

THE OPEN UNIVERSITY OF SRI LANKA B.Sc. Degree Programme / Stand alone courses in Chemistry Level 5 -Assignment Test 2 - 2010 / 2011

CHU 3129/CHE 5129 – INSTRUMENTAL METHODS IN CHEMICAL ANALYSIS

Reg. No	 	• • • • • • • • • •						
			- 1		-	T ₁	Question	marks
						<u>[_1</u>	number	
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Answer all questions in the spaces given. Additional sheets will not be marked.

- 1. (i) Give reasons for the following statements.
 - (a) In gas chromatography, the solvent in which the liquid sample is dissolved should not have a high retention time in the column.
 - (b) The affinity of Ca²⁺ is greater than that of Mg²⁺ for a cation exchanger column.

(c) Samples cannot be directly injected to the column in HPLC.

(ii)	Give two major differences and similarities in Thin Layer Chromatography and Paper Chromatography.
	<u>Differences.</u> 1.
	2.
. •	Similarities 1.
	2.
(iii)	Write a short account on "Eddy diffusion" in column chromatography.
(iv)	Write equations using retention time for the terms "retention factor" and "selectivity factor" in chromatography (the symbols should be defined).

(40 marks)

2.	A student was asked to determine the concentration of the metal ion M ²⁺ in an aqueous solution using Coulometry.									
	M^{2}	⁺ (aq)	+	2e	\rightarrow	M (s)	((0.39 V)		
	(i)	What method	is the p 1?	rinciple	behind	coulome	etry as a	quantitati	ve analytic	al
	(ii)			el a sche ometry.		liagram c	of the in	strumentat	ion of cons	tant
	(iii)) What	is the fi	ınction	of the re	eference	electrod	le?	•	
	(iv)	The st potenti	udent w al. Why	⁄as advi ý?	sed to u	ise a pote	ential mo	ore than th	e theoretica	ıl cell
	(v)	What is quantit	s the pri	inciple i nalytica	behind t I method	he voltar d?	netric n	nethods as	a	

(vi) Give one specific feature in the instrumentation of voltametric methods giving reasons.

3. (i) The concentration of an unknown solution of Cu²⁺(100 cm³) was determined using polarography. The maximum limiting current was 855 mA while the residual current was 5 mA. A 50 ppm Cu²⁺(100 cm³) solution gave a limiting current of 805 mA and the residual current remained the same. What is the concentration of Cu²⁺ in the sample?

(ii) The same solution was subjected to electrogravimetry. Give three factors affecting the deposit.

(iii) Assume that Cd²⁺ ions are also in the solution.

$$Cu^{2+}(aq)$$
 + 2e \rightarrow $Cu(s)$ $E^{\circ} = 0.34 \text{ V}$

$$Cd^{2+}(aq) + 2e \rightarrow Cd(s) E^{0} = -0.40 V$$

(a) Can you determine the concentration of both Cu²⁺ and Cd²⁺ using electrogravimetry? Explain your answer.

-	
	(b) Write down the major steps in the analysis of two metal ions of a mixture using electrogravimetry.
-	
(iv)	Give one major difference and one major similarity in amperometric titrations and photometric titrations.
	tradicio dia protomorro infational
	(25 marks)
•	
	Name
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THE OPEN UNIVERSITY OF SRI LANKA

B.Sc degree programme /Stand Alone courses 2010/2011 CHU 3129/CHE 5129 INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS Answer guide to Assignment test II

- istili. Ikvine verv si serristesia est reservit kantonit tradiciosi 1.(i) (a) If the solvent does not have a high retention time the solvent peaks will not interfere with the sample peaks.
 - (b) Since charges are the same, the affinity depends on the size of the hydrated ion. Smaller the ion larger will be the size of the hydrated ion thus decreasing the affinity. Ca⁺² is larger than Mg⁺². Hydrated ion of Ca⁺² is smaller than Mg⁺². Therefore, the affinity of Ca⁺² is greater than that of Mg⁺².
 - (c) Samples might contain particles which might damage the column. So the samples are passed through the guard column first. The column is run with a high pressure which should not be altered.

(ii) <u>Differences</u>

Thin layer Chromatography Paper Chromatography

er a katemata tusyorida Principle behind the separation is Principle behind separation is partition

(0)

Adsorption stubilishmall Stationary phase is silica Stationary phase is water

Similarities

Both are used in qualitative analysis

Simple and inexpensive to the state of the s A visualization technique is required

- kon tunid saha sahasah sebagaan pelasarsah pelasarsah kan sebagai kata sebagai kan kenderan pelasarsah sebagai (iii) Eddy diffusion (A) is the difference in the rate of diffusion of the mobile phase resulted due to molecules having different pathways in the stationary phase. Eddy diffusion a Diameter of particles Eddy diffusion depends on the uniformity of packing of the stationary phase and the column geometry. When eddy diffusion increases height of the theoretical plates (H) also increases decreasing the efficiency of separation. Therefore, to reduce eddy diffusion (A), particles should be small.
- tor $(K'_A) = \frac{t_A t_M}{t_M}$ Selectivity factor $(\alpha) = \frac{t_B t_M}{t_A t_M}$ $t_A = \text{Retention time of } A$ Retention factor $(K'_A) = \frac{t_A - t_M}{t_M}$ (iv)

 t_B = Retention time of B

 t_M = Retention time of mobile phase

- 2. (i) The total charge \alpha amount of analyte reacted or produced
 - Unit II Page 34 Figure 11.2 (ii)
 - To keep the potential of the working electrode a constant. (iii)
 - To overcome the internal resistance and electrode polarization. (iv)

(v)	Diffusion current a amount of analy	
(vi)	The working electrode has a very small solution (Micro electrode)	all surface area exposed to the
	Reason: 13 1201 140 140 140 140 140 140 140 140 140 1	11 min 1 0 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
son Win ska	The current flowing through the electronallyte concentration will not change	rode is very small. Therefore, the
		one dimensions and ordinate and a
3. hader(i) i ad	ip=ip-ig no alvacion diriffs act	
deercasing far.	$i_{D1} = 855 - 5 = 850 \text{ mA}$ $i_{D2} = 805 - 5 = 800 \text{ mA}$	
	The sufficient of the state of	法的。in citation and appropries
all od m	aulos sai <mark>n jek C</mark> o lagina dishas kaisasa	The state of the s
ille siliyen	vectorial $850 = k C$ it contains the experience	thy figure in the Conserve of the tited operation (i) is a first of the conserve of the conser
	800 = k 50 C/ 50 = 850 / 800	
	C = 53.125 ppm	
	ringromatri Liviri	yangganakak tidak kanggapan
(ii)	Current density	Allangue est unidos sieposiali.
	Stirring Temperature	
	Addion of complexing agents	Legitic et anotique attaches et l
(iii)	Can. Difference of the reduction potential	s should be greater than 0.2 V
	PC基础管理机构设置管理图 15 15 15 15 15 15 15 15 15	的复数的 人名法罗德尔克特 医多种
(iv)	Keep the cathode potential constant	at value needed to reduce one metal
	lon; dry and weigh the electrode and	er deposition; replace the cathode and needed to reduce the other metal ion
dende plate	Dry and weigh again. Take the diffe	rence in weight for the calculations.
plante e	ing different callways fallheistidina	right bisharahga pit dela badhiri (1917). A sa Galarah darin a saka dina kana dari
(v) - 12482-08517/1-27-0	<u>Differences</u> Amperometric titration	Photometric Titration
lasitevost	ladite while the exercise and exit it. The	Measurement is absorbance
of preiso	Measurement is current Analyte (or related substance)	Analyte, reagent or product should
	should be oxidizing or reducing	be able to absorb light
	Similarity	1.25 = ()); purph refirmint († 181 2
	Both are accurate, reproducible and	sensitive.
		politika (j. 1914.)
	tions of enobile places in the second	用好班的 提供 新原本
	to beginning in bottomer stellars for the	
	$(x_1, x_2, x_3, x_4, x_4, x_5, x_5, x_5, x_5, x_5, x_5, x_5, x_5$	注入的原理工程 医原性狂狂的性炎 人名英
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