

DEPARTMENT OF CHEMISTRY & BIOCHEMISTRY

# Analytical Chemistry Lab Manual

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Spring 2018



## **Analytical Chemistry Lab**

### **Lab Information**

Before each lab session, you should prepare by reading the lab manual and the textbook required reading. We expect you to have a good understanding of the purpose, details of the procedure, the use of all chemicals and any significant hazards, and the underlying science of the experiment when you come to lab.

Each Pre-lab recitation will have a prelab. Although your lab notebooks will not be checked before lab drawing up tables in your lab notebook for your data and carrying out the preparation calculations (e.g., mass needed to make up solutions of given molarities) will help you finish quickly and efficiently.

### **Report Format**

Although you are not English majors, it is still very important to be able to communicate your science to others. Writing poor enough to be unclear or confusing will affect your grade, as will lack of regard for grammar, punctuation, and spelling. Learn to use the spell-check facility that is built into your word processing software.

Here are some general comments to help you with your scientific writing. For more detail, we recommend the first chapter of the *ACS Style Guide*.

Scientific writing is not literary writing. You should aim to be brief, precise, and unambiguous. The reader should clearly understand what you are trying to say. Try to keep your verb tense consistent and appropriate. You may use either passive or active voice, but try to be consistent. Avoid using jargon or slang and use full sentences. It is rare that you would need to use first person; i.e., try not to refer to "I", "we", "our", "us", nor should you speak about yourself, e.g., "the student".

### **Title**

The Title should describe the experiment in an appropriate, adequate and concise manner.

### **Abstract**

Is a summary of the goals, objectives and the results of the experiment. It should convey a sense of the full report in a concise and effective manner. Remember, a reader should be able to read the abstract and get a full sense of what was accomplished in an experiment.

### **Introduction**

Connects the basic scientific concepts being tested, or utilized in an experiment with the objectives and purpose of the experiment. Hypotheses and logical reasoning should be given.

### ***Data and Observations***

Report all data, including appropriate units, preferably in tabular form. Any data that was collected digitally (i.e., using computerized data collection) should be printed out graphically as described in the data analysis section of your experiment and included in your report. There is no need to print out the large data tables collected by the computer. Any data that was collected on chart paper should be photocopied onto regular US letter size paper, reduced or enlarged if necessary, labeled appropriately, and included in your report. Do not attach long rolls of chart paper to your report. Include any relevant observations (e.g., color changes, unintended spills, unexpected results, etc.) and note anything you did differently from the procedure given in this manual.

### ***Data Analysis***

See the ***Useful Analysis Techniques*** section of this manual for important information, relevant to your data analysis. Your sample calculations may be hand-written, but must be clear and legible. Follow the analysis steps in this manual, labeling them by number so we can follow what you are doing. You must clearly show each step of your analysis, by writing out each equation used (the formula in words, or symbols with an explanatory key), followed by the calculation plugging in the relevant data, including the appropriate units, even if the equation is explicitly stated in the lab manual. Put a box around, underline, or in some other way highlight the important answers.

Unless you are explicitly told otherwise in the data analysis instructions, if you studied multiple samples, **carry out all calculations for all replicates**; i.e., do not average them together until the very end. Do not show working for all replicate calculations, simply show one complete sample calculation for the first replicate. You may then simply summarize the calculation results for the other replicates in a table or spreadsheet, making sure you label the columns well with informative names and units. It is not necessary to show standard deviation or line of best fit calculations, simply provide the answer from your spreadsheet/calculator. In addition to your calculations, your data analysis section must communicate to the reader what you are doing. There needs to be enough detail and explanation that a reader could follow your steps and reasoning and reproduce your results. State any reasoning processes that you have used that are not described by equations, such as anywhere you used common sense, logic, bizarre thought processes to decide something (like not dropping a data point, choosing which equation best represents your data, etc). Each step of your analysis should be clear and obvious to anyone reading your report.

### ***Results***

This section should report the results of the experiment separately from your calculations in a clear and concise format. Where multiple data sets are used, report each individual determination, the average with appropriate significant figures, the standard deviation, the percent relative standard deviation (%RSD), and the confidence interval for your data. When

using a line-of-best fit to data, report your correlation coefficient. If you Q-test away one of your three replicates, then you cannot report a standard deviation. That's ok, just talk about it in your conclusion.

### **Questions**

Write numbered, coherent answers to all questions found in the lab manual. Use complete sentences and be sure your answer demonstrates understanding of the material. Like the rest of your report, the discussion section must be in your own words. Plagiarism will not be tolerated.

### **Conclusions**

The most scientifically important part of an experiment is your interpretation of your observations. For an analytical chemist, the accuracy and precision of your results is also important. These priorities are reflected in the allocation of some of your lab report grade to these topics. It may seem difficult for you to imagine what more there is to discuss once you have determined something like the % content of ascorbic acid in a Vitamin C tablet; however, here are some guidelines to help you:

Summarize the point of the experiment. (What did you do? Why did you do it?) Discuss your results. (Can you compare to literature values or values given by a manufacturer? Do they seem reasonable? Why or why not?) In the cases where values are available for comparison, you should quantify the difference between your experimental values and the expected values. Rather than simply saying you were "way off", a comment such as your value was 15% larger than the literature value would be more appropriate. List the possible sources of error in your experiment, being as specific as you can in your error descriptions; e.g., rather than saying "operator error", describe exactly what aspect of the experiment you may have done incorrectly. Discuss any problems encountered and suggest ways around them if the experiment was repeated. Evaluate possible sources of error (operator, random, systematic, etc.) in the experiment. Discuss the most significant contributions to the error in this experiment and describe specifically how each of these significant errors would have affected your results (i.e., increased or decreased your answers). Give reasonable methods to eliminate or reduce these errors. Consider the experiment in terms of accuracy, precision, reproducibility, selectivity, and analysis time. Suggest ways to improve your procedure if you were to perform it again.

### **References**

Whenever you use material written by other people to help you create your lab report, you should reference your literature source; e.g., when you look up literature values, when you use reference information from the library, or when you read some information in a textbook to help you answer a discussion question. You should reference this lab manual and your textbook whenever appropriate. All facts, formulas, drawings, graphs, etc. used in the report that you did not create yourself require a citation of their source. Failure to reference this material has an implicit implication that they represent your original work. You should read the **Scholastic Dishonesty** section of this manual to be sure you are clear the policies. Rarely, if ever, will you need to directly quote material. I grade your understanding of what you have read. The ability to

find an appropriate quote is only half the battle, you must then frame it in your own words, applied to the particular experiment you have performed.

Your references should be numbered in consecutive series in order of mention in your report. If a reference is repeated, do not give it a new number; use the original reference number. Reference numbers in the text should be superscripted outside the punctuation of the sentence or phrase to which the citations applies, or after the name of the author if referred to in the text; *e.g.*,

*Quantitation of chlordane in freshwater fish has been reported previously.<sup>1</sup>  
When compared to previous reports of chlordane in fish,<sup>1</sup> the results reported here .....  
Work of Doe et al.<sup>1</sup> has shown that ...*

The complete list of literature citations should be placed at the end of your report, after the conclusions, but before the appendices. List references in numerical order and use the following formats. More information on good reference formatting is explained in the ACS Style Guide, available at the library reference desk. **Journal** references must include the author names, abbreviated journal title, year of publication, volume number (if applicable), and the range of pages of the cited article; *e.g.*,

(1) Smith, A. D.; Moini, M. *Anal. Chem.* 2001, **73**, 240-246.

**Book** references must include the author or editor's name, book title, publisher, city of publication, and year of publication; *e.g.*,

(2) Skoog, D. A.; Holler, F. J.; Nieman, T. A. *Principles of Instrumental Analysis*, 5th ed.; Saunders College: USA, 1998; Chapter 7.

**Website** references must include the author (if known), title of site from browser window title bar, URL, and date accessed; *e.g.*,

(3) U.S. Environmental Protection Agency Home Page, <<http://www.epa.gov/>> (accessed July 1996).

## Useful Analysis Techniques

### Data rejection

When you have a set of data with one data point that seems very different to all the rest, there must be a scientific basis for deciding whether to reject that point. You should not just throw it away without justification. If you have a series of repeated measurements of the same property (e.g., when you run three replicates of an experiment), then the Q-test is a useful statistical way to determine whether one of your points in that series of measurements can be disregarded. The Q-test tables are given in your text. **(The value of Q for three observations is 0.94.)** If you do not have a series of repeated measurements of the same property, then you cannot use the Q-test.

If you have no basis for rejecting a data point, other than some gut level feeling that dropping a particular data point will make all your data look so much better, then this is what you should do: Do the analysis using all your data. Show that the results using all your data are very different than what you would expect. Point out how bad that one data point looks (e.g., the plot is linear except for this one point). Speculate about at least one specific experimental reason that could explain how that data point could have been in error. Redo the analysis dropping that data point. Make self-congratulatory statements about how that fixed all your calculations.

### Data reporting in your lab reports

Most experiments in this course involve running three replicates to reduce the uncertainty in your determined mean, and investigate the precision of your results. Use the mean of your replicates as your final answer; however, carry out all calculations for all replicates. Do not average them together until the very end. The standard deviation of the replicates is used as a measure of how widely distributed your individual measurements have been (and by extension, would be expected to be in the future), and should be reported as a  $\pm$  range about your mean value. Also, we require you to list the percent relative standard deviation (%RSD); i.e., the ratio of the standard deviation to the mean, converted to a percentage. Finally, you should quote the appropriate (stated in the procedure, usually 90 or 95% confidence interval, which is reported as the mean value  $\pm$  a calculated uncertainty value. Although you report both the standard deviation and the confidence interval as ranges about your mean value; they describe very different properties of your data set, and it is important in your discussion to distinguish the meanings of these two different results. Keep in mind that depending on the information sought, one or the other may provide the more appropriate measure of experimental uncertainty. Uncertainty is in the same units as the value, so quote the units after both the value and the uncertainty. If you quote your value in scientific notation, do the same for the uncertainty. When quoting your results in the two required formats (mean  $\pm$  standard deviation and mean  $\pm$  confidence interval), the number of significant figures quoted for the mean may differ for the two formats.

When quoting specific values, you need to use the appropriate **significant figures**; i.e., rather than showing all the digits that have been generated during a calculation, you round your answers in the following way. **When quoting uncertainties, in general you should use only one significant digit**, unless that significant digit would be a "1". In this case only, use two significant

digits. **When quoting your answers**, you want to show all the certain digits, and only the first uncertain digit, thus **you should quote each value such that the last significant figure of your experimental value is of the same order of magnitude as the absolute uncertainty in that value**. If your uncertainty has two significant digits (i.e., the first digit is a "1"), then you should quote the answer such that the last significant figure of your experimental value is of the same order of magnitude as the second significant digit of your uncertainty. When giving your results in a lab report, we would expect you to quote the original experimental data with appropriate significant figures, and to use a reasonable number of significant figures when quoting your final results.

**Never truncate or round numbers during your calculations**, even if some of the intermediate numbers were rounded to significant figures for reporting purposes along the way. Some experiments are treacherously susceptible to error if this rule is not followed.

## Introduction to the use of Analytical Equipment

### Electronic Balances

Unless accuracy is not critical to the procedural step being performed, all weighing in analytical lab is performed using a clean dry **weighing bottle**. Chemicals must not be weighed directly on the pan of any balance. Only weighing bottles, weigh boats and weighing paper should contact the pan. Any chemical that falls on the pan will be weighed along with the chemical in the container but will not be transferred to your experiment, resulting in errors. Never try to weigh an object heavier than the capacity of your balance (160 grams for our Mettler balances).

Temperature can affect the weight of an object. To be weighed accurately, all objects should be at room temperature. A warm weighing bottle will set up convection currents inside the balance case, which will buoy up the balance pan reducing the apparent weight. In addition to the weighing bottle and its contents, a balance also measures the weight of the column of air above the weighing bottle and this air is less dense above a hot object. **Hot objects, such as crucibles, should always be cooled** to room temperature in a desiccator (to prevent adsorption of water) prior to weighing.

### Weighing by Difference

All weighing in analytical lab is performed **by difference**. **We do not use the TARE option** on the electronic balances. This procedure explains how to create triplicate samples of the same solid using weighing by difference.

1. Keep your balance clean. Remove dust from the pan and floor of the balance with a brush before weighing your sample.
2. Weigh your weighing bottle with lid to get an approximate idea of its weight. Do not touch the

weighing bottle with your hands — wear gloves, or hold it using a collar of paper around the neck of the bottle. Close all doors on the balance before making a weight determination. Any air movement can cause variation in weight in the mg range.

3. Many solids increase their weight by taking up water vapor or carbon dioxide from the atmosphere. Some solids are provided predried in a desiccator. Remove the chemical from the desiccator. If you try and transfer chemical from the container while still in the desiccator you are likely to spill chemical into the desiccator. Keep the lid on the desiccator except when actually taking things in or out of it. Do not add chemical to the weighing bottle while it is still on the balance. Place the weighing bottle on the counter beside the balance, and use the spatula provided to transfer sample from its container to your weighing bottle. When you are finished return the chemical container to the desiccator.
4. The weighing bottle lid minimizes absorption of water vapor or carbon dioxide from the air during the weighing process. Use it. Weigh the weighing bottle along with its contents, including the lid, recording the weight using all significant figures. If the difference between this weight and the bottle weight recorded in Step 2 is not the quantity you require (roughly three times a single sample), remove the weighing bottle from the balance and add or remove chemical as appropriate. Do not return unused chemical back into the stock container. Put it in the solid waste.
5. Remove the weighing bottle from the balance and transfer approximately one-third of its contents without loss directly into the first container to be used in your experiment. To transfer the solid, tip the weighing bottle sideways over the container, and use the lid of the weighing bottle to tap gently on the side of the weighing bottle, until an appropriate amount of solid is transferred. Transfer should be made into a container with a large enough mouth (e.g., Erlenmeyer flask) that there is no danger of losing solid during the transfer.
6. Weigh the weighing bottle and its remaining contents, including the lid, recording the weight using all significant figures. **The weight of your first transferred sample is the *difference* between this weight and the weight you measured in Step 4.**
7. Remove the weighing bottle from the balance and transfer approximately one-half of the remaining contents without loss directly into the second container to be used in your experiment.
8. Weigh the weighing bottle and its remaining contents, including the lid, recording the weight using all significant figures. **The weight of your second transferred sample is the *difference* between this weight and the weight you measured in Step 6.**
9. Remove the weighing bottle from the balance and transfer the remaining contents without loss directly into the third container to be used in your experiment.
10. Weigh the weighing bottle and any residual solid, including the lid, recording the weight using all significant figures. **The weight of your third transferred sample is the *difference* between this weight and the weight you measured in Step 8.**
11. When you have finished using the balance, make sure that all weights are recorded and clearly labeled in your notebook, no objects are left on the balance pan, the balance and surrounding bench

top is clean, excess solid is disposed of in the solid waste container, and stock chemicals are returned to their desiccators. Keep the balance room clean, tidy and minimize the paper you bring in there.

If a balance does not behave properly, **report it to the instructor**. Do not try to fix it yourself.

### Volumetric Glassware

In analytical lab all volume measurements are made using **volumetric glassware**. Glass is inert to most chemicals, its coefficient of thermal expansion is small, and measurement is simplified by the transparency of this material. There are many types of glassware; some of which is designed for holding and transferring approximate volumes (e.g., beaker, graduated cylinder, Erlenmeyer flask, graduated pipettes). Volumetric glassware is designed for measuring and transferring accurately known volumes of liquids (e.g., volumetric flasks, volumetric pipettes, burets). The volumetric markings on volumetric glassware are correct to within set tolerances given in Table One below. Note that these tolerances are valid only if the glassware is scrupulously clean.

TABLE I Tolerances for Volumetric Glassware

Volume Capacity mL	Volumetric Flasks	Transfer Pipets	Burets
1	± 0.02 mL		
2	± 0.02 mL	± 0.006 mL	
5	± 0.02 mL	± 0.01 mL	± 0.01 mL
10	± 0.02 mL	± 0.02 mL	± 0.02 mL
25	± 0.03 mL	± 0.03 mL	± 0.03 mL
50	± 0.05 mL	± 0.05 mL	± 0.05 mL
100	± 0.08 mL	± 0.08 mL	± 0.10 mL
200	± 0.10 mL	± 0.10 mL	
250	± 0.12 mL		
500	± 0.20 mL		
1000	± 0.30 mL		

### Burets

A buret is used for titrations and delivers a variable but accurately known volume (i.e., it is TD glassware). Your buret is composed of three major parts: **The Barrel** is the graduated tube used to hold your solution. **The Stopcock** controls the rate of flow of solution out of the buret. **The Tip** is carefully machined to deliver regular size drops.

#### **Cleaning your Buret**

The buret is a delicate and expensive piece of glassware and should be handled carefully. If the buret is broken in any way, it is best to replace it. In particular, each time before using your buret, check that there is no damage to the end of the buret tip. Any chips or cracks in a buret tip will have serious effects on the delivery of the titrant. Return it to the instructor if it needs to be replaced. Burets must be carefully washed with soap and water, to make sure that solutions uniformly drain down the inner

surface of the barrel. A long buret brush may be used in the barrel. Some burets allow you to remove the valve and buret tip to get easier access to both ends of the barrel. The buret is clean when there are no obvious deposits of material and if the buret drains cleanly without leaving droplets of solution on its inner walls. Clean the tip and stopcock by repeated rinsing. If the tip or stopcock becomes clogged, they may be unclogged by **carefully** pushing a piece of small diameter wire through the openings. If applicable, after cleaning, reassemble your buret. Care should be taken to assure that the stopcock is **firmly** seated or leakage will occur. Test for leaks by closing the stopcock, half filling the buret with distilled water, and watching for any liquid seepage.

### ***Rinsing and Filling a Buret***

Once your buret is clean and doesn't leak, it is ready for use. Solutions used in burets are usually standardized (i.e., their concentrations are precisely known), so in order to avoid dilution, you must rinse your buret with your solution before filling; thus:

1. Lower the buret in its holder so the top of the barrel is within easy reach. Using a funnel, pour ~5 mL into the buret from a convenient vessel (a beaker is much better than a 1 liter bottle).
2. Remove your buret from its holder; tip and rotate the barrel so that the solution is brought into contact with the entire inner surface of the buret.
3. Allow the solution to drain completely out through the tip.
4. Repeat with a second 5 mL wash.
5. Close the stopcock and fill the buret to within 1 cm of the top.
6. Let the solution sit in the barrel with the stopcock closed. Make sure that there are no leaks.
7. Make sure that there are no air bubbles in either the barrel or the tip. Tap the barrel lightly to dislodge air bubbles (this is an indication of a dirty buret). To remove air from the tip, open and close the stopcock fully to force the air out. You may have to do this several times.
8. Allow the solution to drain until it is within the graduated portion of the barrel.
9. **Label your buret** with its contents, so you don't get your burets confused.
10. Remove the funnel before taking any readings, or else it may drip additional solution into your barrel, destroying your volume measurement. Record the initial level of the solution in the buret. There is no reason to attempt to start each titration with the solution exactly at 0.00 mL; this practice wastes time and introduces operator bias.

Once a buret has been rinsed and filled, it may be refilled as many times as necessary that same day with the same solution without repeating the above routine. Each day and with each change of solution, the buret should be thoroughly washed with soap and water, rinsed with distilled water, rinsed with the

standard solution and filled as above. Burets are stored upside down so that material does not collect in their tips.

### ***Reading the Buret***

Determinations using a buret involve an initial and final reading; the volume used is the difference between these two readings. Readings must be made correctly and in the same way each time. Remove any partial drops on the buret tip by touching the tip to the inner side of your titration flask. If you have been delivering solution rapidly, with the stopcock fully opened, wait a few seconds for all the liquid to run down the inside walls of the barrel before taking your reading. Readings are made from the bottom of the meniscus (curvature of the liquid in the barrel) for most solutions. With highly colored solutions, you may not see the meniscus and then any other consistent point of reference may be used. All readings with a buret or other volumetric glassware **must be made at eye level**. If you have to look up or down, there will be a serious parallax error involved. Raise or lower your buret in its holder to move the meniscus to your eye level. Your buret is graduated in 0.1 mL intervals. Read it to the nearest 0.01 mL. A plain white index card held behind the buret will make it easier to read. It is also a good idea to place a white piece of paper under your titration flask if the endpoint is being detected by an indicator colour change.

### ***Dispensing Solutions***

**Always hold the valve joint when turning the stopcock**, so the buret will not break and so that you do not pull the valve and tip out of your buret by holding it too tensely. A technique that permits speed, accuracy, and control is as follows: Face your buret with the stopcock handle on the right side of the barrel. Operate the stopcock with your left hand from behind the buret, while swirling your solution with your right hand. Your left thumb and forefinger grasp the stopcock handle, while the other fingers of that hand hold the valve joint and tip steady. (*Reverse for left-handers-like your professor!*)

Treat your first titration as a rough measurement to learn the approximate volume of titrant that will need to be added to reach the endpoint. For subsequent titrations, you can fully open the stopcock at first, allowing a fast stream of solution to enter the titration flask. As the endpoint is approached, the solution should be added more slowly, until it is added dropwise to reach a "perfect" endpoint (each extra drop added results in  $\sim 0.05$  mL of error). Careful stopcock control allows dropwise addition. Be careful to ensure that each drop enters the sample solution. If you touch off a partial drop from the buret tip onto the inner wall of your sample flask, remember to rinse the partial drop down into your sample solution using a wash bottle.

EACH EXPERIMENT IS PROVIDED

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SEPARATE DOCUMENT