059

BSc Degree Programme
Level 5- Assignment Test I- 2023/2024
CYU5308- INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS

Question No.	Marks
1	
2	
Total	
%	

Duration: One hour
Date & Time: 30th December 2023
4.00 pm- 5.00 pm

Registration No.: .....

Answer all questions in the given space. Additional sheets will not be marked.

1. (A) A sample of a factory effluent was analyzed for element X using Atomic Absorption Spectrophotometer. For that, 25.0 mL of 10.0 ppm standard solution was added to 50 mL of the sample solution, and the solution was made up to 100.0 mL. The absorption was 0.600. The sample solution alone gave an absorption of 0.300 in the same conditions. The absorption was not measured at resonance wavelength since there had been a spectral interference.

(i) What do you mean by spectral interference? (10 marks)

(ii) What is the disadvantage of not measuring at resonance wavelength? (10 marks)

(iii) What is the reason for adding the standard solution to the sample solution? (10 marks)

(iv) Calculate the concentration of Zn in the sample. (20 marks)

(B) State the following in brief.

(i) Two advantages of Raman Spectroscopy compared to IR spectroscopy. (10 marks)

(ii) One major difference between Fluorescence and Phosphorescence (10 marks))

(iii) Principle of Mass Spectroscopy (what happens to the sample, what is measured, how the measurements are used) (20 marks)

(C) If you want to use Flame Atomic Emission spectrophotometer as an atomic absorption spectrophotometer, what are the additional parts that you should fix? (10 marks)

2. (A) A solution was contaminated with Cu^{2+}. To determine the concentration of Cu^{2+} using electrogravimetry, a 250.0 mL of this solution was electrolyzed with an applied overvoltage of 0.002 V. The weight of the cathode at the beginning and atter completion of the electrolysis were 201.5 mg and 392.0 mg respectively. (Cu= 65.5)

(i) What do you mean by overvoltage? (10 marks)

(ii)How do you make sure that all the Cu^{2+} in the solution is deposited on the electrode practically? (10 marks)

(iii) Calculate the concentration of Cu^{2+} in the water sample in mol L^{-1}. (12 marks)

(iv) Write down one major difference in measurement and one major difference in instrumentation of coulometry and electrogravimetry. (12 marks)

(B). A student performed a voltametric analysis to measure the concentration of Cd^{2+} in a contaminated water sample. A 10.00 mL water sample was treated with 20.00 mL of 0.5 M KCl and carried out the voltametric analysis. The diffusion current (I_d) observed was 720 mA at the half wave potential ($E_{1/2}$) 1.1 V. Similarly, he repeated the experiment using 10.00 mL of 5.00 ppm of Cd^{2+} standard solution and the diffusion current (I_d) resulted was 240 mA.

(i) What is the reason to add the 0.5 M KCl during this analysis? (10 marks)

(ii) Determine the concentration of the Cd^{2+} in the sample. (14 marks)

(iii) Sketch and label the Voltagram for the sample and indicate $E_{1/2}$ on it. (16 marks)

(C) Thermal analysis was applied to identify the cation of carbonate compound (XCO_3) which undergoes the following reaction at 600°C. Sketch fully labeled thermo- gravimetric curve for the analysis. (16 marks)

$$XCO_3(s) \rightarrow XO(s) + CO_2(g)$$

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1.(A) (i) Interference due to overlapping of maximum absorption line of the analyte and the interfering component.

(ii) Sensitivity will be low.

(iii) To minimize background interference.

(iv) $A = C\varepsilon l$

$0.300 = C_u \varepsilon l$ ⟶ (1)

$0.600 = (C_{u\,(new)} + C_s)\,\varepsilon l$ ⟶ (2)

$C_{u\,(new)} = [(C_u \times 10^{-3} \times 50) + (5 \times 10^{-3} \times 25)] \times 10^{-3}/100$

(1) / (2) $\quad \frac{0.300}{0.600} = \frac{C_u}{[(C_u \times 10^{-3} \times 50) + (10 \times 10^{-3} \times 25)] \times 10^{3}/100}$

$\frac{0.300}{0.600} = \frac{C_u}{(50C_u + 250)/100}$

$C_u = 1.67$ ppm

(B) (i) Two advantages of Raman Spectroscopy compared to IR spectroscopy.

1. Can analyse linear molecules
2. Special cells are not necessary
3. Can determine samples in aqueous solutions.

(ii) One major difference between Fluorescence and Phosphorescence (any one comparison)

1. Flu- occurs with no change of spin of electrons. Pho- - occurs with change of spin of electrons.
2. Flu- fast. Pho- slow.
3. Flu- frequent. Pho- rare.

(iii) Principle of Mass Spectroscopy (what happens to the sample, what is measured, how the measurements are used)

Kinetic energy of fast-moving particles is transferred to the sample molecule resulting in bond breaking. The mass and the relative abundance of the resultant fragments are measured and given as a spectrum. The peaks give information to identify the sample molecule (qualitative analysis/structure determination)

(C) Parts you should fix: HCL and chopper

2. (A) (i) Overvoltage = Difference between thersitical and actual voltage required to carry out the electrolysis.

(ii) Do the experiment until the weight is a constant.

(iii) The concentration of Cu^{2+} in the water sample in mol L^{-1}

The amount of Cu deposited = $392.0 - 201.5\ mg = 190.5\ mg$

The moles of Cu2+ = $\frac{190.5\ mg}{65.5\ g\ mol^{-1}} = 3\ mmol$

Concentration of solution = $\frac{3\ mmol}{250.0\ mL} \times 1000\ mL$

= 12 mmol L^{-1}

(iv)

coulometry analysis	electrogravimetry
Measurement- current and time Number of electrodes- 3	Mass Number of electrodes- 2

(B) (i) The reason to add the 0.5 M KCl-To control the current formed by the migration.

(ii) Concentration of the Cd^{2+} in the sample.

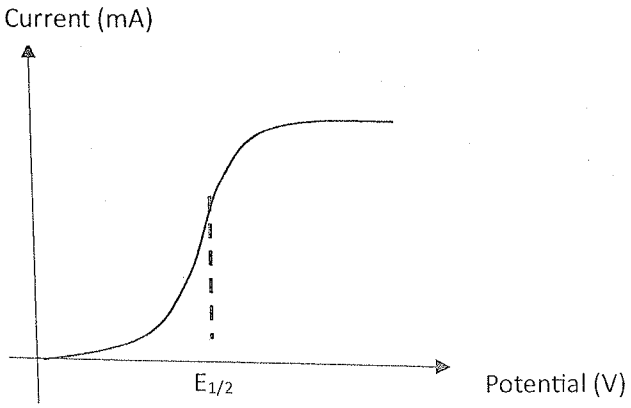
$I_d \alpha\ C$

For sample; $720\ mA = c_x$----------(1)

For 5.00 ppm standard; $240\ mA = 5.00\ ppm$----------(2)

(1)/(2) $\frac{c_x}{5.00\ ppm} = \frac{720\ mA}{240\ mA}$ $\quad c_x = 15\ ppm$

(iii)



(C)