UNIVERSITI
TEKNOLOGI
PETRONAS

FINAL EXAMINATION
JANUARY 2025 SEMESTER

COURSE : **YBB1053 - ANALYTICAL INSTRUMENTATION**
DATE : **10 APRIL 2025 (THURSDAY)**
TIME : **2:30 PM - 5:30 PM (3 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 **FOURTEEN (14)** pages in this Question Booklet including the cover page and Appendices.
- ii. **DOUBLE-SIDED** Question Booklet.
- iii. Graph paper(s) will be provided

1. Atomic spectroscopy is an analytical technique used to determine the concentration of elements in a sample by measuring the interaction between electromagnetic radiation and free atoms. This technique is widely applied in various fields, including environmental analysis, pharmaceuticals, food safety, and metallurgy. Among the different types of atomic spectroscopy, atomic absorption spectroscopy (AAS) and atomic emission spectroscopy (AES) are the most used.

   a. Explain the difference in the mode of measurement between AAS and AES when analyzing trace metals. Your answer should include the principles behind each technique.

   [4 marks]

   b. **FIGURE Q1** shows the absorption spectrum of AAS from an iron hollow cathode lamp.

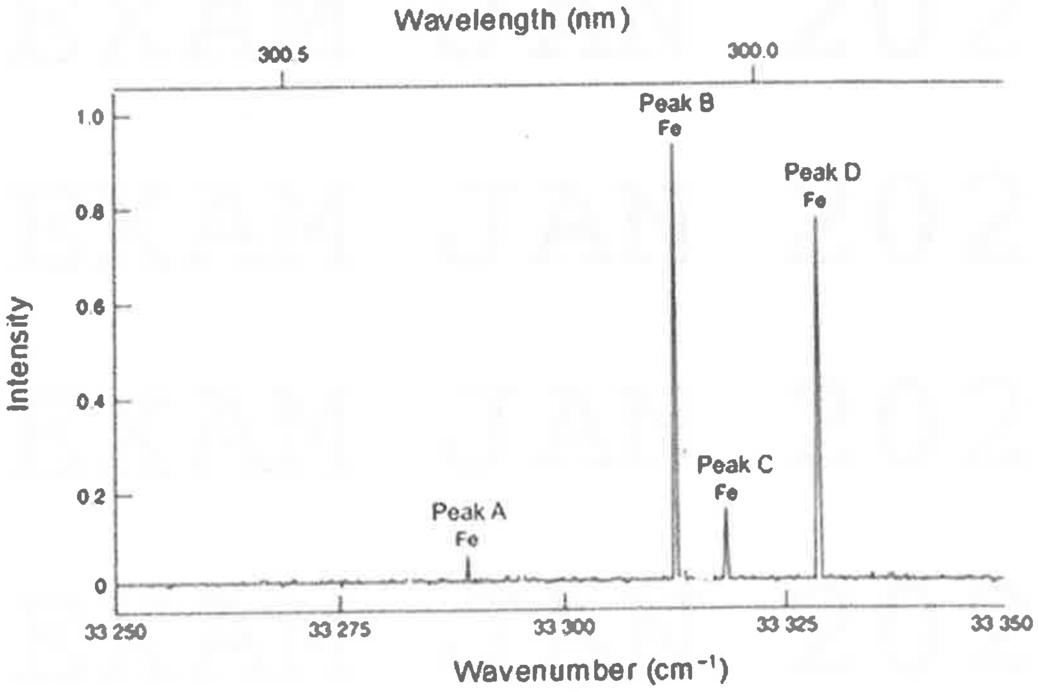


FIGURE Q1: Spectrum of AAS from an iron hallow cathode lamp.

i. Explain the reasons for the sharp peaks in the spectrum at specific wavelengths.

[3 marks]

ii. Select the peak that serves as the reference for the quantitative analysis of iron and justify your selection.

[3 marks]

c. **TABLE Q1** presents the data for external standard solutions of potassium ions obtained using AES. All emission intensities were background-corrected by subtracting the blank intensity.

TABLE Q1: Emission intensity for external standard solutions of potassium.

Concentration of external standard solution (ppm)	**Emission intensity**
1.0	5.0
2.0	10.0
5.0	26.5
10.0	55.2
15.0	81.5
20.0	110.2

i. Plot an appropriate graph using the external standard solution data to derive the equation of the straight line.

[7 marks]

ii. The sample containing potassium ions was analyzed using AES, and the emission intensity was measured as 23.5. Calculate the potassium concentration (in ppm) in the sample using your answer from **part (c)(i)**.

[3 marks]

2. Ultraviolet-visible (UV-vis) spectroscopy is a technique that analyzes light in the ultraviolet and visible regions of the electromagnetic spectrum. It measures the amount of light absorbed or transmitted by a sample relative to a blank reference. UV-vis spectroscopy is based on the electronic transitions in molecules when they absorb light at specific frequencies characteristic of their structure.

   a. Draw a block diagram illustrating the configuration of components in a UV-vis spectrometer. Label all components in the diagram.

   [4 marks]

   b. **FIGURE Q2(i)** shows the molecular structure of Rose Bengal (4,5,6,7-tetrachloro-2',4',5',7'-tetraiodofluorescein) which has a molecular weight of 1017.64 g mol^{-1}. The concentrations and UV-Vis spectra of the external standard solutions of Rose Bengal are shown in **TABLE Q2** and **FIGURE Q2(ii)**, respectively. A sample of Rose Bengal was prepared and analyzed using a UV-Vis spectrometer, where it exhibited an absorbance of 0.786 at lambda maxima (λ_{max}).

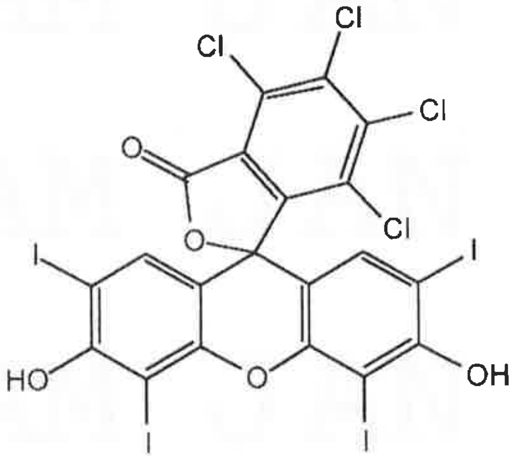


FIGURE Q2(i): Molecular structure of Rose Bengal

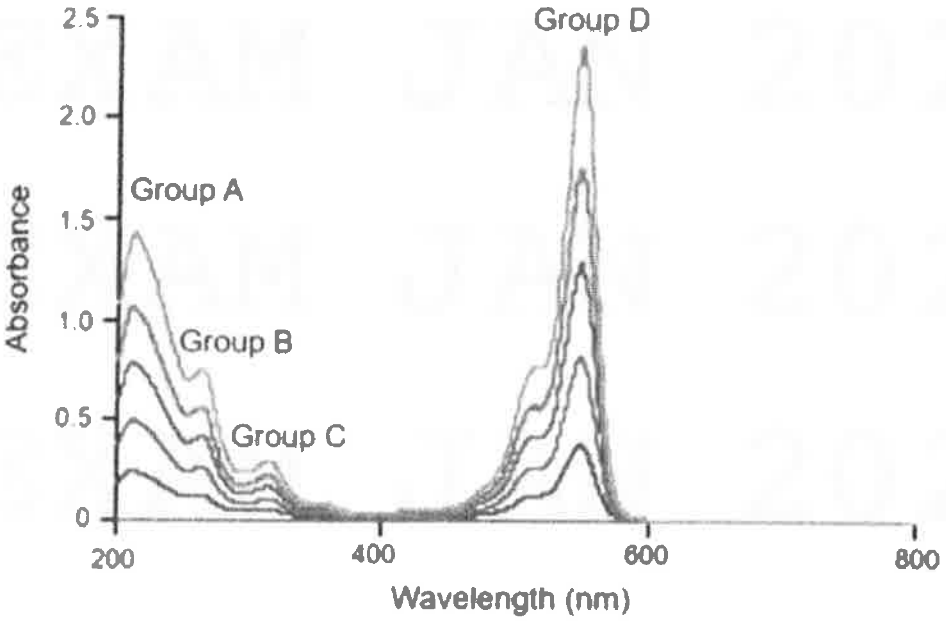


FIGURE Q2(ii): UV-Vis spectra for external standard solutions of Rose Bengal.

TABLE Q2: Absorbance intensity for external standard solutions of Rose Bengal.

Concentration of standard solution (ppm)	**Absorbance intensity**			
	Group A	**Group B**	**Group C**	**Group D**
5.0	0.250	0.120	0.095	0.395
10.0	0.495	0.376	0.165	0.796
15.0	0.850	0.451	0.250	1.350
20.0	1.099	0.599	0.299	1.748
25.0	1.489	0.788	0.375	2.382

i. A UV-vis spectrometer can analyze Rose Bengal both quantitatively and qualitatively. Explain **TWO (2)** factors that support this statement.

[4 marks]

ii. There are four groups of peaks in **FIGURE Q2(ii)**: Group A, Group B, Group C, and Group D. Among these groups, suggest which group best corresponds to λ_{max} and justify your answer.

[3 marks]

iii. Based on your answers in **part (b)(ii)** and the data in **TABLE Q2**, construct the calibration curve and deduce the linear regression equation for the curve.

[7 marks]

iv. Based on your answers in **part (b)(iii)**, calculate the concentration of Rose Bengal in ppm.

[2 marks]

3. Infrared (IR) spectroscopy identifies chemical substances through molecular vibrations, as bonds absorb infrared radiation at frequencies unique to their functional groups. Fourier-transform infrared (FTIR) spectroscopy, a modern variation of IR spectroscopy, uses an interferometer and Fourier transformation to generate interpretable spectra.

   a. Describe the fundamental principle of IR spectroscopy. In your answer, explain how IR radiation interacts with molecules and the type of molecular information that can be obtained from an IR spectrum.

   [5 marks]

   b. Describe **TWO (2)** requirements that a molecular sample must meet to be active in FTIR spectroscopy.

   [4 marks]

   c. The FTIR spectrum of an unknown organic compound shows a strong, broad absorption band around 3400 cm^{-1} and a sharp peak near 1700 cm^{-1}. Identify the functional groups corresponding to these absorptions and propose a possible class of organic compounds.

   [5 marks]

d. Sample X, with the molecular formula $C_6H_{14}O$, was analyzed using FTIR spectroscopy. The resulting FTIR spectrum is shown in **FIGURE Q3(i)**.

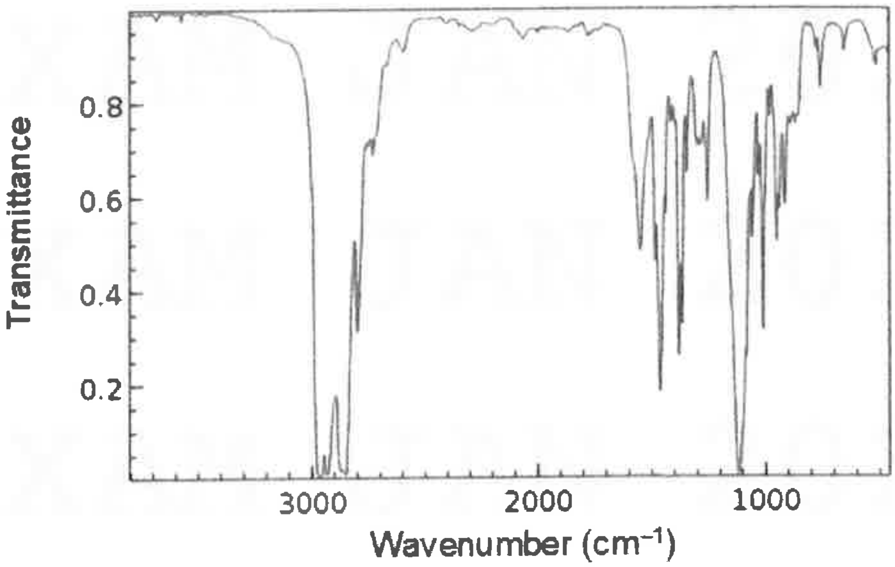
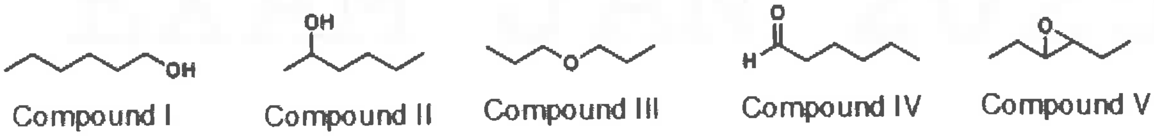


FIGURE Q3(i): FTIR spectrum of $C_6H_{14}O$

Given the compounds in **FIGURE Q3(ii)**, identify **ONE (1)** compound that best corresponds to sample X. Explain your answer.



Compound I Compound II Compound III Compound IV Compound V

FIGURE Q3(ii): Compounds I, II, III, IV and V

[6 marks]

4. Nuclear Magnetic Resonance (NMR) spectroscopy is a powerful analytical technique used to determine the structure, dynamics, and environment of molecules. In organic chemistry, proton NMR (^{1}H-NMR) and carbon-13 NMR (^{13}C-NMR) provide key insights into the structure and environment of hydrogen and carbon atoms, respectively.

   a. Explain the fundamental principles of NMR spectroscopy. In your answer(s), discuss the role of the magnetic field and radiofrequency radiation in generating an NMR signal.

   [5 marks]

   b. The ^{1}H NMR spectrum of an unknown compound shows a triplet at 1.2 ppm with an integration of 3, a quartet at 2.5 ppm with an integration of 2, and a singlet at 4.0 ppm with an integration of 1. Identify the compound and justify your answer.

   [5 marks]

   c. **FIGURE Q4** shows the proton nuclear magnetic resonance (^{1}H-NMR) spectrum of iodoethane (CH_3CH_2I).

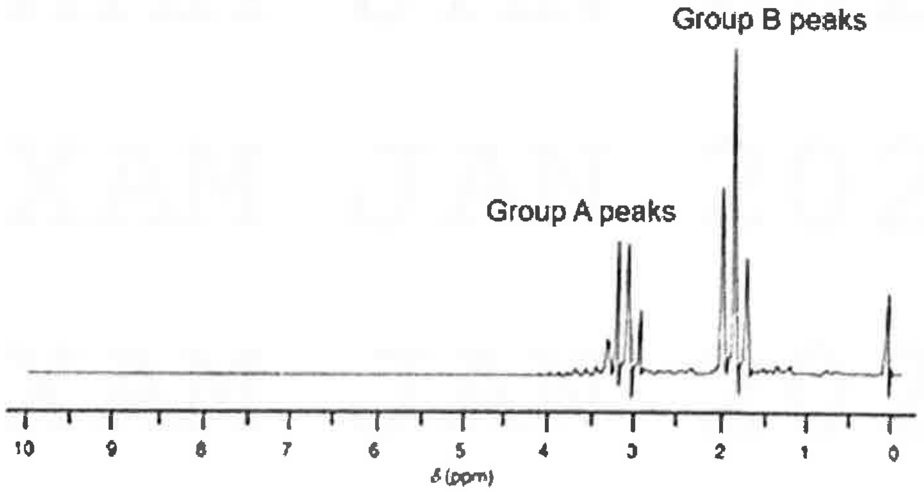


FIGURE Q4: ^{1}H-NMR spectrum of CH_3CH_2I

i. Identify protons that are most shielded. Justify your answers.

[4 marks]

ii. Match the protons with the corresponding peaks in **FIGURE Q3** based on their splitting patterns. Justify your answers.

[6 marks]

5. Chromatography is a versatile analytical technique used to separate, identify, and quantify components within a mixture. Two commonly used chromatographic techniques are gas chromatography (GC) and high-performance liquid chromatography (HPLC).

   a. Construct a block diagram for both GC and HPLC. Identify all the basic components.

   [6 marks]

   b. One key difference between GC and HPLC is the mobile phase. Explain how this difference affects the operational principles in relation to the volatility of the sample.

   [4 marks]

   c. HPLC is used to separate a mixture of three compounds. Explain the effect of the following parameters on the separation: mobile phase composition, flow rate and column temperature.

   [6 marks]

   d. The flame ionization detector (FID) is a commonly used detector in GC. Describe **TWO (2)** key advantages of using FID when analyzing organic compounds.

   [4 marks]

- END OF PAPER-

APPENDIX I

List of Atomic Masses

Elements	Symbol	Atomic No.	Atomic Mass	Elements	Symbol	Atomic No.	Atomic Mass
Actinium	Ac	89	[227]	Mercury	Hg	80	200.59
Aluminium	Al	13	26.98	Molybdenum	Mo	42	95.94
Americium	Am	95	[243]	Neodymium	Nd	60	144.24
Antimony	Sb	51	121.76	Neon	Ne	10	20.18
Argon	Ar	18	39.95	Nickel	Ni	28	58.69
Arsenic	As	33	74.92	Niobium	Nb	41	92.91
Astatine	At	85	[210]	Nitrogen	N	7	14.01
Barium	Ba	56	137.33	Osmium	Os	76	190.23
Berkelium	Bk	97	[247]	Oxygen	O	8	15.99
Beryllium	Be	4	9.01	Palladium	Pd	46	106.42
Bismuth	Bi	83	208.98	Phosphorus	P	15	30.97
Bohrium	Bh	107	[264]	Platinum	Pt	78	195.08
Boron	B	5	10.81	Plutonium	Pu	94	[244]
Bromine	Br	35	79.90	Polonium	Po	84	[209]
Cadmium	Cd	48	112.41	Potassium	K	19	39.10
Caesium	Cs	55	132.90	Radium	Ra	88	[226]
Calcium	Ca	20	40.08	Radon	Rn	86	[222]
Californium	Cf	98	[251]	Rhenium	Re	75	186.21
Carbon	C	6	12.01	Rhodium	Rh	45	102.90
Cerium	Ce	58	140.12	Rubidium	Rb	37	85.47
Chlorine	Cl	17	35.45	Ruthenium	Ru	44	101.07
Chromium	Cr	24	51.99	Samarium	Sm	62	150.36
Cobalt	Co	27	58.93	Scandium	Sc	21	44.96
Copper	Cu	29	63.55	Selenium	Se	34	78.96
Fluorine	F	9	18.99	Silicon	Si	14	28.09
Gadolinium	Gd	64	157.25	Silver	Ag	47	107.87
Gallium	Ga	31	69.72	Sodium	Na	11	22.99
Germanium	Ge	32	72.64	Strontium	Sr	38	87.62
Gold	Au	79	196.97	Sulfur	S	16	32.06
Hafnium	Hf	72	178.49	Tellurium	Te	52	127.60
Helium	He	2	4.00	Terbium	Tb	65	158.92
Holmium	Ho	67	164.93	Thallium	Tl	81	204.38
Hydrogen	H	1	1.01	Thorium	Th	90	232.04
Indium	In	49	114.82	Thulium	Tm	69	168.93
Iodine	I	53	126.90	Tin	Sn	50	118.71
Iridium	Ir	77	192.22	Titanium	Ti	22	47.87
Iron	Fe	26	55.84	Tungsten	W	74	183.84
Krypton	Kr	36	83.80	Uranium	U	92	238.03
Lanthanum	La	57	138.91	Vanadium	V	23	50.94
Lead	Pb	82	207.2	Xenon	Xe	54	131.29
Lithium	Li	3	[6.94]	Ytterbium	Yb	70	173.04
Lutetium	Lu	71	174.97	Yttrium	Y	39	88.91
Magnesium	Mg	12	24.31	Zinc	Zn	30	65.41
Manganese	Mn	25	54.94	Zirconium	Zr	40	91.22

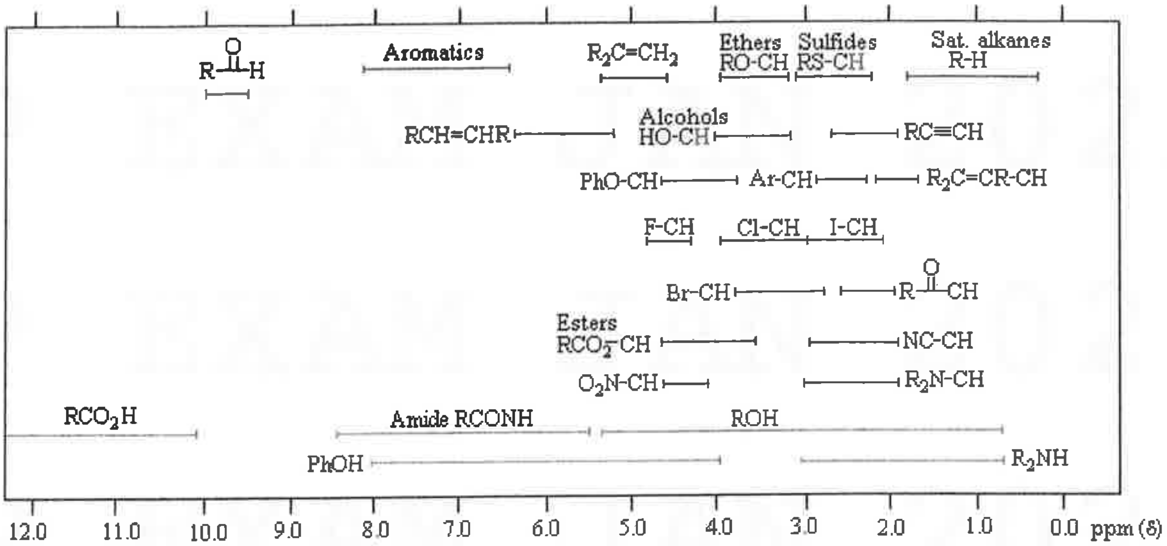
APPENDIX II

IR Absorptions of Common Functional Groups

Functional Group	*Absorption Location (cm^{-1})*	*Absorption Intensity*
Alkane (C–H)	2,850–2,975	Medium to strong
Alcohol (O–H)	3,400–3,700	Strong, broad
Alkene (C=C)	1,640–1,680	Weak to medium
(C=C–H)	3,020–3,100	Medium
Alkyne		
(C≡C)	2,100–2,250	Medium
(C≡C–H)	3,300	Strong
Nitrile (C≡N)	2,200–2,250	Medium
Aromatics	1,650–2,000	Weak
Amines (N–H)	3,300–3,350	Medium
Carbonyls (C=O)	1,720–1,740	Strong
Aldehyde (CHO)	1,720–1,740	
Ketone (RCOR)	1,715	
Ester (RCOOR)	1,735–1,750	
Acid (RCOOH)	1,700–1,725	

APPENDIX III

^{1}H-NMR Chemical shifts



For samples in CDCl$_3$ solution. The d scale is relative to TMS at δ = 0.