CHEMISTRY 440 INSTRUMENTAL METHODS OF ANALYSIS

Southern Connecticut State University

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Texts: Textbook *Principles of Instrumental Analysis, Fifth Edition,* D.A.Skoog, F.J.Holler, T.A.Nieman, Saunders College Publishing (1998)
Laboratory Manual *Chemistry Experiments for Instrumental Methods*, D.T. Sawyer, W.R.Heineman, J.M. Beebe,Wiley (1984)

Introduction: It's the Instrument, stupid!
On December 10, 1996, Robert F. Curl Jr., Sir Harold W. Kroto, and Richard E. Smalley were awarded the 1996 Nobel Prize in Chemistry for their discovery of C_{60}--fancifully named buckminsterfullerene. In his Nobel lecture, Smalley flatly stated: " The INSTRUMENT discovered the fullerenes." He was referring to the laser vaporization, supersonic jet technique that Smalley had invented. Since the time of Galileo and his telescope, new devices, new instrumentation in the hands of the prepared observer, have generated new insights into nature that simply would not have been possible in the absence of the technology.

Course Overview: The course is an introduction to the theoretical background and practical use of modern instruments in the analytical laboratory. After a brief introduction to the use of instrumentation for chemical analysis, the properties of electromagnetic radiation will be discussed. Spectroscopic techniques that will be covered include: Ultraviolet/Visible spectroscopy, Fluorescence and Phosphorescence, Atomic Absorption (Flame and Graphite Furnace) Spectroscopy, Infrared Spectroscopy and Nuclear Magnetic Resonance Spectroscopy. After an introduction to Chromatographic separations, Gas Chromatography and High-Performance Liquid Chromatography will be discussed. Laboratory experiments will demonstrate the scientific method and illustrate basic concepts presented in the lecture portion of the course.

Final Course Evaluation:

Mid-Term Examination	30 %
Final Examination	30 %
Laboratory Reports	40 %

COURSE OUTLINE

I Introduction

Chapter 1: Introduction (*suggested problem 1- 9*)
Chapter 5: Signals and Noise (*suggested problem 5-7*)

II Spectroscopic Techniques

Chapter 6: An Introduction to Spectrometric Methods
(*suggested problems 6-2,6-3,6-4,6-11, 6-12*)
Chapter 7: Components of Optical Instruments (*suggested problem 7-5*)
Chapter 13: An Introduction to Ultraviolet/Visible Molecular Absorption
(*suggested problems 13-1,13-2, 13-5, 13-7, 13-9*)
Chapter 14: Applications of Ultraviolet/Visible Molecular Absorption
Spectrometry (*suggested problems 14-5, 14-6*)

MID-TERM EXAMINATION Thursday MARCH 16

Chapter 16: An Introduction to Infrared Spectrometry
(*suggested problems 16-1, 16-3, 16-9*)
Chapter 17: Applications of Infrared Spectrometry
(*suggested problems 17-2, 17-3, 17-9*)
Chapter 8: An Introduction to Optical Atomic Spectrometry
(*suggested problems 8-8, 8-9*)
Chapter 9: Atomic Absorption and Atomic Fluorescence Spectrometry
(*suggested problems 9-16, 9-20*)
Chapter 19: Nuclear Magnetic Resonance Spectroscopy
(*suggested problems 19-6, 19-7, 19-9, 19-26, 19-27, 19-28, 19-29, 19-30, 19-31, 19-32*)

III Chromatographic Techniques

Chapter 26: An Introduction to Chromatographic Separations
(*suggested problems 26-12, 26-13, 26-14*)
Chapter 27: Gas Chromatography (*if time allows*)
Chapter 28: High-Performance Liquid Chromatography (*if time allows*)

FINAL EXAMINATION Thursday 8 – 10 a.m. MAY 18

The examination questions will be taken directly from the material covered in class. The mid-term examination and the final examination will be two hours long.

LABORATORY EXPERIMENTS

The Laboratory Notebook

As observations are made during a chemical process or as numerical data are recorded and processed, a complete up-to-date record must be made of the observations and data. For these purposes, a laboratory notebook will be kept throughout the semester. The notebook must be permanently bound to prevent the addition or removal of pages. All pages must be numbered consecutively from first page to last page. Your name must be written on the cover of the laboratory notebook. The first few pages of the notebook are to be used as a Table of Contents. As you begin an experiment, make an entry in this table that will permit you to locate information easily e.g. Experiment Title, page number.

Only the right-hand pages of the notebook are to be used for information. Start each experiment on a new, right-hand page. The experiment should include the Title of the experiment, Date, Partner's Name, all Data and Observations that arise during the experiment (including proper units and the appropriate number of significant figures) and all calculations that are required.
The left-hand pages of the notebook are used primarily for quick arithmetic calculations and for taking notes during the instructor's pre-lab instruction, or for recording any special instructions about the experiment.
Mistakes do happen, especially while recording data in a lab notebook. No one expects your experiment to be perfect. If you make a mistake, draw a single line through it and write in the correct value. Never erase data or cover it with correction fluid. Also, never tear pages out of a lab notebook. The lab notebook is a permanent record of what you did in the laboratory, warts and all.
Make sure that the laboratory notebook is kept up-to-date.

The Laboratory Report

The laboratory report is a formal report of what you did in the laboratory. Therefore it should be written in the past tense using the passive voice. It should include only those parts of the experiment actually performed, including any in-lab modifications. **The formal written laboratory report is due two (2) weeks after the experiment has been completed. Late laboratory reports will have five (5) points taken off for each day late , the first time. The second time a report is late, ten (10) points a day will be taken off, etc.** If the instructor is not around to accept a late laboratory report, the student should find another instructor or the department secretary to date and sign the report. **Do not slide late laboratory reports under my office door!**

The laboratory report should include all of the following parts clearly labeled:

Title of Experiment
Name of Experiment and Laboratory Partner
Date Submitted
The Introduction
Experimental Procedure
The Data and the Results
The Discussion of the results
The Conclusion
References

THE INTRODUCTION (20 points)

This section should be a description of the purpose of the experiment and the method used. The introduction contains information required for a complete understanding of the experiment, i.e., historical and theoretical material that is relevant to the experiment. The student is expected to consult the course textbooks, use the library and any other sources of information. The use of any basic equations should be justified here.

THE EXPERIMENTAL PROCEDURE (20 points)

This section should report the main details of the experimental procedure, including the number of runs made and the conditions under which they were carried out (concentration, temperature, etc.) A detailed description of the instrumental method should be given, including a schematic diagram of the instrument used.

THE DATA AND THE RESULTS (20 points)

The results should be presented in both tabular and graphic form. Both the basic information (raw data) and the calculated results should be included in as concise a form as possible. Include a sample calculation (where several sets of data have been obtained, only show a sample calculation for one set). All tables should be titled and all graphs plotted on good quality graph paper. The graphs should be titled with both coordinates labeled. The coordinates should be chosen so that the plotted curve uses most of the graph paper. The curve should be smooth and, therefore, will probably not go through all of the points. Curves should be drawn with a ruler or a French curve. Use the method of Least Squares to determine the slope and intercept of all linear plots. Accompanying the tables and graphs should be text that explains their significance and presents any conclusions that can logically be drawn. Any calculation not explained in the introduction should be explained here.

THE DISCUSSION OF THE RESULTS (20 points)

This is one of the most important sections of the laboratory report. Here the student provides an interpretation of the information just presented in the Results section. Students should draw from the theoretical material presented in the Introduction and relate this to the results obtained. This section should offer possible explanations for the results obtained. Include a discussion of the possible sources of error and their probable magnitude. The number of significant figures in calculated quantities should be consistent with the estimated error of your measurements. Whenever possible, compare your results with theoretical or experimental values from the literature.

THE CONCLUSION (20 points)

The conclusion section should summarize the final results, state the final answer, address the original purpose of the experiment, and briefly indicate whether or not the purpose of the experiment was achieved.

REFERENCES

If you are unsure as to whether or not a reference is required, use the "*eye test*". Where are your eyes while you are writing? If they are looking at another source while you are writing then either a quote or reference citation is required. If you are writing "off the top of your head', then no reference is needed. ***Plagiarism in any form will not be tolerated***.

Any book or article referred to should be indicated at the proper point in the text (a number in parenthesis) and included in a table of References Cited at the end of the report, using proper presentation of literature citations according to the Style Guide of the American Chemical Society.

For example, for a textbook:

> Skoog, D.A., Holler, F. J., Nieman, T. A., *Principles of Instrumental Analysis*, Fifth Edition, Saunders College Publishing, Philadelphia (1998) **pages cited**

For a journal article,

> Snyder, R. and Testa, A. C., *J. Phys. Chem*., **83** , 3041 (1979)

For complete information on references and any other formatting, consult the *Handbook for Authors of Papers in the Journals of the American Chemical Society*, American Chemical Society Publications, Washington, D. C., (1967) or the *ACS Style Guide*.