Unit 4 outcome 2 revision for SAC

Laboratory analysis of organic compounds

- qualitative tests for the presence of carbon-carbon double bonds, hydroxyl and carboxyl functional groups
- applications and principles of laboratory analysis techniques in verifying components and purity of consumer products, including melting point determination and distillation (simple and fractional)
- measurement of the degree of unsaturation of compounds using iodine
- volumetric analysis, including calculations of excess and limiting reactants using redox titrations (excluding back titrations)

Instrumental analysis of organic compounds

- applications of mass spectrometry (excluding features of instrumentation and operation) and interpretation of qualitative and quantitative data, including identification of molecular ion peak, determination of molecular mass and identification of simple fragments
- identification of bond types by qualitative infrared spectroscopy (IR) data analysis using characteristic absorption bands
- structural determination of organic compounds by low resolution carbon-13 nuclear magnetic resonance (^{13}C-NMR) spectral analysis, using chemical shift values to deduce the number and nature of different carbon environments
- structural determination of organic compounds by low and high resolution proton nuclear magnetic resonance (^{1}H-NMR) spectral analysis, using chemical shift values, integration curves (where the height is proportional to the area underneath a peak) and peak splitting patterns (excluding coupling constants), and application of the n+1 rule (where n is the number of neighbouring protons) to deduce the number and nature of different proton environments
- the principles of chromatography, including high performance liquid chromatography (HPLC) and the use of retention times and the construction of a calibration curve to determine the concentration of an organic compound in a solution (excluding features of instrumentation and operation)
- deduction of the structures of simple organic compounds using a combination of mass spectrometry (MS), infrared spectroscopy (IR), proton nuclear magnetic resonance (^{1}H-NMR) and carbon-13 nuclear magnetic resonance (^{13}C-NMR) (limited to data analysis)
- the roles and applications of laboratory and instrumental analysis, with reference to product purity and the identification of organic compounds or functional groups in isolation or within a mixture

Medicinal chemistry

- extraction and purification of natural plant compounds as possible active ingredients for medicines, using solvent extraction and distillation
- identification of the structure and functional groups of organic molecules that are medicines
- significance of isomers and the identification of chiral centres (carbon atom surrounded by four different groups) in the effectiveness of medicines
- enzymes as protein-based catalysts in living systems: primary, secondary, tertiary and quaternary structures and changes in enzyme function in terms of structure and bonding as a result of increased temperature (denaturation), decreased temperature (lowered activity), or changes in pH (formation of zwitterions and denaturation)
- medicines that function as competitive enzyme inhibitors: organic molecules that bind through lock-and-key mechanism to an active site preventing binding of the actual substrate

An unknown hydrocarbon, X, is composed of four carbons. A known mass of this hydrocarbon X is placed in a 250 mL volumetric flask and made to the mark with ethanol to form a 0.0500 M solution.

4 X 20.00 mL aliquots were taken from the volumetric flask placed in four separate c100 mL conical flasks and titrated against a 0.100 M Br_2 solution. The solution in the conical flask remained clear until the end point was reached at which point the solution turned brown. An average titre of 10.01 mL was achieved.

a. Is the hydrocarbon saturated or unsaturated? Justify your answer with a calculation. All working out must be clearly shown in the space below. *3 marks*

b. Below is an organic pathway, fig. 1, starting with hydrocarbon X and the spectra of some of the compounds.

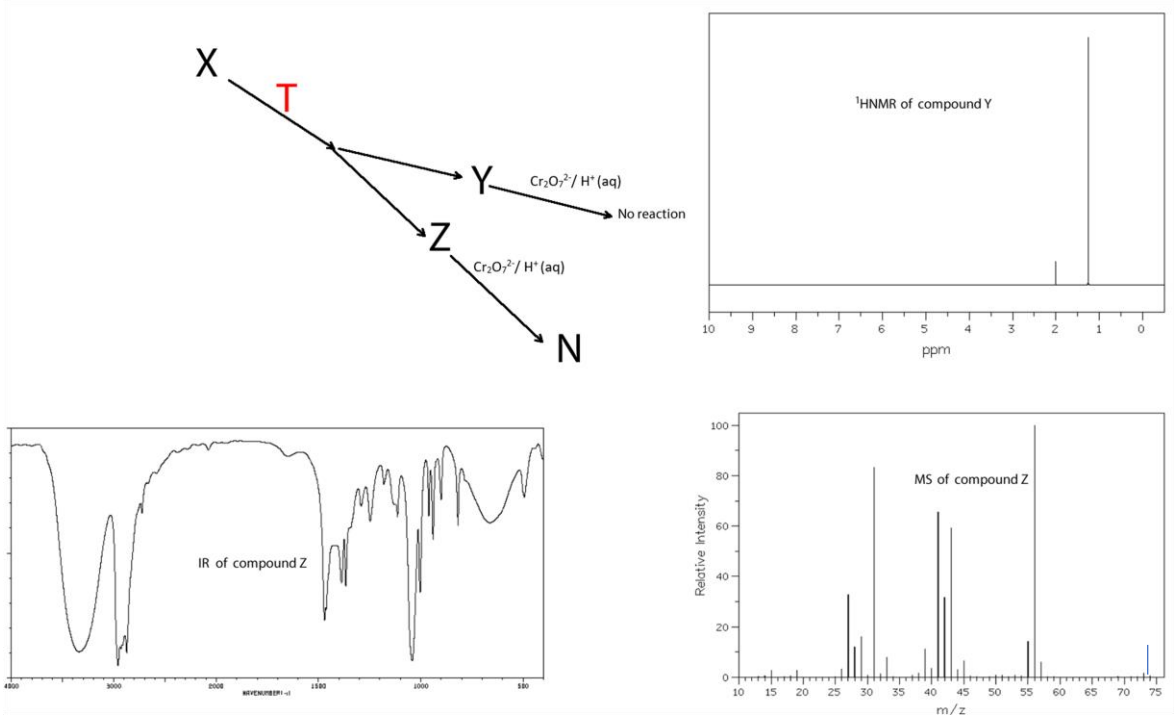

Figure 1

i. Identify the conditions and reagents at T. *1 mark*

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ii. A further reaction of compound Y with $Cr_2O_7^{2-}$ in an acidic solution is not possible. In the space on the right, draw the structural formula and name compound Y *2 marks*

Compound Y _____

iii. To what class of organic compounds does compound Y belong to. *1 mark*

Y _____

iv. An excess amount of $Cr_2O_7^{2-}$ is reacted with compound Z, in an acidified solution, to form compound N. Give the IUPAC name for compound N and identify the class of organic compounds that N belongs to. *2 marks*

IUPCAC name_____-

Class of organic compounds_____

v. After completing a purifying technique to isolate compound N, a student suggested that N might not be a pure substance as it may be contaminated with compound Z . Provide a test that can be conducted to determine the presence of Z and clearly outline the outcomes of the test that shows the presence of Z.

_____

_____

_____*2 marks*

vi. Give the structural formula of the fragment responsible for the peak at 31 m/z in the mass spectrum of compound Z? *1 mark*

_____

vii. Give the IUPAC name and structure for compound Z in the space provided on the right. *2 marks*

Compound Z _____

viii. Give the IUPAC name and structural formula of compound X in the space provided on the right. *2 marks*

Compound X _____

ix. In the ^{1}HNMR spectrum of compound Z, identify the number and type of signals observed. For instance, specify the presence of signals such as 2 triplets, 1 singlet, etc.

_____

_____*2 marks*

1. Cyclooxygenase is an enzyme responsible for catalysing the reaction that produces prostaglandins from arachidonic acid. Prostaglandins trigger inflammation and increase pain sensitivity. Figure 2 illustrates how two different drugs interact with cyclooxygenase. Aspirin chemically modifies the enzyme by reacting with its active site to form a permanent covalent bond, while ibuprofen blocks the entry of arachidonic acid into the active site by forming temporary, reversible bonds with the acitve site.

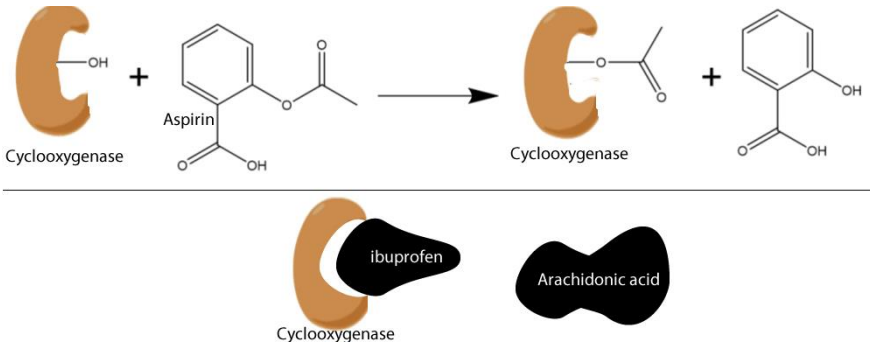

Figure 2 – aspiring and ibuprofen interactions with cyclooxygenase

a. On the set of axes shown in fig 3 draw a clearly labelled graph of a reaction catalysed by: *3 marks*

   i. cyclooxygenase.

   ii. cyclooxygenase with aspirin

   iii. cyclooxygenase with ibuprofen.

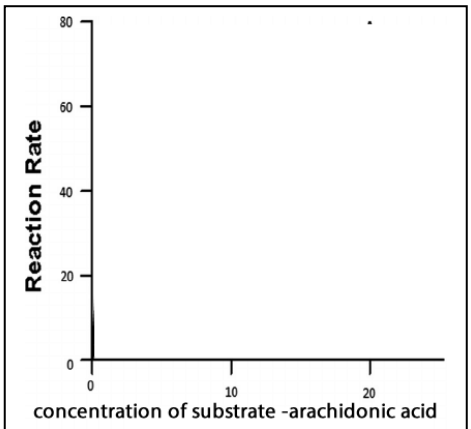

Figure 3

b. Clearly define the term "competitive inhibition" and explain whether aspirin and ibuprofen can be classified as competitive inhibitors.

3 marks

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2. The three substances ibuprofen, paracetamol and aspirin are shown below in fig.4.

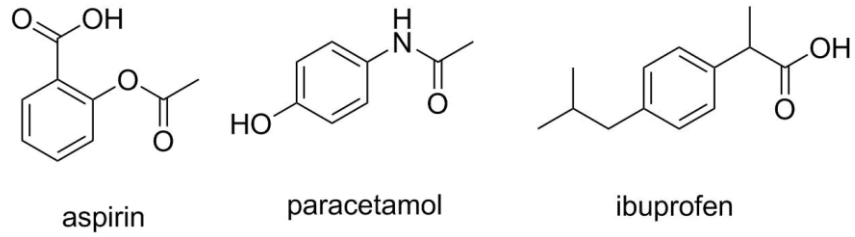

Figure 4

A solution containing the three substances was placed in a reverse phase HPLC column, where the stationary phase is non-polar and the mobile phase is polar.

The chromatogram shown in fig. 5 was obtained.

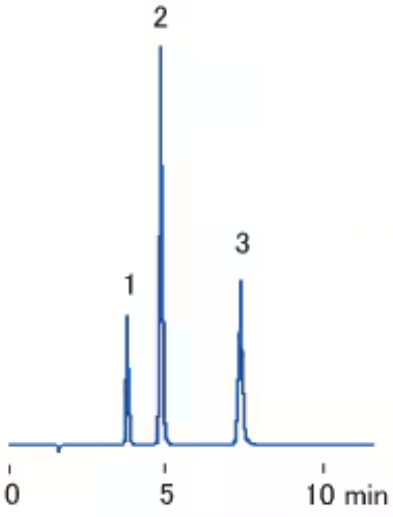

Figure 5

a. Identify the compound that belongs to each peak . *3 marks*

1. _____

2. _____

3. _____

b. Which substance is present in the highest concentration? *1 mark*

_____

c. Explain how each of the following changes to the column would alter the chromatogram in Fig. 5, and provide a justification for your answer.

i. Smaller beads used as the stationary phase.

_____

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_____ *2 marks*

ii. Pressure driving the solution through the column was doubled.

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_____*2 marks*

iii. Temperature of the mobile phase was decreased.

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_____*2 marks*

iv. The sample was diluted prior to being injected into the column.

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_____*2 marks*

v. Normal phase HPLC was used where the mobile phase is non-polar and the stationary phase is polar.

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_____*2 marks*

d. The chromatogram shown in fig 6 was conducted on a different HPLC column under totally different conditions.

i. Substance 2, from the chromatogram shown in fig 5, had a retention time of 5 minutes. Can we be sure it is present in this mixture? Justify your answer.

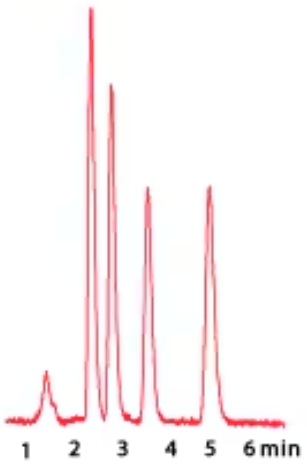

Figure 6

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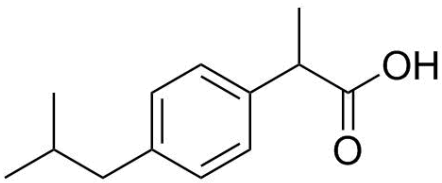
_____*3 marks*

4. Consider the molecule of ibuprofen pictured in fig 7.



ibuprofen

Figure 7

a. How many chiral carbons are present in ibuprofen?_ *1 mark*

_____

b. How many optical isomers must the manufacturer of this product test for harmful side effects in Humans? *1 mark*

_____

c. Define an optical isomer.

_____

_____

_____*1 mark*

d. Circle and label two functional groups in fig. 7 above. *2 marks*

e. A chiral molecule has two different optical isomers (enantiomers). Complete the table below by identifying if the enantiomers behave in similar or different ways to the following . Circle the appropriate response. *7 marks*

Condition	Enantiomer A	Enantiomer B
Interaction with a strong acid	Different reaction Identical reaction	Different reaction Identical reaction
Reaction with a strong base	Different reaction Identical reaction	Different reaction Identical reaction
Interaction with a chiral enzyme	Different reaction Identical reaction	Different reaction Identical reaction
Rotation of plane-polarized light	Rotates polarized light clockwise Identical rotation of polarized light	Rotate polarized light anticlockwise Identical rotation of polarized light
Reaction in a chiral solvent	Different reaction Identical reaction	Different reaction Identical reaction
Melting point in an achiral environment	Different MP Identical MP	Different MP Identical MP
Taste or smell perception	Different / Identical	Different / Identical

5. A student is tasked with determining the concentration of acetic acid in a vinegar solution. A 25.0 mL sample of the vinegar (acetic acid solution) was placed in a 250.0 mL volumetric flask and made to the mark using distilled water. Four 25.0 mL aliquots of the diluted acid solution were transferred into four conical flasks with 2 drops of an appropriate indicator. The solution in each conical flask was titrated against a standard solution of sodium hydroxide (NaOH) of concentration 0.100 M.

The following titres were obtained

- Trial 1: 22.10 mL
- Trial 2: 22.00 mL
- Trial 3: 22.19 mL
- Trial 4: 22.05 mL

a. Write a balanced chemical equation for the reaction taking place in the conical flask.

_____ *2 marks*

b. Calculate the average titre. Show all working out *2 marks*

c. Calculate the amount, in mol, NaOH in an average titre. Give the answer to the right number of significant figures. 2 marks

d. Calculate the mol of acetic acid in the volumetric flask. *2 marks*

e. Calculate the concentration, in %w/v, of acetic acid in the vinegar. *3 marks*

f. The indicator used by the student during the titration was methyl orange. Explain whether the use of methyl orange is appropriate for this titration, and discuss how the choice of this indicator could affect the validity of the results obtained.

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_____*3 marks*

g. Sodium hydroxide is not considered a primary standard.

i. Define what is meant by a primary standard and list four essential criteria that a substance must meet to be considered a primary standard.

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_____*3 marks*

ii. Although NaOH is not a primary standard, a solution of NaOH can still be used as a standard solution. Explain how this is achieved and why it is important for ensuring accurate titrations.

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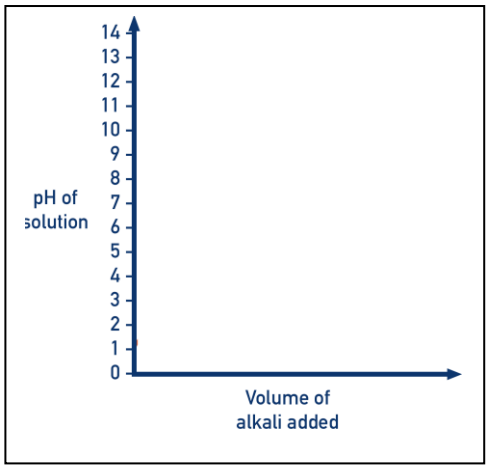
_____*2 marks*

h. A student proposed using sodium bicarbonate ($NaHCO_3$), which is considered a primary standard, to make a standard solution with which to titrate an unknown solution of acetic acid (CH_3COOH). Assess the validity of this suggestion. Discuss whether $NaHCO_3$ is suitable for this titration and address any potential issues that might occur. Use the provided space to draw a pH curve to support your explanation.



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_____*3 marks*

i. Explain the difference between :

- A burette and pipette

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_____*2 marks*

- An aliquot and a titre.

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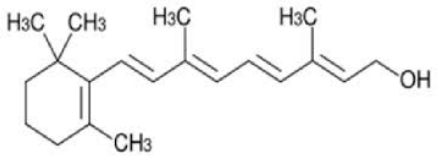
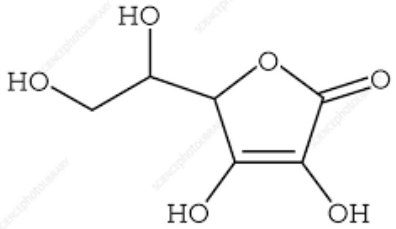
_____*2 marks*

6. A pharmaceutical manufacturer has accidentally mixed a batch of vitamin C with vitamin A. The costly error can be remedied by using a purifying technique called solvent extraction. The structures of both vitamins are shown in fig 7.

a. In the space provided outline a step-by-step process for the purification of each vitamin from the solid mixture in powder form given that you have a ethanol and hexane at your disposal.



Vitamin A



Vitamin C
$C_6H_8O_6$

Figure 7

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_____ 7 marks

b. Describe the difference between steam distillation and fractional distillation and suggest one appropriate use for each process.

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_____ 4 marks