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UNIVERSITY OF SWAZILAND
FIRST SEMESTER EXAMINATION 2008/2009

TITLE OF PAPER	:	**Instrumental Methods For Environmental Analysis - 1**
COURSE CODE	:	**EHS 573**
TIME ALLOWED	:	**Two (2) HOURS**
INSTRUCTIONS	:	**Answer any <u>Four</u> (4) Questions. Each Question Carries 25 Marks**

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

DO NOT OPEN THIS QUESTION PAPER UNTIL PERMISSION TO DO SO HAS BEEN GRANTED BY THE CHIEF INVIGILATOR.

Question 1 (25 marks)

(a) Discuss the significance/advantages of the modern instrumental methods of analysis in modern day environmental studies. [5]

(b) During the analysis of a given environmental sample, 'Selection of the appropriate method' and 'Sampling' are of utmost importance.

(i) Briefly discuss the major figures of merit (or performance characteristics), that would enable you to select an appropriate method for such analysis. [11]

(ii) Sate other useful factors that can be employed in the method selection process. [2]

(iii) Explain the terms 'Sampling' and 'Sample pretreatment'. [2]

(iv) Why is sample pretreatment essential here? Give four common examples. [5]

Question 2 (25 marks)

(a) With the help of a labeled schematic diagram, identify the basic components of an instrument for environmental analysis. Give one function of each of the components indentified. [7]

(b) During the analysis of a species, X, in an aqueous solution, the following calibration data were obtained

Concentration (mg/L)	No. of Replicate Readings, N	Average Analytical Signal, S	Standard Deviation, s
0.00	25	0.0371	0.0079
2.00	5	0.173	0.0094
6.00	5	0.442	0.0084
10.00	5	0.702	0.0084
14.00	5	0.956	0.0085
18.00	5	1.248	0.0110

For this method, calculate:

(i) The analytical sensitivity at each concentration

(ii) The minimum analytical signal, S_m.

(iii) The Calibration sensitivity, **m**.

(iv) The detection limit, c_m.

(v) The % r_s (percentage relative standard deviation), for each of the replicate sets of reading. [18]

(Take k = 3)

Question 3 (25 marks)

(a) Sate the mathematical expression of Beer's law and give the S.I units of all the parameters involved in it. What assumptions are made in deriving this law? [7]

(b) Explain or discuss the following:

(i) Positive and negative deviation from Beer's law. [4]

(ii) The causes and correction/minimization of real and instrumental deviations from Beer's law. [6]

(c) A 6.94×10^{-6} M solution of a complex compound contained in a 1.00-cm cell had a percent transmittance of 31.4 at a given wavelength. Calculate the following:

(i) Absorbance of the solution.

(ii) Molar absorptivity of the complex.

(iii) Absorbance of the same solution in a 5.00-cm cell.

(iv) The cell path that will give a percent transmittance of 20.0

[8]

Question 4 (25 marks)

(a)

(i) What is a spectrophotometer? [2]

(ii) Identify four of the basic components of a spectrophotometer and the corresponding function/s of each of them. [6]

(b) With regards to the monochromator system for a spectrophotometer :

(i) List the components and give the respective functions/s of each of them. [6]

(ii) What are the advantages and weaknesses of 'diffraction gratings' when compared with a 'glass prism'? [3]

(iii) Explain the term 'dispersion of a prism'. Hence briefly discuss the working principles of a prism as a monochromator. [4]

(iv) Sate the factors that enhance the resolution of a 'prism' and the 'diffraction gratings' [4]

Question 5 (25 marks)

The hollow cathode lamp is a vital primary source of radiation in atomic absorption spectrometry. Discuss:

(a) Its features as a sharp line radiation source. [3]

(b) Its structure (configuration) plus a schematic diagram of it. [7]

(c) Its working principles. [10]

(d) The composition and short comings of multielement hollow cathode lamps. [3]

(e) The essence of the cylindrical structure of the cathode tube. [2]

Question 6 (25 marks)

For the elecrothermal atomic absorption spectrophotometry (EAAS), Discuss/Describe:

(a) Its main structural (configurational) features, using a schematic diagram as support. [7]

(b) The stages involved in the atomization process. [9]

(c) Absorbance measurement and use of matrix modifiers. [3]

(d) Its advantages and weakness when compared with the flame atomic absorption spectrometry. [6]

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}

PERIODIC TABLE OF ELEMENTS

GROUPS

RIODS	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
	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
	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
	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
	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
	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.