

CH 424 Instrumental Analysis Spring 2005

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Text: Principles of Instrumental Analysis, 5th edition. Skoog, Holler & Nieman. Saunders, 1998.

Preliminary Course Outline

Instrumental analysis should be the most important course in the education of a chemist. Unfortunately, it is driven by technology, expensive technology, that is. Instruments that were state of the art only a few years ago may take on the role of dinosaurs. As a consequence, we are limited in this course by the available resources in terms of equipment.

At this time, your instructor is still in the process of putting together a series of experiments based on available instrumentation. The operative term is available. Instruments purchased for instructional purposes are a slim picking. We will thus expand and make use of the research type equipment that is scattered throughout the department and around campus. I am still begging my colleagues for time and space. The lecture and lab should go hand in hand. It will take some time to finalize and coordinate both lecture topics and lab experiments. These activities will be delineated in the final course outline.

CH 424 carries a UA writing (W) designation. As a consequence, emphasis will be placed on report writing. In addition, a term paper and its presentation will be required.

Lecture

Handouts will be provided whenever possible. Your instructor will follow the book closely and make frequent references to specific diagrams, plots, etc. As with many instructional resources, our book is far too comprehensive. It is not possible to cover all topics and choices have to be made as to what aspects to emphasize in each chapter and which chapters to eliminate altogether. You will be responsible only for material covered in class. It is thus wise to take note of what is being covered (a highlighter is most useful). You should spend your time in the classroom thinking (and asking questions), not taking notes. Details to follow.

Laboratory

We will be forced to work as one group most of the time. If possible, we will split up in groups of 2 or 3. If possible, real unknowns will be provided for individual students. Each student thus may have individual data to report. In cases where data are acquired by the entire group, data need to be shared. The TA or instructor will make copies of the data point entries of each student and hand these out at the end of the lab. We don't want to use loose sheets. You are asked to buy a ring binder to keep these extra data in some organized form. Even though laboratory results will have to be shared in most cases, it is absolutely essential that each student writes his/her own lab report. Each student is reminded to adhere to the University of Alabama honor code. Laboratory notebooks are to be used to record data (see next section). At the end of each lab period, the TA will have a look at your data and add his initials.

The written report for each laboratory is due at the beginning of the laboratory in the following week. Penalty points will be assessed for lateness (see grading). The instructor will try

to hand out the next lab assignment on the Tuesday lecture preceding the lab. You should read the individual lab handout before coming to the laboratory. There will be only a brief introduction the experiment to be carried out. This presentation will either be done by one of you or by the host of the facility of the research instrumentation we will be using. For the introduction to be done in our laboratory, we will follow the list alphabetically, i.e. Brandon will be first, followed by Timothy..., etc. I will announce when presentations need to be made so that you can prepare accordingly.

Sequence of presenters:

1. Easley, Robert Paul
2. Evans, Brandon W.
3. Kucharski, Timothy John
4. Spruell, Jason Michael
5. Summers, Rachel Joy
6. Whatley, Kelley Suzann
7. Wilson, Jason Brett

Individual Laboratory Notebook

In each experiment, numerical data are recorded and processed. We will try to adhere to standards practiced in industry. A complete up-to-date record must be made of the observations and data. For these purposes, each student needs to keep a laboratory notebook. The notebook must be permanently bound. 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, Etc.

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, and all Data and Observations that arise during the experiment (including proper units and the appropriate number of significant figures) The left-hand pages of the notebook are used primarily taking notes during the instructor's or student's pre-lab introduction.

If you make a mistake, draw a line through it and write in the correct value. Never erase data or cover it with correction fluid. The lab notebook is a permanent record of what you did in the laboratory. Completeness is required. Neatness is only of secondary importance.

Laboratory Report, to be handed in

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

Title of Experiment

Student Name

Name of Experiment

Date Submitted

It includes the following elements:

Introduction

Experimental Procedure

Results and Discussion

Conclusions

References, as needed

Introduction

This section describes the purpose of the experiment and the method used. The introduction contains information required for a complete understanding of the experiment. The student is expected to consult the course textbook and add other sources of information as needed. Only basic equations should be used. Normal length: 100 to 300 words.

Experimental Procedure

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 (instrument used, concentrations, temperature, etc.) A short description of the instrumental method can be given, including a schematic diagram of the instrument used. Do not use this section to elaborate on how an instrument works. Normal length: 100 to 200 words.

Results and Discussion

The results should be presented in either tabular or graphic form (sometimes both). Raw data and calculated results should be included in a concise form. A sample calculation should be included, showing only one sample calculation for one set. The graphs should be titled with both coordinates labeled. The curve should be smooth and, therefore, will probably not go through all of the points. Curves should be drawn. You may want to use one of the commercially available regression programs, if available. Accompanying the tables and graphs should be the text that explains their significance and presents conclusions. Normal length: variable.

The discussion is the most important section of the laboratory report. Provide an interpretation of the information just presented in the Results section. This section should offer possible explanations for the results obtained, good or bad. Possible sources of error and their probable magnitude need to be included. A brief error analysis should be conducted which may include propagation of error. The number of significant figures should be consistent with the estimated error. When possible, compare your results with theoretical or experimental values from the literature. Normal length: variable, but not more than 400 words.

Conclusion

The conclusion section should state the final answer and briefly indicate whether or not the purpose of the experiment was achieved. 1 - 2 sentences.

References

Books or articles referred should be included in a bibliography at the end of the report.

Term Paper: Everyone will be assigned a topic on which to write a term paper. We will hold a drawing on the topics chosen by the instructor. The paper must be at least 10 pages in length (typed double space), and contain a minimum of 15 references, two-third of which must be from journal articles (not books) published within the last four years. The form of the paper and references must adhere to the requirements of the journal Analytical Chemistry review articles. Since the subject of each paper is very specific, a thorough search of the literature is required. References should be current. This sounds like a tall order. It is not. You will have SciFinder at your disposal.

It is suggested to do the literature search as early as possible. The paper is due on the day of the final (Thursday, 5/5/2004 at 8:00 a.m. style). You are encouraged to hand in an outline of the paper 1 to 2 weeks before the due date. Points will be deducted for typographical errors and incomplete sentences. It is obviously to your advantage to read over your final paper careful and to make good use of your word processor's spell checker. Errors of content, use of footnotes, grammar, etc. will affect the overall grade as will poor readability. If time permits I will, at your request, read and comment on the content of the draft prior to final submission.

Please note that some of the topics are instrument oriented whereas others address applications. In either case, it is necessary to focus primarily on instrumentation.

The topics represent cutting edge issues for which adequate analytical instrumentation and methodology may not yet be available. If there are problem, state what they are and how you can get around some of the issues. The term paper gives you a chance to be creative and I think outside of the box! The approximate format is, as follows:

Abstract: 3-5 sentences summarizing what the article is all about.

Theoretical Basis: Review the literature and discuss the fundamental science on which your paper is based. (About 1/3 of the paper).

Implementation and Discussion: Spend some time to discuss the instrumentation to be used and limitations thereof. Think about sampling, sample preparation.

References: The references should be introduced at the appropriate place in the text but only summarized at the end.

Please note that Theoretical Basic and Implementation and Discussion sections take the place of Introduction and Results and Discussion in conventional papers.

Approximate Lecture and Lab Schedule

Week	Chapter	Topic	Lab Experiment
1	1 I Appendix	Introduction Date Treatment	
2	5	Signal and Noise	Calibration of Glassware Detector Optimization
3	6	Introduction Spectrometry	Fe by UV/VIS
4	7 13	Optical Instruments Molecular Spectroscopy	Co and Cr by UV/VIS
5	14 15	Application of UV/VIS Luminescence Spectrometer	Quantitative IR
6	16 17, 18	Infrared Spectrometry Application of IR and Raman	Quantitative IR
7	19	Nuclear Magnetic Resonance	Fluorescence
8	20	Mass Spectrometry	NMR
9	8 9	Atomic Spectrometry Atomic Absorption Spectrometry	MS
10	10 11	Atomic Emission Spectrometry Application of Atomic Spectroscopy	ICP
11	26 27	Chromatography Gas Chromatography	AE
12	28	High Pressure Liquid Chromatography	GC
13		SPRING BREAK	
14	12 21	X-Ray Spectroscopy Surface Spectroscopy	GC/MS
15	22-25	Electrochemistry	HPLC
16	31	Thermal Methods	Ion Chromatography
17	32	Radiochemistry	Electrochemistry

Grading

Instead of conventional 1 hour exams, 6 quizzes, each 20-30 min in length, will be given. The laboratory grade will have a results and a writing component. The term paper will also be graded on both style (writing) and content.

Quizzes	5 x 12	60 (drop lowest score)
Laboratory	15 x (1/2 + 2)	37 1/2 (drop lowest score)
Term paper	15	12 1/2

Another 3 points can be added for classroom performance and interaction.