UNIVERSITY OF SWAZILAND

FACULTY OF HEALTH SCIENCES

Department of Environmental Health sciences

Main examination 2013/14

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;

- Graph papers (3),
- Periodic table,
- A table of scientific constants

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QUESTION 1

Caffein and aspirin are both found in many commercial analgesic preparations. Both compounds absorb in the UV-vis region and the spectra overlap to some extent. An experiment to determine the concentrations of these compounds in a tablet is performed. Using a 1 cm quartz curvette, absorbance spectra of these 2 compounds were obtained from standard solutions of 1 mmol/L and 5 mmol/L of caffein and aspirin, respectively and the UV-vis data are presented in Table 1;

Table 1: UV-vis data for caffeine and aspirin at different wavelengths

Caffeine		**Aspirin**	
Wavelength (nm)	Absorbance (au)	Wavelength (nm)	Absorbance (au)
150	0.3	150	0.4
200	0.5	200	0.5
250	0.3	250	0.6
300	0.2	300	0.3
350	0.3	350	0.4

a) Graphically illustrate the relationship between the wavelength and absorbance, determine the analytical wavelength. [6]

b) Convert the absorbance at the analytical wavelength to percentage transmittance, for each compound. [4]

The absorbances of the 2 compounds were then determined using a UV-vis spectrophotometer using a 1 cm quartz curvette. This was done by preparing standards of known concentrations and running them at the analytical wavelengths determined in (a) above. The results are shown in Table 2 for caffeine and aspirin, respectively;

Table 2: Absorbance values for the two compounds at their analytical wavelengths

Concentration of standards (nM)	**Absorbances for caffeine (au)**		**Absorbances for Aspirin (au)**	
	λ_{max} 1 (nm)	**λ_{max} 2 (nm)**	**λ_{max} 1 (nm)**	**λ_{max} 2 (nm)**
0	0.00	0.00	0.00	0.00
0.05	0.45	0.10	0.30	0.35
0.1	0.90	0.20	0.60	0.70

c) Assuming Beers law holds for this system, **graphically** determine the extinction coefficients for caffeine and aspirin at both analytical wavelengths. [8]

To determine the total absorbances of the compounds at the analytical wavelengths, the tablet was ground and dissolved in appropriate solvents and then diluted to a 200 cm^3 volumetric flask. An aliquot of 10 cm^3 was then transferred into a 1000 cm^3 volumetric flask and diluted to the mark with distilled water. This was then run using a UV-vis spectrophotometer, and the results are presented in Table 3;

Table 3: Absorbance values for the tablet at the analytical wavelengths.

Compound	**Absorbance**	
	λ_{max} 1 (nm)	λ_{max} 2 (nm)
Mixture	1.11	1.03

d) Determine the concentration (M) of caffeine, [4]

e) Determine the concentration (M) of aspirin. [3]

QUESTION 2

a) Give the mathematical expression of Beer-Lambert's law and give the SI units of all the parameters appearing therein. [3]

b) Give any five (5) assumptions made during the derivation of Beer-Lambert's law. [5]

c) A 0.11 M solution contained in a 1.00 cm cell had a percentage transmittance of 31.4 at 324.7 nm wavelength. Calculate:

i. Absorbance of the solution. [2]

ii. Molar absorptivity of the solution. [2]

iii. The cell path that will give a percentage transmittance of 20.0. [2]

iv. The energy of one photon of the radiation at the specified wavelength. [2]

An aliquot of the above solution was taken to prepare a 0.05 M solution in a 500 mL volumetric flask.

v. What is an aliquot? [1]

vi. Calculate the volume, in L, used to prepare the new concentration. [3]

vii. Will the new concentration have a higher or lower percentage transmittance than the original solution? Justify your reasoning. [2]

viii. Calculate the mass of the compound used to prepare the original solution, given that its molar mass is 180.13 $gmol^{-1}$. [3]

QUESTION 3

It is suspected that a mining operation is disposing a toxic metal (lead) into a stream in trace amounts (in the ppb range). You are working in the analytical laboratory of the Department of Water Affairs and tasked with determining the amount of the metal in the water.

a) **List**, in order, the 5 important analytical steps that you would consider when undertaking the task. [5]

Which of the following would you prefer to use and **why**?

b) Wet chemistry techniques or instrumental techniques. [2]

c) An AAS or an ICP-MS [2]

An atomic absorption spectrophotometric technique was used to determine the concentration of the lead in the water sample. Prepared working standards were determined together with the unknown sample and the results are shown below.

Concentration of standard (mg/L)	**Signal output**
0.8	0.9
0.6	0.45
0.4	0.22
0.2	0.11
0	0
Water sample	0.34

d) Determine the concentration (mg/L) of the unknown sample using the **graphical method**. [8]

e) For the light source used in AAS,

i. Give an example of a commonly used light source. [1]

ii. Draw and fully label its schematic diagram, [4]

iii. Explain its working principle in atomizing the lead atoms. [3]

QUESTION 4

For the FAAS, a mixture of combustible gases is ignited at temperature ranges from 2100 to 2800 °C.

a) Give an example of a gas mixture that can achieve these temperatures, clearly showing the oxidant and fuel. [2]

b) Give two (2) functions of the flame. [2]

c) Explain the processes that take place in the nebulizer in FAAS. [5]

d) Explain, using a fully labeled schematic diagram, the working principle of a PMT detector commonly used in FAAS. [9]

e) **State** the most significant difference between FAAS and ETAAS, hence **explain** the important processes that take place during atomization in ETAAS [7]

QUESTION 5

a) For the ICP-OES,

i. Draw a fully labeled schematic diagram of the plasma, clearly indicating the different temperature regions. [5]

ii. Hence explain the processes involved during ignition of the plasma. [8]

iii. Give any five (5) advantages of the ICP torch over other flames. [5]

b) Describe briefly the following;

i. Absorption of EMR by an atom of Na, [2]

ii. Absorption of EMR by a molecule of CO_2, [3]

iii. The differences between atomic absorption and atomic emission. [2]