Universiti Tun Hussein Onn Malaysia

# UNIVERSITI TUN HUSSEIN ONN MALAYSIA

## FINAL EXAMINATION
## SEMESTER II
## SESSION 2013/2014

| | | |
|---|---|---|
| COURSE NAME | : | ANALYTICAL CHEMISTRY |
| COURSE CODE | : | DAS 12403 |
| PROGRAMME | : | 1 DAU |
| EXAMINATION DATE | : | JUNE 2014 |
| DURATION | : | 2 HOURS 30 MNUTES |
| INSTRUCTION | : | ANSWER **FOUR (4)** QUESTIONS ONLY |

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

**Q1** (a) Explain the following terms;

(i) analyte
(ii) solvent
(iii) solution

(3 marks)

(b) A typical quantitative analysis involves a sequence of steps. List the steps involved in the quantitative analysis and explain each of the steps briefly.

(7 marks)

(c) Given four laboratory apparatus to be used in titrimetric analysis: pipette, burette, conical flask and volumetric flask. Answer the following questions.

(i) Explain the use of each apparatus

(4 marks)

(ii) Arrange the apparatus in ascending order in terms of accuracy

(4 marks)

(iii) Explain the reason of the answer in (ii).

(2 marks)

(d) State five (5) laboratory rules to be strictly followed when you are in the chemistry laboratory.

(5 marks)

**Q2** (a) Determine the concentration of the following solution in molarity (M) and % w/v.

(i) 0.805 mol of NaCl dissolved in 3.55 L deionised water
(ii) 2.053 g of $CuSO_4.5H_2O$ dissolved in 250.0 ml distilled water

(Na=23.000 g/mol, Cl=35.453 g/mol, Cu=63.546 g/mol,S= 32.066 g/mol, O = 16.000 g/mol, H = 1.000 g/mol)

(8 marks)

(b) A solution of $KNO_3$, 500.0 ml with 0.35 M concentration, is needed. Describe how you would prepare the above solution

(i) from pure, solid $KNO_3$ ($KNO_3$ = 101.105 g/mol)

(4 marks)

(ii) from a solution of $KNO_3$ that is 4.375 M

(4 marks)

(c) A water sample taken from a lake was analyzed for pH value. Ten replicate measurements of the pH were recorded as 2.80, 3.10, 2.85, 2.65, 2.88, 3.20, 3.05, 3.15, 2.90 and 2.95.

(i) Explain the importance of performing replicate measurements.

(1 marks)

(ii) Calculate the mean of this analysis.

(2 marks)

(iii) Calculate the standard deviation of the data.

(2 marks)

(iv) Find the relative standard deviation (RSD) of the data.

(2 marks)

(v) Conclude on the standard deviation results obtained in (iii).

(2 marks)

Given : $\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$, $s = \sqrt{\frac{\sum_{i=1}^{n} (xi - \bar{x})^2}{n-1}}$

**Q3** (a) An experiment was performed by a Lab Technician following the equation below;

$$a H_2SO_4 + b KOH \longrightarrow c + d$$

The concentration of KOH solution used in the titration was 0.1022 M and 25.00 ml of KOH was exactly neutralized by 29.04 ml of the $H_2SO_4$. Answer the following questions.

(i) Write a balance equation. Find ***a, b, c*** and ***d.***

(2 marks)

(ii) Calculate the number of moles of KOH in 25 ml solution.

(2 marks)

(iii) Find the number of moles of $H_2SO_4$ that neutralized the KOH solution.

(2 marks)

(iv) Find the concentration (M) of the $H_2SO_4$ solution.

(2 marks)

(v) Based on the titrant and the analyte in the equation above, state the type of the titration that was performed and draw the titration curve.

(3 marks)

(b) Gravimetric analysis is one of the quantitative analysis in the Analytical Chemistry.

(i) Define gravimetric analysis.

(ii) Explain the mechanism of precipitation in gravimetric analysis briefly.

(4 marks)

(iii) A 50 ml sample of water is analysed for its calcium content by precipitating and collecting calcium oxalate ($CaC_2O_4$). The precipitate of $CaC_2O_4$ is slowly heated to 900°C. On heating, the oxalate initially loses water to form anhydrous calcium oxalate. As the temperature continues to be raised, the calcium oxalate decomposes to form calcium carbonate ($CaCO_3$) and carbon monoxide (CO). As the temperature is raised still further the calcium carbonate decomposes to finally leave calcium oxide (CaO) and carbon dioxide ($CO_2$). (One mole of $CaC_2O_4$ gives one mole of CaO). The data is collected and recorded as in **Table Q3** below;

**Table Q3**

| | |
|---|---|
| Mass of crucible before collection of precipitate | 25.7824 g |
| Mass of crucible after precipitation, heating, and cooling | 25.9625 g |

Calculate the concentration of Ca in the water sample in part per million (ppm). (Ca = 40.078 g/mol, O = 16.000 g/mol)

(10 marks)

**Q4** (a) Using your own word and understanding, differentiate the following electrochemical methods based on their working-concepts and application:

(i) Electrogravimetric

(ii) Potentiometric

(iii) Voltammetry

(6 marks)

(b) Free bromine ($Br_2$) can be measured in aqueous solutions using an electroanalytical method. **Figure Q4 (b)** illustrates the bromine working electrode in use (wires and liquid junctions have been omitted for clarity). Answer the following questions based on the figure. (Note that stir bar and magnetic stirrer are present in the diagram).

(i) Write a half-cell reaction to explain reduction of bromine to bromide ion in aqueous solution ($E^\circ = 1.087$ V).

( 3 marks)

(ii) For the concentrations indicated in the figure, calculate the half-cell potential for the bromine working electrode at 25 °C.

(4 marks)

(c) For an electrochemical cell,

$$Mg(s) + 2Ag^{+}(0.028\ M) \rightarrow Mg^{2+}(0.0157\ M) + 2Ag(s)$$

(i) Write the cell notation.

(2 marks)

(ii) Write the Nernst equation at anode, $E_a$ and cathode, $E_c$.

(4 marks)

(iii) Calculate $E_{cell}$.
($E^{\circ}_{Mg^{2+}/Mg} = -2.372\,V, E^{\circ}_{Ag^{+}/Ag} = 0.7996\,V$)

(6 marks)

5 (a) An infrared spectroscopy is one of the analytical methods used to identify the unknown compounds in a sample. Explain the following;

(i) the electromagnetic radiation range for infrared

(1 marks)

(ii) the types of vibrational modes when molecules absorb infrared radiation

(4 marks)

(iii) the transmission versus absorption of infrared spectroscopy (include a graphic representation in your answer).

(3 marks)

(iv) An infrared spectroscopy was performed to identify the functional group of compound A. The empirical formula of the compound was analyzed to be $C_5H_{10}O$ with molar mass of 86 grams per mole. Based on the infrared spectrum of the compound A in **Figure 5(a)** and **Table Q5(a)**, identify the functional groups present in the compound, predict the structure and name the compound A.

(7 marks)

(b) Atomic Absorption Spectroscopy (AAS) can be used to determine the lead concentration in soil collected from the side of a road. A student prepared standard lead solutions for comparison and the absorbance of each solution was measured. A road-side soil sample was also prepared. The results are shown in **Table Q5(b)**.

**Table Q5(b)**

| Sample | Concentration (ppm) | Absorbance |
|---|---|---|
| Blank | 0.00 | 0.00 |
| Standard 1 | 1.00 | 0.17 |
| Standard 2 | 2.00 | 0.34 |
| Standard 3 | 3.00 | 0.48 |
| Standard 4 | 4.00 | 0.65 |
| Standard 5 | 5.00 | 0.83 |
| Sample | $x$ | 0.58 |

(i) Plot the data and draw the graph.
(ii) Determine the concentration of lead ($x$) in road-side soil sample from the graph drawn in (i).

(10 marks)

**Q6** (a) Explain the information obtained from a mass spectrometer of a gas chromatography that is useful in

(i) qualitative analysis
(ii) quantitative analysis

(6 marks)

(b) Define the following terms related to chromatography:

(i) mobile phase and stationary phase
(ii) liquid chromatography and gas chromatography
(iii) chromatogram

(12 marks)

(c) (i) In a gas chromatography analysis, an injection containing benzene at a concentration of 2000 µg/mL is made and results in a peak area of 100,000. Calculate the response factor for benzene.

(3 marks)

(ii) As a continuation of (i), an unknown concentration of benzene sample solution was injected and resulted in a peak area of 57,000. Calculate the amount of benzene present in the unknown sample solution.

(4 marks)

**-END OF QUESTIONS-**

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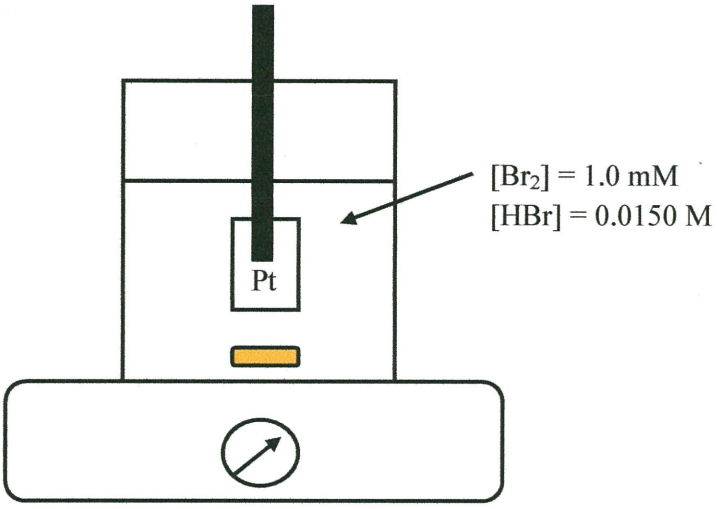


**Figure Q4(b)** : Bromine working electrode

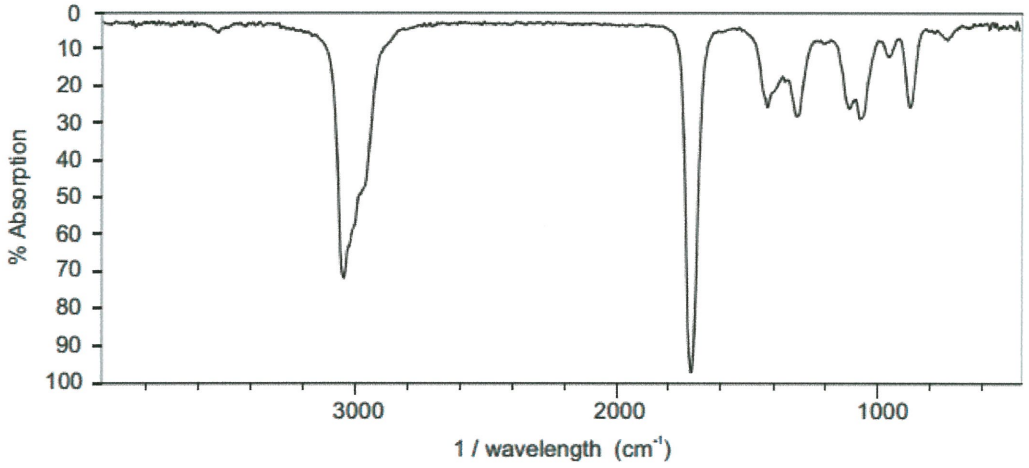


**Figure Q5(b)** : The infrared spectrum of compound A

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**Table Q5(b)** : Infrared Correlation Chart

| | Type of Vibration | | Frequency ($cm^{-1}$) | Intensity |
|---|---|---|---|---|
| C - H | Alkanes | (stretch) | 3000-2850 | s |
| | $-CH_3$ | (bend) | 1450 and 1375 | m |
| | $-CH_2-$ | (bend) | 1465 | m |
| | Alkenes | (stretch) | 3100-3000 | m |
| | | (out-of-plane bend) | 1000-650 | s |
| | Aromatics | (stretch) | 3150-3050 | s |
| | | (out-of-plane bend) | 900-690 | s |
| | Alkyne | (stretch) | ~3300 | s |
| | Aldehyde | | 2900-2800 | w |
| | | | 2800-2700 | w |
| C - C | Alkane | not interpretatively useful | | |
| C = C | Alkene | | 1680-1600 | m-w |
| | Aromatic | | 1600 and 1475 | m-w |
| C≡C | Alkyne | | 2250-2100 | m-w |
| C = O | Aldehyde | | 1740-1720 | s |
| | Ketone | | 1725-1705 | s |
| | Carboxylic Acid | | 1725-1700 | s |
| | Ester | | 1750-1730 | s |
| | Amide | | 1670-1640 | s |
| | Anhydride | | 1810 and 1760 | s |
| | Acid Chloride | | 1800 | s |
| C - O | Alcohols, Ethers, Esters, Carboxylic Acids, Anhydrides | | 1300-1000 | s |
| O - H | Alcohols, Phenols | | | |
| | Free | | 3650-3600 | m |
| | H-bonded | | 3500-3200 | m |
| | Carboxylic Acids | | 3400-2400 | m |
| N - H | Primary and Secondary Amines and Amides | | | |
| | | (stretch) | 3500-3100 | m |
| | | (bend) | 1640-1550 | m-s |
| C - N | Amines | | 1350-1000 | m-s |
| C = N | Imines and Oximes | | 1690-1640 | w-s |
| C≡N | Nitriles | | 2260-2240 | m |
| X = C = Y | Allenes, Ketenes, Isocyanates, Isothiocyanates | | 2270-1950 | m-s |
| N = O | Nitro ($R-NO_2$) | | 1550 and 1350 | s |
| S - H | Mercaptans | | 2550 | w |
| S = O | Sulfoxides | | 1050 | s |
| | Sulfones, Sulfonyl Chlorides, Sulfates, Sulfonamides | | 1375-1300 and<br>1200-1140 | s<br>s |
| C - X | Fluoride | | 1400-1000 | s |
| | Chloride | | 800-600 | s |
| | Bromide, Iodide | | <667 | s |