



**NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY**

**FACULTY OF APPLIED SCIENCES**

**DEPARTMENT OF APPLIED CHEMISTRY**

**ANALYTICAL III**

**SCH 4206**

**First Semester Examination Paper**

**December 2017**

This examination paper consists of 5 pages

**Time Allowed: 3 hours**

**Total Marks: 100**

**Examiner's Name: Dr. A. Maringa**

**INSTRUCTIONS**

- a) Answer ALL questions in section A and any three (3) questions in section B.
- b) Section A carries 10 marks and each question in section B carries 20 marks.

**Special requirements: Calculator**

**MARK ALLOCATION**

<b>QUESTION</b>	<b>MARKS</b>
<b>SECTION A: 1.</b>	<b>40</b>
<b>SECTION B: 2.</b>	<b>20</b>
3.	<b>20</b>
4.	<b>20</b>
5.	<b>20</b>
<b>TOTAL POSSIBLE MARKS</b>	<b>100</b>

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## **SECTION A**

1. (a). Distinguish between essential water and nonessential water. [4 marks]
- (b). The following wet-ashing procedure was used to measure arsenic in organic soil samples by atomic absorption spectroscopy: A 0.1 g sample was heated in a 150-mL Teflon bomb in a microwave oven for 2.5 min with 3.5 mL of 70% HNO<sub>3</sub>. After the sample cooled, a mixture containing 3.5 mL of 70% HNO<sub>3</sub>, 1.5 mL of 70% HClO<sub>4</sub>, and 1.0 mL of H<sub>2</sub>SO<sub>4</sub> was added and the sample was reheated for three 2.5-min intervals with 2-min unheated periods in between. The final solution was diluted with 0.2 M HCl for analysis. Explain why HClO<sub>4</sub> was not introduced until the second heating. [4 marks]
- (c). What types of contamination and changes in composition can occur during crushing and grinding? [6 marks]
- (d). A study of the possible relationship between traffic density and concentration of lead, cadmium and zinc in roadside soils, made use of the following sampling plan: Samples of surface soil (0-10 cm) were collected at perpendicular distances of 1, 5, 10, 20, 30 m from the roadway. At each distance, 10 samples were taken from different locations and mixed to a single sample. What type of sampling plan is this? Explain why this is an appropriate sampling plan [6 marks]
- (e). What are the reasons for installing automatic analysers to replace manual methods? [5 marks]

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(f).

**Table 1: Selected Inorganic and Organic Masking Agents for Metal Ions**

Masking Agent	Elements Whose Ions Can Be Masked
CN <sup>-</sup>	Ag, Au, Cd, Co, Cu, Fe, Hg, Mn, Ni, Pd, Pt, Zn
SCN <sup>-</sup>	Ag, Cd, Co, Cu, Fe, Ni, Pd, Pt, Zn
NH <sub>3</sub>	Ag, Co, Ni, Cu, Zn
F <sup>-</sup>	Al, Co, Cr, Mg, Mn, Sn, Zn
S <sub>2</sub> O <sub>3</sub> <sup>2-</sup>	Au, Ce, Co, Cu, Fe, Hg, Mn, Pb, Pd, Pt, Sb, Sn, Zn
Tartrate	Al, Ba, Bi, Ca, Ce, Co, Cr, Cu, Fe, Hg, Mn, Pb, Pd, Pt, Sb, Sn, Zn
Oxalate	Al, Fe, Mg, Mn
Thioglycolic	Cu, Fe, Sn

Source: Meites, L. *Handbook of Analytical Chemistry*, McGraw-Hill: New York, 1963

Using the Table 1, suggest a masking agent (with reasons) for the analysis of Fe in the presence of Al. [5 marks]

(g). Samples need to be stored prior to analysis. Describe the factors that you will consider in order to store organic trace pollutant samples. [10 marks]

## **SECTION B**

2. (a) What is meant by fusion as a method for sample dissolution? [2 marks]
- (b) Why is it important to reduce the size of solid particles present in a sample? [3 marks]
- (c) What are the hazards associated with the use of diethyl ether and what specific precautions are taken? [5 marks]
- (d) Differentiate between solid–liquid extraction, liquid–liquid extraction, and solid phase extraction. [10 marks]
3. (a) What is the minimum distribution coefficient that permits removal of 99% of a solute from 50.0 mL of water with
- i. Two 25.0 mL extractions with toluene? [5 marks]
- ii. Five 10.0 mL extractions with toluene? [5 marks]
- (b) If 30.0 mL of water that is 0.0500 M in Q is to be extracted with four 10.0 mL portions of an immiscible organic solvent, what is the minimum distribution coefficient that allows transfer of all but the following percentages of the solute to the organic layer:
- i.  $1.00 \times 10^{-4}$  [5 marks]
- ii.  $1.00 \times 10^{-3}$  [5 marks]
4. (a) What do you think are the relative merits of laboratory and field analyses? [8 marks]
- (b) Microwave assisted extraction (MAE) method was used to extract semi volatile organic compounds. Describe the procedure involved and highlight the advantages and disadvantages of MAE. [12 marks]

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5. (a). An analytical technique for copper, lead, cadmium and zinc in water, as described in the chemical literature, involved dividing the filtered sample into five aliquots. The treatment of the aliquots (prior to ASV analysis) was as follows:
- i. Strong chemical oxidation and UV irradiation.
  - ii. No pretreatment.
  - iii. Weak chemical oxidation.
  - iv. Passage through a chelating resin then UV irradiation.
  - v. Extraction using an organic solvent, and UV irradiation of the aqueous phase.

Which species are determined in each aliquot? [10 marks]

- (b). A number of samples to be analyzed for traces of a common solvent are taken from a river flowing through a highly polluted area. The samples are transported to the laboratory for analysis by gas chromatography. Which steps of the procedure would need to be monitored in order to ensure that the sample was not contaminated? What quality control procedure could you introduce to ensure reliability of the analytical result? [10 marks]

***End of question paper!!!!***

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