UNIVERSITI TEKNOLOGI PETRONAS

FINAL EXAMINATION
MAY 2024 SEMESTER

COURSE : CEB4032 - ANALYTICAL CHEMISTRY

DATE : 13 AUGUST 2024 (TUESDAY)

TIME : 2.30 PM - 4.30 PM (2 HOURS)

INSTRUCTIONS TO CANDIDATES

1. Answer **ALL** questions in the Answer Booklet.
2. Begin **EACH** answer on a new page in the Answer Booklet.
3. Indicate clearly answers that are cancelled, if any.
4. Where applicable, show clearly steps taken in arriving at the solutions and indicate **ALL** assumptions, if any.
5. **DO NOT** open this Question Booklet until instructed.

Note :

i. There are **THIRTEEN (13)** pages in this Question Booklet including the cover page and appendices.

ii. **DOUBLE-SIDED** Question Booklet.

1. a. Define the precipitation gravimetry and propose a methodology to achieve the precipitate desired for this process.

[5 marks]

b. A 0.3063 g sample of Alpha metal, containing aluminium, magnesium, and other metals, was dissolved and treated to prevent interferences by the other metals. The aluminium and magnesium were precipitated with 8-hydroxyquinoline. After filtering and drying, the mixture of $Al(C_9H_6NO)_3$ and $Mg(C_9H_6NO)_2$ was found to weigh 3.9154 g. The mixture of dried precipitates then was ignited, converting the precipitate to a mixture of Al_2O_3 and MgO. The weight of this mixed solid was found to be 0.5022 g. Molar mass of the compounds is given in the **TABLE Q1**. Calculate the %w/w Al and %w/w Mg in the alloy.

TABLE Q1: Molar mass

Compound	Molar mass ($gmol^{-1}$)
Al_2O_3	101.9600
$Al(C_9H_6NO)_3$	459.4500
MgO	40.3040
$Mg(C_9H_6NO)_2$	312.6100

[12 marks]

c. The efficiency of a particular catalyst is highly dependent on its zirconium content. The starting material for this preparation is received in batches with purity between 58% and 84% $ZrCl_4$. Routine analysis based on precipitation of AgCl is feasible, having been established that there are no sources of chloride ion other than the $ZrCl_4$ in the sample. Given that molar mass of AgCl and $ZrCl_4$ are 143.32 $gmol^{-1}$ and 233.03 $gmol^{-1}$, respectively.

i. Calculate the sample mass that should be taken to ensure an AgCl precipitate weighs at least 0.50 g.

[3 marks]

ii. If this sample mass is used, determine the maximum mass of AgCl that can be expected in this analysis.

[2 marks]

iii. Propose the sample mass that should be taken to have the percentage of $ZrCl_4$ exceed the mass of AgCl produced by a factor of 100.

[3 marks]

2. a. Glucose levels are routinely monitored in patients with diabetes. Glucose concentration in a patient with moderately elevated albumin level was determined in different months using a spectrophotometric analysis. The patient was placed on a low-sugar diet to lower blood glucose levels. The following results tabulated in **TABLE Q2** were obtained during a study to determine the effectiveness of the diet.

TABLE Q2: Glucose concentration by month

Time	Glucose concentration mg/L
Month 1	1292, 1306,1259, 1283, 1301, 1267, 1300
Month 2	1092, 1075, 1122, 1101, 1091
Month 3	888, 905, 879, 922, 900
Month 4	899, 845, 850, 874, 877, 900, 858

i. For each month, calculate the mean, the sum of squared deviations and the standard deviation for the method.

[10 marks]

ii. Recommend the effectiveness of this diet on the patient. Justify your answer.

[5 marks]

b. Lithium is in the alkali-metal group and commonly used in batteries, ceramics, air-conditioning, grease, electric cars, and in pharmaceutical products. Lithium can be found in continental brine waters, geothermal waters, and oil-gas field brines.

i. Propose and briefly explain a suitable instrument used to determine lithium in water sample.

[5 marks]

ii. Calculate the mass of hydrogen (1.000 $gmol^{-1}$) formed by the complete reaction of 80.570 g of Lithium (6.941 $gmol^{-1}$) with water.

[5 marks]

3. a. **TABLE Q3a** tabulates a set of data for a calibration curve used last year to correlate calcium [Ca] concentration with a reading on an atomic emission machine. At the end of this experiment, household tap water was tested and readings in **TABLE Q3a** was obtained. From the data, calculate the concentration of Ca in the tap water and determine the uncertainty in this number. Include a complete spreadsheet for your analysis and state assumptions or adjustments required to be made in order to obtain the final number.

TABLE Q3a: Data analysis and calibration curve data

[Ca] ppm	Emission run 1	Emission run 2
493	1.035	1.040
370	0.905	0.910
247	0.735	0.745
123	0.512	0.526
0	0.000	0.010
Tap water	0.790	0.810

[10 marks]

b. The gas chromatography data were obtained for two compounds separated on a 2 m pack column are tabulated in **TABLE Q3b.**

TABLE Q3b: Retention time and width

Compound	Retention time, t_r (s)	Width at the base, w (s)
A	350.0	19.8
B	370.0	20.3

i. Calculate the number of theoretical plates (N) for each compound and justify the different number of theoretical plates for the different compounds.

[5 marks]

ii. If the retention time for an unretained solute is 50.0 s, evaluate the resolution and selectivity factors for the above compounds and suggest method to improve the resolution.

[5 marks]

iii. Estimate the height of theoretical plates to achieve resolution (R_s) of 1.5 without increasing the length of the column and time required to elute substance B on the column.

[5 marks]

4. a. **FIGURE 4Qa** shows chromatograms of five standards and one sample. Each standard and sample contain the same concentration of an internal standard, which is 2.50 mg/mL. For the five standards, the concentrations of analyte are 0.20 mg/mL, 0.40 mg/mL, 0.60 mg/mL, 0.80 mg/mL, and 1.00 mg/mL, respectively.

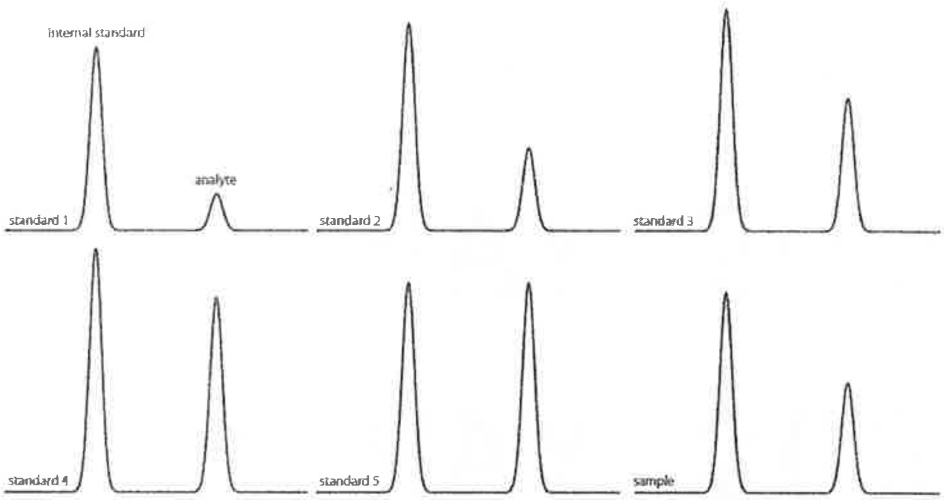


FIGURE Q4a: Chromatograms

i. Determine the concentration of analyte in the sample by (a) ignoring the internal standards and creating an external standards calibration curve, and by (b) creating an internal standard calibration curve.

[5 marks]

ii. For each approach, report the analyte's concentration and the 95% confidence interval. Use peak heights instead of peak areas.

[5 marks]

b. Determine the best molecule structure that corresponds to the IR spectrum in **FIGURE Q4b** below with molecular formula of $C_6H_{14}O$.

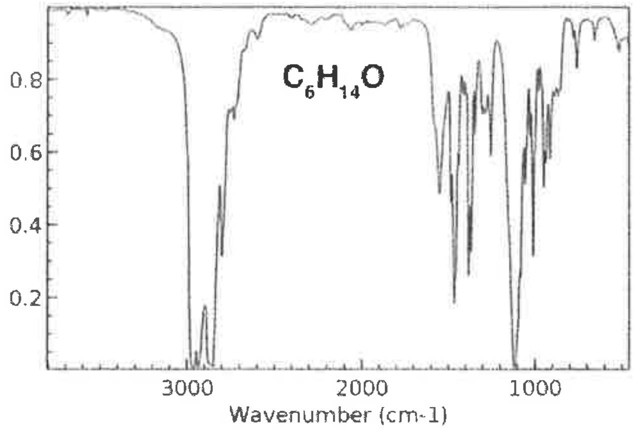


FIGURE Q4b: IR spectrum

[5 marks]

c. Organic compounds ($C_xH_yO_z$) with a molecular weight of 72.11 $gmol^{-1}$ produced two different IR spectra, A and B as shown in **FIGURE Q4c** and **FIGURE Q4d,** respectively. Analyse the spectra by considering **ALL** possible functional groups. Identify and state the **IUPAC** name of both compounds.

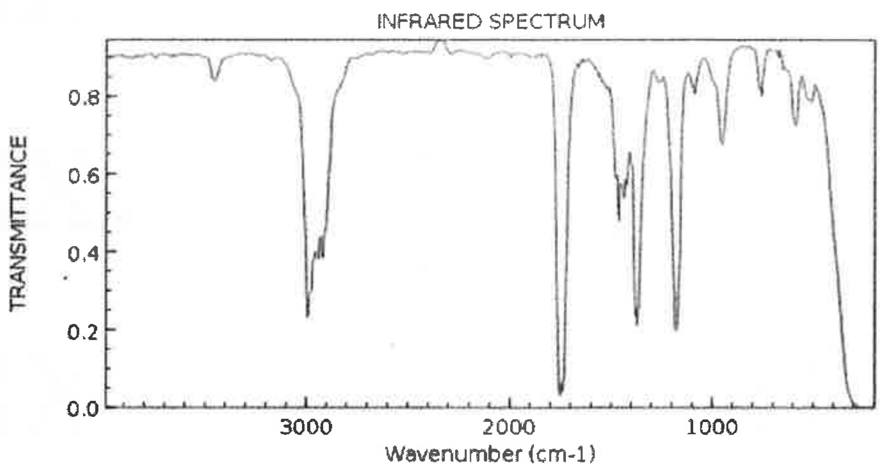


FIGURE Q4c: Spectrum A

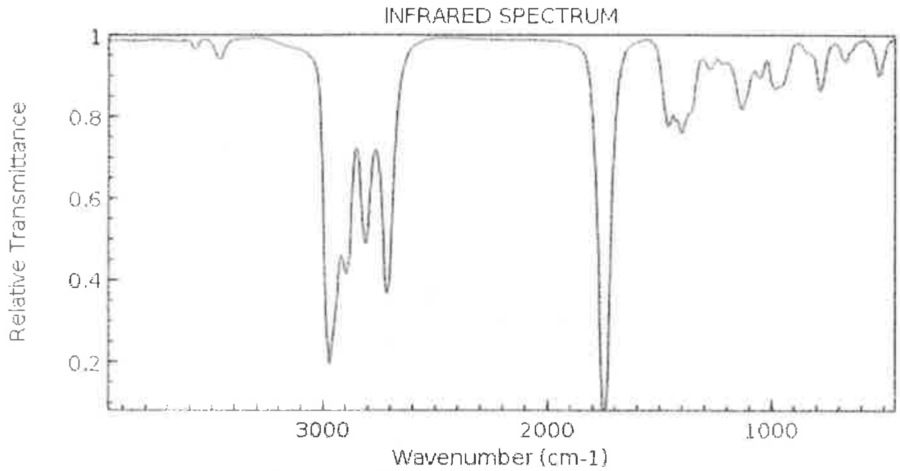


FIGURE Q4d: Spectrum B

[10 marks]

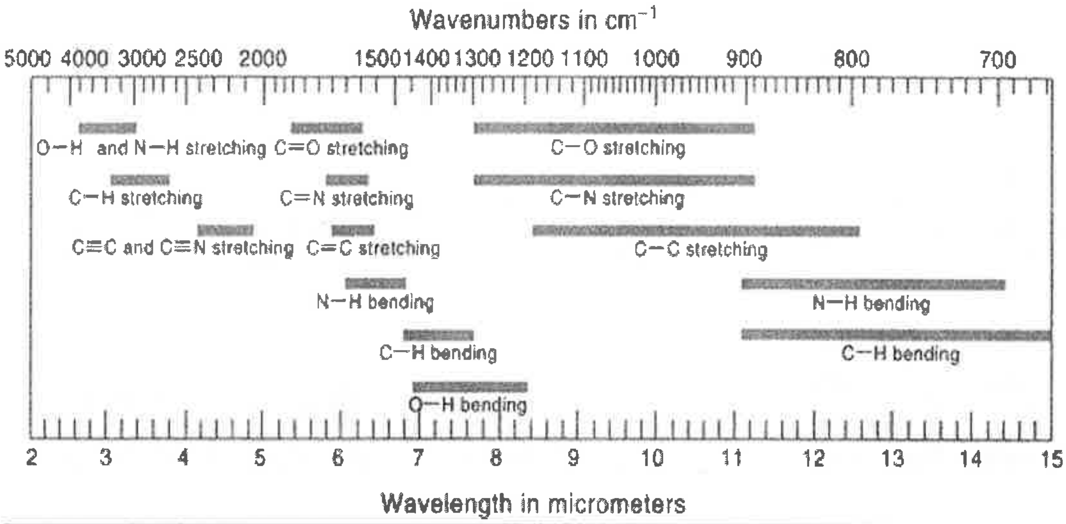
–END OF PAPER–

APPENDIX A

Table A1: FTIR Group Frequencies for Organic Functional Groups

Bond	**Type of Compound**	**Frequency Range, cm^{-1}**	**Intensity**
C-H	Alkanes	2850-2970	Strong
C-H	Alkenes	3010-3095 675-995	Medium strong
C-H	Alkynes	3300	Strong
C-H	Aromatic rings	3010-3100 690-900	Medium strong
O-H	Monomeric alcohols, phenols Hydrogen-bonded alcohols, phenols Monomeric carboxylic acids Hydrogen-bonded carboxylic acids	3590-3650 3200-3600 3500-3650 2500-2700	Variable Variable, sometimes broad Medium broad
N-H	Amines, amides	3300-3500	medium
C=C	Alkenes	1610-1680	Variable
C=C	Aromatic rings	1500-1600	Variable
	Alkynes	2100-2260	Variable
C-N	Amines, amides	1180-1360	Strong
[illegible]	Nitriles	2210-2280	Strong
C-O	Alcohols, ethers,carboxylic acids, esters	1050-1300	Strong
C=O	Aldehydes, ketones, carboxylic acids, esters	1690-1760	Strong
NO_2	Nitro compounds	1500-1570 1300-1370	Strong

TABLE A2: Simple correlations of group vibrations to regions of infrared absorption



APPENDIX B

List of Formula

Beer's Law

$A_{Xs} = \varepsilon_x b[X]_s$
$A_{Ys} = \varepsilon_y b[Y]_s$
$A_{Zs} = \varepsilon_z b[Z]_s$
$A_m = \varepsilon_x b[X] + \varepsilon_y b[Y] + \varepsilon_z b[Z]$

% Analyte

$$\% A = \frac{\text{weight of ppt x gravimetric factor (G}}{\text{weight of sample}}$$

Area of Gaussian peak

$= 1.064 \times h \times W_{1/2}$

$= 1.064 \times h \times 2.35\,\sigma$

Adjusted retention time, $t'_r = t_r - t_m$

Relative retention, $\alpha = t'_{r2}/t'_{r1} = k'_2/k'_1$

Capacity factor, $k' = (t_r - t_m)/t_m = t'_r/t_m$

Number of plates, $N = 16\, t_r^2/w^2 = 5.55\, t_r^2/w_{1/2}^2$

Plate height, $H = L/N$

Resolution $= \Delta t_r/w_{av}$

$$= \frac{\sqrt{N}}{4}\left(\frac{\alpha-1}{\alpha}\right)\left(\frac{k'_2}{1+k'_{av}}\right)$$

Response factor:

$$\frac{A_X}{[X]} = F\left(\frac{A_S}{[S]}\right)$$

Al= 26.98 g/mol

N = 14 g/mol

S =32.07 g/ mol

O = 16 g/mol

Cl= 35.45 g/mol