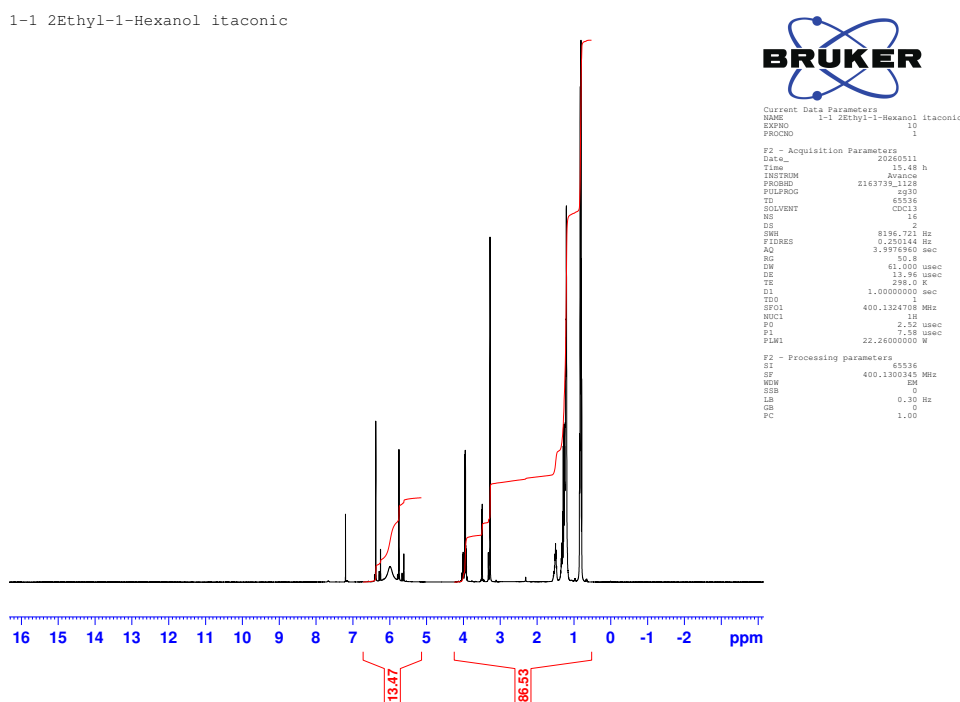


Figure A.11: ^1H NMR From reaction to form 1-Undecyl Lactate

Figure A.12: ^{13}C NMR From reaction to form 1-Undecyl LactateFigure A.13: ^1H NMR From reaction to form Mono(2-ethylhexyl) itaconate

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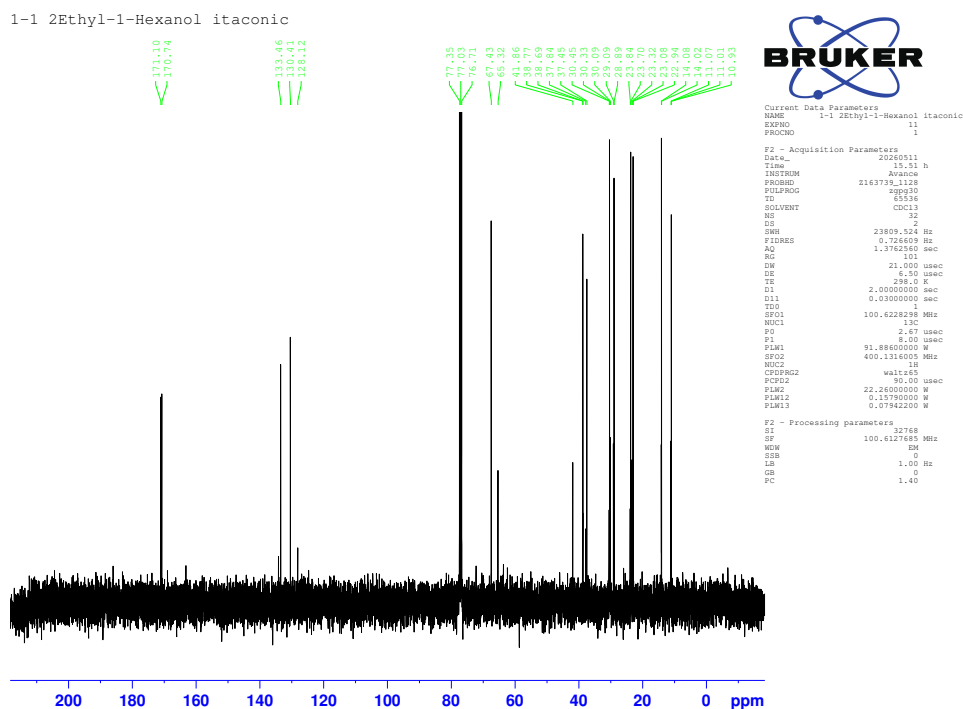


Figure A.14: ^{13}C NMR From reaction to form Mono(2-ethylhexyl) itaconate

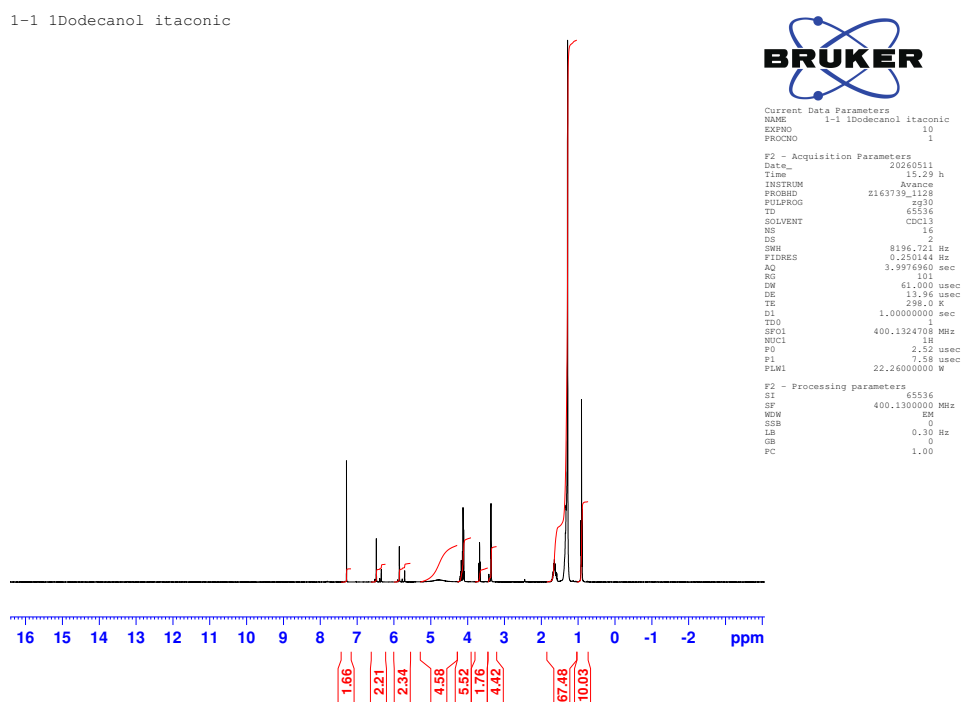
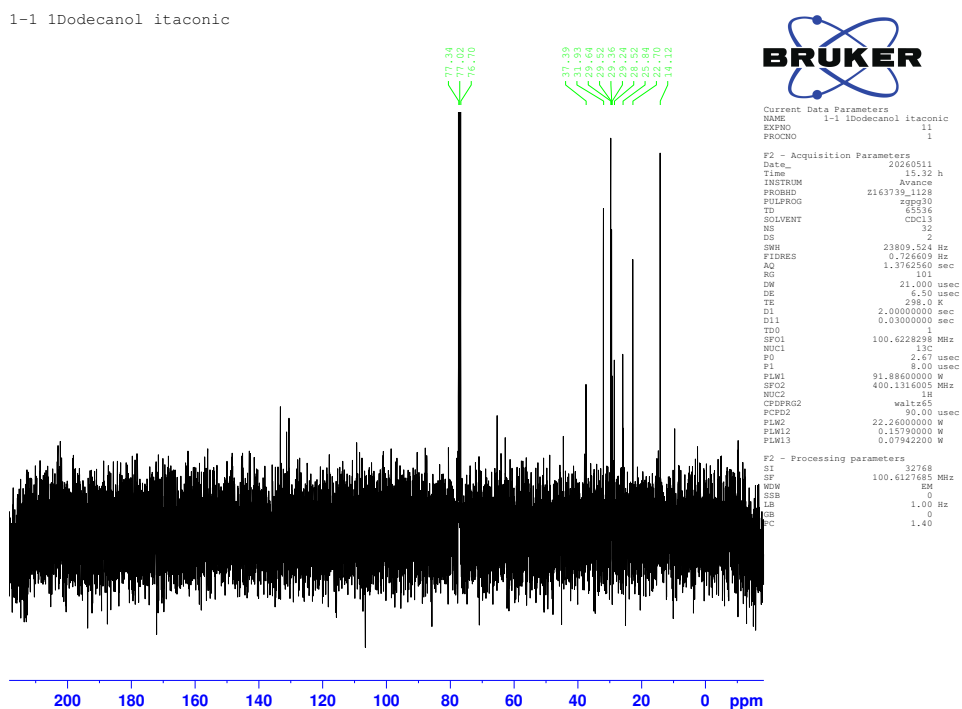
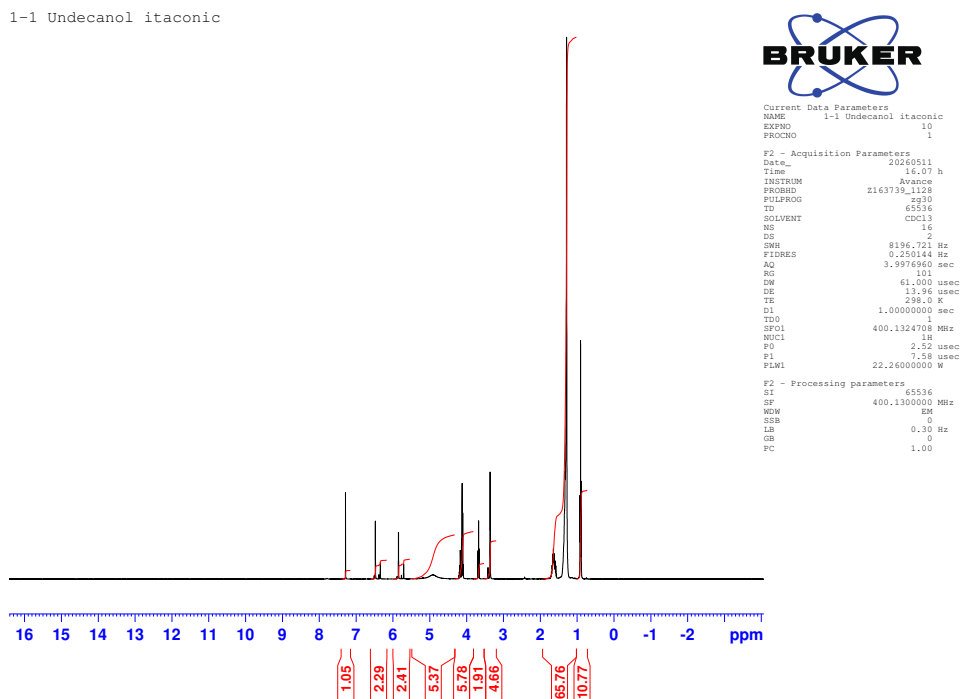


Figure A.15: ^1H NMR From reaction to form Mono(Dodecyl) itaconate

Figure A.16: ^{13}C NMR From reaction to form Mono(Dodecyl) itaconateFigure A.17: ^1H NMR From reaction to form Mono(Undecyl) itaconate

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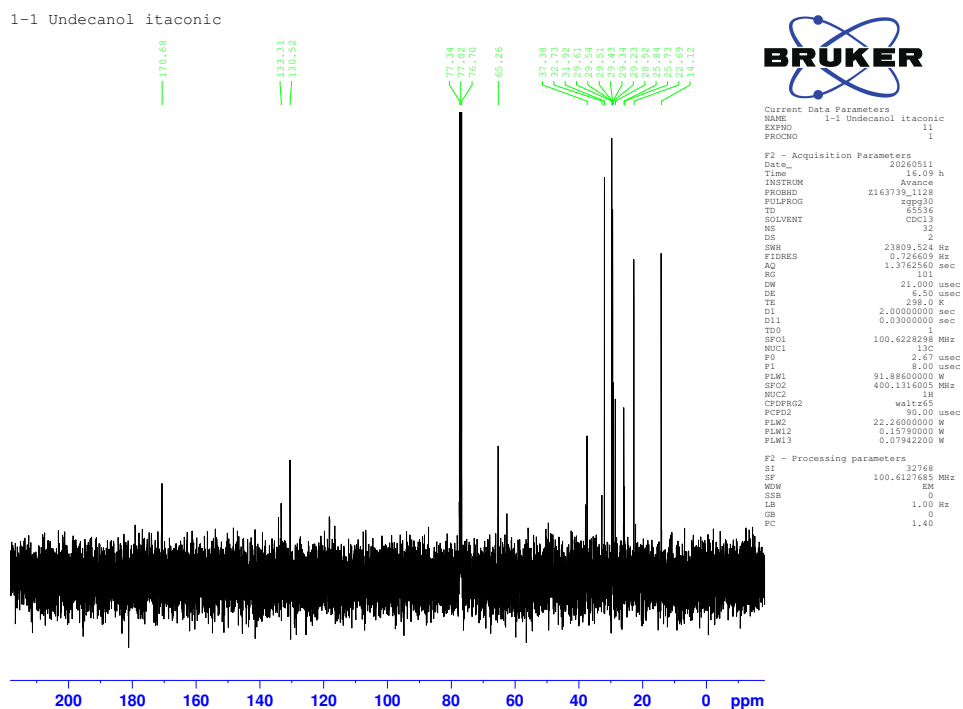


Figure A.18: ^{13}C NMR From reaction to form Mono(Undecyl) itaconate

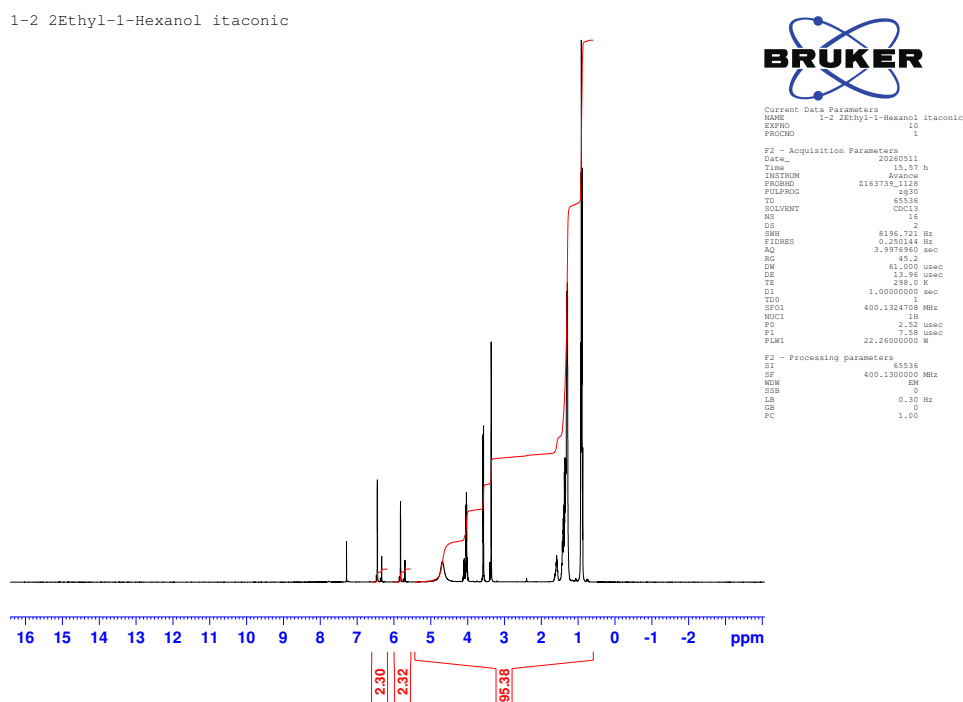


Figure A.19: ^1H NMR From reaction to form Di(2-ethylhexyl) itaconate

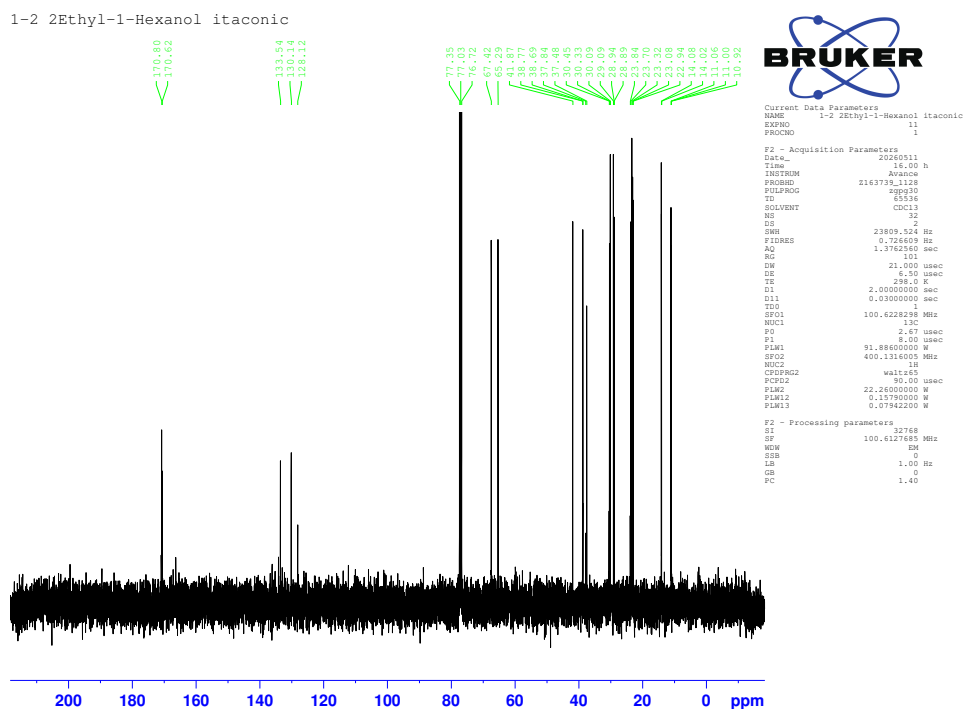


Figure A.20: ^{13}C NMR From reaction to form Di(2-ethylhexyl) itaconate

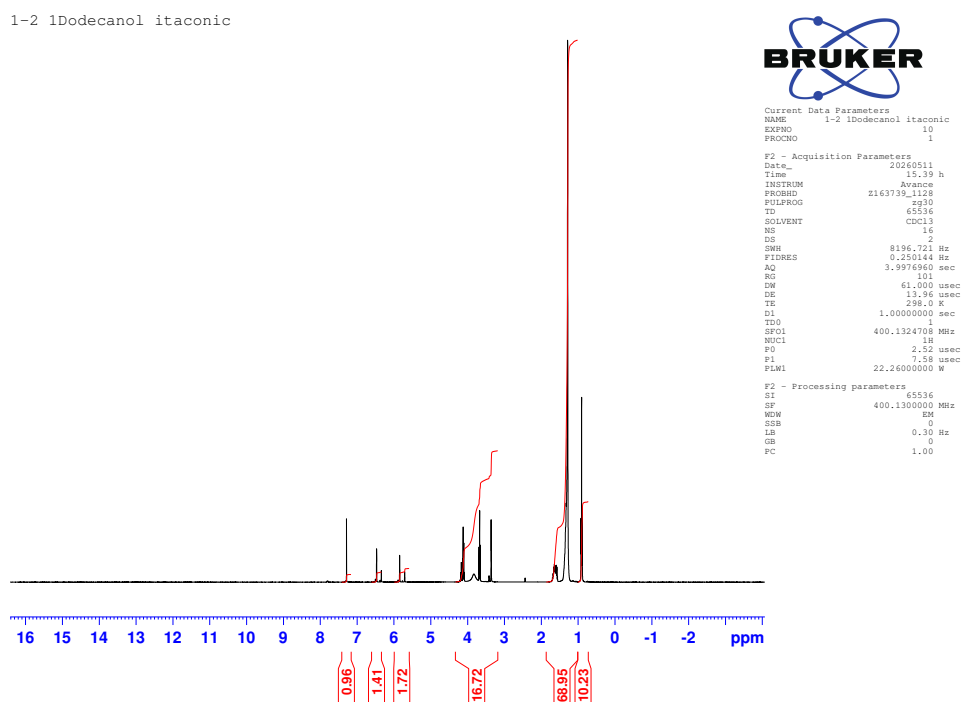


Figure A.21: ^1H NMR From reaction to form Di(Dodecyl) itaconate

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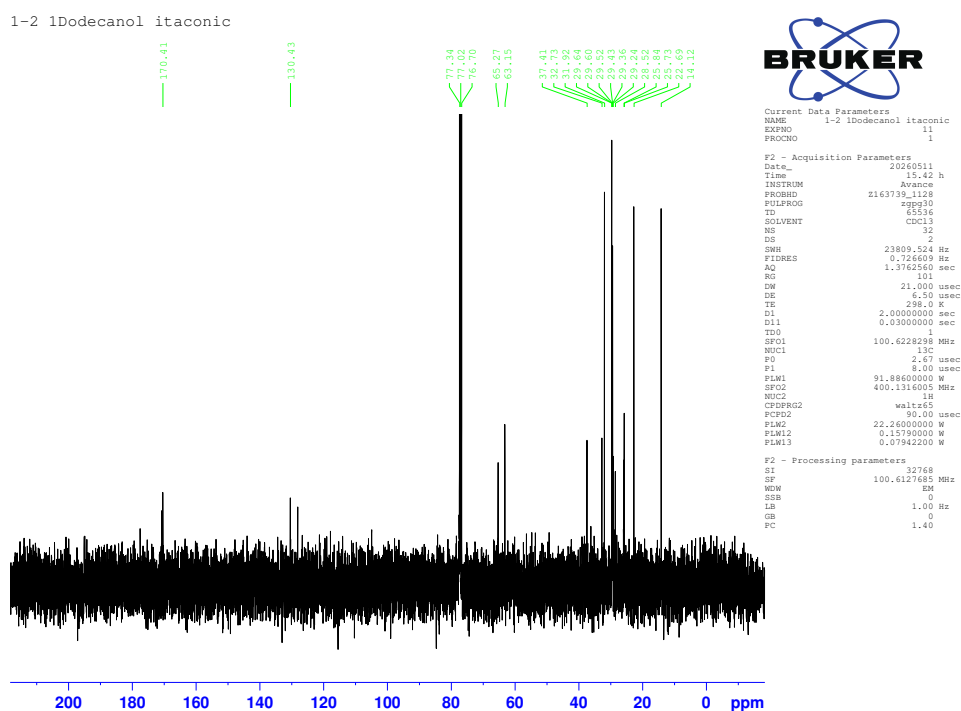


Figure A.22: ^{13}C NMR From reaction to form Di(Dodecyl) itaconate

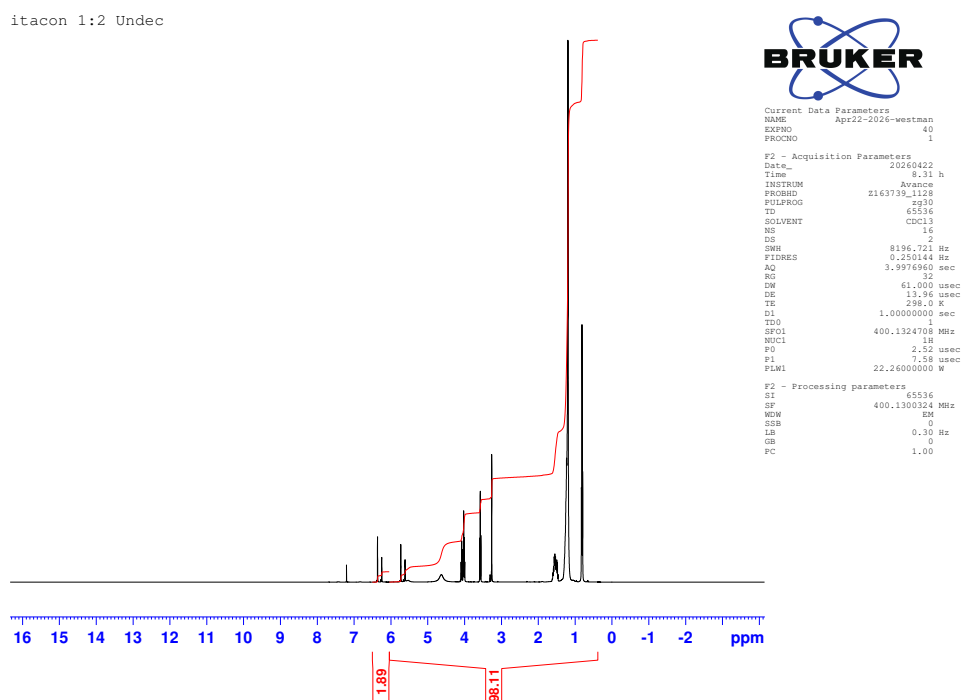
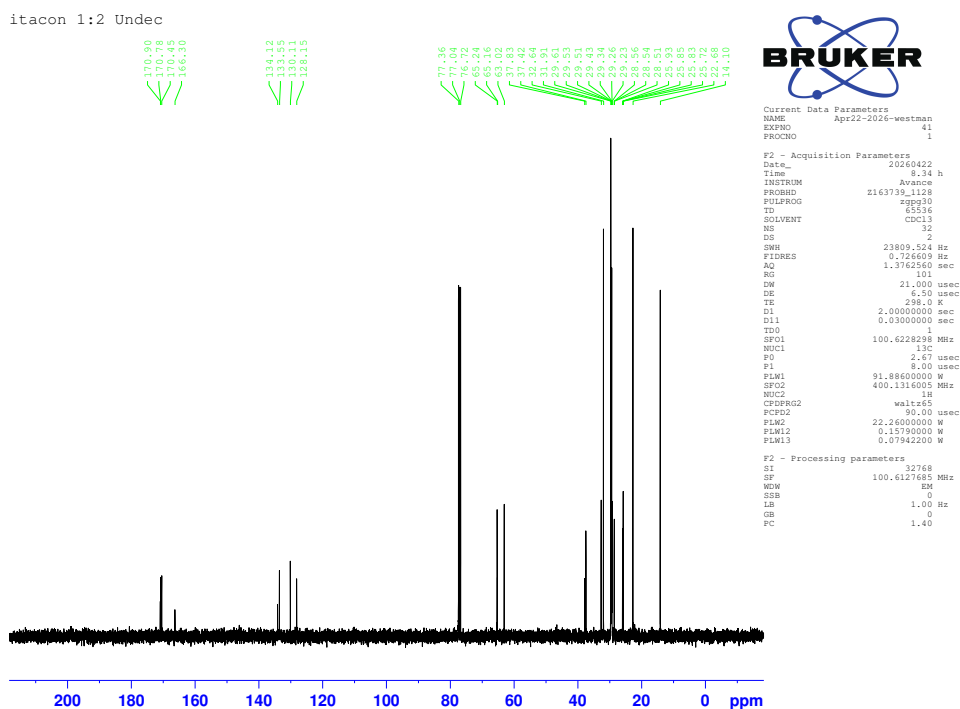
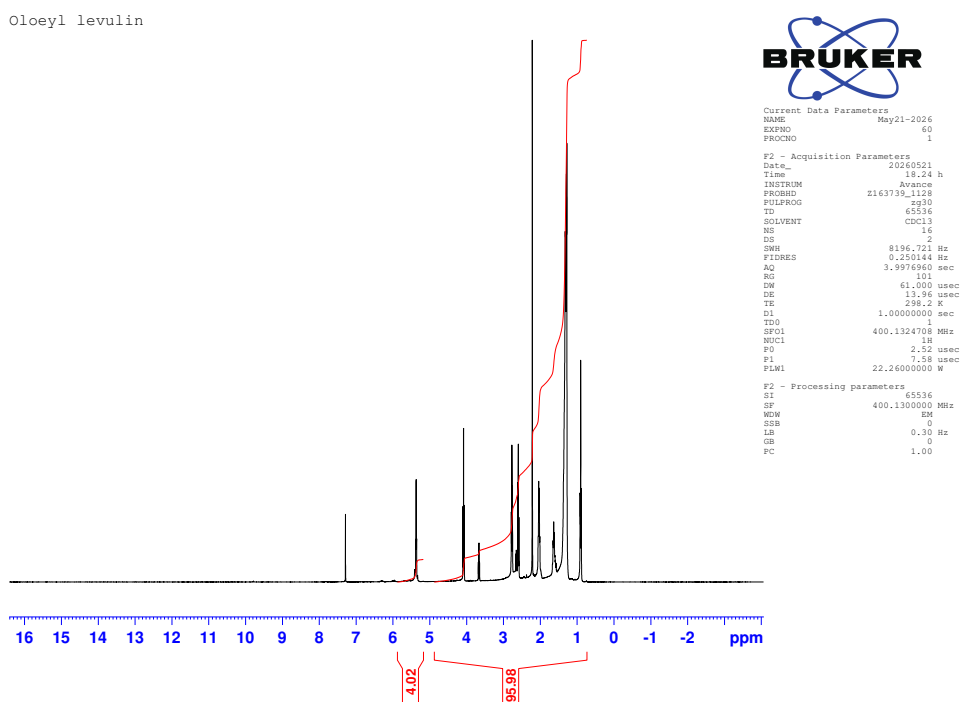


Figure A.23: ^1H NMR From reaction to form Di(Undecyl) itaconate

Figure A.24: ^{13}C NMR From reaction to form Di(Undecyl) itaconateFigure A.25: ^1H NMR From reaction to form Oleyl levulinate

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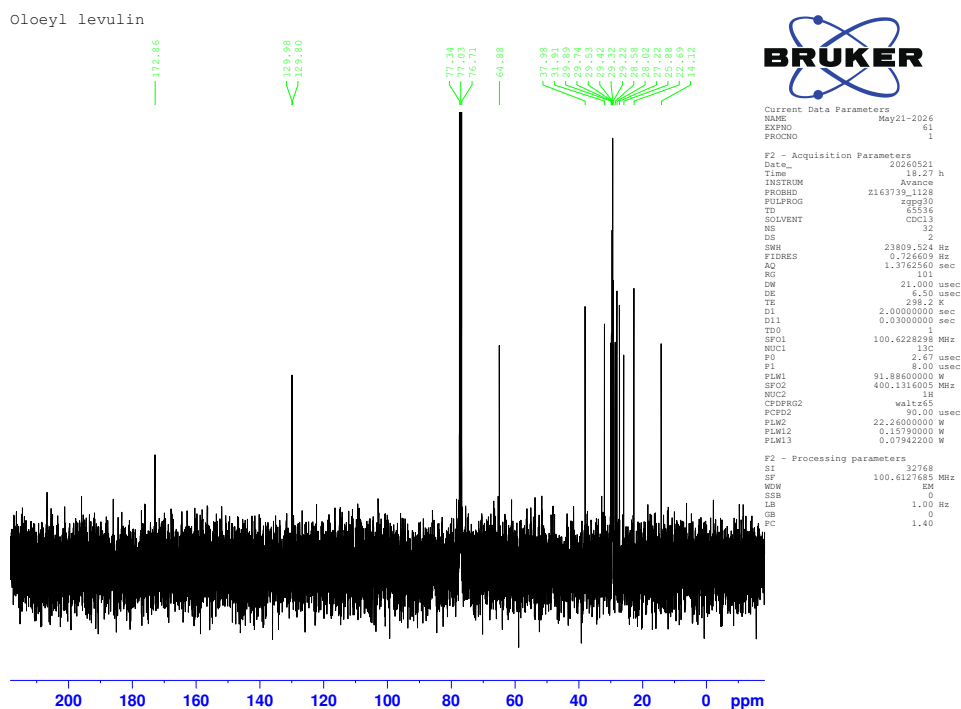


Figure A.26: ^{13}C NMR From reaction to form Oleoyl Levulinate

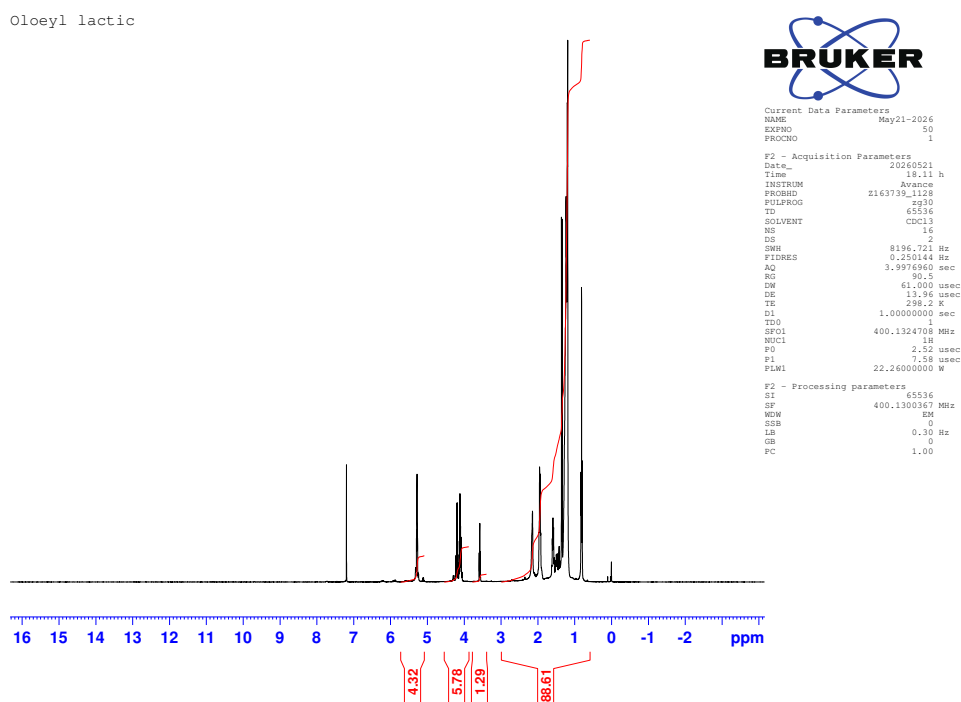
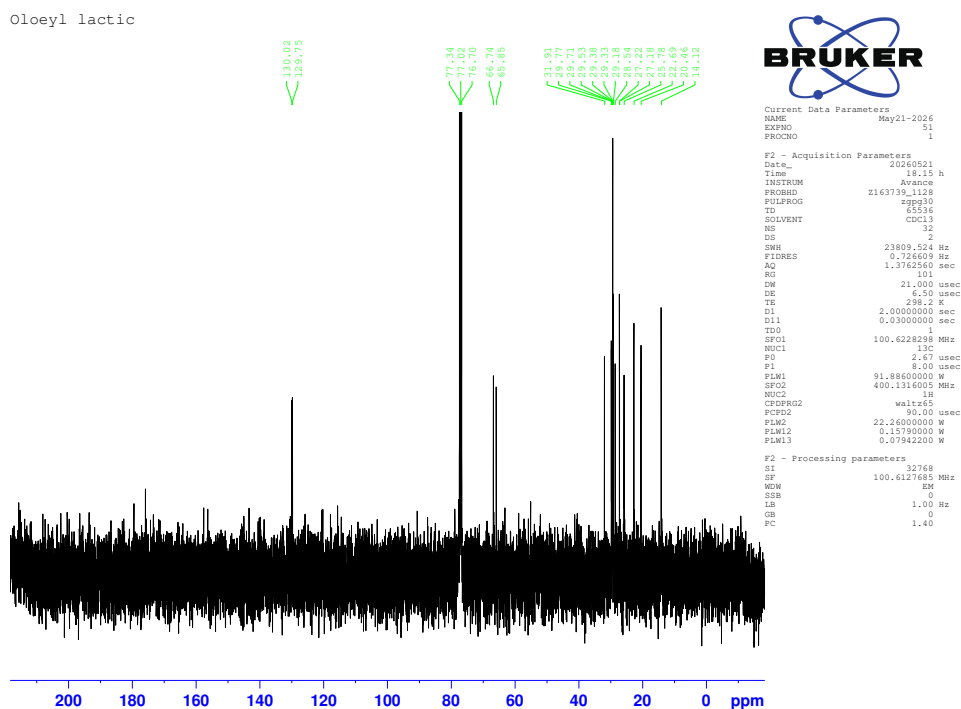
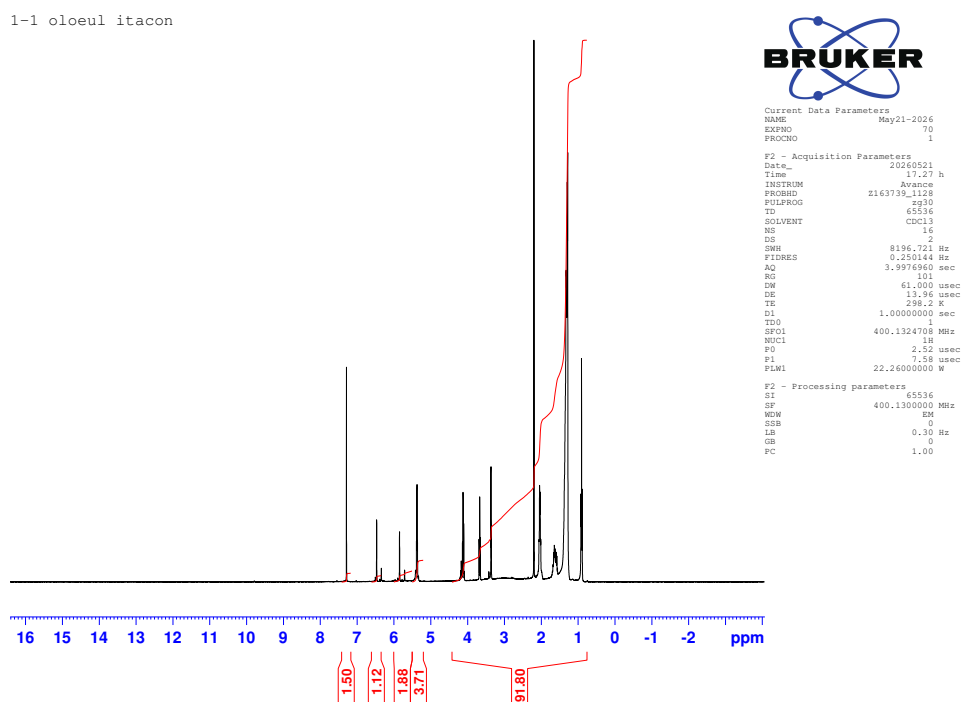


Figure A.27: ^1H NMR From reaction to form Oleoyl Lactate

Figure A.28: ^{13}C NMR From reaction to form Oleyl LactateFigure A.29: ^1H NMR From reaction to form Mono(Oleyl) Itaconate

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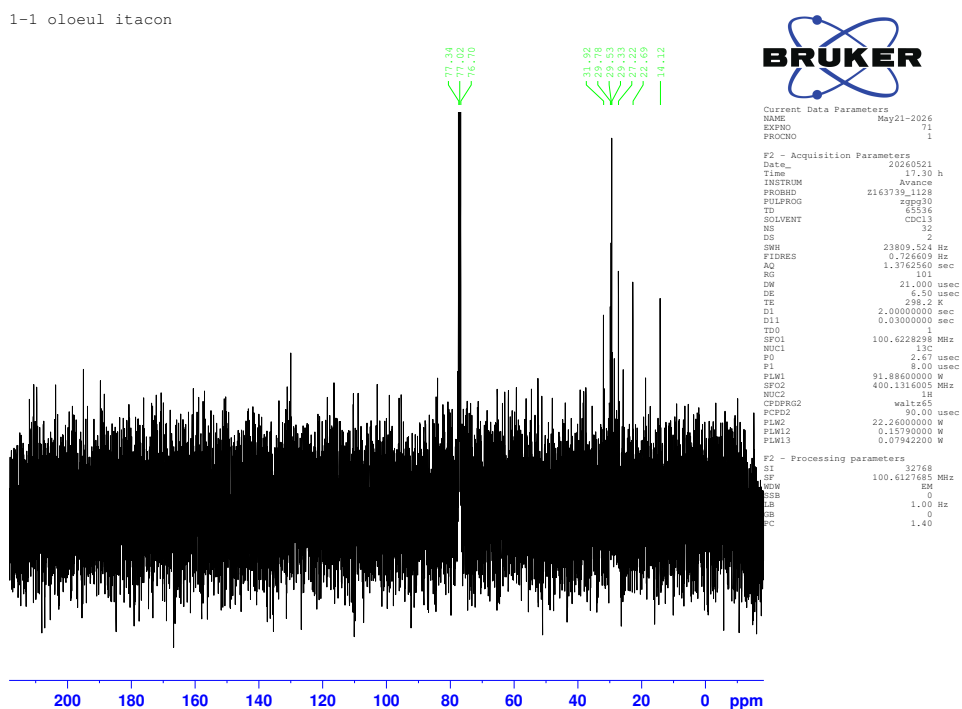


Figure A.30: ^{13}C NMR From reaction to form Mono(Oleyl) Itaconate

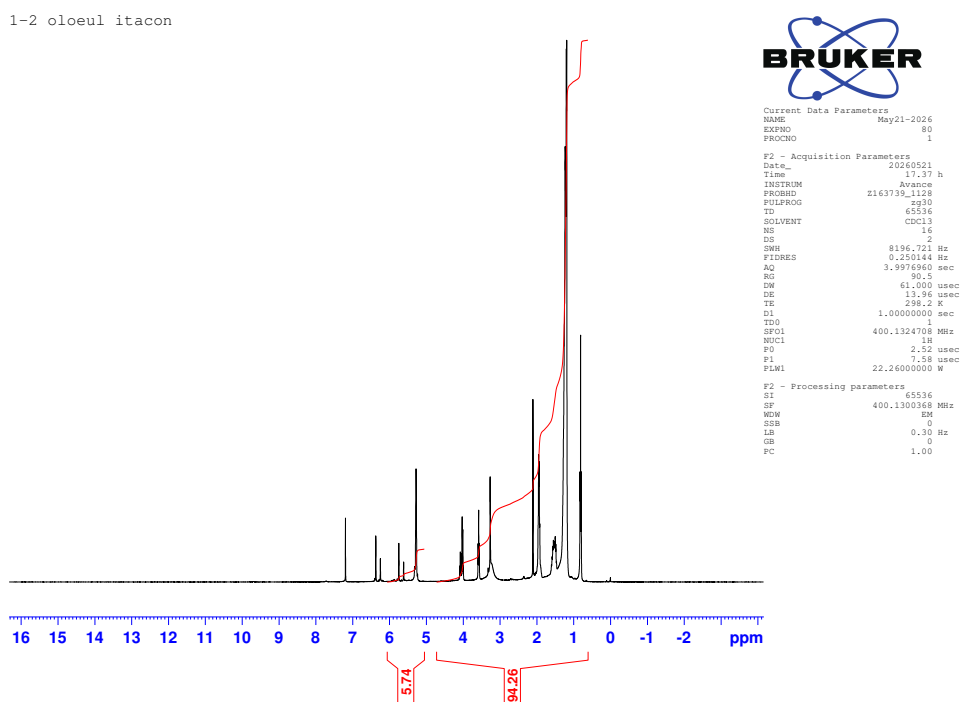


Figure A.31: ^1H NMR From reaction to form Di(Oleyl) Itaconate

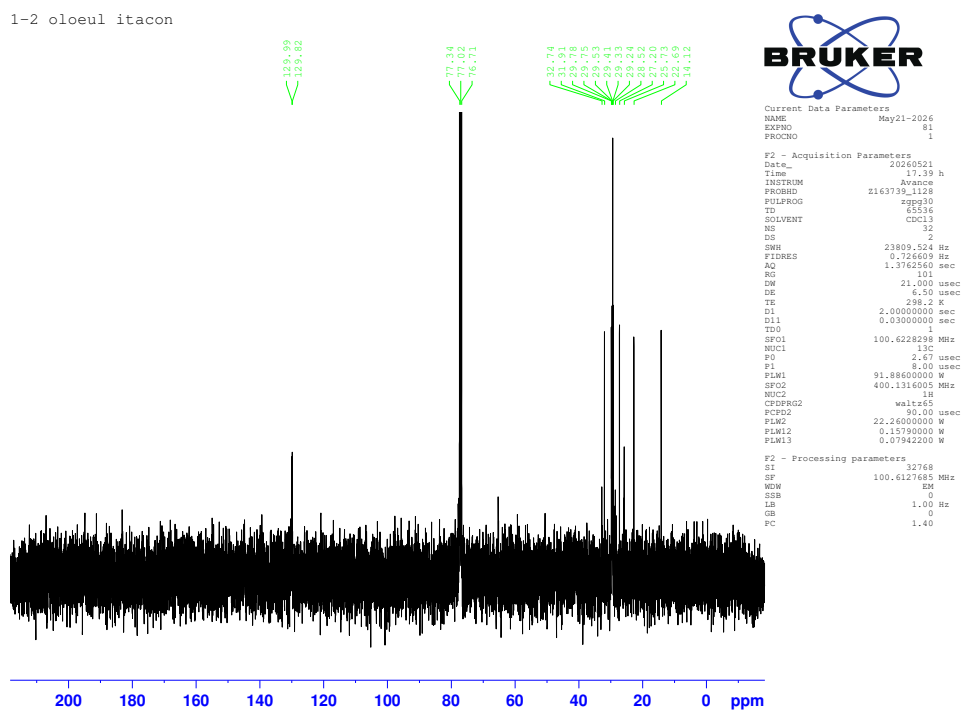


Figure A.32: ^{13}C NMR From reaction to form Di(Oleyl) Itaconate

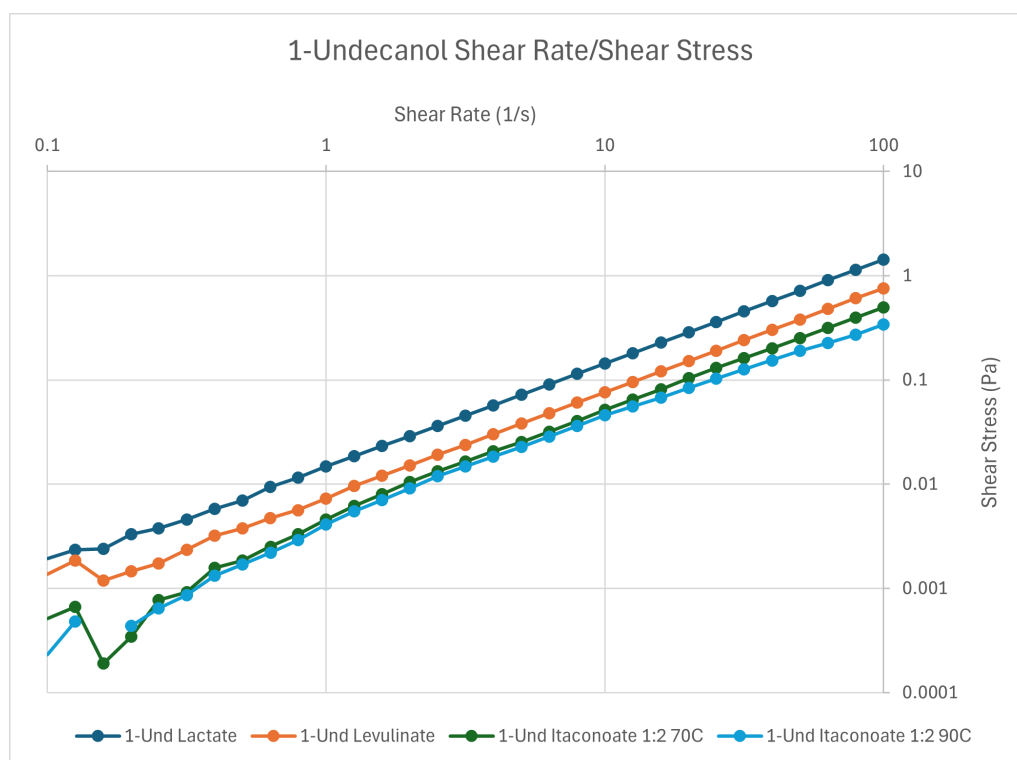


Figure A.33: Graph showing Shear Rate/Shear Stress for 1-Undecanol Products.

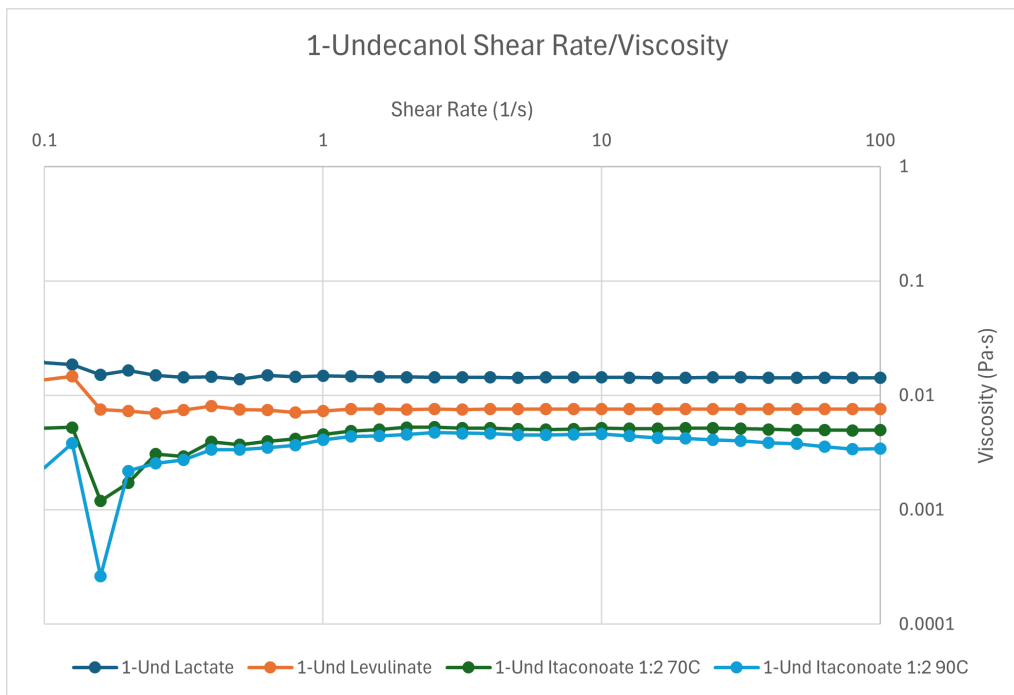


Figure A.34: Graph showing Shear Rate/Viscosity for 1-Undecanol Products.

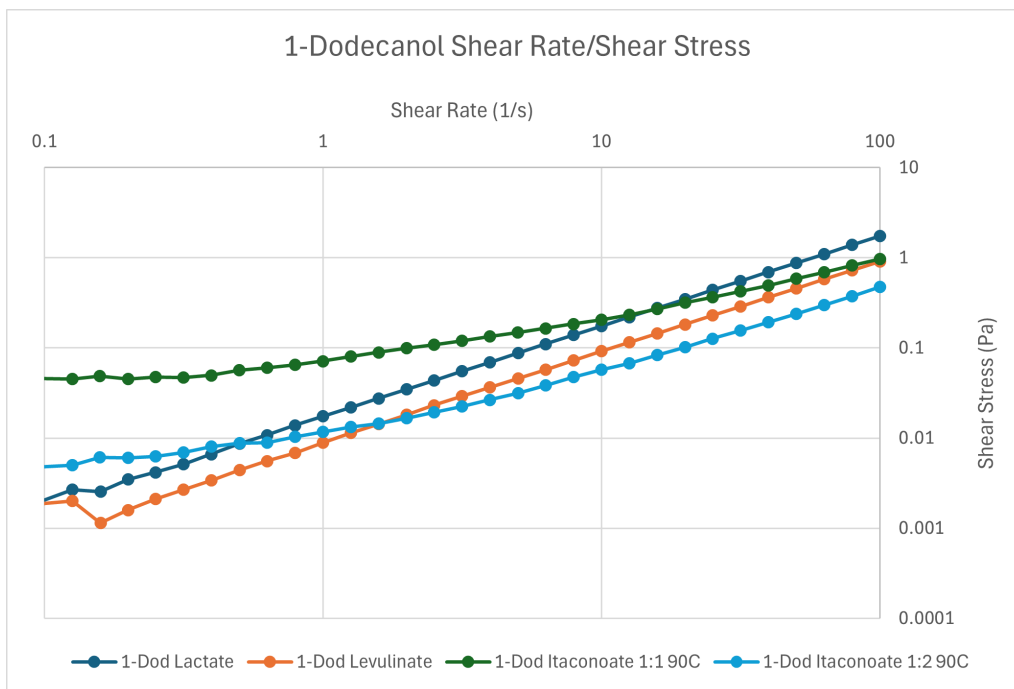


Figure A.35: Graph showing Shear Rate/Shear Stress for 1-Dodecanol Products.

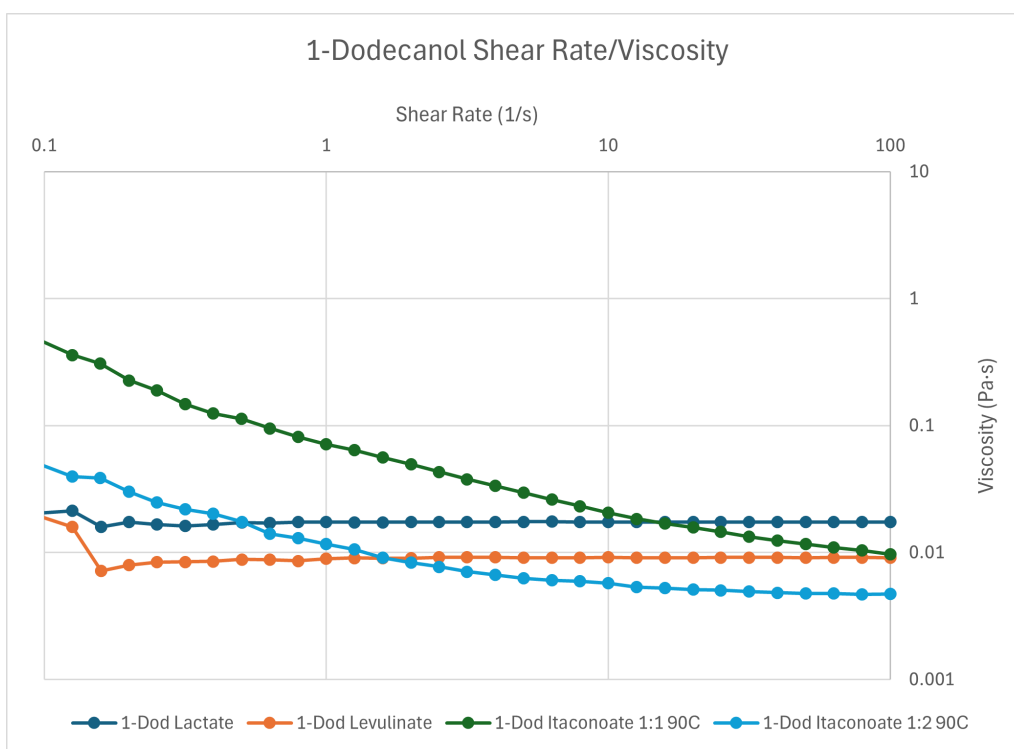


Figure A.36: Graph showing Shear Rate/Viscosity for 1-Dodecanol Products

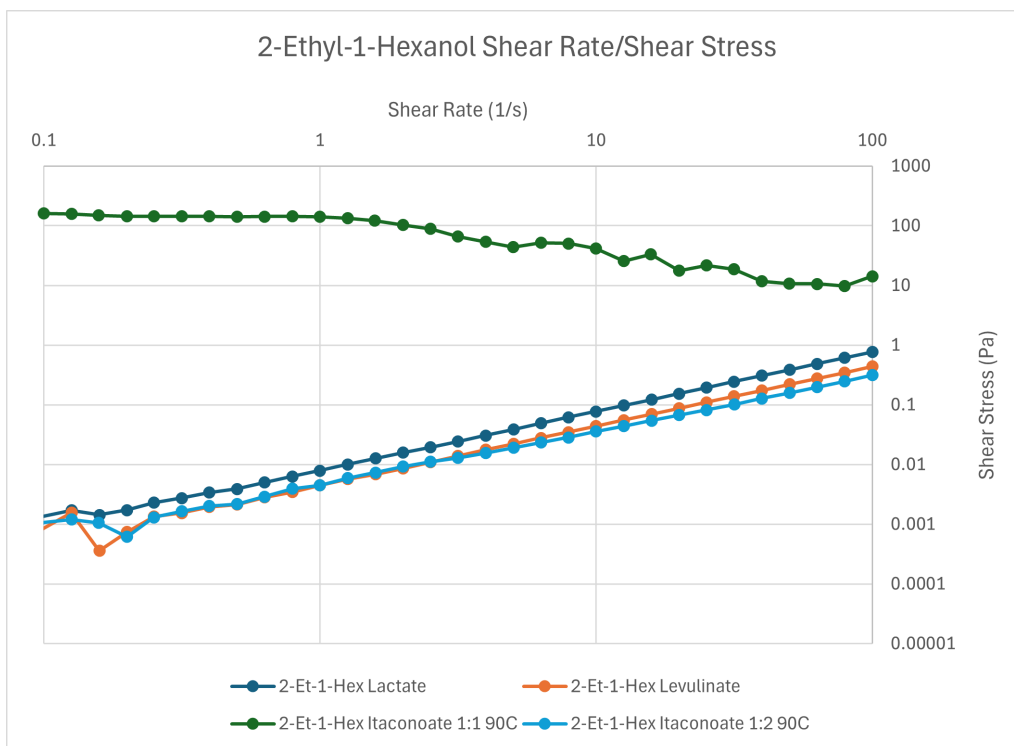


Figure A.37: Graph showing Shear Rate/Shear Stress for 2-Ethyl-1-Hexanol Products.

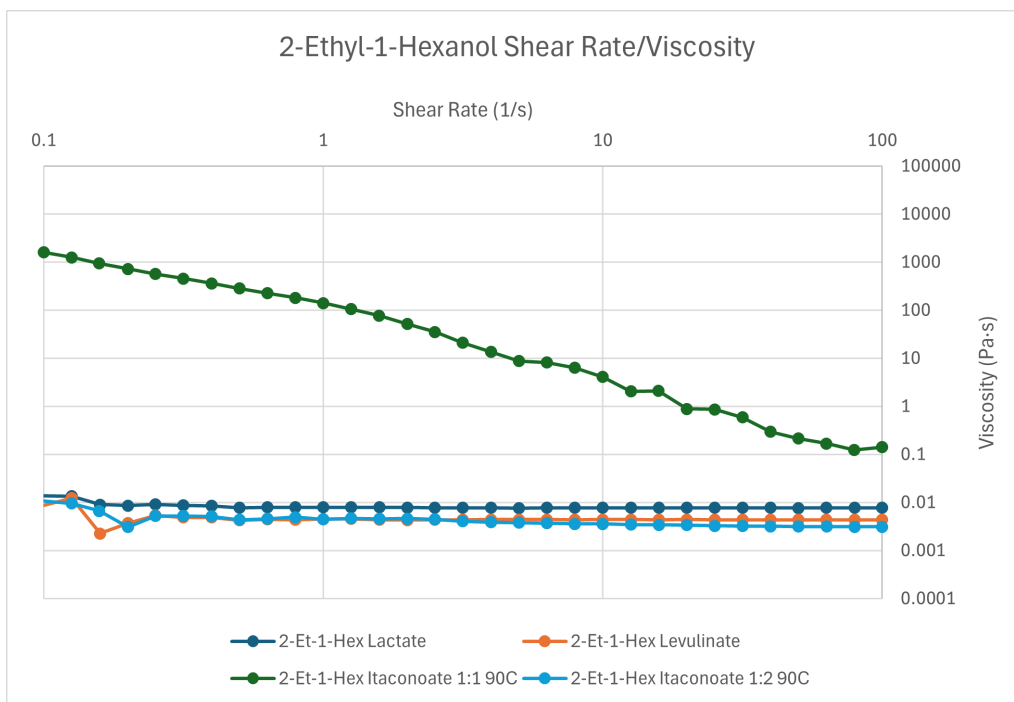


Figure A.38: Graph showing Shear Rate/Viscosity for 2-Ethyl-1-Hexanol Products.

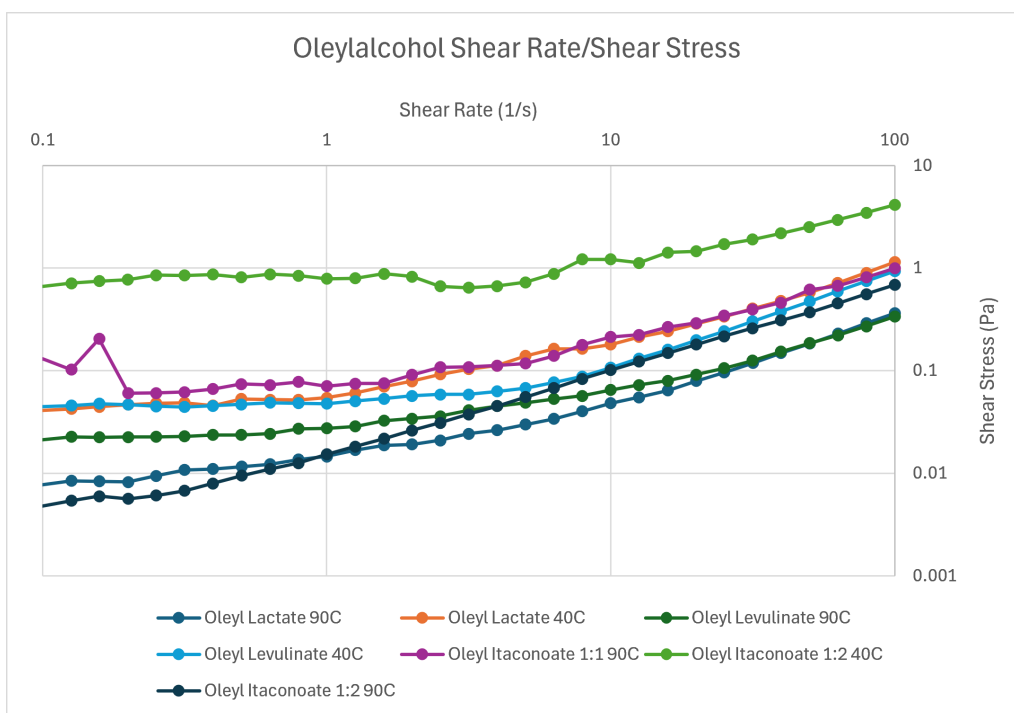


Figure A.39: Graph showing Shear Rate/Shear Stress for Oleylalcohol Products.

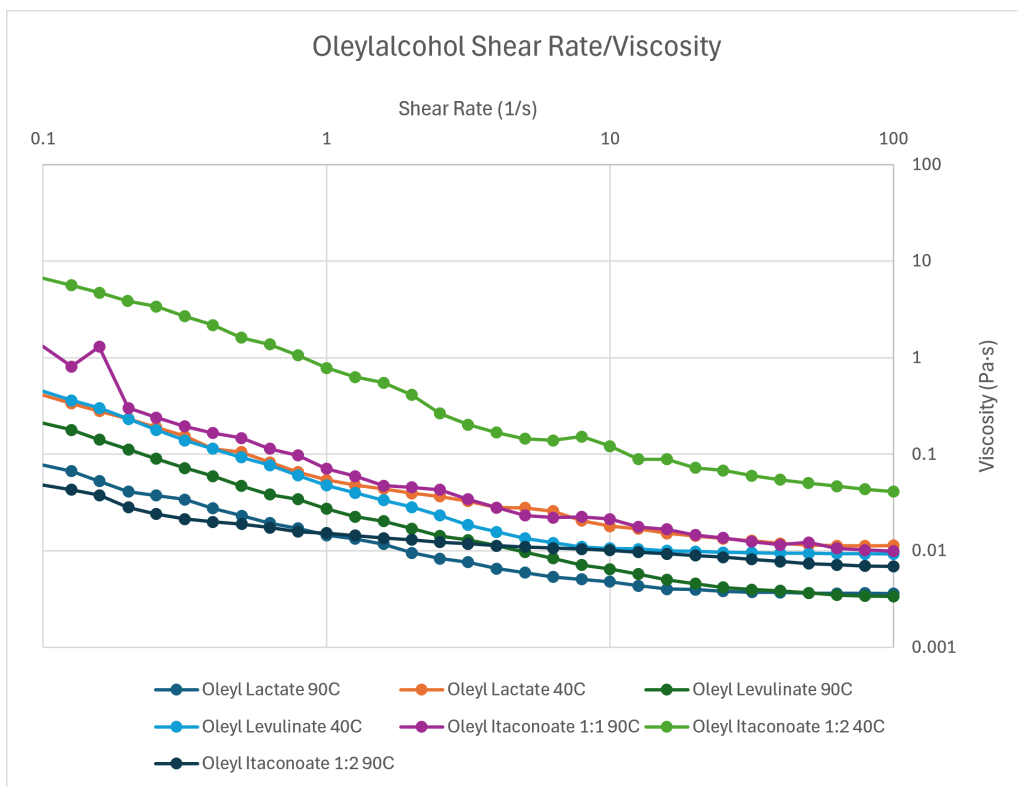


Figure A.40: Graph showing Shear Rate/Viscosity for Oleyl Alcohol Products.

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