# UNIVERSITY OF SWAZILAND

## FACULTY OF HEALTH SCIENCES

### Department of Environmental Health Sciences

### Supplementary Examination 2014/15

Title : Instrumental methods for environmental analysis

Code : EHM 204

Time : 2 hours

Marks : 100

Instructions:

1. Answer any 4 questions,
2. Each question weighs 25 marks,
3. Start each question on a fresh page,
4. Diagrams and graphs should be large and clearly well labeled,
5. Non-programmable scientific calculators may be used.

Additional material;

- Periodic table,

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## QUESTION 1

A sample of imported baby milk is suspected of being contaminated with melamine – an organic compound that causes kidney failure in babies (Figure 1). As afood technologist working for the Swaziland Standards Authority (SWASA) you are tasked with verifying if the levels of melamine in the baby milk are within the legal limits.

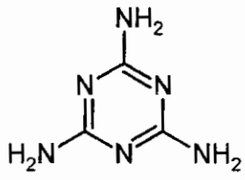


Figure 1: Chemical structure of melamine

a) State the analytical steps that need to be followed in undertaking this task. [5]
b) Looking at the structure of the compound, explain why this compound cannot be analysed using atomic absorption/emission spectroscopy. [3]
c) If UV-vis spectroscopy is the preferred method of analysis where all spectra are recorded in the liquid phase in a 1-cm cuvette,
   i. Explain how $\lambda_{max}$ (the wavelength maximum absorption) can be obtained. [4]
   ii. Give the most common material types that can be used to make cuvettes employed as sample containers in a UV spectrometer. [3]
   iii. Given that the cut-off for water is 190. Define the term 'cut-off'; hence explain why water cannot be a good solvent for the analysis. [3]
   iv. Give two (2) other properties of a good solvent for UV-vis applications. [2]
   v. The output signal from the analysis shows a transmittance of 0.743 at 540 nm. Assuming that Beer's law is obeyed, calculate the concentration of melamine ($molL^{-1}$), given that its molar absorptivity is 25 $Lmol^{-1}cm^{-1}$. [5]

## QUESTION 2

a) Explain the difference between 'real deviations' from Beer's law and those that are due from instrumental and chemical factors. [3]

b) Explain the observed differences in spectra between atomic and molecular absorptions. [3]

c) A sophisticated ultraviolet/visible/near-IR instrument has a wavelength range of 185 to 3000 nm.

   i. What are its wavenumber and frequency ranges? [4]

   ii. Hence, calculate the energy (J) of a photon emitted at this wavelength. [2]

d) A coloured solution is transferred into a 1 cm cuvette and placed in a UV-vis spectrometer. At 465 nm the sample shows an absorbance of 0.79.

   i. Calculate the percentage of light that is absorbed. [3]

   ii. Hence, determine how much light is transmitted. [2]

e) Draw a large, clearly labeled schematic diagram of a double beam UV spectrometer. [6]

f) What are the advantages of a double beam over a single beam UV spectrometer? [2]

## QUESTION 3

A pharmaceutical company is suspected of disposing effluent waste that contains the pollutant metal lead, into a river. Pharmaceutical wastes are considered difficult matrices to extract analytes from. Therefore, to determine the concentration of the metal, a series of standard solutions are made by adding 0.1, 0.2, 0.3, 0.4 and 0.5 mL of a 10 $mgL^{-1}$ lead stock solution to 100 mL aliquots of the unknown solution after sample pre-treatment. The analysis was replicated and the following results were obtained:

| Sample | Vol. of standard (mL) | Absorbance (au) | Blank absorbance |
|---|---|---|---|
| **1** | 0 | 0.28 | 0.01 |
| **2** | 0.1 | 0.38 | 0.01 |
| **3** | 0.2 | 0.55 | 0.02 |
| **4** | 0.3 | 0.67 | 0.02 |
| **5** | 0.4 | 0.76 | 0.01 |
| **6** | 05 | 0.90 | 0.02 |

a) Define the following terms as applied above:

   i. Analyte,

   ii. Aliquot,

   iii. Matrix,

   iv. Stock solution,

v. Corrected absorbance,
vi. Blank absorbance,
vii. Replicating an analysis [7]

b) Explain the phrase; 'extraction of analyte from matrix' with respect to atomic absorption spectrometry. [1]
c) Briefly explain how the lead metal can be extractedfrom soil samples into aqueous solutions prior to instrumental analysis. Justify your choice of technique. [6]
d) Explain the role of the blank absorbance in the analysis. [1]
e) Calculate the concentration of lead at each standard addition in $\mu gL^{-1}$. [5]
f) In AAS the phrase 'concentration is below detection limits' is common. Explain the meaning of this phrased. What steps would you undertake to correct this problem?[3]
g) What other figures of merit are employed to express the precision of an instrument.[2]

## QUESTION 4

For the Electrothermal atomic absorption spectrometer (ETAAS) or graphite furnace,

a) State its advantages in elemental determinations, [4]
b) Discuss the stages involved in the atomization process of a sample, [8]
c) What is the role of the argon gas? [2]
d) Draw and fully label a schematic for a hollow cathode lamp. [5]
e) Explain the operation of a photomultiplier tube detector, using a schematic diagram to illustrate. [6]

## QUESTION 5

a) What are the advantages of an ICP flame over conventional flames? [2]
b) Give three (3) types of plasma commonly employed to generate a flame in ICP-AES. [3]
c) What are the advantages of ICP-AES over the AAS technique? [5]
d) Explain the concept of nebulization in atomic absorption spectrometry using an appropriate schematic diagram. [5]
e) What is a monochromator? [2]
f) For a spectrometer, list the components of a monochromator and state the respective functions of each component given. [8]

| Group | 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 |
| Period 1 | 1 H 1.008 | | | | | | | | | | | | | | | | | 2 He 4.003 |
| 2 | 3 Li 6.94 | 4 Be 9.01 | | | | | | | | | | | 5 B 10.81 | 6 C 12.01 | 7 N 14.01 | 8 O 16.00 | 9 F 19.00 | 10 Ne 20.18 |
| 3 | 11 Na 22.99 | 12 Mg 24.31 | | | | | | | | | | | 13 Al 26.9 | 14 Si 28.09 | 15 P 30.97 | 16 S 32.06 | 17 Cl 35.45 | 18 Ar 39.95 |
| 4 | 19 K 39.10 | 20 Ca 40.08 | 21 Sc 44.96 | 22 Ti 47.90 | 23 V 50.94 | 24 Cr 52.01 | 25 Mn 54.9 | 26 Fe 55.85 | 27 Co 58.71 | 28 Ni 58.71 | 29 Cu 63.54 | 30 Zn 65.37 | 31 Ga 69.7 | 32 Ge 72.59 | 33 As 74.92 | 34 Se 78.96 | 35 Br 79.91 | 36 Kr 83.80 |
| 5 | 37 Rb 85.47 | 38 Sr 87.62 | 39 Y 88.91 | 40 Zr 91.22 | 41 Nb 91.22 | 42 Mo 95.94 | 43 Tc 98.9 | 44 Ru 101.1 | 45 Rh 102.9 | 46 Pd 106.4 | 47 Ag 107.9 | 48 Cd 112.4 | 49 In 114.8 | 50 Sn 118.7 | 51 Sb 121.8 | 52 Te 127.6 | 53 I 126.9 | 54 Xe 131.3 |
| 6 | 55 Cs 132.9 | 56 Ba 137.3 | 71 Lu 174.9 | 72 Hf 178.5 | 73 Ta 180.9 | 74 W 183.8 | 75 Re 186.2 | 76 Os 190.2 | 77 Ir 192.2 | 78 Pt 195.1 | 79 Au 196.9 | 80 Hg 200.6 | 81 Tl 204.4 | 82 Pb 207.2 | 83 Bi 208.9 | 84 Po 210 | 85 At 210 | 86 Rn 222 |
| 7 | 87 Fr 223 | 88 Ra 226.0 | 103 Lr 257 | 104 Unq | 105 Unp | 106 Unh | 107 Uns | 108 Uno | 109 Une | | | | | | | | | |

NON-METALS

METALLOIDS

METALS

| | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lanthanides | 57 La 138.9 | 58 Ce 140.1 | 59 Pr 140.9 | 60 Nd 144.2 | 61 Pm 146.9 | 62 Sm 150.9 | 63 Eu 151.3 | 64 Gd 157.3 | 65 Tb 158.9 | 66 Dy 162.5 | 67 Ho 164.9 | 68 Er 167.3 | 69 Tm 168.9 | 70 Yb 173.0 |
| Actinides | 89 Ac 227.0 | 90 Th 232.0 | 91 Pa 231.0 | 92 U 238.0 | 93 Np 237.1 | 94 Pu 239.1 | 95 Am 241.1 | 96 Cm 247.1 | 97 Bk 249.1 | 98 Cf 251.1 | 99 Es 254.1 | 100 Fm 257.1 | 101 Md 258.1 | 102 No 255 |