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How to Read and Interpret 1H-NMR and 13C-NMR Spectrums

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ABSTRACT

Nuclear magnetic resonance spectroscopy or NMR is a chemical instrument that can be used to evaluate the structure of a chemical compound other than FTIR, GC-MS, and HPLC. NMR spectroscopy commonly used for compound analysis is ^{1}H-NMR and ^{13}C-NMR. Techniques can be used to determine the structure conformation, the number of protons, and the number of carbons in the structure of a chemical compound. So far, there have been many publications related to the use of this spectroscopic technique. However, the steps in reading and interpreting the spectra of both ^{1}H-NMR and ^{13}C-NMR are not described in detail. Thus, in this paper, we described the steps in reading and interpreting the ^{1}H-NMR and ^{13}C-NMR spectra based on the level of difficulties: (1) simple compounds, (2) fairly complex compounds, (3) more complex compounds, and (4) very complex compounds.

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1. INTRODUCTION

A nuclear magnetic resonance spectrometer (NMR) is a chemical instrument used to obtain information regarding the structure and conformation of a chemical compound. NMR spectroscopy is a fairly good method of elucidation in determining the structure of organic compounds. NMR spectroscopy utilizes the interaction between the nucleus which acts as a small magnet and an external magnetic field, making it applicable for evaluating chemical bonds and the nuclear environment (Dayrit & de Dios, 2017). The signal obtained from NMR spectroscopy provides information about the interactions between nuclei and electrons as well as interactions between nuclei, which can help to determine the structure of a chemical compound (Hilal et al., 2017). The resulting NMR spectrum is a collection of one or more resonant peaks at a certain frequency.

There are two types of NMR spectroscopy, namely ^{1}H-NMR and ^{13}C-NMR. One of the important pieces of information that the ^{1}H-NMR spectrum shows is the chemical shifts of the different types of protons in the sample, whereas ^{13}C-NMR can provide structural information related to a compound based on the chemical shifts of various types of carbon. Apart from being used to determine the structure of chemical compounds, NMR spectroscopy can also be used in advanced medical imaging techniques, such as MRI. NMR has now become an analytical technology that can be applied in many disciplines of research, medicine, and various industries (Hameed et al., 2017).

Many studies have described the application of ^{1}H-NMR and ^{13}C-NMR spectroscopy in the analysis of the structure of a chemical compound, especially organic compounds such as the analysis of levodopa compounds using ^{1}H-NMR conducted by Talebpour et al. (2004), analysis of eugenol compounds. which were extracted from cloves using ^{1}H-NMR and ^{13}C-NMR (Thirukumaran et al., 2014), pyrazole-based ligand analysis (Bouroumane et al., 2021), and others. In addition to qualitative use, NMR spectroscopy can also be used for quantitative analysis purposes (Fernandez-Pastor et al. (2019)). However, the reading and interpretation of the ^{1}H-NMR and ^{13}C-NMR spectra were not described in detail.

Based on our previous studies on the data interpretation of FTIR and adsorption isotherm (Nandiyanto et al., 2019; Ragadhita & Nandiyanto, 2021), the purpose of this study was to explain how to read and interpret the ^{1}H-NMR and ^{13}C-NMR spectra through step-by-step exposure in determining simple compounds, fairly complex compounds, more complex compounds, and very complex compounds. Interpreting is very important since it can allow researchers and practitioners for further analyses (Mohamad et al., 2021).

2. CURRENT THEORIES FOR ^{1}H-NMR AND ^{13}C-NMR SPECTRUM

2.1 Spectrum in the ^{1}H-NMR and ^{13}C-NMR Analysis Result

^{1}H-NMR spectroscopy is an analytical method used to determine the structure of a compound based on the type of proton or hydrogen. The ^{1}H-NMR spectrum provides information regarding the number of proton types in a compound and the environmental properties of each type of hydrogen proton. According to Harwood and Claridge (1997), there is some important information appearing in the ^{1}H-NMR spectrum, such as

1) The proton resonance is distributed along the frequency axis. Each proton is

in a different chemical environment characterized by its chemical shift (δ).

2) Different peaks in the spectrum can be seen appearing with different intensities which related to the number of protons giving rise to the signal.
3) Multiple proton resonances can interact with neighboring atoms. The degree of interaction or coupling is indicated by the coupling constant (J).

The absorption peak that appears in the ^{1}H-NMR spectrum is represented by the difference in the resonance frequency of a nucleus against the standard in units of ppm or chemical shift (δ). The value of chemical shift (δ) is influenced by several factors, such as: (1) inductive effect, (2) bond anisotropy, and (3) hydrogen bond formation. The schematic of the ^{1}H-NMR spectral peaks for various types of proton absorption is shown in **Figure 1**.

Figure 1 shows that the inductive effect of an electronegative atom such as oxygen and nitrogen causes peaks to appear in large chemical shifts, known as de-shielded. This can occur because an electronegative atom such as O has the direction of the electron cloud circulation in the same direction as the external magnetic field, thus giving the effect of magnetic field induction. In addition to the induction effect of the presence of electronegative atoms such as N and O, chemical shifts are also influenced by the anisotropy of a chemical bond, such as compounds with alkene (C=C), alkynes (C≡C), carbonyl (C=O), and aromatic (Ar) groups. The peaks appear at a greater chemical shift in the presence of the double bond.

In contrast to ^{1}H-NMR, the absorption peak shown in the ^{13}C-NMR spectrum provides structural information based on the chemical shifts of various types of carbon in a chemical compound. The schematic of the ^{13}C-NMR spectral peaks for various types of proton absorption is shown in **Figure 2**. The chemical shift of carbon is determined by the type of carbon bond itself. The carbonyl carbon (C=O) is highly de-shielded and has a larger chemical shift value, the carboxylate and ester groups have smaller chemical shift values. Ketone and aldehyde groups have a chemical shift value of around 200 ppm, while aromatic carbon has a chemical shift value of between 110–160 ppm. Carbon with double bonds has a chemical shift value between 100–50 ppm, metine, methylene, and methyl have a chemical shift value between 10–50 ppm.

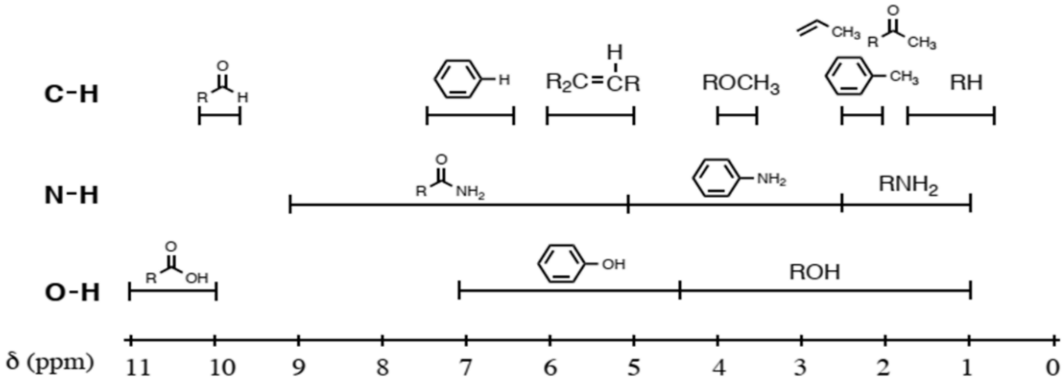

Figure 1. Chemical shift (δ) of the ^{1}H-NMR spectra

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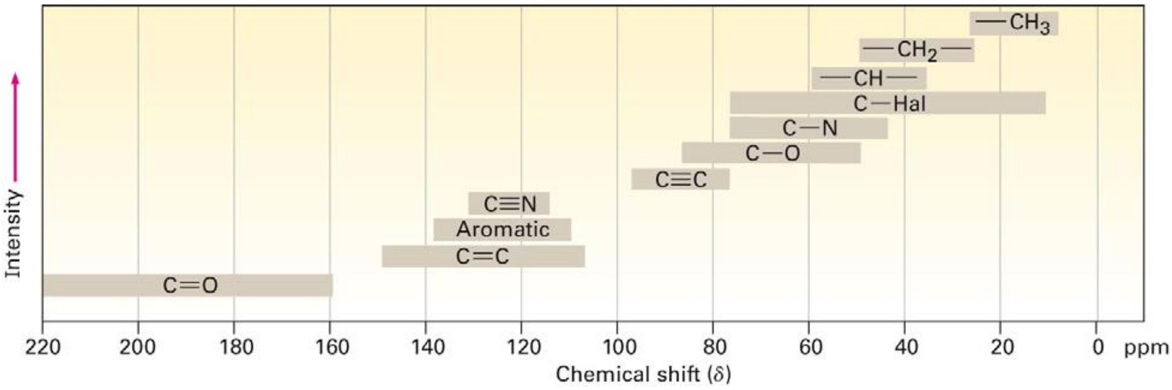


Figure 2. Chemical shift (δ) ^{13}C-NMR spectra

2.2 Step-by-step Analysis Procedure

2.2.1 ^{1}H-NMR Spectra

There are six primary steps in reading and interpreting the ^{1}H-NMR spectrum, including:

1) **Step 1:** Identify the number of signals that appear by observing the chemical environment of the structure of the compound being analyzed. The signal that appears represents the difference in the chemical environment of the hydrogen atom in a molecule. For example, **Figure 3** shows the structure of the compound p-cymene.

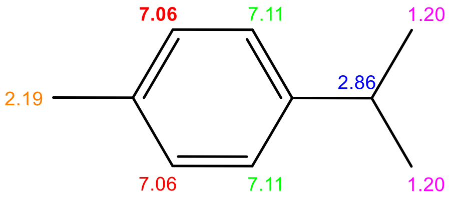


Figure 3. Structure of p-cymene

   The p-cymene structure shows the presence of CH and CH_3 groups that are in the same chemical environment as shown in **Figure 3**. CH and CH_3 groups that are in the same chemical environment are shown as one spectrum peak so that the p-cymene compound give rise to five spectral peaks. ^{1}H-NMR.

2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. This identification is done to determine the multiplicity or peak pattern that appear in the 1H-NMR spectrum.

   There are several patterns of signal multiplicity in the ^{1}H-NMR spectrum as follows:

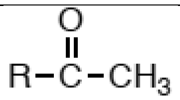
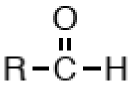
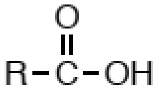
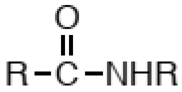
   a) Singlet: Protons without neighboring protons that are not magnetically equivalent show a single peak in the ^{1}H-NMR spectrum.
   b) Doublet: Protons with one non-equivalent neighboring proton give rise to a peak that is split in half or double.
   c) Triplet: A proton with two neighboring protons that are not equivalent to each other give rise to a peak that is split into three.
   d) Quartet: A proton with three neighboring protons that are not equivalent to each other give rise to a peak that is split into four.

3) **Step 3:** Identify the signal integration value. Integration shows the relative number of H obtained from the measurement of the length of each peak. The greater the integration value, the more protons that generate the signal.

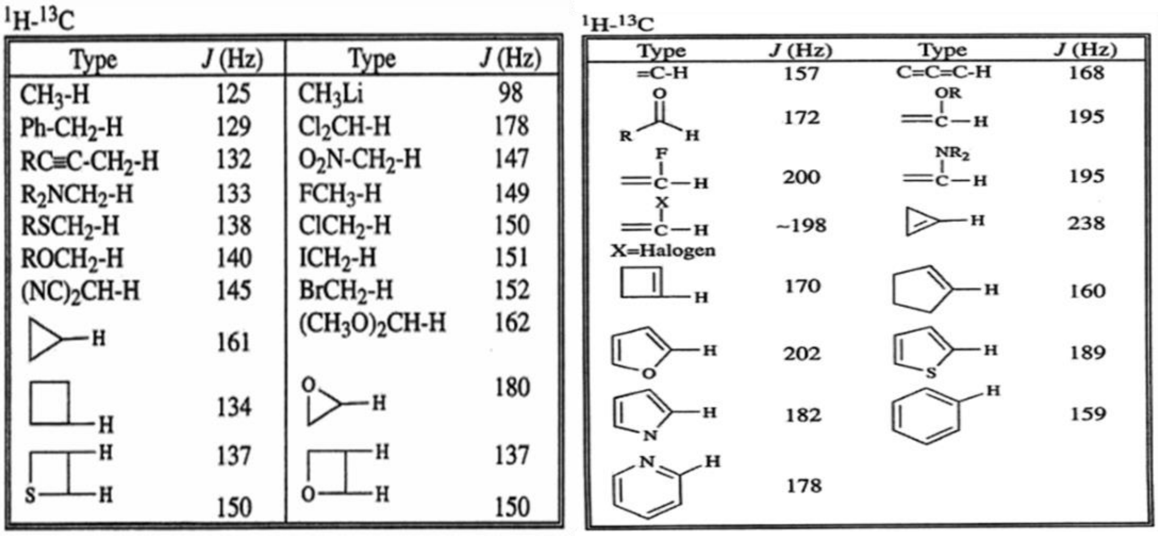
4) **Step 4**: Identify the peaks based on the chemical shift value (δ). Chemical shift values for certain types of protons in a

particular group. The chemical shift value (δ) of a nucleus arises as a result of the presence of electrons in a molecule which forms a shielding effect on the spin of the nucleus. An atom with a low or near TMS chemical shift value is called shielded, while a high or distant chemical shift (δ) value with TMS is called de-shielded. The value of chemical shift (δ) is influenced by several factors, such as: (1) inductive effect, (2) bond anisotropy, and (3) hydrogen bond formation. The chemical shift values in ^{1}H-NMR spectroscopy are shown in **Table 1**.

Table 1. Chemical shift ^{1}H-NMR

Type of Bond	Chemical shift (δ) (ppm)	Description
$R-CH_3$	0.9	*Alkyl (methyl)*
$R-CH_2-R$	1.3	*Alkyl (methylene)*
R_3C-H	1.5 – 2	*Alkyl (methine)*
	1.8	*Alylic*
 $R-\overset{\overset{O}{\parallel}}{C}-CH_3$	2 – 2.3	*CH α – carbonyl (C=O)*
$Ar-CH_3$	2.3	*Benzylic (C-Ph)*
$RC\equiv C-H$	2.5	*Alkynyl*
R_2N-CH_3	2 – 3	*CHα – N*
$R-CH_2-X$	2 – 4	*CHα – halogen (Cl, Br, I)*
$RO-CH_3$	3.8	*CHα – oxygen*
$R-CH_2-F$	4.5	*CHα – fluorine*
$Ar-H$	7.3	*Aromatic*
 $R-\overset{\overset{O}{\parallel}}{C}-H$	9.7	*Aldehyde*
ROH	0.5 – 5	*Alcohol*
ArOH	4 – 7	*Phenol*
 $R-\overset{\overset{O}{\parallel}}{C}-OH$	10 – 13	*Carboxylic acid*
RNH_2	0.5 – 5	*Amine*
$ArNH_2$	3 – 5	*Aniline*
 $R-\overset{\overset{O}{\parallel}}{C}-NHR$	5 – 9	*Amide*

5) **Step 5:** Identify the coupling constant (J). The coupling constant ^{13}C-^{1}H has a value ranging from 125 to 250 Hz depending on the character of the C to H bond and the C and C bond. This step can be done if the coupling constant data is shown on the spectrum. The value of the coupling constant is shown in **Figure 3**. The value of the coupling constant (J) in **Figure 3** reflects the existence of the bonding environment of a nucleus. The J value of a proton is so specific that a lot of information can be retrieved. For example, a double bond can take two forms, namely cis and trans. For trans double bonds have a J value between 12-18 Hz, cis between 6-11 Hz, and geminal between 0–3 Hz. The J value of an aromatic also provides important information about the position of the functional group in an aromatic. The proton signal of a benzene derivative with an ortho position has a J value of 7.5 Hz, a meta of about 1.5 Hz and a para has a J value of 0.7 Hz, whereas a naptalene with an ortho position has a J value of about 8.3, meta 1.3, and para 0.7 Hz.
6) **Step 6:** From steps 1 - 5, the structure of an organic compound can be determined. The analysis results were combined and concluded the structural results from the NMR spectrum. First, after the number of proton types and the chemical environment of the protons are known in step 1, the molecular formula based on their bond with H can be determined. Second, the signal multiplicity analysis provides information on how many hydrogen atoms are present in the adjacent carbon atoms. Finally, the pieces of the molecule are combined to form a structural formula for the compound. The coupling constant obtained in the previous step shows the interactions between the protons and the chemical shift values obtained are then compared with the chemical shift table values (**Table 1**) to identify their functional groups.



^{1}H-^{13}C

Type	J (Hz)	Type	J (Hz)
CH_3-H	125	CH_3Li	98
Ph-CH_2-H	129	Cl_2CH-H	178
RC≡C-CH_2-H	132	O_2N-CH_2-H	147
R_2NCH_2-H	133	FCH_3-H	149
$RSCH_2$-H	138	$ClCH_2$-H	150
$ROCH_2$-H	140	ICH_2-H	151
$(NC)_2CH$-H	145	$BrCH_2$-H	152
	161	$(CH_3O)_2CH$-H	162
	134		180
	137		137
	150		150

^{1}H-^{13}C

Type	J (Hz)	Type	J (Hz)
=C-H	157	C=C=C-H	168
	172		195
	200		195
X=Halogen	~198		238
	170		160
	202		189
	182		159
	178		

Figure 3. Coupling constant value (J)

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2.2.2 ^{13}C-NMR Spectra

The steps for interpreting the ^{13}C-NMR spectrum of a compound are almost the same as determining the ^{1}H-NMR spectrum and are described as follows:

1) **Step 1**: Identify the number of signals that appear on the ^{13}C-NMR spectrum. The number of carbon atoms (C) can be determined by looking at the number of peaks that appear and the chemical environment of the carbon in the compound. Just like ^{1}H-NMR, carbon atoms that are in a chemical environment appear as the same peak. For example, the pentane-2,4-dione compound shown in **Figure 4** has five carbon atoms with two of them in the same chemical environment giving rise to three peaks of the ^{13}C-NMR spectrum.

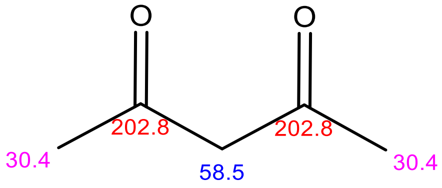
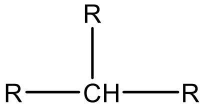
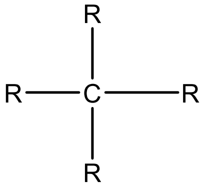
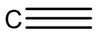
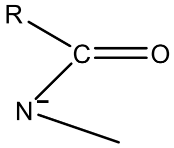
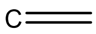
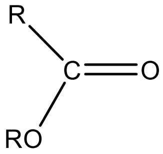
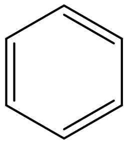
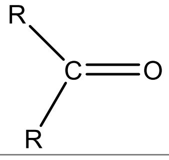


Figure 4. Structure of pentane-2,4-dione

2) **Step 2:** Identify the chemical shift value (δ) that appears in the ^{13}C-NMR spectrum. To predict the chemical shear value that appear in the ^{13}C-NMR spectrum, you can use the references in **Table 2**.

Table 2 Chemical shift ^{13}C-NMR

Type of Carbon	Approximate Chemical Shift (ppm)	Type of Carbon	Approximate Chemical Shift (ppm)
$(CH_3)_4Si$	0	C-I	0 – 40
$R\text{-}CH_3$	8 – 35	C-Br	25 – 65
$R\text{-}CH_2\text{-}R$	15 – 50	C-Cl	35 – 80
	20 – 60	C-N	40 – 60
	30 – 40	C-O	50 – 80
	65 – 85		165 – 175
	100 – 150		165 – 175
	110 – 170		205 – 220

3) **Step 3:** The structure of an organic compound can be determined by combining the results of the analysis based on steps 1 and 2. First, after the amount of carbon and its chemical environment are known, the molecular formula of a compound can be determined. Then, the chemical shift values are compared with the chemical shift table values (Table 2) and the pieces of the molecules are combined to form a structural formula for the compound.

3. EXPERIMENTAL METHOD

To understand how to read and interpret the ^{1}H-NMR and ^{13}C-NMR spectra, this paper explained step by step reading the ^{1}H-NMR and ^{13}C-NMR spectra through step-by-step exposure in determining simple compounds, fairly complex compounds, more complex compounds, and very complex compounds. The simple compound used are: methane, methanol, acetylene, n-octane, and iso-butane. The fairly complex compound used are: toluene and naphtalene. The more complex compound used are: eugenol (Thirukumaran et al., 2014). The very complex compound used are: $L_1 – L_4$ ligands (Bouroumane et al., 2021).

Thirukumaran et al. (2014) conducted NMR spectroscopic analysis of eugenol compounds extracted from clove plants. NMR spectroscopy was performed using a Joel spectrometer with tetramethylsilane (TMS) as the internal standard and samples were prepared using $CDCl_3$ and (Bouroumane et al., 2021) synthesize N-alkylated pyrazolyl compound (L_1-L_4) via one-step process by condensation of (3,5-dimethyl-1Hpyrazol-1-yl) methanol A with a appropriate primary amines using DMSO solvent system and the structure of L_1-L_4 compound are shown in **Figure 5**.

4. RESULTS AND DISCUSSION

Determination of the structure of an organic compound can be done by analyzing the resulting ^{1}H-NMR and ^{13}C-NMR spectral patterns. NMR spectrum analysis can be performed based on the step-by-step analysis described in section 2.2. Analysis was carried out on the ^{1}H-NMR spectrum to determine the number and type of H atoms present in the structure of the compound. Then, the analysis of the ^{13}C-NMR spectrum was carried out to obtain information regarding the number and type of carbon atom bonds. Thus, the structure of an organic compound can be determined by combining the results of ^{1}H-NMR and ^{13}C-NMR spectrum analysis.

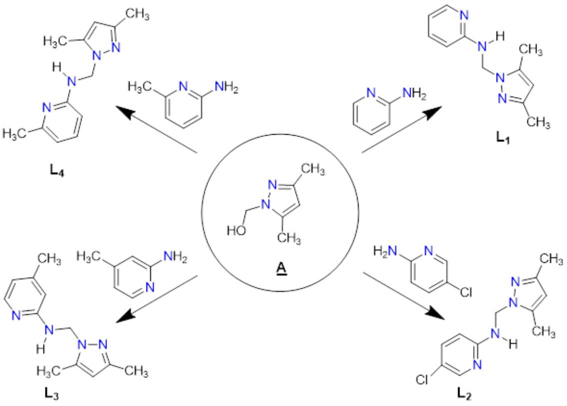
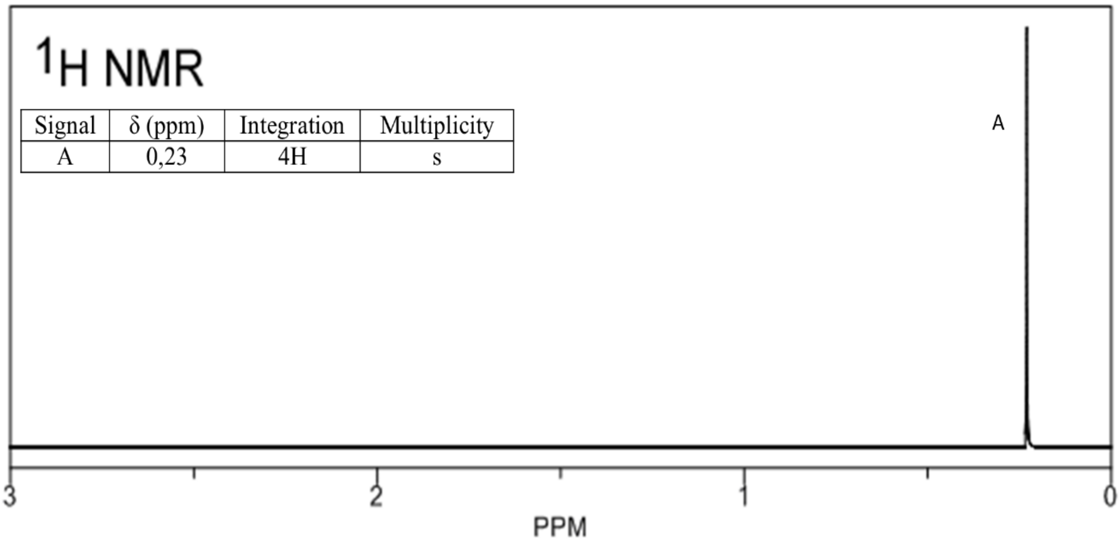


Figure 5. Structure of prepared mono-alkylated pyrazole **L_1-L_4** (Bouroumane et al., 2021)

4.1. NMR Analysis of Simple Compound

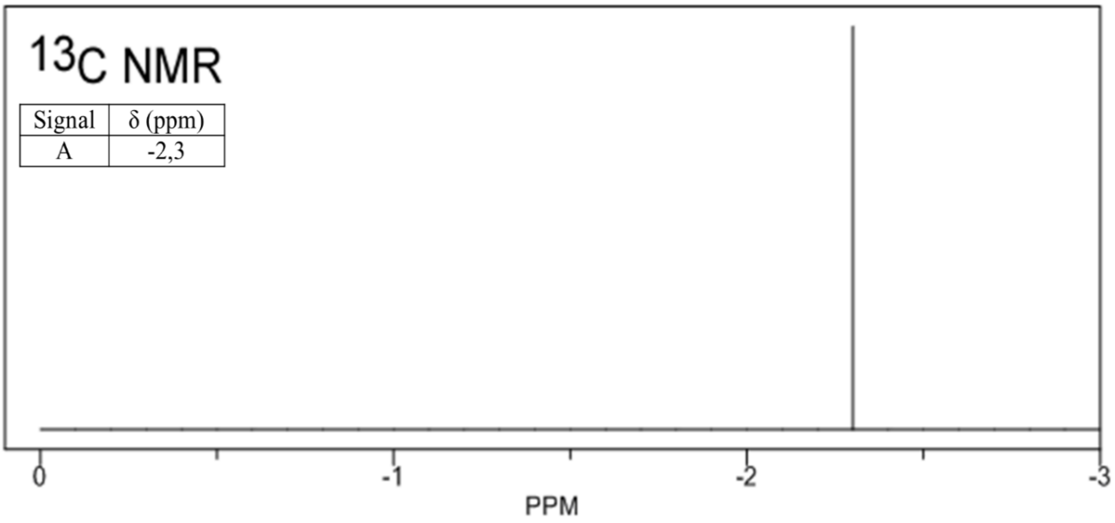
4.1.1. NMR Spectra of Methane

^{1}H-NMR and ^{13}C-NMR spectra of methane are shown in **Figures 6 and 7**, respectively. Based on a step-by-step analysis of how to read and interpret the ^{1}H-NMR and ^{13}C-NMR spectrum, the interpretation is described in the following.



Signal	δ (ppm)	Integration	Multiplicity
A	0,23	4H	s

Figure 6. Methane ^1H-NMR Spectra
(https://scilearn.sydney.edu.au/OrganicSpectroscopy/?type=NMR&page=Examples, retrieved on 29 October 2020)



Signal	δ (ppm)
A	-2,3

Figure 7 Methane ^{13}C-NMR spectra
(https://scilearn.sydney.edu.au/OrganicSpectroscopy/?type=NMR&page=Examples, retrieved on 29 October 2020)

4.1.1.1. ^1H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 6** presents one peak (A), indicating a signal originating from one type of proton.

2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 6** shows signal (A) appears as a singlet peak, indicating the absence of a proton in the neighboring C atom.

3) **Step 3:** Identify the signal integration value. **Figure 6** shows the peak (A) appears with the integration value for four H atoms, showing that the four H atoms are in the same chemical environment. Thus, they appear as one peak.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, **Figure 6** displays the peaks appear at 0.23 ppm close to the chemical shift value of the methyl R-CH_3 (0.9 ppm). However, in step 3 it is explained that the peaks have integration for four H atoms, so that there is no inductive effect or bond anisotropy effect, causing the peaks to appear at a smaller chemical shift (0.23 ppm).

4.1.1.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. **Figure 7** shows one peak that appears (A) which indicates a signal originating from one type of carbon.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, **Figure 7** shows the peaks appear at -2.3 ppm related to ^{13}C sp^3.

The ^{1}H-NMR analysis shows the signal appears as a singlet peak with integration values for four H atoms and appears at 0.23 ppm, whereas the ^{13}C-NMR analysis showed that there was one peak indicating the presence of one carbon atom and appeared at -2.3 ppm referring to the C sp^3 atom. Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes in **Figures 6 and 7** are combined, a CH_4 (methane) compound is formed.

4.1.2. NMR Spectra of Methanol

^{1}H-NMR and ^{13}C-NMR spectra of methanol are shown in **Figures 8 and 9**, respectively.

4.1.2.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 8** displays two peaks that appears (A) and (B) which indicates a signal originating from two types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 8** shows signal (A) and (B) appears as a singlet peak, respectively indicating the absence of a proton in the neighboring C atom.
3) **Step 3:** Identify the signal integration value. **Figure 8** shows the peak (A) appears with the integration value for three H atoms, meanwhile peak (B) appears with the integration for one H atom.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, the value of the proton chemical shift can be determined. **Figure 8** shows the peak (A) appear at 3,48 ppm to 3 × ^{1}H on *sp*^{3} CH_3 group bonded to the electronegative OH group and peak (B) appear at 4.3 ppm related to ^{1}H on OH group as a broad peak caused by the ^{1}H rapidly exchanges with ^{1}H atoms in the solvent.

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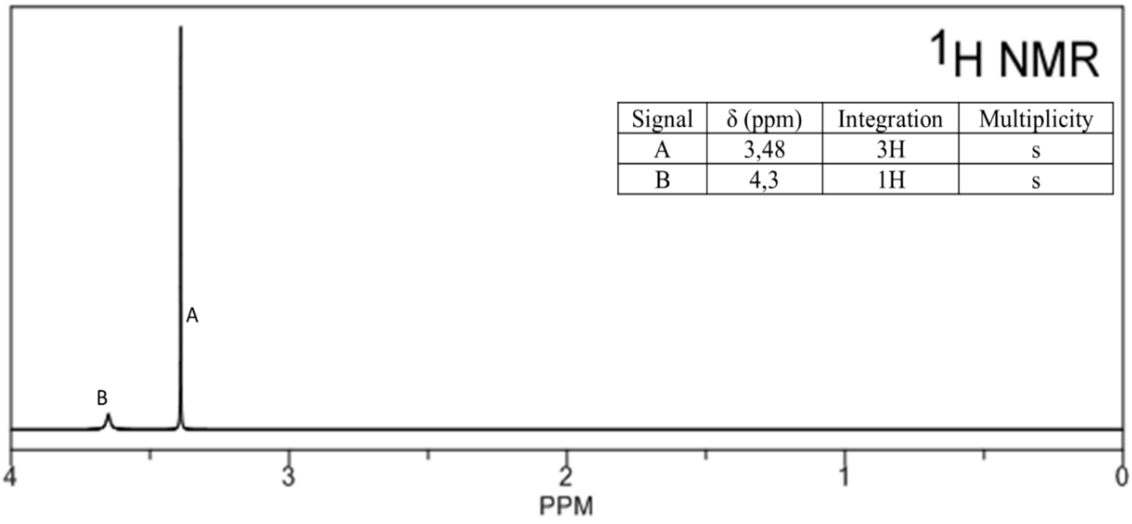
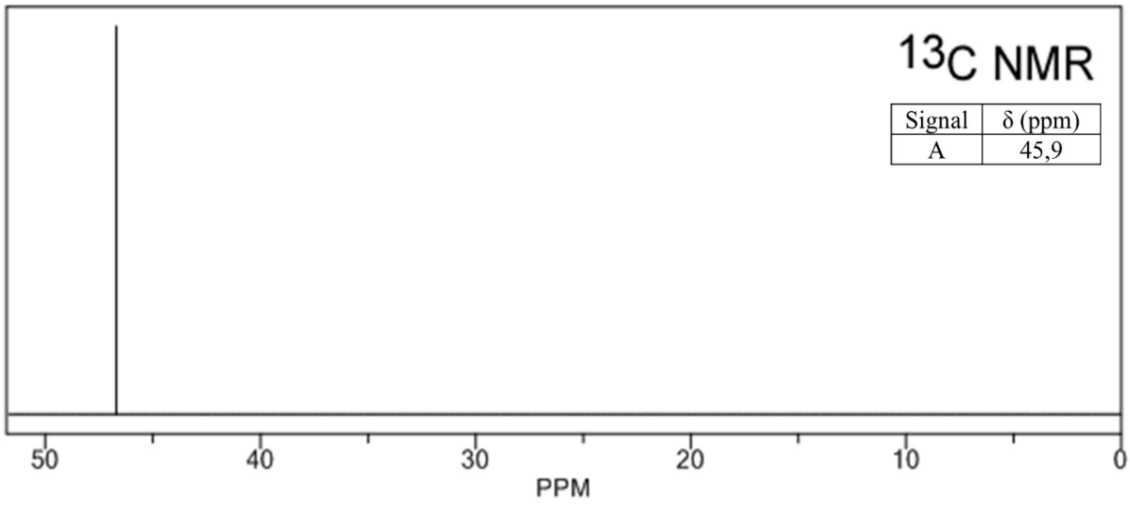


Signal	δ (ppm)	Integration	Multiplicity
A	3,48	3H	s
B	4,3	1H	s

Figure 8 Methanol ^{1}H-NMR Spectra
(https://scilearn.sydney.edu.au/OrganicSpectroscopy/?type=NMR&page=Examples, retrieved on 29 October 2020)



Signal	δ (ppm)
A	45,9

Figure 9 Methanol ^{13}C-NMR Spectra
(https://scilearn.sydney.edu.au/OrganicSpectroscopy/?type=NMR&page=Examples, retrieved on 29 October 2020)

4.1.2.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. **Figure 9** shows one peak that appears (A) which indicates a signal originating from one type of carbon.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, **Figure 8** shows the peaks appear at 45.9 ppm related to ^{13}C on sp^3 CH_3 group bonded to the electronegative OH group.

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 8 and 9**, respectively, the ^{1}H-NMR analysis shows the signal appears as two peaks (A) and (B) at 3.48 and 4.4 ppm. Meanwhile, the ^{13}C-NMR analysis showed that there was one peak at 45.9 ppm indicating ^{13}C on sp^3 CH_3 group bonded to the electronegative OH group. Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes in **Figures 8 and 9** are combined, a CH_3OH (methanol) compound is formed.

4.1.3. NMR Spectra of Acetylene

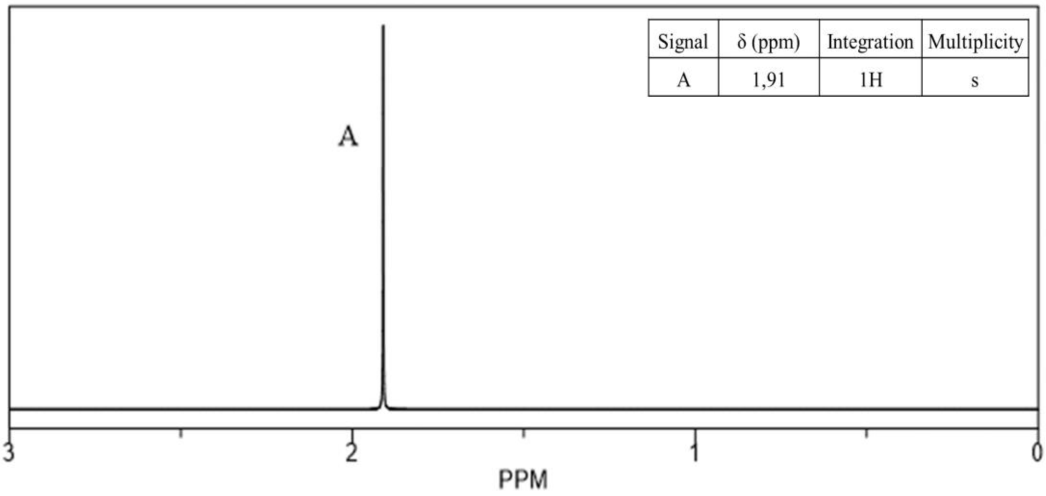
^{1}H-NMR and ^{13}C-NMR spectra of acetylene are shown in **Figures 10 and 11**, respectively.

4.1.3.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 10** shows one peak that appears (A) which indicates a signal originating from one type of proton.
2) **Step 2:** Identify the multiplicity of signals. **Figure 10** presents signal (A) appears as a singlet peak indicating the absence of a proton in the neighboring C atom.
3) **Step 3:** Identify the signal integration value. **Figure 10** shows the peak (A) appears with the integration value for one H atom.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, the value of the proton chemical shift can be determined. **Figure 8** shows the peak appears at 1.91 ppm. The C-H bond generally occurs at 0.2 – 0.9 ppm. The peak that appears for the CH bond at greater chemical shift (1.91 ppm) appears due to the anisotropy effect of the triple bond between two carbon atoms -C≡C-.

4.1.3.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. **Figure 11** shows one peak that appears (A), which indicates a signal originating from one type of carbon.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, the value of the carbon chemical shift can be determined. **Figure 11** shows the peaks appear at 71.9 ppm related to ^{13}C sp (C≡C).



Signal	δ (ppm)	Integration	Multiplicity
A	1,91	1H	s

Figure 10 Acetylene ^{1}H-NMR Spectra

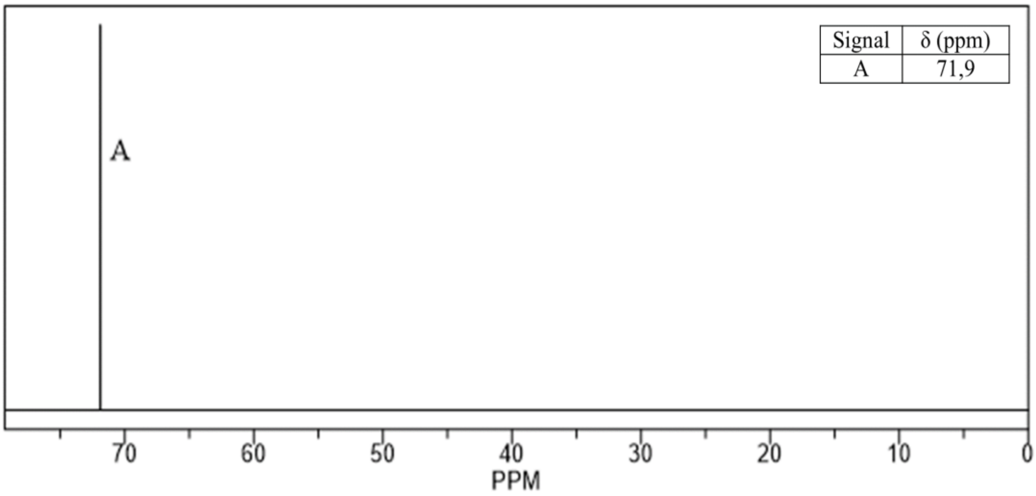


Figure 11 Acetylene ^{13}C-NMR Spectra

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 10 and 11**, respectively, the ^{1}H-NMR analysis shows the signal appears as a singlet peak with an integration value for one H atoms and appears at 1.91 ppm. Meanwhile, the ^{13}C-NMR analysis showed that there was a peak indicating the presence of one type of carbon atom and appeared at 71.9 ppm referring to the C sp atom (C≡C). Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes in **Figures 8 and 9** are combined, a C_2H_2 (acetylene) compound is formed and its structure shown in **Figure 12**.

$HC\equiv CH$

Figure 12 Structure of Acetylene (Voronin et al., 2018)

4.1.4. NMR Spectra of n-Octane

^{1}H-NMR and ^{13}C-NMR spectra of n-octane are shown in **Figures 13 and 14**, respectively.

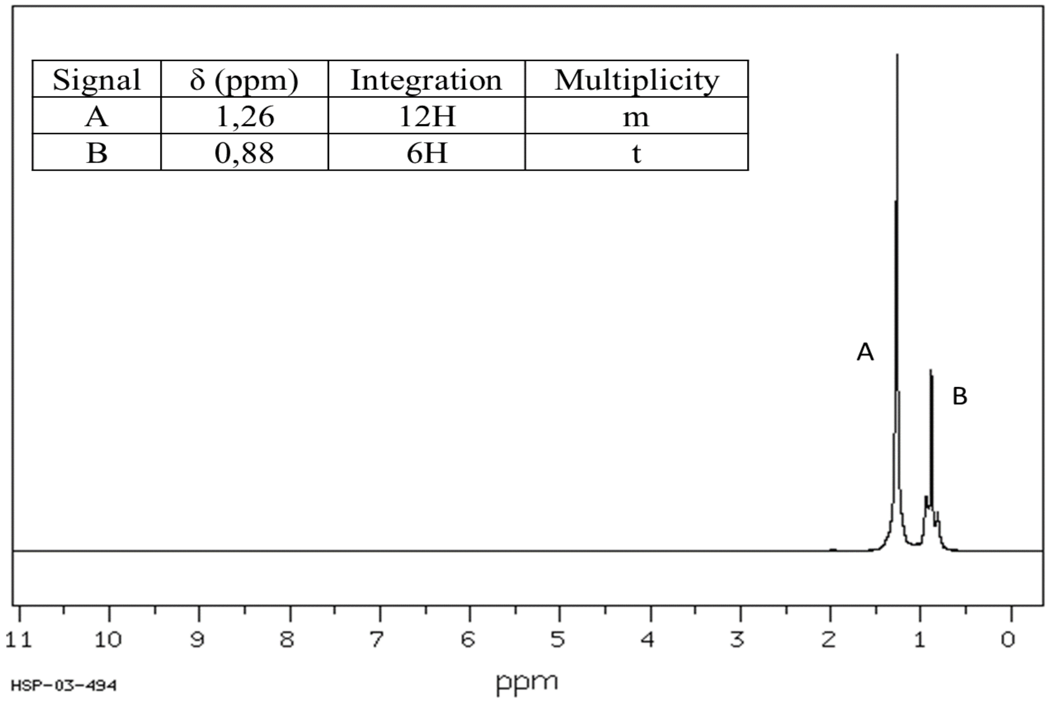
4.1.4.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 13** shows two peaks that appears (A) and (B) which indicates a signal originating from two types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 13** shows signal (A) appears as a multiplet peak indicates the presence of several protons on the neighboring carbon and signal (B) appears as a triplet peak indicates the presence of two protons on the neighboring carbon.
3) **Step 3:** Identify the signal integration value. **Figure 13** shows the peak (A) appears with the integration value for twelve H atom and peak (B) appears with the integration value for six H atom.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in Table 1, the value of the proton chemical shift can be determined. **Figure 13** shows the peak (A) appears at 1.26 ppm related to the chemical shift value of the methylene (R-CH_2-R) and peak (B) appears at 0.88 ppm related to the chemical shift value of the methyl (R-CH_3).

4.1.4.2. ^{13}C-NMR Spectra Analysis

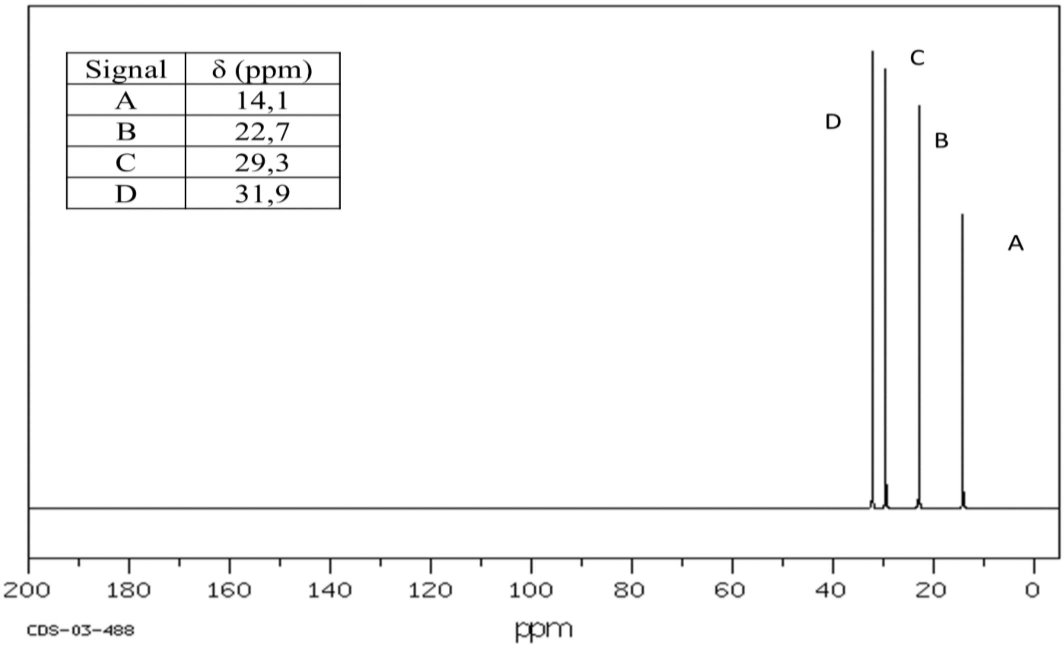
1) **Step 1:** Identify the number of signals. **Figure 14** shows four peaks that appears (A, B, C, D), indicating a signal originating from four types of carbon.

2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, **Figure 14** shows the peak (A) at 14.1 ppm related to ^{13}C sp^3 methyl (-CH_3), peak (B, C, and D) appeared at 22.7; 29.3; and 31.9 ppm, respectively, relating to ^{13}C sp^3 methylene (-CH_2-).



Signal	δ (ppm)	Integration	Multiplicity
A	1,26	12H	m
B	0,88	6H	t

Figure 13 n-octane ^{1}H-NMR Spectra (https://www.chemicalbook.com/SpectrumEN_111-65-9_13cnmr.htm, retrieved on 29 October 2020)



Signal	δ (ppm)
A	14,1
B	22,7
C	29,3
D	31,9

Figure 14 n-octane ^{13}C-NMR Spectra (https://www.chemicalbook.com/SpectrumEN_111-65-9_13cnmr.htm, retrieved on 29 October 2020)

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 13 and 14**, respectively, the ^{1}H-NMR analysis shows the signal (A) appears as a multiplet peak with an integration value for twelve H atoms and appears at 1.26 ppm ($R-CH_2-R$). Peak (A) indicates the presence of 6 methylene $–(CH_2)_6–$. Whereas signal (B) appears as a triplet peak indicating the presence of two protons on the neighboring carbon with integration values for six H atoms and appears at 0.88 ppm ($R-CH_3$). Peak (B) indicates the presence of methyl bound to methylene (CH_3-CH_2-R). Meanwhile, the ^{13}C-NMR spectrum shows the appearance of four peaks (A, B, C, and D), each of which indicates the presence of ^{13}C sp^3 bonds for methyl ($-CH_3$) and methylene ($-CH_2-$). Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes are combined, an n-octane compound (C_8H_{18}) is formed and its structure is illustrated in **Figure 15**.

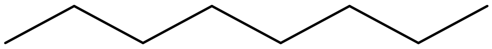


Figure 15 n-octane structure (https://pubchem.ncbi.nlm.nih.gov/compound/octane#section=Information-Sources, retrieved on 29 October 2020)

4.1.5. NMR Spectra of Isobutene

^{1}H-NMR and ^{13}C-NMR spectra of isobutene are shown in **Figures 16 and 17**, respectively. Based on a step-by-step analysis of how to read and interpret the ^{1}H-NMR and ^{13}C-NMR spectrum, the following conclusions are:

4.1.5.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 16** shows two peaks that appears (A) and (B) which indicates a signal originating from two types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 16** shows signal (A) appears as a doublet peak indicates the presence of one proton on the neighboring carbon and signal (B) appears as a multiplet peak indicates the presence of several protons on the neighboring carbon.
3) **Step 3:** Identify the signal integration value. **Figure 16** shows the peak (A) appears with the integration value for nine H atom and peak (B) appears with the integration value for one H atom.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, **Figure 16** shows the peak (A) at 0.89 ppm related to the chemical shift value of the methyl ($R-CH_3$) and peak (B) appears at 1.74 ppm related to the chemical shift value for methyne (R_3C-H).

4.1.5.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. **Figure 17** shows four peaks appearing (A) and (B), indicating a signal originating from two types of carbon.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, **Figure 17** shows the peak (A) appeared at 24.3 ppm, relating to ^{13}C sp^3 methyl ($-CH_3$) and peak (B) at 25 ppm related to C sp^3 methyne (-CH-).

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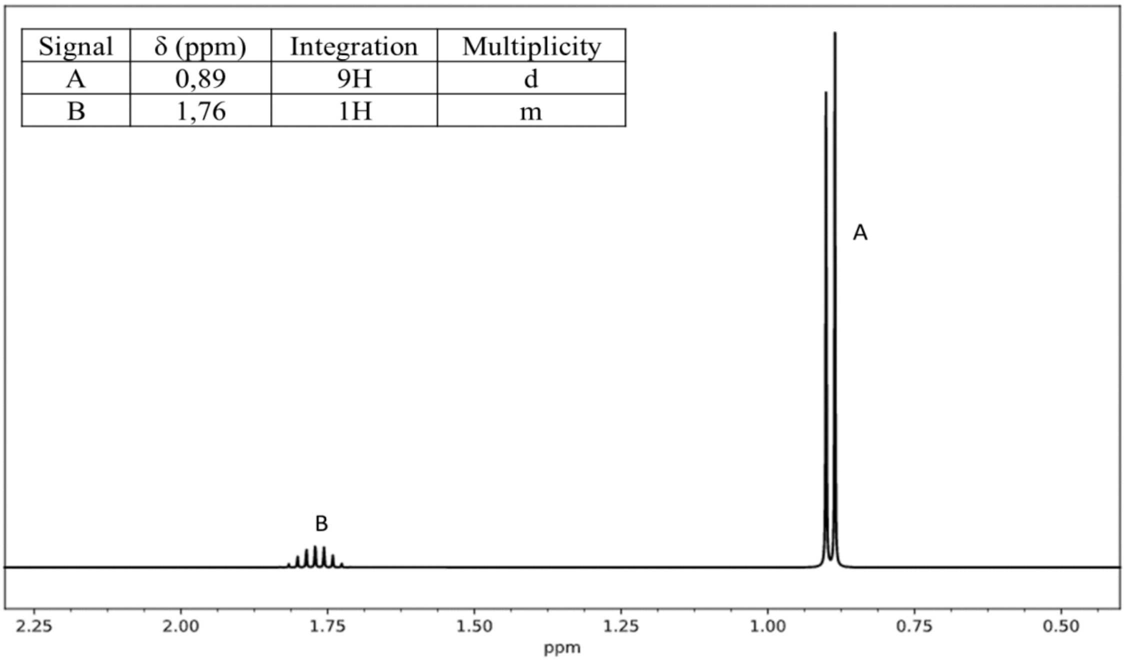
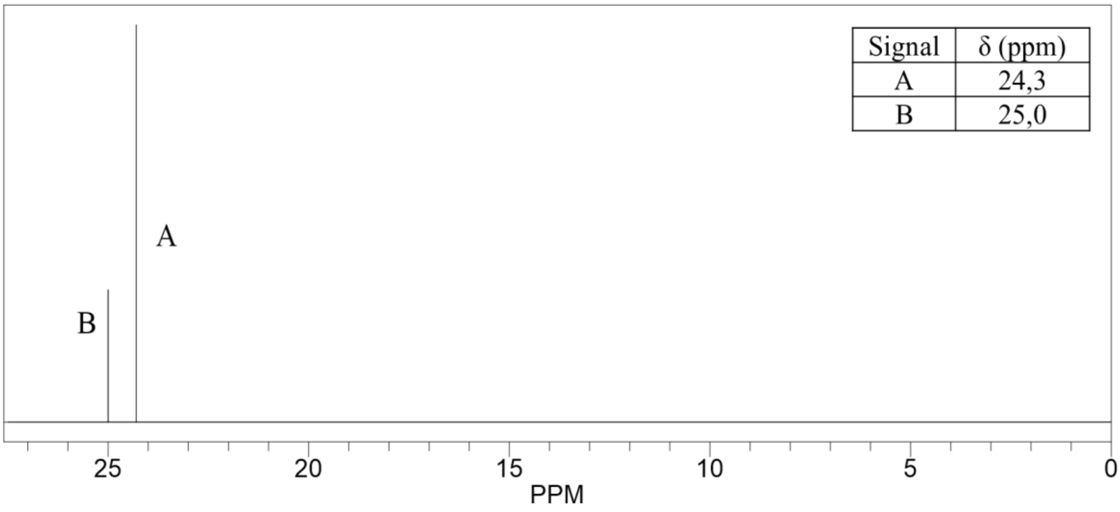


Signal	δ (ppm)	Integration	Multiplicity
A	0,89	9H	d
B	1,76	1H	m

Figure 16 Isobutane ^{1}H-NMR Spectra (Abraham & Mobli, 2008)



Signal	δ (ppm)
A	24,3
B	25,0

Figure 17 Isobutane ^{13}C-NMR Spectra

Based on analysis in **Figures 16 and 17**, respectively, the ^{1}H-NMR analysis shows the signal (A) appears as a doublet peak indicating the presence of one proton on the neighboring carbon with an integration value for nine H atoms and appears at 0,89 ppm (R-CH_3). It can be concluded that peak (A) indicates the presence of 3 methyl 3(CH_3), whereas signal (B) appears as a multiplet peak indicating the presence of several protons on the neighboring carbon with integration values for one H atom and appears at 1.74 ppm (R_3C-H). Peak (B) indicates the presence of methyne bound to three methyl (CH_3). Meanwhile, the ^{13}C-NMR spectrum shows the appearance of two peaks (A) and (B), indicating the presence of ^{13}C sp^3 bonds for methyl (-CH_3) and methyne (-CH-). Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes are combined, an isobutane compound (C_4H_{10}) is formed (see **Figure 18**).

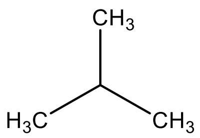


Figure 18 Structure of isobutane (https://www.ebi.ac.uk/chebi/searchId.do?chebiId=43907#:~:text=Isobutylene%20(or%202%2Dmethylpropene),is%20of%20considerable%20industrial%20value, retrieved on 29 October 2020)

4.2. NMR Analysis of Fairly Complex Compound

4.2.1. NMR Spectra of Benzene

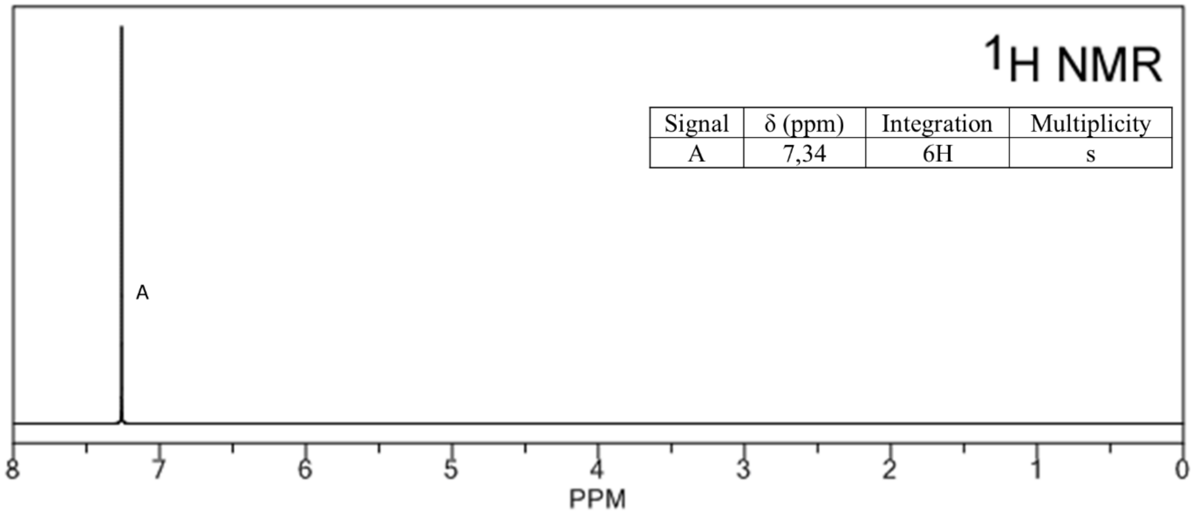
^{1}H-NMR and ^{13}C-NMR spectra of benzene are shown in **Figures 19 and 20**, respectively.

4.2.1.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 19** shows one peak that appears (A) which indicates a signal originating from one type of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 19** shows signal (A) appears as a singlet peak indicating the absence of a proton in the neighboring C atom. Signal is not split by coupling as all ^{1}H environments are the same.
3) **Step 3:** Identify the signal integration value. **Figure 19** shows the peak (A) appears with the integration value for six H atoms. All ^{1}H environments are the same causes the signal to appear as one peak.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, the value of the proton chemical shift can be determined. **Figure 19** shows the peak (A) appear at 7.34 ppm related to 6×^{1}H on sp^2 CH groups.

4.2.1.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. **Figure 20** shows one peak that appears (A) which indicates a signal originating from one type of carbon.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, the value of the carbon chemical shift can be determined. **Figure 20** shows the peaks appear at 128.5 ppm related to ^{13}C on sp^2 CH groups.



Signal	δ (ppm)	Integration	Multiplicity
A	7,34	6H	s

Figure 19 Benzene ^{1}H-NMR Spectra (https://scilearn.sydney.edu.au/OrganicSpectroscopy/?type=NMR&page=Examples, retrieved on 29 October 2020)

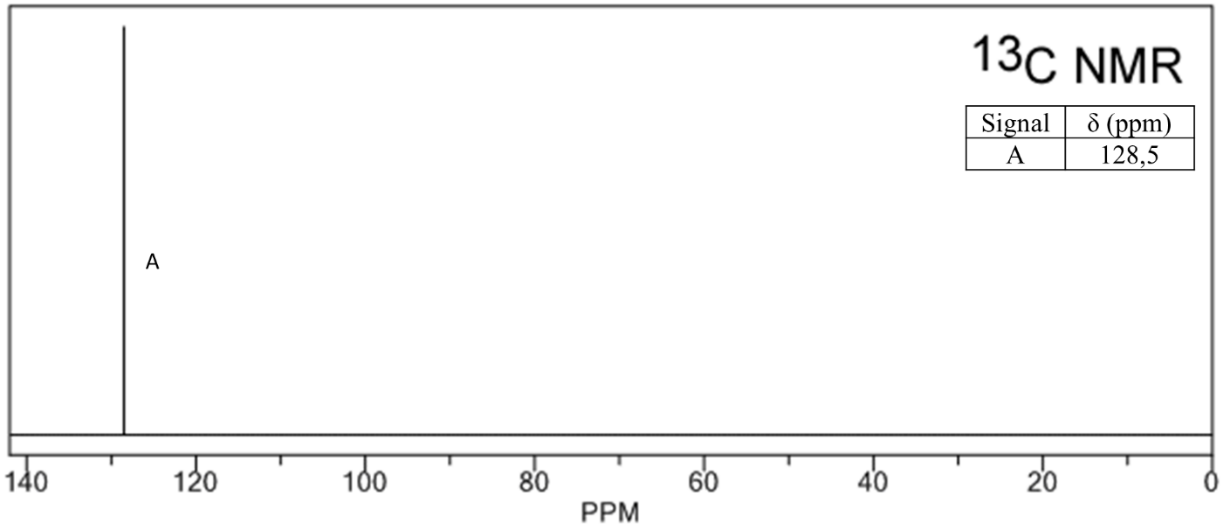


Figure 20 Benzene ^{13}C-NMR Spectra (https://scilearn.sydney.edu.au/OrganicSpectroscopy/?type=NMR&page=Examples, retrieved on 29 October 2020)

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 19 and 20**, respectively, the ^{1}H-NMR analysis shows the signal appears as one peak at 7.34 ppm with integration for six H atoms. The signal is not split by coupling due to all ^{1}H environments are the same. Meanwhile, the ^{13}C-NMR analysis showed that there was one peak at 128.5 ppm indicating ^{13}C on sp^{2} CH groups. Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes in **Figures 19 and 20** are combined, a C_6H_6 (benzene) compound is formed and its structure shown in **Figure 21**.

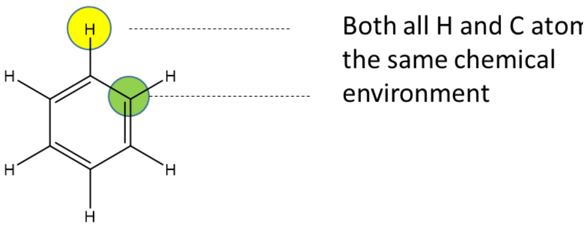


Figure 21 Structure of benzene

4.2.2. NMR Spectra of Naphtalene

^{1}H-NMR and ^{13}C-NMR spectra of naphthalene are shown in **Figures 22 and 23**, respectively.

4.2.2.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 22** shows two peaks that appears (A) and (B) which indicates a signal originating from two types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 22** shows both of signal (A) and (B) appears as a multiplet peak indicating presence of several protons in the neighboring C atom.
3) **Step 3:** Identify the signal integration value. **Figure 22** shows both of peak (A) and (B) appears with the integration value for four H atoms.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, the value of the proton chemical shift can be determined. **Figure 22** shows the peak (A) appear at 7.32 ppm related to 4 × ^{1}H on sp^{2} CH groups. Similar to peak (A), peak (B) appears at 7.67 ppm related to 4 × ^{1}H on sp^{2} CH groups.

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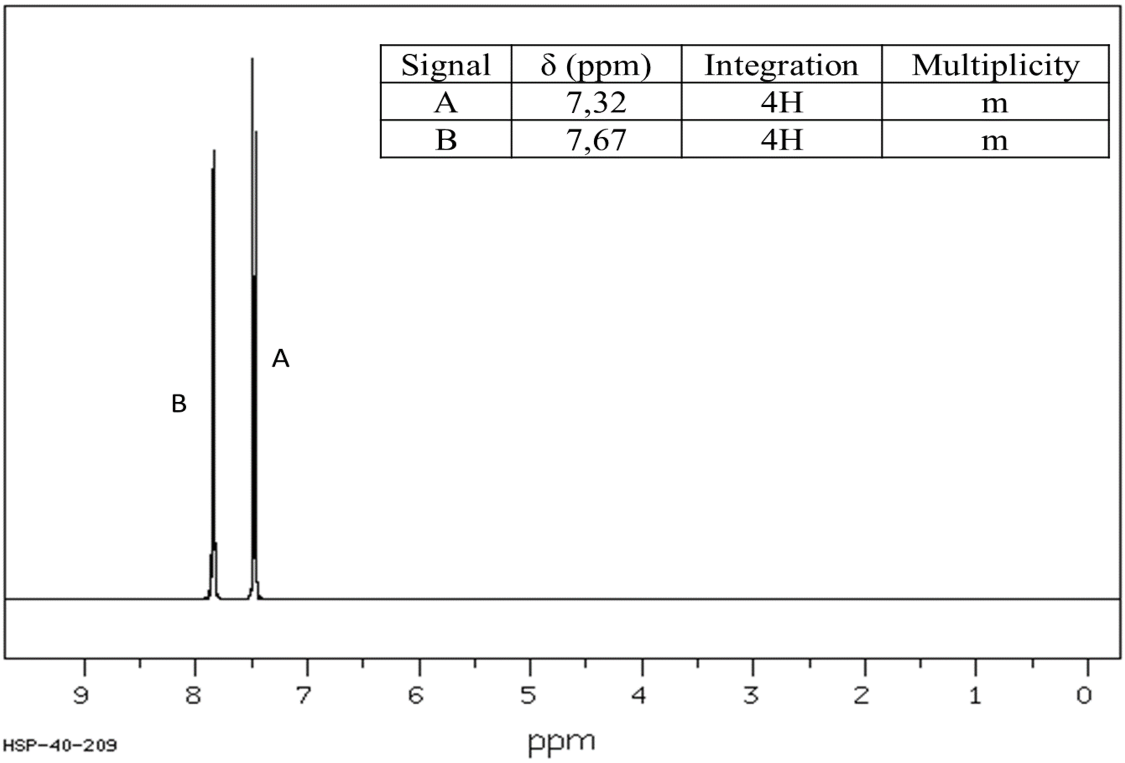
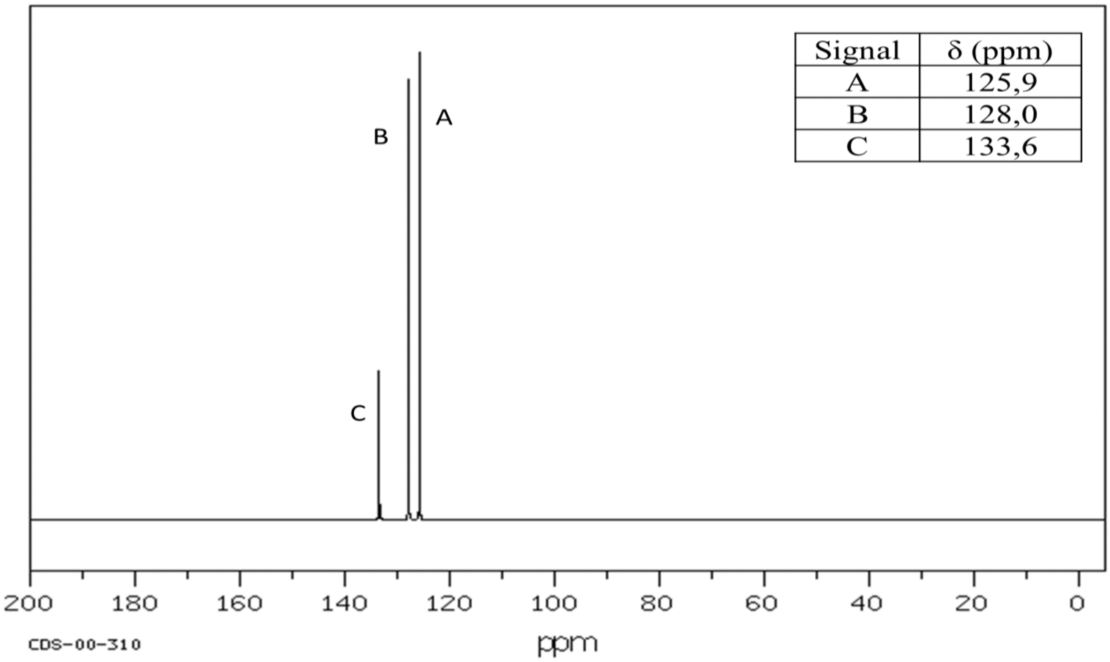


Signal	δ (ppm)	Integration	Multiplicity
A	7,32	4H	m
B	7,67	4H	m

Figure 22 Naphthalene ^{1}H-NMR Spectra
(https://www.chemicalbook.com/SpectrumEN_111-65-9_13cnmr.htm, retrieved on 29 October 2020)



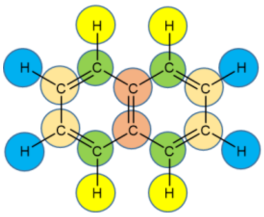
Signal	δ (ppm)
A	125,9
B	128,0
C	133,6

Figure 23 Naphthalene ^{13}C-NMR Spectra
(https://www.chemicalbook.com/SpectrumEN_111-65-9_13cnmr.htm, retrieved on 29 October 2020)

4.2.2.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. **Figure 23** shows three peaks that appears (A), (B), and (C) indicating the signal from three types of carbon.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, **Figure 23** shows the peak (A), (B), and (C) at 125.9; 128.0; and 133 ppm respectively. All peaks related to ^{13}C on sp^{2} CH groups.

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 22 and 23**, respectively, the ^{1}H-NMR analysis shows the signal appears as two peaks at 7.32 and 7.67 ppm with both of integration for fours H atoms. Meanwhile, the ^{13}C-NMR analysis showed that there was three peaks at 125.9; 128.0; and 133 ppm respectively indicating ^{13}C on sp^{2} CH groups. Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes in **Figures 22 and 23** are combined, a $C_{10}H_{8}$ (naphthalene) compound is formed and its structure shown in **Figure 24**.



* Atoms of the same color indicate the same chemical environment

Figure 24 Structure of naphthalene

4.3. NMR Analysis of More Complex Compound

4.3.1. NMR Spectra of Eugenol

^{1}H-NMR and ^{13}C-NMR spectra of eugenol are shown in **Figures 25 and 26**, respectively. Based on a step-by-step analysis of how to read and interpret the ^{1}H-NMR and ^{13}C-NMR spectrum, the following conclusions are:

4.3.1.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 25** shows eight peaks that appears which indicates a signal originating from eight types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 25** presents signal (a), (b), and (c) appears as a singlet peak indicating absence of protons in the neighboring C atom. Meanwhile signal (d), (g), and (h) appears as a doublet peak indicating presence of one protons in the neighboring C atom and signal (e) appears as a multiplet peak peak indicating presence of several protons in the neighboring C atom. Interestingly, signal (f) appears as a double doublet peak which indicates the presence of a proton that is attached to the same C atom, but has a very slightly different chemical shift value.
3) **Step 3:** Identify the signal integration value. **Figure 25** shows peak (a), (c), (e), (g), and (h) appears with the integration value for one H atom, peak (d) and (f) appears with the integration value for two H atoms, and peak (b) appears with the integration value for three H atoms.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ).

Based on the reference data in **Table 1**, **Figure 25** shows the singlet peak at 3.82 ppm is due to the −OCH3 protons, the doublets at 3.21 and 5.05 ppm as well as the multiplet at 5.95 ppm are assigned to the allyl protons [$-CH_2-CH=CH_2-$], the peak that appear at 5.66 ppm related to −OH protons, and the peak that appears in the range of 6.5 to 7.0 ppm is related to aromatic protons. The peak at 7.2 ppm is due to the solvent peak ($CDCl_3$).

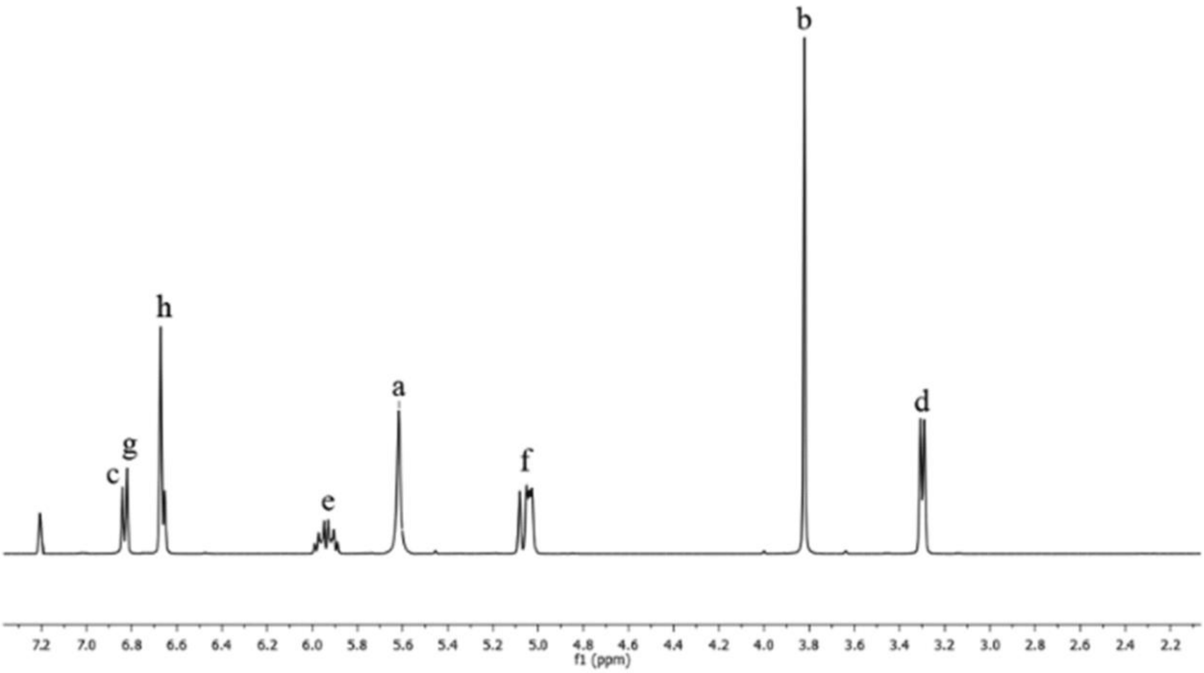


Figure 25 Eugenol ^{1}H-NMR spectrum analysis (Thirukumaran et al., 2014)

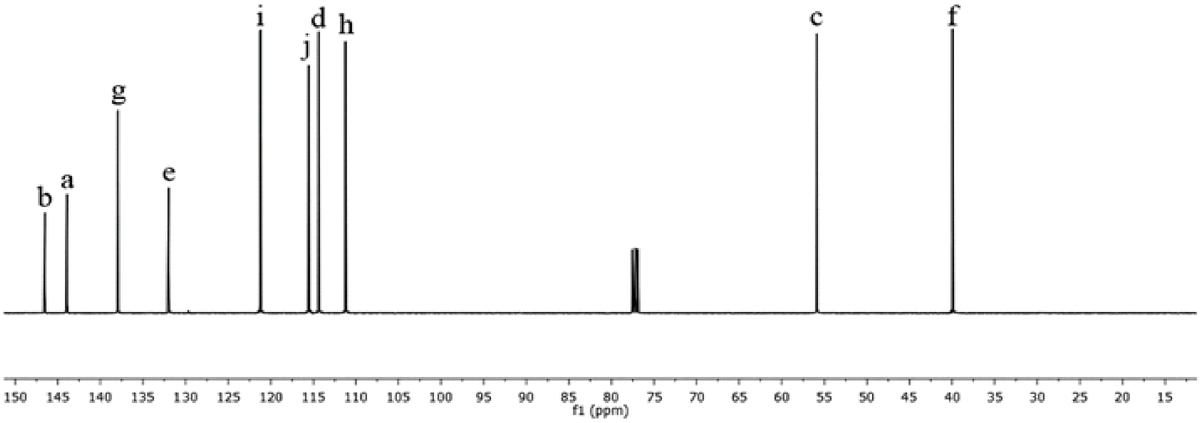


Figure 26 Eugenol ^{13}C-NMR spectrum analysis (Thirukumaran et al., 2014)

4.3.1.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. **Figure 26** shows nine peaks that appears which indicates a signal originating from nine types of carbon.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, the value of the carbon chemical shift can be determined. **Figure 26** shows the peak at 56 ppm (methoxy carbons ($-OCH_3$)), as well as 115; 137; and 40 ppm (alkyl carbons [$-CH_2-CH=CH_2$]), and the peak that appears at around 120−150 ppm is related to aromatic carbons.

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 25 and 26**, respectively, the ^{1}H-NMR analysis shows the signal appears as eight peaks each representing a different type of proton bond, such as $-OCH_3$ protons, −OH proton, allyl protons [$-CH_2-CH=CH_2-$], and aromatic protons. Similar to ^{1}H-NMR analysis, the ^{13}C-NMR analysis showed that there was nine peaks each representing a different type of carbon bond, such as methoxy carbons ($-OCH_3$) at 56 ppm, allyl carbons [$-CH_2-CH=CH_2$] at 115 ppm, 137 ppm, and 40 ppm, and aromatic carbon around 120−150 ppm. Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes in **Figures 25 and 26** are combined, a $C_{10}H_{12}O_2$ (eugenol) compound is formed and its structure presented in **Figure 27**.

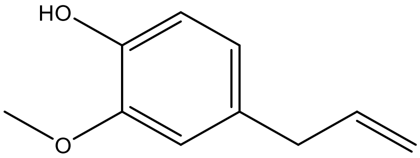


Figure 27 Structure of eugenol (Thirukumaran et al., 2014)

4.4. NMR Analysis of Very Complex Compound

4.4.1. NMR Spectra of N-((3,5-dimethyl-1H-pyrazol-1-yl)methyl)pyridin-2-amine (L_1 Ligand)

^{1}H-NMR and ^{13}C-NMR spectra of L_1 ligand are shown in **Figures 28 and 29**, respectively.

4.4.1.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 28** shows nine peaks that appears which indicates a signal originating from nine types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 28** shows the signals appear with varying multiplicities, indicating the presence of multiple protons in neighboring C atoms.
3) **Step 3:** Identify the signal integration value. **Figure 28** displays several proton integrations, ranging from 1H, 2H, and 3H.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, the value of the proton chemical shift can be determined. **Figure 28** shows the peaks at 8.02; 7.41; 6,63; and 6.56 ppm related to aromatic protons (^{1}H on sp^{2} CH groups), peak at 7,57 ppm is related to proton from amine groups (N-H), peak at 5.54 ppm is related to ^{1}H on sp^{3} CH_2 groups and bonded to electronegative atoms, peak at 5.74 ppm is related to aliphatic protons (^{1}H on sp^{2} CH groups), and peaks at 2.36 and 2.07 ppm related to ^{1}H on sp^{3} CH_3 groups.

4.4.1.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. From the analysis of the ^{13}C-NMR spectrum of the ligand L_1 compound, it was shown that eleven peaks appeared indicating the existence of eleven types of carbon atoms.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, the value of the carbon chemical shift can be determined. **Figure 29** shows that the peaks that appear at 157.6; 146.4; and 139.3 ppm related to ^{13}C on sp^{2} bond with electronegative atom such as N, peaks at 147.7; 137.5; 113.4; and 105.1 related to ^{13}C on sp^{2} CH, peaks at 13.8 and 11.2 ppm related to ^{13}C on sp^{3} methyl (CH_3), and peak at 53.9 ppm related to ^{13}C on sp^{3} CH_2 bonded to electronegative atom.

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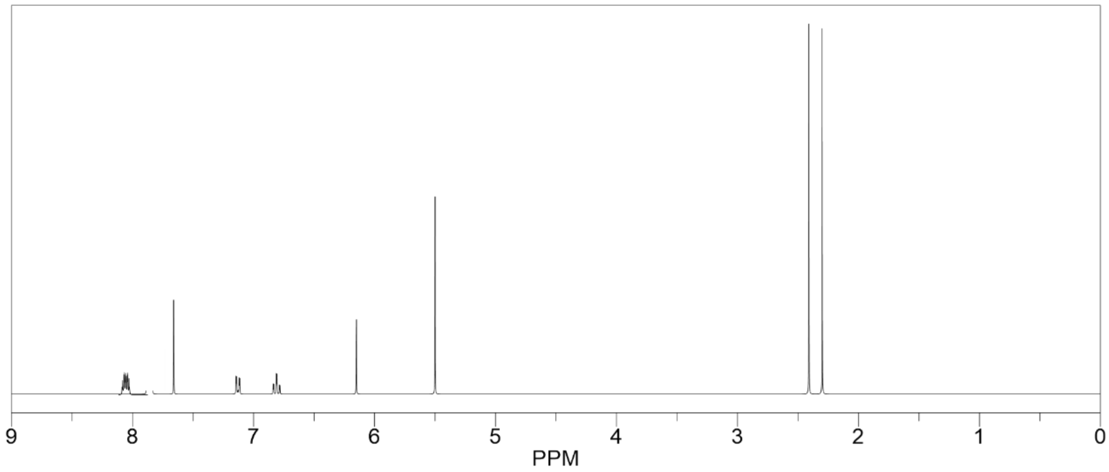
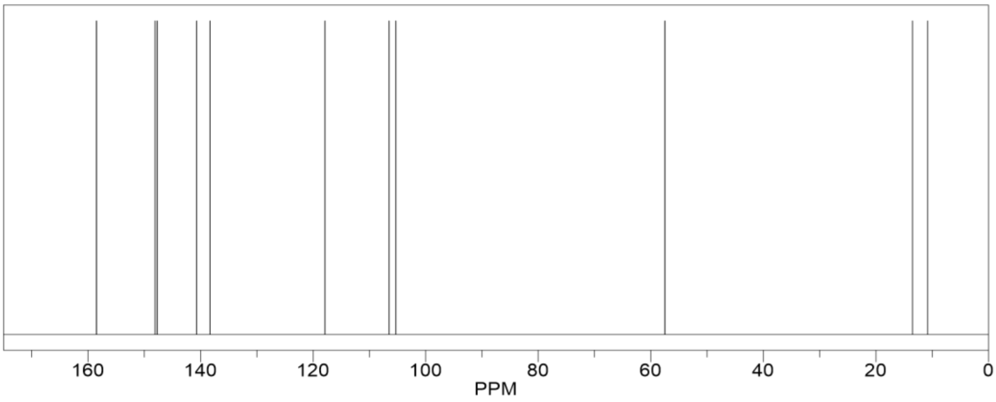


Chemical Shift δ (ppm)	Multiplicity	Integrity
8,02	dd	1H
7,57	t	1H
7,41	td	1H
6,63	d	1H
6,56	td	1H
5,74	s	1H
5,54	d	2H
2,36	s	3H
2,07	s	3H

Figure 28 L_1 ligand ^{1}H-NMR spectra (Bouroumane et al., 2021)



δ (ppm)
157,6
147,7
146,4
139,3
137,5
113,4
109,1
105,1
53,9
13,8
11,2

Figure 29 L_1 ligand ^{13}C-NMR spectra (Bouroumane et al., 2021)

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis above, the ^{1}H-NMR analysis shows the signal appears as nine peaks at 8.02 and 6.63 ppm as doublet peaks as well as 7.41 and 6.56 ppm as a triplet peak related to aromatic protons (^{1}H on sp^2 CH groups), triplet peak at 7.57 ppm related to proton from amine groups (N-H), doublet peak at 5.54 ppm related to ^{1}H on sp^3 CH_2 groups bonded with electronegative atom, singlet peak at 5.74 ppm related to aliphatic protons (^{1}H on sp^2 CH groups), and singlet peaks at 2.36 and 2.07 ppm related to ^{1}H on sp^3 CH_3 groups. Meanwhile, the ^{13}C-NMR analysis showed that there were peaks at 157.6; 146.4; and 139.3 ppm related to ^{13}C on sp^2 bond with electronegative atom, peaks at 147.7; 137.5; 113.4; and 105.1 ppm related to

^{13}C on sp^2 CH peaks at 13.8 and 11.2 ppm related to ^{13}C on sp^3 methyl (CH_3), and peak at 53.9 ppm related to ^{13}C on sp^3 CH_2 bonded to electronegative atom. Thus, if the ^{1}H-NMR and ^{13}C-NMR analyzes are combined, N-((3,5-dimethyl-1H-pyrazol-1-yl)methyl)pyridin-2-amine compound is formed and its structure shown in **Figure 30**.

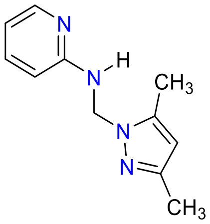


Figure 30 Structure of L_1 ligand (Bouroumane et al., 2021)

4.4.2. NMR Spectra of 5-chloro-N-((3,5-dimethyl-1H-pyrazol-1-yl)methyl)pyridin-2-amine (L_2 Ligand)

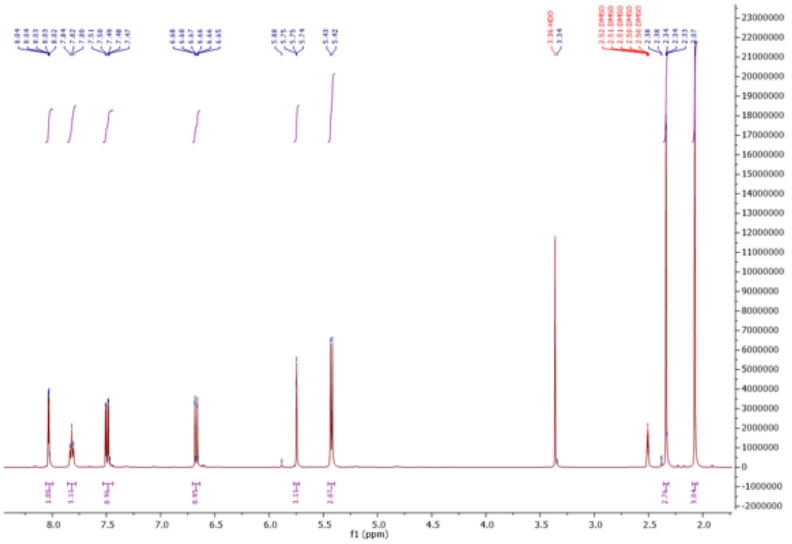
^{1}H-NMR and ^{13}C-NMR spectra of L_2 ligand are shown in **Figures 31 and 32**, respectively. Based on a step-by-step analysis of how to read and interpret the ^{1}H-NMR and ^{13}C-NMR spectrum, the following conclusions are:

4.4.2.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 31** shows eight peaks that appears which indicates a signal originating from eight types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 31** presents the signal appear with varying multiplicities, indicating the presence of multiple protons in neighboring C atoms.
3) **Step 3:** Identify the signal integration value. **Figure 31** shows several proton integrations, ranging from 1H, 2H, and 3H.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, the value of the proton chemical shift can be determined. **Figure 31** shows the peaks at 8.03; 7.49; and 6.66 ppm related to aromatic protons (^{1}H on sp^2 CH groups), a peak at 7,81 ppm is related to proton from amine groups (N-H), a peak at 5.42 ppm is related to ^{1}H on sp^3 CH_2 groups and bonded to electronegative atoms, peak at 5.74 ppm related to aliphatic protons (^{1}H on sp^2 CH groups), and peaks at 2.33 and 2.07 ppm related to ^{1}H on sp^3 CH_3 groups.

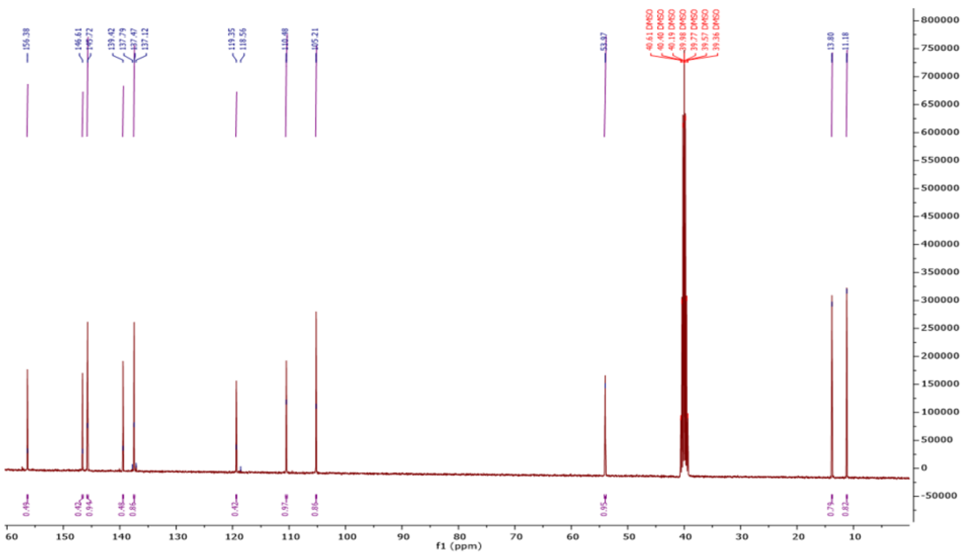
4.4.2.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. From the analysis of the ^{13}C-NMR spectrum of the ligand L_1 compound, it was shown that eleven peaks appeared indicating the existence of eleven types of carbon atoms.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, the value of the carbon chemical shift can be determined. **Figure 32** show that the peaks that appear at 156.3; 145.7; and 139.4 ppm related to ^{13}C on sp^2 bond with electronegative atom such as N, peaks at 146.6; 137.4; 119.3; and 105.2 ppm related to ^{13}C on sp^2 CH, peaks at 13.8 and 11.1 ppm related to ^{13}C on sp^3 methyl (CH_3), and peak at 53.9 ppm related to ^{13}C on sp^3 CH_2 bonded to electronegative atom. A significant difference in chemical shift compared to the L1 ligand was seen at the peak of 119.3 ppm. In L_1 ligand, the peak appears at 113.4 ppm. In L_2 ligands, the peaks appear at a larger chemical shift. This is probably due to the presence of electronegative substituents such as halogens.



δ (ppm)	Multiplicity	Integrity
8,03	dd	1H
7,81	t	1H
7,49	dd	1H
6,66	dd	1H
5,74	s	1H
5,42	d	2H
2,33	s	3H
2,07	s	3H

Figure 31 L_2 ligand ^{1}H-NMR spectrum analysis (Bouroumane et al., 2021)

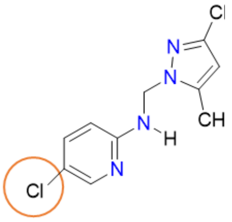


δ (ppm)
156,3
146,6
145,7
139,4
137,4
119,3
110,4
105,2
53,9
13,8
11,1

Figure 32 L_2 ligand ^{13}C-NMR spectrum analysis (Bouroumane et al., 2021)

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 31 and 32**, respectively, the ^{1}H-NMR analysis shows the signal appears as nine peaks at 8.03 and 6.66 ppm as doublet peaks and 7.49 ppm as a triplet peak that related to aromatic protons (^{1}H on sp^2 CH groups), triplet peak at 7.81 ppm that related to proton from amine groups (N-H), doublet peak at 5.42 ppm that related to ^{1}H on sp^3 CH_2 groups bonded with electronegative atom, singlet peak at 5.74 ppm that related to aliphatic protons (^{1}H on sp^2 CH groups), and singlet peaks at 2.33 and 2.07 ppm that related to ^{1}H on sp^3 CH_3 groups. Meanwhile, the ^{13}C-NMR analysis showed that there were peaks at 156.3;

146.6; and 139.4 ppm that related to ^{13}C on sp^2 bond with electronegative atom, peaks at 145.7; 137.5; and 105.2 ppm that related to ^{13}C on sp^2 CH, peaks at 13.8 and 11.1 ppm that related to ^{13}C on sp^3 methyl (CH_3), peak at 53.9 ppm that related to ^{13}C on sp^3 CH_2 bonded to electronegative atom, and peak at 119.3 ppm that related to ^{13}C on sp^2 bonded to halogen atom such as Cl. Thus, if the ^{1}H-NMR and ^{13}C-NMR analyses are combined, 5-chloro-N-((3,5-dimethyl-1H-pyrazol-1-yl) methyl) pyridin-2-amine compound is formed and its structure shown in **Figure 33**.



Halogen (Cl) at C^5 Pyridine

Figure 33 Structure of L_2 ligand (Bouroumane et al., 2021)

4.4.3. NMR Spectra of N-((3,5-dimethyl-1H-pyrazol-1-yl)methyl)-4-methylpyridin-2-amine (L_3 Ligand)

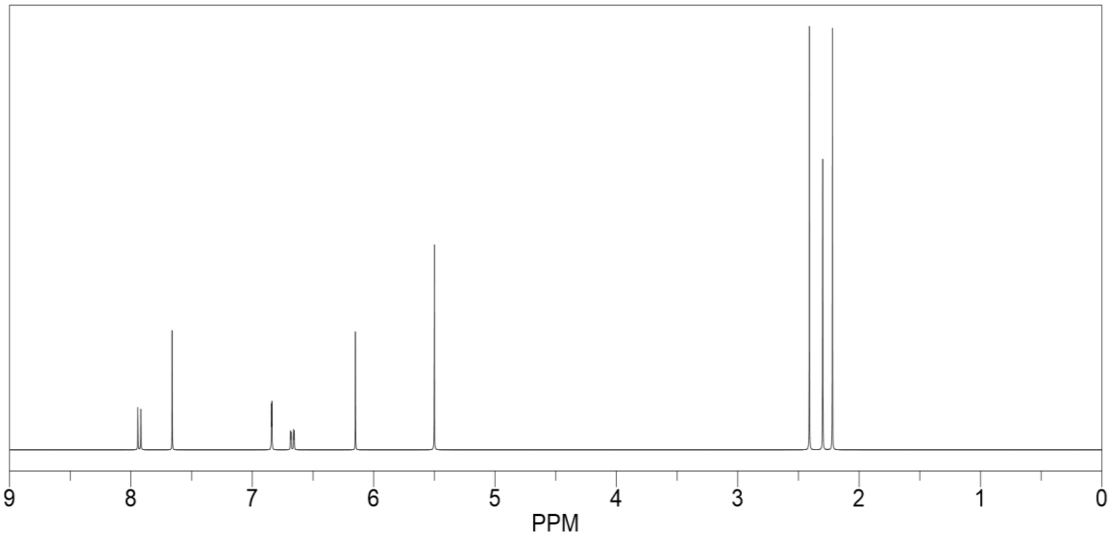
^{1}H-NMR and ^{13}C-NMR spectra of L_3 ligand are shown in **Figures 34 and 35**, respectively.

4.4.3.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 34** shows seven peaks that appears which indicates a signal originating from seven types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 34** shows the signals appear with varying multiplicities, indicating the presence of multiple protons in neighboring C atoms.
3) **Step 3:** Identify the signal integration value. **Figure 34** shows several proton integrations, ranging from 1H, 2H, and 3H.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in Table 1, the value of the proton chemical shift can be determined. **Figure 34** shows the peak at 7.89 ppm appear due to aromatic proton (^{1}H on sp^2 CH groups), peak at 7.46 ppm appear due to proton from amine groups (N-H), peak at 6.52 – 6.32 ppm appear due to ^{1}H on sp^2 CH groups, a peak at 5.73 ppm appear due to aliphatic protons (^{1}H on sp^2 CH groups), peak at 5.44 appear due to ^{1}H on sp^3 CH_2 groups and bonded to electronegative atoms, and peaks at 2.36 and 2.08 ppm appear due to aromatic and aliphatic ^{1}H on sp^3 CH_3 groups, respectively.

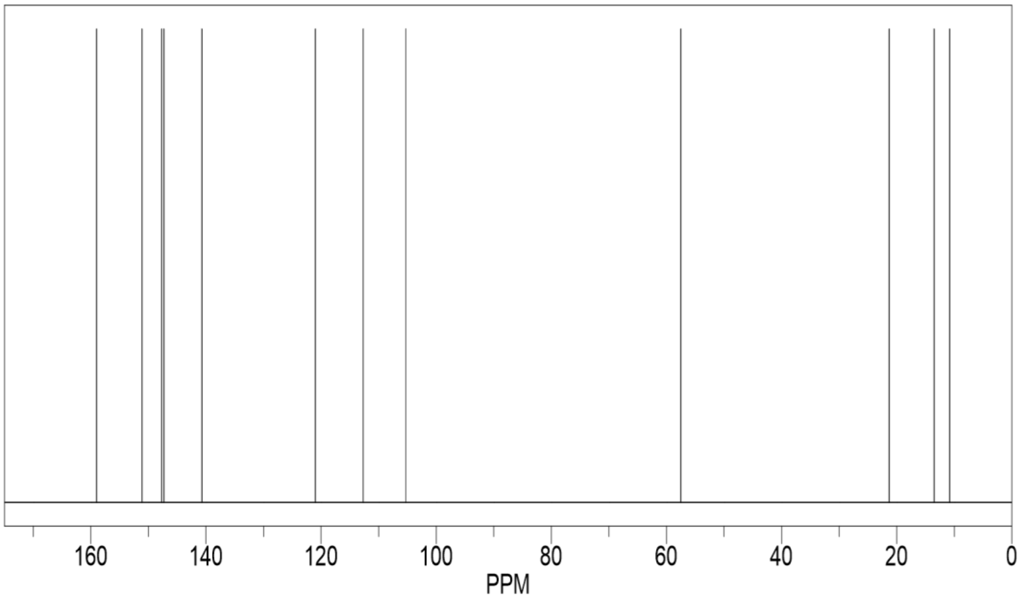
4.4.3.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. From the analysis of the ^{13}C-NMR spectrum of the ligand L_3 compound, it was shown that twelve peaks appeared indicating the existence of twelve types of carbon atoms.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, the value of the carbon chemical shift can be determined. **Figure 35** show that the peaks similar to L_1 and L_2 ligands. However, in **Figure 35**, it is shown that there is a peak that appears at 21,0 ppm indicating the presence of methyl carbon in the aromatic ring while L_1 and L_2 do not show a peak for sp^3 methyl carbon in the aromatic ring.



Chemical Shift δ (ppm)	Multiplicity	Integrity
7,89	d	1H
7,46	t	1H
6,52 – 6,32	m	2H
5,73	s	1H
5,44	d	2H
2,36	s	3H
2,08	s	3H

Figure 34 L_3 ligand ^{1}H-NMR spectra (Bouroumane et al., 2021)



δ (ppm)
157,8
147,8
147,4
146,4
139,3
115.0
109,1
105,0
54,0
21,0
13,8
11,2

Figure 35 L_3 ligand ^{13}C-NMR spectrum (Bouroumane et al., 2021)

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Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 34 and 35**, the ^{1}H-NMR analysis shows the signal appears as seven peaks : doublet peak at 7.89 ppm related to aromatic proton (^{1}H on sp^{2} CH groups), triplet peak at 7,46 ppm related to proton from amine groups (N-H), multiplet peak at 6.52 – 6.32 ppm related to ^{1}H on sp^{2} CH groups, singlet peak at 5,73 ppm related to aliphatic protons (^{1}H on sp^{2} CH groups), doublet peak at 5,44 related to ^{1}H on sp^{3} CH_2 groups and bonded to electronegative atoms, and singlet peaks at 2.36 and 2.08 ppm related to aromatic and aliphatic ^{1}H on sp^{3} CH_3 groups. Meanwhile, the ^{13}C-NMR analysis are similar to L_1 and L_2 ligand but show peak that appears at 21.0 ppm indicating the presence of methyl carbon in the aromatic ring while L_1 and L_2 do not show a peak for sp^{3} methyl carbon in the aromatic ring. Thus, if the ^{1}H-NMR and ^{13}C-NMR analysis shown in **Figures 34 and 35** are combined, N-((3, 5-dimethyl-1H-pyrazol-1-yl)methyl)-4-methylpyridin-2-amine compound is formed and its structure is shown in **Figure 36**.

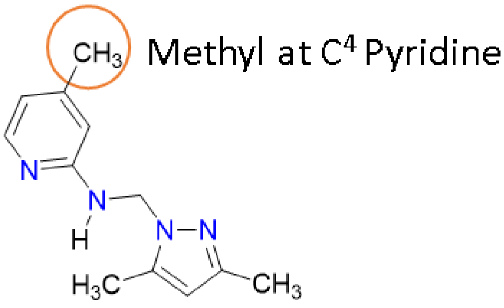


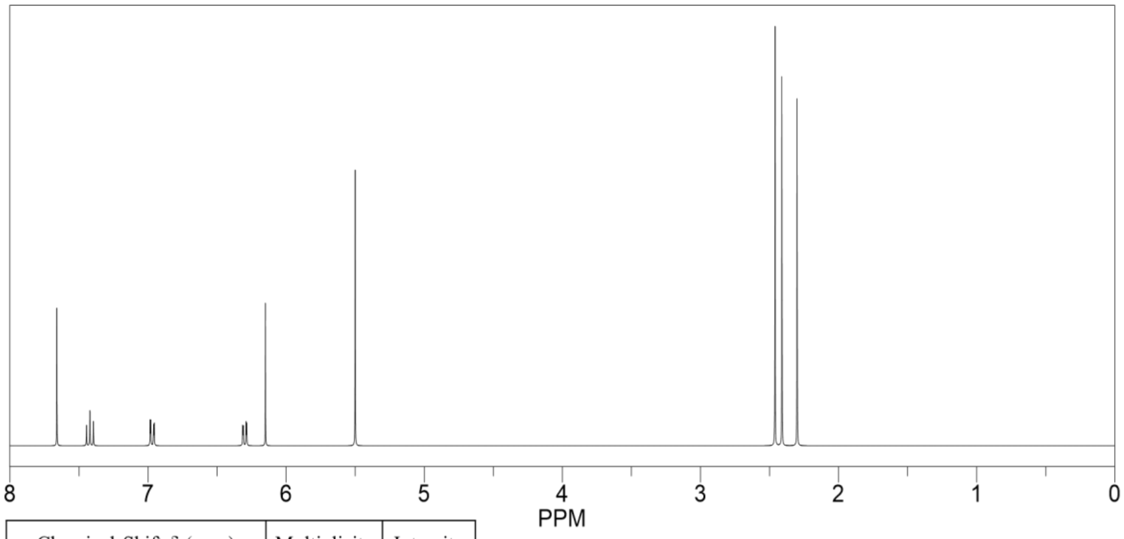
Figure 36 Structure of L_3 ligand (Bouroumane et al., 2021)

4.4.4. NMR Spectra of N-((3,5-dimethyl-1H-pyrazol-1-yl)methyl)-6-methylpyridin-2-amine (L_4 Ligand)

^{1}H-NMR and ^{13}C-NMR spectra of L_3 ligand are shown in **Figures 37 and 38**, respectively.

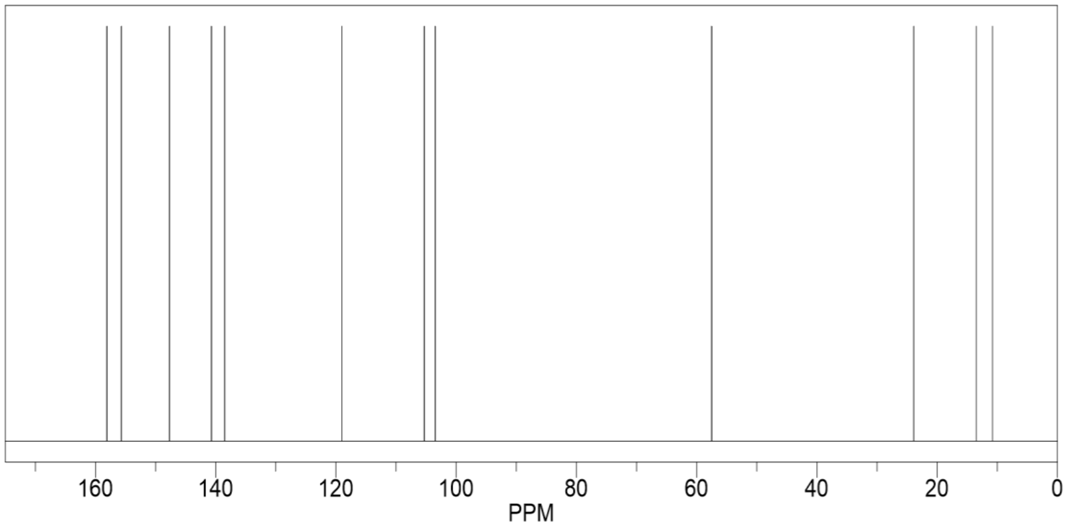
4.4.4.1. ^{1}H-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals that appear. **Figure 37** shows seven peaks that appears which indicates a signal originating from seven types of proton.
2) **Step 2:** Identify the multiplicity of signals that arise due to the presence of neighboring protons. **Figure 37** shows the signals appear with varying multiplicities, indicating the presence of multiple protons in neighboring C atoms.
3) **Step 3:** Identify the signal integration value. **Figure 37** shows several proton integrations, ranging from 1H, 2H, and 3H.
4) **Step 4:** Identify the peaks based on the chemical shift value (δ). Based on the reference data in **Table 1**, **Figure 37** shows the similar result to L_3 ligand ^{1}H-NMR analysis. However, **Figure 37** shows a slightly different chemical shift. The peak at 7.5 ppm appear due to proton from amine groups (N-H), peaks at 7.3 ppm as well as 6.42 – 6.44 ppm appear due to aromatic proton (^{1}H on sp^{2} CH groups), peak at 5.73 ppm appear due to aliphatic protons (^{1}H on sp^{2} CH groups), peak at 5.40 appear due to ^{1}H on sp^{3} CH_2 groups and bonded to electronegative atoms, and peaks at 2.43 and 2.28 ppm appear due to aromatic and aliphatic ^{1}H on sp^{3} CH_3 groups, respectively.



Chemical Shift δ (ppm)	Multiplicity	Integrity
7,5	t	1H
7,3	t	1H
6,42 – 6,44	d	2H
5,73	s	1H
5,40	d	2H
2,43	s	3H
2,28	s	3H

Figure 37 L_4 ligand ^{1}H-NMR spectra (Bouroumane et al., 2021)



δ (ppm)
157,0
155,9
146,4
139,4
137,9
112,4
105,9
104,9
53,8
24,4
13,8
11,1

Figure 38 L_4 ligand ^{13}C-NMR spectrum (Bouroumane et al., 2021)

4.4.4.2. ^{13}C-NMR Spectra Analysis

1) **Step 1:** Identify the number of signals. **Figure 38** shows twelve peaks appeared indicating the existence of twelve types of carbon atoms.
2) **Step 2:** Identify the chemical shift value (δ). Based on the reference data in **Table 2** and **Figure 2**, the value of the carbon chemical shift can be determined. **Figure 38** shows that the peaks similar to L_3 ligand. However, **Figure 38** shows the peak that represent C sp^3 methyl appears at higher chemical shift (24.4 ppm) probably due to different position at aromatic ring.

Based on the ^{1}H-NMR and ^{13}C-NMR spectrum analysis in **Figures 37 and 38**, respectively, the ^{1}H-NMR analysis shows the signal appears as seven peaks : triplet peak at 7.5 ppm related to proton from amine groups (N-H), triplet peak at 7.3 ppm related to aromatic proton (^{1}H on sp^2 CH groups), doublet peak at 6.42 – 6.44 ppm related to ^{1}H on sp^2 CH groups, singlet peak at 5.73 ppm related to aliphatic protons (^{1}H on sp^2 CH groups), doublet peak at 5.44 ppm related to ^{1}H on sp^3 CH_2 groups and bonded to electronegative atoms, and singlet peaks at 2.43 and 2.28 ppm related to aromatic and aliphatic ^{1}H on sp^3 CH_3 groups, respectively. Meanwhile, the ^{13}C-NMR analysis are similar to L_3 but show peak that appears at greater chemical shift (24,4) ppm for methyl carbon in the aromatic ring probably due to different position at aromatic ring. Thus, if the ^{1}H-NMR and ^{13}C-NMR analysis shown in **Figures 37 and 38** are combined, N-((3,5-dimethyl-1H-pyrazol-1-yl)methyl)-6-methylpyridin-2-amine compound is formed and its structure is presented in **Figure 39**.

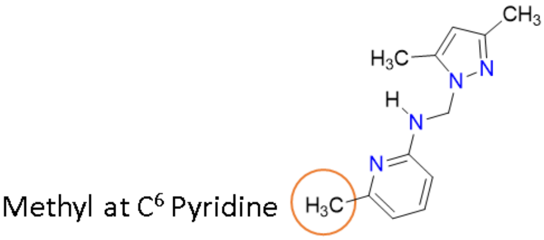


Figure 39 Structure of L_4 ligand (Bouroumane et al., 2021)

5. CONCLUSION

This study shows the simplest way to understand the results of ^{1}H-NMR and ^{13}C-NMR spectroscopic analysis. This study was conducted by describing the step-by-step how to read and interpret ^{1}H-NMR and ^{13}C-NMR spectra of several organic compounds based on the level of difficulty: (1) simple compounds, (2) fairly complex compounds, (3) more complex compounds, and (4) very complex compounds. With this paper, we believe that this paper can be used as a basis for understanding and interpreting ^{1}H-NMR and ^{13}C-NMR data.

6. AUTHORS' NOTE

Authors declare that there is no conflict of interest regarding the publication of this article. Authors confirmed that the paper was free of plagiarism.

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