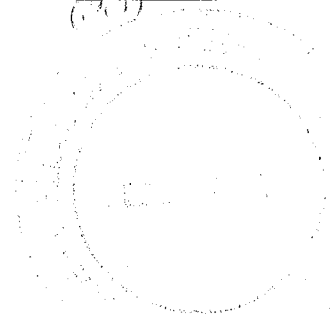




Uva Wellassa University, Sri Lanka
BTech. Science and Technology
End Semester Examination – December, 2008
CHE 311-2: Analytical Techniques and Instrumentation



Answer for **four (04) questions only**. All questions carry equal marks

Total number of questions: Five (05)

Time: Two hours

Question 1: (total 100 marks)

(a) Define **briefly** following terms. (20 marks)

- | | |
|-----------------------------------|------------------------------|
| (i). <i>X-ray fluorescence</i> | (ii). <i>Auger electrons</i> |
| (iii). <i>Satellite electrons</i> | (iv). <i>Photo-electron</i> |

(b). In each of the following cases, state which radiation is potentially the most dangerous (20 marks)

(i). X-rays of wavelengths 1.5 Å or X-rays of wavelength 20 Å

(c). What happens when a monochromatic X-ray beam is incident on the following? (20 marks)

- | | |
|---------------------------------------|------------------------------|
| 1. <i>A stationary single crystal</i> | 3. <i>A rotating crystal</i> |
| 2. <i>A crystalline powder</i> | 4. <i>Powdered glass</i> |

(d). Write down Bragg equation and define all terms. (40 marks)

In an experiment using CsCl, it was found that the first order reflection from the (100) planes was detected when the planes were inclined at 10.78° to the X-ray beam of wavelength 1.542 Å. Given that the unit cell is cubic, calculate its volume.

Question 2: (total 100 marks)

- (a). Define X-ray fluorescence yield. What is the competing process that influence fluorescence yield of an element? (20 marks)
- (b). Name the important components of a conventional wavelength dispersive spectrometer and state their functions. (40 marks)
- (c). Write an equation for the Beer's law of X-ray absorption and identify each term in it. (20 marks)
- (d). In XRF different crystals are used to differentiate X-ray wavelengths. Determine whether TOPAZ and EDDT crystals are capable of resolving $Mn K_{\alpha}$ and $Cr K_{\beta}$ lines using the following data: (20 marks)

TOPAZ	$2d$	=	2.712 Å
EDDT	$2d$	=	8.808 Å
$Mn K_{\alpha}$	λ	=	2.103 Å

Question 3: (total 100 marks)

- (a). List important interactions that occur when an electron beam strikes a sample and explain conditions under which these occur. Which of the types you have listed are of use in scanning electron microscopy? (10 marks)
- (b). This question tests your understanding about magnification by a Scanning electron microscope (SEM). A SEM enlarges the image in a unique and very simple way. Let us assume that the beam of column scans an area of 20 cm square and that the CRT screen is also 25 μm square (10 marks).
- (i). What is the magnification factor?
- (ii). Define the term resolution. How it relate to magnification?
- (c). Use the following data to determine which of the following radiation would produce X-ray fluorescence in Fe (40 marks).

$Cr K_{\alpha}$	λ	=	2.2897 Å
$Cu K_{\alpha}$	λ	=	1.5418 Å

K absorption edge of Fe = 1.743 Å

- (d). Using following data, select the metal, which would remove $Co K_{\beta_1}$ from the output of Co X-ray tube (40 marks).

$$Co K_{\beta_1} : \lambda = 1.6208 \text{ \AA}$$

K absorption edge of Ni	=	1.4880 Å
K absorption edge of Fe	=	1.7430 Å

Question 4 (100 marks)

- (a) Define atomic absorption, emission and fluorescence phenomena (20 marks).
- (b) The characteristic yellow light emission of sodium seen in flames containing sodium salt or from sodium street lamps occur at a wavelength of 589 nm. For photons of this wavelength calculate (i) frequency (ii) wave number (iii) energy in J (10 marks).
- (c) Describe the essential feature of hollow cathode lamp used in atomic absorption spectrometer (you may take copper hollow cathode lamp as an example) (20 marks)
- (d) The characteristic concentration is a useful measure of how sensitive a technique is. It is the concentration of an element which gives to 1% absorption or 99% transmission of the incidence radiation. What is the corresponding value of absorbance when $I_t = 99\%$? (10 marks)
- (e) What stands for ICPS? What is plasma? How is it generated in ICPS (explain Radio frequency ICPS torch operation) (20 marks)

Question 5 (100 marks)

- (a) List types of errors encountered in instrumental analysis? (20 marks)
- (b) Write equations to calculate precision and accuracy of a given analytical method (20 marks)
- (c) What is spike analysis? (20 marks)
- (d) Cadmium in a sample was analyzed by the method of known addition using ICP. In one experiment the following results were obtained:

<u>Sample</u>	<u>Absorbance</u>
Sample	0.078
Sample + 0.3 ppm Cd	0.140
Sample + 0.6 ppm Cd	0.205

The same sample was analyzed by AAS and following results were obtained:

<u>Sample</u>	<u>Absorbance</u>
Sample	0.090
Sample + 0.3 ppm Cd	0.160
Sample + 0.6 ppm Cd	0.240

For each of the results calculate the concentration of Cd in the sample. Is there any interference from the matrix of the sample? Explain your answers obtained under ICP and AAS measurements (40 marks)

