



NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY
DEPARTMENT OF APPLIED CHEMISTRY
END OF FIRST SEMESTER EXAMINATIONS - DECEMBER 2004
ANALYTICAL CHEMISTRY III - SCH 4206
TIME: 3 HOURS

INSTRUCTIONS TO CANDIDATES

Answer **All** questions from Section A and **Any Three** from Section B.
Section A carries 40 marks and Section B carries 60 marks.
Total Marks - 100

SECTION A

1. The average particle diameter of an ore sample is 2.0 mm. It is estimated that the stibnite content ($d_{Sb_2S_3} = 4.5 \text{ g.cm}^{-3}$, 71.7 % Sb) is approximately 2.0%; the remainder has a density of 3.0 g.cm^{-3} and contains about 1% Sb.
 - (i) How many particles of the ore should be taken if the relative standard deviation due to sampling is to be 1% or less? [4 marks]
 - (ii) What should the weight of the gross sample be? [3 marks]
 - (iii) To what diameter must the particles be ground in order to yield a sample for analysis that weighs 0.750g and has the same number of particles as the gross sample? [3 marks]
2. (a) Derive an expression used for determining the efficiency of multiple extractions [6 marks]
- (b) A proposed method for the determination of chemical oxygen demand (COD) of wastewater was compared with the standard (mercury salt) method. The following results were obtained for a sewage effluent sample.

	Mean (mgL ⁻¹)	Standard Deviation (mgL ⁻¹)
Standard Method	72	3.31
Proposed Method	72	1.51

For each method eight determinations were made. Is the precision of the proposed method significantly greater than that of the standard method? [4 marks]

3. Describe the following membrane sample clean-up techniques

- (i) Reverse osmosis [4 marks]
- (ii) Ultrafiltration [4 marks]
- (iii) Dialysis [4 marks]

4. With the aid of well-labelled diagram explain how a flow injection apparatus could be employed in the determination of calcium in a water sample. [8 marks]

SECTION B

5. (a) Differentiate between dry ashing and wet ashing [2 marks]
- (b) What sources of error are to be anticipated when decomposing and dissolving a sample. [8 marks]
- (c) You are presented with sample containing volatile analytes (e.g As, Hg, S etc). Which methods would you choose for the digestion of such a sample? Explain the basis of your selection taking particular consideration of the advantages of the chosen methods. [10 marks]
6. It is desired to evaluate the contamination level of a river that passes close an industrial processing plant. Various elements are suspected to be present in trace amounts. Suggest a method that could be developed for such a problem, focusing on sampling, sample pre-treatment and the analytical method of choice. [20 marks]
7. (a) Sketch a flow injection system that might be employed for determining lead in the aqueous effluent from an industrial plant based upon extraction of lead ions with a carbon tetrachloride solution of dithizone, which reacts with lead ion to form an intensely colored product. [10 marks]

(b) Give a comparison of discrete analyzers and continuous-flow analyzers. What are the general advantages of automatic analysis? [10 marks]

8. A 0.150M aqueous solution of the weak organic acid HA was prepared from the pure compound, and three 50.0 mL aliquots were transferred to 100 mL volumetric flasks. Solution 1 was diluted to 100 mL with 1.0 M HClO₄, solution 2 was diluted to the mark with 1.0 M NaOH, and solution 3 was diluted to the mark with water. A 25.0 mL aliquot of each was extracted with 25.0 mL of *n*-hexane. The extract from solution 2 contained no detectable trace of A-containing species, indicating that A⁻ is not soluble in the organic solvent. The extract from solution 1 contained no ClO₄⁻ or HClO₄ but was found to be 0.0454 M in HA. The extract from solution 3 was found to be 0.0225 M in HA. Assume that HA does not dissociate or associate in the organic solvent, and calculate:

(a) The distribution ratio for HA between the two solvents. [4 marks]

(b) The concentration of the species HA and A⁻ in aqueous solution 3 after extraction. [4 marks]

(c) The dissociation constant of HA in water. [4 marks]

(d) What is supercritical fluid extraction? What advantages does it offer? [8 marks]

End of question Paper!!!