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UNIVERSITY OF RUHUNA – FACULTY OF ALLIED HEALTH SCIENCES

DEPARTMENT OF PHARMACY

FOURTH BPHARM PART I EXAMINATION – NOVEMBER/DECEMBER 2023

PH 4123 PHARMACEUTICAL ANALYSIS – SEQ PAPER

TIME: TWO HOURS

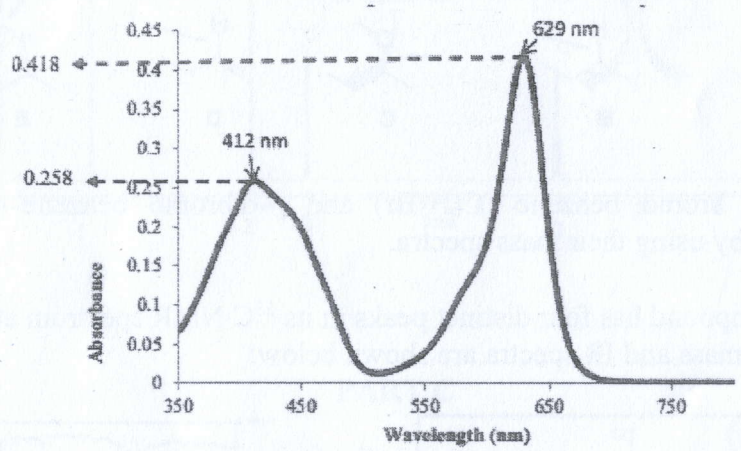
INSTRUCTIONS

- Answer all questions in parts **A, B, C,** and **D.**
- No paper should be removed from the examination hall.
- Do not use any correction fluid.
- Use illustrations where necessary.

PART A

01.

1.1 UV-vis spectrum of an unknown beverage sample is shown below:

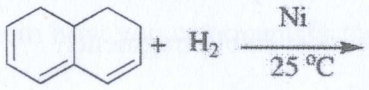


1.1.1 Calculate the %T of the solution based on the tallest peak. (***10 marks***)

1.1.2 What colour would you predict the beverage to be? (***05 marks***)

1.1.3 State what type of electronic transition is responsible for each of these peaks. (***10 marks***)

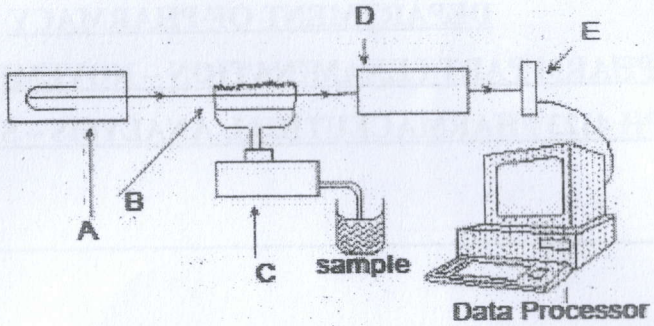
1.2 Partial hydrogenation of the triene shown below results in two compounds, **A** and **B** both of molecular formula $C_{10}H_{14}$. Compound **A** shows a λ_{max} = 235 nm and **B**, 275 nm. Assign the structures by calculating λ_{max} values using Woodward-Fieser rules. (***25 marks***)



A and B

1.3 Explain the occurrence of fluorescence and phosphorescence with the aid of a Jablonski diagram. (***20 marks***)

1.4 Schematic diagram of an Atomic Absorption Spectrometer (AAS) is shown below:

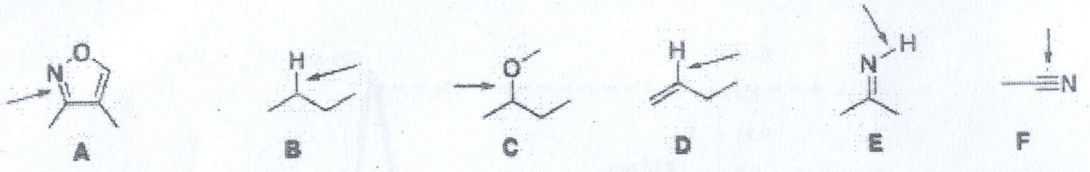


1.4.1 Label all the components of the diagram. ***(15 marks)***

1.4.2 Can this instrument be used as an atomic emission spectrometer? Sketch the instrumental components and their arrangement of an atomic emission spectrometer. ***(15 marks)***

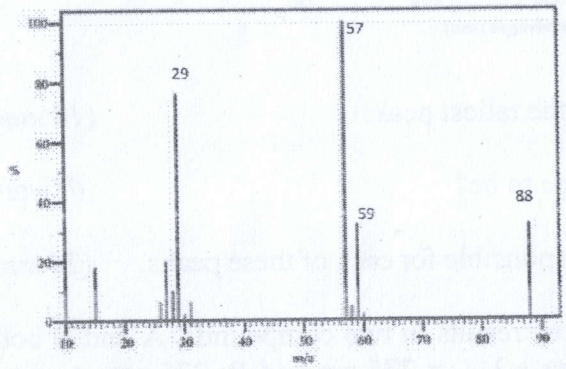
02.

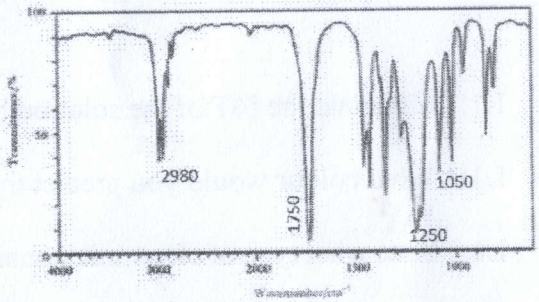
2.1 Rank the following bonds (indicated by arrows) in the order of increasing wavenumber. ***(10 marks)***



2.2 Explain how bromo benzene (C_6H_5Br) and p-dibromo benzene ($C_6H_4Br_2$) could be distinguished by using their mass spectra. ***(10 marks)***

2.3 An organic compound has four distinct peaks in its ^{13}C NMR spectrum at δ 9.2, 27.5, 51.5 and 174.9 ppm. Its mass and IR spectra are shown below:





2.3.1 Identify the parent and base peaks in the mass spectrum. ***(10 marks)***

2.3.2 What are the molecular structures of the most stable fragments? ***(05 marks)***

2.3.3 Deduce the structure of the compound giving reasons. ***(10 marks)***

2.3.4 Assign as many bonds as possible to their corresponding bands in the IR spectrum. ***(05 marks)***

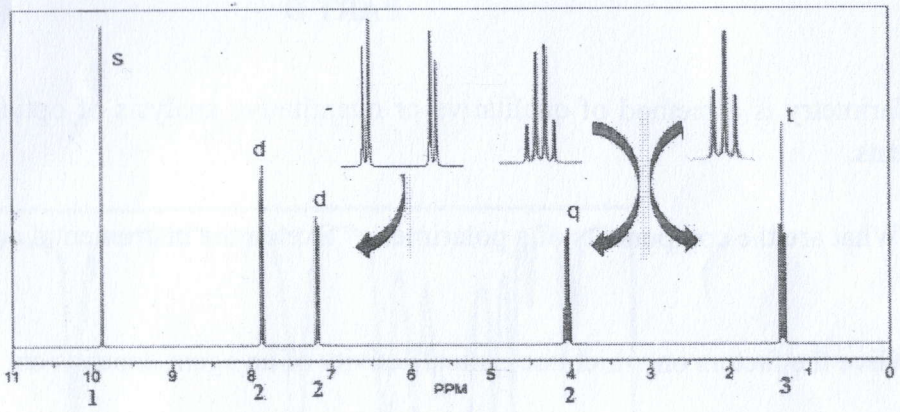
PART B

2.4 Answer the following questions on NMR spectroscopy.

2.4.1 Briefly explain the use of spin-spin coupling for the structure elucidation of organic compounds. ***(10 marks)***

2.4.2 Assume that you have been given two ^1H NMR spectra that were recorded for two molecules of 1-*chloroethylbenzene* (**A**) and 4-*chloroethylbenzene* (**B**). Explain how you would assign the two spectra for the molecules **A** and **B**. ***(15 marks)***

2.4.3 ^1H NMR spectrum of compound **P** ($C_9H_{10}O_2$) is shown below. Spin multiplicity of each peak is indicated top of the peak. Giving reasons, suggest a plausible chemical structure for the compound **P**. Integration values of the spectrum are displayed under the scale.
(25 marks)



PART C

03.

3.1 Assume that a drug **X** is **partially** metabolized to the metabolite **Y** in the liver and completely excreted along with **Y** via urine.

3.1.1 If you use a C_{18} reversed phase HPLC column and aqueous methanol as the mobile phase for separation of **X** and **Y** in a urine sample, state the order of elution of **X** and **Y**. Briefly explain the reason. ***(20 marks)***

3.1.2 Briefly explain how you can quantify the concentrations of **X** and **Y** in a urine sample.
(30 marks)

3.2 List five detectors used in gas chromatography. ***(15 marks)***

3.3 State how the following factors affect the separation of compounds in column chromatography. (*20 marks*)

3.3.1 Number of theoretical plates
3.3.2 Length of the column
3.3.3 Size of the silica particles
3.3.4 Thickness of a theoretical plate

3.4 State the chromatographic method that you would use for the following purposes. (*15 marks*)

3.4.1 Detection of a volatile pesticide in a food sample
3.4.2 To monitor the progress of an organic reaction
3.4.3 To assess the purity of the fractions obtained from column chromatography
3.4.4 To purify an antibody
3.4.5 To assess the molecular weight distribution pattern of a polymer sample

PART D

04.

4.1 Polarimetry is a method of qualitative or quantitative analysis of optically active chemical agents.

4.1.1 What are the components of a polarimeter? Sketch the instrumental components of it. (*15 marks*)

4.1.2 Give the factors on which the optical activity of an agent depends on. (*18 marks*)

4.1.3 Write down the meaning of the following expression pertaining to the polarimetry.

$$[\alpha]_D^{25} = +3.12^\circ$$ (*21 marks*)

4.2 Draw the conductance vs volume titration curve for the titration of acetic acid with ammonium hydroxide. Giving reasons, explain the shape of the graph and mark the end point. (*20 marks*)

4.3

4.3.1 Devise a cell in which the cell reaction is $Mn(s) + Cl_2(g) \rightarrow MnCl_2(aq)$. (*10 marks*)

4.3.2 Write down the cell notation for the above cell. (*06 marks*)

4.3.3 Give the half reaction of each electrode of the cell. (*06 marks*)

4.3.4 Deduce the standard potential of the Mn^{2+}(aq)/Mn(s) couple if the standard cell potential of the cell is 2.54 V and the standard reduction potential of Cl_2(g) is 1.36 V. (*04 marks*)

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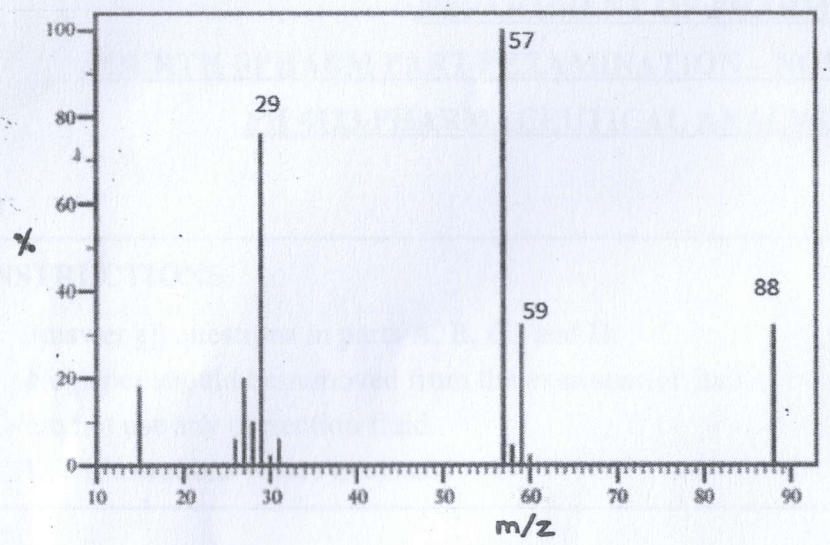
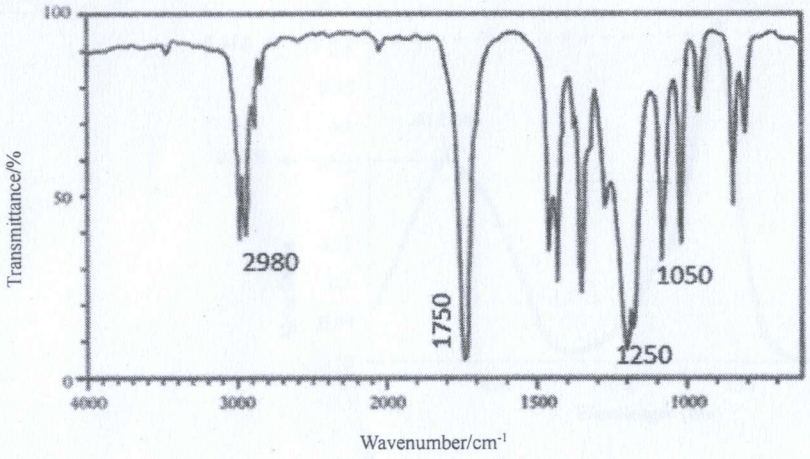

100
80
60
40
20
0
%
57
29
59
88
10
20
30
40
50
60
70
80
90
m/z


100
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0
Transmittance/%
2980
1750
1250
1050
4000
3000
2000
1500
1000
Wavenumber/cm-1