Uva Wellassa University, Sri Lanka
End Semester Examination – March 2011
SCT 334-2 Analytical Techniques and Instrumentation

Time: Two (02) hours

Total five (05) questions
Answer any four (04) questions.
You may use standard symbols/ abbreviations without a definition

01. a. Define following terms as appeared in instrumental analysis:
   i. Accuracy
   ii. Precision
   iii. Spike analysis

(15 marks)

b. A certain instrumental technique has a standard deviation of 1.0 %. How many replicate measurements are necessary if the standard error of the mean is to be 0.01%?

(15 marks)

c. List three error types commonly encountered in instrumental analysis? Briefly state sources of these errors. How can they be minimized/ or eliminated?

(20 marks)

d. The mercury in samples of fish taken from the Mahaweli River was determined by atomic absorption method, and following results were obtained: (in ppm) 1.80, 1.58, and 1.64.

   i. Calculate the 95% confidence limits for the mean value?
   ii. How many replicate measurements would be needed to decrease the 95 % confidence interval to ± 0.07 ppm?

(50 marks)

02. a. Define <u>briefly</u> following terms.
   i. X-ray fluorescence
   ii. Auger electrons
   iii. Satellite electrons
   iv. Photo-electron

(25 marks)

15

b. Distinguish between the roentgen, gray, and sievert.

In each of the following cases, state which radiation is potentially the most dangerous: X-rays or α particles

(25 marks)

c. Write down Bragg equation and define terms in it

(25 marks)

d. Using the following data, select the metal which would remove $CoK_1\beta_1$ from the output of a Co X-ray tube. Co $K\beta_1$: $\lambda = 1.6208$ Å

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

(25 marks)

03. a. Define X-ray fluorescence yield. What is the competing process that influen[redacted] fluorescence yield of an element?

(20 marks)

b. Name the important components of a conventional energy dispersive spectrometer and state their functions.

(20 marks)

c. Write an equation for the Beer's law of X-ray absorption and identify each term in it.

(20 marks)

d. A company sent a sample of iron ore to the laboratory for assessment. The sample was finely ground and divided into four roughly equal amounts. To three of these were added known amounts of pure Fe_2O_3, and intimately mixed. The samples were then pressed into discs under identical conditions and placed in an X-ra[redacted] spectrometer set on the first order FeKα line. The results obtained are shown below:

Sample	Mass of ore / g	Mass of added Fe_2O_3 / g	Counts /10s
1	5.0123	0.0000	9213
2	5.0231	0.5614	11998
3	4.9937	1.2504	14804
4	5.0021	2.1502	17715

i. Use the data to determine the percentage of iron in the ore. The relative atomic masses of Fe and O are 55.847 and 15.999 respectively.

ii. Would you consider this to be high or a low grade iron Fe = 55.847, O =15.999)

(40 marks)

04. a. Briefly explain the principle behind the external standard method of phase analysis by XRD data

(20 marks)

b. Mixtures of quartz and KCl were prepared. The intensity of a particular quartz line (I_o) was determined for each sample and for an unknown mixture of quartz and KCl. The data obtained are presented below:

% weight of quartz	Io, units
20	31
35	69
60	186
75	315
85	482
Unknown	129

The intensity of a sample of pure quartz (I_{PO}) was 630 units. Intensity measurements were recorded under identical conditions.

i. Prepare a calibration graph of I_o/ I_{PO} against % weight of quartz, and use it to determine the composition of the unknown.

ii. Calculate the composition of the unknown given the following mass absorption coefficients:

$(\mu_m)_{quartz}$ = 34.9 $cm^2\ g^{-1}$

$(\mu_m)_{KCl}$ = 124.0 $cm^2.\ g^{-1}$

(40 marks)

c. Would you expect to obtain normal X ray powder patterns with the following samples?

i. Ice

ii Charcoal

iii. A radio active sample

iv. $FeSO_47H_2O$

(20 marks)

d. List the essential components of powder X-ray diffractrometer. You may illustrate the answer with a block diagram.

(20 marks)

15

05. a. The characteristic yellow emissions of sodium seen in flames containing sodium salts occur at a wavelength of 589 nm. For photons of this wavelength calculate

i. The frequency

ii. The wave number

iii. The energy of one mole of sodium atoms

(25 marks)

b. Show with the aid of energy level diagrams following processes: absorption, emission and fluorescence.

i. Draw block diagrams to illustrate the working principles of AAS, AFS and AES.

ii. The characteristic concentration is a measure of how sensitive a technique is. It is the concentration of an element which gives rise to 1% absorption of the incident radiation. What is the corresponding value of absorbance (show all work).

(25 marks)

c. 5.00 cm^3 aliquots of waste water were analyzed for cadmium by standards addition method and making the resulting solution of up to a volume of 10.0 cm^3. Use the data below to determine the concentration of cadmium in the original water samples by a standards addition plot?

Added Cd / $mg.dm^{3-}$	Absorbance
0.0	0.070
0.2	0.112
0.4	0.156
0.6	0.194

(25 marks)

d. Explain the process of atomization when NaCl solution is injected into the system?

(25 marks)