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The Roasting Procedure and Its Characteristics Using Solid-State NMR: Case Study of Bengkulu Robusta Coffee

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Case Report

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Abstract

Solid-state Nuclear Magnetic Resonance (SS NMR) spectroscopy revealed considerable chemical changes in Bengkulu Robusta coffee roasting. The ^{1}H MAS NMR spectra showed increased caffeine peak strength at 3.2 ppm after roasting, indicating higher caffeine content. Aliphatic chains were observed in the region of 1.4–1.8 ppm, and the 2D HETCOR approach confirmed correlations between proton and carbon signals at 1.8 ppm–30.58 ppm and 1.5 ppm–30.15 ppm, respectively. Melanoidins, which give the coffee a brown color, were found at 4.5 ppm. The ^{13}C MAS NMR spectra showed kahweol at 77 ppm, a chemical found in Arabica coffee, suggesting a Robusta-Arabica blend. The presence of succinate and carboxylic acid peaks at 60.3 ppm and 171.7 ppm indicated Maillard reaction products. These findings demonstrate solid-state NMR's fast and non-destructive ability to characterize roasting-induced chemical alterations. The study shows coffee producers and customers how roasting conditions affect coffee quality and authenticity.

1. INTRODUCTION

Coffee roasting is a pivotal process of transforming raw coffee beans into an aromatic and flavorful beverage millions worldwide enjoy. Roasting is a critical thermal process widely used in the food industry, particularly in the preparation of coffee, cocoa, nuts, and grains, as it significantly influences the flavor, aroma, color, and texture of the final product. The complex chemical transformations that occur during roasting—such as Maillard reactions, caramelization, and the breakdown of cellular structures—contribute to the development of desirable sensory characteristics. Understanding these transformations at the molecular level is essential for optimizing roasting conditions and ensuring consistent product quality. Solid-state Nuclear Magnetic Resonance (NMR) spectroscopy has emerged as a powerful analytical technique for investigating the structural and dynamic properties of solid food matrices. Unlike solution-state NMR, solid-state NMR enables the analysis of non-soluble, heterogeneous materials, making it particularly suitable for studying roasted food products. The roasting process imparts coffee's characteristic taste and aroma and determines its chemical composition and health benefits [1–3]. Among the various methods available, the roasting of Robusta coffee beans, particularly from unique regions like Bengkulu, Indonesia, offers an intriguing case for study due to its distinctive flavor profile and high caffeine content, the importance of roasting, the scientific understanding of how specific roasting procedures affect the final quality of coffee remains limited, especially concerning the molecular-level changes that occur during the process [4, 5].

Solid-state Nuclear Magnetic Resonance (SS NMR) spectroscopy presents a powerful tool for exploring molecular changes [6–8]. Unlike earlier analytical methods, solid NMR allows for the detailed investigation of coffee's solid-state components, including the complex interactions between its organic compounds. Furthermore, the tool offers several additional benefits, including the simplicity of material preparation, elimination of the need for solutions, and a relatively straightforward operational process [7, 9]. The solid NMR to the study of coffee roasting, researchers can gain faster insight into how roasting

conditions influence the chemical composition of coffee beans, ultimately affecting their sensory characteristics and potential health benefits.

Several previous studies have shown the content of organic compounds in coffee. Researchers [2, 10] generally said caffeine and chlorogenic acid are commonly found in coffee. Caffeine, also known as 1,3,7-trimethylxanthine, is a natural purine alkaloid. However, it is important to acknowledge that the substances examined by the researchers displayed variances based on the precise kind, the origin of the coffee, and the testing method and phase (liquid/solid) employed. According to another study [5], there are more than a hundred different compounds, from the bean to the roast.

This study focuses on good coffee roasting procedures based on the experience of coffee lovers and the application of solid NMR to analyze the roasting product of Bengkulu Robusta coffee, a variety renowned for its bold flavor and resilience. This research aims to identify the compounds generated during the roasting process of Bengkulu Robusta coffee beans using a simple and rapid analytical technique. The achieve this by exploring how specific parameters and roasting conditions impact the beans' chemical composition. Through this case study, we seek to contribute to the broader understanding of coffee roasting science, providing valuable insights for coffee producers and consumers who value high-quality coffee.

2. EXPERIMENTAL

2.1. Materials

Green Robusta coffee beans were obtained directly from farmers in Tes village, South Lebong District, Lebong Regency, Bengkulu Province, Indonesia.

2.2. Roasting Machine and Its Procedure

The coffee roasting machine in focus boasts a capacity of 10 kg per batch and features a rotating flat cylinder design. Its roasting cylinder is constructed from 3 mm thick 304 stainless steel with a diameter of 450 mm, complemented by a stirrer made from a 2 mm thick stainless-steel plate. The feeder and seed production hopper are crafted from 1.5 mm stainless steel. Heating is provided by an LPG gas burner, utilizing an indirect system to ensure even roasting. The machine includes a digital temperature thermostat indicator and a gas solenoid valve for precise control. Centrifugal fans are installed to manage dust, smoke, and bark removal.

The drive system consists of a 1/2 PK electric motor gear operating at 380 volts, with a variable speed inverter that takes a 220-volt electrical input. Transmission is facilitated through a gear chain, V-belt, pulley, and a type 60 reducer. The frame is made from a 4x4 box pipe, the body wall is constructed from a 2 mm plate, and the cylindrical blanket is made of 1.2 mm thick stainless steel.

The initial step in the roasting process involves verifying the presence of gas, electrical connections, and the condition of the roasting hopper within the roasting machine. This procedure serves as a

precautionary measure following the previous operation, particularly in cases where coffee residue may remain in the hopper. It is imperative to ensure that all components are thoroughly cleaned and properly arranged. Press the "on" button to start the coffee roaster, which will automatically preheat to approximately 80°C. Measure out about 250 grams of green coffee beans and fill the roaster hopper with them. Set the gas valve to half open and roast the beans for 7 minutes at 175°C. They will turn yellow, emit a baked bread scent, and start to crack. Afterward, turn off the shutdown button and wait a minute. Then, transfer the beans to a container to release CO_2 for 2–3 minutes before placing them in a glass jar.

2.3. Solid-State NMR Spectroscopy

Green and roasted coffee beans must first be ground and filtered using a 24 cm x 4.5 cm filter before being measured using the NMR instrument. The ^{1}H and ^{13}C Magic-Angle Spinning (MAS) NMR spectra were obtained at a frequency of 500 MHz using a JEOL JNM-ECZ500R solid-state NMR spectrometer. The measurements were conducted using a zirconia rotor with a diameter of 4 mm. The rotational frequency of the rotor was 6 kHz. The spectra were obtained using 0.1 us pulses, 5 and 15 s relaxation delay, and 5 and 100 scans for proton and carbon measurement, respectively. Meanwhile, for HETCOR, the main parameters are ^{13}C for X_domain, ^{1}H for Y_domain, 5 s relaxation delay, and 4320 scans.

3. RESULTS AND DISCUSSION

3.1. ^{1} H (Proton) Spectra of MAS NMR

Figure 1 showcases a sample ^{1}H NMR spectrum of green and roasted coffee beans. By combining 1D NMR analyses with experiments from earlier research, five components were established[11–14]. The coffee bean extract spectra under both circumstances were dominated by several compounds, as depicted in Fig. 1. As is generally known, the most popular compound is caffeine. In the spectra, both peaks in green and roasted beans at 3.2 ppm. In a specific study of caffeine synthesis [15], peaks were found that were approximately the same as in this study. After roasting, the peak intensity slightly increases, indicating increased caffeine levels. A study by Hečimović et al.[16] demonstrated that the roasting process, conducted at a light temperature of approximately 180°C, increased caffeine levels. This finding aligns with the results of our NMR method and interpretation.

Then, the aliphatic chains and amine groups were identified in the range of 1.4 to 1.8 and 2.2 to 2.5 ppm, respectively. Figure 3 shows the next phase in this investigation: proton integration using Solid-state NMR. It has revealed the presence of alkane compounds in two distinct chains. The interpretation of Solid-state Nuclear Magnetic Resonance (SS NMR) data indicates the presence of alkane compounds, a finding that aligns with previous studies in the field [17]. The solid-state NMR technique can detect various types of carbon bonds. While alkane compounds may contribute minimally to the taste and aroma of coffee, they are detected due to their presence in reactions that naturally occur in the coffee membrane and during coffee beans' roasting [18].

Next is Melanoidins, compounds that appear in the range of 4.5 ppm, responsible for giving the coffee a brownish colour [19]. As the temperature and roasting time increase, the intensity of this compound will continue to increase. It is also responsible for the distinctive aroma of coffee. The Maillard reaction [13, 19], a reaction between components with carbonyl groups, including melanoidins, carboxylic acids, esters, and others with amino acids, is the mechanism by which this occurs.

In addition, other studies [20] indicate that chlorogenic acid is attached to melanoidin during roasting. This finding is supported by research [21], demonstrating that the acid exhibits the same peak intensity (chemical movement) as melanoidin in our proton spectra. Of all the compounds mentioned according to the proton NMR spectra, Kahweol is of particular note. This aromatic compound strongly appears in Arabica [7, 12] coffee, not Robusta. So, based on this solid-NMR investigation, it is suspected that the coffee being studied is a mixture of Robusta-Arabica coffee and not pure Robusta coffee.

Robusta coffee is grown at an altitude of 500–750 meters above sea level[22, 23], while Arabica coffee is grown at 800–1500 [24, 25], or even 1000–1700 meters above sea level [26].

3.2. ^{13}C (Carbon) Spectra of MAS NMR

Figure 2 showcases a sample 13C NMR spectrum of green and roasted coffee beans. An aliphatic chain has been identified in the initial peak range of the proton spectra, then strengthened by observing this group peak in the carbon spectra at 14 to 34.5 ppm. Subsequent peaks were identified as ester compounds at 60 ppm and kahweol at 77 ppm. Caffeine was also detected as an aromatic carbon compound at 127 ppm. The final peak was identified as carboxylic acid at 172 ppm. This carbon interpretation [11, 27] amplifies the proton interpretation.

Notably, ester compounds and carboxylic acids possess a carbonyl group (C = O), which is crucial for the Maillard reaction. Further analysis and study determined that the chemical shifts observed in the NMR spectra of ester and carboxylic acid groups were consistent with the presence of succinic and succinate groups. The conclusion is demonstrated by the peak integration of the proton, identified as a proton from succinic acid in the second and third branches, with an integration area of 0.14 (Fig. 3). Meanwhile, the peak of the carboxylic acid and ester was elucidated as succinate in Fig. 4, respectively.

A report [28] indicates that succinic acid can be extracted through the skin of Arabica coffee. It further confirms our research on the blend of Robusta and Arabica coffee in the components deployed. Some reports [29–31] reveal many succinic acid and succinate applications. Among the most significant of these are: (1) Succinic acid is a useful regulator of acidity, anti-inflammatory, and antioxidant for the skin; (2) succinate acts as a flavor enhancer in food products and as a stabilizer in medicines. Furthermore, it is noteworthy that both can be employed as raw materials for biodegradable plastics.

3.3. 2D NMR: Hetero Correlation (HETCOR)

Figure 5 showcases the 2D NMR HETCOR analysis, which found two significant correlations between proton and carbon signals. The proton signal at 1.8 ppm corresponded to the carbon signal at 30.58 ppm, whereas the proton signal at 1.5 ppm corresponded to the carbon signal at 30.15 ppm. The carbon signal about 30 ppm is typically assigned to carbon atoms in the -CH_2- group, which is a crucial component of the aliphatic chain in the lipid or fatty acid structure. Previous studies have linked the ^{13}C signal in this range to methylene (CH_2) groups in aliphatic environments [11, 32].

The minor discrepancy in proton shift readings (1.8 ppm to 1.5 ppm) suggests changes in the chemical environment around the -CH_2- group. Protons at 1.8 ppm exhibit minor deshielding, indicating that the -CH_2- group is closer to electron-withdrawing functional groups like carbonyl or aromatic groups. In contrast, the protons at 1.5 ppm show a more aliphatic environment with little influence from electron-drawing groups [11, 32]. The direct link between protons and carbons supports the analysis that these shift variations reflect local differences in the chain structure of alkanes, derivative products of lipids under investigation.

The 2D HETCOR NMR results not only validate the structural alterations of aliphatic components during roasting but underscore their potential significance in flavour chemistry and quality evaluation. The observed carbon changes at around 30 ppm may relate to lipid oxidation or breakdown products, which further modify the composition of roasted coffee [5, 33]. These interactions exhibit the complex chemical composition of roasted coffee, wherein aliphatic and aromatic changes play a role in flavour creation and serve as potential quality indicators for coffee authentication.

4. CONCLUSION

The roasting process of Bengkulu Robusta coffee induced notable chemical alterations, as demonstrated by solid-state NMR spectroscopy. The examination revealed elevated caffeine levels, the existence of alkanes, and the synthesis of melanoidins, which enhance the coffee's distinctive flavour, fragrance, and colour. The identification of kahweol, commonly present in Arabica coffee, indicates a potential blend of Robusta and Arabica beans. These findings underscore the efficacy of solid-state NMR in detecting chemical alterations generated by roasting, offering significant insights for coffee growers and consumers. This study highlights the essential influence of roasting parameters on coffee quality and authenticity, facilitating the development of more sophisticated methods and analysis in coffee science.

Declarations

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Figures

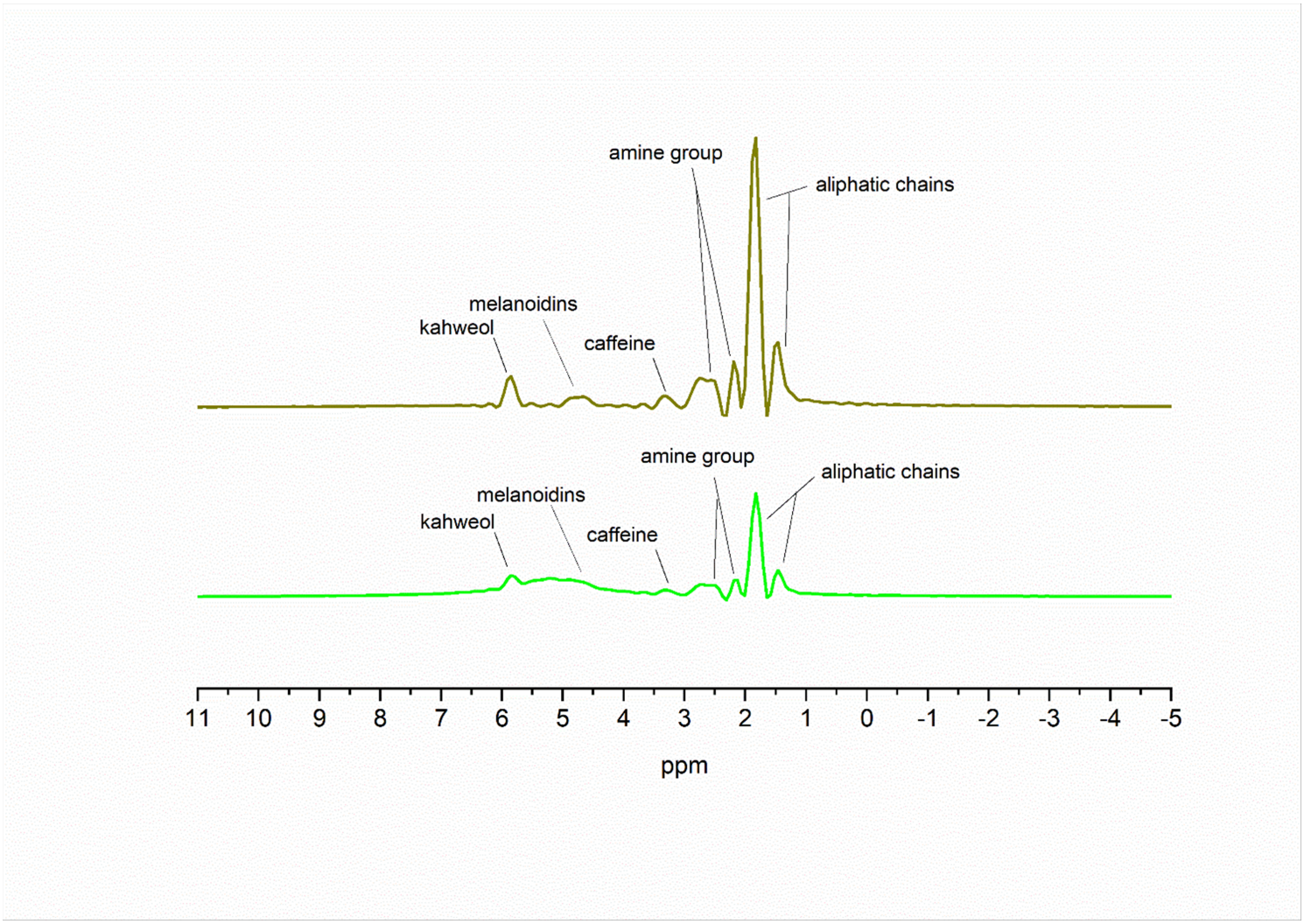


Figure 1

^{1}H spectra of green (down) and roasted (top) beans coffee

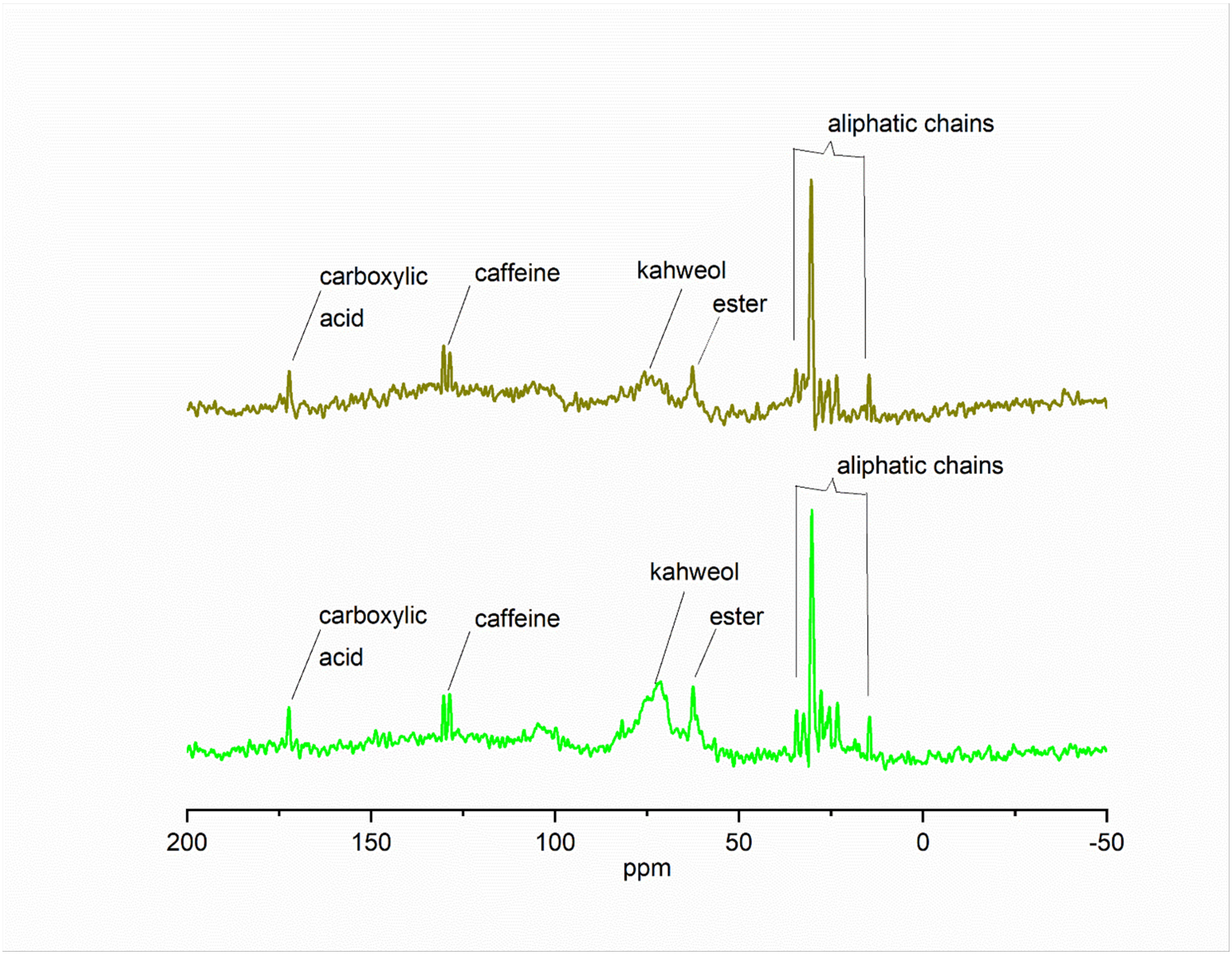


Figure 2

^{13}C spectra of green (down) and roasted (top) beans coffee

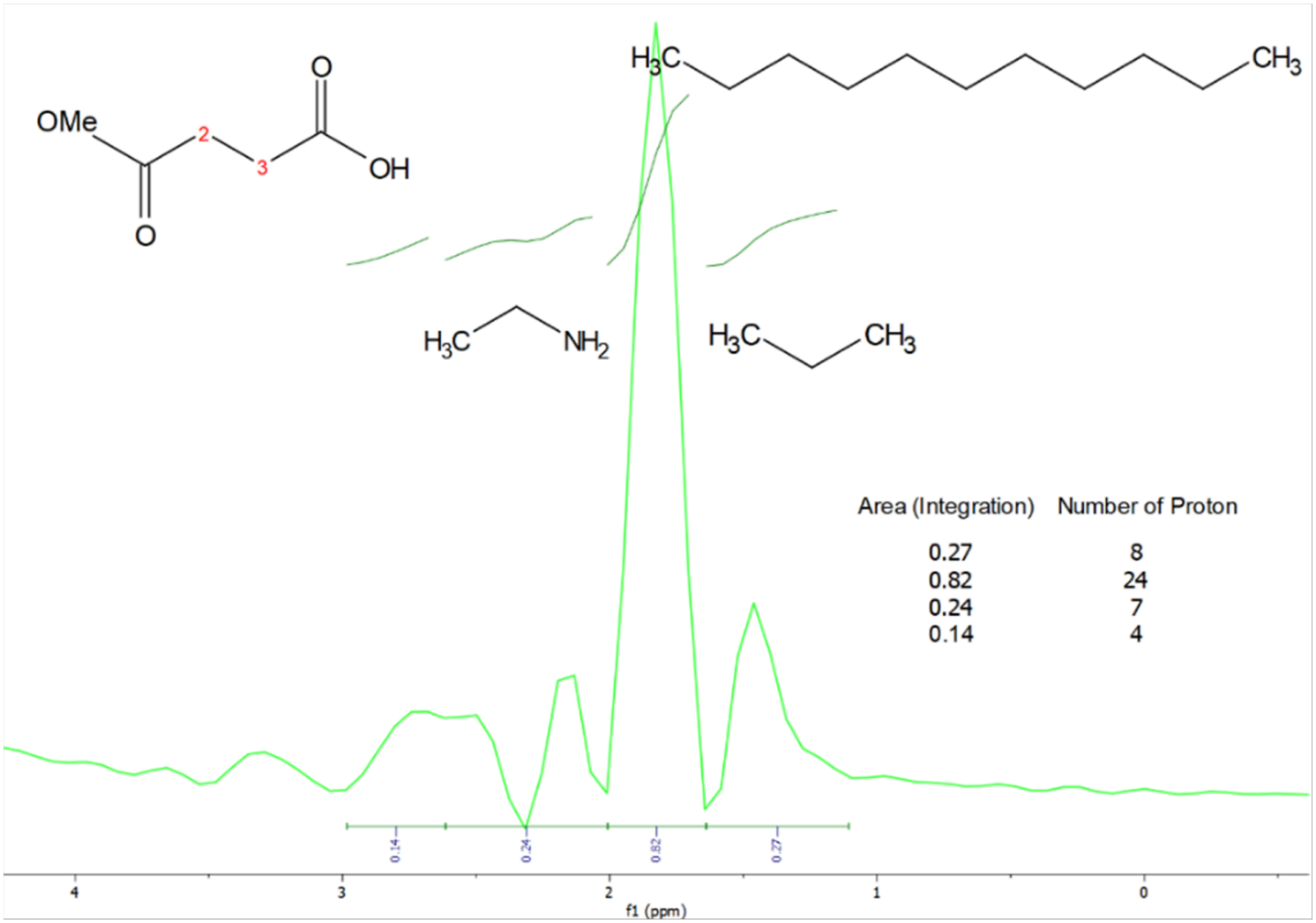


Figure 3

Elucidated Structure between 0-4 ppm on ^{1}H spectra

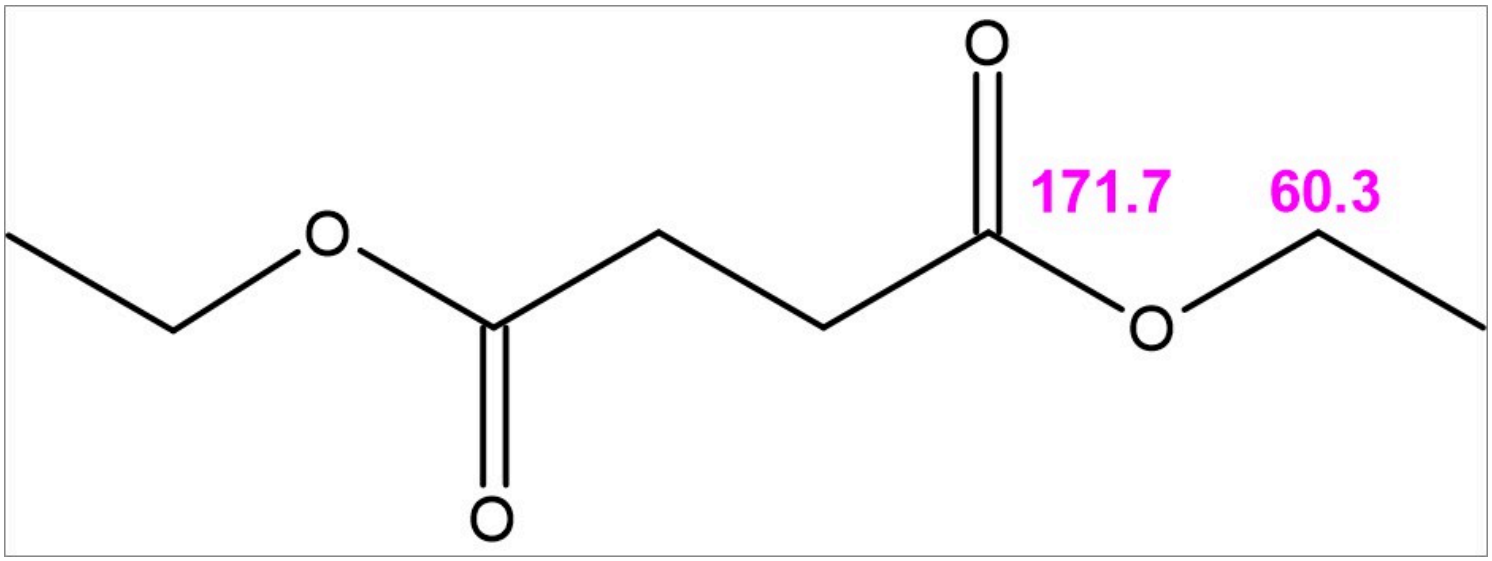


Figure 4

Succinate at 171.7 and 60.3 ppm

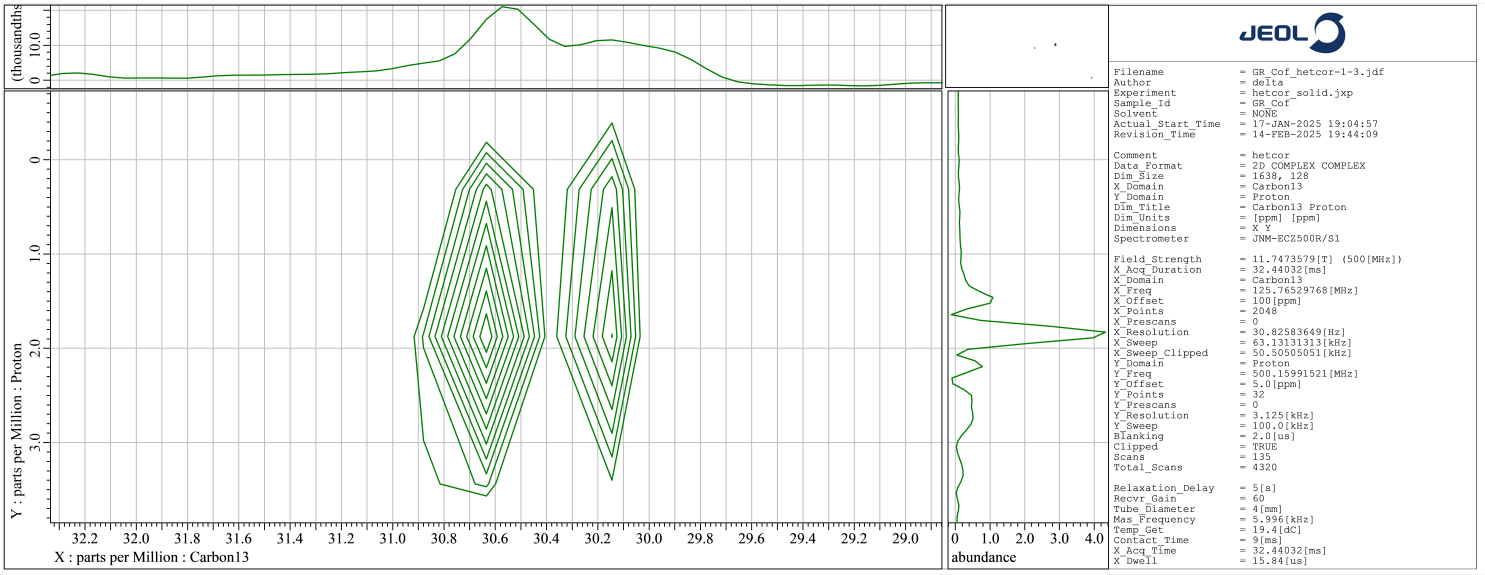


Figure 5

2D HETCOR (Relationship between ^{1}H and ^{13}C nuclei)