UNIVERSITY OF SWAZILAND
Faculty of Health Sciences

DEGREE IN ENVIRONMENTAL HEALTH SCIENCES
FINAL EXAMINATION PAPER 2005

TITLE OF PAPER : INSTRUMENTAL METHODS FOR ENVIRONMENTAL ANALYSES

COURSE CODE : EHS 537

DURATION : 3 HOURS

MARKS : 100

INSTRUCTIONS : READ THE QUESTIONS & INSTRUCTIONS CAREFULLY

: ANSWER ONLY **FIVE** QUESTIONS

: EACH QUESTION CARRIES 20 MARKS.

: WRITE NEATLY & CLEARLY

: NO PAPER SHOULD BE BROUGHT INTO OR OUT OF THE EXAMINATION ROOM.

: BEGIN EACH QUESTION ON A SEPARATE SHEET OF PAPER.

: APERIODIC TABLE AND OTHER USEFUL DATA HAVE BEEN PROVIDED WITH THIS PAPER

DO NOT OPEN THIS QUESTION PAPER UNTIL PERMISSION IS GRANTE BY THE INVIGILATOR.

Question 1 (20 marks)

a) Differentiate between the following terms for an analytical instrument
   i) sensitivity and detection limit
   ii) calibration sensitivity, **m** and analytical sensitivity, γ.
   iii) precision and accuracy. (6)

b) Explain the term 'Applicable concentration Range' of an analytical method. Illustrate this concept with a labeled diagram and define all the parameters involved. (6)

c) Why is it essential to pre-treat an environmental sample prior to final analysis? (6)

d) The Swazi Paper Mills has approached you to assist in analyzing their factory effluent for the presence and levels of some known environmental pollutants. **State** sequentially the steps you would take in handling this problem. (5)

Question 2 (20 marks)

a) For a beam of electromagnetic radiation passing through an absorbing medium, which of the following of the radiation beam's parameters, 'wavelength' or 'frequency' is influenced by the medium's density? Illustrate your answer with a labeled diagram. (3)

b) State Beer's Law. Give its mathematical expression and state the S.I. Units of all the terms involved. (5)

c) For a species that absorbs radiation in accordance to Beer's Law, show graphically the variation patterns of its concentration with its absorbance (A), transmittance, T and $Log_{10}T$. (5)

d) A solution that absorbs radiation in accordance with Beer's Law has a molar absorptivity of 750 $M^{-1}cm^{-1}$ and an absorbance reading of 0.563. If the absorbance measurement is made using a 1.00 cm cell, calculate:
   i) The concentration of the solution
   ii) The %T (% transmittance). (7)

Question 3 (20 marks)

For the Electrothermal Atomic Absorption Spectrophotometer, EAAS, (Graphite furnace),

a) Draw and label its schematic diagram. (3)

b) Give three of its advantages and two if its disadvantages over the flame AAS. (5)

c) Discuss the stages involved in the atomization process of a sample. (8)

d) Account for the uses of 'the following inert gas' and 'the Matrix modifier' during analysis involving the use of this method. (4)

Question 4 (20 marks)

a) For the following spectrophotometric methods:
FAAS, FAES, AFS, ICP, ICP-AFS
i) Classify them into two groups of 'emission' or 'absorption' method.
ii) State the quantity measured in each case. (5)

b) Using the 'Spectronic 20' as a typical example of a single beam spectrophotometer.
i) Draw and label the schematic diagram of its optical train. (6)
ii) State the material used for its source of radiation, the wavelength dispersing medium, and the detector (3)
iii) Why is it referred to as 'a single beam instrument'? (1)

c) Give a brief description of the working principles of the FAES (flame atomic emission) spectrometry. (5)

Question 5 (20 marks)

a) Give at least one difference in the instrumental design for the following atomic spectroscopic methods of analysis.
i) AAS and FES; ii) AAS and AFS; iii) AFS and FES. (4)

b) Which is more sensitive to flame temperature stability, AAS or FES and why? (3)

c) Give five advantages of ICP (Inductively Coupled Plasma) spectroscopic method of analysis over the other conventional spectroscopic methods. (5)

d) Briefly describe the working principles of ICP. (8)

Question 6 (20 marks)

a) What is solvent extraction? (!)

b) Define K_D (the distribution coefficient) and D (the distribution ratio). State any difference/s between them. (4)

c) Briefly describe the procedure for the extraction of a solute contained in a 50.0mL aqueous phase using 100.0mL carbon tetrachloride. (5)

d) Using an appropriate expression, identify the factors that influence the distribution ration (D), of an acid that is monomeric in both aqueous and organic phases and whose anion does not penetrate the organic layer. (4)

e) i) Ninety percent of a certain solute is extracted when equal volumes of aqueous and organic phases are used. What will be the percent extracted if the volume of the organic phase is doubled? (4)

ii) If, instead of doubling the volume of the organic phase, the extraction is carried out twice using the same volume of the organic phase, which of the two procedure would you prefer and why? (2)

Question 7 (20 marks)

a) Define the following chromatographic terms:
   i) Retention time, t_R
   ii) Retention volume, V_R (2)

b) Using an illustrative choromatogram, discuss how chromatographic methods can be employed for both quantitative and qualitative analysis of a sample. (4)

c) Draw and label the schematic diagram of a 'Gas Chromatograph' (GC) (4)

d) For the GC, discuss
   i) The main features of a packed column. (4)
   ii) The function and the ideal properties of the solid support for the column. (3)
   iii) The function and the ideal properties of the liquid phase for the column. (3)

Question 8 (20 marks)

a) Distinguish between 'Thin Layer Chromatography' (TLC) and 'Paper Chromatography' from the following points of view:
   i) the nature of the mobile phase.
   ii) the nature of the stationary phase
   iii) resolution and sensitivity. (6)

b) Define R_f value, with regards to qualitative analysis in planar chromatography. (2)

c) For the analysis of a polar substance using the TLC method, give a brief procedure for the:
   i) TLC plate preparation (6)
   ii) Identification of the separated components on the TLC plate. (6)

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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	$R_\infty = m_e e^4/8h^3 c\varepsilon_0^2$	$1.097\,37 \times 10^{5}$ 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}

PERIODIC TABLE OF ELEMENTS

GROUPS

PERIODS	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
	IA	IIA	IIIB	IVB	VB	VIB	VIIB		VIIIB		IB	IIB	IIIA	IVA	VA	VIA	VIIA	VIIIA
1	1.008 H 1																	4.003 He 2
2	6.941 Li 3	9.012 Be 4										Atomic mass → Symbol → Atomic No. →	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				TRANSITION ELEMENTS							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								

*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.