NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY

FACULTY OF APPLIED SCIENCES

DEPARTMENT OF APPLIED CHEMISTRY

ANALYTICAL CHEMISTRY II – SCH 2106

End of Second Semester Examination Paper

December 2024

This Examination Paper consists of 6 pages

Time Allowed:	3 hours
Total Marks:	100
Special Requirement:	Calculator
Internal Examiner:	Ms E. Bere
External Examiner:	Dr. G. Mehlana

INSTRUCTIONS

1. Answer ALL questions in Section A and any three (3) questions in Section B

2. Questions in Section A carries 40 marks and each question in Section B carries 20 marks

MARK ALLOCATION

QUESTION	MARKS
1.	40
2.	20
3.	20
4.	20
5.	20
TOTAL POSSIBLE MARKS	**100**

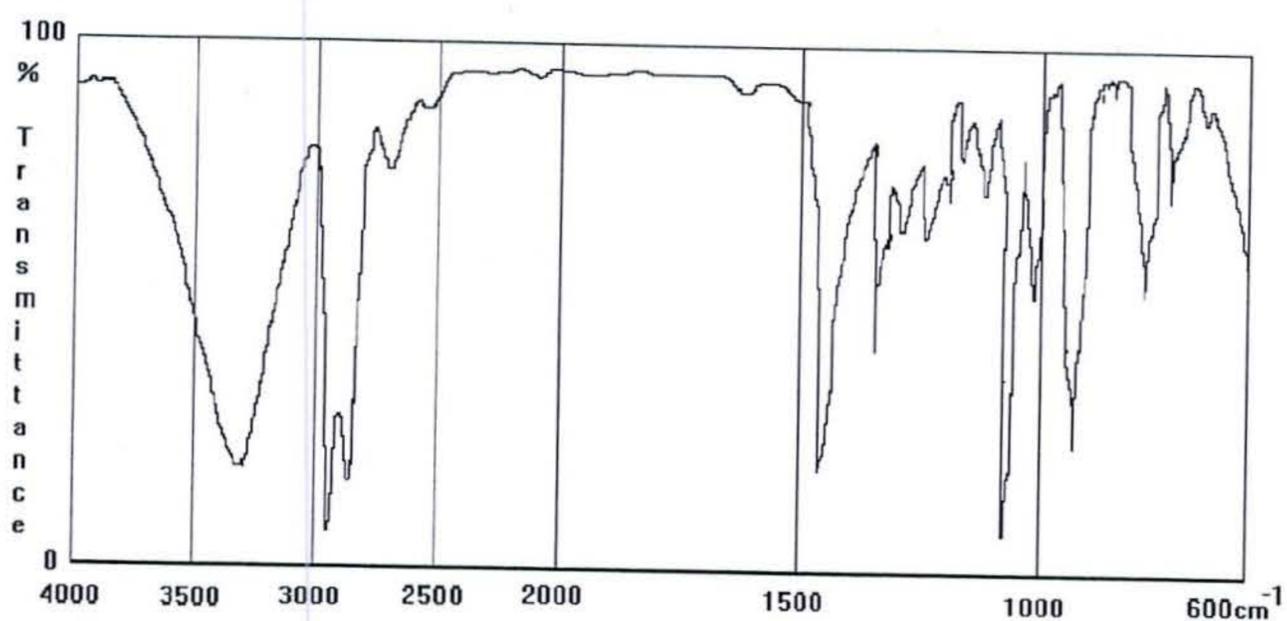**Copyright: National University of Science and Technology, 2024**

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SECTION A

1. a). Define the following terms when used in spectroscopy:

 i. Atomization

 ii. Chemical interferences

 iii. Stokes shift

 iv. Spectral interferences **[8 marks]**

 b) i) define Beer Lambert law **[2 marks]**

 ii) what makes this law useful? **[3 marks]**

 iii) identify factors that cause deviations from linearity. **[5 marks]**

 c) A portable photometer with a linear response to radiation registered 75.5 µA with a blank solution in the light path. Replacement of the blank with an absorbing solution yielded a response of 23.7 µA Calculate:

 i) the absorbance of the sample solution.

 ii) the transmittance to be expected for a solution that has twice the concentration of the sample solution. **[4 marks]**

 d). i) Define the term *chromatography* **[2 marks]**

 ii) using appropriate examples, describe the difference between planar and column chromatography **[4 marks]**

 iii) Why is high pressure needed in HPLC? **[2 marks]**

 e). Define the following terms:

 i) stationary phase

 ii) mobile phase

 iii) elution **[6 marks]**

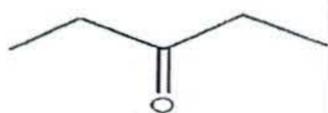 f) What are the 2 types of solvent systems used in column chromatography?

 [4 marks]

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SECTION B

2. a) From the spectrum and the IR peak location index:

i) Characterize the major peaks.

ii) Identify possible functional groups and list them.

iii) Identify the structure from the list of possibilities.
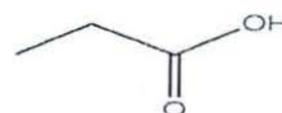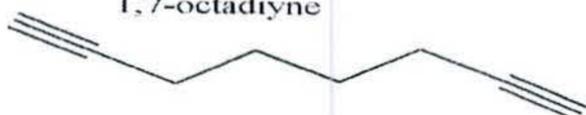

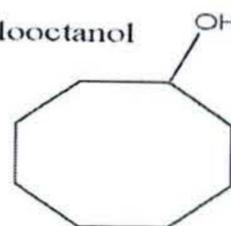List of possible structures:

3-pentanone

1-hexene

propanoic acid

1,7-octadiyne

Cyclooctanol

[5 marks]

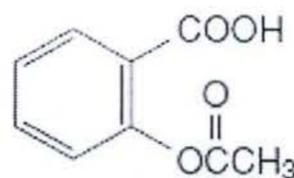b) Describe any 2 methods used to prepare a solid sample for IR analysis

[4 marks]

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c)   A student carefully prepared a solid organic compound for IR analysis. On running, the spectra showed none of the expected peaks. Explain what could have caused this.   **[1 mark]**

d)   Salicylic acid is a compound used to synthesize aspirin.
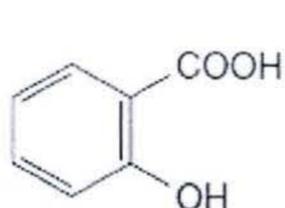
salicylic acid   **acetylsalicylic acid**

What differences would be expected in the infrared spectra of salicylic acid and acetylsalicylic acid?   **[4 marks]**

e)   Infrared spectroscopy can be used for both quantitative and qualitative analysis. Explain why it is mainly used in qualitative analysis   **[2 marks]**

f)   Pure hexane has negligible ultraviolet absorbance above a wavelength of 200 nm. A solution prepared by dissolving 25.8 mg of benzene in hexane and diluting to 250.0 mL had an absorption peak at 256 nm with an absorbance of 0.266 in a 1.000-cm cell. Find the molar absorptivity of benzene at this wavelength.   **[4 marks]**

3.   a)   Draw a block diagram of a FAAS spectrophotometer and discuss the functions of each major component   **[10 marks]**

   b)   A 5.00-mL sample of blood was treated with trichloroacetic acid to precipitate proteins. After centrifugation, the resulting solution was brought to a pH of 3 and was extracted with two 5-mL portions of methyl isobutyl ketone containing the organic lead complexing agent APCD. The extract was aspirated directly into an air-acetylene flame yielding an absorbance of 0.454 at 283.3 nm. Five-millilitre aliquots of standard solutions containing 0.240 and 0.475 ppm Pb were treated in the same way and yielded absorbances of 0.412 and 0.642. Calculate

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the concentration Pb (ppm) in the sample assuming that Beer's law is followed.

[10 marks]

4.   What are the principal advantages and the principal limitations of each of the detectors:

a) thermal conductivity,

b) flame ionization,

c) electron capture,

d) thermionic, and

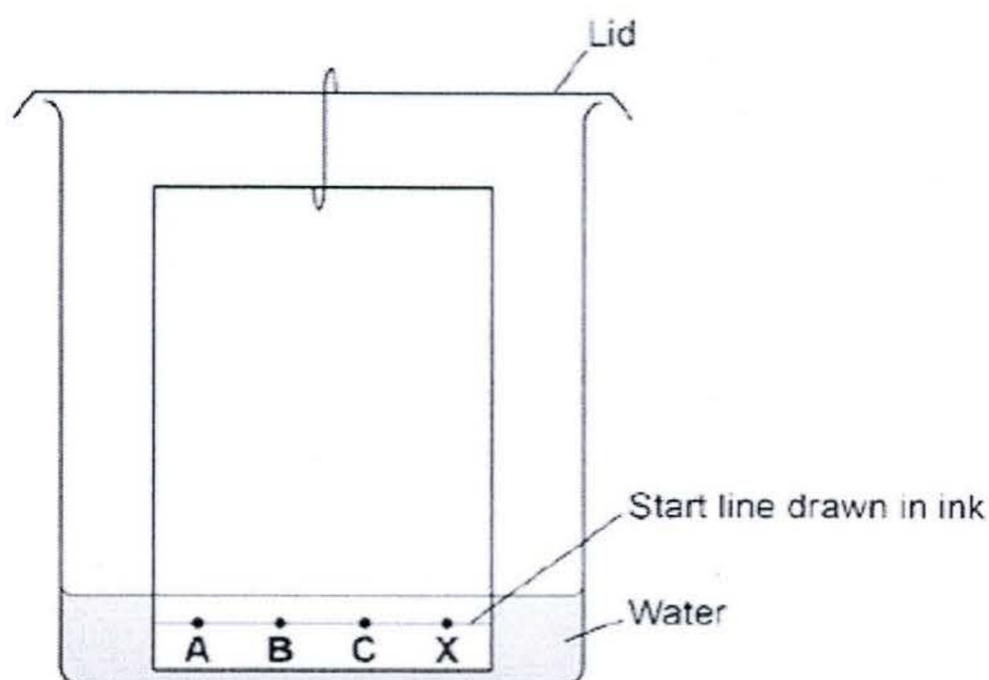e) photoionization. **[20 marks]**

5.   A student investigated a food colouring using paper chromatography.

This is the method used.

- Put a spot of food colouring X on the start line.
- Put spots of three separate dyes, A, B and C, on the start line.
- Place the bottom of the paper in water and leave it for several minutes.

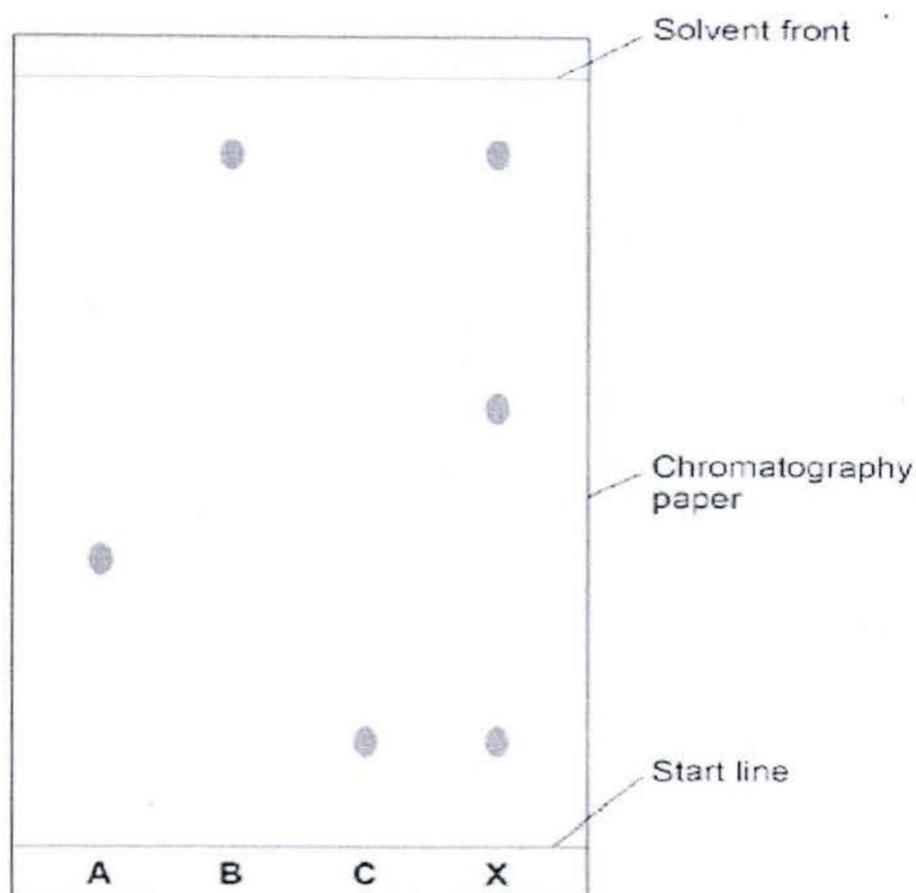Figure 1 shows the apparatus the student used.

Figure 1

a) Give two mistakes the student made in setting up the experiment.   **[2 marks]**

b) Another student set the experiment up correctly. Figure 2 shows the student's results.

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Figure 2

i) How many dyes were in X? [1 mark]

ii) Which dye is not a constituent of X? [1 mark]

iii) Calculate the value for Rf for dye A. [2 marks]

c) TLC and paper chromatography are often used for qualitative rather than quantitative analysis. Describe how you can use TLC for quantitative analysis

[4 marks]

d) What is temperature programming in GC? How does it gain an advantage over single T separations? [4 marks]

e) What is gradient elution and how does this differ from an isocratic one? What advantage does gradient elution have over isocratic separations? [4 marks]

f) What is/are the advantage(s) of using an autosampler instead of performing manual injections in gas chromatography? [2 marks]

END OF QUESTION PAPER

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