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 ENGINEERING TECHNOLOGY

Semester: FIVE

Date: MAY, 1986

Author: J. S. KORREY

New: _____ Revision: X

APPROVED: _____ June 12/86.

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	50 - 59
R	49 OR LESS

Weighting:	
TEST #1	20
TEST #2	20
ASSIGNMENTS	10
THEORY	50%
LAB	50%

Lab Grade:

5 labs at @ 20 each = 100 marks
1 lab seminar = 15 marks

115 marks

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 and/or late assignments will NOT be accepted. Reports are due within two weeks after dates established by the instructor at lab completion.

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.

Rewrites: Students with an "I" grade (50-59%) may rewrite if:

1. They have written all the tests.
2. They have turned in their assignments on time.
3. They were in attendance for at least 85% of the Theory classes and 100% of ALL scheduled lab sessions, and then only at the discretion of the instructor.
4. Rewrites will be an exam on the whole semester's work in theory and lab.

TEXTBOOK:

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

Supplemental Texts and References:

Marr and Cresser, Environmental Chemical Analysis, International Textbook Company, 1983.

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.

UNIT I: METHODS

8 hours

1-1 Choosing a Method

1-2 Criteria for Selecting a Method

1-3 Sources of Error in Trace Analysis

1-4 Sampling

1-5 Dissolution and Decomposition of Samples

1-6 Separation

1-7 Determination

The Analysis of Water: What is Required?

1-8 The Storage of Samples and Prevention of Contamination

1-9 Selected Analytical Methods for Water Quality Control

UNIT II: Electrochemistry

8 hours

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

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)

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. Other Voltammetric Techniques

11. TEST #1

UNIT III: Optical Methods of Analysis IR AND U.V.

6 hours

1. Choice of Wavelength (LAB)

2. Simultaneous Determination of Two or More Components (LAB)

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

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

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

5. Qualitative and Quantitative Analysis

6. 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 - Photomultiplier Tubes

7. General Principles of:

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

8. Other Optical Techniques (Time Permitting)

   (a) Fluorescence
   (b) X-Ray Diffraction

UNIT IV: Atomic Absorption and Emission Spectroscopy

1. Comparison of Flame Photometry, Atomic Absorption and Emission

2. Advantages and Disadvantages of A.A.

3. Interferences (including Matrix Effect)

4. Atomization, fuels

5. 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 V: 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)

2 hours "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

"D" - Detectors

A. Differential B. Integral

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

2 hours 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

TEST #2

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

a) 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 peak 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).

ELECTROMETRIC METHODS:

1. 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.

2. Amperometric Titration 6 Hours

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

3. Polarography

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

   b) Determination of phosphates in waste water effluent.

4. Acidity/Alkalinity

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

One lab seminar (10 marks) will be required of each student. It will be on the first instrument assigned. The seminar will be during the third week of the experiment and it will be in two parts.

1. Explain principles of the instrument to class & instructor.

2. Explain operation of the instrument to the class instructor.