OBJECTIVES

for

CHM 231-5

J. S. Korrey
MAY, 1986

CHM 231-5

Unit I: METHODS

After completion of this unit, the student should be able to:

1. Select a suitable method for analysis based on certain criteria.
2. List the criteria useful in making a decision.
3. Define the sources of error in trace analysis, and give an explanation of each.
4. Distinguish between repeatability and reproducibility.
5. Explain what is meant by sensitivity and detection limit.
6. Discuss the importance of "speed" on an analysis.
7. List two factors of prime importance under the heading "Scale of Working".
8. List the four steps into which most analytical methods can be broken down.
9. Explain, why choice of containers is so important in trace analysis.
10. List the various methods used for cleaning glassware.
11. Choose the best container for the storage of various types of samples for analysis.
12. List the limits of impurities in reagents used for trace analysis.
13. State the errors that occur due to sampling.
14. Describe how a sample may vary in composition during storage or processing.
15. Define "Fluxes".
16. State, why fluxes are so important in analytical chemistry.
17. List the two categories into which fluxes are classed.

Unit II: ELECTROCHEMISTRY

After completing this section, the student should be able to:

1. Draw an electrochemical cell showing half-cells, salt bridge, and external circuit.

2. Balance redox equations, and show half-reactions and cell diagrams together with the direction of electron flow, anode and cathode.

3. Write down any cell in the shorthand notation.

4. Describe the function of a salt bridge.

5. Show how the table of standard potentials can be developed, using the abitrary standard hydrogen half-cell.

6. Use standard reduction potentials to predict the strength of oxidizing and reducing agents.

7. Obtain the potential of any cell from the standard reduction potentials, and explain why it is independent of the stoichiometry of the redox equation.

8. Use standard reduction potential data to predict the spontaneity of redox reactions.

9. Calculate the equilibrium constant of a cell process, given the cell potential.

10. Calculate the potential of a cell in which the concentrations of electrolytes differ, using the Nernst Equation. Calculate the concentrations within such a cell, given the cell potential.

11. Draw an electrolytic cell showing electrode processes at the anode and cathode, and the direction of charge flow.

12. From the redox of an electrolytic cell, and the current passed in a given time, calculate the amount of chemical reaction produced. For example, you will be able to calculate how much Cl_2 is evolved in the electrolysis of NaCl, given the current and time.

13. Predict the order of precedence of half-reactions in an electrolytic cell at both anode and cathode, using standard reduction potentials.

14. Calculate the theoretical decomposition potential of a solution, given the molar concentration of the solution.

15. a) Determine the amount of substance deposited on a cathode by passage of a current for a specified time.

b) Determine the time required to deposit a certain amount of substance from solution under specified conditions.

c) Determine the quantity of electricity required for deposition or liberation of a certain quantity of substance at an electrode.

16. List five variables that influence the properties of deposits, and give a brief explanation of each.

17. Describe the phenomenon of Polarization.

18. Determine the pH of a solution, so that the concentration of metal can be reduced to a low value before hydrogen evolution occurs.

19. Define, i.e., write out concise definitions for each of the following:

    a) Faraday
    b) Coulomb
    c) ampere
    d) equivalent weight
    e) decomposition potential
    f) back e.m.f.
    g) polarization
    h) overvoltage
    i) IR drop (Ohmic Resistance of solution)
    j) current density
    k) limited cathode potential
    l) depolarizer

20. Draw fully labelled diagrams and compare

    a) the hydrogen
    b) calomel
    c) silver-silver chloride
    d) Weston cell
    e) glass indicator electrodes with respect to:

        i) construction
        ii) chemical make-up
        iii) useful pH range
        iv) interferences, if any
        v) general utility under all operating conditions
        vi) usefulness in solvents other than water

21. Write the half-cell reactions which occur when each of the electrodes in objective No. 1 (a), (b) and (c) are coupled with the glass electrode in part (e).

22. Write the shorthand designation of each of the cells produced in objective No. 2.

23. Derive an equation relating the potential of the glass-calomel electrode system to the pH of the solution.

24. Describe the uses and limitations of the hydrogen electrode.

25. State four properties of a good reference electrode.

26. Give a brief explanation of how the glass electrode measures pH.

27. Explain, what is meant by the term "junction potential", as it applies to pH measurement with the glass electrode.

28. List five errors that affect pH measurements with the glass electrode. Give an explanation of each and how they may be avoided.

29. Define the terms:

    a) residual current
    b) diffusion current
    c) limiting current
    d) half-wave potential
    e) current maxima

30. Give a brief explanation of the principle of polarography.

31. Give four factors regarding the choice of a supporting electrolyte.

32. State the advantages and disadvantages of the dropping mercury electrode.

33. Give a method by which current maxima may be eliminated.

34. State the effect of oxygen on polarographic waves.

Unit III: OPTICAL METHODS

After completing this unit, the student should be able to:

1. Define such terms as frequency, velocity, wavelength, wave number, radiant energy, micron, Angstrom unit, and nanometer.

2. Explain the difference between a photometer and a spectrometer.

3. Elaborate on the difference between emission and absorption spectroscopy.

CHM 231-5 Objectives...5

4. List the five main regions of the electromagnetic spectrum in which the different forms of radiant energy may be grouped or classified.

5. Explain, how the five forms of radiant energy interact with matter.

6. Explain or define such terms as: Monochromatic light, radiant power, transmittance, absorbance, absorbtivity.

7. State Lambert's Law, and Beer's Law.

8. Solve problems based on the Beer-Lambert Laws.

9. Explain how spectra are produced:

    a) by atoms
    b) by molecules

10. Convert units of wavelength in Angstroms to microns, millimicrons and centimeters.

11. Derive an equation for energy in terms of wave numbers, and determine the energy associated with 200 wave number units.

12. State the four basic components required by a piece of apparatus to perform an analysis.

13. List three requirements of a radiation source.

14. List the various devices employed for restricting radiation, and explain how each functions.

15. Explain, what happens to white light, when it makes contact with a diffraction grating.

16. Give an explanation of what happens to light beams of different wavelength and frequency, after contracting the diffraction grating.

17. Explain, in what way the various knobs on the colorimeter affect the light beam which is passed through the sample holder.

18. Explain, what is meant by the term "bandwidth".

19. Explain, why the effective bandwidth of 20 mu is constant over the entire wavelength region.

20. Give a reason why a solution appears red in colour.

21. Review the Beer and Lambert Laws of absorption, and make use of them in calculations.

22. Account for deviations from Beer's Law.

23. Show that A = 2 - log %T, starting with the Beer-Lambert relationship.

$$A = \log \frac{P_o}{P} =$$

24. Draw a schematic diagram of a double-beam optical system as used in U.V. molecular absorption spectroscopy.

25. State the advantages of the double-beam system, over the single beam system.

26. Name, and give brief outline of sources of U.V. and visible radiation with the usable wavelength region of each.

27. List the materials used in sample cells for U.V.-visible radiation, and explain why each is used for a particular region of the electromagnetic spectrum.

28. Name the various types of detectors used for U.V.-visible radiation, and give a brief description of each.

29. State four requirements for the absorption of IR radiation by molecules.

30. Describe the various ways in which two atoms, joined by a chemical bond, may vibrate in a molecule.

31. Name the two most popular sources of IR radiation, and give a brief explanation of each.

32. List the common types of detectors used in IR spectroscopy, and give a brief description of their make-up, and how they function.

33. State the factors which govern choice of solvent for use in IR spectroscopy.

34. Explain, how a slurry or mull, and the KBr pellet method are used to obtain IR absorption spectra on solid samples.

Unit IV: ATOMIC ABSORPTION SPECTROSCOPY

After completing this module, the student should be able to:

1. Explain, why atomic absorption lines are very narrow.

2. a) Give reasons why a hollow cathode is used instead of a hydrogen lamp as the radiation source in A.A. spectrophotometers.

   b) Draw a schematic diagram of a hollow cathode.

3. a) Explain, why modulation is necessary for accurate results in A.A. spectroscopy.

   b) Describe, how modulation is achieved in the Unicam SP-90 A.A. units.

4. Give an explanation of why atomic absorption is not used for qualitative analysis.

5. a) State the causes of chemical interference in A.A. spectroscopy.

   b) Give three examples of substances that cause interference in Objective 5(a).

6. Explain the difference between sensitivity and detection limit.

7. a) List the different types of burners available for use in A.A. spectroscopy.

   b) Give a brief description of each of the burners listed in part (a).

   c) Describe the advantages and disadvantages of each of the burners listed in part (a).

8. Discuss the desirability of using an organic solvent to dissolve a particular sample for A.A. analysis. Consider the effect on solubility, flammability, flow rate, fuel/oxidant ratio, etc.

9. Describe the effect produced by too high, and too low source current upon absorbance readings.

10. Describe the effect of too high a source current, upon the lifetime of a hollow cathode lamp.

11. a) Describe the effect of too high, or too low a slit width setting has upon spectral bandwidth.

    b) Explain, how the slit setting in (a) part affect the absorbance readings of the sample.

12. Explain, how you would optimize the analytical wavelength on the SP-90 A.A. unit.

Describe the effects of variations in the control settings on absorbance by the following parameters.

- Gain
- Scale expansion
- Fuel to oxidant ratio
- Burner positioning
- Aspiration rate (cc/min)

13. Given a set of data, calculate this concentration of metal in percent, mg/liter, micrograms/liter, moles, etc., and be able to convert one set of units to another.

Unit V: GAS CHROMATOGRAPHY

General Objectives:

The student should study the general principles of gas liquid chromatography, and learn to apply those principles to the qualitative and quantitative analysis of organic liquid mixtures.

Specific Objectives:

The student should be able to:

1. Define such terms as:

   a) Stationary phase
   b) mobile phase
   c) migration medium
   d) retention time
   e) resolution
   f) efficiency
   g) origin
   h) base line
   i) dead volume
   j) peak
   k) chromatography

2. Describe the mechanism by which solute species are separated in the chromatographic process.

3. List four properties of a good stationary phase.

4. Select the appropriate stationary phase for any particular analytical problem.

5. Describe the function of the various components that comprise a gas-liquid chromatograph.

6. List five precautions that should be taken in the preparation of a column.

7. Give the sequence of operations necessary in making an injection.

8. Give three precautions to be taken when handling and storing a precision syringe.

9. Choose, which of the following stationary phases you would use to separate a mixture of acetone (b.p. 56^{o}) and methanol (b.p. 65^{o}):

   a) Apiezon L (non-polar)
   b) dinonyl phthalate (semi-polar)
   c) Carbowax 20,000 (polar)

10. Write down the formulae for <u>three</u> of the following:

    a) Number of theoretical plates
    b) Peak Resolution
    c) Partition Coefficient
    d) Peak Area Ratio
    e) Retention time

11. Describe briefly, what happens after a sample has been injected into the apparatus, concluding your account with the production of a visual trace.

12. Describe, which part of a peak gives a measure of efficiency of the column.

13. Write down the two main classes of detectors, and draw a sketch of the type of chromatogram produced by each.

14. a) Name two types of detectors most commonly used.

    b) Explain how each type produces a signal.

15. Give a brief account of the way in which (a) Polarity, and (b) Hydrogen Bonding can help in a chromatographic analysis.

16. In a chromatographic analysis explain:

    a) What effect would a low temperatue have on
       - efficiency
       - retention times

    b) What would be the first choice of oven temperature

    c) Assuming the temperature were kept constant, how would a change in the flow of carrier gas affect the separation of two components

17. Show how you would identify a component quiltatively by g.l.c.

18. Describe, which property of a peak gives a measure of the quantity of that component present in the sample.

19. Interpret a chromatogram, and identify the components.

20. List four requirements of a solid support.

21. State the disadvantages of using hydrogen as a carrier gas.

22. List the factors which affect retention time.