UNIVERSITY OF SWAZILAND

Faculty of Health Science

Department of Environmental Health Sciences

Final Examination 2007

TITLE OF PAPER	:	INSTRUMENTAL METHODS FOR ENVIRONMENTAL ANALYSIS
COURSE CODE	:	EHS 537
DURATION	:	3 HOURS
MARKS	:	100
INSTRUCTIONS	:	READ THE QUESTIONS & INSTRUCTIONS CAREFULLY
	:	ANSWER FIVE QUESTIONS
	:	EACH QUESTION CARRIES 20 MARKS
	:	NO PAPER SHOULD BE BROUGHT INTO NOR OUT OF THE EXAMINATION ROOM
	:	BEGIN EACH QUESTION ON A SEPARATE SHEET OF PAPER

DO NOT OPEN THE QUESTION PAPER UNTIL PERMISSION HAS BEEN GRANTED BY THE INVIGILATOR.

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Question 1 (20 marks)

(a). Give five advantages of modern instrumental methods of analysis over the older classical methods. [5]

(b). Explain the term 'Calibration'. Give two examples of physical properties of the sample(analyte), that can be used for calibration and measurement of the analyte concentration. State the instrument employed in each case. [3]

© (i). Suppose that Cadbury Swaziland approaches you to help in analyzing their factory effluents for some bye products suspected to be environmental pollutants. State, in a sequential form, the steps you would take to tackle this analytical problem, and offer them a satisfactory report of your findings. [5]

(ii) Give a detailed discussion of any three of the stated steps in c(i) above. [7]

Question 2 (20 marks)

(a). Sensitivity and detection limit are two of the numerical criteria for selecting analytical methods:

(i) Explain these two terms.

(ii). Identify and distinguish between the two types of sensitivities associated with instrumental techniques.

(iii). State four other numerical criteria that can be employed for selecting an analytical method. [7]

(b). The aqueous solution of a sample, A, was analyzed using an instrumental method. The calibration data obtained are shown in the following table:

Concentration (ppm)	0.00	2.00	6.00	10.00	14.00	18.00
No. of Replications, N	25	5	5	5	5	5
Mean Analytical Signal, S	0.031	0.173	0.442	0.702	0.956	1.248
Standard Deviation, s	0.0079	0.0094	0.0084	0.0084	0.0085	0.0110

For this method, calculate:

(i) The minimum analytical signal, $\mathbf{S_m}$.

(ii) The calibration sensitivity, **m** and

(iii) The detection limit, $\mathbf{c_m}$. [13]

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Question 3 (20 marks)

(a) (i) State Beer's law. [1]

(ii). Define all the parameters contained in the Beer's law and give their corresponding S.I Units. [4]

(iii). What assumptions were made in deriving this law? [3]

(iv). Demonstrate the applicability of this law to a multicomponent system. What specific condition must be fulfilled for its applicability in this respect? [3]

(b). A 2.63 x 10^{-5} M solution containing tris(1,10-phenanthroline) iron(II) was analyzed at 508nm, using a 2.00-cm cell.

- (i) What is the absorbance of the solution at 508nm?
- (ii) Estimate the percent transmittance (%T), of the solution at 508nm.
- (iii) What should be the absorbance and the corresponding %T of this solution if a 5.00-cm cell is now used? [9]

Question 4 (20 marks)

(a) What is a spectrophotometer? State four of its basic components and their functions respectively. [7]

(b) What is the requirement for a cell material before it can be used for a particular spectral region? [1]

© Give two examples of materials generally used for the following regions:
(i) UV & Visible; (ii) IR [2]

(d) In respect of 'A Mull' and 'A KBr Pellet' used during spectroscopic analysis:

- (i) State the nature of samples for which they are used and their respective spectral regions of application.
- (ii) Briefly describe how they are prepared. [6]

(e) What are the necessary precautionary measures that need be taken when using a cell during a UV-Visible spectroscopic analysis? Why are these measures crucial? [4]

Question 5(20 marks)

(a) Define the term 'Source' with regards to atomic spectroscopic methods. Give two examples and state four of its idealized goals. [6]

(b). What are the major limitations of atomic spectroscopic methods? [2]

© For the flame atomic absorption spectrophotometer (FAAS):

- (i). What analyte property is measured and in what units? [1]
- (ii). Draw and label a schematic diagram of the 'Atomic absorption spectrophotometer' [4]
- (iii) Briefly describe its working principles. [7]

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Question 6 (20 marks)

(a) (i) Explain the term 'Chromatography' [1]

(ii) Give a summary of what happens generally during chromatographic analysis. [5]

(b) What is a chromatogram and how is it employed for both qualitative and quantitative analysis of a sample? [5]

© Define the capacity factor, k of a solute in terms of the retention time, t_r and the dead time, t_m. [1]

(d) What is column efficiency? State the factors that influence it. [4]

(e) Calculate the width at half the height of the peak, ($W_{1/2}$), for a solute whose retention time is 213s on a column with 4.5×10^3 theoretical plates. Determine the expected extrapolated base width of the peak, W. [6]

Question 7 (20 marks)

(a) For the 'Gas Liquid Chromatograph (GC):

(i) Draw and label its schematic diagram. [4]

(ii) What is the basic requirement for a mobile phase material? Give four commonly employed examples. [3]

(iii) Give two structural differences between a packed column and an open tubular column. Supply three advantages of the latter over the former. [5]

(iv) State the function and four ideal properties of the stationary phase (the liquid phase). [5]

(b) Mention the various aspects of the society where chromatographic methods have been employed as analytical tools. Give one industry in Swaziland where this method is being routinely used. [3]

General data and fundamental constants

Quantity	Symbol	Value
Speed of light†	c	$2.997\,924\,58 \times 10^{8}$ m s^{-1}
Elementary charge	e	$1.602\,177 \times 10^{-19}$ C
Faraday constant	$F = eN_A$	9.6485×10^{4} C mol^{-1}
Boltzmann constant	k	$1.380\,66 \times 10^{-23}$ J K^{-1}
Gas constant	$R = kN_A$	$8.314\,51$ J K^{-1} mol^{-1}
		$8.205\,78 \times 10^{-2}$ dm^3 atm K^{-1} mol^{-1}
		62.364 L Torr K^{-1} mol^{-1}
Planck constant	h	$6.626\,08 \times 10^{-34}$ J s
	$\hbar = h/2\pi$	$1.054\,57 \times 10^{-34}$ J s
Avogadro constant	N_A	$6.022\,14 \times 10^{23}$ mol^{-1}
Atomic mass unit	u	$1.660\,54 \times 10^{-27}$ kg
Mass of		
electron	m_e	$9.109\,39 \times 10^{-31}$ kg
proton	m_p	$1.672\,62 \times 10^{-27}$ kg
neutron	m_n	$1.674\,93 \times 10^{-27}$ kg
Vacuum permeability†	μ_0	$4\pi \times 10^{-7}$ J s^2 C^{-2} m^{-1}
		$4\pi \times 10^{-7}$ T^2 J^{-1} m^3
Vacuum permittivity	$\varepsilon_0 = 1/c^2\mu_0$	$8.854\,19 \times 10^{-12}$ J^{-1} C^2 m^{-1}
	$4\pi\varepsilon_0$	$1.112\,65 \times 10^{-10}$ J^{-1} C^2 m^{-1}
Bohr magneton	$\mu_B = e\hbar/2m_e$	$9.274\,02 \times 10^{-24}$ J T^{-1}
Nuclear magneton	$\mu_N = e\hbar/2m_p$	$5.050\,79 \times 10^{-27}$ J T^{-1}
Electron g value	g_e	$2.002\,32$
Bohr radius	$a_0 = 4\pi\varepsilon_0\hbar^2/m_e e^2$	$5.291\,77 \times 10^{-11}$ m
Rydberg constant	$R_\infty = m_e e^4/8h^3c\varepsilon_0^2$	$1.097\,37 \times 10^{5}$ cm^{-1}
Fine structure constant	$\alpha = \mu_0 e^2 c/2h$	$7.297\,35 \times 10^{-3}$
Gravitational constant	G	$6.672\,59 \times 10^{-11}$ N m^2 kg^{-2}
Standard acceleration of free fall†	g	$9.806\,65$ m s^{-2}

† Exact (defined) values

Prefixes

f	p	n	μ	m	c	d	k	M	G
femto	pico	nano	micro	milli	centi	deci	kilo	mega	giga
10^{-15}	10^{-12}	10^{-9}	10^{-6}	10^{-3}	10^{-2}	10^{-1}	10^{3}	10^{6}	10^{9}

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PERIODIC TABLE OF ELEMENTS

GROUPS

PERIODS	1 IA	2 IIA	3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8 VIIIB	9 VIIIB	10 VIIIB	11 IB	12 IIB	13 IIIA	14 IVA	15 VA	16 VIA	17 VIIA	18 VIIIA
1	1.008 H 1																	4.003 He 2
2	6.941 Li 3	9.012 Be 4											10.811 B 5	12.011 C 6	14.007 N 7	15.999 O 8	18.998 F 9	20.180 Ne 10
3	22.990 Na 11	24.305 Mg 12											26.982 Al 13	28.086 Si 14	30.974 P 15	32.06 S 16	35.453 Cl 17	39.948 Ar 18
4	39.098 K 19	40.078 Ca 20	44.956 Sc 21	47.88 Ti 22	50.942 V 23	51.996 Cr 24	54.938 Mn 25	55.847 Fe 26	58.933 Co 27	58.69 Ni 28	63.546 Cu 29	65.39 Zn 30	69.723 Ga 31	72.61 Ge 32	74.922 As 33	78.96 Se 34	79.904 Br 35	83.80 Kr 36
5	85.468 Rb 37	87.62 Sr 38	88.906 Y 39	91.224 Zr 40	92.906 Nb 41	95.94 Mo 42	98.907 Tc 43	101.07 Ru 44	102.91 Rh 45	106.42 Pd 46	107.87 Ag 47	112.41 Cd 48	114.82 In 49	118.71 Sn 50	121.75 Sb 51	127.60 Te 52	126.90 I 53	131.29 Xe 54
6	132.91 Cs 55	137.33 Ba 56	138.91 *La 57	178.49 Hf 72	180.95 Ta 73	183.85 W 74	186.21 Re 75	190.2 Os 76	192.22 Ir 77	195.08 Pt 78	196.97 Au 79	200.59 Hg 80	204.38 Tl 81	207.2 Pb 82	208.98 Bi 83	(209) Po 84	(210) At 85	(222) Rn 86
7	223 Fr 87	226.03 Ra 88	(227) **Ac 89	(261) Rf 104	(262) Ha 105	(263) Unh 106	(262) Uns 107	(265) Uno 108	(266) Une 109	(267) Uun 110								

Atomic mass → 10.811
Symbol → B
Atomic No. → 5

TRANSITION ELEMENTS

*Lanthanide Series	140.12 Ce 58	140.91 Pr 59	144.24 Nd 60	(145) Pm 61	150.36 Sm 62	151.96 Eu 63	157.25 Gd 64	158.93 Tb 65	162.50 Dy 66	164.93 Ho 67	167.26 Er 68	168.93 Tm 69	173.04 Yb 70	174.97 Lu 71
**Actinide Series	232.04 Th 90	231.04 Pa 91	238.03 U 92	237.05 Np 93	(244) Pu 94	(243) Am 95	(247) Cm 96	(247) Bk 97	(251) Cf 98	(252) Es 99	(257) Fm 100	(258) Md 101	(259) No 102	(260) Lr 103

( ) indicates the mass number of the isotope with the longest half-life.