UNIVERSITY OF SWAZILAND

FACULTY OF HEALTH SCIENCES

Department of Environmental Health sciences

Supplementary examination 2012/13

Title : Instrumental methods for environmental analysis

Code : EHM 212

Time : 2 hours

Marks : 100

Instructions:

1. Answer all questions,
2. Each question weighs 25 marks,
3. Start each question on a fresh page,
4. Non-programmable scientific calculators may be used,

Additional material;

- Graph paper,
- Periodic table,

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QUESTION 1

a) Give the term that is best described by the following statements:

i) The process by which analytes move up a chromatographic plate, [1]

ii) An example of a stationary phase in TLC. [1]

iii) An example of a solid support in TLC. [1]

iv) A technique for the visualization of TLC spots. [1]

v) The distance between the centre of the spots over the average diameter of the spots is called..... [1]

vi) The ratio of the amount of the solute in the stationary phase to the amount in the mobile phase [1]

b) Define the following terms as applied in chromatography, giving the appropriate equations where necessary:

i) Retention volume, V_R, [2]

ii) Resolution, [2]

c) The following functional groups are arranged in order of increasing polarity;

$-CH=CH_2$, -X, -OR, -CHO, $-CO_2R$, $-NR_2$, $-NH_2$, -OH, $-CONR_2$, $-CO_2H$.

Briefly describe, with illustrations where possible, the procedure for the separation of an ink mixture that contains $-CH=CH_2$, –CHO and $-CO_2H$ using Thin Layer Chromatography. In your discussion show or explain the following points;

i. the solvent front,
ii. the origin,
iii. the solid support,
iv. the stationary phase.
v. the orientation of the spots after separation and the mechanism of separation,
vi. How the identity of the separated compounds is made. [10]

d) Give the important mechanisms responsible for the isolation/separation of compounds in chromatography. [5]

QUESTION 2

a) A flame photometer was used to determine the calcium concentration of a water sample. The instrument was calibrated via a standard additions method, and the responses obtained are given below:

Standard addition concentration (mg/L)	Instrument output (arbitrary units)
0	12
3	16
5	27
10	37
15	49
20	61

Assuming that no interferences are present, determine the calcium concentration within the original sample. [8]

b) The distribution coefficient, K_D, of an organic salt between hexane and water is 90. A quantity of 0.1 moles of the salt is dissolved in 100mL water. Determine the number of moles of the salt that will remain within the aqueous phase following extractions using;
   i) 100 mL [4]
   ii) Four 25 mL aliquots of hexane. [4]
   iii) Compare your results and conclude which determination is preferred and why. [2]

c) Given that at 20°C only 0.24g of an organic acid dissolves in 100 mL water, but 2.70g of the same acid dissolves in 100mL of ether, calculate the value of the partition coefficient. [3]

d) Give 4 important scenarios where TLC is used. [4]

QUESTION 3

a) Define the following terms or acronyms as applied in Gas Chromatography (GC);
   i) Chromatography
   ii) SCOT
   iii) GSC
   iv) HETP
   v) Theoretical plates
   vi) Elution [6]

b) In GC, analyte separation occurs in the columns.
   i) Explain why the column is housed in a temperature programmable oven. [2]
   ii) Explain the differences between open tubular and packed columns, with labeled illustrations where possible. [6]
   iii) Give an example of an adsorbent used as a packing in GC columns. [1]

c) Briefly outline the properties of a good mobile phase in GC, giving an example. [5]

d) An unretained solute passes through a chromatography column in 3.7min and the analyte

requires 8min. Calculate the adjusted retention time and the capacity factor for the analyte. [5]

QUESTION 4

a) The distribution ratio for palladium(II)chloride between 3M HCl and tri-n-butyl phosphate (TBP) is 2.3. How many times must 15 mL of a 5.0×10^{-3} M of $PdCl_2$ be extracted with fresh 5.00 mL portions of TBP in order to remove 99.5 % of the metal? [5]

b) Using appropriate illustrations compare normal calibration curves and standard additions methods and their use in elemental determinations.

i) Clearly explain how a normal calibration curve is obtained. [4]

ii) Clearly explain how one uses standard additions method to determine concentrations of unknown. [4]

iii) Under what conditions does the standard additions method provide more accurate analytical information than the calibration curve method? [3]

c) Describe in detail how you would separate a mixture of sugar in vegetable oil. In your discussion, include the following important points:

- which one is the analyte, and which is the aqueous or organic solvent, (1)
- what apparatus / equipment to use, (1)
- what safety precautions to ensure, (2)
- the extraction procedure, (4)
- how you would ensure that 100 % of the analyte was extracted. (1)

QUESTION 5

a) With reference to Gas Chromatography (GC), briefly discuss;

i) The main features of open and tubular columns, [6]

ii) The main advantages of open tubular columns over packed columns, [4]

iii) The functions and ideal properties of the solid support and stationary phase, [5]

iv) The important property and example of a mobile phase. [2]

b) For the ECD GC detector discuss,

i) Its function,

ii) The factors determining its choice,

iii) Its desirable properties. [4]

c) i) What is a chelating agent? [1]

ii) Write an equation for the formation of a metal chelate (complex) and identify the reactant and product. [3]

PERIODIC TABLE OF THE ELEMENTS

http://www.periodni.com

GROUP

PERIOD

PERIOD	1 IA	2 IIA	3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8	9 VIIIB	10	11 IB	12 IIB	13 IIIA	14 IVA	15 VA	16 VIA	17 VIIA	18 VIIIA
1	1 1.0079 **H** HYDROGEN																	2 4.0026 **He** HELIUM
2	3 6.941 **Li** LITHIUM	4 9.0122 **Be** BERYLLIUM											5 10.811 **B** BORON	6 12.011 **C** CARBON	7 14.007 **N** NITROGEN	8 15.999 **O** OXYGEN	9 18.998 **F** FLUORINE	10 20.180 **Ne** NEON
3	11 22.990 **Na** SODIUM	12 24.305 **Mg** MAGNESIUM											13 26.982 **Al** ALUMINIUM	14 28.086 **Si** SILICON	15 30.974 **P** PHOSPHORUS	16 32.065 **S** SULPHUR	17 35.453 **Cl** CHLORINE	18 39.948 **Ar** ARGON
4	19 39.098 **K** POTASSIUM	20 40.078 **Ca** CALCIUM	21 44.956 **Sc** SCANDIUM	22 47.867 **Ti** TITANIUM	23 50.942 **V** VANADIUM	24 51.996 **Cr** CHROMIUM	25 54.938 **Mn** MANGANESE	26 55.845 **Fe** IRON	27 58.933 **Co** COBALT	28 58.693 **Ni** NICKEL	29 63.546 **Cu** COPPER	30 65.38 **Zn** ZINC	31 69.723 **Ga** GALLIUM	32 72.64 **Ge** GERMANIUM	33 74.922 **As** ARSENIC	34 78.96 **Se** SELENIUM	35 79.904 **Br** BROMINE	36 83.798 **Kr** KRYPTON
5	37 85.468 **Rb** RUBIDIUM	38 87.62 **Sr** STRONTIUM	39 88.906 **Y** YTTRIUM	40 91.224 **Zr** ZIRCONIUM	41 92.906 **Nb** NIOBIUM	42 95.96 **Mo** MOLYBDENUM	43 (98) **Tc** TECHNETIUM	44 101.07 **Ru** RUTHENIUM	45 102.91 **Rh** RHODIUM	46 106.42 **Pd** PALLADIUM	47 107.87 **Ag** SILVER	48 112.41 **Cd** CADMIUM	49 114.82 **In** INDIUM	50 118.71 **Sn** TIN	51 121.76 **Sb** ANTIMONY	52 127.60 **Te** TELLURIUM	53 126.90 **I** IODINE	54 131.29 **Xe** XENON
6	55 132.91 **Cs** CAESIUM	56 137.33 **Ba** BARIUM	57-71 **La-Lu** Lanthanide	72 178.49 **Hf** HAFNIUM	73 180.95 **Ta** TANTALUM	74 183.84 **W** TUNGSTEN	75 186.21 **Re** RHENIUM	76 190.23 **Os** OSMIUM	77 192.22 **Ir** IRIDIUM	78 195.08 **Pt** PLATINUM	79 196.97 **Au** GOLD	80 200.59 **Hg** MERCURY	81 204.38 **Tl** THALLIUM	82 207.2 **Pb** LEAD	83 208.98 **Bi** BISMUTH	84 (209) **Po** POLONIUM	85 (210) **At** ASTATINE	86 (222) **Rn** RADON
7	87 (223) **Fr** FRANCIUM	88 (226) **Ra** RADIUM	89-103 **Ac-Lr** Actinide	104 (267) **Rf** RUTHERFORDIUM	105 (268) **Db** DUBNIUM	106 (271) **Sg** SEABORGIUM	107 (272) **Bh** BOHRIUM	108 (277) **Hs** HASSIUM	109 (276) **Mt** MEITNERIUM	110 (281) **Ds** DARMSTADTIUM	111 (280) **Rg** ROENTGENIUM	112 (285) **Cn** COPERNICIUM	113 (. . .) **Uut** UNUNTRIUM	114 (287) **Fl** FLEROVIUM	115 (. . .) **Uup** UNUNPENTIUM	116 (291) **Lv** LIVERMORIUM	117 (. . .) **Uus** UNUNSEPTIUM	118 (. . .) **Uuo** UNUNOCTIUM

RELATIVE ATOMIC MASS (1)

GROUP IUPAC — GROUP CAS

ATOMIC NUMBER — SYMBOL — ELEMENT NAME

Example: 13 IIIA — 5 10.811 **B** BORON

Metal · Semimetal · Nonmetal

Alkali metal · Alkaline earth metal · Transition metals · Lanthanide · Actinide

Chalcogens element · Halogens element · Noble gas

STANDARD STATE (25 °C; 101 kPa)

Ne - gas · Fe - solid · Hg - liquid · (. . .) - synthetic

LANTHANIDE

57 138.91 **La** LANTHANUM	58 140.12 **Ce** CERIUM	59 140.91 **Pr** PRASEODYMIUM	60 144.24 **Nd** NEODYMIUM	61 (145) **Pm** PROMETHIUM	62 150.36 **Sm** SAMARIUM	63 151.96 **Eu** EUROPIUM	64 157.25 **Gd** GADOLINIUM	65 158.93 **Tb** TERBIUM	66 162.50 **Dy** DYSPROSIUM	67 164.93 **Ho** HOLMIUM	68 167.26 **Er** ERBIUM	69 168.93 **Tm** THULIUM	70 173.05 **Yb** YTTERBIUM	71 174.97 **Lu** LUTETIUM

ACTINIDE

89 (227) **Ac** ACTINIUM	90 232.04 **Th** THORIUM	91 231.04 **Pa** PROTACTINIUM	92 238.03 **U** URANIUM	93 (237) **Np** NEPTUNIUM	94 (244) **Pu** PLUTONIUM	95 (243) **Am** AMERICIUM	96 (247) **Cm** CURIUM	97 (247) **Bk** BERKELIUM	98 (251) **Cf** CALIFORNIUM	99 (252) **Es** EINSTEINIUM	100 (257) **Fm** FERMIUM	101 (258) **Md** MENDELEVIUM	102 (259) **No** NOBELIUM	103 (262) **Lr** LAWRENCIUM

(1) Pure Appl. Chem., **81**, No. 11, 2131-2156 (2009)
Relative atomic masses are expressed with five significant figures. For elements that have no stable nuclides, the value enclosed in brackets indicates the mass number of the longest-lived isotope of the element. However three such elements (Th, Pa and U) do have a characteristic terrestrial isotopic composition, and for these an atomic weight is tabulated.