

Parker

CHEMISTRY 260

Spring 2001

Book required : "Spectrometric Identification of Organic Compounds," by Silverstein, Bassler, and Morrill

Recommended: any modern instrumental analysis textbook (also on library reserve)

Topics covered from Silverstein and Bassler will include ultraviolet and visible spectroscopy, infrared spectroscopy, NMR spectrometry, and mass spectrometry. Topics covered from the other book will include fluorescence spectroscopy, atomic spectroscopy (emission, absorption, and fluorescence), gas and liquid chromatography, electrochemistry, and other miscellaneous techniques (ESCA, X-ray and electron spectroscopy), thermal analysis, radiochemical analysis, polarimetry, and ESR spectroscopy). Topics covered from handouts will include statistics, data analysis, model fitting, and optimization.

There will be a mid-term exam covering spectroscopy; it will be given the week after spring break. There will be no lab that week; the mid-term exam may be taken anytime that week, including the regular lab time. There will be an exam at the end of the semester that will cover only those topics covered since the mid-term.

There will be a problem set assigned from Silverstein and Bassler; this is to be treated like a take-home exam. The assignment will be given before spring break and will be due Friday, March 20 (the Friday after spring break). You may use only your book(s) and lecture notes for the problem set.

The Honor Code applies to the exams, the problem set, and the lab reports. It is permissible to discuss lab reports prior to writing them up, but the report itself must be your work alone. The lab report for Experiment III will be written jointly by the pair of students who worked together on the experiment. The two students will turn in one report and both students will receive the same grade on the report.

Grading:	Mid-term exam	25 %
	Final exam	25 %
	Problem set	10 %
	Lab	40 %

If you do not take the mid-term exam the week it is assigned, you will receive a zero. If you miss the due date of a lab report or the problem set, you will lose 5 points per day unless you receive permission to turn in the assignment later.

EMORY UNIVERSITY



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LAB SCHEDULE

Jan. 18	Introduction, discussion of scientific writing
Jan. 25	Experiment I (UV-VIS)
Feb. 1	Experiment II (UV-VIS)
Feb. 8	Experiment III (IR-NMR-MS); rough draft due on Exp. I-II (UV-VIS)
Feb. 15	Experiment III (IR-NMR-MS); report due on Exp. I-II (UV-VIS)
Feb. 22	Experiment IV (AA); rough draft due on Exp. III (IR-NMR-MS)
Mar. 1	Experiment V (GC); report due on Exp. III (IR-NMR-MS)
Mar. 8	No lab; report due on Exp. IV (AA)
Mar. 15	Spring break; no lab
Mar. 22	No lab; mid-term exam; problem set due
Mar. 29	Experiment VI (TLC); report due on Exp. V (GC)
Apr. 5	Experiment VII (HPLC)
Apr. 12	Experiment VIII (statistics); report due on Exp. VI-VII (LC)
Apr. 19	No lab; report due on Exp. VIII (statistics)

CHEMISTRY 260 LAB

- 1. Before the lab.** You should come into the lab with some knowledge of the experiment to be performed. This means having read the experiment, performed any preliminary calculations necessary, and reviewed your class notes and the relevant textbook sections.
- 2. During the lab.** The experiment must be performed and completed during the lab period. Work efficiently and make good use of your time. You are expected to be present in the lab until the experiment is finished and to participate in all parts. Most of the glassware and chemicals needed can be obtained from the stockroom. Chemical preparation will be carried out in the prep room; use the hood when necessary. The instruments are expensive and must be handled carefully. If an instrument is broken, this not only involves the expense of repairing it, but may also entail down time before service can be arranged.
- 3. After the lab.** Before leaving the lab, all equipment must be cleaned and put away. Any equipment obtained from the stockroom must be returned.
- 4. Safety.** Proper safety precautions must be observed at all times. This means wearing approved safety glasses, no eating or drinking in the lab or instrument room, wearing proper protective clothing (e.g., no sandals), not wearing contact lenses, and no unauthorized experimentation.
- 5. Reports.** All data collected in lab should be recorded in a notebook. All people in a lab will work on the same experiment and instrument. This means everybody must participate in each part of the procedure -- preparation, instrument operation, and clean-up. The report itself is to be your work alone -- any collaboration is a violation of the Honor Code. (For Experiment III, two students will work together to prepare a single report; both students will receive the same grade.) In most cases, there will be only one original record of the data obtained directly from the instrument. If so, one person should turn in the original with his or her report and the others should mention whose report includes this record. If desired, you may copy or photocopy the data for your own report.

6. Report style. A written report is required for each technique investigated. The report is due at the beginning of the lab as noted on the schedule. The report should include:

- a. Your name and the names of the others who worked on the experiment.
- b. Date of experiment and date of report.
- c. Number and title of experiment.
- d. Technique and theory.
- e. Description of apparatus used.
- f. Brief description of the problem investigated.
- g. Data (normally organized into tables).
- h. Graphs, equations, diagrams, etc., where appropriate.
- i. Results
- j. Discussion and conclusions.

Reports must be written in proper scientific English -- using passive voice and past tense where appropriate. Reports should be word processed and double-spaced; they must be neat and legible with correct spelling, grammar, and punctuation. Normally, you should avoid writing anything in -- use the word processor instead.

7. Grading. Reports will be graded on a 100-point basis. You will be graded on the report itself (style, organization, completeness), your data, your results, and how you obtained your results. The most important aspect, though, is the writing. Some of the grading will, by the nature of the reports, be subjective. If you want, you may turn in a draft of your first two reports for me to look over before you turn in these reports for a grade. Late reports will be penalized 5 points per day (or any fraction thereof) unless you receive permission to turn in a report late. The grading will become stricter during the semester since it is expected that your writing will improve.

Your lab grade will be computed by averaging the report grades and an evaluation of your performance in lab. The evaluation includes some factors as being on time, being prepared, working efficiently, finishing on time, leaving the lab and instrument room in proper condition, participating in all parts of the experiments, handling the instruments properly, and observing the safety rules.

EXPERIMENT I

VISIBLE SPECTROSCOPY

Chemicals: 0.2-M NiCl_2

Apparatus: Shimadzu 1601 spectrophotometer, plastic cuvettes

Procedure:

Prepare a series of dilutions of the nickel solution: 0.1 M, 0.05 M, and 0.025 M.

Select "Spectrum" mode on the instrument. Place the 0.2-M nickel solution in a cuvette in the front cell holder and an empty cuvette in the rear holder. Select absorbance for the Y-axis with a maximum of 1.00. Obtain the spectrum of the nickel solution by scanning from 700 nm to 400 nm. Determine the wavelength of maximum absorbance using the zoom and peak functions.

Select "Photometric" mode on the instrument and go to the wavelength you found. Measure the absorbance of each of the nickel solutions, including the unknown. From the standard solutions, construct a Beer's Law curve of absorbance vs. concentration. If any of the points appears to be too far off the line, discard it.

Using the standard curve, determine the concentration of the unknown nickel solution.

No report is due until you finish Experiment II. In your report for this experiment, you will need to include the spectrum of nickel, the wavelength selected, the Beer's Law plot, all the absorbance readings, and the concentration of the unknown.

EXPERIMENT II

SIMULTANEOUS MULTI-COMPONENT ANALYSIS USING UV SPECTROSCOPY

Chemicals: pheniramine maleate (PAM), 80 $\mu\text{g/mL}$ in 0.010-M HCl
phenylephrine hydrochloride (PEH), 200 $\mu\text{g/mL}$ in 0.010-M HCl
0.010-M HCl
commercial Dristan[®] sample

Apparatus: Shimadzu 1601 spectrophotometer, quartz cuvettes.

Procedure:

Prepare a series of dilutions using 0.010-M HCl: PAM -- 40, 20, 10, and 5 $\mu\text{g/mL}$; PEH -- 100, 50, 25, and 12.5 $\mu\text{g/mL}$. Also, dilute the Dristan[®] sample 1:200 with the 0.010-M HCl (this means 1 mL diluted to 200 mL total volume).

Select "Spectrum" mode on the instrument. Scan each of the standard substances. Select two wavelengths to use where each substance is at or near its maximum.

Select "Photometric" mode on the instrument. Measure the absorbances of all the solutions at each of the two wavelengths (4 solutions X 2 substances X 2 wavelengths); also measure the absorbance of the diluted Dristan[®] solution at each wavelength. For each substance, construct a Beer's Law curve at each wavelength (2 substances X 2 wavelengths); determine the absorptivity (slope) of each curve.

Solve for the concentrations of the unknown solution using the matrix method.

Once you have the concentrations in the diluted Dristan[®], you need to calculate what the concentrations were before dilution and then convert from $\mu\text{g/mL}$ to %:

Multiply by 200 to account for the dilution. Divide by 10^6 to convert from $\mu\text{g/mL}$ to g/mL (since 1 mL of dilute aqueous solution has a mass of 1 g). Multiply by 100 to convert to %.

In the report, include all spectra, Beer's Law curves, and absorbance readings. Show how you solved the equations using matrices. Give the concentrations in the original Dristan[®]. Discuss the theory of uv-visible absorption and the instrument.

EXPERIMENT III

QUALITATIVE ANALYSIS

Chemicals: unknown organic compound
TMS

Apparatus: Varian EM360A NMR spectrometer, NMR tubes
Perkin-Elmer Paragon 500 IR spectrometer, salt plates

Procedure:

Each pair of students will be given 2 pure, liquid, organic unknowns; they will contain only carbon, hydrogen, oxygen, and/or nitrogen.

Fill an NMR tube about 1/3 full with your unknown; add an equal amount of TMS. Obtain the NMR spectrum, including the integration trace. Clean out the NMR tube with acetone.

Place a drop of the sample between two salt plates. Obtain an infrared spectrum. Clean the salt plates with chloroform.

You will be provided with mass spectral data for your unknowns. You will be provided with carbon-13 NMR data for one of your unknowns.

Determine the identity of your unknowns, including structure and proper name. Each peak in the NMR spectra must be assigned and the shift and area explained. The major peaks in the IR and mass spectra must also be assigned. After you have identified the unknown, go back and explain how it would (or would not) be expected to give the observed spectra.

Predict what the C-13 NMR spectrum of your other unknown would look like.

This is a two-week experiment, with the report due after the second week.

In your report, include a discussion of the theory and instrumentation of scanning proton NMR spectrometry and IR spectroscopy. It is not necessary to discuss the instrumentation of C-13 NMR or mass spectrometry since you did not use these instruments, but you should discuss the theory. Be sure that once you have identified your unknowns, you compare the shift, area, and multiplicity of each NMR peak with the predicted values and that you assign the major mass and IR spectral peaks.

Each pair of students will work together to identify their unknowns and to write up the report. Only one report will be accepted from the pair and both students will receive the same grade.

EXPERIMENT IV

ATOMIC ABSORPTION SPECTROSCOPY

Chemicals: lead nitrate

Apparatus: Buck 200A atomic absorption spectrometer

Procedure:

Prepare a solution of 20-ppm lead, if not already prepared. From this solution, prepare standard solutions of 1, 2, 5, and 10 ppm lead.

Prepare standard addition solutions. Mix equal quantities of your unknown with each of the standards and deionized water (0 ppm Pb), giving 5 standard addition solutions (e.g., one such solution might be 1 mL of unknown + 1 mL of 10 ppm Pb).

Locate the lead wavelength (283.3 nm). Set zero while aspirating deionized water. Aspirate the standards, unknown, and standard addition solutions and record the absorbances.

Construct a standard curve and determine the concentration of the unknown. Also construct a plot of the standard addition solutions (absorbance vs. ppm added) and determine the concentration of the unknown from this graph.

In your report, include the 2 plots, absorbances, and the concentration of the unknown from the 2 methods. Discuss the theory and instrumentation.

BUCK 200A ATOMIC ABSORPTION SPECTROMETER

1. Turn the instrument on and adjust the lamp current to be around 5 mA.
2. If necessary, tune in the wavelength by adjusting the wavelength selector control for maximum deflection on the energy meter.
3. In general, the lamp will not need to be aligned and the burner position will not need to be adjusted. If this is not the case, see the manual. There is a slit width control at the back of the instrument. The chart inside the lamp compartment gives the setting for each element.
4. Make sure the loop in the drain line is filled with water.
5. Turn the oxidant selector to air. Turn on the air tank main valve. If necessary, adjust the regulator for a pressure around 40 psi. The oxidant flow control on the AA should be set at around 5-6.
6. **MAKE ABSOLUTELY CERTAIN THAT AIR IS FLOWING THROUGH THE BURNER BEFORE TURNING ON THE FUEL!** Turn on the acetylene at the tank and turn on the fuel switch on the spectrometer. If necessary, adjust the regulator for a pressure of 5 psi. The fuel flow control on the AA should be approximately 5.
7. Ignite the flame using the lighter. If necessary, adjust the air and/or acetylene flow controls.
8. Select Auto-Zero mode. Aspirate deionized water. Press the zero button and continue aspirating the blank until the display reads zero. Aspirate the solutions and read the absorbance from the meter. **NOTE:** It is often better to switch to integration mode. This integrates the signal for 7 seconds, giving less noise in the reading. After switching to integration mode, aspirate a solution and press the read button. In 7 seconds, the display will show the reading for that solution. That reading will be held until you press the read button again.
9. To shut down the AA, first turn off the acetylene with the fuel switch on the spectrometer. Turn off the acetylene tank valve. Open the fuel switch until both gauges on the acetylene tank read 0, and then close the fuel switch. Turn off the air tank valve. When these gauges read 0, turn the oxidant selector to the center position.
10. Turn off the power.