CHUKA



UNIVERSITY

## UNIVERSITY EXAMINATIONS

# EXAMINATION FOR THE AWARD OF DEGREE OF DOCTOR OF PHILOSOPHY IN CHEMISTRY

## CHEM 941: ELECTRO ANALYTICAL TECHNIQUES AND VALIDATION

**STREAMS: PhD (CHEMISTRY)** 

**TIME: 3 HOURS** 

DAY/DATE: WEDNESDAY 14/08/2019

2.30 P.M - 5.30 P.M.

**INSTRUCTIONS** Answer **All** Questions

### **QUESTION ONE: [20 MARKS]**

1. (a) Give a brief explanation on the importance of voltammetric method as a technique for trace analysis of pollutants in the environmental sample. [3<sup>1</sup>/<sub>2</sub> Marks]

(b) Bring out clearly the tasks for analytical Chemistry in the environmental field and the criteria placed on the analytical methods for environmental sample analysis.  $[3\frac{1}{2} Marks]$ 

(c)Discuss how stripping voltammetric technique using pencil graphinte electrodes can be applied for the analysis of available copper, zinc and manganese content in soil samples from Mwea Rice Scheme. Also, explain how you can;

- (i) Select the best method for digestion
- (ii) Establish the accuracy of the technique [7 Marks]
- (d) (i) Derive an equation relating cell potential and current in reference to voltammetric technique for a solution with only;

(i) Oxidized from	[2 Marks]
(ii) Reduced form	[½ Mark]

(ii) The following data were obtained from a linear scan hydrodynamic voltammogram of a reversible reduction reaction.

E(VVSSCE0.372	-0.382 -0.420	0 -0.410 -0.435
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	0.358					
Current (A)	0.37	0.97	1.1	3.48	4.20	4.97

The limiting current was 5.15 A. Show that the reduction is reversible and determine values of n and for . [2 Marks]

(iii) The differential pulse polarographic analysis of a mixture of indium and cadmium in 0.1 M Hcl is complicated by the overlap of their respective voltammograms. The peak potential for indium is at -0.557V and that for cadmium is at -0.597V. When a 0.800 ppm indium standard is analyzed, ip (in arbitrary units) is 200.5 at 0.557V and 87.5 at -0.597 V. A standard solution of 0.793 ppm cadmium has a ip of 58.5 at -0.557 V. Calculate the concentration of indium and cadmium in a sample if ip is 167.0 at a potential of -0.557V and 99.5 at a potential of -0.597 V. All potentials are relative to a saturated Ag/Agcl reference electrode. [1½ Marks]

#### **QUESTION TWO: [20 MARKS]**

- (a) Explain using suitable examples why voltammetric techniques are coupled with spectroscopic techniques. [4<sup>1</sup>/<sub>2</sub> Marks]
- (b) Discuss how cyclic voltammetry when combined with EPR or UV-VIS-NIR spectroscopy can help to search for new organic materials that can be used in organic electronics with desirable performance or stability. [4<sup>1</sup>/<sub>2</sub> Marks]
- (c) Write short notes on Aptamer based biosensor Array. [4½ Marks]
- (d) (i) Justify the statement given below;
  "Electrochemical Biosensors is a solution to pollution detection with reference to Environmental contaminants".

(ii) Discus some of the problems which have prevented electrochemists to build electrochemical systems for oxidative purification of effluents and some of the remedies for this. [2 Marks]

#### **QUESTION THREE: [20 MARKS]**

- (a) Explain why a combination of fast-scan cyclic voltammetry with scanning electrochemical microscopy is likely to be a powerful tool for chemical imaging compared with the combination of anodic voltammetry at mercury microelectrodes with scanning electrochemical microscopy. [5 Marks]
- (b) Discuss into detail the stepwise validation of analytical techniques for the determination of various inorganic and organic substances in the environmental samples. [7 Marks]

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(c) (i) Give reasons to why analytical measurements in flowing liquids are steadily gaining importance in all fields, including environmental analysis. [2 Marks]

(ii) Outline the most important trends for future development of electrochemical flow detection. [2 Marks]

(iii) State two major challenges facing analyst when determining the residual pharmaceuticals in environmental water samples using capillary electrophoresis (CE) and also suggest ways of overcoming these challenges. [2 Marks]

(d) Discuss the application of capillary electrophoresis in environmental analysis. [2 Marks]