

Examining the structural changes in the organic molecules during hydrolysis with simulated ^1H NMR using JSPEC View (Jmol)

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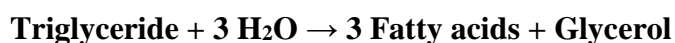
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1. Introduction

Hydrolysis is a type of chemical reaction in which water is used to break apart a larger molecule into smaller molecules. The term "hydrolysis" comes from the Greek words "hydro" meaning water, and "lysis" meaning to break apart. In a hydrolysis reaction, a molecule of water (H_2O) is split into a hydrogen ion (H^+) and a hydroxide ion (OH^-), and these ions are used to break the bonds of the larger molecule. The end products of a hydrolysis reaction are typically a smaller molecule and either an alcohol or an acid, depending on the reaction conditions.

There are two main types of hydrolysis reactions: acid hydrolysis and base hydrolysis. In an acid hydrolysis reaction, the hydrogen ion (H^+) comes from an acid, such as hydrochloric acid (HCl), and the hydroxide ion (OH^-) comes from water. In a base hydrolysis reaction, the hydroxide ion (OH^-) comes from a base, such as sodium hydroxide (NaOH), and the hydrogen ion (H^+) comes from water.

Another method of hydrolysis is with the assistance of enzymes, one such example is hydrolysis reaction of a triglyceride (a type of fat molecule) into glycerol and fatty acids. When a triglyceride is treated with water and an enzyme called lipase, the ester bonds that link the glycerol and fatty acid molecules are broken, and the triglyceride is hydrolyzed into its component parts. Another example is the hydrolysis of a protein into its component amino acids, which can happen by the action of enzymes called proteases.



It is also important to note that hydrolysis can also refer to the breakdown of a complex molecule through the addition of water in a specific way, like breaking apart a sugar molecule by adding a water molecule.

Another example is the breakdown of an ester into a carboxylic acid and alcohol by adding water in presence of a mineral acid like HCl. The reverse reaction, in which an alcohol and an acid or a carboxylic acid and alcohol are combined to form an ester or an amide, is called a condensation reaction.

There are also proteins hydrolysis in which a protein is broken down by water and enzymes into smaller peptides and amino acids, this process is important for the digestion of proteins in the body. In general, hydrolysis reactions are reversible, meaning that the products of the reaction can be recombined under the right conditions to form the original molecule.

Hydrolysis reactions are also used in many industrial processes. For example, during the production of ethanol from biomass, enzymes are used to hydrolyse cellulose and hemicellulose in the biomass into simpler sugars which are then fermented to produce ethanol.

Overall Hydrolysis is a versatile process that can be used to break down a wide range of compounds such as carbohydrates, proteins, lipids, and even some synthetic compounds.

In this project we have performed the hydrolysis of benzamide using sodium hydroxide- base catalysed hydrolysis reaction in a real time lab.

2. Objectives:

- To perform the hydrolysis of benzamide to get benzoic acid. (Real –time)
- To demonstrate the detailed mechanism of hydrolysis process
- To create 3D structures for various organic molecules in Jmol interface
- To evaluate the structural changes in the reactant and product with JSPEC view in Jmol MOL
- To analyse the hydrolysis reactions in a few more molecules such as sucrose, ethyl acetate, methyl acetate, starch etc.
- To interpret the ¹HNMR obtained from JSPEC view of Jmol

3. EXPERIMENTAL PROCEDURES AND METHODS

Apparatus required:

- Round Bottom flask
- Water bath
- Measuring cylinder
- 250 ml beaker glass rod
- Buchner funnel
- 250 ml of conical flask
- Suction pump

Chemicals required:

- Benzamide - 4gm
- Concentrated Hydrochloric acid - 5ml
- 10% Sodium Hydroxide solution – 20ml

Procedure:

- About 4 g of benzamide is mixed with 20 ml of sodium hydroxide solution taken in a RB flask.
- The solution was kept in a water bath for about 30 minutes at 78° C.
- Sodium benzoate was formed with the evolution of ammonia.
- The heating was stopped when the evolution of ammonia ceased.
- The flask was then cooled and the contents were poured into 200 ml of water taken in a beaker, concentrated hydrochloric acid was added with constant stirring till the solution becomes distinctly acidic (tested with blue litmus which turns red).
- The precipitate benzoic acid was filtered with a suction pump using a Buchner funnel, washed with cold water and dried.

4. THE PROCESS OF HYDROLYSIS OF BENZAMIDE



Step (i): 4 ml of NaOH was taken in a 100ml in RB flask



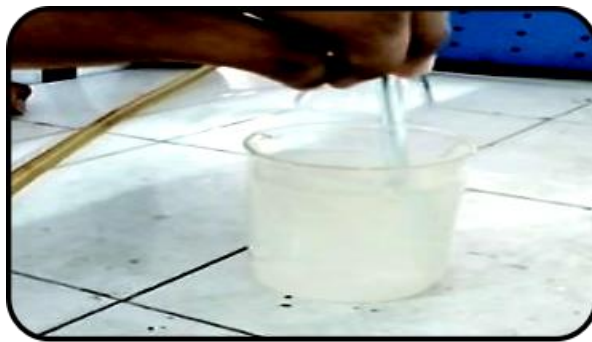
Step (ii): 4g of Benzamide added into the RB flask and homogenized



Step (iii): The mixture kept in water bath about 78°C for 30 minutes



Step (iv): The mixture was cooled at room temperature for few minutes



Step (v): 5 ml of HCl was added to 200ml of water in a beaker



Step (vi): Benzamide + NaOH mixture and HCl + water mixture was mixed well till it dissolves



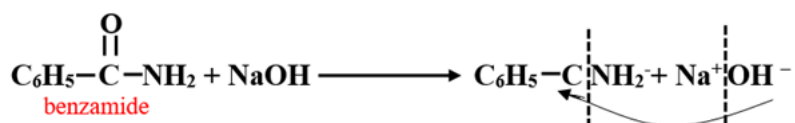
Step (vii): The process of filtration



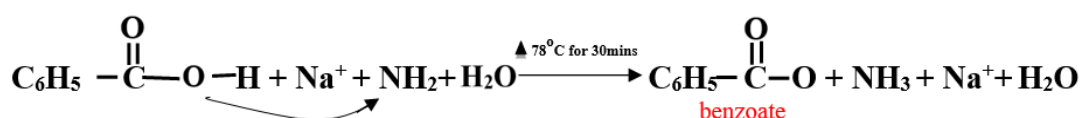
Step (viii): The filtered product benzoic acid

4.1. Mechanism of Hydrolysis:

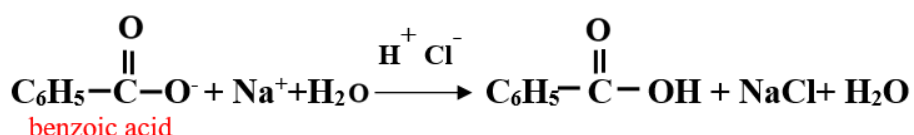
- i) When benzamide reacts with sodium hydroxide, ion exchange occurs



- ii) Sodium and ammonia, were removed by heating it at 78°C for 30 mins



- iii) Sodium benzoate will react with dilute HCl to form benzoic acid and sodium chloride



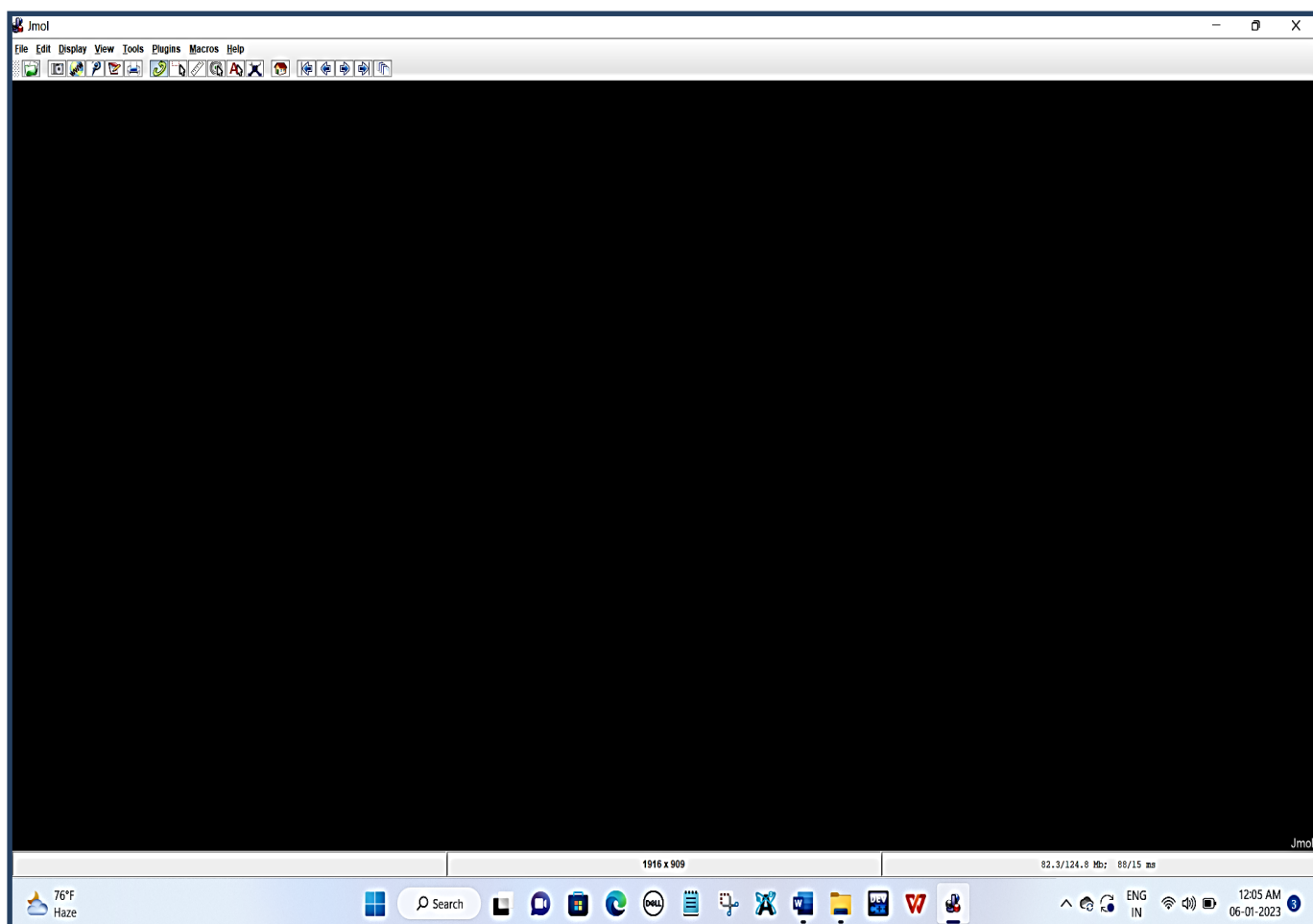
5. INSTALLATION OF Jmol

- Dealing with molecular structure
- Jmol is an open-source Java viewer for chemical structures and biomolecules in 3D
- It is free and open source software, written in Java, it runs on Windows, Mac OS X, Linux and Unix systems.



5.1. Procedure for installation of Jmol

- First install Jmol on your desktop or on your pc.
- If you need any help about Jmol you can refer to this link : www.jmol.sourceforge.net
- At first we installed Jmol on our pc.
- At first it looks like this



About the compound

Compound name: Benzamide

Chemical formula: $\text{C}_7\text{H}_7\text{NO}$

Molar mass: $121.139 \text{ g}\cdot\text{mol}^{-1}$

Melting point: 127 to 130 °C

Boiling point: 288 °C

5.2. Procedure for creating 3D structure of benzamide in Jmol

Step 1: Open Jmol in your desktop or in your pc

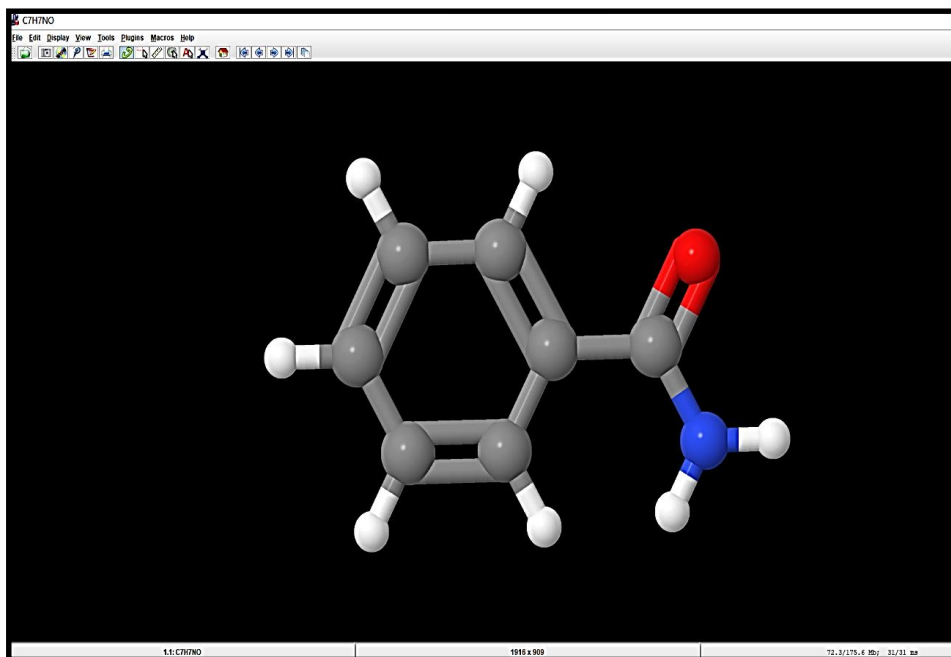
Step 2: Go to file and then click 'Get MOL'

Step 3: A dialogue box opens with the provision to type the molecule

Step 4: Type the name of the molecule as “benzamide” and click enter

Step 5: The 3D structure of benzamide was imported from the databas

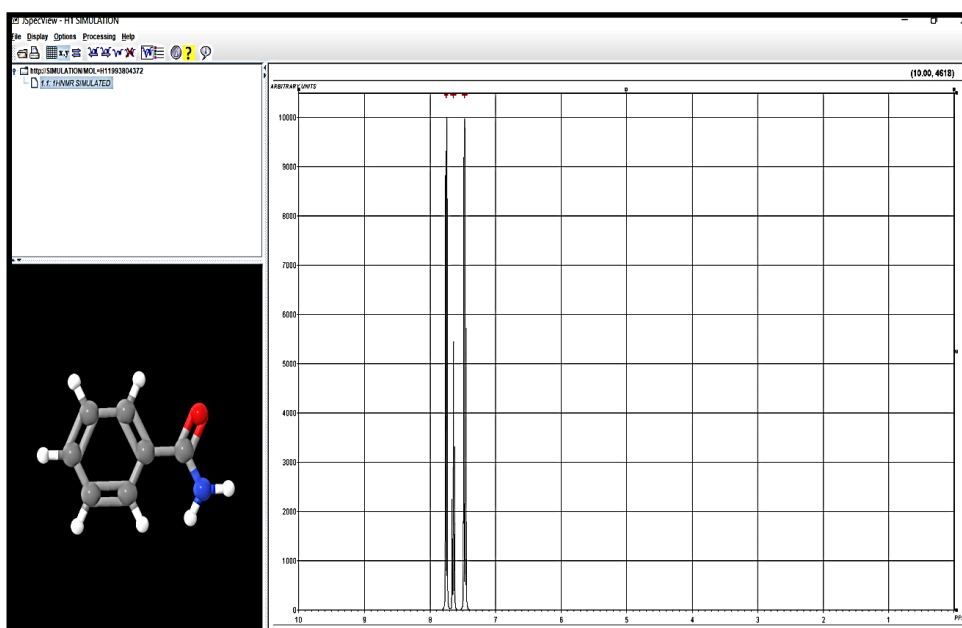
5.3. Procedure for obtaining 1HNMR of benzamide in JSPEC view of Jmol



Step 1: Open the Jmol interface window with the 3D structure of benzamide

Step 2: In the menu bar go to “tool” menu click the spectra inside that choose 1HNMR

Step 3: New JSPEC view dialogue box opens with 1HNMR spectra of benzamide



About the compound:

Compound name: Benzoic Acid

Chemical formula: $C_7H_6O_2$

Molar mass: 122.123 g/mol

Melting point 122 °C

Boiling point 250 °C

5.4. Procedure for creating 3D structure of benzoic acid in Jmol

Step 1: Open Jmol in your desktop or in your pc

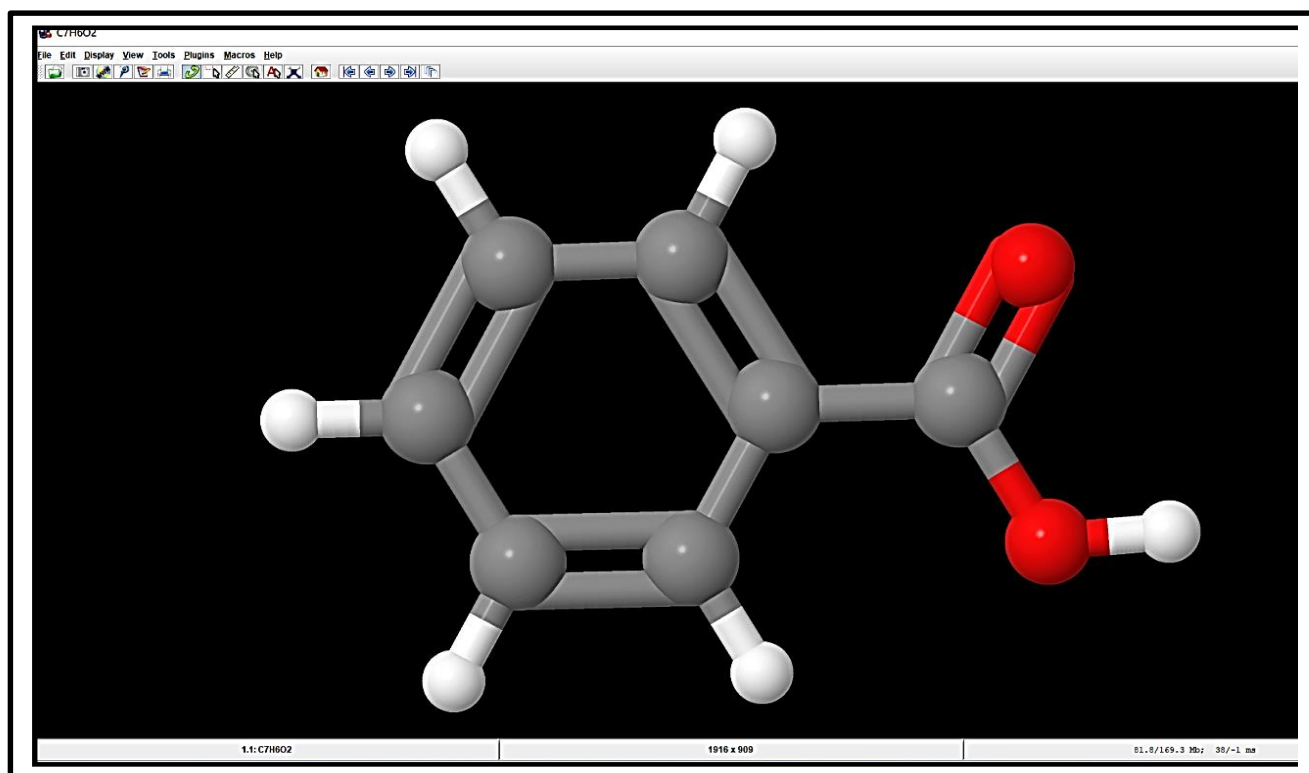
Step 2: Go to file and then click ‘Get MOL’

Step 3: A dialogue box opens with the provision to type the molecule

Step 4: Type the name of the molecule as “benzoic acid” and click enter

Step 5: The 3D structure of benzoic acid was imported from the database

THE STRUCTURE OF BENZOIC ACID IN Jmol



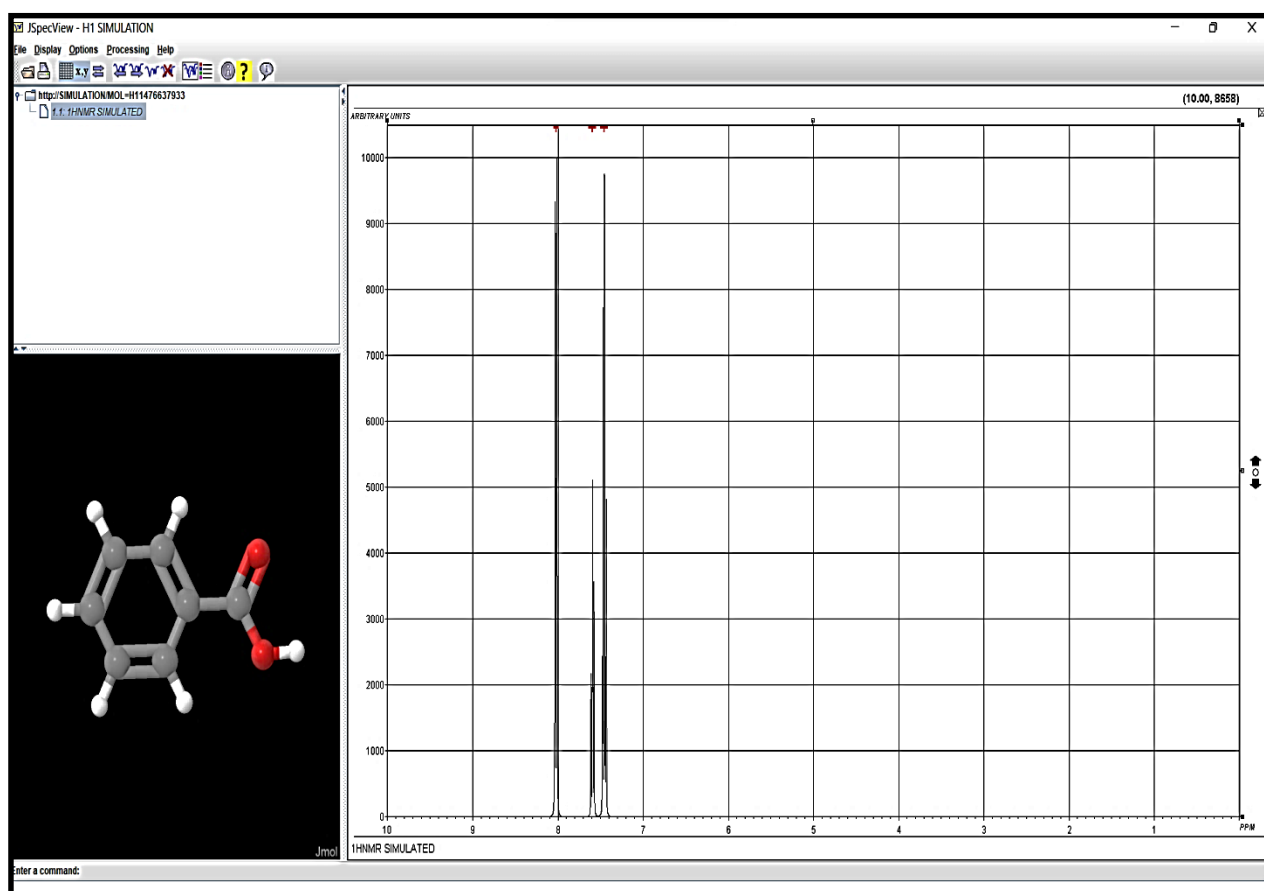
5.5. Procedure for obtaining ¹HNMR of benzoic acid in JSPEC view of JMOL

Step 1: Open the Jmol interface window with the 3D structure of benzoic acid

Step 2: In the menu bar go to “tool” menu click the spectra inside that choose ¹HNMR

Step 3: New JSPEC view dialogue box opens with ¹HNMR spectra of benzoic acid

¹HNMR OF BENZOIC ACID IN JSPEC VIEW OF JMOL



About the compound:

Compound name:	Methyl Acetate
Chemical formula:	$\text{C}_3\text{H}_6\text{O}_2$
Molar mass:	$74.079 \text{ g}\cdot\text{mol}^{-1}$
Melting point	-98°C
Boiling point	56.9°C

5.6. Procedure for creating 3D structure of methyl acetate in Jmol

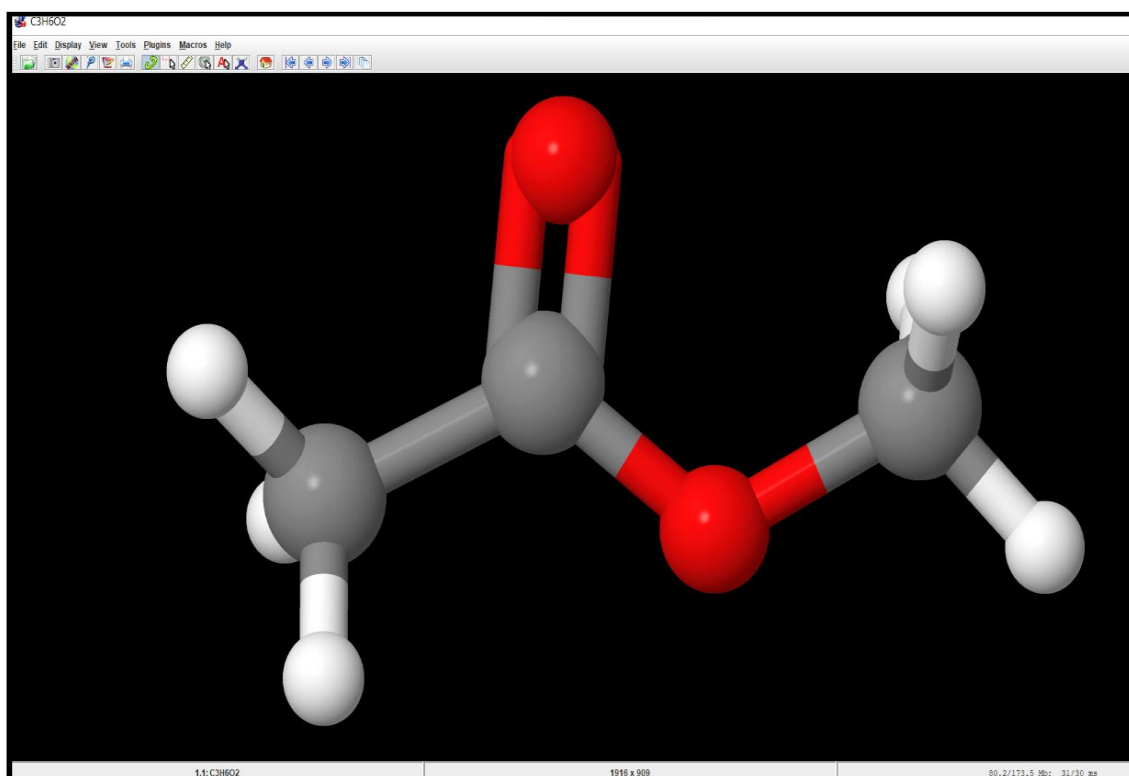
Step 1: Open Jmol in your desktop or in your pc

Step 2: Go to file and then click ‘Get MOL’

Step 3: A dialogue box opens with the provision to type the molecule

Step 4: Type the name of the molecule as “methyl acetate” and click enter

Step 5: The 3D structure of methyl acetate was imported from the database

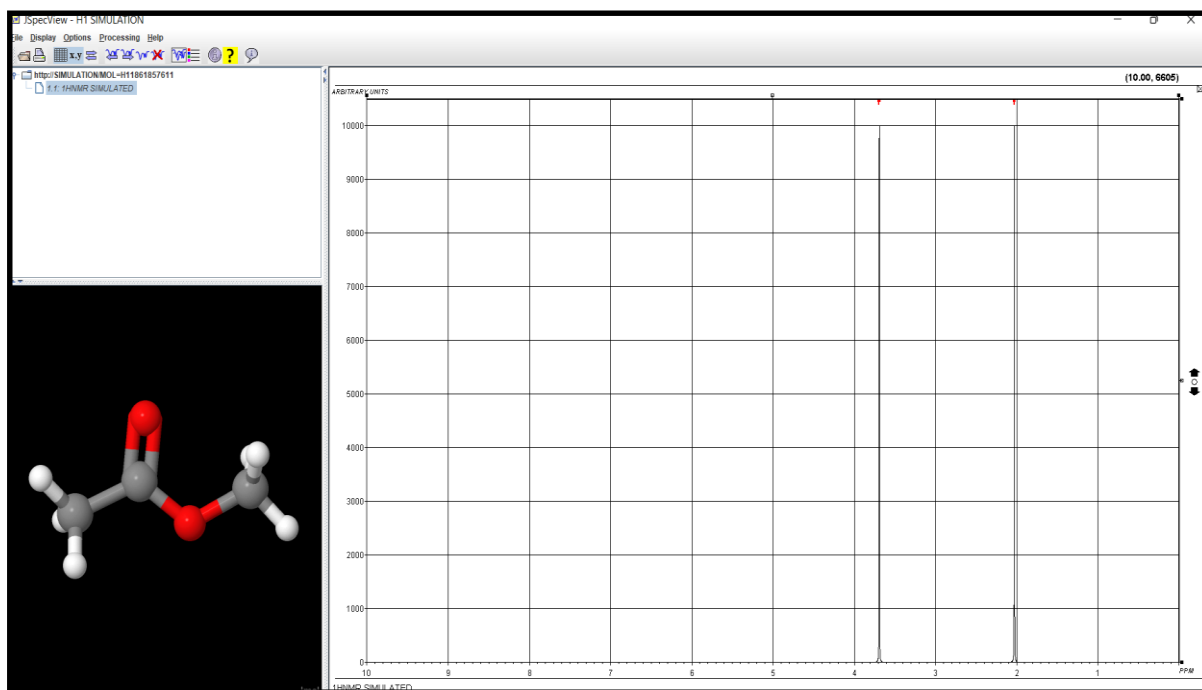
3D STRUCTURE OF METHYL ACETATE CREATED IN Jmol

5.7. Procedure for obtaining ^1H NMR of methyl acetate in JSPEC view of Jmol

Step 1: Open the Jmol interface window with the 3D structure of methyl acetate

Step 2: In the menu bar go to “tool” menu click the spectra inside that choose ^1H NMR

Step 3: New JSPEC view dialogue box opens with ^1H NMR spectra of methyl acetate



About the compound:

Compound name: acetic acid

Chemical formula: CH_3COOH

Molar mass: $60.052 \text{ g}\cdot\text{mol}^{-1}$

Melting point 16 to 17 °C

Boiling point 118 to 119 °C

5.8. Procedure for creating 3D structure of acetic acid in Jmol

Step 1: Open Jmol in your desktop or in your pc

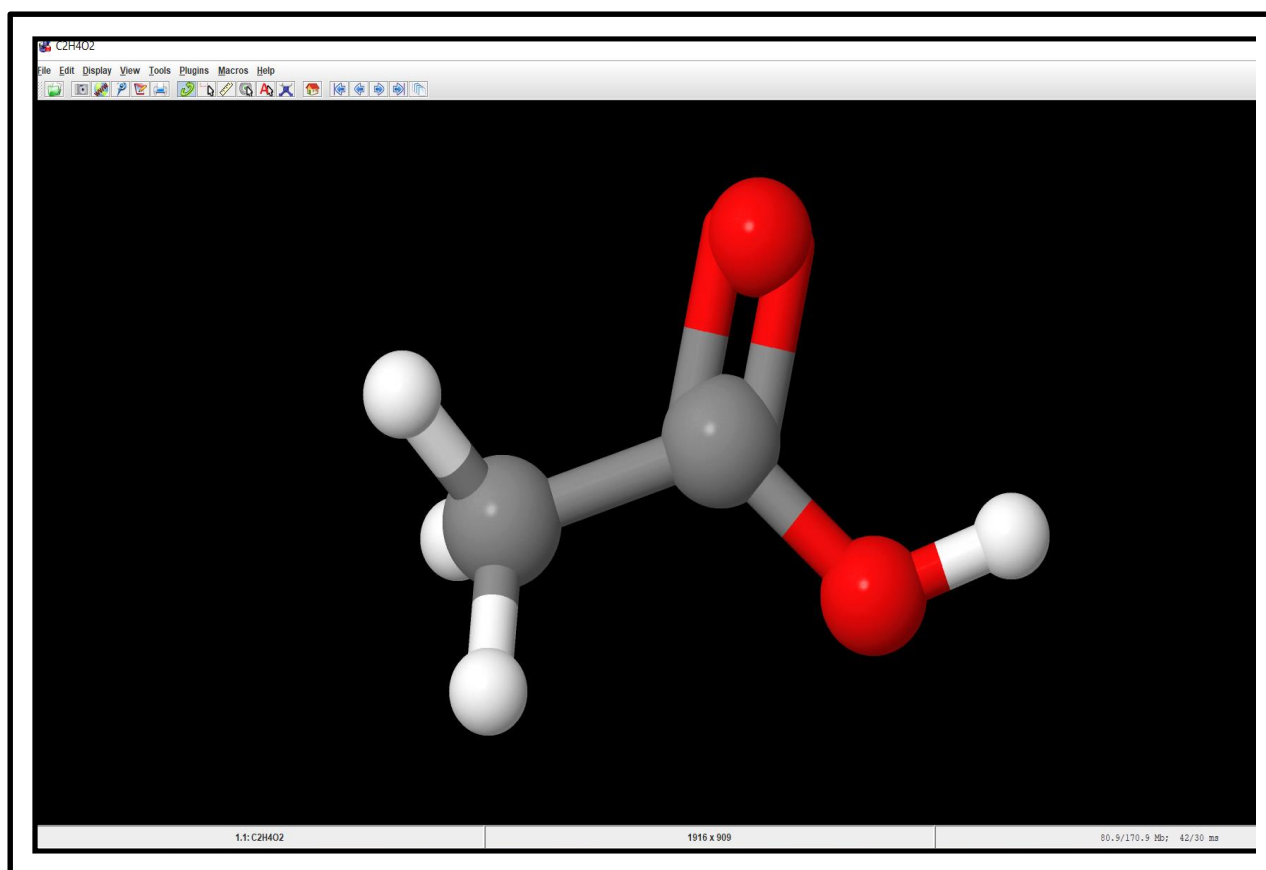
Step 2: Go to file and then click ‘Get MOL’

Step 3: A dialogue box opens with the provision to type the molecule

Step 4: Type the name of the molecule as “acetic acid” and click enter

Step 5: The 3D structure of acetic acid was imported from the database

THE STRUCTURE OF ACETIC ACID IN Jmol



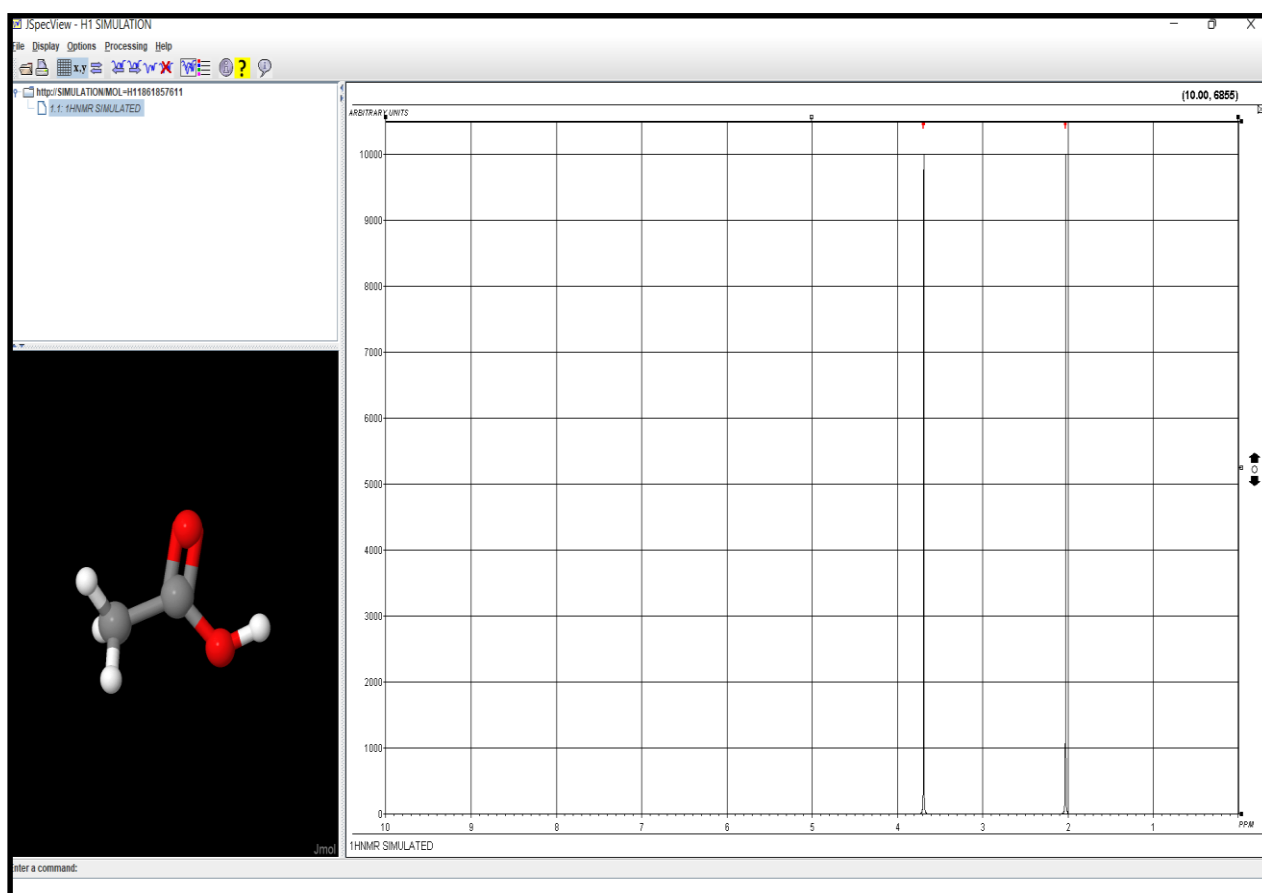
5.9. Procedure for obtaining ^1H NMR of acetic acid in JSPEC view of Jmol

Step 1: Open the Jmol interface window with the 3D structure of acetic acid

Step 2: In the menu bar go to “tool” menu click the spectra inside that choose ^1H NMR

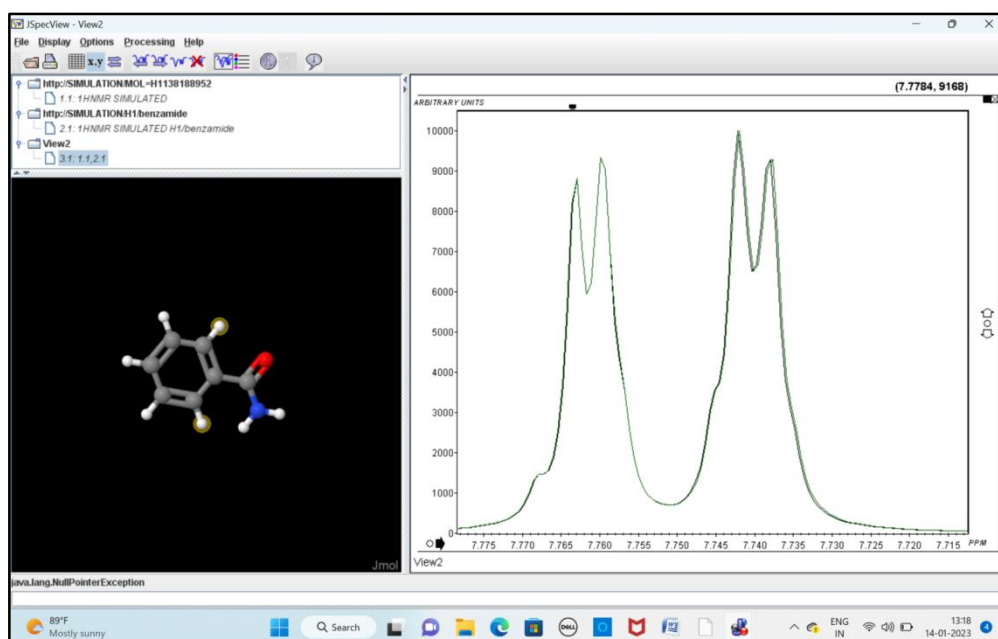
Step 3: New JSPEC view dialogue box opens with ^1H NMR spectra of acetic acid

^1H NMR OF ACETIC ACID IN JSPEC VIEW OF Jmol

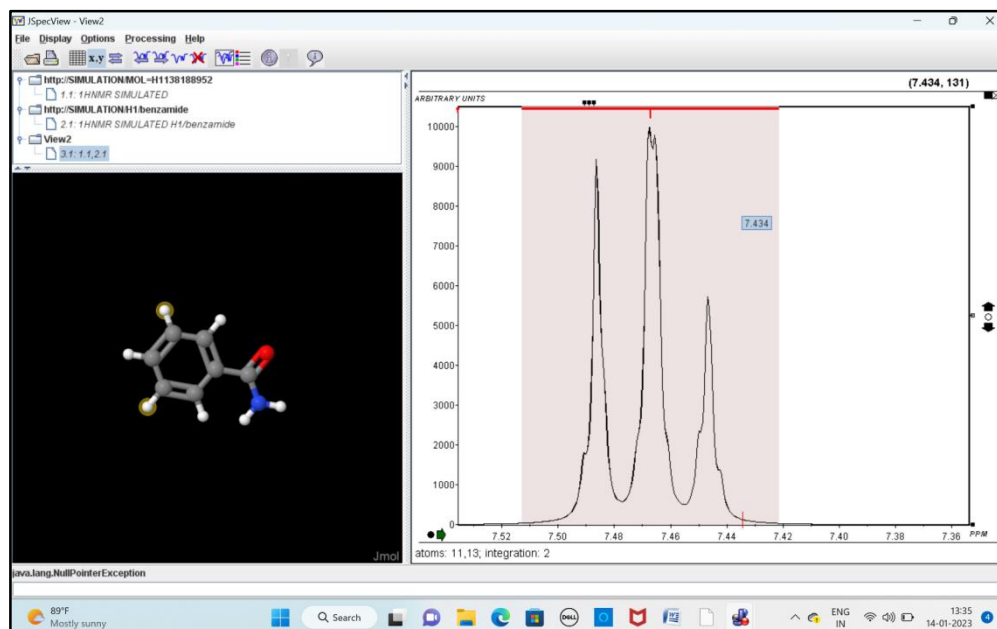


6. Result and Discussion

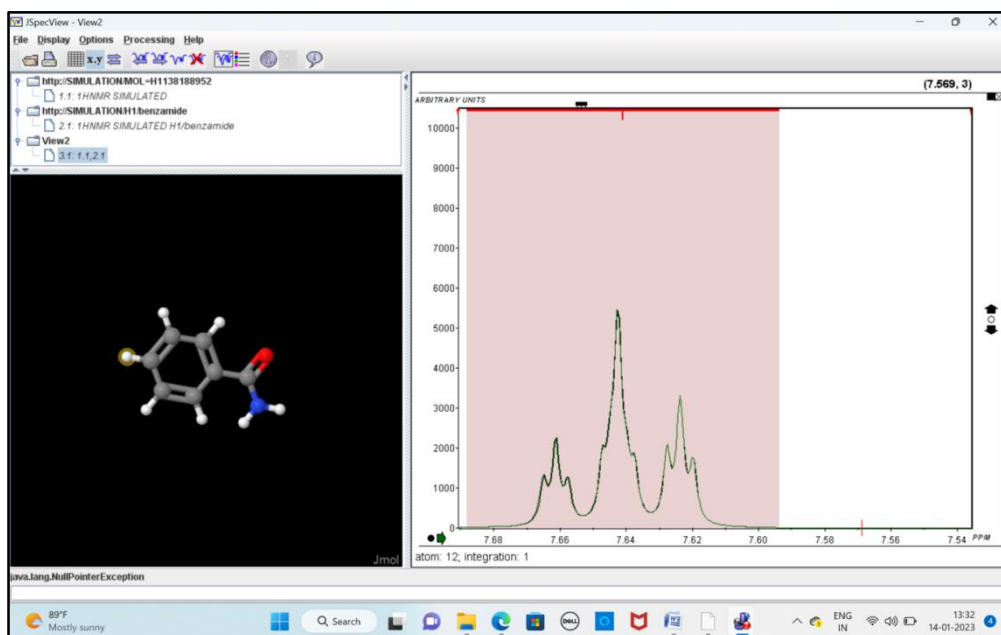
6.1. Interpretation of ^1H NMR of benzamide molecule obtained from JSPEC view of Jmol



- Doublet peak was obtained for both the ortho hydrogens as they have only one equivalent hydrogen in their neighbouring position
- Position of the peaks were 7.742 and 7.763 PPM

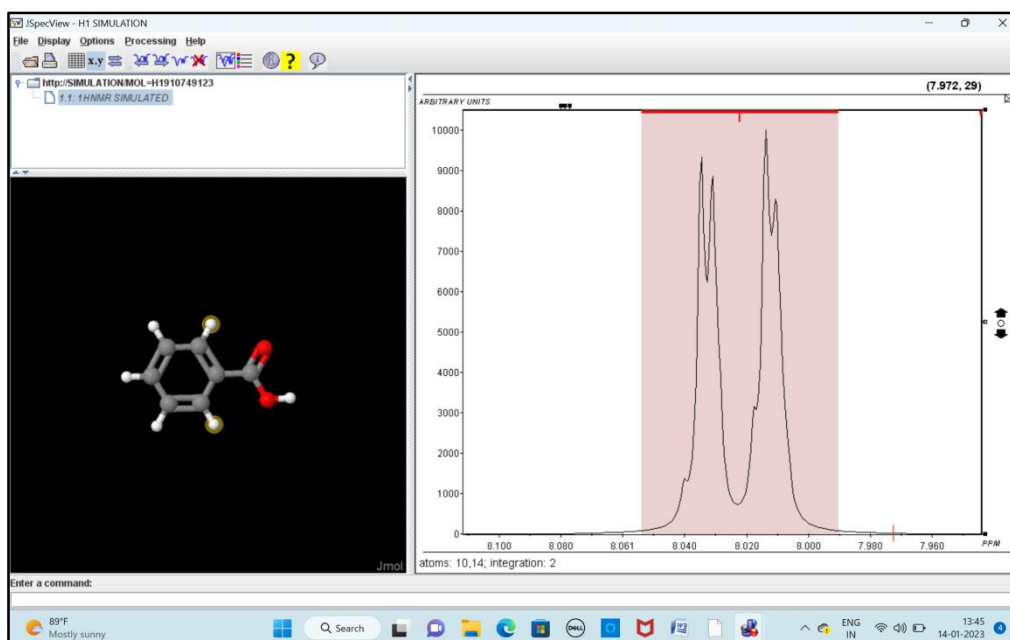


- Triplet peak was obtained for both the meta hydrogen atoms as they have two equivalent hydrogen in its neighbouring position
- The peak position was at 7.468 PPM

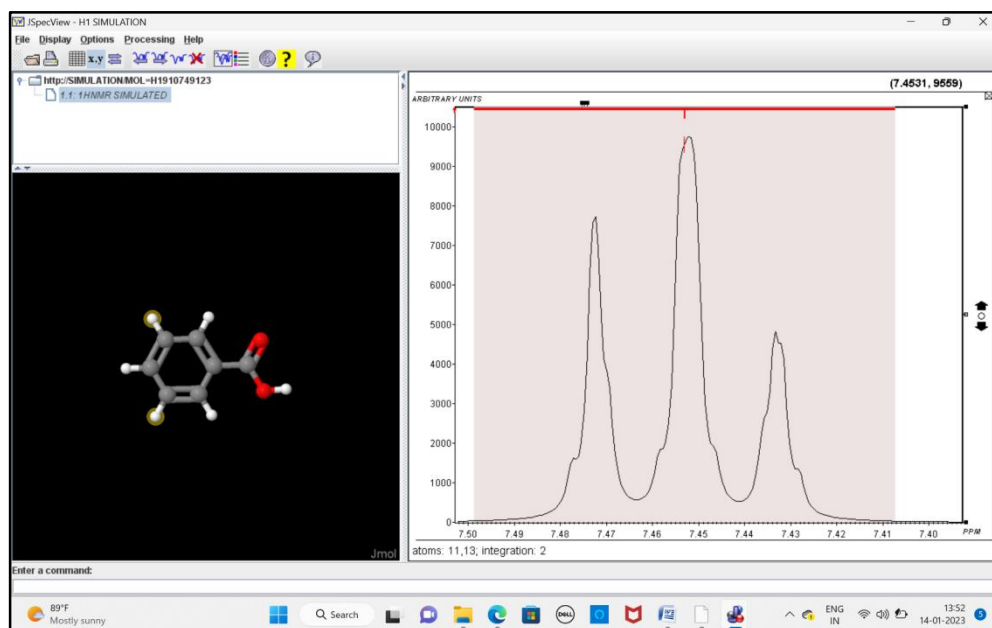


- Triplet peak was obtained for the para hydrogen atom as they have two equivalent hydrogen in its neighbouring position
- The peak position was at 7.642 PPM

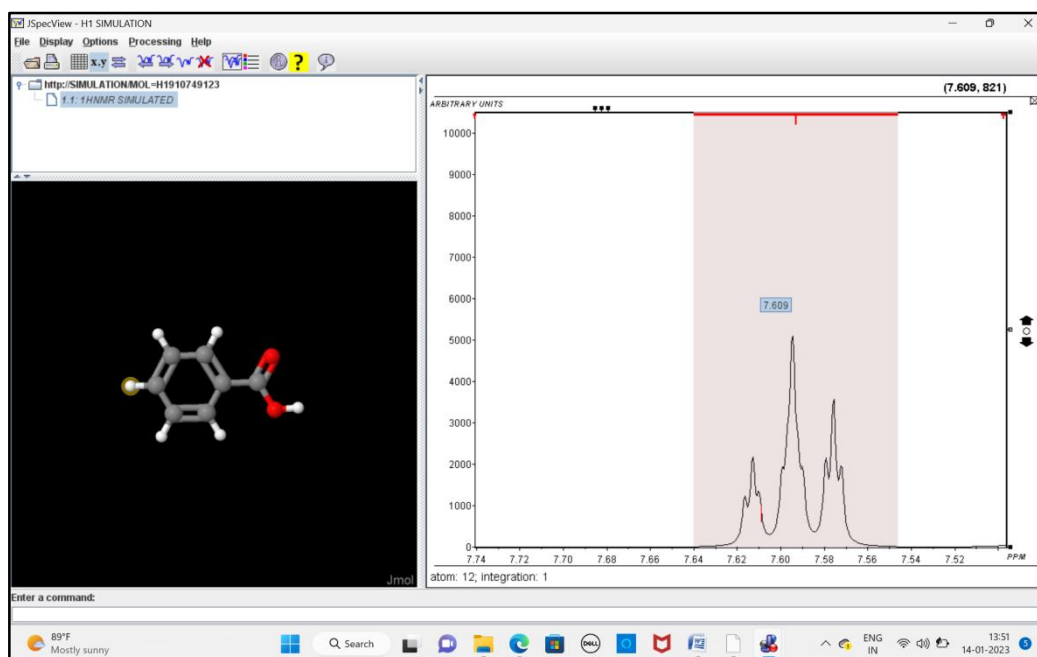
6.1.1 Interpretation of ^1H NMR of Benzoic acid molecule obtained from JSPEC view of Jmol



- Doublet peak was obtained for both the ortho hydrogen atoms as they have only one equivalent hydrogen in their neighbouring position
- Position of the peaks were 8.014 and 8.035 PPM



- Triplet peak was obtained for the above highlighted hydrogen atom as they have two equivalent hydrogen in its neighbouring position
- The peak position was at 7.595 PPM

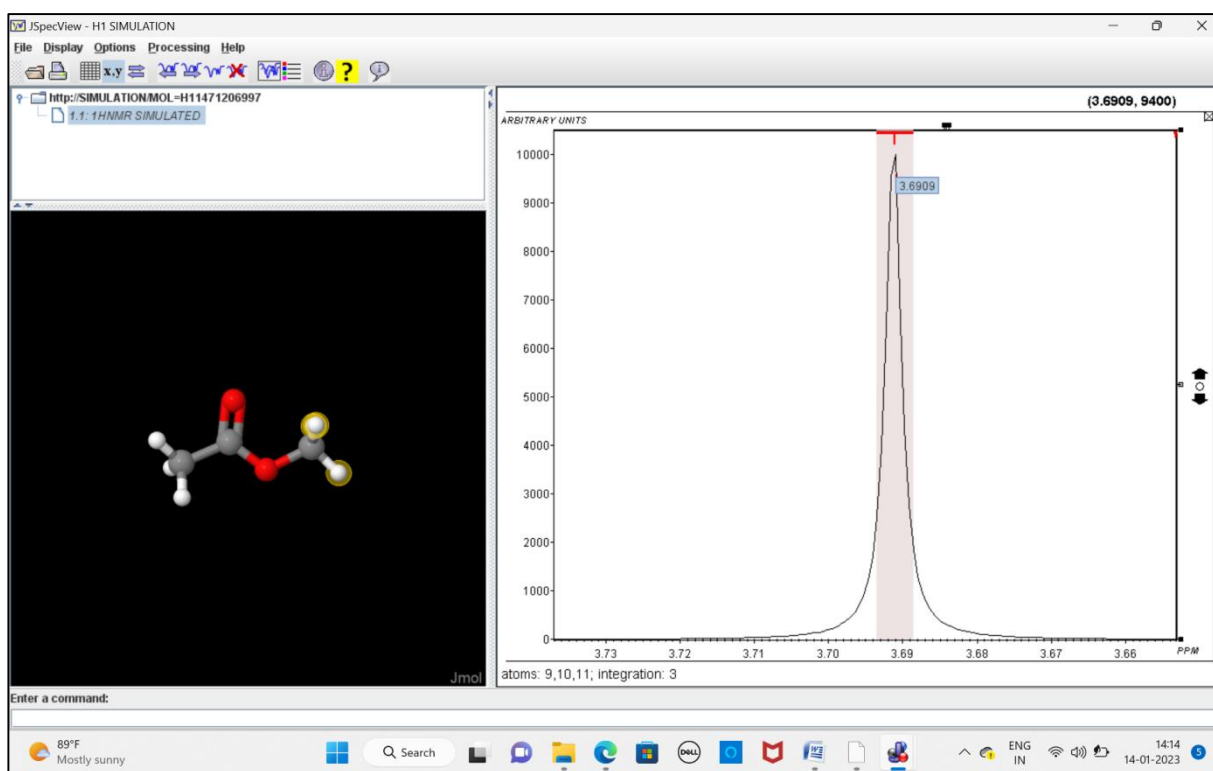


- Triplet peak was obtained for the above highlighted hydrogen atom as they have two equivalent hydrogen in its neighbouring position
- The peak position was at 7.457 PPM

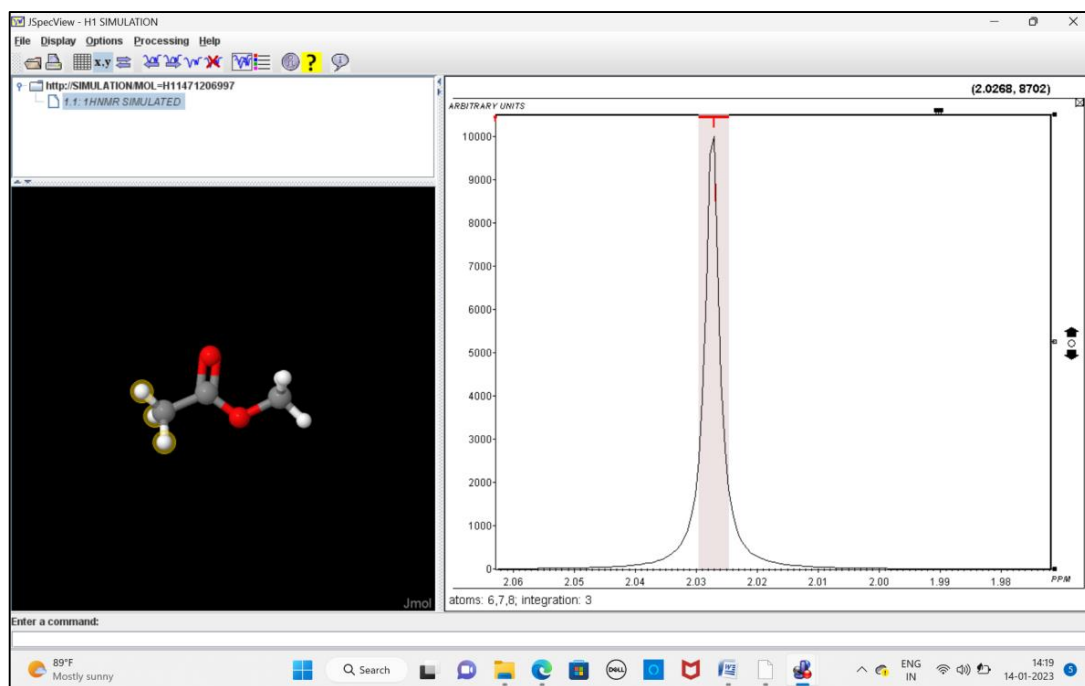
6.1.2 Comparison of ^1H NMR spectra of Benzamide and Benzoic acid:

- The peaks obtained for benzamide were in the range of 7-7.6 PPM due to the influence of the amide group
- The peaks obtained for the product benzoic acid were in the range similar to that of the benzamide but the peaks corresponding to the hydrogen's next to the carboxylic group in the benzoic acid was observed around 8 PPM due to the influence of $-\text{COOH}$ group
- The shift in the peak value suggest that the product formed is having a carboxylic acid group

6.2 Interpretation of ^1H NMR of methyl acetate molecule obtained from JSPEC view of Jmol

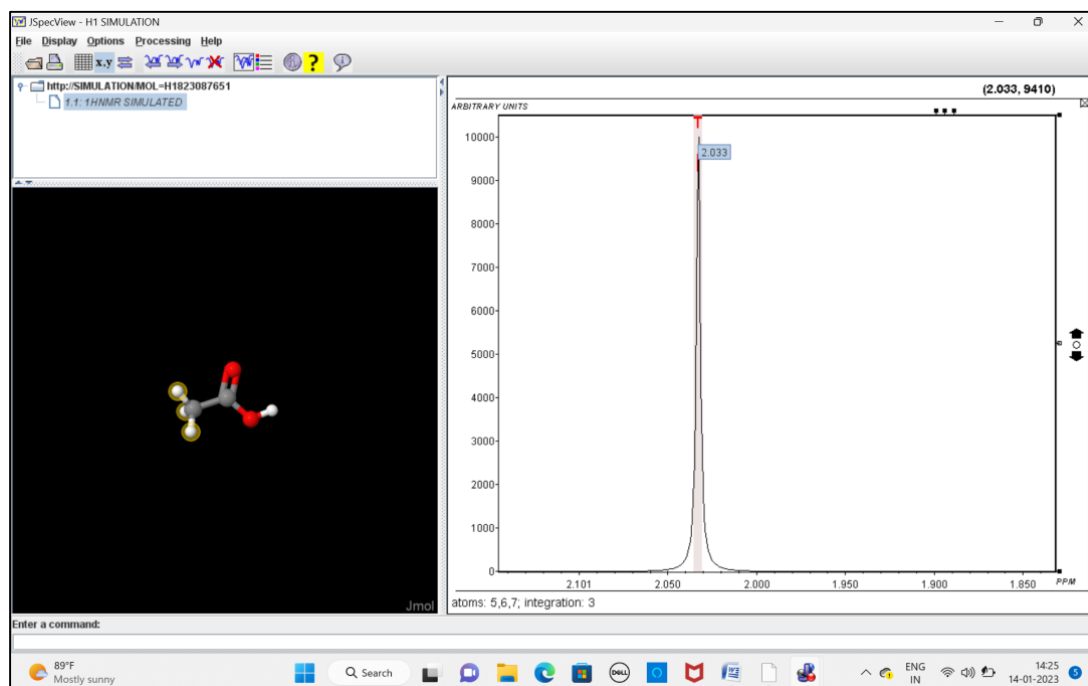


- Singlet was obtained for the protons highlighted above as they don't have any protons in the neighbouring carbon
- The peak value 3.69 suggest that it is a methoxy proton



- A singlet was obtained for the highlighted protons as they don't have any protons in the neighbouring carbon
- The peak value 2.02 suggest that it is a methyl proton

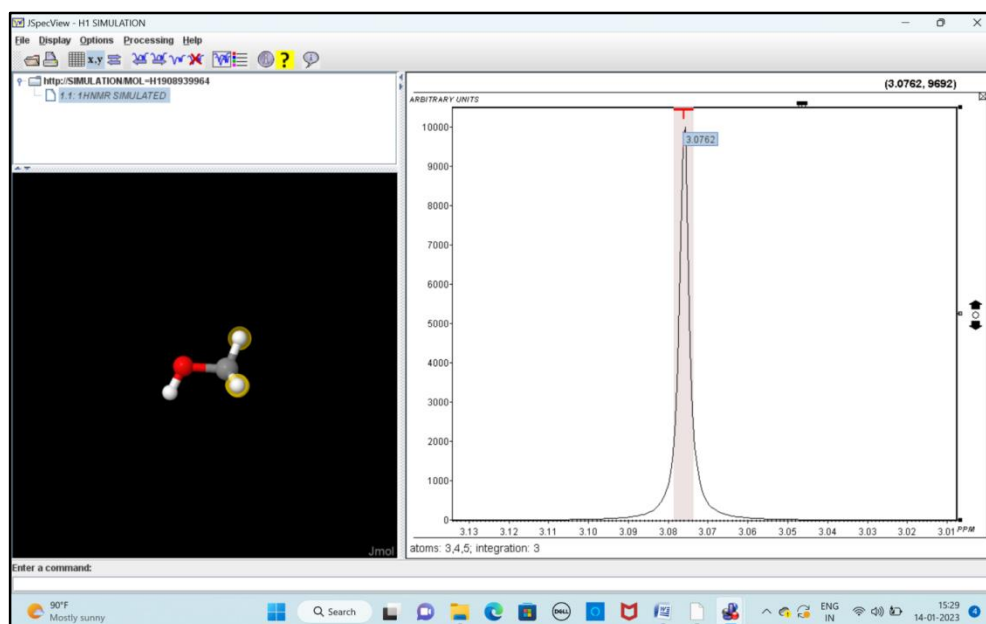
6.2.1 Interpretation of ¹H NMR of acetic acid molecule obtained from JSPEC view of Jmol



- Acetic acid molecule shows a single peak at 2.03 for the methyl protons

- Only a singlet peak was obtained as no neighbouring protons were in the molecule

6.2.2 Interpretation of ¹H NMR of Methyl alcohol molecule obtained from JSPEC view of Jmol



●A

single-singlet peak was obtained for the protons highlighted above as they don't have any other protons in the neighbouring carbon

- The peak value 3.07 suggest that it is a methoxy proton

6.2.2 Comparison of ¹H NMR spectra of methyl acetate and acetic acid:

- In the reactant molecule methyl acetate two singlet peaks were obtained corresponding to methyl proton and methoxy proton
- In case of the product acetic acid only a single peak corresponding to the methyl proton alone obtained which confirms that the methoxy group was not present in the product acetic acid
- The another product methyl alcohol shows a singlet peak at 3.07 which corresponds to the methoxy proton and it suggests that the methoxy group from methyl acetate was broken to form the methyl alcohol molecule

7. Conclusion

The hydrolysis process involves the breakdown of molecules into smaller units. In this project we hydrolysed Benzamide molecules and the product obtained was Benzoic acid. The amide group was broken and converted to carboxylic acid group with the evolution of ammonia. When methyl acetate was hydrolysed it was broken down into acetic acid and methanol molecules.

Acknowledgement

We wish to express our gratitude to our KARE Management for providing the facilities for fabrication. We also wish to express our heartfelt thanks to Dr. Koteswara Rao Anne, Director Academics, Dr.N.Rajini, Dean Freshman Engineering and Dr. P. Sivaranjana project guide for their encouragement and support.