

Introductory Details

Preparation for the Laboratory

The laboratory part of this course is intended to give the student exposure to as many instrumental methods as possible during 60 hours of laboratory work. It is absolutely essential that the student be prepared for a given experiment by reading the laboratory outline before starting that laboratory work. Failure to do this will undoubtedly result in your failing to complete the laboratory, and, therefore, receive a poor grade. The laboratory outlines have been made as detailed as possible with respect to operation of the instruments, and, in theory, you should be able to proceed without the assistance of a TA if you are properly prepared.

Laboratory Work

Experiments have been set up for students working in small groups. Be sure to have a work plan that makes efficient use of your time. Lengthy sample preparation procedures should be started early in the six hours assigned to a given experiment. All students are expected to record laboratory observations and any numerical data in a hard-bound laboratory notebook. Each page should be titled, dated and signed. Results that are obtained from recorders, such as spectra, current-voltage curves, and chromatograms, do not have to be run twice for each student operating in a team. However, you may want to attach these results to your report, so photocopying may be necessary. If it is not too time-consuming, run the experiment twice so that you can check the reproducibility of your data. At the end of each lab, the TA must be asked to initial the pages of your lab book on which new data have been recorded.

The Laboratory Report

One of the most important skills you should be developing in this course is writing laboratory reports. As a student, the preparation of a well-written report requires careful consideration of the content of the report, hopefully leading to fuller understanding of the concepts involved in the experiment. In your future career, the ability to write clear, concise and well-organized reports will ensure success.

The reports submitted should be written using the standard format described below. Normally, they should be typewritten and double-spaced. The required format is:

- *Title page:* Title of experiment, course number, author, and date work was carried out, name of supervising TA, date report is submitted.
- *Introduction:* A statement of the purpose of the experiment; this requires 2 or 3 paragraphs at least.
- *Theory:* A brief paragraph describing the theoretical background for the analytical technique used; this section should contain a reference to the appropriate pages of the textbook, or other source material if you prefer.
- *Experimental:* A schematic or a box diagram is encouraged. The major pieces of equipment used should be listed. Also list standard solutions and reagents used to calibrate the instrument, and the nature and origin of any unknown samples analyzed. It is NOT necessary to copy experimental instructions from the laboratory manual, but you should refer to it here.
- *Data:* Summarize your data in numbered tables and graphs. Remember to label both axes on a graph, and to give it a title. Graphs should be referred to as Fig. 1, Fig. 2, etc., and tables as Table I, Table II, etc.
- *Results:* Present the results of your experiment based on the above data, showing all steps in calculations needed to reach a numerical result. Include an analysis of errors. The data should be analyzed to determine the amount of the unknown in the original sample.
- *Discussion:* Comment on the results. Explain any lack of success or limitations in the experiment. Discuss possible improvements if you see any.

References: List any references referred to in the report. This list should always include the textbook and the laboratory manual. In the text of the report, the reference is cited as a numbered footnote in parentheses: for example, "The drying procedure followed that outlined by Vogel (3)." 3. A.I. Vogel, Textbook of Quantitative Inorganic Analysis. Third Edition, Longmans, London (1961), p. 221.

Note: These report guidelines are not the same as those presented on the lab evaluation form.

Lab Evaluation Form

The [lab evaluation form](#) **must** be printed out and attached to your report between the Title page and the Introduction. This will be the criterion used for grading your lab report, although lab-specific questions will be introduced as necessary.

Here are a few more pointers

BE BRIEF AND CONCISE! This is one of the most difficult aspects of good writing to master. Read over your first draft and be ruthless in chopping out superfluous words, phrases, or sentences. Learn to be economical in expressing yourself. The total length of the lab report should be less than 15 pages. Short is sweet!

WATCH YOUR GRAMMAR! You may be a brilliant scientist in the lab, but your credibility will be diminished if you can't communicate your results. Science has two parts: doing novel and reproducible experiments, and being able to tell other people what you did!

WATCH YOUR SPELLING! You will not be marked down for the occasional typographical error, but a report full of spelling errors will be penalized. Take the time to proofread the draft of your text at least once.

Some particular comments on writing a scientific report:

- Avoid the use of personal pronouns, especially "I" and "we."
- Use the past passive tense in the Experimental and Results sections.
- All tables should be numbered and titled. The table headings should include the units.
- All figures should be numbered and titled. If the figure is a graph, the axes should be clearly labelled, including the units. If more than one set of data is included on the graph, use different symbols and a legend to identify them.
- Tables and figures should be presented on different pages, not included with the text. More than one table or figure can be placed on a page, however. Be sure to refer to any table or figure in the text, e.g. "The current and potential values from which E and n were calculated are reported in Table 2, and plotted in Figure 3."
- All equations presented in the text should be numbered and all symbols defined, as shown in the example below:

"The diffusion-limited current, i_d , is related to the bulk analyte concentration, c_a , by the following expression:

$$i_d = nFSD_a^{1/2}c_a/(\pi\Delta t)^{1/2} \quad (1)$$

where n is the number of electrons transferred, F is the Faraday constant, S is the electrode area, D_a is the diffusion coefficient of the analyte ion, and t is the time during which the current is sampled."

Remember, the all-important criteria in determining the quality of the report are that it be clear, concise, and provide enough information so that anyone else with your level of experience could repeat your work without consulting you. Students without experience in writing reports are often concerned about the length of the report and the time they take to write it. The first report may take longer, but with experience you will find that a good report can be written in a few hours. The most important aspect is that it presents the data and their analysis clearly and concisely. This can usually be done in 4 or 5 pages. If you have access to a word processor, you should use it to prepare reports. Editing is then very easy, and a neat copy is easily produced.

Finally, you are strongly advised to consult with the TA grading a given report before writing it. The TA can show you the important points to be emphasized in evaluating your data and will discuss his/her grading scheme. A list of TAs with their grading assignments and office hours is posted in the lab.

Your laboratory report is due one week after the lab is complete. The late hand-in penalty is **10 points (10%) per day!**

Lab Report Outline

(See pages 1-3 of Laboratory Manual)

I. Title page.

Include the title of experiment, course number, your name, and partners name(s), date of experiment performed, date lab report submitted and supervising TA.

II. Introduction.

Brief statement of purpose, which should indicate what was analyzed and the technique used. Limit to three to five sentences.

III. Theory

Describe the general theory used in the experiment. You may find that sections in your text and other textbooks could provide useful information in explaining the theory. Reference your sources of information.

IV. Experimental

Write down the major equipment used and list out the experimental conditions. DO NOT just reference the manual in terms of set up and procedure for this experiment. Plots will include copies of the chromatogram and mass spectrum of each standard and each unknown mixture.

V. Results & Discussion

Tables will include data (retention times and mass spectral information) obtained for standards used and labeled unknowns. From this information, you should be able to determine the compounds present in your unknowns.

Describe and explain your observations based on the experimental results. Also include possible structures of the standards and unknowns used in the experiment, based on your observations. Identify what your unknown mixtures contain. In your discussion, you should be able to determine the origin of the major peaks in the mass spectra. Use the references previously mentioned as an aid.

Explain possible sources of error and possible solutions to correct those problems.

VI. Conclusion

VII. References

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