

CONFIDENTIAL



UNIVERSITI TUN HUSSEIN ONN MALAYSIA

**FINAL EXAMINATION
SEMESTER I
SESSION 2019/2020**

COURSE NAME : ANALYTICAL CHEMISTRY
COURSE CODE : DAS 22403
PROGRAMME : DAU
EXAMINATION DATE : DECEMBER 2019 / JANUARY 2020
DURATION : 2 HOURS 30 MINUTES
INSTRUCTIONS : ANSWER ALL QUESTIONS IN
SECTION A AND ONE (1)
QUESTION IN SECTION B

TERBUKA

THIS QUESTION PAPER CONSISTS OF **EIGHT (8)** PAGES

CONFIDENTIAL

SECTION A

Q1 A 500 ml stock standard solution containing 2.370 g of 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 KMnO_4 stock standard solution that you need to pipette for each concentration of working solution. Redraw the table and complete the **Table Q1(a)**. Given $\text{MW} = 158.034$.

(14 marks)

(b) Plot the graph of absorbance versus concentration of KMnO_4 working solutions and 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 KMnO_4 . Determine the concentration of KMnO_4 in the samples if the following absorbance were obtained as in **Table Q1(c)**.

(4 marks)

Q2 (a) (i) Write the Beer's Law equation. State the unit for each parameter.

(4 marks)

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

(5 marks)

(b) **Table Q2(b)** shows the UV/Vis analysis of **five (5)** standard chemicals at different concentrations. Answer the following questions.

(i) Redraw the table and fill in the table completely. Show all the calculations.

(14 marks)

(ii) Determine the value of molar absorptivity, ϵ if the pathlength of the cuvette is 1cm.

(2 marks)

TERBUKA

Q3 (a) Answer the followings.

(i) Define high performance liquid chromatography (HPLC) (3 marks)

(ii) **Three (3)** major components of HPLC and their functions (3 marks)

(iii) Differentiate between HPLC and classical liquid chromatography (2 marks)

(b) 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. (8 marks)

(c) The amount of caffeine in an analgesic tablet was determined by HPLC using a normal calibration curve. Standard solutions of caffeine were prepared and analyzed using a $10\mu\text{L}$ fixed-volume injection loop. Results for the standards are summarized in **Table Q3(c)**.

(i) Draw the calibration curves and find the correlation between caffeine concentration and peak area. (7 marks)

(ii) The sample was analyzed by HPLC and the peak area for the caffeine was found to be 21469. Based on the analysis, calculate the caffeine concentration. (2 marks)

TERBUKA

SECTION B

Q4 (a) Answer the followings.

(i) Definition of functional group.

(2 marks)

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

(4 marks)

(iii) Draw the structure of carboxylic acid and alkene group.

(4 marks)

(b) Calculate the absorbance of an IR peak with 25% of transmittance.

(4 marks)

(c) An unknown compound is analyzed using fourier transform infrared (FTIR). A strong, sharp peak is observed at a frequency of 1750cm^{-1} . Identify the functional group present and draw the structure.

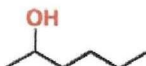
(3 marks)

(d) Match the given IR spectrum shown in **Figure Q4(d)** to one of the following compounds. Label at least **three (3)** absorbance bands (or absence thereof) in the IR that allow you to conclusively identify the compound.

(i)



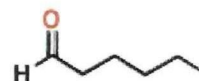
(ii)



(iii)



(iv)



(8 marks)

TERBUKA

Q5 (a) Answer the following.

(i) Definition of chromatography (2 marks)

(ii) Differentiate the isocratic and gradient pump used in chromatographic instruments. (4 marks)

(iii) Distinguish the stationary and mobile phase function used in chromatography. (4 marks)

(b) The concentration of five standards may be determined by gas chromatography mass spectrophotometer (GCMS). Each standard contains the same concentration of an internal standard which is 2.50 mg/mL. For the five standards, the concentrations of analyte as listed in **Table Q5(b)**. Calculate the peak height ratio by using internal standard and draw the calibration curve. (6 marks)

(c) An internal standard is used in the lab experiments using gas chromatography (GC) instead of high performance liquid chromatography (HPLC). Explain the reason for this statement. (3 marks)

(d) The following questions regard to the chromatograms shown in Figure **Q5(d)**. Choose the column that

(i) has greater efficiency. (2 marks)

(ii) gives a greater selectivity factor (for X and Y). (2 marks)

(iii) gives greater resolution between X and Y. (2 marks)

Justify your answer for each Q5(d) (i), (ii) and (iii).

-END OF QUESTIONS-

TERBUKA

CONFIDENTIAL**FINAL EXAMINATION**SEMESTER / SESSION : SEM 1 / 2019/2020
COURSES : ANALYTICAL CHEMISTRYPROGRAMME : DAU
COURSES CODE : DAS 22403**Table 1 Q1(a) : The volume of KMnO_4 stock standard solution to be pipetted for each working standard solution concentration.**

No	Concentration of KMnO_4 working solution (M)	Volume of KMnO_4 Stock Solution (ml)	Absorbance, A
1	0.00		0.000
2	0.600×10^{-4}		0.105
3	1.200×10^{-4}		0.212
4	2.4×10^{-4}		0.402
5	3.6×10^{-4}		0.597
6	5.0×10^{-4}		0.825

Table Q1(c) : The absorbance of KMnO_4 stock standard solution with concentration.

No	Sample	Absorbance of KMnO_4	Concentration, M
1	A	0.165	
2	B	0.666	

Table Q2(b) : 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
1.50		0.3		

TERBUKA

CONFIDENTIAL

FINAL EXAMINATION

SEMESTER / SESSION : SEM 1 / 2019/2020
 COURSES : ANALYTICAL CHEMISTRY

PROGRAMME : DAU
 COURSES CODE : DAS 22403

Table Q3(c) : HPLC analysis of samples

Caffeine concentration (ppm)	HPLC peak area
50.0	8354
100.0	16925
150.0	25218
200.0	33584
250.0	42002

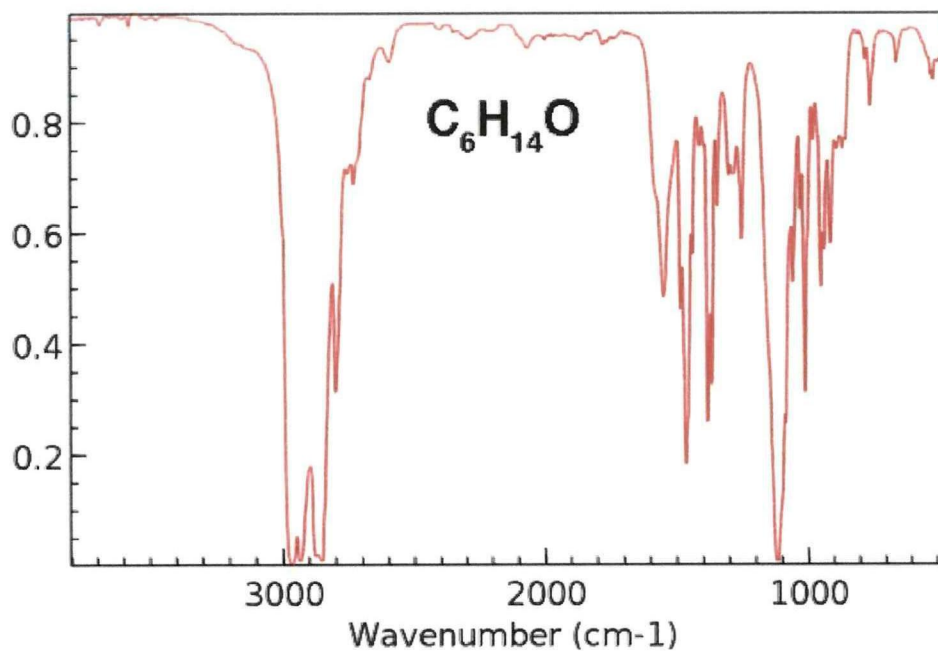


Figure Q4(d)

TERBUKA

Scanned with CamScanner

FINAL EXAMINATION

SEMESTER / SESSION : SEM 1 / 2019/2020
 COURSES : ANALYTICAL CHEMISTRY

PROGRAMME : DAU
 COURSES CODE : DAS 22403

Table Q5(b): Peak heights for each standard

Standard	Internal standard	Analyte	Peak height ratio
0.20	35	7	
0.40	41	16	
0.60	44	27	
0.80	48	39	
1.00	41	21	

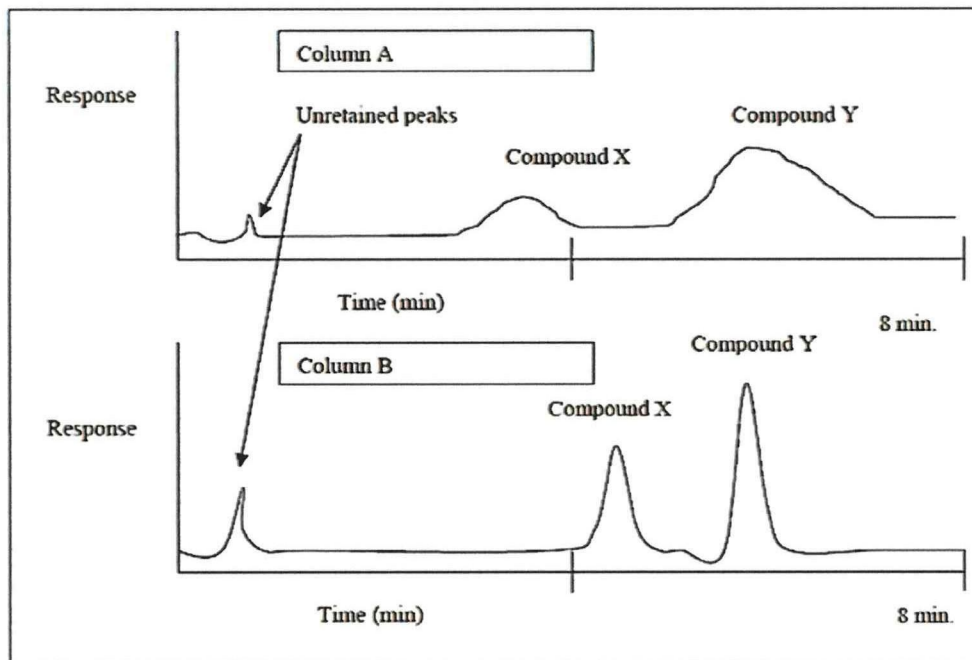


Figure Q5(d)

TERBUKA

TERBUKA