

UNIVERSITY OF JAFFNA, SRI LANKA
FACULTY OF ALLIED HEALTH SCIENCES
THIRD YEAR SECOND SEMESTER EXAMINATION IN BPharmHons-2022
PHAPA 3244 PHARMACEUTICAL ANALYSIS

Date: 25. 06.2024

Time: 3 Hours

ANSWER ALL THE SIX QUESTIONS

1. 1.1 Define distillation. (10 Marks)
- 1.2 State the principle of distillation. (20 Marks)
- 1.3 List the types of distillation techniques and briefly explain them. (50 Marks)
- 1.4 Briefly discuss the applications of distillation methods mentioned in section 1.3. (20 Marks)

2. 2.1 Give the basic principle of Thin Layer Chromatography and Paper chromatography (20 Marks)
- 2.2 List the difference in between the Paper and thin Layer chromatography. (30 Marks)
- 2.3 Explain how the synthesis of aspirin could be monitored with Thin Layer chromatography technique. (50 Marks)

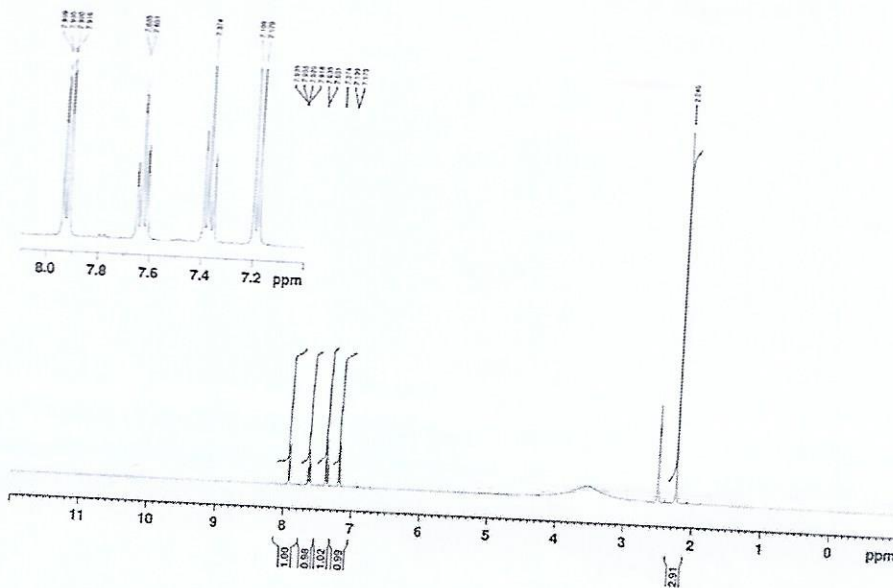
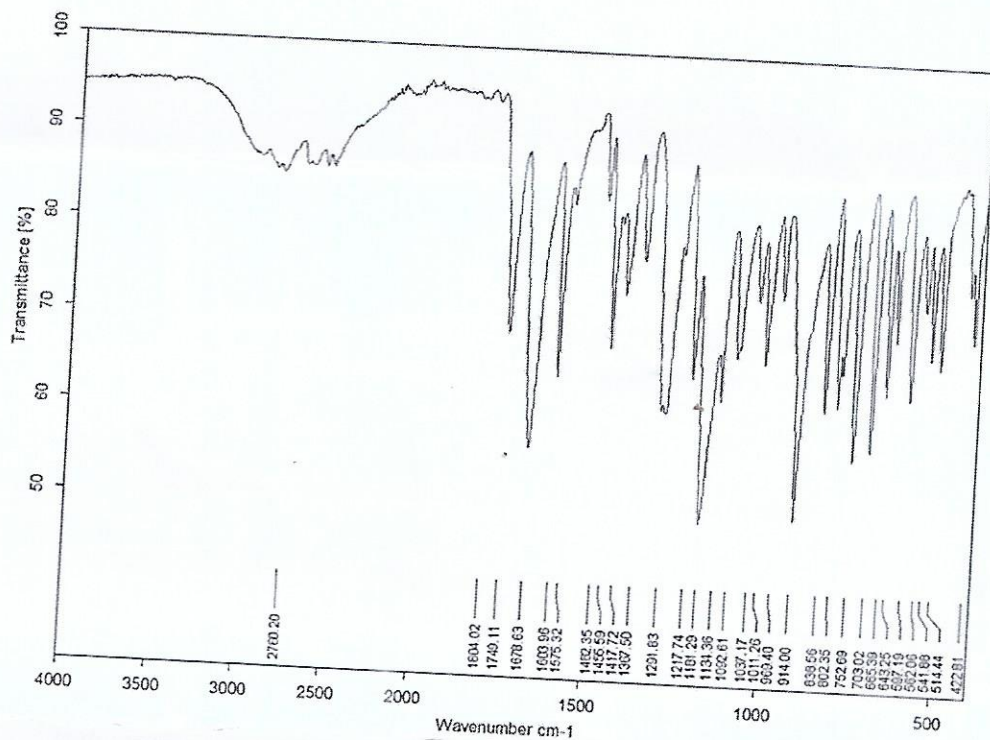
3. 3.1 3.1.1 State the principle of Mass Spectrometry. (20 Marks)
- 3.1.2 Briefly describe the instrumentation of Mass Spectrometry. (20 Marks)
- 3.1.3 Explain Mc Lafferty rearrangement with an example. (20 Marks)

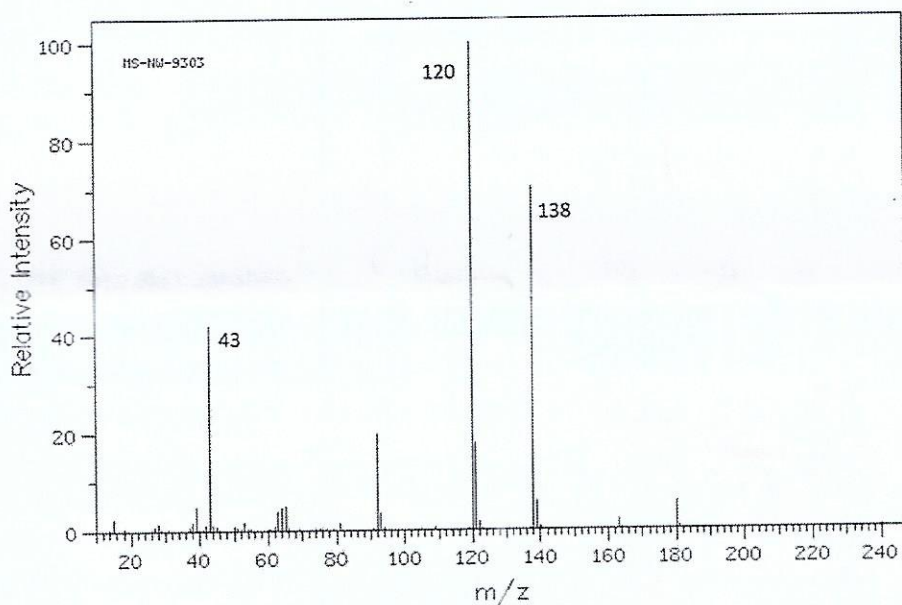
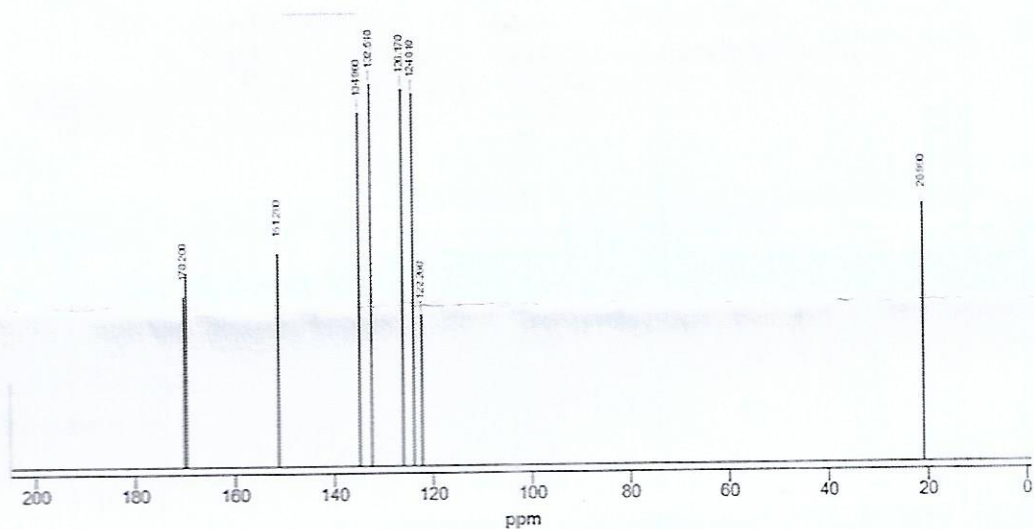
- 3.2 Write a short account on the followings: (40 Marks)
 - 3.2.1 Nephelometry.
 - 3.2.2 Flame photometry

4. 4.1 Define fingerprint region. (10 Marks)
- 4.2 4.2.1 State the theory of Infrared spectroscopy. (20 Marks)
- 4.2.2 Briefly explain why not all the compounds give peak in IR Spectroscopy. (20 Marks)
- 4.3 A sample, para-nitrophenol was reduced with NaBH₄, and then it was treated with anhydride, produced paracetamol. Briefly discuss how the IR spectroscopy technique could be used to ensure the synthesis of paracetamol. (50 Marks)

5

A Compound has a molecular formula of $C_9H_8O_4$. The structure of the compound is analysed with IR, 1H -NMR, ^{13}C -NMR and Mass spectroscopy. The analysed data of the spectra are given below.





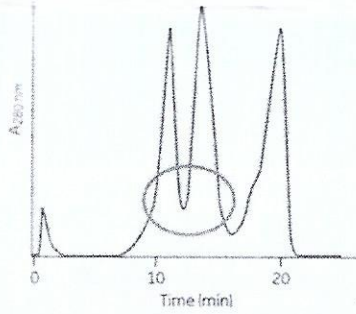
- 5.1 Determine the degree of unsaturation of the compound. **(10 Marks)**
- 5.2 Assign the spectra of IR, $^1\text{H-NMR}$, $^{13}\text{C-NMR}$ and Mass. **(70 Marks)**
- 5.3 Deduce the structure of the compound. **(20 Marks)**
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- 6 6.1 Write a short account on Ultra Performance Liquid chromatography (UPLC). **(30 Marks)**
- 6.2 6.2.1 State the principle of ion exchange chromatography. **(10 Marks)**
- 6.2.2 Briefly discuss the factors that influence the separation of the compounds in ion exchange chromatography. **(30 Marks)**

6.3 Briefly discuss the following ion exchange chromatograms of a sample containing different compounds.

6.3.1

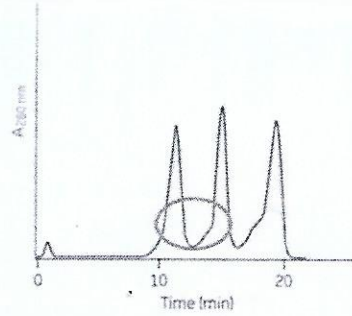
(A) Sample load: 10 mg

Column: SOURCE™ 30S, 5 mm x 50 mm (i.d. x h)



(B) Sample load: 1 mg

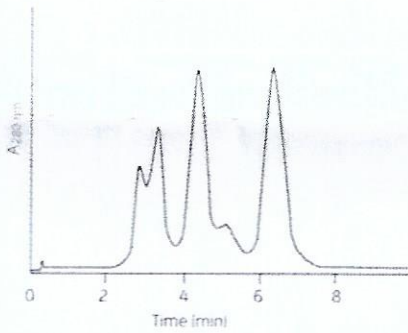
Column: SOURCE 30S, 5 mm x 50 mm (i.d. x h)



6.3.2

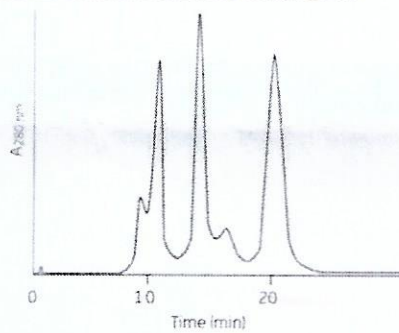
(A) Flow rate: 13 mL/min

Column: SOURCE™ 30Q, 10 mm x 50 mm (i.d. x h)



(B) Flow rate: 4 mL/min

Column: SOURCE 30Q, 10 mm x 50 mm (i.d. x h)



(30 Marks)