



**UNIVERSITI TUN HUSSEIN ONN MALAYSIA**

**FINAL EXAMINATION  
(ONLINE)  
SEMESTER I  
SESSION 2020/2021**

COURSE NAME : ANALYTICAL CHEMISTRY  
COURSE CODE : DAS 22403  
PROGRAMME CODE : DAU  
EXAMINATION DATE : JANUARY / FEBRUARY 2021  
DURATION : 2 HOURS 30 MINUTES  
INSTRUCTIONS : ANSWER ALL QUESTIONS

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THIS QUESTION PAPER CONSISTS OF SEVEN (7) PAGES

**Q1** A 500 ml stock standard solution containing 2.370 g of  $\text{KMnO}_4$  standard was readily prepared by the Laboratory Assistant. You are required to prepare five working solutions at  $0.600 \times 10^{-4} \text{ M}$ ,  $1.200 \times 10^{-4} \text{ M}$ ,  $2.4 \times 10^{-4} \text{ M}$ ,  $3.6 \times 10^{-4} \text{ M}$  and  $5.0 \times 10^{-4} \text{ M}$  in 250 ml volumetric flasks

(a) Calculate the volume of  $\text{KMnO}_4$  stock standard solution that you need to pipette for each concentration of working solution. Redraw and complete **Table Q1(a)**. Given the molecular weight of  $\text{KMnO}_4 = 158.034 \text{ g/mol}$ .

(14 marks)

(b) Plot the calibration graph of absorbance versus concentration of  $\text{KMnO}_4$  working solutions. From the calibration curve, draw conclusion based on the curve trend by referring to the equation of the calibration curve.

(7 marks)

(c) Two samples contain unknown concentrations of  $\text{KMnO}_4$ . Determine the concentration of  $\text{KMnO}_4$  in the samples if the absorbance were obtained as in **Table Q1(c)**.

(4 marks)

**Q2** (a) A spectrometer records the degree of absorption by a sample at different wavelengths and the resulting plot of absorbance ( $A$ ) versus wavelength ( $\lambda$ ) is known as a spectrum. Sketch the spectrum in order to show the correlation between absorbance and wavelength.

(3 marks)

(b) Draw the diagram to show the instrument components in UV-Visible Spectrophotometer. Name each component.

(5 marks)

(c) **Table Q2(c)** shows the UV/Vis analysis of **four (4)** standard chemicals at different concentrations.

(i) Redraw and complete the table. Show all calculations.

(14 marks)

(ii) Determine the value of molar absorptivity ( $\epsilon$ ) if the pathlength of the cuvette is 2 cm.

(3 marks)

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**Q3** (a) Answer the following.

(i) Describe the function of the Fourier Transform Infrared (FTIR) Spectroscopy instrument. (1 mark)

(ii) Explain **two (2)** types of vibrational modes. (4 marks)

(iii) In basic components of spectroscopic instruments, distinguish the different components available in UV Vis and FTIR spectroscopy. (2 marks)

(b) (i) Calculate the absorbance of an IR peak with 36% of transmittance. (3 marks)

(ii) Convert  $4.98 \times 10^{13} \text{ s}^{-1}$  of frequency into wavenumber. (3 marks)

(c) An unknown compound is analyzed using FTIR. A strong, sharp peak is observed at a frequency of  $1750 \text{ cm}^{-1}$ .

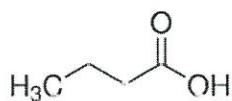
(i) Identify the functional group present. (1 mark)

(ii) Draw the identified structure. (1 mark)

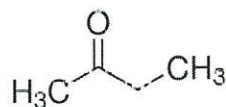
(d) Ali has analysed ethylbenzoate using FTIR instruments and the molecular structure is as shown in **Figure Q3(d)**. Discuss the absorbance bands that must appear in the IR spectrum. (4 marks)

(e) Analyze and match the given IR spectrum shown in **Figure Q3(e)** to one of the following compounds. Illustrate at least **two (2)** absorbance bands (or absence thereof) in the IR that allow you to conclusively identify the compound.

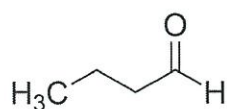
(i)



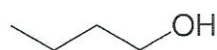
(ii)



(iii)



(iv)



- (6 marks)
- Q4** (a) High Performance Liquid Chromatography (HPLC) is an advanced type of Liquid Chromatography (LC).
- (i) Differentiate between HPLC and classical LC. (2 marks)
- (ii) State the function of column. (1 mark)
- (b) You are required to prepare a mobile phase with polarity index of 8.5. Explain how you can prepare this mobile phase using ethanol and water. Given the polarity index for ethanol – 4.3 and water – 10.2. (5 marks)
- (c) Calculate the number of theoretical plates  $N$  and the plate height  $H$ , when the retention time is 15.20 minutes, half of the base width (given in minutes) is 0.55 minutes and the column length is 25cm. (6 marks)
- (d) By referring to the chromatogram in **Figure Q4(d)**, assuming that barbital is more polar than phenobarbital which is more polar than talbutal. Based on information given, identify either this experiment run under normal or reverse phase conditions. Justify your answer. (3 marks)
- (e) **Figure Q4(e)** shows the results from chromatography instrument by using different columns which are A and B. The resolution of Column B is much better than Column A.
- (i) Describe how to increase the resolution of Column A. (4 marks)
- (ii) Predict the polarity of compound X and Y if this experiment used silica (polar) as stationary phase. Justify your answer by explaining the type of phase condition used. (4 marks)

-END OF QUESTIONS-

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COURSE NAME: ANALYTICAL CHEMISTRYPROGRAMME CODE: DAU  
COURSES CODE: DAS 22403**Table 1 Q1(a) : The volume of  $\text{KMnO}_4$  stock standard solution to be pipetted for each working standard solution concentration**

No	Concentration of $\text{KMnO}_4$ working solution (M)	Volume of $\text{KMnO}_4$ Stock Solution (ml)	Absorbance, A
1	0.00		0.000
2	$0.600 \times 10^{-4}$		0.105
3	$1.200 \times 10^{-4}$		0.212
4	$2.4 \times 10^{-4}$		0.402
5	$3.6 \times 10^{-4}$		0.597
6	$5.0 \times 10^{-4}$		0.825

**Table Q1(c) : The absorbance of  $\text{KMnO}_4$  stock standard solution with concentration**

No	Sample	Absorbance of $\text{KMnO}_4$	Concentration, M
1	A	0.165	
2	B	0.666	

**Table Q2(c) : UV/Vis analysis of samples**

Absorbance at 454 nm	I	$I_0$	T	Concentration (mol/L)
0.00		0.3		
0.15		0.3		
0.50		0.3		
1.00	0.03	0.3		0.04

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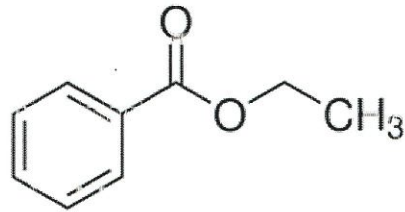


Figure Q3(d)

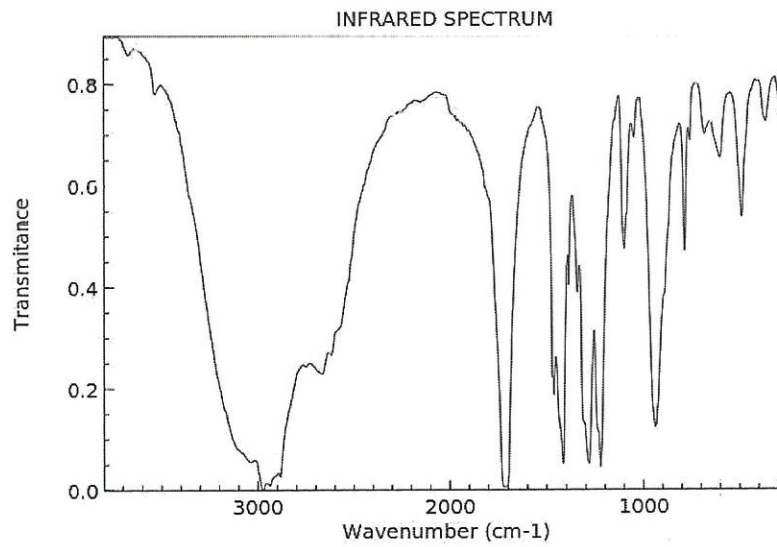
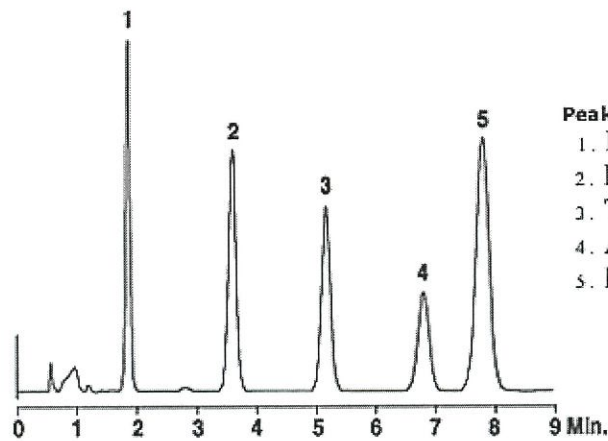


Figure Q3(e)



- Peak Identification
- 1. Barbitol (0.25 mg/ml)
  - 2. Phenobarbital (0.1 mg/ml)
  - 3. Talbutol (0.3 mg/ml)
  - 4. Amobarbital (0.25 mg/ml)
  - 5. Mephobarbital (0.1 mg/ml)

Figure Q4(d)

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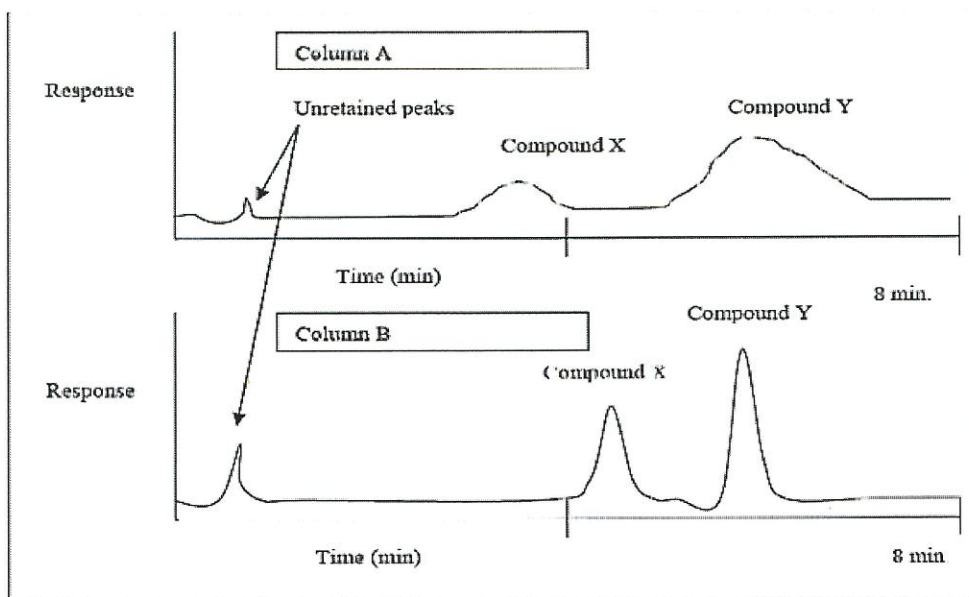


Figure Q4(e)

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