DEPARTMENT OF CHEMISTRY UNIVERSITY OF ESWATINI

C404/CHE411 ADVANCED ANALYTICAL CHEMISTRY II

MAY 2019 FINAL EXAMINATION

Time Allowed:

Three (3) Hours

Instructions:

- 1. This examination has six (6) questions and one data sheet. The total number of pages is four (4) including this page.
- 2. Answer any four (4) questions fully; diagrams should be clear, large and properly labelled. Marks will be deducted for improper units and lack of procedural steps in calculations.
- 3. Each question is worth 25 marks.

Special Requirements

- 1. Data sheet
- 2. Graph paper

YOU ARE NOT SUPPOSED TO OPEN THIS PAPER UNTIL PERMISSION TO DO SO HAS BEEN GIVEN BY THE CHIEF INVIGILATOR.

a) i) With the aid of a diagram, use the ion exchange theory to explain how a pH glass membrane electrode works.	[5]
ii) Explain how a pH glass electrode can be used to measure Na ⁺ ions	[3]
iii) Write the Nernst expression for an ideal pH glass electrode, and show that unit calibrations in the readout a increments of 59 mV.	are in [5]
b) The data below was obtained when a ion-selective electrode was immersed in a series of standard solutio fluoride buffered in TISAB.	ns of
$ \begin{array}{c cccc} & & & & & & & & & & & & & & \\ \hline & & & & & & & & & & & & & & \\ \hline & & & & & & & & & & & & \\ \hline & & & & & & & & & & & \\ \hline & & & & & & & & & & & \\ \hline & & & & & & & & & & \\ \hline & & & & & & & & & \\ \hline & & & & & & & & & \\ \hline & & & & & & & & & \\ \hline & & & & & & & & & \\ \hline & & & & & & & & & \\ \hline & & & & & & & & & \\ \hline & & & & & & & & & \\ \hline & & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & & \\ \hline & & & & & & & \\ \hline & & & & & \\ \hline & & & & & \\ \hline & & & & & $	
0.98×10^{-4} -48.4 0.96×10^{-3} -18.7	
1.11×10^{-2} -10.0	
1.05×10^{-1} +37.7	[6]
i) Describe each component of TISAB and explain its function in this analysis	
ii) What is the concentration of F in the sample if it gave a reading of -50.3mV	[6]
QUESTION 2 [25]	
a) For the UO ₂ ²⁺ /U ⁴⁺ system in acid,	
i) Write down the balanced redox half cell reaction.	[4]
ii) Write down the Nernst expression.	[3]
iii) Calculate the concentration of U ⁴⁺ at pH=3 if the potential measured for a 0.2351M UO ₂ ²⁺ solution is 0.	.562V. [6]
b) Use equations to describe the anodic and cathodic reactions taking place during electrodeposition measurement of copper in an unknown solution.	in the [6]
c) A dilute solution of mixture of reduced and oxidized forms of Vanadium is subjected to controlled portion electrolysis. At -0.255 V vs SHE, 90.52 coulombs are required to complete electrolysis of the oxidised form. The potential is then shifted to -0.325V vs SHE, where controlled potential electrolysis requires 144.8 could calculate the concentration of V ³⁺ and V ²⁺ in the original 100-mL solution.	III V .
QUESTION 3 [25]	
a) What is the role of a reference electrode in potentiometry?	[1]
b) Describe and state the Nernst response equations for the following:	
 i) Electrode of the First Kind ii) Electrode of the Second Kind iii) Electrode of the Third Kind 	[2] [3] [3]
c) Discuss each of the two (2) main requirements of reference electrodes in potentiometry.	[4]
d) i) With the aid of a diagram, explain how a saturated calomel electrode is fabricated, and explain the role component in the electrode.	of each
ii) Write down its half cell reaction and Nernst expression.	[3]

		2
	iii) State its standard electrode potential and typical input impendance.	[2]
	iv) State two advantages, and two disadvantages of using this reference electrode over the other commonly ones	used [4]
Q	UESTION 4 [25]	
a)	Describe the term "overpotential" in relation to the polarography technique, and explain why overpotent desirable in this electroanalytical technique.	ial is [4]
b)	Voltammetry in the upper right quadrant can be complicated by the presence of dissolved oxygen in solution.	
	i) Use chemical equations to explain the origin of oxygen waves.	[4]
	ii) Describe two (2) ways by which oxygen waves eliminated in voltammetry?	[2]
c)	i) Use diagrams to explain the origins of "non-faradaic" current in polarography.	[2]
	ii) Use a diagram to illustrate the dependence of "non-faradaic" current on time during the lifetime of a me drop in polarography.	rcury [2]
	iii) Use a diagram to illustrate the dependence of "faradaic" current on time during the lifetime of a mercury in polarography.	drop [2]
	iv) Use a diagram to illustrate the effect of concentration on "non-faradaic" current during the lifetime mercury drop in polarography.	of a [2]
d)	Sometimes useful information can be derived from the rising portion of the polarographic wave, for example number of electrons involved in the reduction. For benzoquinone, the following data were obtained in the portion of a polarographic wave:	e, the rising
	$E \text{ vs SCE (V)} I (\mu A)$	
	+0.210 +0.190 +0.170 +0.150 0.591 0.146 4.646 6.299	
	i) Calculate the value of n if $i_{d,max} = 7.008 \mu A$. ii) What is E°?	[6] [1]
Ω	UESTION 5 [25]	

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a) (i) What is the origin of polarographic maxima in voltammetry?

[2]

(ii) How are polarographic maxima eliminated in polarography?

[2]

- b) For each of the following techniques, indicate, on a voltage-time plot, when sampling of the signal is carried out. Draw the shape of the resultant voltammogram, and indicate the typical resolution (in Volts) and detection limit (in mol/L).
 - i) Tast polarography.

[4]

ii) Differential pulse polarography.

[4]

c) i) Draw a schematic diagram of the apparatus used in Anodic Stripping Voltametry (ASV).

[3]

- ii) Assume that ASV is being carried out on an environmental sample containing the toxic element cadmium. Use equations to describe the chemical processes taking place at each of the four steps involved in the ASV of the sample.
- iii) Explain why ASV is considered superior over most analytical techniques in terms of detection limits. [2]
- iv) Suppose tap water is scanned in a polarographic cell without deaeration, and the following is observed: $E_{1/2} = -0.05 \text{ V}$; $i_{d, \text{ ave}} = 2.54 \,\mu\text{A}$, when the rate of flow of mercury is 4.00 mg/sec and the drop interval is 3 sec. Calculate the concentration of oxygen in the tap water in the ppm units (diffusion coefficient $-2.12 \times 10^{-5} \, \text{cm}^2/\text{sec}$.

QUESTION 6 [25]

- a) A solution of 0.5M Ag^+ in $1M H^+$, resistance 0.5 Ω , is to be electrodeposited to 99.9999% completion with 4A in an open cell (partial pressure of O_2 in air = 0.2 atm). In the equation $E_{app} = E_{cathode} + IR + \omega$ used to ascertain the potential at which electrodeposition will occur:
 - i) Calculate E_{cathode}.
 ii) Calculate E_{anode}.
 iii) Calculate the IR drop.
 iv) Describe the term ω, and explain its origins in electrogravimetry using suitable equations.
- b) Explain the origins of electromotive efficiency in pH glass membrane electrodes. [2]
- c) Consider a biamperometric titration in which Fe²⁺ is titrated with Ce⁴⁺ according to the reaction:

 Fe²⁺ + Ce⁴⁺ Fe³⁺ + Ce³⁺ Given that the Fe³⁺/Fe²⁺ couple gets reduced at more negative potential than the Ce³⁺/Ce⁴⁺ couple,
 - i) Sketch the current-potential curves for point at which the fraction titrated is 0.1, 0.5, 1.0 and 1.2, assuming an impressed voltage of 100 mV across the electrodes. [8]
 - ii) Sketch the biamperometric titration curve for this system. [2]

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*Lanthanide Series **Actinide Series	11 137.33 Ba 56 226.03 Rn 88	8 40.078 Ca 20 8 87.62 Sr 38	0 24.305 Mg 12	9.012 Be		
cs ics	3 138.91 *La 57 3 (227) **Ac 89	\$ 44.956 Sc 21 88.906 Y 39				ω Land
140.12 Cc 58 232.04 T11 90	178.49 Fif 72 (261) Rt 104	47.88 Ti 22 91.224 Zr 40			- T	IVR 4
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permittivity		೬₃ = 1/ <i>ε</i> ²ಬ್ಗ					•
Bohr magneton $\mu_{3}=e\hbar/2m$, 9.27402×10^{-24} J $^{-1}$ Nuclear $\mu_{N}=e\hbar/2m$, 5.05079×10^{-27} J $^{-1}$ Electron g g , 2.00232 . Sonr radius $a_{2}=4\pi\epsilon_{2}\hbar^{2}/m$, 5.29177×10^{-11} m Rydberg $R_{2}=m_{1}\epsilon^{2}/8h^{3}c$ 1.09737×10^{5} cm $^{-1}$ Fine structure $c=\mu_{0}\epsilon^{2}c/2h$ 7.29735×10^{-3} Crevitational G $5.672.59\times10^{-11}$ N m 2 kg $^{-2}$ Standard g $9.806.65.m$ s $^{-2}$ of free fall; g	permittivity			-			
Nuclear $\mu_{\rm M}=e \hat{n}/2m_{\rm p}$ 5.050 $79 \times 10^{-27} {\rm J}^{-1}$ Electron g value g 2.002 g 2. Soin radius $e_{\rm p}=4\pi \epsilon_{\rm p} \hat{n}^2/m_{\rm p} t$ 5.291 $77 \times 10^{-17} {\rm m}$ Rycberg $R_{\rm m}=m_{\rm p}e^4/8h^3c$ 1.097 $37 \times 10^5 {\rm cm}^{-1}$ Fine structure $c=\mu_0e^2c/2h$ 7.297 35×10^{-2} Crevitational g 5.672 $59 \times 10^{-11} {\rm N} {\rm m}^2 {\rm kg}^{-2}$ Standard g 9.806 $65 {\rm m} {\rm s}^{-2}$ Standard g 9.806 $65 {\rm m} {\rm s}^{-2}$ of free fall g 9.806 g 3. I	Bobe magnetor						
Electron g g , g		•				•	
value 2.00232 . Sohr radius $a_0 = 4\pi\epsilon_0 \hbar^2/m_e t$ 5.291 77 × 10 ⁻¹¹ m. Rydberg $R_e = m_e t/8 \hbar^2 c$ 1.097 37 × 10 ⁵ cm ⁻¹ Fine structure $c = \mu_0 e^2 c/2 \hbar$ 7.297 35 × 10 ⁻² Crevitational G 6.672 59 × 10 ⁻¹¹ N m ² kg ⁻² Standard G 9.806.65 m s ⁻² acceleration of free fall; G		$\mu_{\rm H}={\rm e}\pi/2m_{\rm p}$	5.05079×	10-11	1		
Sonr radius $a_2 = 4\pi\epsilon_2 \hat{n}^2/m_e t$ 5.291 77 × 10 ⁻¹¹ m Hydberg $R_e = m_e \epsilon^4/8h^3 c$ 1.097 37 × 10 ⁵ cm ⁻¹ Fine structure $c = \mu_0 \epsilon^2 c/2h$ 7.297 35 × 10 ⁻¹ Gravitational G 6.572 59 × 10 ⁻¹¹ N m ² kg ⁻² Standard G 9.806.65 m s ⁻² acceleration of free fall? I Exact (defined) values femto pico nano micro milli centi deci kilo mega giga	Electron g	g.,	-2 003 22			-	÷
Rydberg $R_{-} = m_{+}e^{4}/8h^{3}c$ $1.09737 \times 10^{-11} \text{m}$ constant $C = \mu_{0}e^{2}c/2h$ 7.29735×10^{-2} Constant $C = \mu_{0}e^{2}c/2h$ 7.29735×10^{-2} Constant $C = \mu_{0}e^{2}c/2h$ 7.29735×10^{-2} $C = \mu_{0}e^{2}c/2h$ 7.29735×10^{-2} $C = \mu_{0}e^{2}c/2h$ $C =$		area commentes	<u></u>			•	`. `
constant $R_{\star} = m_{\star}e^{4}/8h^{3}c$ $1.09737 \times 10^{5} cm^{-1}$ Fine structure constant $c = \mu_{0}e^{2}c/2h$ 7.29735×10^{-2} Gravitational Gravitational Goostant Standard g		$a_0 = 4\pi \epsilon_0 \hat{n}^2/m_e \epsilon$	5.291 77 x	:10 ⁻¹¹ m		_	
Fine structure $c = \mu_0 e^2 c/2h$ $7.297.35 \times 10^{-2}$ Constant $6.672.59 \times 10^{-11} \text{N m}^2 \text{kg}^{-2}$ Constant $9.806.65 \text{m s}^{-2}$ Standard $9.806.65 \text{m s}^{-2}$ of free fall; $1 = \frac{1}{2} \text{n} \mu \text{m} \text{c} \text{d} \text{k} \text{M} \text{G}$ Prefixes femto pico nano micro milli centi deci kilo mega giga		$R_{-} = m_{1} e^{4}/8h^{3}c$					
Constant Gravitational Gravitational Gostant Standard Standard guestion guestion guestion of free fall† function pico nano micro milli canti deci kilo mega giga			- ,-				
Standard Standard Standard G.87259 x 10 ⁻¹¹ N m ² kg ⁻² Standard Standard G.80665 m s ⁻² coceleration of free fall† f	constant	$c = \mu_0 \epsilon^* c/2h$	7.297 35 ×	.10-3			
scoeleration of free fall† f		G	6.672 59 x	10 ⁻¹¹ N	m² kg-²		
of free fall† f	Standard i	g	Q gne c-	_~7	`	-	- .
f p n μ m c d k M G Prefixes femto pico nano micro milli centi deci kilo mega giga	acceleration		<u></u>	·			
f μ m c d k M G Prefixes femto pico nano micro milli centi deci kilo mega giga	or treplatti					. t Exact (defined)	Volume
femto pico nano micro milli centi deci kilo mega giga	£ . 1	•				· · · · · · · · · · · · · · · · · · ·	-aidez -
femto pico nano micro milli centi deci kilo mega giga	11	μ in	c q	k	M G	Profive	
10 ⁻¹⁵ 10 ⁻¹² 10 ⁻⁵ 10 ⁻⁶ -0 ⁻⁷	femto pico nano	micro milli	centi i deci	kīlo -	_	richikes	
10 10 10	10 ⁻¹⁵ 10 ⁻¹² 10 ⁻⁵			-			
•					in. 16,		

APPENDIX C. POTENTIALS OF SELECTED HALF-REACTIONS AT 25 °C

A summary of exidation/reduction half-reactions attained in order of decreasing exidation strength and useful for selecting reagent systems.

	and property of the state of	
Half-reaction		E'.(Y)
F2(g) + 2H + 2e	=/2HF	3.06;
O ₃ + 2H ⁺ + 2e ⁻		2.07
	= 2502	2.0(
	.⊨ Ag±	2.00
H ₂ O ₂ + 2H + 2e	≠ 2H ₂ O	1.77
	= $MnO_2(s) + 2H_2Q$	170
Ce(IV) + e	= Ct(IH) (in 1M HClO ₄)	. 1.61
H,10, + H+ + 2e7	$= 10^{-1} + 3H_2O$	1.6 : •
Bi_2O_4 (bismuthate) + $4H^+ + 2e^+$		1.59
BrO ₃ + 6H ⁺ + 5e ⁻	$=\frac{1}{2}Br_2 + 3H_2O$	1.52 ···
	$= Mn^{2+} + 4H_2O$	1.51
	$= Pb^{2+} + 2H_2O$	1.455
	= 2Cl7	1.36
- Cr ₂ O ₇ - + 1414 + 6e-	$= 2Cr^{3+} + 7H_1O \cdot \dots \cdot$	133
$MnO_2(s) + 4H^f + 2e^-$	$= Mn^{2+} + 2H_2O$	1.23
	= 2H ₂ O	1.229
$10\frac{1}{3} + .6H^{+} + 5e^{-}$	$=\frac{1}{2}I_2 + 3H_2O$	1.20
$Br_2(l) + 2e^{-l}$	$= 2Br^{-}$	1.065
$ICl_{2}^{-} + e^{-}$	$=\frac{1}{2}I_2 + 2CI^-$	1.06
$YO_{2}^{+} + 2H^{+} + e^{-}$	= YO2+ +~H2O	1,00
$HNO_2 + H^+ + e^-$	$= NO(g) + H_2O$	1.00
$NO_3^{-} + 3H^{+} + 2e^{-}$	$= HNO_2 + H_1O$	0.94
$2Hg^{2+} + 2e^{-}$	$= Hg_2^{2+}$	0.92
$Cu^{2+} + I^{-} + e^{-}$	= Cul(s)	0.86
Ag* + e" :	= Ag .	0.799
Hg ₂ ²⁺ + 2e ⁻	= 2Hg	0.79
Fc ^{3.+} + e ⁻	= .Fc ²⁺	0.771
$O_2(g) + 2H^+ + 2e^-$	$= H_2O_2$	0.682
2HgCl ₂ + 2e ⁻	$= Hg_2Cl_2(s) + 2CI^-$	0.63 -
$Hg_2SO_4(s) + 2e^{-s}$	$= 2Hg + SO_4^{2}$	0.615
Sb2O5 + 6H+ + 4e-	$= 2SbO^{+} + 3H_{2}O$.	0.581
$H_3AsO_4 + 2H^+ + 2e^-$	= HAsO2 + 2H2O	· 0.559
$\overline{I_3} + 2e^-$.	= 31	0_545
Cu ⁺ + e ⁻	= Cu	0.52
VO ²⁺ + 2H ⁺ + ε	$= V^{3+} + H_2O$	0.337
$F_{c}(CN)_{c}^{3} + e^{-}$	$= Fe(CN)_6^{1-}$	0.36
$Cu^{2+} + 2e^{-}$	= Cu	0.337
$UO_{2}^{2+} + 4H_{1}^{+} + 2e^{-}$	$= U^{2+} + 2H_2O$	0.334
		(continued)

		* ; " ;
APPENDIX C (continued)		
Half-reaction		E° (V)
$Hg_2CI_2(s) + 2e^-$	$= 2Hg + 2Cl^{\frac{1}{2}}$	0.2676
$BiO^{+} + 2H^{+} + 3e^{-}$	$= Bi + H_2O$. 0.32
AgCI(s)+e	. = Ag + Cl	. 0.2222
$SbO^+ + 2H^+ + 3e^-$.	$= Sb + H_2O.$	0:212
$CuCl_3^{2-} + e^-$	$= Cu + 3Cl^{-}$	0.178
$SO_{4}^{2-} + 4H^{+} + 2e^{-}$	$= SO_2(a\bar{q}) + 2H_2O$	0.17
$\operatorname{Sn}^{-r} + 2e^{-r}$	$=$ Sn^{2+}	0.15
$S + 2H^{+} + 2e^{-}$	$= H_2S(g)$	0.14
$TiO^{2+} + 2H^+ + e^-$	$= Ti^{3+} + H_1O$	0.10
S ₄ O ₆ + 2e	$= 2S_2O_3^{1-\cdots}$	0.08
$AgBr(s) + e^{-}$	= Ag + Br ⁻	0.071
$2H^{+} + 2e^{-}$	= H ₂ .	0.0000
$\cdot Pb^{z+} + 2e^{-}$	= Pb	-0.126
$Sn^{2+} + 2e^{-}$	= Sn '	-0.136
$AgI(s) + e^{-}$	= Ag + I -	-0.152
$Mo^{3+} + 3e^-$	= Mo approx	_ •
$N_1 + 5H^+ + 4e^-$	$= H_2NNH_3^+$	-0.23
$Ni^{2+} + 2e^{-}$	= Ni	-0.246
$V^{3+} + e^{-}$	= Y ²⁺	-0 ₋₂₅₅
$Co^{2+} + 2e^{-}$	= Co	-0.277
$Ag(CN)_z^- + e^-$	$= Ag + 2CN^-$	-0.31
$Cd^{2+} + 2e^{-}$	= Cđ	-0.403
$Cr^{3+} + e^{-}$	= Cr ² *	-0.41
$Fe^{2+} + 2e^{-}$	= Fe	-0.440
$2CO_2 + 2H^+ + 2e^-$	$= H_2C_2O_4$	-0.49
$H_3PO_3 \div 2H^+ + 2e^-$	$= HPH_2O_2 + H_2O$	-0.50
$U^{4+} + e^{-}$	$= U_{2+}$.	-0.61
$Zn^{z+} + 2e^{-}$	= Zn	-0.763
$Cr^{z+} + 2e^{-}$	= Cr	-0.91
$Mn^{2+} + 2e^{-}$	= Mn	-1.18
Zr4+ + 4e-	= Zr · · ·	-1.53
$Ti^{3+} + 3e^{-}$	= Tī .	-1.63
$A1^{3+} + 3e^{-}$	= Al .	- i.66
$Th^{4+} + 4e^{-}$	= Th	-1.90
$Mg^{2+} + 2e^{-}$	= Mg	-2.37 .
$La^{3+} + 3e^-$	= La	-2.52
$Na^{+} + e^{-}$	≕ Na_	-2.714
$Ca^{2+} + 2e^{-}$	= Ca	-287
$Sr^{2+} + 2e^-$	= Sr ·	-289
K+ + e-	= K	- 2.925
Li ⁺ +e ⁻	= Li	-3.045