

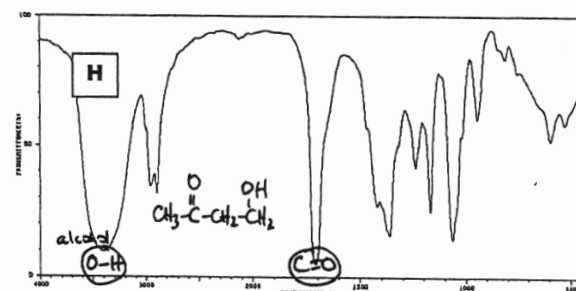
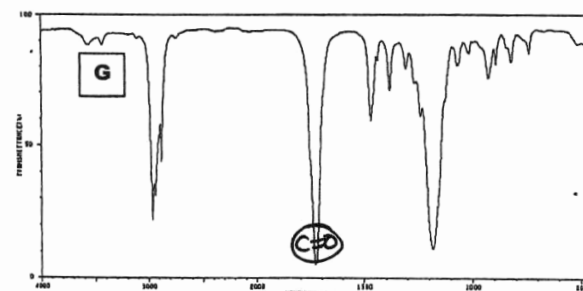
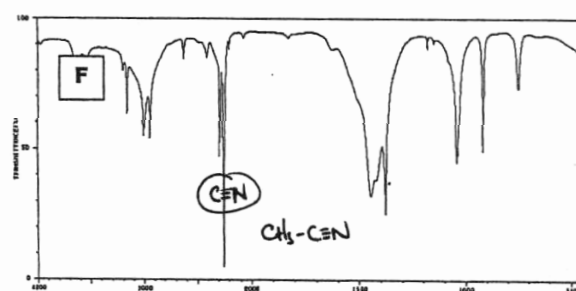
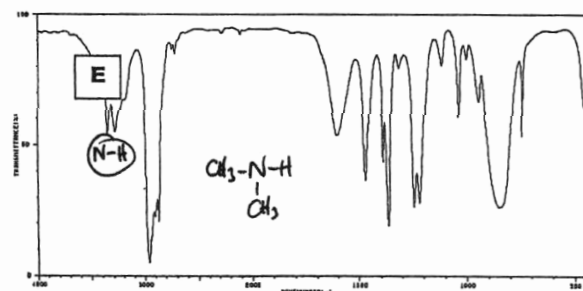
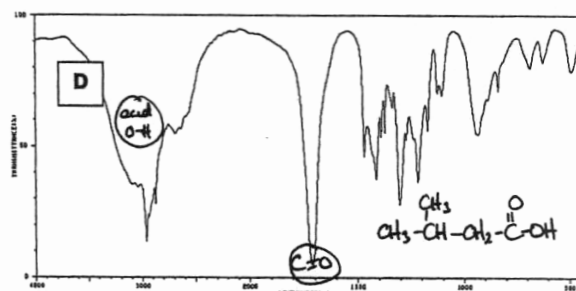
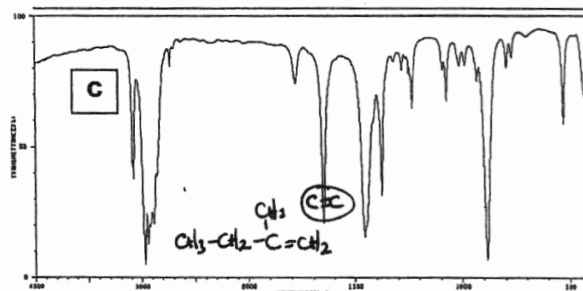
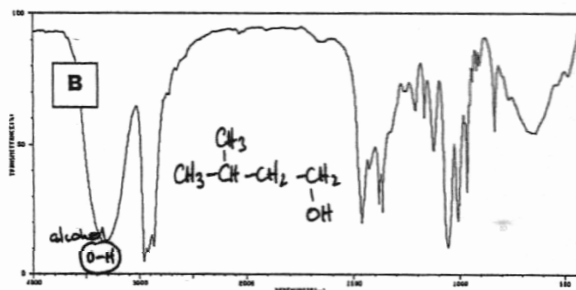
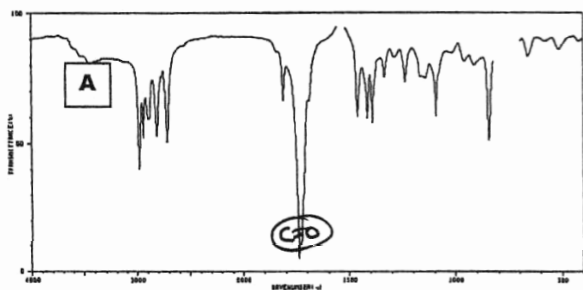
Answers

IR TASK 1

The IR spectra of eight compounds are shown. The compounds are:

- | | | | |
|------------------------------|------------------------|------------------------|-----------------------|
| dimethylamine (E) | 2-methylbut-1-ene (C) | 3-methylbutan-1-ol (B) | 4-hydroxybutanone (H) |
| 3-methylbutanoic acid (D) | butyl methanoate (A/G) | ethanenitrile (F) | propanal (G/C) |

Decide which spectrum belongs to which compound and draw the molecule next to the spectrum. You may not be able to decide between two of the compounds.



IR TASK 2

The IR spectra of six compounds are shown. The compounds are:

butanoic acid (E)

butanone (C)

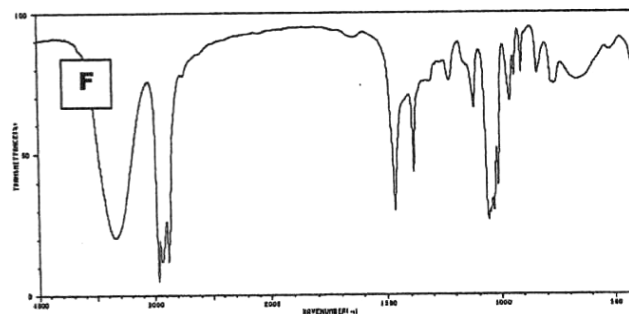
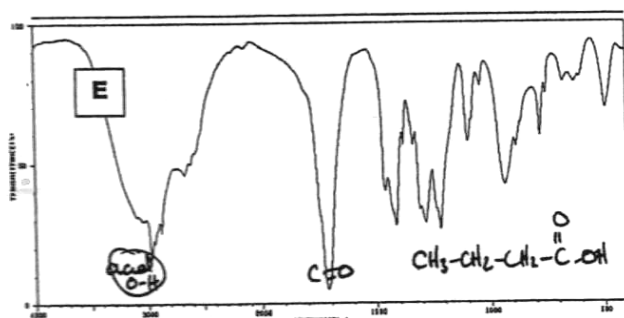
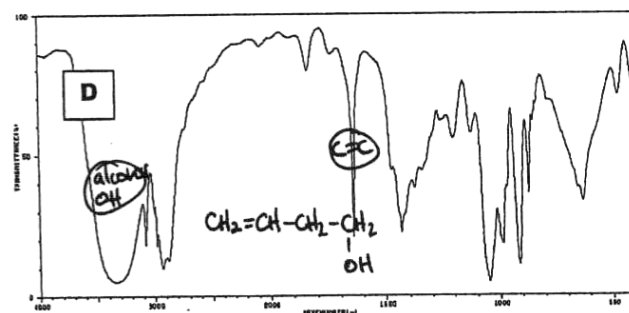
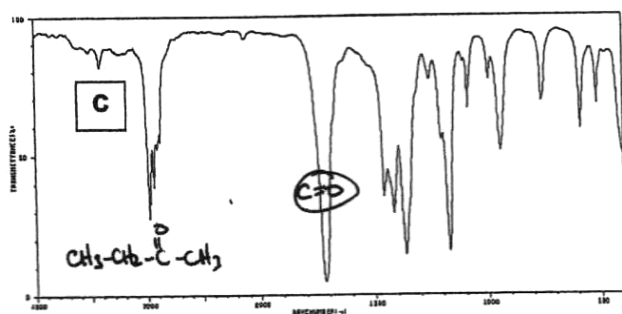
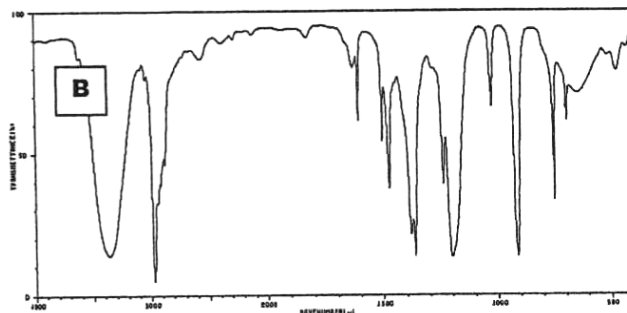
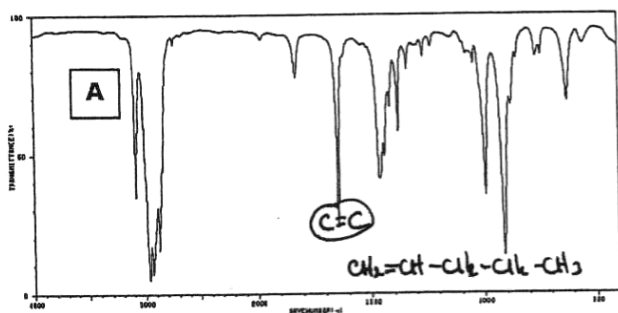
but-3-en-1-ol (D)

2-methylpropan-2-ol (B)/(F)

2-ethylbutan-1-ol (B)/(F)

pent-1-ene (A)

- a) Decide which spectrum belongs to which compound and draw the molecule next to the spectrum. You may not be able to decide between two of the compounds.

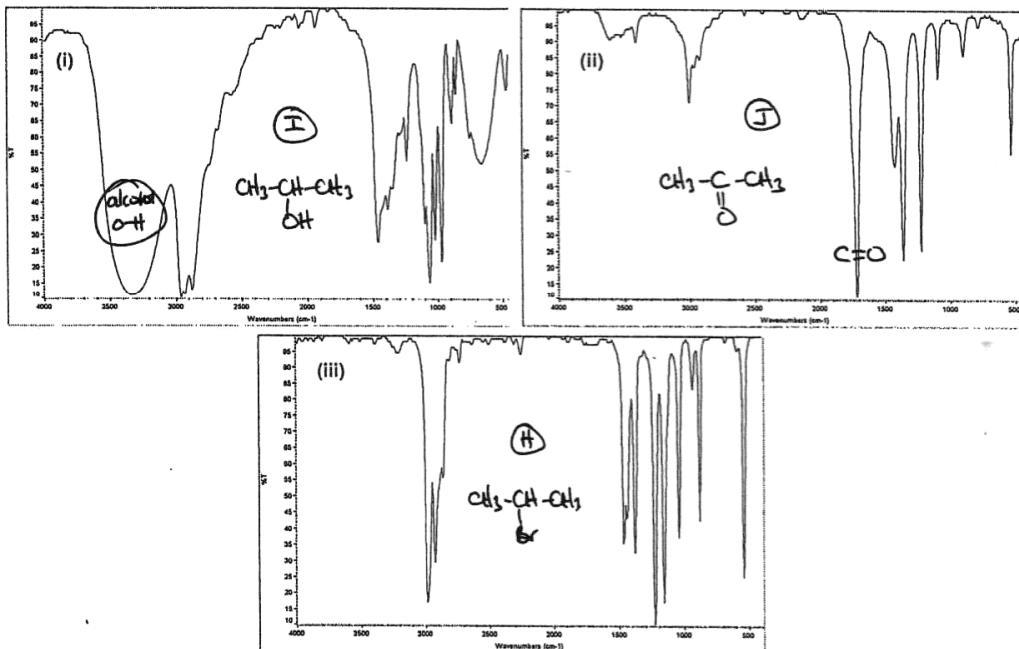
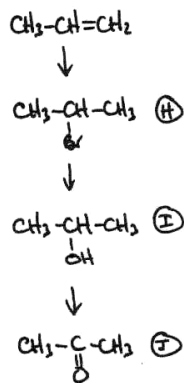


- b) For the compounds that cannot be distinguished by use of functional group signals, explain how the infra-red spectra could be used to distinguish the compounds.

(B)/(F) - use fingerprint region
 - compare to spectrum of known sample
 - look for exact match

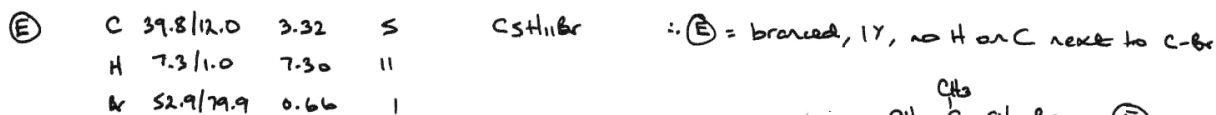
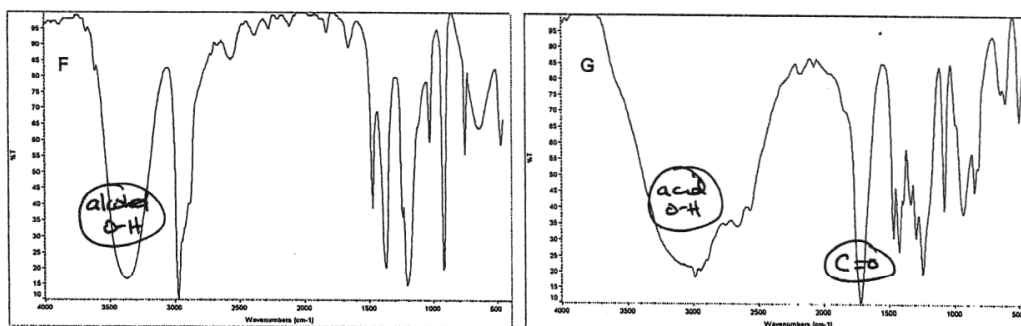
IR TASK 3

- 1) Propene reacts with HBr to form H. H reacts with sodium hydroxide to form I, and I reacts with warm acidified potassium dichromate (VI) to form J. The infra-red spectra of H, I and J are given below, but it does not indicate which is which. Identify the three compounds H, I and J, using the infra-red spectra below, and decide which spectrum belongs to which compound.



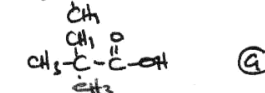
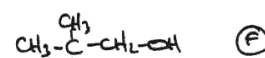
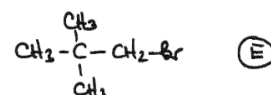
- 2) Compound E, which is a branched chain haloalkane, was found to have the composition by mass of 39.8% C, 7.3% H, and 52.9% Br. There were two peaks for the molecular ions in the spectrum at 150 and 152, of approximately equal intensity. E reacts with sodium hydroxide to form F, whose infra-red spectrum is shown. F does not undergo dehydration with concentrated sulphuric acid.

F reacts further with acidified potassium dichromate (VI) to form G, whose infra red spectrum is also shown. Draw the structures and name E, F and G. Identify the species responsible for the peaks at 150 and 152 in the mass spectrum of E.



(F) alcohol - not dehydrated ∴ no H on C next to OH

(G) carboxylic acid ∴ (F) = 1^o alcohol



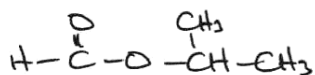
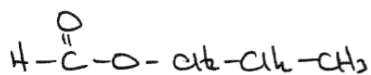
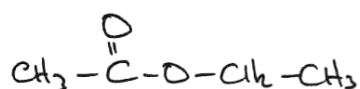
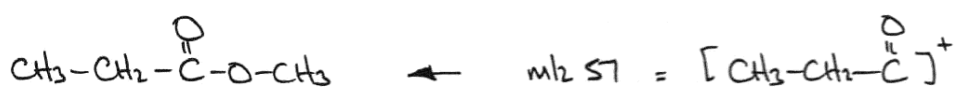
12

MASS SPECTROSCOPY - TASK 1

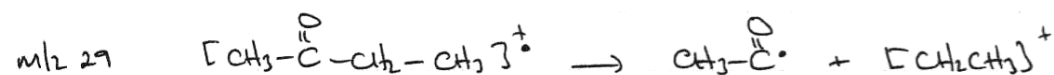
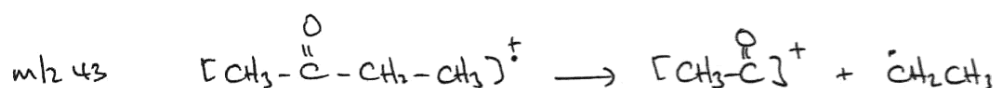
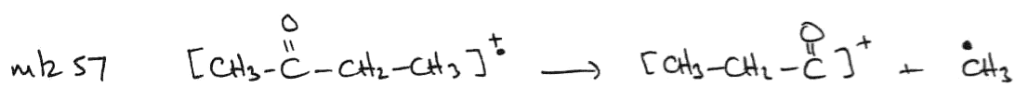
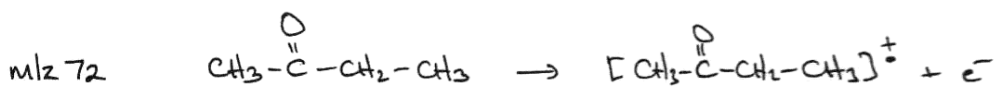
- ①
- a) $[\text{CH}_3\text{-CH}_2\text{-CH}_3]^{\dagger} \rightarrow [\text{CH}_3\text{-CH}_2]^{\dagger} + \dot{\text{C}}\text{H}_3$
m/z 29
- b) $\text{CH}_3\text{-CH}_2\text{-CH}_3 \rightarrow [\text{CH}_3\text{-CH}_2\text{-CH}_3]^{\dagger} + e^{-}$
m/z 44
- c) $\text{CH}_3\text{-}\overset{79}{\text{Br}}\text{-CH}_3 \rightarrow [\text{CH}_3\text{-}\overset{79}{\text{Br}}\text{-CH}_3]^{\dagger} + e^{-}$
m/z 122
- d) $[\text{CH}_3\text{-}\overset{\text{Br}}{\text{C}}\text{-CH}_3]^{\dagger} \rightarrow [\text{CH}_3\text{-CH-CH}_3]^{\dagger} + \dot{\text{B}}\text{r}$
m/z 43
- e) $[\text{CH}_3\text{-}\overset{\text{O}}{\parallel}{\text{C}}\text{-O-CH}_2\text{-CH}_3]^{\dagger} \rightarrow [\text{CH}_3\text{-}\overset{\text{O}}{\parallel}{\text{C}}]^{\dagger} + \dot{\text{O}}\text{CH}_2\text{-CH}_3$
m/z 43
- f) $[\text{CH}_3\text{-}\overset{\text{CH}_3}{\underset{\text{Cl}}{\text{C}}}\text{-CH}_3]^{\dagger} \rightarrow [\text{CH}_3\text{-}\overset{\text{CH}_3}{\text{C}}\text{-CH}_3]^{\dagger} + \dot{\text{C}}\text{l}$
m/z 57
- g) $[\text{CH}_3\text{-CH}_2\text{-}\overset{\text{O}}{\parallel}{\text{C}}\text{-O-CH}_3]^{\dagger} \rightarrow [\text{CH}_3\text{-CH}_2\text{-}\overset{\text{O}}{\parallel}{\text{C}}]^{\dagger} + \dot{\text{O}}\text{-CH}_3$
m/z 57

②

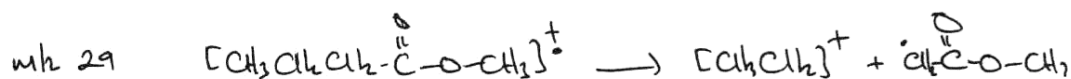
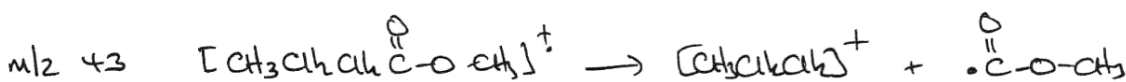
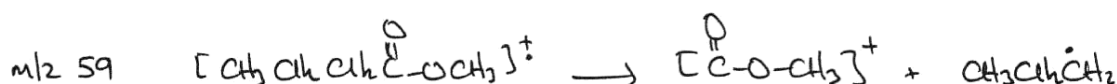
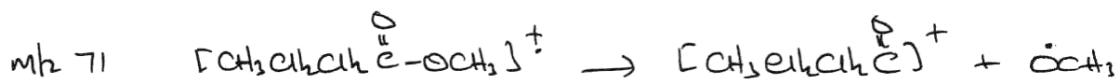
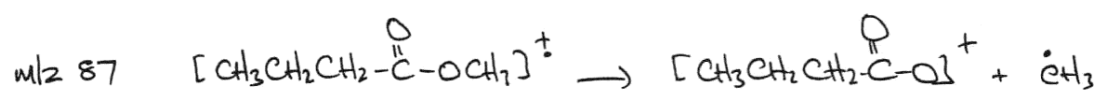
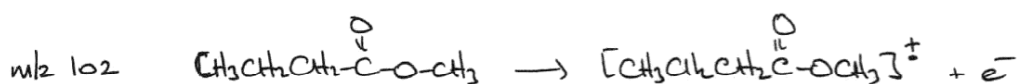
Possible esters:



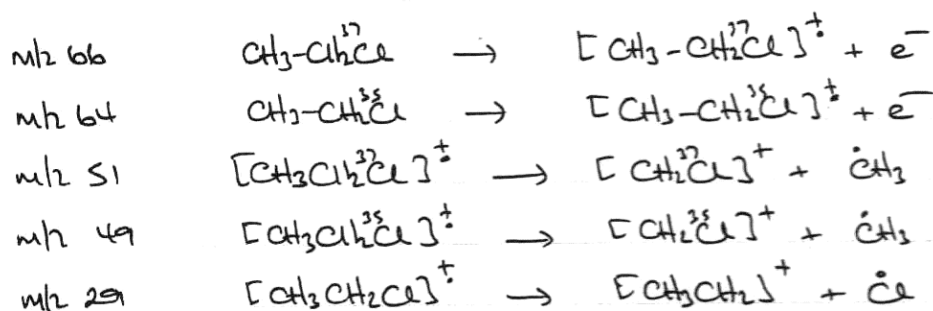
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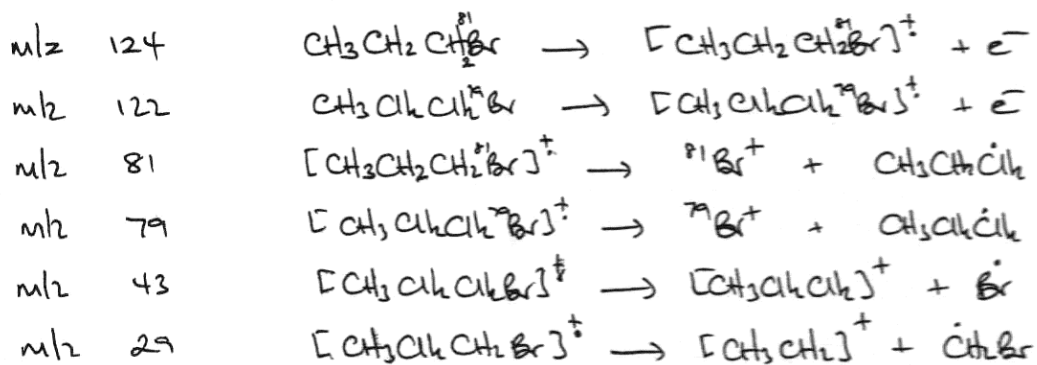
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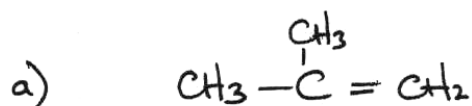
⑤



⑥



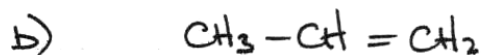
¹H NMR SPECTROSCOPY - TASKS 1/2/5



②

3 : 1

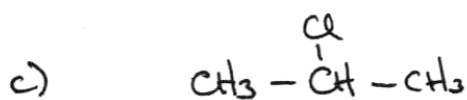
s s



③

3 : 1 : 2

d m d



②

6 : 1

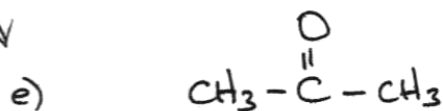
d m



③

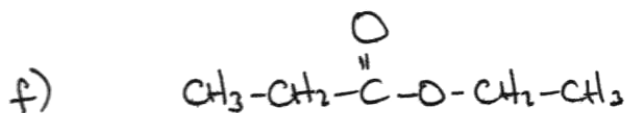
3 : 2

t q



①

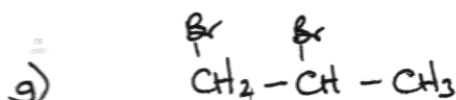
s



④

3 : 2 : 2 : 3

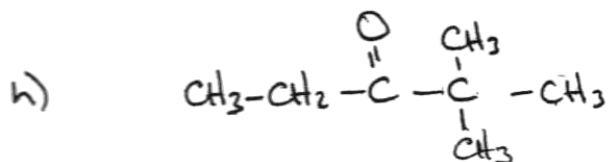
t q q t



③

2 : 1 : 3

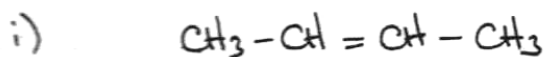
d m d



④

3 : 2 : 9

t q s



②

3 : 1

d q

¹H NMR SPECTROSCOPY - TASK 3

A $0.6 : 3.6 : 1.2 : 1.8 = 1 : 6 : 2 : 3$

B $0.5 : 0.5 : 0.75 : 0.75 = 2 : 2 : 3 : 3$

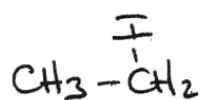
C $0.8 : 2.4 = 1 : 3$

D $1.2 : 1.8 = 2 : 3$

¹H NMR SPECTROSCOPY - TASK 4

a)	$\text{CH}_3 - \text{CH}_2 - \overset{\text{O}}{\parallel}{\text{C}} - \text{OH}$	③	CH ₃	3	0.7-1.2
			CH ₂	2	2.1-2.6
			OH	1	10.0-12.0
b)	$\text{CH}_3 - \text{CH}_2 - \overset{\text{O}}{\parallel}{\text{C}} - \text{H}$	③	CH ₃	3	0.7-1.2
			CH ₂	2	2.1-2.6
			$\overset{\text{O}}{\parallel}{\text{C}} - \text{H}$	1	9.0-10.0
c)	$\text{CH}_3 - \overset{\text{CH}_3}{\underset{ }{\text{CH}}} - \text{CH}_3$	②	CH ₃	6	0.7-1.2
			CH	1	3.1-4.2
d)	$\text{CH}_3 - \overset{\text{CH}_3}{\underset{ }{\text{CH}}} - \text{CH}_2 - \text{CH}_3$	⑤	$\text{CH}_3 - \text{CH}$	6	0.7-1.2
			CH	1	1.4-1.6
			CH ₂	2	1.2-1.4
			$\text{CH}_3 - \text{CH}_2$	3	0.7-1.2
e)	$\text{CH}_3 - \overset{\text{CH}_3}{\underset{ }{\text{C}}} = \text{CH}_2$	②	CH ₃	3	0.7-1.2
			CH ₂	2	4.5-6.0
f)	$\text{CH}_3 - \text{CH}_2 - \overset{\text{O}}{\parallel}{\text{C}} - \text{O} - \text{CH}_3$	③	CH_3CH_2	3	0.7-1.2
			CH ₂	2	2.1-2.6
			CH ₃ O	3	3.1-3.9

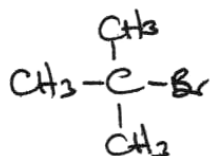
¹H NMR SPECTROSCOPY - TASK 6



②

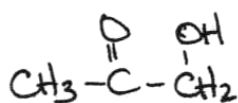
3 : 2
t q

Ⓒ



①

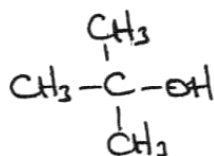
Ⓐ



③

3 : 2 : 1
s s s

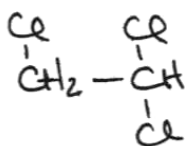
Ⓕ



②

9 : 1
s s

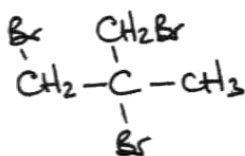
Ⓔ



②

2 : 1
d t

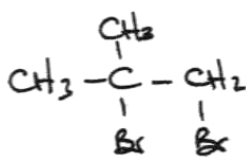
Ⓖ



②

4 : 3
s s

Ⓑ

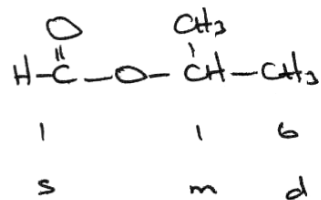
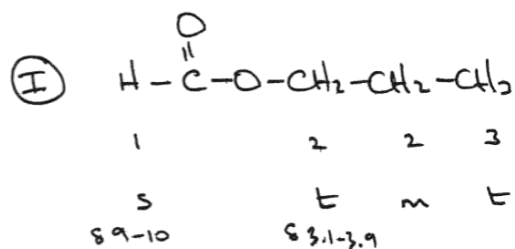
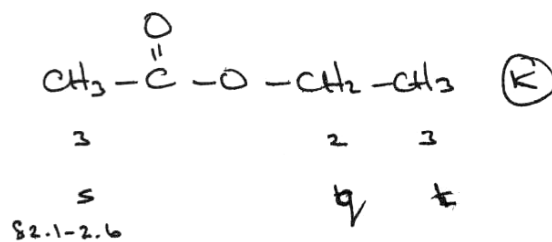
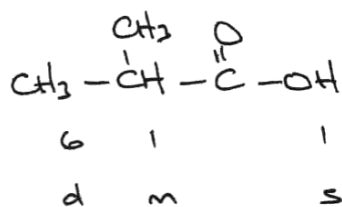
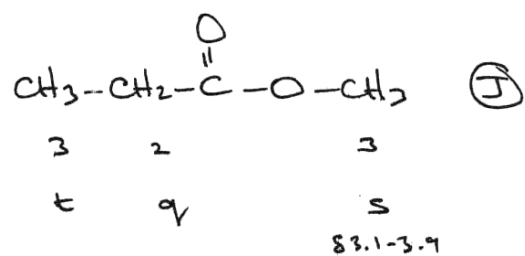
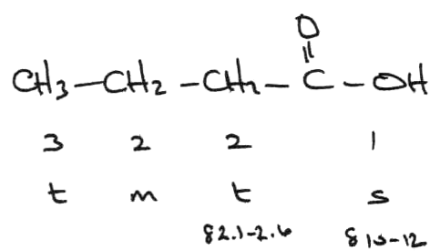


②

3 : 1
s s

Ⓓ

¹H NMR SPECTROSCOPY - TASK 7



¹H NMR – TASK 8

Complete the table about each compound.

1) butanone	$\text{CH}_3-\text{CH}_2-\overset{\text{O}}{\parallel}{\text{C}}-\text{CH}_3$			
protons	CH ₃ -CH ₂	CH ₃	COCH ₃	
relative intensity	3	2	3	
δ / ppm	0.7-1.2	2.1-2.6	2.1-2.6	
multiplicity	triplet	quartet	singlet	
coupled to	CH ₂	CH ₃		

2) methyl ethanoate	$\text{CH}_3-\overset{\text{O}}{\parallel}{\text{C}}-\text{O}-\text{CH}_3$			
protons	CH ₃ -C=O	O-CH ₃		
relative intensity	3	3		
δ / ppm	2.1-2.6	3.7-4.1		
multiplicity	s	s		
coupled to				

3) methylpropane	$\text{CH}_3-\overset{\text{CH}_3}{\text{C}}-\text{CH}_2-\text{CH}_3$			
protons	CH ₃	CH		
relative intensity	9	1		
δ / ppm	0.7-1.2	1.4-1.6		
multiplicity	d	m		
coupled to	CH	3 x CH ₃		

4) 2,2-dimethyl pentan-3-one	$\text{CH}_3-\overset{\text{CH}_3}{\underset{\text{CH}_3}{\text{C}}}-\overset{\text{O}}{\parallel}{\text{C}}-\text{CH}_2-\text{CH}_3$				
protons	CH₃-CH₂	CH	(CH₃)₂	CH₂	CH₃
relative intensity	12	2	9	2	3
δ / ppm	0.7-1.2	2.1-2.6	0.7-1.2	2.1-2.6	0.7-1.2
multiplicity	d	m	s	q	t
coupled to	CH	2 x CH₃		CH₂	CH₃

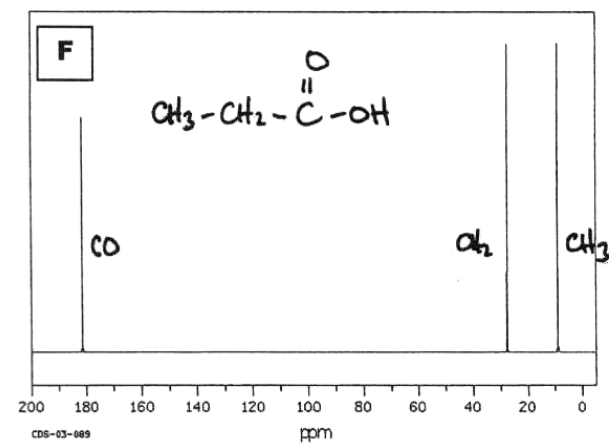
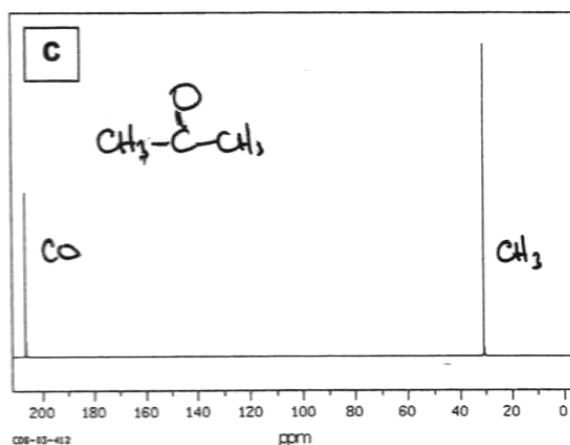
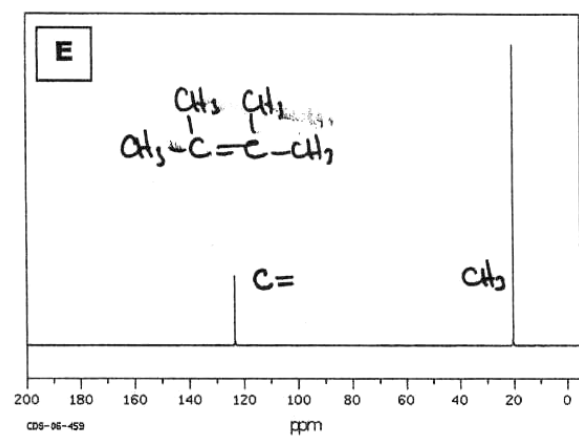
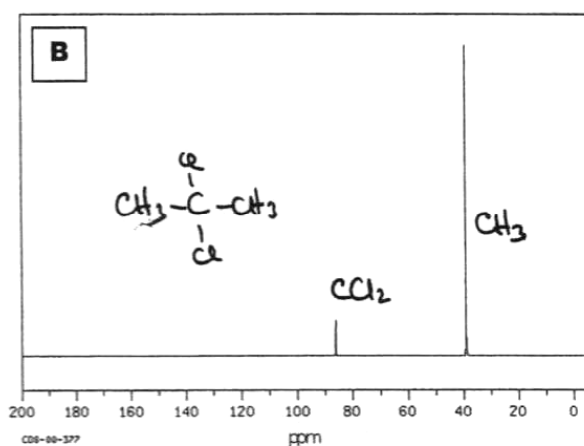
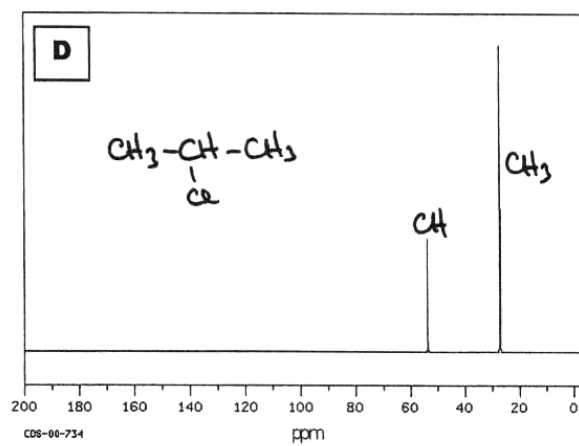
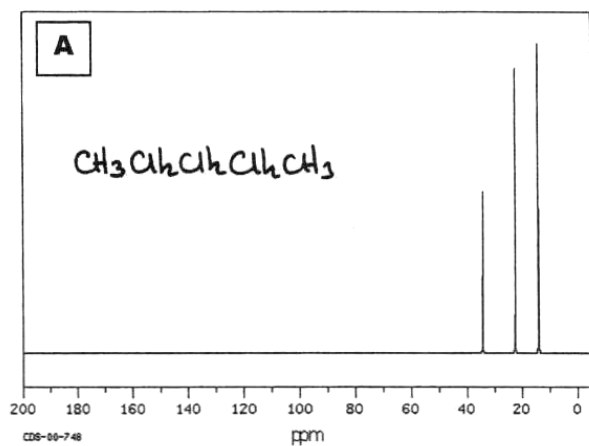
5) ethyl ethanoate	$\text{CH}_3-\overset{\text{O}}{\parallel}{\text{C}}-\text{O}-\text{CH}_2-\text{CH}_3$				
protons	CH ₃ -CO	CH ₂	CH ₂ -CH ₃		
relative intensity	3	2	3		
δ / ppm	2.1-2.6	3.1-3.9	0.7-1.2		
multiplicity	s	q	t		
coupled to		CH ₂	CH ₃		

6) butane	$\text{CH}_3-\text{CH}_2-\text{CH}_2-\text{CH}_3$				
protons	CH ₃	CH ₂			
relative intensity	6	4			
δ / ppm	0.7-1.2	1.2-1.4			
multiplicity	t	q			
coupled to	CH ₂	CH ₂			

¹³C NMR SPECTROSCOPY - TASK 1

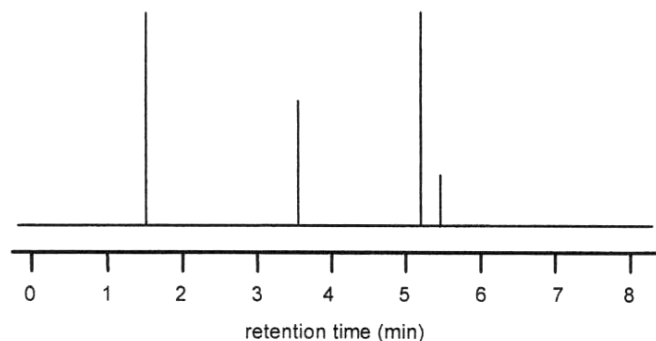
a)	$\text{CH}_3-\overset{\text{CH}_3}{\text{C}}=\text{CH}_2$	(3)	CH_3 C CH_2	5-40 90-150 90-150
b)	$\text{CH}_3-\text{CH}=\text{CH}_2$	(3)	CH_3 CH CH_2	5-40 90-150 90-150
c)	$\text{CH}_3-\overset{\text{O}}{\text{C}}-\text{CH}_3$	(2)	CH_3 C	5-40 10-70
d)	$\text{CH}_3-\overset{\text{O}}{\text{C}}-\text{CH}_2$	(2)	CH_3 CO	20-50 190-220
e)	CH_3-NH_2	(1)	CH_3	25-60
f)	$\text{CH}_3-\text{CH}_2-\overset{\text{O}}{\text{C}}-\text{O}-\text{CH}_2\text{CH}_3$	(5)	CH_3 CH_2 CO CH_2 CH_3	5-40 20-50 160-185 50-90 5-40
g)	$\overset{\text{Br}}{\text{CH}_2}-\overset{\text{Br}}{\text{CH}_2}-\text{CH}_3$	(3)	CH_2Br CH_3	10-70 5-40
h)	$\text{CH}_3\text{CH}_2-\overset{\text{O}}{\text{C}}-\text{O}-\overset{\text{CH}_3}{\underset{\text{CH}_3}{\text{C}}}-\text{CH}_3$	(5)	CH_3 CH_2 CO C CH_3	5-40 20-50 160-185 50-90 5-40
i)	$\text{CH}_3-\text{CH}=\text{CH}-\text{CH}_3$	(2)	CH_3 CH	5-40 90-150

¹³C NMR SPECTROSCOPY - TASK 2



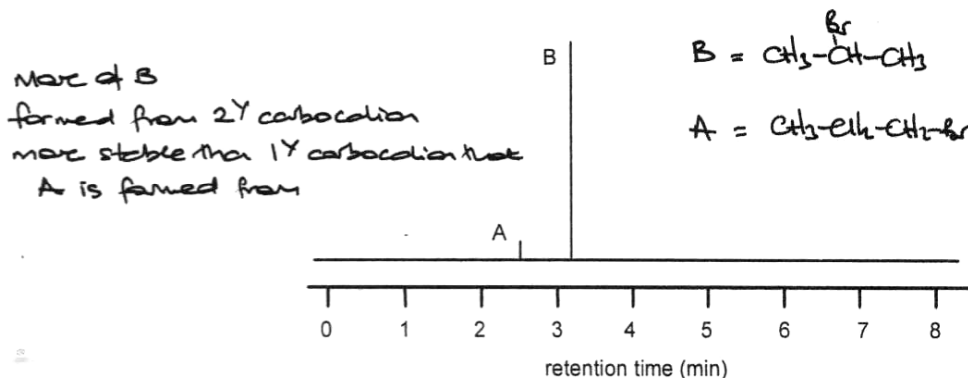
CHROMATOGRAPHY – TASK 1

1) A mixture of volatile liquids was analysed by gas chromatography. The chromatogram is shown.



- How many substances were in the mixture? 4
- What is meant by the term *retention time*? time to pass through column
- What is the stationary phase in gas chromatography? powder coated with non volatile liquid
- What is the mobile phase in gas chromatography? unreactive gas (eg N₂, Ar)
- Why are substances separated by gas chromatography? substances have different relative affinities for stationary + mobile phases

2) Propene was reacted with hydrogen bromide forming a mixture of products. This mixture was analysed by gas chromatography. The chromatogram is shown. Give the structure of both A and B and explain the relative abundance of the two compounds in the mixture.



3) Column chromatography is a very useful of separating substances for analysis.

- What type of mixtures are separated by column chromatography? soluble substances
- What is the stationary phase in column chromatography? powder (eg SiO₂ or Al₂O₃)
- What is the mobile phase in column chromatography? solvent
- Why are substances separated by column chromatography? different relative affinity for mobile + stationary phases of each crystals