

UNIVERSITI TUN HUSSEIN ONN MALAYSIA

FINAL EXAMINATION SEMESTER II SESSION 2022/2023

COURSE NAME : ANALYTICAL CHEMISTRY

COURSE CODE : DAK 12603

PROGRAMME CODE : DAK

EXAMINATION DATE : JULY / AUGUST 2023

DURATION : 2 HOURS AND 30 MINUTES

INSTRUCTION : 1. ANSWER ALL QUESTIONS.

2. THIS FINAL EXAMINATION IS CONDUCTED VIA **CLOSED BOOK.**

3. STUDENTS ARE **PROHIBITED** TO CONSULT THEIR OWN MATERIAL OR ANY EXTERNAL RESOURCES DURING THE EXAMINATION

CONDUCTED VIA CLOSED BOOK.

THIS QUESTION PAPER CONSISTS OF SEVEN (7) PAGES



- Q1 Lina was instructed by her laboratory supervisor to prepare a stock solution consisting of 23.4 g of chemical A. Lina carried out the task by dissolving the solute in a solvent and diluting the solution to a final volume of 250 ml. In addition, the stock solution was diluted in different dilution ratios to produce the calibration curve. Assume that the molar mass of chemical A is 142 g/mol.
 - (a) Differentiate the term solution, solvent, and solute.

(3 marks)

(b) Determine the molarity of the stock solution prepared.

(5 marks)

(c) State two (2) functions of calibration curve.

(2 marks)

- (d) The equipment used in Lina laboratory has been properly calibrated and all the steps for the solution preparation has been carefully followed to obtain the final result with high accuracy and high precision.
 - (i) Distingush the term accuracy and precision.

(2 marks)

(ii) Identify **two (2)** type of errors that could occur in analytical laboratory with **example** for each type of the error.

(4 marks)

(iii) Discuss **two (2)** approaches that should be taken to verify the accuracy and precision of the result obtained.

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(2 marks)

(e) Traceability is an important part of measurement in the analytical field. Lina is aware that the measurements she makes in the laboratory must be traceable and should be based on similar measurement standards used by other analytical laboratories around the world. Define the term traceability and name one advantage of traceability.

(2 marks)



- Q2 (a) UV-Vis spectroscopy utilized electromagnetic waves of visible light and ultra-violet range to provide an excitation source for material analysis. The result of the measurement is often given in the value of absorbance vs wavelength.
 - (i) State the relationship of absorbance and transmittance in the form of an equation.

(1 mark)

(ii) Determine the typical wavelength range applicable for UV-Vis spectroscopy measurement.

(1 mark)

- (b) Mike has given a solution containing an blue colored chemical compound dissolved in ethanol. The concentration of the solution is not known but is in the range of 0.01 M to 0.02 M. Mike needs to determine the exact value of the sample concentration and intends to measure the concentration using the UV-Vis spectroscopy available in his laboratory.
 - (i) State the suitable solvent to be used as blank in the procedure that Mike conducted.

(1 mark)

(ii) Define **two (2)** precautions that must be taken during sample preparation and during cuvette handling to obtain reliable result for UV-Vis spectroscopy measurement.

(2 marks)

(iii) Elucidate the essential preparation steps and procedures that Mike must conduct to obtain the exact solution concentration.

(6 marks)

- (iv) Calculate the value of absorbance corresponds to 45.0% transmittance (2 marks)
- (v) If a 0.01 M solution gives 45.0% transmittance at some wavelength, calculate the absorbance for a 0.02 M solution of this substance. Given the path length is 1 cm.

(4 marks)

- (c) In a separate occasion, the client sent Mike a new solution that differ in color.
 - (i) Identify if the maximum absorption wavelength (λ_{max}) would differ for the new chemical compared to the blue sample.

(1 mark)

(ii) Justify your answer on Q2(c)(i).

(2 marks)



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- Q3 (a) FTIR is a commonly used equipment for analytical measurement involving various industries such as oil and gas, pharmaceutical and food.
 - (i) Provide **two** (2) important applications of FTIR in industry.

(2 marks)

(ii) Various hydrocarbons and their derivatives are produced by the oil and gas industries. Specify **three** (3) possible functional groups that could be obtained from this industry's products. For each functional group mentioned, provide an example of the product.

(6 marks)

(iii) In a specific incident, water is believed to contaminate certain hydrocarbon produced in a facility. Discuss **two (2)** changes in FTIR spectra due to the contamination.

(2 marks)

- (b) Ali needs to identify the presence of caffeine in coffee powder. Based on this statement,
 - (i) Suggest the instrument used for Ali to solve the problem.

(1 mark)

(ii) Justify your answer in Q3(b)(i).

(1 mark)

(iii) According to Figure Q3(b), discuss two (2) functional groups with wavenumber in order to assist Ali identify the presence of caffeine in coffee powder.

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(6 marks)

(c) Identify **one** (1) difference between the infrared spectrum of carboxylic acid and alcohol other than in their fingerprint regions.

(2 marks)

Q4 (a) High-Performance Liquid Chromatography (HPLC) separates compounds dissolved in a liquid sample and allows qualitative and quantitative analysis of what components and how much of each component is contained in the sample. Briefly retrieve the principle of HPLC with a simple sketch of a diagram.

(5 marks)

(b) Mobile phase supply system in HPLC provides a solvent for the system and governs the separation process. The solvent used as the mobile phase should be filtered and degassed before the analysis. State the function of filtration and degassed steps.

(2 marks)

You are required to prepare a mobile phase with a polarity index of 9.5. Explain how you can prepare this mobile phase using toluene and water. Given the polarity index for toluene = 2.4 and water = 10.2.

(5 marks)

- (d) Predict the order of elution for the separation of butanone, butanol and butene when using silica as the stationary phase. Describe the type of HPLC mode and explain your answer for the order of elution. Given that the molecule polarity is in **Figure Q4(d)**.

 (4 marks)
- (e) Calculate the number of theoretical plates N when the retention time is 15.20 minutes and half of the base width is 0.55 minutes.

(4 marks)

- Q5 (a) The choice of carrier gas often is determined by the needs of the instrument detector.
 - (i) State the function of detector.

(2 marks)

(ii) Distinguish the functionality between the mobile phase and the stationary phase.

(4 marks)

(b) A capillary column requires a different style of injector to avoid overloading the column with the sample. Discuss **one** (1) type injector used in gas chromatographymass spectroscopy (GC-MS).

(2 marks)

(c) Figure Q5(c) elucidates an example of a grapefruit oil analysis by utilizing multiple detectors; (a) Flame ionization (FID), (b) Flame thermionic detector (FTD) and (c) Flame photometric Detector (FPD) at the same time. Express your argument upon the criteria of a detector system suitable for analyzing grapefruit oil via GC-MS.

(4 marks)

(d) Fatima is responsible for identifying and quantifying the unknown sample. She is unsure whether to employ GC or GC-MS. Recommend an appropriate tool and justify your answer in order to assist her.

(4 marks)

(e) Mass spectroscopy (MS) is a technique for determining the mass of atoms and molecules. Depending on the method used to separate ions, there are numerous types of analyzers available. Explain **one** (1) type of mass analyzer used in GC-MS, including its benefits and limitations.

(4 marks)

-END OF QUESTIONS-

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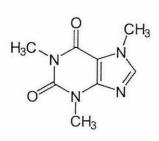


Figure Q3(b)

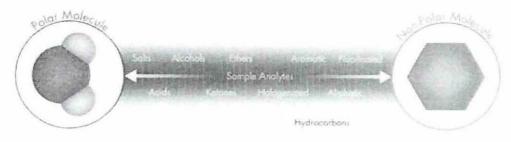


Figure Q4(d)

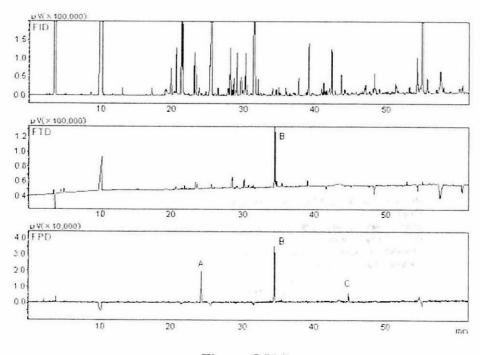


Figure Q5(c)



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THE DATASHEET PROVIDED WITH THE QUESTIONS

Table 1

Infrared Absorption

Bond	Location	Wavenumber/cm ⁻¹
C-H	Alkenes	2850–2950
	Alkenes, arenes	3000–3100
C-C	Alkanes	750–1100
C=C	Alkenes	1620–1680
aromatic C=C	Arenes	Several peaks in range 1450–1650 (variable)
C=O	Aldehydes	1720–1740
	Ketones	1705–1725
	Carboxylic acids	1700-1725
	Esters	1735-1750
	Amides	1630-1700
	Acyl chlorides and acid anhydrides	1750–1820
C-O	Alcohols, ethers, esters and carboxylic acids	1000–1300
C≡N	Nitriles	2220–2260
C–X	Fluoroalkanes	1000–1350
	Chloroalkanes	600-800
	Bromoalkanes	500-600
О-Н	Alcohols, phenols	3200-3600 (broad)
	Carboxylic acids	2500-3300 (broad)
N—H	Primary amines	3300–3500
	Amides	ca. 3500