

Lecture Policy

5.310 Laboratory Chemistry has three components:

- 1) the safety lectures and safety quiz
- 2) the experiments carried out in the laboratory and associated quizzes/ reports
- 3) the classroom lectures and two 50 minutes lecture quizzes.

Classroom lectures are given at 10:00 on Tuesday and Thursday mornings as shown in the Lecture Schedule.

No graphing or other calculators with extensive memory will be permitted for the lecture quizzes. Please see the course website for the list of acceptable calculators.

The purpose of the lecture component is to provide the background and theoretical understanding necessary to execute and understand the experiments. Lectures will cover the chemistry involved in the experiments, the theoretical background of various techniques and procedures used in the experiments and the instrumental methods of analysis used throughout the semester. Error analysis and use of the computer to perform the more elaborate calculations will also be covered.

A list of **required readings** is provided in direct support of the lecture material. It will prove most valuable to students to look over the readings prior to the associated lectures. These readings will be found in the course textbook and in books on reserve in the library or in the reading room (4-449).

It is expected that students will attend **all** of the lectures scheduled for the course. Lecture attendance may be considered in the case of borderline grades.

Topics for the Lecture Quizzes:

Lecture Quiz Part I:

Separations:

- Recrystallization
- Distillation (simple, fractional, vacuum)
- Sublimation
- Chromatographic methods
 - Thin Layer chromatography
 - Column chromatography
 - Gas-liquid chromatography

Identification and quantification of material:

- Melting point

Equivalent weight
UV-VIS Spectroscopy
Infrared Spectroscopy
Refractometry
Polarimetry

Lecture Quiz Part II:

Error Analysis

Synthesis and derivatization

Tosyl derivative

Ferrocene and Acetylation

Semicarbazone derivative

Kinetics

Potentiometric Titration

5.310 Required Reading:

Required text: MHSM Mohrig, Hammond, Schatz and Morrill *Techniques in Organic Chemistry* (Freeman ISBN 0-7167-6638-8, 2003)

The following texts are available on reserve in the library (O.R.), in the Reading Room 4-449 (R.R.), in the chemistry stockroom (C.S.). (Note the same topics may be found in other editions)

SWH Skoog West and Holler *Fundamentals of Analytical Chemistry* 7th ed. (6th ed.)

S&L Skoog and Leary, *Principles of Instrumental Analysis* 4th ed

Oxtoby Oxtoby et. al. *Principles of Modern Chemistry* 5th edition

Atkins Atkins and Jones *Chemical Principles: The Quest for Insight* 2nd ed.

SFH J. I. Steinfeld, , J. S. Francisco, and W. L. Hase, *Chemical Kinetics and Dynamics*, 2nd ed. Prentice-Hall, Upper Saddle River, N.J., 1999.

Lecture/Topic	Reading
Lecture #1 Introduction/ Unknown Amino Acid	Lab Manual: Sections I, II, III and Experiment 1 (Unknown Amino Acid) MHSM Ch. 1 Safety pp.2-15 MHSM Ch. 2 Laboratory Glassware pp.16-23 MHSM Ch. 3 Laboratory Notebook pp.23-27 MHSM Ch. 4 Using Handbooks and On-line Databases pp.27-31 MHSM Ch. 5 Measuring Mass and Volume pp.32-40 MHSM Ch. 6.2 Heating ... pp.42-43 (hot plates), pp.46-47 water baths MHSM Ch. 6.3 Cooling methods p.47 SWH Balances pp. 780-791 (pp. 797-806) SWH Handling Chemicals p. 779 (p. 795) optional -JWZ Buchner funnels and filtration Ch. 13 pp. 94-98
Lecture #2 Unknown Amino Acid	MHSM Ch. 8.5 Pasteur filter pipets pp.65-66 MHSM Ch. 9 Recrystallization pp.78-89 MHSM Ch. 10 Melting Points and Melting Point Ranges pp.93-103 MHSM Ch. 3.2 Calculation of Percent Yield pp. 26-27 SWH Burets pp. 798-805 (pp.820-825) (omit sections on pipets for now) Atkins Ch. 10 Acid and Bases Ch. 11 Aqueous Equilibria especially Titrations (p.590-603)
Lecture #3 Error Analysis	SWH Ch. 2 Errors in Chemical Analysis and Ch. 3 Statistical Evaluation of Data OR S&L Appendix 1 Evaluation of Analytical Data
Lecture #4 UV-VIS spectroscopy	<u>Review:</u> Oxtoby Ch 17.1 General Aspects of Molecular Spectroscopy Oxtoby Ch 17.3 Excited Electronic States [Oxtoby Ch 18.5 The Spectrochemical Series...] OR Atkins Ch3.13 Orbitals in Polyatomic Molecules p. 152-156 Major Techniques 2 Ultraviolet and Visible Spectroscopy p 168-169 Ch.16 The Electronic Structures of Complexes p. 889-900 <u>Read:</u> SWH Ch. 22 Spectroscopic Methods of Analysis pp. 497-519 (pp. 508-528) SWH Ch. 24 Molecular Absorption Spectroscopy pp. 557-572 (pp. 561-568) optional SWH Ch. 23 Instruments for Optical Spectroscopy OR S&L Ch. 5 Properties of Electromagnetic... pp.58-61, 72-75 S&L Ch. 7 ...Molecular UV/VIS spectroscopy pp. 123-147 S&L Ch. 8 Application of UV/VIS spectroscopy pp. 150-164 optional S&L Ch. 6 Instruments for Optical Spectroscopy

Lecture #5 Infrared Spectroscopy	MHSM Ch. 18 Infrared Spectroscopy pp. 197-219 Oxtoby Ch 17.2 Vibrations and Rotations of Molecules OR Atkins Major Technique 1 Infrared Spectroscopy p 100-111 optional S&L Ch. 12 Infrared Absorption Spectroscopy
Lecture #6 Ferrocene	Lab Manual: Experiment 2 (Ferrocene) MHSM Ch. 11.4 Fractional distillation pp. 118-121 MHSM Ch 2.2 Greasing ground glass joints pp.18-19 MHSM Ch. 6.2 Heating ... pp.42-43 (heating mantles) MHSM Ch. 7.1 Refluxing a mixture pp.48-50 MHSM Ch. 12 Sublimation pp.133-135 Oxtoby 5.6 Phase Diagrams OR Atkins 8.6 Phase Diagrams p 401-403
Lecture #7 Chromatography	MHSM Ch. 15 Thin-Layer Chromatography pp.153-160, Summary p.162 MHSM Ch. 16 Gas-Liquid Chromatography pp. 163-177 MHSM Ch. 17 Liquid chromatography pp. 178-189 Oxtoby 9.8 Distribution of a Single Species between Immiscible Phases: Extraction and Separation Processes OR Atkins Major Technique 4 Chromatography p 456-459
Lecture #8 Essential Oils	Lab Manual: Experiment 3 (Essential Oils) MHSM Ch. 11 Boiling Points and Distillation pp.104-129 Oxtoby 6.6 Mixtures and Distillation OR Atkins 8.17 Vapor Pressure Lowering p 427-429 Binary Liquid Mixtures p. 438-442 MHSM Ch. 13 Refractometry pp. 136-140 MHSM Ch. 14 Optical Activity and Polarimetry pp. 140-147 Atkins Box 16.2 and Figure 16.28 Optical activity, Plane polarized light p 886-887
Lecture #9, 10 Potentiometric I, II	Lab Manual: Experiment 5 (Potentiometric Titration) Oxtoby Ch. 10 Acid-Base Equilibria-especially 10.6 Acid-Base Titration Curves and 10.7 Polyprotic Acids OR Atkins Aqueous Equilibria especially Polyprotic Titrations p 604-609 Oxtoby Ch. 12 Electrochemistry-especially 12.3 Concentration Effects and the Nernst Equation (pH meters) OR Atkins Ch. 12 Electrochemistry especially 12.10 The Nernst Equation (p. 662-664) 12.11 Ion-Selective electrodes (p.664-665) Additional readings from SWH as listed in the laboratory manual

Lecture #11,12 Kinetics I, II	Lab Manual: Experiment 4 (Kinetics) Definition of the Rate of a Chemical Reaction SFH pp 1 –3 Order and Molecularity of a Reaction SFH pp 3 –6 Second order Reactions SFH pp 8 –9 Reaction Mechanisms SFH pp 17 –18 Effects of Ionic Strength on Reactions Between Ions: SFH pp 133 –136. Pipets and Burets SWH pp. 820-825 Experiment #29 Sime, R. J. <i>Physical Chemistry: Methods, Techniques, and Experiments</i>, Saunders College Publishing: Philadelphia, PE, 1990; pp 628-640 (available in Room 4-449). SWH Pipets and Burets pp. 798-805 (pp.820-825)
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5.310 Required Videos from the MIT Digital Laboratory Techniques Manual

Video	Unknown Amino Acid	Ferrocene	Essential Oils	Kinetics	Potentiometric Titration
1. Volumetric Techniques	Buret only			Volumetric Flask, Pipet	Pipet, Buret
2. Titration	Required				Required
3. TLC basics		Required			
4. TLC advanced					
5. Reaction Work-up I: Extracting, Washing and Drying					
6. Reaction work-up II: Using the Rotovap					
7. Filtration	Required	Required	Required		
8. Sublimation		Required			
9. Recrystallization	One solvent system only				
10. Column Chromatography		Required			
11. Use of balances	Required	Required	Required	Required	Required