

PHYSICAL CHEMISTRY LABORATORY

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The document you are reading gives you the general guidelines that are to be followed in the physical chemistry laboratory and the format for your lab notebook.

1 General Guidelines

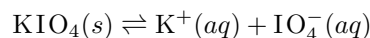
1. First and foremost, ATTENDANCE IS MANDATORY. Absence is only permitted for medical reasons, which has to be supported by a certificate. You will be docked 25% of the total marks for every experiment that you are absent. No repeat turn will be granted for an experiment missed due to absence.
2. You are expected to be in the lab ON TIME.
3. Wearing a lab coat is MANDATORY.
4. If time permits, lab notebooks should be turned in before you leave the lab. Otherwise they are due on the following Monday before 5 pm (except during Minors) in the lab.
5. An important part of the laboratory is the oral examination on the objective, the theoretical basis, the experimental method, the expected results or in other words anything under the sun. You will be examined at random on any experiment that you may have performed or are performing upto that point. COME PREPARED. Reading the handout is not considered adequate. Some background reading from a physical chemistry textbook is generally expected.
6. You will be graded for your pre-lab preparation, your effort, your laboratory skills, and your laboratory report.

2 Laboratory Notebook Format

Use a folder as the lab notebook (NO NOTEBOOKS, PLEASE). On completion of an experiment add the pages to the folder. The format for the lab notebook is given below.

1. Title and Date
2. Introduction: The introduction contains three important pieces of information. (1) The objective of the experiment or in other words "What is being investigated in this experiment?" (2) The theoretical basis of the experiment and (3) a description of the method that will be used to achieve this objective. Please do not copy this from the lab manual or from your predecessor. Write in your own words.

Here is an example. In this experiment we study the solubility of KIO_4 , a sparingly soluble salt and the effect of the ionic strength on it. The equilibrium under consideration is



for which the equilibrium constant may be written as

$$K = \frac{a_{\text{K}^+} a_{\text{IO}_4^-}}{a_{\text{KIO}_4}}$$

where a stands for activity. The activities are related to the concentrations as $a = \gamma c$ and the γ 's in turn are related to the ionic strength, at least in some limiting situations, by the relation

$$\log \gamma_{\pm} = -Az_+z_-\sqrt{I}.$$

The experiment involves measuring the concentration of IO_4^- in 5 saturated solutions of KIO_4 with varying amounts of an inert salt, NaNO_3 , which serves to change the ionic strength. The IO_4^- is estimated in each case by an iodometric titration against $\text{Na}_2\text{S}_2\text{O}_3$ after liberating the iodine present quantitatively by the addition of potassium iodide in acid medium.

THESE TWO SECTIONS ARE TO BE COMPLETED BEFORE YOU COME TO CLASS.

3. Data and Observations: This section will be written as you perform the experiment; the data you collect and the observations you make are recorded here. If necessary, make a flow chart (or any other device to jog your memory) of the procedure to be followed. View this as an opportunity to organize your thoughts and not to reproduce the procedure in the handout. Perhaps the easiest way to collect your data and observations is to paraphrase the procedure as you go through the steps and then enter your data and observations for that step. Simply enter a one- or two-line statement to describe the operation and then your observations for that step. To do this you must have read the procedure more than once, discussed with people who have done the experiment before you and most importantly, made a mental picture of how you intend to proceed and the types of information you will be collecting. Most importantly, for every step in the procedure answer for yourself the question "Why am I doing what I am doing?" We discourage you from looking at the procedure in the handout during the course of the experiment. This is a sure sign that you have not done the pre-lab preparation.

Write all the observations and data for the experiment in this section. Data and observations will only be recorded IN PEN. As you record your data and observations this section may become messy and unorganized which is to be expected. If you make a mistake score it off and make a note of the error made. Avoid overwriting. No fairing of your rough data and observations is allowed or required. RECORDING DATA OR OBSERVATIONS ON SCRAPS OF PAPER OR BACKS OF NOTEBOOK WILL NOT BE TOLERATED.

Here is an example. Procedure: 1. Prepare 50 mL of a stock solution of 100 ppm quinine bisulphate in 4 M sulfuric acid. 2. Make 10 solutions, each of 10 mL, of quinine bisulphate with concentrations varying from 1 to 10 ppm. 3. Measure the fluorescence intensities with the 10 ppm solution as standard. 4. Measure the decrease in fluorescence intensity for the 10 ppm solution on the addition of increasing volume of 0.1 M NaCl

Preparation of 50 mL of 4 M sulphuric acid: Provided conc. sulphuric acid. Take — mL and make it up to 50 mL.

Preparation of 100 ppm quinine sulfate: Done in two steps. Prepare 1000 ppm and dilute. Weighed — mg and diluted to 100 mL.

And so it goes.

THIS SECTION IS DONE DURING THE LAB PERIOD.

4. Calculations and Results: If your data and observation has become cluttered or disorganized, this is the place to clean it up and present it with clarity. Use tables, if and when required.

Show all set-ups for each type of calculation: be explicit. If you have to perform the same calculation more than once you do not have to write the set-up for each one, but it should be clear as to which set-up correlates to which calculation.

Here is an example. The solubility experiment discussed earlier requires you to calculate I , the ionic strength. The ionic strength was calculated from the expression $I = \sum_i c_i z_i^2$ where the contributing species are K^+ , IO_4^- , Na^+ , NO_3^- . The first two are known from the estimation and the last two from the amount of added salt.

The activity coefficient was calculated using the expression ...

Be sure to include the final results of all your calculation and to highlight the final answers in some fashion. Support them with the required graphs. Use your own or a spreadsheet program (Microsoft Excel for example) to perform the least squares fits.

Finally, state your results to the question you set out to answer in the objective.

THIS SECTION IS TO BE COMPLETED EITHER IN THE LABORATORY, IF THE EXPERIMENT FINISHES EARLY, OR AT HOME.

5. Discussion and Conclusions: The discussion is used to explain your results you presented in the previous section. Ask yourself provocative questions like: Do your results make sense? Is there some data missing? Did your results conform to your expectations? If the data from an experiment is straightforward and self-explanatory this section may be brief. However, most of the time you will have to explain to the reader why you obtained a particular result, especially if your result is different from that expected. Discuss your results in the context of what you stated in the Introduction.

Explain any sources of error and how they affect your results. Discuss any improvements that may be made to the experiment or procedural errors in the laboratory manual.

Answer any during-lab questions that may have been raised by the instructor.

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