Year 12 Instrumental Analysis of Organic Compounds Topic Test

Question 1 (6 marks)

(a) What is the difference between the molecular ion peak and a fragment ion? **2 marks**

(b) What information does the base peak provide in a mass spectrum? **2 marks**

(c) A compound containing chlorine shows two molecular ion peaks at m/z 120 and 122 in a 3:1 ratio. What does this indicate about its isotopic composition? **2 marks**

Question 2 (8 marks)

(a) What is the purpose of the fingerprint region between 1500-500cm^{-1}? **2 marks**

(b) How can you distinguish between an aldehyde and a ketone using IR spectroscopy? **2 marks**

(c) What absorption would you expect for an amide carbonyl compared to an ester carbonyl? **2 marks**

(d) A student claims their compound to be an amide due to the presence of a C=O peak at 1650cm^{-1}. No other peak was present on the spectrum. Is the students claim correct? **2 marks**

Question 3 (7 marks)

(a) Why is tetramethylsilane, TMS, used as a reference standard? **2 marks**

(b) How does electronegativity of nearby atoms influence chemical shift? **2 marks**

(c) Two isomers, propanal and propan-2-one, have the same molecular formula. How would their ^{1}H NMR spectra differ? **2 marks**

(d) A ^{13}C NMR spectrum shows only three signals for a molecule with formula $C_4H_{10}O$. Suggest a possible structure. **1 mark**

Question 4 (7 marks)

(a) What is the basic principle that allows chromatography to separate components of a mixture? **2 marks**

(b) What does retention time (t_r) represent in chromatography? **1 mark**

(c) A chromatogram shows two peaks with very similar retention times. What does this suggest about the separation? **2 marks**

(d) You are separating a mixture of three compounds using HPLC. Increasing the mobile-phase polarity causes all retention times to decrease. Explain why. **2 marks**

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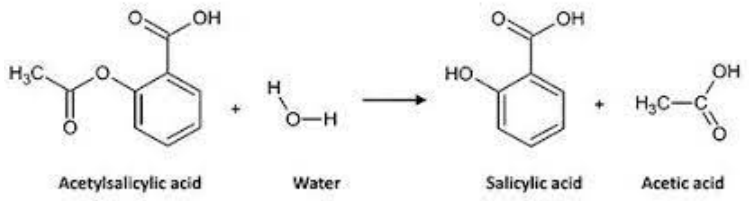
Question 5 (9 marks)

Aspirin, also known as acetylsalicylic acid, is a type of nonsteroidal anti-inflammatory drug that can treat mild to moderate pain, inflammation or fever. When aspirin is exposed to moisture, heat, or alkaline or acidic conditions, it undergoes hydrolysis, breaking down into salicylic acid and acetic acid (ethanoic acid).



A group of students used thin-layer chromatography to identify and separate the active ingredients and possible contaminants in an over-the-counter aspirin tablet. The general purity of the tablet can also be tested in TLC.

Method

Prepare the Sample:

1. Crush the aspirin tablet and dissolve a small amount (~100 mg) in 5 mL of ethanol
2. Filter the solution to remove insoluble fillers

Apply the Sample:

3. Draw the origin baseline about 2 cm from the bottom of the TLC plate
4. Spot the sample on the baseline using a capillary tube
5. Spot a pure sample of acetylsalicylic acid and salicylic acid on the TLC plate and label as reference 1 and 2
6. Allow it to dry and spot again to concentrate

Develop the Chromatogram:

7. Place the paper in the chromatography chamber containing the solvent (ethanol) and cover with a watch glass
8. Let the solvent rise until it's about 2cm below the top of the paper

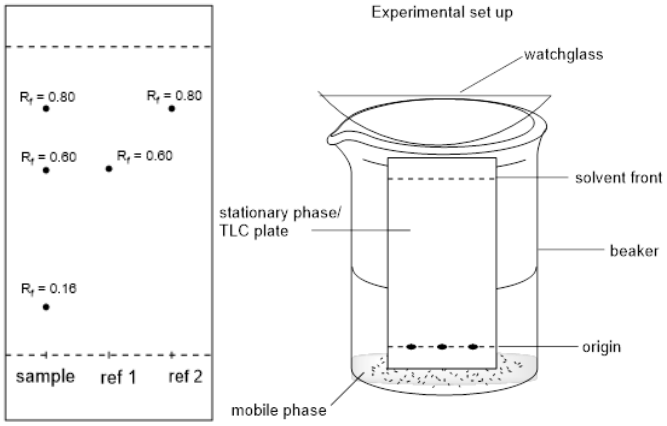
9. Remove and mark the solvent front immediately

10. Allow the TLC plate to air dry

Visualization:

11. Use a UV lamp to view spots. Alternatively, place the dry paper in iodine vapour to stain organic compounds

Results



(a) Based on the TLC plate results, how many components can be identified in the sample of aspirin tablet? **1 mark**

(b) Between acetylsalicylic acid and salicylic acid, identify which components belong to reference 1 and reference 2 on the TLC plate. Justify your answer. **3 marks**

(c) What is the purpose of the watch glass listed in step 7? **1 mark**

(d) Is the tablet of aspirin tested in this experiment pure? Justify your answer referring to the results and chromatography principles. **3 marks**

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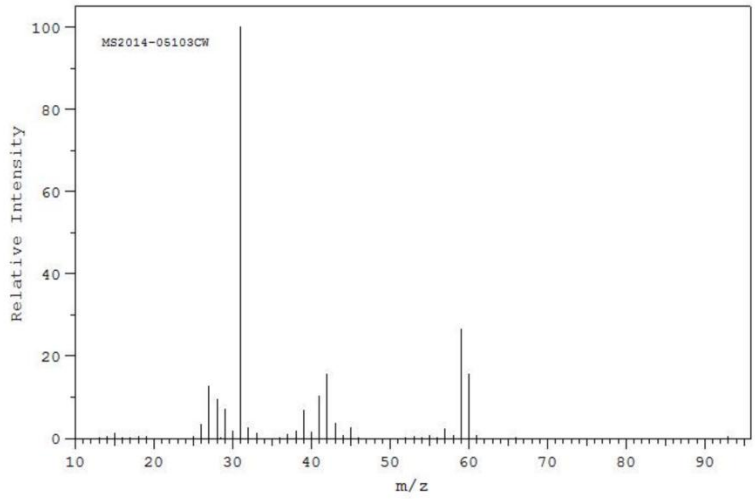
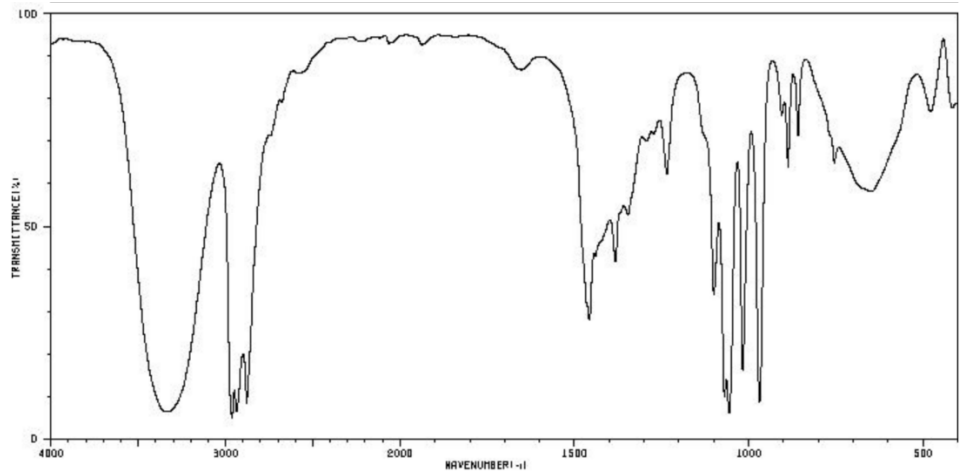
Question 6

An organic compound under analysis has the molecular formula of C_3H_6O. Using the mass, IR and NMR spectra provided, deduce the structure and name of the compound. **4 marks**

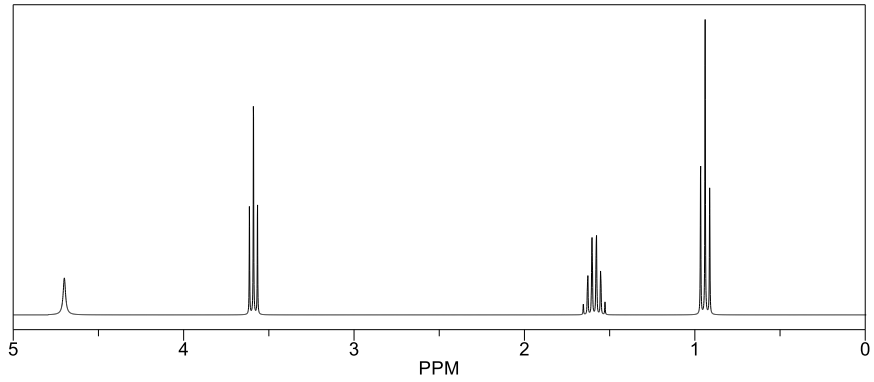
Mass spectrum

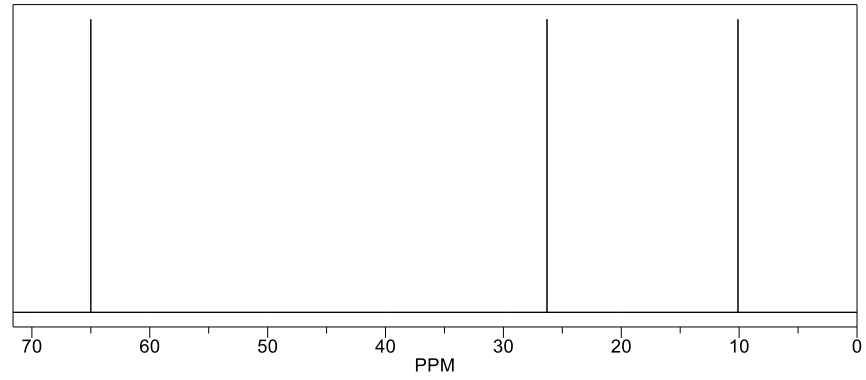


Infrared spectrum



NMR spectrums

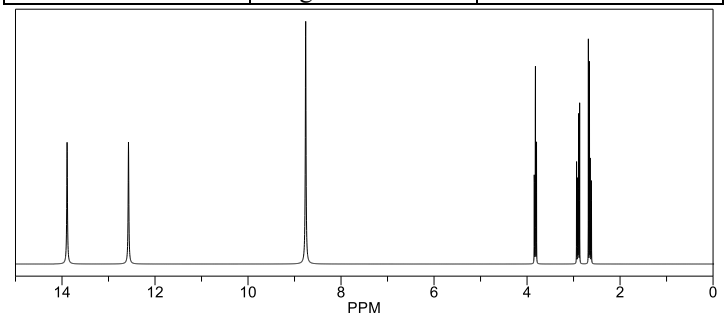




Question 7 (4 marks)

An unknown substance has molecular formula, $C_4H_7NO_4$. Determine its structure using the following information in the table of the proton NMR data. The carbon NMR has 4 peaks and the infrared spectrum has a broad band at 3100cm^{-1} and a sharp band at 1690cm^{-1}.

Chemical shift (ppm)	Peak splitting	Relative peak area
2.7	Doublet	2
3.8	Triplet	1
8.8	Singlet	2
12.6	Singlet	1
13.9	Singlet	1



Question 8 (10 marks)

An athlete has been requested to provide a blood sample to a sports drug testing agency to test the levels of a steroid consumed. Using HPLC, the results are shown below.

Concentration (mg L^{-1})	**Peak area (mm^2)**
21.0	0.12
46.0	0.23
63.0	0.36
86.0	0.46
Blood sample	0.28

(a) In the space below, construct a calibration curve for the data in the table. **2 marks**

(b) What is the concentration of the steroid in the athlete's blood sample? **1 mark**

(c) Determine the concentration of the steroid in µg L^{-1}. **1 mark**

(d) HPLC is distinguished from the other types of chromatography because it uses pressure generated from a pump. Why is this an important feature? **2 marks**

(e) The chromatogram generated from this blood sample shows broad overlapping peaks. Comment on the quality of the HPLC run and suggest two improvements that can be made. **3 marks**

(f) During qualitative analysis of the blood sample, the chromatogram was compared to the HPLC run of a pure sample of the steroid. To compare the chromatograms, what must be considered? **1 mark**