UNIVERSITY OF SWAZILAND

Faculty of Health Science

Department of Environmental Health Science

Main Examination

Dec 2012

Title of paper: Instrumental Methods for Environmental Analysis-I

Course code: EHS 573/EHM 204

Time allowed: 2 HOURS

Marks allocation: 100 Marks

Instructions:

1) ANSWER **ANY FOUR QUESTIONS**
2) Each question is weighted 25 marks
3) Write neatly and clearly
4) 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) Identify the major advantages of the modern instrumental methods of analysis in environmental studies, when compared with the old classical methods. [5]

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

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

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

(iii) Explain what the terms 'Sampling' and 'Sample pretreatment' mean? [2]

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

Question 2 (25 marks)

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

(b) While analyzing a species X in an aqueous solution, the calibration data obtained are as follows:

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.

(Take k = 3) [18]

Question 3 (25 marks)

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

(ii) Using a schematic diagram, discuss the concept of positive and negative deviation from Beer's law. [4]

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

(b) A 6.94 x 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) For the monochromator system of 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) What are the factors that enhance the resolution of 'prisms' and 'diffraction gratings'? [4]

Question 5 (25 marks)

For the hollow cathode lamp of a flame atomic absorption spectrophotometer(FAAS), 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 of a sample. [9]

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

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

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.

General data and fundamental constants

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

† 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}