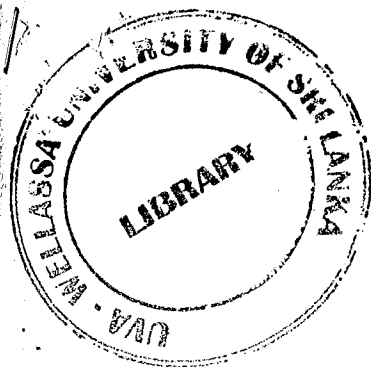




Uva Wellassa University, Sri Lanka
End Semester Examination – February 2012
SCT 334-2 Analytical Techniques and instrumentation
Time: Two (02) hours



Total five (05) questions

Answer any four (04) questions

Calculators are allowed

All symbols carry standard meanings. You may use standard symbols without definition

1.
 - i. List three types of errors commonly encountered in instrumental analysis? Briefly state what are the sources of these errors? How can they be minimized or eliminated? (25 marks)
 - ii. (a) The mercury in samples of fish taken from the Mahaweli River was determined by atomic absorption method, and following results were obtained: 1.80, 1.58 and 1.64 ppm. Calculate the 95% confidence limits for the mean value? (25 marks)

(b) How many replicate measurements would be needed to decrease the 95 % confidence interval to ± 0.07 ppm? (25 marks)
 - iii. A new procedure for the rapid determination of sulfur in kerosene was tested and sulfur was found to be in 0.123 % of the sample. The results were $S (\%) = 0.112, 0.118, 0.115$ and 0.119 . Do the data indicate the presence of bias in the method? (25 marks)

2.

i. Define following processes:

- (a) Atomic absorption (b) Atomic emission
(c) Atomic fluorescence (d) Resonance fluorescence

Layout essential components of the instruments based on these principles (25 marks)

ii. All elements give absorption lines. Why following elements cannot be analyzed by atomic absorption method:

- (a) Oxygen (b) Fluorine (c) Argon (25 marks)

iii. What is the selection rule of atomic electron transition? (25 marks)

iv. Explain the principles of following methods:

- (a) Hydride formation
(b) Cold vapor determination of Hg (25 marks)

3.

(i) Define X-ray fluorescence yield. What is the competing process that influence fluorescence yield of an element? (20 marks)

(ii) Name the important components of a conventional energy dispersive spectrometer and state their functions. (20 marks)

(iii) Write an equation for the Beer's law of X-ray absorption and identify each term in it. (20 marks)

- (iv) 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. Three of these samples 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-ray spectrometer set on the first order $\text{FeK}\alpha$ 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

- (a) 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.

- (b) Would you consider this to be high or a low grade iron?
(Fe = 55.847, O = 15.999)

(40 marks)

4.

- (i) Define following terms in chromatography:

- (a). Mobile phase (b). Retention time
(c). Selectivity factor (d). Distribution coefficient

(30 marks)

- (ii) List two assumptions made in the theoretical plate model used in chromatography.

(30 marks)



- (iii) Substances A and B were found to have retention times of 16.40 and 17.63 min. respectively on a 30.0 cm column. An unretained species passed through the column in 1.30 min. The peak widths (at base) for A and B were 1.11 and 1.21 min., respectively.

Calculate,

(a). Column resolution

(b). Average number of theoretical plates in the column

(Hint: $N = 16 \left(\frac{t_R}{W} \right)^2$)

(40 marks)

5.

- (i) State important interactions that occur when an electron beam strikes the specimen. Which of these interactions are important in Scanning Electron Microscopy (SEM)?

(25 marks)

- (ii) What is the monochromatic electron beam? How it can be generated?

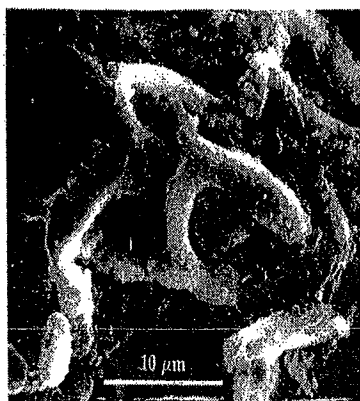
(Note: A labeled diagram of electron column is required in the answer)

(25 marks)

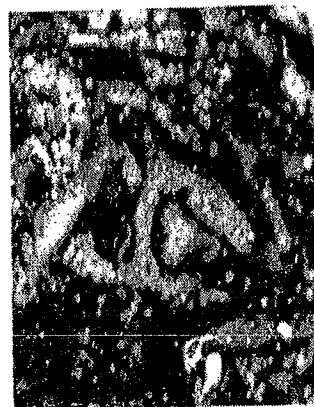
- (iii) Assume the CRT display of a SEM is 20 cm wide. What scan width would be required to achieve a magnification of 40 000?

(25 marks)

- (iv) Following fungi sample was imaged by SEM. Both images represent the same fungi.



A



B

- (a) For morphological analysis of fungi which image is recommended?
Why?
- (b) For compositional analysis of fungi which image is recommended?
Why?

(25 marks)



t- Table

Degrees of freedom (N-1)	80%	90%	95%	99%
1	3.08	6.31	12.7	63.7
2	1.89	2.92	4.30	9.92
3	1.64	2.35	3.18	5.84
4	1.53	2.13	2.78	4.60
5	1.48	2.02	2.57	4.03
6	1.44	1.94	2.45	3.71
7	1.42	1.90	2.36	3.50
8	1.40	1.86	2.31	3.36
9	1.38	1.83	2.26	3.25
19	1.33	1.73	2.10	2.88
59	1.30	1.67	2.00	2.66
∞	1.29	1.64	1.96	2.58

z- Table

Confidence level, %	z
50	0.67
68	1.0
80	1.29
90	1.64
95	1.96
96	2.00
99	2.58
99.7	3.00
99.9	3.29