NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY
DEPARTMENT OF APPLIED CHEMISTRY
BACHELOR OF SCIENCE HONOURS DEGREE
SUPPLEMENTARY EXAMINATION – AUGUST 2014
ANALYTICAL CHEMISTRY III – SCH 4206
TIME: 3 HOURS

INSTRUCTIONS TO CANDIDATES
Answer **ANY FIVE** questions out of **SIX** questions provided.
Each question carries 20 marks.

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1. Copper (II) reacts with the chelating agent HL to give a complex CuL_2 that is readily soluble in chloroform. A spectrophotometric study revealed that when a 1.00×10^{-4}M aqueous solution of copper (II) was extracted with $CHCl_3$ that was 0.010M in H_2L, the analytical concentration of copper in the two phases was identical at pH 5.65

   (a) Write equations describing the equilibria in the system, assuming that dissociation of CuL_2 in the organic phase is negligible. [4 marks]

   (b) Calculate K_D [4 marks]

   (c) Calculate the distribution ratio for the system at pH 6.00. [4 marks]

   (b) If 50.0mL of 5.00×10^{-5}M Cu^{2+} in a pH 6.00 buffer were to be extracted with 25.0mL portions of 0.010M H_2L in $CHCl_3$, how many extractions would be required to remove 99% of the copper from the aqueous phase? [4 marks]

   (e) Repeat the calculations in part (d) for 99.9% removal. [4 marks]

2. (i) Discuss the differences and advantages that microwave digestion has over wet ashing using conventional heating. [10 marks]

   (ii) With the aid of a diagram, describe the vessels used for moderate pressure microwave digestion. [10 marks]

3. You are assigned to investigate the extent of lead contamination in soil samples. Discuss the possible sources of lead contamination in the environment, and the effects of lead on human health. Explain how you would design a quantitation method for the determination of lead taking into account that it is present in the sample in trace concentration. [20 marks]

4. (a) In the analysis of environmental samples, briefly describe what do the following terms mean and how are they determined experimentally?

   (i) Total Organic Content (TOC) [5 marks]

   (ii) Chemical Oxygen Demand (COD) [5 marks]

(b) What is a masking agent and how does it function? [5 marks]

(c) What are the steps involved in obtaining a laboratory sample? [5 marks]

5. (i) What is a super critical fluid? [2 marks]

(ii) Discuss the advantages of supercritical fluid extraction (SFE) over solvent extraction. [6 marks]

(iii) Differentiate between the following terms as used in solvent extraction.

(a) An exhaustive and countercurrent extraction. [4 marks]

(b) Distribution coefficient and distribution ratio. [4 marks]

(iv) What are the main disadvantages of solvent extraction? [4 marks]

6. (a) A coating that weighs at least 3.00 mg is needed to impart adequate shelf life to a pharmaceutical tablet. A random sampling of 250 tablets revealed that 14 failed to meet this requirement.

(i) Use this information to estimate the relative standard deviation for the measurement. [2 marks]

(ii) What is the 90 % confidence for the number of unsatisfactory tablets? [3 marks]

(iii) Assuming that the fraction of rejects remains unchanged, how many tablets should be taken for inspection to ensure a relative standard deviation of 10% in this measurement. [4 marks]

(b) In a comparison of two methods for the determination of chromium in rye grass, the following results (mg kg^{-1}) were obtained:
Method 1: mean = 1.48; standard deviation = 0.28
Method 2: mean = 2.33; standard deviation = 0.31
For each method five determinations were made. Do these two methods give results having means which differ significantly? [5 marks]

(c) There are two general methods of dealing with interferences. Describe these and give specific examples. [6 marks]

End of question Paper!!!