

Establishing a Spectroscopic Analysis Procedure for Identifying the Molecular Structure of Organic Compounds to Enhance Chemistry Students' Problem-Solving Skills

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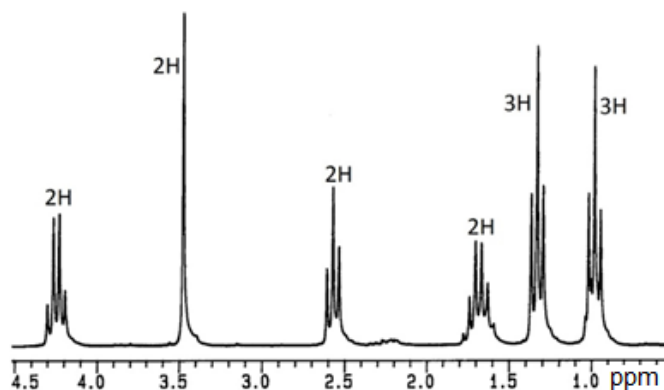
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Received December 01, 2024; Revised January 01, 2025; Accepted January 08, 2025

Abstract This study aims to guide students in quickly determining the structure of organic compounds based on spectral data obtained from spectral databases. We have established a spectral analysis process that combines mass spectrometry (MS), infrared spectroscopy (IR), ultraviolet-visible spectrum (UV-Vis), and nuclear magnetic resonance spectroscopy (NMR) spectral interpretation techniques to determine the molecular structure of common organic compounds. This process is applied to third-year chemistry education majors to enhance their problem-solving skills in spectral analysis for determining the structure of organic compounds. The research results show that the new process has improved the accuracy and reduced the time for spectral analysis to determine the structure of organic compounds, which is considered a challenging task for chemistry students. Participating students have shown significant progress in improving their problem-solving skills by analyzing spectral data to predict the structure of the target organic compound. This new spectral analysis process not only provides an effective tool for determining the molecular structure of organic compounds but also plays a significant role in chemistry student training, helping them develop the necessary skills to solve complex problems in the field of chemistry.



Keywords: Mass spectrometry, infrared spectroscopy, ultraviolet-visible spectrum, nuclear magnetic resonance spectroscopy, organic compound molecular structure, problem-solving skills

Cite This Article: Cao Cu Giac, and Tran Van Thanh, "Establishing a Spectroscopic Analysis Procedure for Identifying the Molecular Structure of Organic Compounds to Enhance Chemistry Students' Problem-Solving Skills." *World Journal of Chemical Education*, vol. 13, no. 1 (2025): 1-6. doi: 10.12691/wjce-13-1-1.

1. Introduction

Reading and analyzing MS, IR and NMR spectra is an indispensable skill for third-year chemistry students when studying organic chemistry. Each type of spectrum provides specific information about the molecular structure of a

compound, helping us to accurately determine the composition and bonding of atoms in the molecule.

1.1. Mass Spectrometry

(i) Principle: Compound molecules are ionized and fragmented into smaller ions. MS spectrum shows the mass of parent ions and fragment ions. [1]

(ii) Information provided: [2]

- Molecular mass: The M^+ peak shows the molecular mass of the compound.

- Molecular formula: Based on the M^+ peak and isotopes, the approximate molecular formula can be deduced.

- Molecular structure: Fragment ions provide information about functional groups and bonds in the molecule.

(iii) Spectrum reading techniques: [3, 4]

- Identify the M^+ peak: The peak with the highest intensity and usually the peak with the highest mass.

- Analyze fragment ions: Identify fragment ions characteristic of different functional groups.

- Use the odd number rule: The M^+ peak of a compound containing an odd number of nitrogen atoms is usually an odd number.

- Compare with standard spectra: Compare the obtained spectrum with standard spectra to determine the structure.

1.2. Infrared Spectrum

(i) Principle: Molecules absorb infrared radiation at characteristic frequencies, corresponding to the vibrations of chemical bonds. [5,6]

(ii) Information provided: [7]

- Functional groups: Absorption bands at characteristic frequency regions for different functional groups (eg: C=O, O-H, N-H).

- Hydrogen bonds: Hydrogen bonds change the position and intensity of absorption bands.

(iii) Spectrum reading techniques: [8]

- Identification of absorption bands: Identify absorption bands with strong intensity and characteristic positions.

- Comparison with frequency table: Compare absorption bands with standard frequency table to determine the functional groups present.

- Fingerprint region analysis: Fingerprint region (below 1500 cm^{-1}) provides detailed information about molecular structure.

1.3. Ultraviolet-visible Spectrum

(i) Principle: Molecules absorb radiation in the ultraviolet-visible region with wavelengths, causing electron excitation and the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions. [9]

(ii) Information provided: [10]

The presence of isolated or conjugated double bonds or aromatic rings in the molecule.

(iii) Spectral reading technique: [11,12]

- Molecules with many conjugated double bonds, the maximum absorption wavelength (λ_{max}) shifts to the longer wavelength region.

- Molecules with isolated double bonds, the maximum absorption wavelength (λ_{max}) shifts to the shorter wavelength region.

- Molecules that do not contain multiple bonds usually do not have strong absorption signals in the UV-Vis region, or if they do, the absorption signal is very weak and located in the shorter wavelength region.

1.4. Nuclear magnetic Resonance Spectra

(i) Principle: Nuclei with non-zero spins (such as ^1H , ^{13}C) when placed in a magnetic field will absorb energy at different frequencies. [13,14]

(ii) Information provided: [15,16]

- Number of protons/carbons: The number of signal peaks indicates the number of non-equivalent protons/carbons in the molecule.

- Chemical environment: The position of the signal peak indicates the chemical environment of the proton/carbon.

- Chemical bonds: The coupling constant provides information about the chemical bonds between protons.

(iii) Spectral reading techniques: [17]

- Counting the number of signal peaks: Counting the number of signal peaks to determine the number of protons/carbons.

- Determining chemical positions: Comparing the chemical positions of the signal peaks with standard values.

- Splitting analysis: Analyzing the splitting of signal peaks to determine chemical bonds.

- Area integration: Integrate the areas of the signal peaks to determine the atomic number ratio of the proton types.

To understand spectra, students need:

- Basic knowledge of organic chemistry: Understand molecular structure, types of bonds, functional groups.

- Understand the working principles of spectroscopic techniques: Know how these techniques provide information about molecular structure.

- Be familiar with frequency tables, chemical values: Use these tables to compare and analyze data.

- Practice a lot: The more you practice, the more familiar you will be with the types of spectra and how to interpret them.

Note: Reading and analyzing spectra is a process that requires patience and experience. Students can start by analyzing simple spectra and gradually move on to more complex spectra.

2. Research Method

The study uses spectral data analysis methods to determine the structure of organic compounds:

- MS: Determine molecular mass and characteristic ions to deduce molecular structure.

- IR: Determine characteristic absorption bands and correlate them with functional groups in the molecule.

- UV-Vis: Determine characteristic absorption bands and calculate related parameters (e.g. maximum absorption, extinction coefficient).

- NMR: Analyze signals and determine the number and type of protons/carbons in the molecule.

3. Results and Discussion

3.1. Analysis Flowchart

To guide students in identifying problems and solving exercises on determining the structure of organic compounds, we propose the following 5-step analysis process:

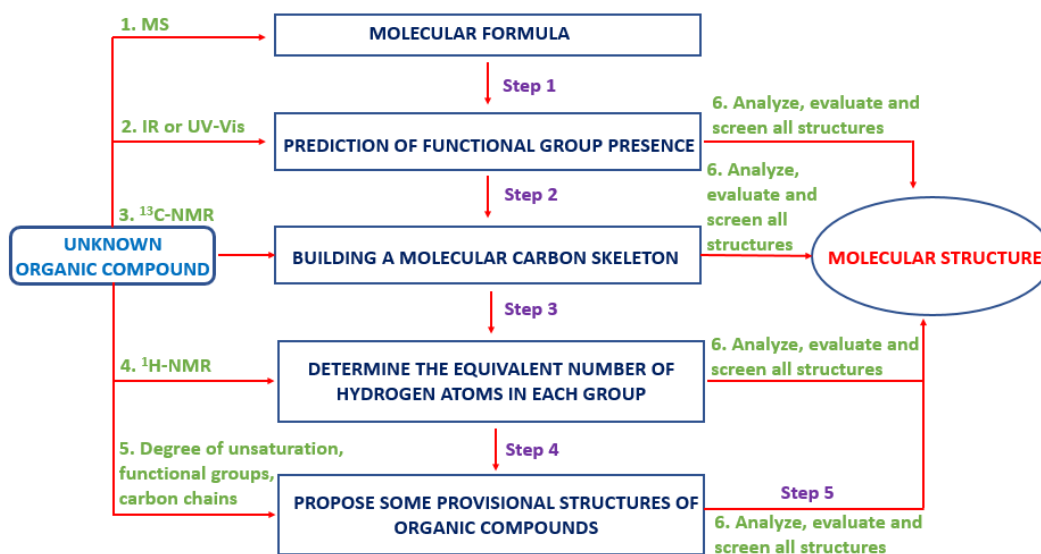


Figure 1. Procedure for analyzing exercises to determine the structure of organic compounds based on spectral data

Step 1. Predict the presence of functional groups

(i) Based on the relative intensity and quantity (m/z) of the mass spectrum (MS), determine the molecular formula of the organic compound.

(ii) From the wavenumber (ν) and the characteristics of the absorption peaks (peaks with strong, medium or weak absorption intensity; peaks with broad-obtuse or sharp-sharp peaks, ...) in the IR spectrum, predict that molecule X may have the following functional groups: OH, C=O, COOH, COOR, NH₂, NH, ...

(iii) From the maximum absorption wavelength ($\lambda_{\max}$) of the electron transitions: $\pi \rightarrow \pi^*$ or $n \rightarrow \pi^*$ in the UV-Vis spectrum, predict the presence of isolated or conjugated double bonds or aromatic rings in the molecule.

Step 2. Construct the carbon skeleton (carbon chain) of the molecule

Based on the number of peak groups and chemical shifts (δ) in each carbon group of the ¹³C-NMR spectrum, determine the number of equivalent or non-equivalent carbon atoms, thereby constructing the carbon skeleton (carbon chain) of the molecule.

Step 3. Determine the number of hydrogen atoms in each group

Based on the intensity ratio, along with the characteristics of each peak type (singlet, doublet, triplet, quarter, ... multiplet peak) and the chemical shift (δ) of the ¹H-NMR spectrum, determine the number of equivalent or non-equivalent hydrogen atoms in each group. In this way, we can assign the number of hydrogen atoms to the carbon skeleton of the molecule (built in Step 2). [18]

Step 4. Propose some provisional structures of the organic compound molecule

By combining the information collected from steps 1, 2 and 3, propose some isomers with the same functional group of the organic compound molecule.

Step 5. Determine the correct structure of the organic compound molecule

Since each substance only establishes a unique set of spectral data, comparing all spectral data: IR, UV-Vis, NMR and especially fragment ions in the MS spectrum, we eliminate inappropriate structures to confirm the correct structure of the organic compound molecule [19].

Note: It is not necessary to follow each step of the procedure completely, depending on whether the molecular structure is simple or complex. If the molecules are simple and to save on spectroscopy costs, we only need to use one of the two NMR spectra or no UV-Vis spectra.

3.2. Some Applications

Problem 1. The elemental analysis results of an organic substance (X) are as follows: %C = 60.76%; %H = 8.86%, the rest is of the element oxygen. The MS, IR, ¹³C-NMR and ¹H-NMR spectra of (X) are shown in Figures 2, 3, 4 and 5. Determine the structural formula of (X).

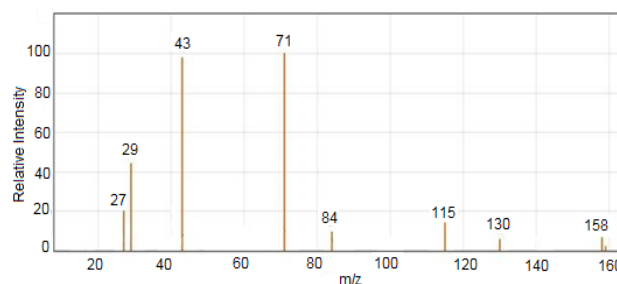


Figure 2. Mass spectrum of an unknown organic compound (X) [20]

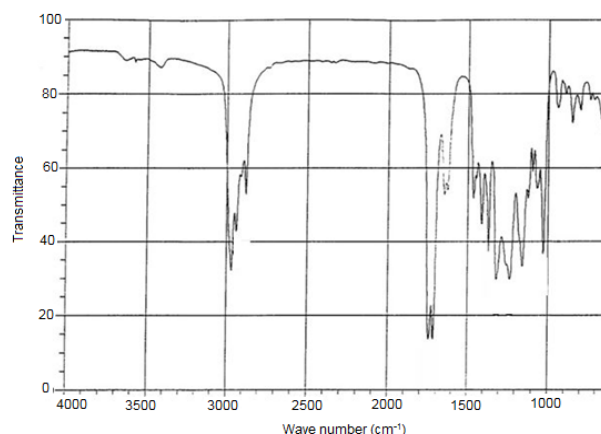


Figure 3. Infrared spectrum of an unknown organic compound (X) [21]

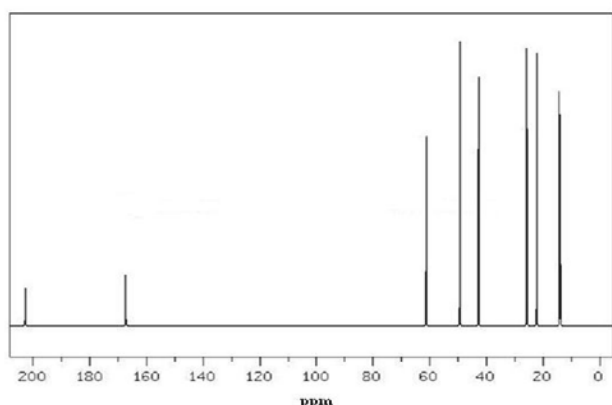


Figure 4. ^{13}C -NMR spectrum of an unknown organic compound (X)

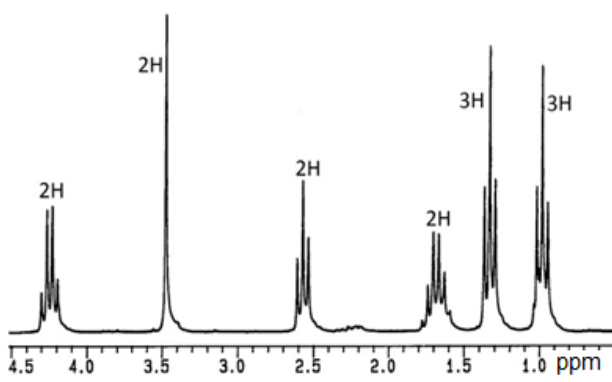


Figure 5. ^1H -NMR spectrum of an unknown organic compound (X) [21]

Guide students to solve problems:

Step 1. Predict the presence of functional groups

(i) Reading the MS spectrum, we see:

- There is a peak with m/z value of 158, next to it is a peak with m/z of 159 with relatively low intensity and no peak has m/z value greater than 159.
- In particular, there are two peaks with m/z of 43 (95%) and m/z of 71 (100%), probably peaks of fragment ions $[\text{C}_3\text{H}_7]^+$ and $[\text{C}_3\text{H}_7 + 28]^+$.
- Therefore, we predict that the m/z value of 158 is of the molecular ion peak ($[\text{M}]^+$) and 159 is of the carbon 13 isotope molecular ion peak ($[\text{M} + 1]^+$). So (X) has a molecular mass of 158 (amu).

$$\begin{cases} \%C = \frac{12x}{158} \times 100 = 60.76 \\ \%H = \frac{y}{158} \times 100 = 8.86 \\ \%O = \frac{16z}{158} \times 100 = 30.38 \end{cases} \rightarrow \begin{cases} x = 8 \\ y = 14 \\ z = 3 \end{cases} \rightarrow \text{X} : \text{C}_8\text{H}_{14}\text{O}_3$$

(ii) The IR spectrum does not have an OH group and has two peaks with strong absorption intensity at $\nu = 1715 \text{ cm}^{-1}$ (C=O group) and $\nu = 1745 \text{ cm}^{-1}$ (-COOR group); The narrow band at $\nu = 3000\text{--}2850 \text{ cm}^{-1}$ is the vibration signal of the C-H single bond.

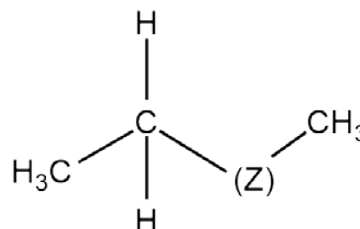
On the other hand, the ^1H -NMR spectrum does not have a proton signal of the CHO group ($\delta = 9.5\text{--}10.1 \text{ ppm}$).

So (X) could be a polyfunctional organic compound containing both a ketone group and an ester group.

Step 2. Construct the carbon skeleton of the molecule

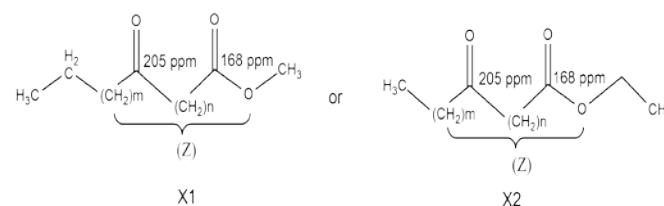
Processing the ^{13}C -NMR spectrum reading results, we get:

- Molecule (X) has 8 non-equivalent carbon atoms (because there are eight different peaks);
- At the two ends of the molecule there are groups: $\text{CH}_3\text{-CH}_2\text{-}$ and $\text{CH}_3\text{-}$ (because there are three peaks located close to the TMS standard substance with small chemical shifts ($\delta = 10\text{--}25 \text{ ppm}$)). So the preliminary structure of (X) is



- Assign $\delta = 205 \text{ ppm}$ and $\delta = 168 \text{ ppm}$ to the two carbon atoms in the C=O group and the COOR group. This is completely consistent with the prediction in the IR spectrum about the presence of these two functional groups.

So the skeleton of molecule (X) can have two following cases:



(where $m + n = 3$)

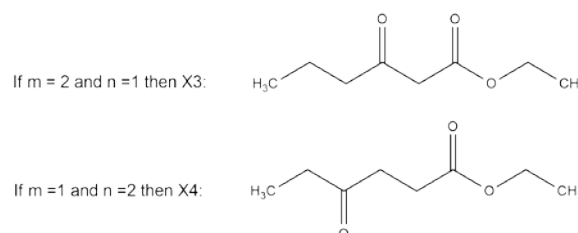
Step 3. Determine the number of hydrogen atoms in each group

Processing the ^1H -NMR spectrum reading results, we get:

If X is an ester of methyl alcohol (structure X1), then the three equivalent protons of the CH_3O group will have the largest chemical shift and the signal will be a singlet. This is not suitable because the ^1H -NMR spectrum of (X) gives a quartet signal of the $\text{CH}_3\text{CH}_2\text{O}$ group. So (X) will have the structure X2.

Step 4. Propose some provisional structures of molecule X

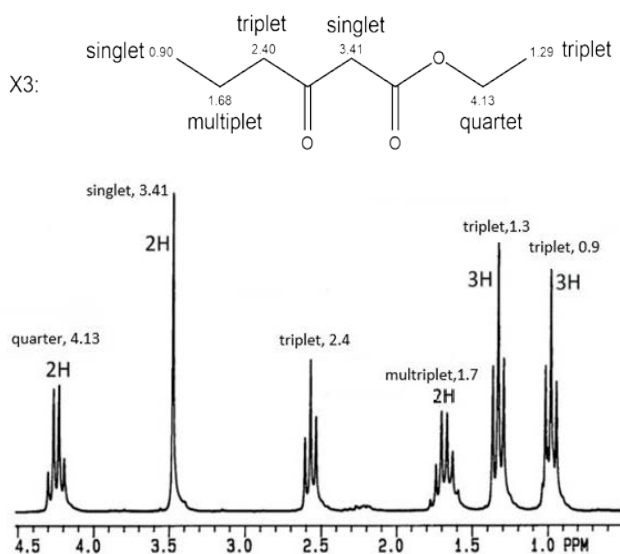
- Unsaturation degree of X: $\Delta = \frac{8 \times 2 + 2 - 14}{2} = 2$
- Because (X) has 2 groups C=O and COOR, (X) has an open chain and there are only sigma bonds between the carbon atoms. So (X) can have the following two structural isomers:



Step 5. Determine the correct structure of molecule X

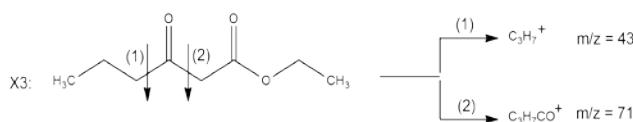
(i) Check the ^1H -NMR spectrum again:

If we assign the chemical shift along with the signal of each peak to the structures X3 and X4, we get:



Thus, in the structure X4 there is no singlet peak and moreover there are 2 triplet peaks next to each other and thus not consistent with the measured spectrum of (X).

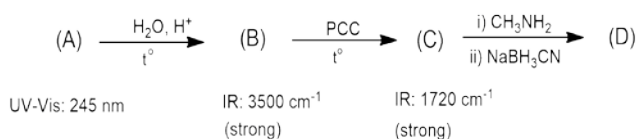
(ii) Recheck the MS spectrum:



As mentioned above, the fragmentation process of substance (X) in the MS method shows 2 peaks 43 and 71, which account for a very large proportion. Compared with the structure of X3 and its possible fragmentation, we see that this structure is completely reasonable.

So through all the steps of spectrum analysis and re-checking the data, we conclude that (X) can only be the compound: Ethyl 3-oxohexanoate.

Exercise 2. Organic compound (D) consists of 3 elements C, H, N, has optical activity and has a molecular mass of 149 (amu). Hydrocarbon (A) is converted into substance (D) through the following 3 steps:



Determine the structures of compounds A, B, C, and D in the above scheme. The ^1H -NMR spectrum of compound (D), in addition to other types, also shows 3 types of aromatic protons. The ^{13}C -NMR spectrum has 8 signals, including 4 signals at $\delta = 120\text{--}140$ ppm.

Guide students to solve problems:

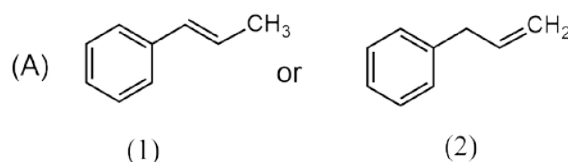
(i) Compound (D) has an odd molecular weight, indicating an odd number of nitrogen atoms in its molecule. Additionally, the ^1H -NMR spectrum of (D) shows 3 types of aromatic protons, and the ^{13}C -NMR spectrum exhibits 8 signals, including 4 signals with chemical shifts between 120–140 ppm, suggesting that (D) has only one substituent on the benzene ring. Therefore, the formula of (D) can be expressed as: $\text{C}_6\text{H}_5\text{RN}_x$ ($x = 1, 3, 5$).

$$M_D = 149 = 12 \times 6 + 5 + R + 14x$$

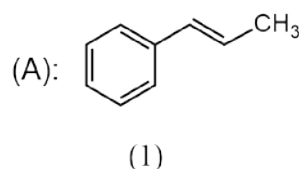
$$\Leftrightarrow 72 = R + 14x \rightarrow \begin{cases} x = 1 \\ R = 58 (\text{C}_4\text{H}_{10}) \end{cases} \rightarrow \text{D: } \text{C}_6\text{H}_5\text{C}_4\text{H}_{10}\text{N}$$

(ii) Analysis of the reaction scheme and IR spectra reveals that compound (B) is an alcohol and compound (C) is a carbonyl compound. Both compounds possess one fewer carbon atom than compound (D) and lack nitrogen. Based on this information, the molecular formulas for compounds (B) and (C) are determined to be $\text{C}_6\text{H}_5\text{C}_3\text{H}_7\text{O}$ and $\text{C}_6\text{H}_5\text{C}_3\text{H}_5\text{O}$, respectively.

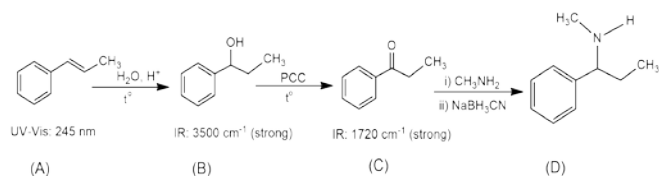
(iii) Given these findings, compound (A) can be inferred to be an aromatic hydrocarbon containing an unsaturated three-carbon side chain. Several possible structures for compound (A) are:



(iv) The UV-Vis spectrum of (A) has $\lambda_{\text{max}} = 245$ nm, corresponding to the transition of the conjugated double bond, so the correct structural formula of substance (A) is



So the structures of substances A, B, C, D in the diagram above are:



4. Conclusion

Through this research, we have successfully established an efficient spectroscopic analysis procedure for determining the molecular structure of organic compounds. This procedure includes UV-Vis, IR, NMR, and MS spectroscopic analysis steps, data processing, and result interpretation.

The application of this procedure in teaching has yielded positive results:

- Students have significantly improved their ability to apply theoretical knowledge to practical work, especially in interpreting spectra and determining molecular structures.
- Students' problem-solving skills have been noticeably enhanced as they have had to deal with complex data and make scientifically-based decisions.
- Students' confidence in approaching chemical research problems has also been boosted.

We have demonstrated that combining UV-Vis, IR, NMR, and MS spectroscopic methods into a unified procedure is an effective way to determine the molecular structure of common organic compounds. However, to apply this procedure to more complex compounds, further research on advanced data processing techniques and supporting software is needed.

Statement of Competing Interests

The Authors have no competing interests.

ACKNOWLEDGMENTS

We would like to thank the School of Education - Vinh University for supporting us in this research.

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