SAULT COLLEGE OF APPLIED ARTS & TECHNOLOGY

SAULT STE. MARIE, ONTARIO

COURSE OUTLINE

Course Title: INSTRUMENTAL ANALYSIS II

Code No.: CHM 231-5

Program: WATER RESOURCES TECHNOLOGY

Semester: FIVE

Date: JUNE, 1984

Author: J. S. KORREY

New: _____ Revision: X

APPROVED: _____ _____
Chairperson Date

CALENDAR DESCRIPTION

ANALYTICAL CHEMISTRY - INSTRUMENTAL ANALYSIS II
COURSE NAME

CHM 231-5
COURSE NUMBER

PHILOSOPHY/GOALS:

Instrumental analysis II expands on topics covered in the first instrumental course (CHM 221-5) and in addition, the following will be discussed:

U.V-Visible & I.R. Spectrophotometry, Atomic Absorption and Emission, Gas Chromatography, Liquid Chromatography, Electrochemistry, Specific Ion Electrodes and Polarography.

METHOD OF ASSESSMENT:

A	80 - 100
B	70 - 79
C	60 - 69
I	59 or less

Weighting:

Theory	50%
Lab	50%

Theory: The grade is the sum of all tests and assignments. Tests will include all work up to the time of the test.

Labs: Late labs will be penalized 10% per week. Late assignments will NOT be accepted.

Attendance: Your grade will be greatly affected by attendance at scheduled classes. 85% is required at theory classes while 100% is mandatory for all labs. Serious illness (doctor's care) is the only valid excuse.

TEXTBOOK:

Robinson, James W., Undergraduate Instrumental Analysis, Third Edition, Revised and Expanded, Marcel Dekker Inc., N.Y., 1982.

Braun, Robert, D., Introduction to Chemical Analysis, McGraw-Hill, 1982.

TEXT & REFERENCES

*1. Undergraduate Instrumental Analysis by James W. Robinson, Marcel Dekker, 3rd edition.

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

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

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

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

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

7. Atomic Absorption Spectroscopy by R.J. Reynolds & K. Aldous _ Charles Griffon & Co. Ltd.

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

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

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

*STUDENT TEXT

COURSE OUTLINE

INSTRUMENTAL ANALYSIS II

CHM 231-5

UNIT I: Electrochemistry

1. Review of Electrochemical Theory

2. Electrical Properties of Cells

   (a) E.M.R.
   (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. Calomel
      2. Silver-Silver Chloride

   (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) End Point Determination

      1. Location of the End Pt. (including graphical methods)

   (b) Classes of Chemical Titrations

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

UNIT III: Polarography

1. Basic Principles

2. Interpretation of Polargraphic Waves

3. Half Wave Potentials

4. Polarographic Maxima

5. Factors Affecting Diffusion Current

6. The Dropping Mercury Electrode

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

7. Removal of Dissolved Oxygen

8. Polarograms for Mixtures of Reactants

9. Evaluation Methods

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

10. Organic Polargraphic Analysis

11. Diffusion Polargraphy

12. Applications

UNIT IV: Optical Methods of Analysis

1. The Electromagnetic Spectrum

2. Definition of Terms: (Review covered in Instrumental I)

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

3. Fundamental Laws of Photometry (Review - covered in Instrumental I)

   (a) Beer's Law
   (b) Lambert's Law
   (c) Combined Beer-Lambert Law

4. Failures of the Beer-Lambert Law.

   (a) Chemical
   (b) Instrumental

*5. Choice of Wavelength (LAB)

*6. Simultaneous Determination of Two or More Components (LAB)

7. Relative Concentration Error

8. Photometric Titrations

9. Basic Principles of teh 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 of Photovoltaic Cells
        2. Photoemission Tubes
        3. Photomultiplier Tubes
        4. Thermal

13. General Principles of:

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

UNIT V: 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 VI: Gas Chromatography

"A" - Operating Parameters: (Discussed in Instrumental I)

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

"B" - Theoretical (Discussed in Instrumental I)

1. Peak Area
2. Retention Time
3. Adjusted Retention Time
4. Separation
5. Resolution
6. Efficiency (Van Deemter Equation)

"C" - Column Technology

1. Choice of Solid Support
2. Particle Size
3. Stationary Phase Loading
4. Choice of Stationary Phase

5. Preparation of Packing Material
6. Packing the Column

Detectors:

A. Differential B. Integral

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

UNIT VII: Liquid Chromatography (Time Permitting)

1. Comparison of Liquid vs Gas Chromatography
2. Instrumentation and Methods
3. Types of HPLC
4. Solvents
5. Gradients
6. Analytical Procedures
7. Preparative Procedures
8. Quantitation
9. Detectors

LABORATORY EXPERIMENTS FOR
CHM 231-5
ANALYTICAL CHEMISTRY
INSTRUMENTAL ANALYSIS II

TIME: 3 hours/week x 15 weeks = 45 hours

Due to the limited amount of instrumentation available, the following experiments will be performed on a rotating basis.

A. Atomic Absorption and Emission Spectrophotometry:

1. Absorption: 6 Hours

a) Determination of heavy metals (e.g. Zn or Pb) in plant effluent.

b) Determination of Manganese in natural waters.

2. Emission: 3 Hours

Determination of Salinity (Na and K) in industrial wastes.

B. Gas Chromatography 6 Hours

a) Determination of hydrocarbon contaminants in water using the Pye-Unicam SP-90 A.A.S. or, Perkin Elmer 3920 A.A.S.

b) Determination of industrial pollutants (solvents) in waste water.

C. Spectrophotometric Methods

I. Spectronic 20 Colourimeter 9 Hours

a) Determine the relative response of the phototube, colourimeter and lamp intensity.

b) Determine the optimum wavelength to use in an analysis

c) Study applicability of Beer's Law

d) Determine the concentration of an unknown Cr (III) Solution obtained from plating shop effluent.

e) Determine the concentration of Chromium (III) and Cobalt (II) in a sample obtained from mining effluent.

-or-

f) Determination of sulphate in surface waters and ground water.

II. Coleman Spectrophotometer (U.V.-Vis.) 6 Hours

a) Determination of Phenols (Method 510D) in industrial waters and drinking water.

-or-

b) Determination of Phosphates in natural waters and waste waters (Method 425D).

-or-

c) Determination of Nitrogen (Nitrate) in polluted natural waters and water supplies (Method 419A).

III. Unicam SP1000 IR Spectrophotometer 6 Hours

a) Record the IR absorption spectrum of Hexane. Identify the absorption bands caused by:
- the C H stretching frequency
- the C H bending frequency
- teh C C stretching frequency

b) Record the IR spectrum of heptane.
Note the similarity to the spectrum of hexane. Would it be possible to distinguish between these compounds based on their IR system?

c) Record the IR spectrum of n-butanol.
Note the O H stretching peak and the C OH stretching peak.

- Repeat using i-butanol and t-butanol.

- Note the change with the C OH stretching peak but little change with the O H stretching peak.

- Could this change be used to distinguish among primary, secondary and tertiary alcohols?

d) Record the IR spectrum of n-butylamine.
Note the N H stretching pear and the C N stretching peak

Repeat with sec-butylamine and tert-butylamine.

IV. Carbon Analysis

Determination of organic, inorganic and total carbon in surface waters, waste water and saline water by use of carbon analyzer (Beckman 915B Tocamaster).

e) Determination of grease and oil in water and waste water by a partition _ Infrared Method (Method 502B and E)

ELECTROMETRIC METHODS:

I. Specific Ion Electrodes — 6 Hours

a) Determination of fluoride in drinking water.

b) Determination of chloride in drinking water.

c) Determination of nitrate in natural waters.

d) Determination of ammonia in drinking water, clean surface water and waste water effluent.

e) Determination of sodium in water by the Method of Standard Addition.

II. Amperometric Titration — 6 Hours

Determination of free, total and combined residual chlorine in water and waste water (Method 409C) using the chlorine Titrimeter.

III. Polarography

a) Determination of heavy metals (Zn, Cu, Pb, Cd) in waste water effluent.

b) Determination of phosphates in waste water effluent.

IV. Acidity/Alkalinity

Determination of acidity/alkalinity of water/waste water using the Titroprocessor.