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Experiment No :-> 07

1.) Aim:- To determine the structure of an unknown compound A using spectroscopic data

Given Molecular formula of the given compound is $C_9H_{10}O$

2.) Theory:-

1.) Spectroscopy is the study of the interaction of the E.M. radiation with the matter

2.) Spectroscopy is an important tool for the qualitative and quantitative analysis of an unknown compound

3.) In the modern structure analysis the powerful spectroscopy methods of I.R. and N.M.R play a major role

4.) In order to determine the structure of an unknown compound it may be required to comline a variety of spectroscopic data

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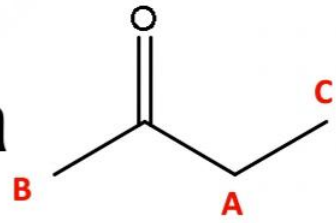
IR frequencies for important Functional Groups

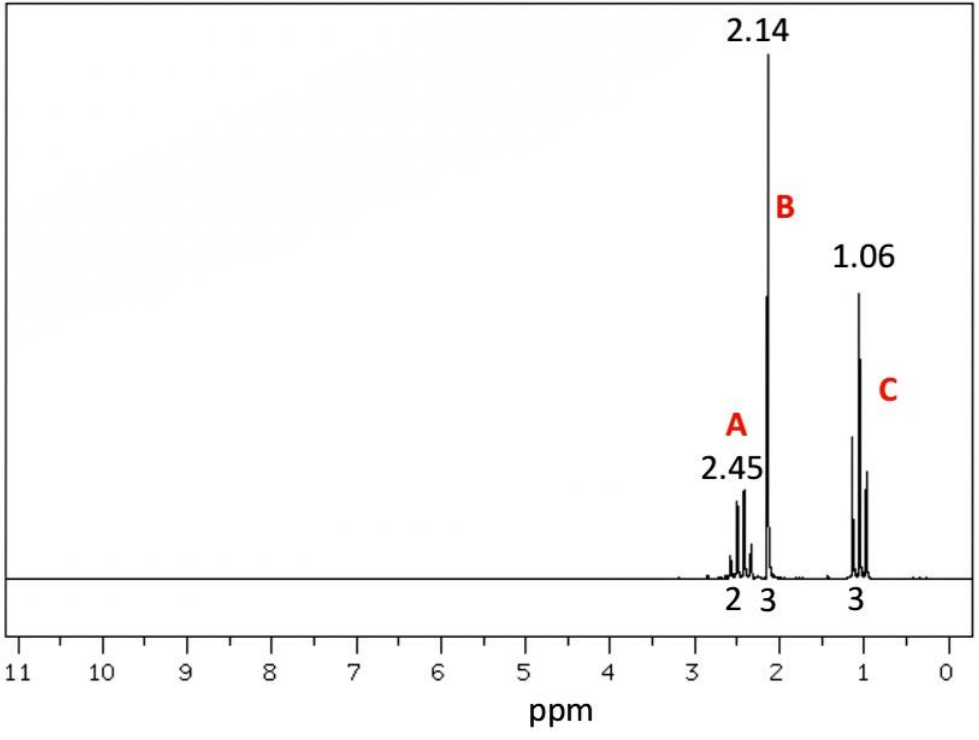
Functional Group	Frequency (cm^{-1})
OH stretch (H_2O)	3700–3100
OH stretch (alcohol)	3600–3200
carboxylic acid OH stretch	3600–2500
N–H stretch	3500–3350
≡C–H stretch	~3300
=C–H stretch	3100–3000
–C–H stretch	2950–2840
–C–H aldehydic	2900–2800
C≡N stretch	~2250
C≡C stretch	2260–2100
C=O aldehyde	1740–1720
C=O anhydride	1840–1800, 1780–1740
C=O ester	1750–1720

Functional Group	Frequency (cm^{-1})
C=O ketone	1745–1715
C=O amide	1700–1500
C=C alkene	1680–1600
C=C aromatic	1600–1400
CH_2 bend	1480–1440
CH_3 bend	1465–1440, 1390–1365
C–O–C stretch	1250–1050
C–OH stretch	1200–1020
NO_2 stretch	1600–1500 and 1400–1300
C–F	1400–1000
C–Cl	800–600
C–Br	750–500
C–I	~500

Example: NMR spectra

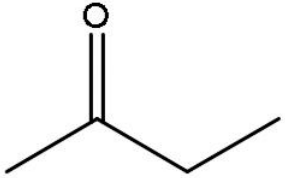


2-butanone



Peak	Integration	Chemical shift (δ)	Multiplicity	Adjacent Hs
A	2	2.45	q	3
B	3	2.14	s	0
C	3	1.06	t	2

Example: IR spectra



2-butanone

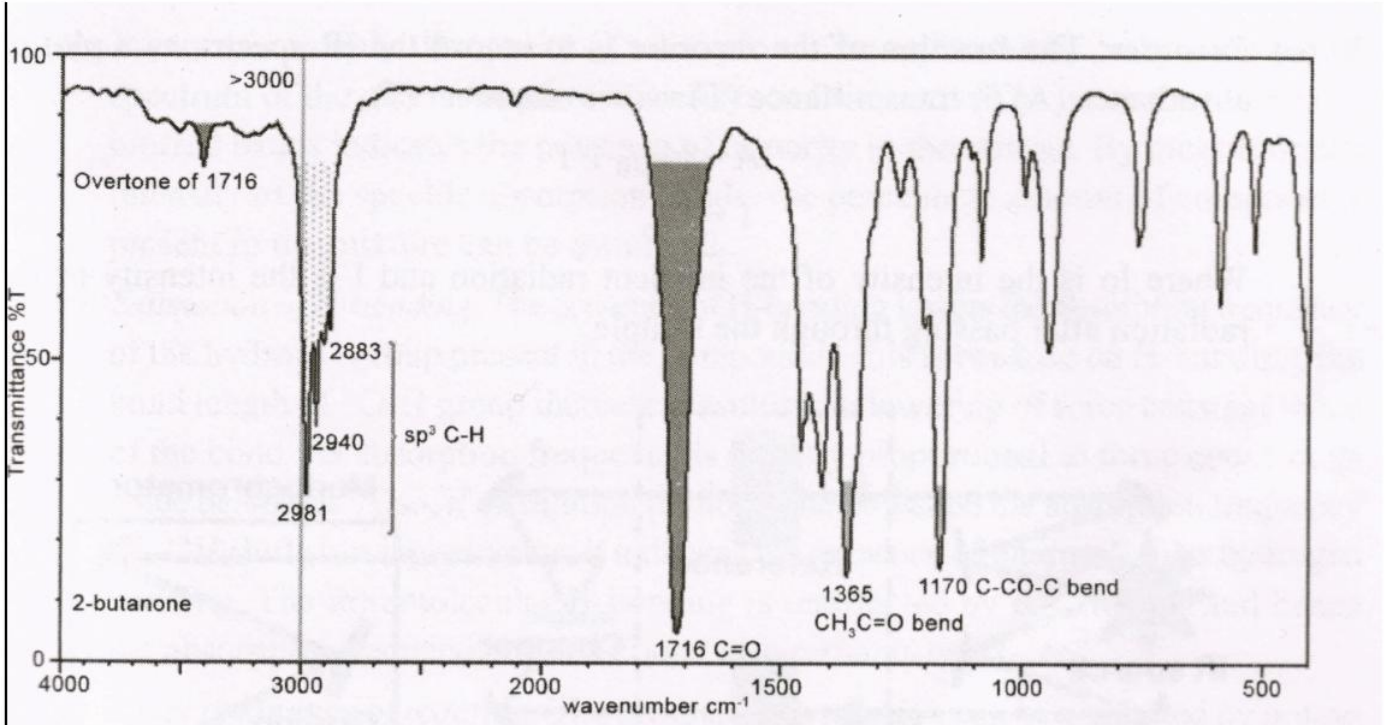


Fig. 2.7: IR spectra of 2-butanone

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✱) Infrared Spectroscopy

1) Infrared spectroscopy is especially useful in identifying the functional groups in an unknown compound. This is a type of functional group spectroscopy

2) Conventionally the I.R. spectrum is a plot of inheritance transmittance (%T) vs wave numbers (cm^{-1})

3) The parts peaks/signals observed in the spectrum can be used for the detection of various important functional groups such as alcohol (-OH), ($-NH_2$), (-CO-) etc in unknown samples

4) The IR spectrum is interpreted by referring to the reference tables containing the info regarding the functional groups & their corresponding wave numbers and the presence of functional groups is analysed accordingly

✱) ^{1}H-NMR Spectroscopy

1) Proton nuclear magnetic resonance is very important & widely used analytical technique

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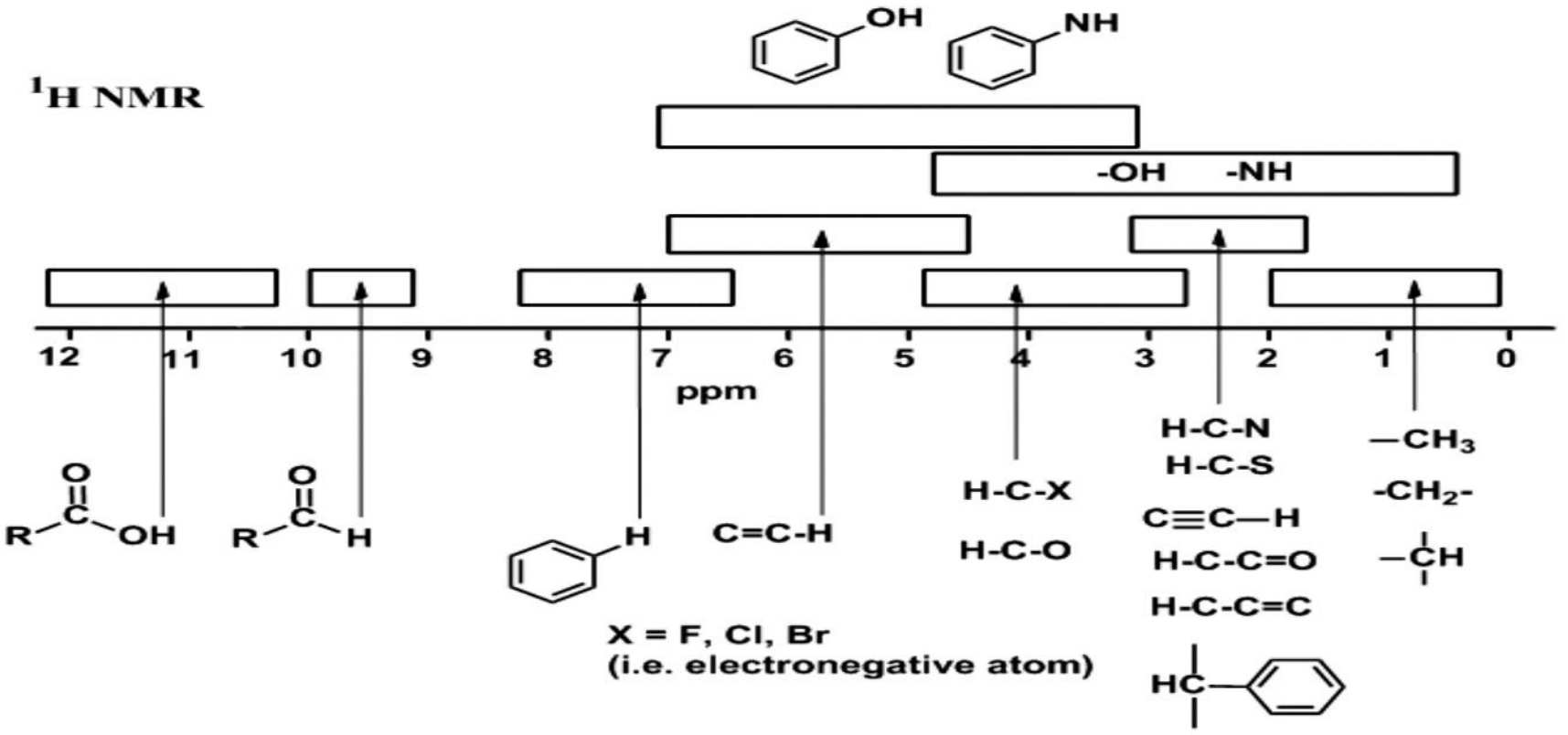
Characteristic ^{1}H-NMR Chemical shifts (δ)



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2) It is primarily used in the characterization of organic molecules

3) H-NMR spectrum is a plot of signal intensity vs chemical shift (δ)

4) The info ~~form~~ from the HNMR spectrum is extracted & is often combined with the info from other spectroscopic techniques such as IR, for the characterisation of unknown compounds

☆ Interpretation of H-NMR spectroscopy spectrum

Important info includes

1) No of signals/peaks (No of H environments)

2) Type of Hs (Aliphatic/Aromatic/Functional) based on chemical shifts

3) No of Hs each type (Integration)

4) No of adjacent Hs (splitting/multiplicity of each signal)

3) Analysis & Interpretation

a) Probable structure for unknown compound A is

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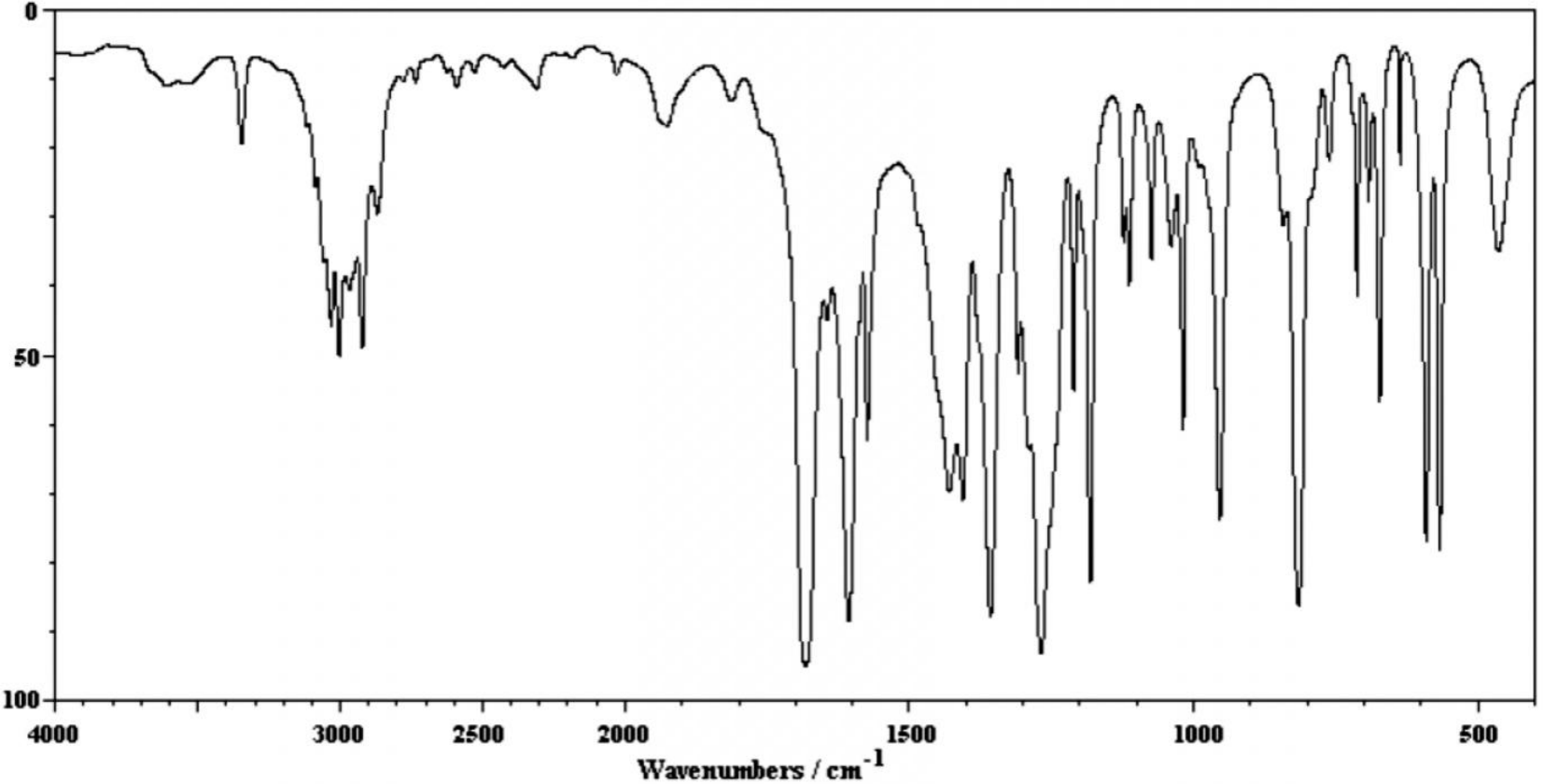
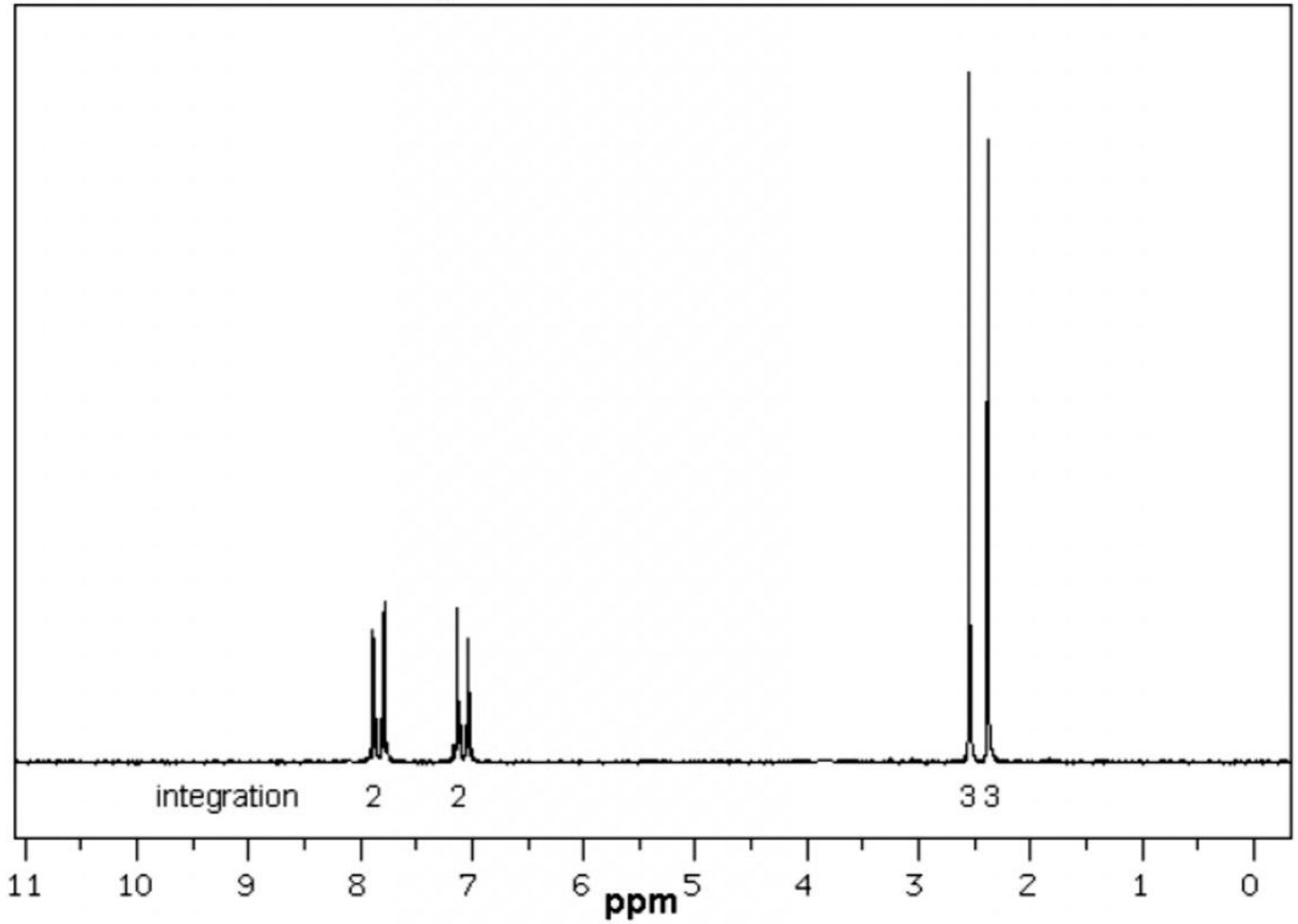
IR Spectrum of the Unknown Compound A



NMR Spectrum of the Unknown Compound A



Observations

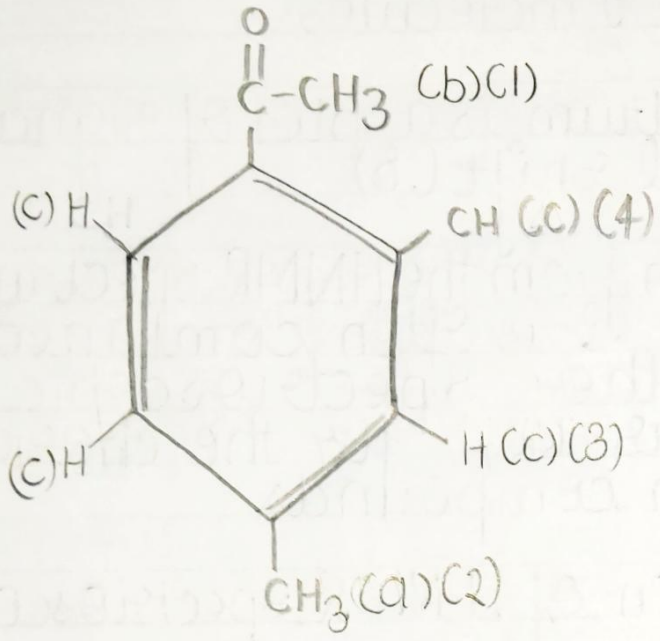
1) IR Spectrum

Sr No	Wave (cm^{-1}) Number	Possible Group	Explanation	Group Present
1	3100-3210	-CH (Alkyl)	Medium & Sharp	-CH (a)
2	2360-2930	=CH (Alkyl/arom)	Medium & Sharp	-CH (c)
3	1690-1750	-C=O	High & Broad	-CH=O (d)
4	1560-1620	-CH (attached to C=O)	High & Broad	-CH (b)

2) NMR-Spectrum

S.No	Chemical Shift	Possible Group	Explanation	Group present
1	2.4-2.6	-CH (attached to C=O)	High & sharp	-CH (1)
2	2.3-2.5	-CH (Alkyl)	High & sharp	-CH (2)
3	6.9-7.1	-CH (Attached to Benzene)	High & sharp	-CH (3)
4	7.3-8.1	-CH (Attached to Benzene near C=O)	Low & sharp	-CH (4)

Therefore, the structure of the compound is



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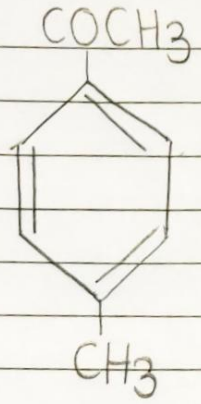
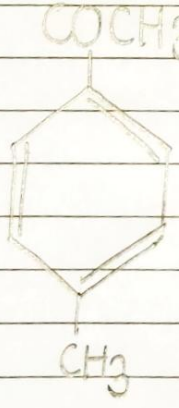


b) Does it satisfy the observation from IR and HNMR spectra → Yes

4) Results:→ Based on the available spectroscopic data (IR & HNMR) the proposed structure of the Unknown compound is //Ans



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