UNIVERSITI TEKNOLOGI PETRONAS

FINAL EXAMINATION
JANUARY 2025 SEMESTER

COURSE	**:**	**CEB4032/CFB3032 - ANALYTICAL CHEMISTRY/ANALYTICAL INSTRUMENTATION**
DATE	**:**	**12 APRIL 2025 (SATURDAY)**
TIME	**:**	**9.00 AM - 11.00 AM (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 **EIGHT (8)** pages in this Question Booklet including the cover page and appendices.

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

1. a. Two analysts perform replicate sets of lead (Pb^{2+}) determinations of a same river water sample. The concentration of lead in ppm are shown in **TABLE Q1.**

TABLE Q1: Lead concentration in river water sample.

	Pb^{2+} concentration (ppm)			
Analyst 1	0.045	0.048	0.046	0.050
Analyst 2	0.047	0.044	0.048	0.045

i. Calculate the coefficient of variation for each replicate.

[10 marks]

ii. Discuss **TWO (2)** reasons that can cause different results in **part a(i).**

[4 marks]

iii. Suggest and explain the basic principle of the instrument used during the analysis.

[6 marks]

b. Magnesium reacts with hydrochloric acid to produce magnesium chloride and hydrogen. Calculate the grams of hydrogen (MW = 1.00 g/mol) released when 60.0 g of magnesium (MW = 24.31 g/mol) reacts completely with excess hydrochloric acid.

[5 marks]

2. a. Mercury (II) ions, Hg^{2+} (MW = 200.59 g/mol) in a 0.8142 g sample was precipitated with an excess of paraperiodic acid, H_5IO_6. The resulting precipitate, $Hg_5(IO_6)_2$ (MW = 1448.76 g/mol) was filtered, washed, dried, weighed and 0.4114 g was recovered.

i. Construct an experimental outline to produce $Hg_5(IO_6)_2$ from the reaction between mercury (II) ions and paraperiodic acid and show the chemical balance of the reaction.

[8 marks]

ii. Calculate the percentage of mercury (II) ions (Hg^{2+}) in the original sample.

[5 marks]

b. Compound A and compound B produced two different Fourier-transform infrared spectroscopy (FTIR) spectra as shown in **FIGURE Q2b(i)** and **FIGURE Q2b(ii).** The molecular weight of compound A and compound B are 46.07 g/mol and 60.05 g/mol, respectively. Given that, the molecular formula for both compounds are ($C_xH_yO_z$), analyze the spectra by considering **ALL** possible functional groups. Identify and state the **IUPAC** name of both compounds.

[12 marks]

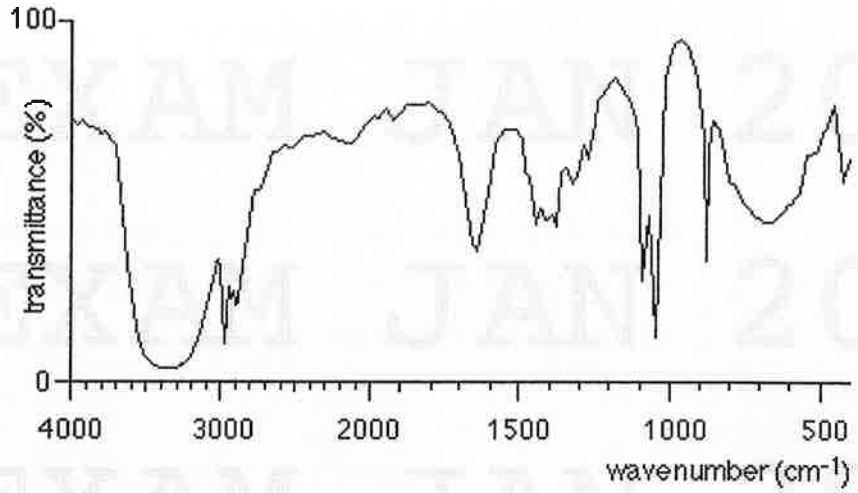

FIGURE Q2b(i): FTIR for compound A.

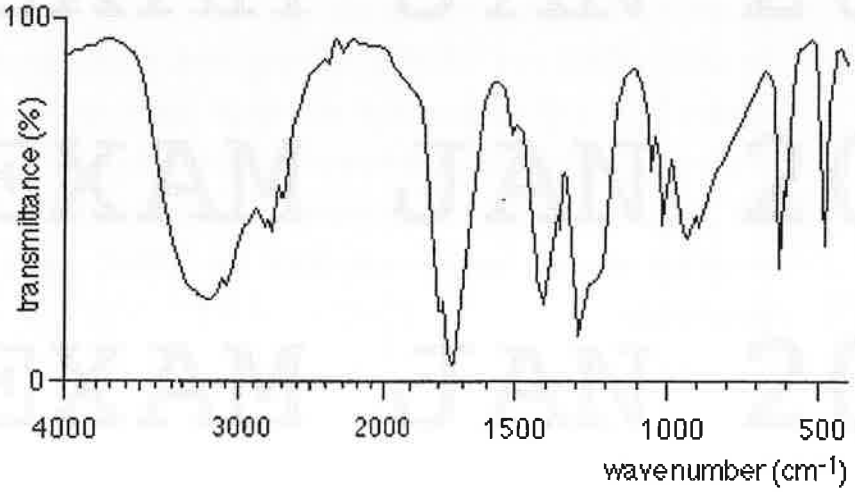

FIGURE Q2b(ii): FTIR for compound B.

3. a. Using energy level diagram, discuss the similarity and compare the absorption of radiation between an atomic substance and a molecular substance.

[8 marks]

b. Atomic spectroscopy is the study of the electromagnetic radiation absorbed and emitted by atoms.

i. Differentiate atomic emission spectroscopy (AES) and atomic absorption spectroscopy (AAS).

[6 marks]

ii. In Atomic Absorption Spectroscopy (AAS), the absorbance of iron (Fe) decreases in the presence of high concentrations of sulfate ions (SO_4^{2-}). This occurs due to the formation of stable iron-sulfate complexes, which reduce the availability of free Fe atoms for absorption. Explain the reason(s) and suggest **TWO (2)** method to overcome this observation.

[6 marks]

c. The Beer-Lambert law is the linear relationship between absorbance (A) and concentration (c) of an absorbing species. Derive the mathematical expression that correlates absorbance with concentration (c) and path length (b) of a chemical sample.

[5 marks]

4. a. Chromatography is a powerful separation technique used to analyze complex mixtures by distributing components between a mobile phase and a stationary phase. The separation occurs due to differences in affinity between the sample components and the stationary phase, allowing qualitative and quantitative analysis. Using schematic diagrams, discuss the basic principle of Gas Chromatography (GC) and Liquid Chromatography (LC).

[10 marks]

b. An experiment was conducted to separate four different compounds (benzene, aniline, *n*-hexane and phenol) using high performance liquid chromatography (HPLC).

i. Distinguish the difference between 'normal phase' and 'reversed phase'. Sketch the diagrams to support your answer.

[7 marks]

ii. Rank and discuss the order of the four compounds would be eluted from an HPLC column containing a reversed-phase packing.

[8 marks]

–END OF PAPER–

APPENDIX A

FTIR Group Frequencies for Organic Functional Groups

Type of Compound	**Frequency Range, cm^{-1}**	**Intensity**
Alkanes	2850-2970	Strong
Alkenes	3010-3095 675-995	Medium strong
Alkynes	3300	Strong
Aromatic rings	3010-3100 690-900	Medium strong
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
Amines, amides	3300-3500	medium
Alkenes	1610-1680	Variable
Aromatic rings	1500-1600	Variable
Alkynes	2100-2260	Variable
Amines, amides	1180-1360	Strong
Nitriles	2210-2280	Strong
Alcohols, ethers,carboxylic acids, esters	1050-1300	Strong
Aldehydes, ketones, carboxylic acids, esters	1690-1760	Strong
Nitro compounds	1500-1570 1300-1370	Strong

APPENDIX B

List of Formula

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, $N = 16\ t^2_r/w^2 = 5.55\ t^2_r/w^2_{1/2}$

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:

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