UNIVERSITY OF SWAZILAND
FACULTY OF HEALTH SCIENCES
SUPPLEMENTARY EXAMINATION, 2006

TITLE OF PAPR	**:**	**Instrumental Methods for Environmental Analysis**
COURSE CODE	**:**	**EHS 537**
TIME	**:**	**Three Hours**
INSTRUCTIONS	**:**	**Answer any FIVE(5) questions. Each question carries 20 marks.**

A periodic table and other useful data have been provided with this paper.

You are not supposed to open this paper until permission to do so has been granted by the Chief Invigilator

Question 1 (20 marks).

a) State the steps (in sequential form), that should be followed in solving a typical analytical problem. (6)

b) (i) Using a labeled schematic diagram, identify the basic components of a typical analytical instrument (5)

(ii) Discuss briefly the functions of any two of these components and give an example in a named instrument (5)

b) State the salient performance characteristics (selection criteria) for an analytical instrument. (5)

Question 2 (20 marks)

a) Give one type of radiation source for each of the following wavelength regions of the electromagnetic spectrum.
(i) Visible (ii) UV and (iii) IR. (3)

b) State the necessary precautions in the handling and maintenance of a sample cuvet for UV spectrophometric analysis. (4)

c) The amount of iron in an aqueous sample was determined spectrophotometrically by preparing the 1.10-phenathroline complex with a λ_{max} of 508 nm. The Fe(II) stock solution was prepared by transferring 0.702g of $Fe(NH_4)_2.(SO_4)_2.6H_20$ into 1-L volumetric flask originally containing 2.5 mL H_2SO_4, and then diluted to mark with distilled water. The working standards were prepared by transferring 1.00-, 2.00-, 5.00- and 10.00- mL portions of the stock solution to separate 100.00 mL volumetric flasks. 10.00 mL of the sample was put in another 100 mL flask, and equal amounts of hydroxyl ammonium chloride followed by phenanthroline solution were added to each flask and then diluted to mark with water. A blank was prepared in a similar way in another 100 mL volumetric flask. The following table shows the absorbance readings obtained for both standards and sample when measured in a 1cm cuvette against the blank at 508 nm.

Solution	Standard 1	Standard 2	Standard 3	Standard 4	Sample
Absorbance	0.041	0.081	0.205	0.420	0.232

(i) Calculate the amount of iron in the sample in ppm. (10)

(ii) What would be the absorbance of the sample if a 4 cm cuvette was used? (3)

Question 3 (20 marks)

a) For a spectrophotometer, list the components of its monochromator system , and give the respective functions of each named component. (6)

c) For the prism as a component of the monochromator system:

(i). State the spectral region where it works best and why.
(ii). State the factors that increase its resolution. (4)

c) (i) State the factors that increase the resolution of 'diffraction gratings' as a component of the monochromator system of a spectrophotometer.
(ii) Give example of a spectrophotometer which uses the diffraction grating monochromator.
(iii) What advantage/s does it have over the prism monochromator system? (5)

d) What monochromator prism material would you recommend for the following spectral regions:
(i) Visible. (ii) UV (iii) IR ?
Account briefly for your choice. (5)

Question 4 (20 marks)

a) What is meant by "interference" with regards to flames and furnances? (1)

b) For the 'chemical', 'ionization' and 'spectral' types of interferences.
(i) Explain their causes
(ii) Discuss the steps normally taken to correct or eliminate each of them.
(iii). Discuss the steps usually taken to correct or eliminate 'ionization interference. (12)

c) (i) Give two examples each of a 'cool flame' and a hot flame'
(ii) Offer an explanation for the following observation. "Although chemical interferences are more prevalent in 'cool' flames, yet this flame is preferred for the analysis of alkali metals" (7)

Question 5 (20 marks)

a) Detail the basic principles of solvent extraction of metals as metal chelates and give an example of a chelating agent. (5)

b) State the expression for the distribution ratio, D, of a metal between an aqueous and an organic phase during solvent extraction using a chelating agent. What conclusions can be drawn from this expression? (5)

c) A 1.0×10^{-7} M aqueous Cu (II) was being extracted into CCl_4 by 0.1mM *dithizone*. Given that $K_{DL} = 1.1 \times 10^4$; $K_{DM} = 7.0 \times 10^4$; $Ka = 3.0 \times 10^{-5}$; $K_f = 5.0 \times 10^{22}$ and n = 2. Calculate:

 (i) The distribution ratio for this extraction at pH =1.0 (5)

 (ii) The fraction of Cu(11) remaining in the aqueous phase when 100ml of the 1.0×10^{-7} M aqueous Cu(11) is extracted once with 50ml of the CCl_4 at pH = 1.0. (5)

Question 6 (20 marks)

a) In gas chromatography (GC) what is column efficiency? How is its value affected by N, the number of theoretical plates, and H, the plate height? What other factors affect it? (5)

b) What is temperature programming in GC? Use a graphical illustration to show how it affects the resolution, R_s, the retention time, t_r and the number of solutes eluted during a GC analysis. (5)

c) What are the functions and the ideal properties of the liquid stationary phase for GC analysis? (5)

d) Give four general applications of 'Gas Chromatographic analysis'. Give an example of an industry in Swaziland where this method is being used on routine basis. (5)

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.

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