SAULT COLLEGE
of Applied Arts and Technology
Sault Ste. Marie

COURSE OUTLINE

INSTRUMENTAL METHODS OF ANALYSIS

CHM 206 - 4

revised June 1981 by J. Korrey

TEXT & REFERENCES

1. Instrumental Methods of Analysis by Willard, Merritt & Dean, 5th edition, D. Van Nostrand & Co. Inc.

2. Fundamentals of Analytical Chemistry by Skoog & West - Holt, Rinehart & Winston.

3. Principles of Instrumental Analysis by Skoog & West - Holt, Rinehart & Winston.

4. Gas Chromatography by C. Simpson - Kogan Page, London

5. A Programmed Introduction to Gas-Liquid Chromatography by J. B. Pattison, 2nd edition, Heyden & Son, Ltd.

6. Atomic Absorption Spectroscopy by R. J. Reynolds & K. Aldous - Charles Griffon & Co. Ltd.

7. Applications of Absorption Spectroscopy of Organic Compounds by John Dyer, Foundations of Modern Organic Chemistry - Prentice-Hall

8. Practical Polarography by Heyrovsky, J. & Zuman, P. - Academic Press

9. Atomic Absorption Spectroscopy by J. W. Robinson - Marcel Dekker Inc.

COURSE OUTLINE

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UNIT I: Electrochemistry

1. Review of Electrochemical Theory
2. Electrical Properties of Cells
   - (a) E.M.F.
   - (b) Conductance
   - (c) Ohm's Law
   - (d) Faraday's Law
   - (e) Coulomb's Law
3. Types of Half Cells
4. Nernst Equation

UNIT II: Electrometric Methods of Analysis

1. Summary of Methods
2. Different Kinds of Electrodes:
   - (a) Reference Electrodes
     1. Hydrogen Gas
     2. Calomel
     3. Silver-Silver Chloride
     4. Weston Cell
   - (b) Indicator Electrodes
     1. Glass Membrane Electrodes
     2. Solid State
     3. Liquid Ion - Exchange
     4. Gas Sensing
     5. Special Purpose (enzyme electrodes)
3. Measurement of pH:
   - (a) Basic Principles
   - (b) How a glass electrode measures pH
   - (c) Applications of the glass electrode
   - (d) Errors in pH measurement with the glass electrode

4. Buffers

5. Calibration of pH meters

6. Potentiometric Titrations:

   (a) Principles of the Potentiometer

   (b) Potentiometric Titration Methods

      1. Location of the End Pt. (including graphical methods)
      2. Use of Two Indicating Electrodes
      3. Titrations at Constant Electrolysis Current

   (c) Classes of Chemical Titrations

      1. Acid-Base Reactions (in aqueous and non-aqueous media)
      2. Oxidation - reduction
      3. Precipitation (Ion combination reactions)

UNIT III: Separations by Electrolysis

1. Basic Principles

2. Completeness of Depositions

3. Overpotentials

4. Anode Processes

5. Constant Current Electrolysis

6. Separations with Controlled Electrode Potentials

7. Constant Voltage Electrolysis

8. Characteristics of the Deposit

9. Physical & Chemical Factors of Importance in Electrodeposition

UNIT IV: Polarography

1. Basic Principles

2. Interpretation of Polarographic Waves

3. Ilkovic Equation

4. Half Wave Potentials

5. Polarographic Maxima

6. Factors Affecting Diffusion Current

7. The Dropping Mercury Electrode

   (a) Characteristics
   (b) Advantages
   (c) Disadvantages

8. Removal of Dissolved Oxygen

9. Polarograms for Mixtures of Reactants

10. Evaluation Methods

    (a) Direct Comparison
    (b) Standard Addition
    (c) Internal Standard

11. Organic Polarographic Analysis

UNIT V: Optical Methods of Analysis

1. The Electromagnetic Spectrum

2. Definition of Terms:

   Absorbance, Absorptivity, Molar Absorptivity, Transmittance, Frequency, Velocity, Wavelength, Wavenumber, Radiant Energy and Related Units of Measurements, etc.

3. Fundamental Laws of Photometry

   (a) Beer's Law
   (b) Lambert's Law

4. Failures of the Beer-Lambert Law.

   (a) Chemical
   (b) Instrumental

5. Choice of Wavelength

6. Simultaneous Determination of Two or More Components

7. Relative Concentration Error

8. Photometric Titrations

9. Basic Principles of the Absorption of Infrared Radiation to include:

   (a) Molecular Vibrations
   (b) Requirements for IR Absorption

10. Methods of Handling Gaseous, Liquid and Solid Samples.

11. Qualitative and Quantitative Analysis

12. Comparison of Ultra-Violet, Visible and Infrared Spectrophotometers with Respect to the Following:

   (a) Radiant Energy Sources - Tungsten Lamp, Hydrogen Discharge Lamp, Nernst Glowers, Globars

   (b) Monochromators (Dispersing Devices) - Filters, Prisms and Gratings

   (c) Sample Containers

   (d) Detectors -

      1. Barrier Layer or Photovoltaic Cells
      2. Photoemission Tubes
      3. Photomultiplier Tubes
      4. Thermal

13. General Principles of:

   (a) Colorimeters
   (b) Single and Double Beam Spectrophotometers

UNIT VI: Atomic Absorption and Emission Spectroscopy

1. Origin of Emission Spectra

2. Excitation Methods

3. Comparison of Flame Photometry, Atomic Absorption and Emission

4. Advantages and Disadvantages of A.A.

5. Interferences (including Matrix Effect)

6. Instrumentation

   (a) Single Beam Systems
   (b) Double Beam Systems

7. Radiation Sources

8. Modulation

9. Atomization

10. Monochromators

11. Detectors

12. Analytical Parameters

Choice of Analytical Wavelength Adjustment:

Source - H.C. Lamp Current
- H.C. Lamp Alignment

Atomizer - Gas Composition
- Sample Flow Rate
- Burner Alignment

Monochromator - Wavelength Adjustment
- Slits Adjusted

Amplifier Gain - Maximum Signal
- Lowest Noise

UNIT VII: Gas Chromatography

"A" - Operating Parameters:

1. Temperature Effect
2. Sample Size Effect
3. Carrier Gas Effect
4. Column Selection
5. Detector Selection
6. Flow Rate

"B" - Theoretical

1. Peak Area
2. Retention Time
3. Adjusted Retention Time
4. Separation
5. Resolution
6. Efficiency (Van Deemter Equation)
7. Choice of Solid Support
8. Particle Size
9. Stationary Phase Loading

10. Choice of Stationary Phase
11. Preparation of Packing Material
12. Packing the Column
13. Detectors:

A. Differential

(a) F.I.D.
(b) Thermal Conductivity
(c) Electron Capture
(d) Gas Density

B. Integral

Titration Cell

14. Kovats Retention Index