KAT 347/3 – Electroanalytical Methods

Course Objectives: 1) To understand the electroanalytical technique of analysis namely potentiometry and voltammetry.
2) To understand various voltammetric techniques including poloragraphy and amperometry.

Topic	Content	Number of lecture hours	Expected outcome – upon completion of this course, the student should be able to:
1. General overview	Brief overview on techniques, i.e potentiometry and voltammetry	2	• Acquire a general overview of potentiometry and voltammetry
2. Potentiometry	Definition for ion selective/sensitive electrode (ISE), Nomenclature, Cell schemes, Half cell, Concept of electrode potential, Output signal, E/mV and Nernst equation	2	• Understand that the analysis is carried out at a controlled current, i.e. I = 0. Know the pros and cons of this technique.
	Electrode sensitivity, Calibration plot (E/mV vs Log c) , Nernst slope, Semi-log graph paper, Grans ruler (Beckman's)	2	• Know parameters that affect sensitivity of any ISE e.g. slope, response & recovery times, limit of detection (LOD), signal to noise ratio.
	Electrode selectivity (K_{ij}^{pot}), Nicolsky-Eisenmann equation, Determination – mixed and separate solution, Relationship of K_{ij}^{pot} with LOD, pH profile and total ionic adjustment buffer (TISAB)	2	• Understand that the performance of an ISE also depends on its capability to sense the primary ions only (analytes) not the secondary ions (interferences).
	Membrane and mechanism – exchange and redox – therein, Non-nernstian response, Donnan equilibrium and potential.	2	• Know the significance of the membrane in ISE construction. Be able to predict which mechanism is involved in producing a signal output, depending on oxidation state of the primary ions. Understand what sub- and super-Nernstian responses are.
	Membrane, Solid and liquid – Homogeneous and heterogeneous. Preparation, Mechanism and improvisation. Examples: glass, fluoride, Ca and K electrodes.	4	• Understand how the electrodes are generally prepared. Know how to improve their selectivities. Know the terms: doped single crystal salts, acid and base error, macrocyclic and ionophores.

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3. Voltammetry	Automation of analysis involving ISE. Flow injection analysis (FIA), ISFET/MOSFET and sensor arrays	2	• Understand the requirements for real sample analysis using solid state type – coated wire electrode (CWE) and size miniaturization. Know about chemometric analysis
	Definition for voltammetry, Electrochemical cell, Polarography, Output signal, I/mA and Illkovic equation	2	• Understand that the analysis is carried out at a controlled potential, i.e. at a fixed E or in a known potential range. Pros and cons of this technique.
	Electrochemical series, Standard potential (E^0), electroactivity, Electrode/redox process,	1.5	• Understand basic electrochemistry .
	Phenomenon of various layers in solution around inert electrodes. Types of current generated by the electrodes processes.	1.5	• Understand concepts of double, diffusion and depletion layers. Realize that the output signal is the summation of Faradaic as well as non-Faradaic currents.
	Mass transfer of solutes/analytes to the electrode surface, Polarization and overpotential (η)	1	• Know that diffusion parameters are significant in many analyses. Understand that an excess voltage is sometimes essential to start an analysis.
4. Classic Polarography	Dropping mercury electrode (DME), Characteristics – purity, Strength and Weaknesses. Instrumentation and sample cell requirements. Ohmic drop (iR).	2	• Know the precautions needed when handling mercury (Hg). Know how to purify Hg. Understand the requirements of a classic polarographic analysis.
	I/E plot, Polarographic waves, Quantative (diffusion current, I_d) and Qualitative (half potential, $E_{1/2}$) analysis, Calibration plot.	2	• Understand how the analysis is done.

Topic	Content	Number of lecture hours	Expected outcome – upon completion of this course, the student should be able to:
	Checking of reversibility of electrode process at a DME by graph and Meites method.	1	• Know that an irreversible process is unwanted in any voltammetric analysis
5. Advanced Polarography	Pulse and stripping techniques. Exploitation of the growth of Hg drop. In depth discussion on various ways of doing stripping analysis. I/E plot. Solid mercury electrode, i.e. mercury thin film electrode (MTFE)	4	• Be able to decide which electrochemical method to use for their analysis. Know that MTFE is the best electrode for polarographic analysis.
6. Amperometry	Techniques using solid (non-mercury) electrodes which also include MTFE. Cyclic voltammogram (CV). Kinetic and Mechanistic study, reversibility and Diagnostic test.	3	• Know that this technique is only used for analysis if the concepts in polarography are well understood.
	Introduction on chemically modified electrodes (CME) and miniaturization of the electrodes.	2	• Know the current trends in electrochemical methods of analysis.
	TOTAL	**36**	