

**NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY**  
**DEPARTMENT OF APPLIED CHEMISTRY**  
**SUPPLEMENTARY EXAMINATIONS – JULY 2004**  
**ANALYTICAL CHEMISTRY III – SCH 4206**  
**TIME – 3 HRS**

**INSTRUCTIONS TO CANDIDATES**

Answer **Any Four** questions.  
Each question carries 25 marks.

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1. (a) The distribution coefficient for X between chloroform and water is 9.6. Calculate the concentration of X remaining in the aqueous phase after 50.0 ml of 0.150M X is treated by extracting with the following quantities of chloroform
- (i) One 40.0 ml portion
  - (ii) Two 20.0 ml portions
  - (iii) Four 10.0 ml portions
  - (iv) Eight 5.00 ml portions [15 marks]
- (b) Differentiate between sorbed water, adsorbed water and occluded water. [6 marks]
- (c) What fluxes are suitable for the determination of alkali metal silicates? [4 marks]
2. (a) Derive the expression that relates the distribution ratio, D, to the distribution coefficient,  $K_d$  and the dissociation constant  $K_a$ . [10 marks]
- (b) In a liquid-liquid extraction using organic chelating reagents the distribution ratio is dependent on pH and on the concentration of the chelating reagent. Using relevant equilibrium expressions and appropriate equations derive an expression, which support this statement. [15 marks]
3. (a) 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  $3.0 \text{ g.cm}^{-3}$  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? [5 marks]
  - (ii) What should the weight of the gross sample be? [2 marks]

- (iii) To what diameter must be particles ground in order to yield a sample for analysis that weighs 0.750 g and has the same number of particles as the gross sample? [3 marks]
- (b) Describe the sources of error in decomposition and dissolution of samples. [8 marks]
- (c) Explain why microwave digestion is more efficient than conventional methods. What other advantages does microwave digestion offer? [7 marks]
4. (a) Using clearly labelled diagrams describe three discrete automatic systems for sampling and sample definition of fluids. [15 marks]
- (b) Explain how traces of manganese, lead and zinc could be separated from major components of the sample using precipitation methods. [5 marks]
- (c) Briefly describe three types of solvent extraction procedures. [5 marks]
5. (a) Lead forms a neutral complex  $PbQ_2$  with the ligand Q. The constant  $K_{ex}$  for the distribution of this complex between water and  $CCl_4$  has been found experimentally to be  $2.0 \times 10^4$ . A 25.0 mL aliquot of an aqueous solution that is  $5.00 \times 10^{-4}$  in  $Pb^{2+}$  and 0.500 M in  $HClO_4$  is extracted with two 10.0 mL portions of  $CCl_4$  that are 0.0250 M in HQ. Calculate the percentage of the unrecovered  $Pb^{2+}$  in the aqueous solution. [7 marks]
- (b) A proposed method for the determination of the chemical oxygen demand of wastewater was compared with the standard (mercury salt) method. The following results were obtained for a sewage effluent sample:

|                 | Mean mg/L | Standard Deviation mg/L |
|-----------------|-----------|-------------------------|
| 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? [7 marks]

- (c) Discuss the steps involved in a typical analytical quantitative analysis. [11 marks]

**END OF QUESTION PAPER!!!**