Laboratory Procedures
Advanced Placement Chemistry, Edmund Burke School, 2014-2015

Chemistry is an experimental science, and an advanced chemistry course would be incomplete without a major laboratory component. In this course, we will carry out complex experiments that require skillful lab techniques, careful observations, precise quantitative measurements, and moderately detailed mathematical analyses. The laboratory part of this class is designed to emulate a college chemistry laboratory course, in order to prepare you for the advanced level of laboratory techniques your future studies will require. In addition, the experiments will help you master the underlying theory and problem-solving methods that are relevant to each particular topic in the class.

All documentation of your work in the laboratory will take place in your **laboratory notebook**, in which you will take notes, record data as you work, perform calculations, and complete your final laboratory reports. If you wish to obtain college credit for Advanced Placement work in chemistry, your college or university will require evidence of your skill and achievements in the laboratory; your notebook may provide such documentation.

I. Setting up your laboratory notebook:

Your laboratory notebook, a permanent record of everything you do in the lab, is permanently-bound, with carbonless duplicate sets of pages that can be removed and submitted for each final lab report. Write your name on the outside and inside front cover of the notebook. For each experiment we do, list in the Table of Contents on the first page the following: the date you begin the experiment, a brief title for the experiment, and the pages on which you begin and complete the experiment. Be sure to update the Table of Contents for each experiment. ***DON'T* LOSE YOUR LAB NOTEBOOK!!!**

II. Preparing for an experiment:

Before we perform the experiment, you will receive a (pink) lab experiment handout, which will contain all of the background information, safety hazards, and procedural details for the experiment, and which will provide guidelines for data collection and analysis. Read the lab experiment handout carefully, and use it to prepare your **pre-lab write-up**. The pre-lab write-up should be written in dark ink directly in your notebook; be sure to put the periodic table that comes with the lab notebook *underneath* the duplicate page before you begin writing. Your pre-lab write-up should include the following:

(a) The experiment number and title; the date on which you begin the experiment; your name, and your lab partners' initials. All of these should be written in the blue boxes at the top of the page on which you start the write-up.

(b) An introduction, in which you briefly state the purpose of the experiment, and how you plan to carry it out. The introduction should also include chemical equations to represent any reactions involved in the experiment, and mathematical formulas you will use in your data analysis.

(c) Written solutions to pre-lab questions, if any are assigned. Do not re-write the question in your lab notebook, but you should show all of your work, include units, round to the correct number of significant figures, etc.

(d) A brief experimental procedure, written as a list of bulleted steps. You should refer to your lab handout for specific, step-by-step instructions *during* the actual lab; your pre-lab should merely have a brief description of the general method, rather than lengthy, detailed directions. A modestly experienced chemist should be able to look at your experimental procedure and have a pretty good idea of what you plan to do. Your procedure should also describe major safety hazards associated with the experiment, and also state how to avoid, and what to do in case of, an accident.

(e) A data table in which you will record all of the data measured in the experiment. Leave lots of space between entries, in case you need to re-record a datum. If you plan to make repeated measurements, you should set aside one column of data for each experimental "run". You should also leave at least half a page for **observations**.

***Everything up to and including a blank data table MUST be written in your lab notebook BEFORE you come to class to perform the experiment*!**

III. Performing the experiment:

Be sure to bring your lab notebook and experiment handout to class on days we're scheduled to do lab experiments, and be prepared to get to work as soon as you arrive. Here's what happens on "lab day":

(a) I will briefly discuss the experimental procedure, demonstrate the equipment and instrumentation we'll use, and quickly check to see that your pre-lab write-up is complete. You'll be broken into lab groups, and instructed to start working.

(b) As you work on the experiment, you'll follow the procedure described in the lab handout, recording all measurements with proper units, to the correct number of significant figures. If you find it necessary to modify your data table during the experiment, by all means do so. It's your notebook, after all. If you make a mistake, simply draw ONE STRAIGHT LINE through the error, and continue by writing the correct datum or calculation underneath it or next to it. NEVER erase, completely blacken out, or (heaven forbid) use white-out in your lab notebook. I fully expect that you will make some errors in, and slop some corrosive chemicals all over, your notebook. Don't worry – your lab notebook wouldn't be authentic without them! Also, be sure to record concise but descriptive observations throughout the course of the experiment.

(c) If time permits, you may start your data analysis (see below) in class. You should try to complete at least some preliminary calculations before leaving the lab; if one of your measurements is grossly in error, you may need to repeat it.

(d) When you're finished with all of your measurements and observations, clean up your glassware and equipment according to the instructions in the lab handout, put away any chemicals or instrumentation, and WASH YOUR HANDS.

IV. Completing and submitting your lab report:

To complete your lab report, follow these steps:

(a) Finish the data analysis: show all of your calculations clearly and explicitly, include correct units in your calculations and on each final result, and round the final answer to the correct number of significant figures. If you made several determinations of the same value (e.g., a molar mass or an equilibrium constant), report the average value (i.e., the mean), and the percent deviation. If there is an "accepted value" of the quantity you measured, be sure to calculate the percent error in your measurement. If the experiment requires that you perform many repetitive calculations of the same type, you may use a spreadsheet program; simply print out two copies of the spreadsheet, tape one into your lab notebook and include one with your final report. (However, you must show at least one calculation done "by hand", in explicit detail.) Likewise, if your analysis requires a graph, print out two copies of the graph, tape one into your notebook and include one with the report. Remember that all graphs should have descriptive titles and labeled axes, with correct units on the axes.

(b) Answer in writing any post-lab questions assigned. You need not re-copy the questions, but you should show all work in your calculations, and include proper units and the correct number of significant figures in your calculations and in your final answer.

(c) Write a conclusion, which should begin with the statement: "In this lab experiment, I learned that . . . ", or something to that general effect, in which you concisely state WHAT YOU FOUND (e.g., your experimentally-determined numerical value of a physical quantity). You should also include a discussion of the theory that was demonstrated in the experiment, and how the purpose of the

experiment was fulfilled (if in fact it was). You should also describe *specific* possible sources of experimental error, and discuss how they could affect your final result (i.e., do they make the value you obtained larger or smaller than the accepted value?). Remember that human error exists in all experiments, and should *not* be mentioned as a source of error unless they cause a significant fault. Significant figures and calculation errors are NOT valid sources of experimental error! In this section, you should also discuss what you might do differently, if you were to repeat the experiment. I would also appreciate suggestions for modifications to the experiment. Finally, if there is an accepted value for your final result, you should report the percentage error you calculated in the Data Analysis section.

(d) To finish and hand in the lab report, sign your name and write the date the report is submitted on the bottom of the last page of the report, carefully tear out the duplicate copy sheets of all pages of the report, staple them together with any spreadsheets, graphs or other supporting documentation, and submit the entire report.

If your handwriting is particularly difficult to read, you should type any or all sections of the lab report; however, you must submit the duplicate copy sheets from your lab notebook. See me for more detailed guidelines if you wish to type your reports.

I encourage you to work collaboratively with your classmates on pre-lab questions, data analysis and post-lab questions, but the precise wording of all sections of your lab report should come from you and you alone. **Copying sections of another student's lab report verbatim is considered to be an academic integrity violation, and will be treated as such.**

Attached are copies of a sample Table of Contents and a sample lab report. Use these as models for setting up your lab notebook and writing lab reports!

V. Assessment:

As stated in the course syllabus, the laboratory section of this class is normally worth 15 – 20% of your trimester grade, depending on the number of lab experiment we do in a trimester. Each lab will normally be graded on a 25 point scale (although this might vary with length of the experiment and write-up), with typical point distributions and grading standards as follows:

(a) Title, Date and Introduction: 5 points. Points will be deducted if you omit the title, date performed, or any important equations and formulas from the Introduction section.

(b) Pre-lab Questions: 5 points. Pre-lab questions will be graded on correctness.

(c) Procedure, Data and Observations: 5 points. Points will be deducted if data are omitted, or not shown with units or the correct number of significant figures. Points will also be deducted if no observations are included.

(d) Data Analysis: 5 points. Points will be deducted if calculations or graphs are unclear, incomplete, incorrect or missing.

(e) Post-Lab Questions and Conclusion: 5 points. Points will be deducted if the conclusion does not have sufficient detail, or omits important information (such as final result, percent error, etc.). Post-lab questions will be graded on accuracy.

Lab reports that are submitted with incomplete or dramatically incorrect data analysis or conclusions will be assessed a point penalty and returned for you to complete. If your lab report is not completed on the due date (and you do not have a valid excuse), you must stay in after-school study hall that day and each subsequent day until the lab report is complete. Point penalties are as follows: forgetting to bring your lab book to class on the day of the lab – 20%; turning in a lab report late – 10% per school day; failing to complete the pre-lab write-up before class – 10%. Please remember that **you are required to complete and submit all lab reports in order to obtain credit for the course!**

VI. List of Laboratory Experiments, 2014-2015:

First Trimester (chapters 1-6, 13)

#1: Synthesis of a Coordination Complex of Nickel

#2: Determination of the Mass Percentage of Nickel in a Coordination Complex

#3: Determination of the Mass Percentage of Chloride in a Coordination Complex

#4: Determination of the Mass Percentage of Ammonia in, and the Chemical Formula of, a Coordination Complex

#5: Determination of the Mass Percentage of H_2O_2 in a Commercial Solution of Hydrogen Peroxide – a Guided Inquiry Experiment

#6: Determination of the Molar Mass of a Volatile Liquid

#7: Application of Hess's Law to the Determination of the Enthalpy of Formation of Magnesium Oxide

#8: Spectrophotometric Determination of the Equilibrium Constant for the Formation of an Iron-Thiocyanate Complex

Second Trimester (chapters 14-17)

#9: Standardization of a Solution of Sodium Hydroxide

#10: Determination of the Equivalent Weight and K_a Value of an Unknown Weak Acid

#11: Preparation and Interpretation of Titration Curves

#12: Preparation and Properties of Buffer Solutions

#13: Temperature Dependence of the K_{sp} Value of Calcium Hydroxide: Determination of the Standard Enthalpy and Entropy Changes in a Chemical Reaction

#14: Determination of Equivalent Mass by Electrolysis

Third Trimester (chapters 7-12)

#15: Spectrophotometric Determination of the Rate Law for the Reaction of Phenolphthalein with Sodium Hydroxide

#16: Determination of the Enthalpy of Vaporization of Water

#17: Determination of Molar Mass by Freezing Point Depression

VII. Laboratory Safety:

As always, the most important rule in this lab is: **SAFETY FIRST!** Never begin an experiment until you fully understand all of the safety hazards involved, and *exactly* what to do in order to avoid accident and injury. Review the safety rules (below) before each and every experiment.

A. General Lab Safety Guidelines:

(1) Never begin an experiment until you fully understand all of the safety hazards involved, and *exactly* what to do in order to avoid accident and injury. Start the experiment ONLY after the teacher has given clear instructions and has indicated that you may begin work.

(2) Follow all instructions carefully; never perform any experiment that is not specifically authorized by the instructor, and do not work in the laboratory if the instructor is not present.

(3) Leave bags, coats and books at your desk. You should have only a pen, your lab experiment handout, your lab notebook and any other materials specified in the procedure at your lab work station.

(4) Know the location of each of the following: emergency exits, fire extinguisher, eyewash fountain, safety shower, fume hood, fire blanket, sand and baking soda

(5) No horseplay in the laboratory! Students who endanger themselves and others by behaving inappropriately in the lab will be asked to leave class, and will receive appropriate penalties.

(6) If you are unsure about ANY procedure or materials, ASK YOUR TEACHER!

B. Appropriate Attire:

(1) Wear safety glasses whenever you are in the laboratory part of the classroom.

(2) Wear closed-toe shoes, and clothing that minimizes exposed skin, whenever you're doing experiments in the lab. You are encouraged to wear old clothes on days we perform laboratory experiments.

(3) You are encouraged to wear a lab apron and latex gloves whenever we are using corrosive chemicals. (Be sure to fold up the apron and return it to the cabinet when you're finished.)

C. Glassware Hazards:

(1) Whenever possible, secure glassware by clamping it to a ring stand. Keep beakers, flasks and other glassware away from the edge of the bench top, where they might accidentally be knocked off.

(2) Work deliberately, carefully and thoughtfully; rushing through the experiment leads to broken glassware.

(3) If you accidentally break a piece of glassware, inform your instructor, who will clean it up. DO NOT pick up pieces of broken glass yourself!

D. Chemical Hazards:

(1) Do not eat, drink or chew gum in the laboratory.

(2) Never remove any chemicals or equipment from the laboratory area.

(3) Work deliberately, carefully and thoughtfully; rushing through the experiment leads to chemical spills.

(4) Any chemicals that emit toxic, corrosive or offensive fumes should ONLY be used in the fume hood.

(5) Never fully inhale the vapors of any substance. If it is necessary to smell something, gently waft a small amount of the vapors toward your nose.

(6) If you accidentally spill a small amount of chemical on the bench top, inform your instructor immediately – do not attempt to clean it up yourself. Acid spills can be neutralized with solid baking soda.

(7) If you accidentally spill a chemical on exposed skin, IMMEDIATELY wash with large quantities of water. Acid spills should be neutralized by rinsing the exposed area with baking soda solution; base spills should be neutralized by rinsing the exposed area with vinegar.

(8) If you splash any chemical solution in your eyes, IMMEDIATELY go to the eyewash fountain and bathe your eyes in running water until the ambulance arrives.

(9) If you spill toxic or corrosive chemicals on your clothing, remove the article of clothing and rinse off in the sink or under the safety shower. If you spill toxic or corrosive chemicals over a large part of your body, rinse off under the safety shower before removing the affected clothing.

E. Fire and Heat Hazards:

(1) Use a "hot-hand" protector or thick gloves to handle hot glassware. Whenever possible, allow glassware to cool to room temperature before handling.

(2) Do not light a Bunsen burner unless and until you are instructed to do so.

(3) Place the Bunsen burner toward the center of the bench top, keep flammable materials (especially chemicals and solvents) far away while the burner is lit, and turn the gas off as soon as you're finished using the burner. Tie back long hair, and avoid loose clothing.

(4) Never leave a lit Bunsen burner unattended. Know where the emergency gas shutoff is located, and be prepared to use it in case of an accident.

(5) Any time you see a fire, inform your instructor immediately. If a small amount of flammable solvent or material catches on fire, quickly but carefully smother it with an overturned beaker or sand. If a larger fire breaks out, use a fire extinguisher, pointed at the base of the fire. If a large section of the room is in flames, leave immediately through the nearest exit, and pull the fire alarm. The last people out should close the doors.

(6) If your clothing catches on fire, "stop, drop and roll", wrap yourself in the fire blanket, or stand under the safety shower while someone pulls the handle to extinguish the flames.