CH455/6 Analytical Chem: Lab Manual Excerpts — Fall 04

Course Instructors	Dr. Paul McCord (CH455) .... pmccord@mail.utexas.edu .... Phone: 471-6522, Welch 5.239 Prof. Jason Shear (CH456) .... jshear@mail.utexas.edu .... Phone: 232-1454, Welch 3.426
Laboratory Director	Dr. Ruth Shear .... DrRuth@mail.utexas.edu .... Phone: 471-5755 WEL 3.106
Stockroom	Angie Steelman .... aloving@mail.utexas.edu .... Phone: 471-3928, WEL 2.128
Head TA	Jose Arellano .... gj.arellano@cm.utexas.edu
Lectures	MWF 12 – 1 pm .... 2.304 (CH455), WEL 2.312 (CH456)
Laboratory Sessions	T 9:30 am – 12:30 pm, W 9 – 12 pm, MTWTh 2 – 5 pm, MTWTh 7 – 10 pm .... WEL 2.116 Attend one lab session per week, based on your class unique number. See the calendar in this manual for your experiment schedule.
Office Hours	Office hours for your professor will be announced in class. TA office hours are held in WEL 2.112 and will be posted by the lab. Do not disturb your TAs outside these times, except by appointment; they have lives outside Analytical Lab. You may disturb the Lab Director any time you can find her.
Textbook	Daniel C. Harris, *Quantitative Chemical Analysis*, 6th ed.
Required Supplies	This lab manual Combination lock Lab notebook with duplicate numbered pages
Lab Notebook	The format and content of your lab notebook is described in a section of this manual. As your grade is dependent on your notebook, we recommend reading that section.
Experiments	You will carry out one experiment each lab session, following the procedures in this lab manual. ***Lab reports are due at the start of your next lab session***. A penalty will be given for late reports. Reports may be turned in to any TA in class or office hours, to the Stockroom, or the report box across the hall from the Stockroom door. Follow the format given in this manual. A sample report is supplied.
Safety & Waste	Safe lab practices and waste disposal procedures are described in this manual. Unsafe, imprudent and careless activities will result in you being removed from the lab. Continued noncompliance could result in a reduced lab grade or course failure.
Appeals	Your TA will make decisions concerning grades, attendance, and other policy matters. Should you disagree with a TA, you may take the matter to the Lab Director.
Plagiarism	For definitions of nonacceptable behaviour, such as plagiarism, and the UT policy regarding such activities, please see <http://deanofstudents.utexas.edu/sjs/academicintegrity.html>

Fall 2004 CH455 Analytical Lab Schedule (WEL 2.116)

CH455	Monday	Tuesday	Wednesday	Thursday
Morning		52925 **Selynda**		
Afternoon		52930 **Carina**	52935 **Jose**	
Evening		52940 **Sarah**		52945 **Myles**

Analytical Office Hours Schedules (WEL 2.112)

	Monday	Tuesday	Wednesday	Thursday	Friday
8–9			**Ganesh** (CH456)		
9–10	**Nicole** (CH456)	*9:30 am* CH456 TA Meeting		*9:30 am* **Selynda** (CH455)	**Bella** (CH456)
10–11		CH456 TA Meeting	CH455 TA Meeting		
11–12			CH455 TA Meeting *11:30 am*	**Ben** (CH456)	
12–1	Lecture		Lecture		Lecture
1–2				**Sarah** (CH455)	
2–3	**Carina** (CH455)	**Amanda** (CH456)			
3–4	**Myles** (CH455)				
4–5					
5–6				**Jose** (CH455)	

Jose Arellano	gj.arellano@cm.utexas.edu
Myles Gardner	myles.gardner@mail.utexas.edu
Carina Gunder	cgunder@mail.utexas.edu
Sarah Pierce	spierce1@mail.utexas.edu
Selynda Soto	selynda@mail.utexas.edu
Jose Arellano (Head TA)	gj.arellano@cm.utexas.edu
Ruth Shear (Lab Director)	DrRuth@mail.utexas.edu

Fall 2004 CH456 Analytical Lab Schedule (WEL 2.116)

CH456	Monday	Tuesday	Wednesday	Thursday
Morning			52960 **Nicole**	
Afternoon	52965 **Amanda**			52970 **Bella**
Evening	52975 **Ben**		52980 **Ganesh**	

Analytical Office Hours Schedules (WEL 2.112)

	Monday	Tuesday	Wednesday	Thursday	Friday
8–9			**Ganesh** (CH456)		
9–10	**Nicole** (CH456)	*9:30 am* CH456 TA Meeting		*9:30 am* **Selynda** (CH455)	**Bella** (CH456)
10–11		CH456 TA Meeting	CH455 TA Meeting		
11–12			CH455 TA Meeting *11:30 am*	**Ben** (CH456)	
12–1	Lecture		Lecture		Lecture
1–2				**Sarah** (CH455)	
2–3	**Carina** (CH455)	**Amanda** (CH456)			
3–4	**Myles** (CH455)				
4–5					
5–6				**Jose** (CH455)	

Amanda Gerlach	amandagerlach@mail.utexas.edu
Nicole Green-Mower	n_green-mower@mail.utexas.edu
Ben Hahn	bhahn@mail.utexas.edu
Ganesh Vijayaraghavan	ganeshv@mail.utexas.edu
Bella Yao	bellayao@mail.utexas.edu
Jose Arellano (Head TA)	gj.arellano@cm.utexas.edu
Ruth Shear (Lab Director)	DrRuth@mail.utexas.edu

From “Laboratory Procedures”

Prelab Preparation

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. Instead of submitting a handwritten prelab report, you must pass an oral Prelab Quiz at the start of each lab session to demonstrate your understanding of the material. In addition to the general list of points above, specific topics that you may expect to be quizzed on are listed at the start of each experiment.

Each prelab quiz is administered by a CH455 TA (not necessarily your class TA). TAs may choose to give quizzes individually, or in small groups. If you clearly have not read the manual and textbook, or have not attempted to understand the experiment, then you will be told that you will have to perform the experiment during a makeup session. As long as an honest attempt has been made to read and understand the experiment, you will be allowed to perform your experiment. As well as acting as an entry pass to performing your experiment, the prelab quiz will be worth 20% of your lab report grade for that experiment. Although no prelab report is required, 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.

During the long semesters (Fall and Spring), the lab will be open for you to take your quiz starting 30 minutes before your lab session commences. You are not required to come early, but once you pass the quiz, you will be allowed into the lab to start preparing to do your experiment. You may pick up equipment from the stockroom, wash your glassware, etc.; however, you may not start using chemicals until after the TA introductory lecture, which will be given ten minutes after your lab session commences. During the summer session when the lab meets for four hours, not three, we will start the quizzes at the start of your lab session, and the TA introductory lecture will be given at a time deemed appropriate by your TA after your lab session commences.

From “Lab Reports”

You will write a lab report on each of the experiments you perform during this course. Use the required readings to help you complete each lab report following the ***Report Format*** section below. Follow the data analysis steps in your lab manual. Number all graphs, drawings, spreadsheets, etc. and refer to them in the appropriate part of the report. Don’t forget to answer all discussion questions asked in the manual for the experiment.

When handing in your report, submit the original (white) pages, not the carbon copies, from your lab notebook, attached to printouts of all work done on a computer. Make sure you keep a copy of all computer work submitted. You will have a carbon copy of all work in your notebook; thus, any report you have submitted should be able to be recreated by you in the case of loss or other catastrophe.

Report Format

Although you are not doing an English degree, 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-processor. 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*, which is available on reserve in the library. 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”.

Twenty percent of each lab report grade is based on your presentation, formatting, and layout. Although we don’t require it, we recommend that you use a word processor and spreadsheet software to produce your reports. All graphs must be created using graphing software, not hand-drawn. Any section not produced on a computer must be hand-written in your lab notebook. Do not use pencil. See the ***Your Lab Notebook*** section of this manual for more detail. There is no penalty for turning in a completely handwritten (excluding graphs and photocopied data) report if your writing is neat and legible and your report is well organized and clearly laid out. Conversely, using a computer does not automatically guarantee you full presentation points. Use a legible (at least 12 pt) font, lay things out well, label table columns, etc.

A ***Sample Lab Report*** is included in this manual as a guideline for what we are expecting. The following components are required in each of your lab reports:

Report Summary Sheet

Attach the appropriate **Summary Sheet** from the back of this manual to the front of your Report. Your TA should have filled part of it in during your lab class. You need to complete the top section and then fill in the summary of your results, using the appropriate significant figures and units.

Purpose

Brief summary of the principles demonstrated in this experiment (what you are going to do and why).

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

Follow the analysis steps in this manual, labeling them by number so we can follow what you are doing. 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. You do not need to show all working for all replicate calculations. Show one complete sample calculation for the first replicate, writing out the each formula in words first, then plugging in the relevant data, including the appropriate units, clearly showing each step of your analysis. See the sample report for examples of what we are expecting. 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. We recommend that you actually perform each step of the sample calculation with a calculator, rather than just writing in the results your excel spreadsheet gives you. This way your sample calculation acts as a check of your excel spreadsheet, so you can detect any errors you have made in your formulae. It is not necessary to show standard deviation or line of best fit calculations, just give the answer from your spreadsheet/calculator. Your sample calculations may be hand-written, but must be clear and legible. See the **Useful Analysis Techniques** sections of this manual for important information, relevant to your data analysis. If the manual has told you to plot something, then that means you must include the plot in 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 90% confidence interval for your data. When using a line-of-best fit to data, report your correlation coefficient. See the **Error & Statistical Analysis** section of this manual for more information. 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.

Discussion 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 on our policies. Rarely, if ever, will you need to directly quote material. We 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.[1]
When compared to previous reports of chlordane in fish,[1] the results reported here ……
Work of Doe *et al.*[1] 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).

From "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 ± 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 90% confidence interval, which is reported as the mean value ± a calculated uncertainty value.

Although you report both the standard deviation and the 90% 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 ± standard deviation and mean ± 90% 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. Note: We do not require you to use the error propagation techniques we use in physical chemistry labs.

From "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* supplied at room temperature by the Stockroom. 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. This is particularly a problem in humid Austin. We supply these solids 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 Stockroom**. 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, burettes). 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.

Burettes

A burette is used for titrations and delivers a variable but accurately known volume (i.e., it is TD glassware). Your burette 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 burette. *The Tip* is carefully machined to deliver regular size drops.

Cleaning your Burette

The burette is a delicate and expensive piece of glassware and should be handled carefully. If the burette is broken in any way, it is best to replace it. In particular, each time before using your burette, check that there is no damage to the end of the burette tip. Any chips or cracks in a burette tip will have serious effects on the delivery of the titrant. Return it to the Stockroom if it needs to be replaced. Burettes must be

carefully washed with soap and water, to make sure that solutions uniformly drain down the inner surface of the barrel. A long burette brush may be used in the barrel. Some burettes allow you to remove the valve and burette tip to get easier access to both ends of the barrel. The burette is clean when there are no obvious deposits of material and if the burette 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 burette. 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 burette with distilled water, and watching for any liquid seepage. Stopcock grease is *not* required when using a Teflon stopcock. If you have a glass stopcock and feel you need grease, you may get some from the Stockroom.

Rinsing and Filling a Burette

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

1. Lower the burette in its holder so the top of the barrel is within easy reach. Using a funnel, pour ~5 mL into the burette from a convenient vessel (a beaker is much better than a 1 liter bottle).
2. Remove your burette from its holder; tip and rotate the barrel so that the solution is brought into contact with the entire inner surface of the burette.
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 burette 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 burette). 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 burette* with its contents, so you don't get your burettes 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 burette. 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 burette 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

burette should be thoroughly washed with soap and water, rinsed with distilled water, rinsed with the standard solution and filled as above. Burettes are stored upside down so that material does not collect in their tips.

Reading the Burette

Determinations using a burette 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 burette 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 burette 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 burette in its holder to move the meniscus to your eye level. Your burette is graduated in 0.1 mL intervals. Read it to the nearest 0.01 mL. A plain white index card held behind the burette 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 Solution

Always hold the valve joint when turning the stopcock, so the burette will not break and so that you do not pull the valve and tip out of your burette by holding it too tensely. A technique that permits speed, accuracy, and control is as follows: Face your burette with the stopcock handle on the right side of the barrel. Operate the stopcock with your left hand from behind the burette, 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.)

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 ~ 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 burette tip onto the inner wall of your sample flask, remember to rinse the partial drop down into your sample solution using a wash bottle.

Sample Lab Report

Purpose

The purpose of this experiment is to prepare a standardized solution of NaOH for use in future experiments. Direct titration against a primary standard (KHP) will be used to determine the concentration accurately.

Data and Observations

KHP

1. Wt. Bottle + KHP ____________g
2. Wt. Bottle + remaining KHP ____________g
3. Wt. Bottle + remaining KHP ____________g
4. Wt. Bottle + residual KHP ____________g

NaOH Standardization

1. Volume NaOH ____________mL
2. Volume NaOH ____________mL
3. Volume NaOH ____________mL

HCl Standardization

1. HCl Volume ____________ mL NaOH Volume ____________ mL
2. HCl Volume ____________ mL NaOH Volume ____________ mL
3. HCl Volume ____________ mL NaOH Volume ____________ mL

Observations

Write your observations here, noting which part of the experiment they apply to. Alternately, you can write them interspersed between the data as you make the observations. Be sure to note any deviations from the procedure that took place, such as repeating a portion of the experiment due to mistakes, etc.

Data Analysis

All calculations can be hand written, NEATLY! Just one sample calculation for each step is necessary; don't do a calculation for each replicate sample. Make sure you write out the formula in words first, before plugging in the numbers. You don't need to show calculations for average, standard deviation or % RSD.

Step 1: *Moles of KHP*

Mass of KHP transferred to flask 1

= Mass of KHP in Weighing bottle (g) – Mass of Weighing bottle after transfer (g)

= 2.9321 g – 2.1080 g = 0.8241 g KHP

No of moles KHP in flask 1

$$= \frac{\text{Mass of KHP transferred to flask (g)}}{\text{MW KHP (g mol}^{-1})} = \frac{0.8241 \text{ g}}{204.23 \text{ g mol}^{-1}} = 4.035 \times 10^{-3} \text{ mol KHP}$$

Similarly, No of moles KHP in flask 2 = _____ mol; No of moles KHP in flask 3 = _____ mol

Step 2: *NaOH Molarity*

Concentration NaOH

$$= \frac{1 \text{ mole NaOH}}{1 \text{ mole KHP}} \times \text{Moles of KHP (mol)} \times \frac{1}{\text{Volume NaOH (L)}}$$

$$= \frac{1 \text{ mole NaOH}}{1 \text{ mole KHP}} \times 4.035 \times 10^{-3} \text{ mol} \times \frac{1}{0.03582 \text{ L}} = 0.1126 \text{ M NaOH}$$

Similarly, NaOH Conc using flask 2 = _____ (M); NaOH Conc using flask 3 = _____ (M)

Average NaOH Conc = _____ (M)

Step 3: *HCl Concentration*

Concentration HCl

$$= \text{Avg Conc. NaOH (mol L}^{-1}) \times \text{Volume NaOH (L)} \times \frac{1 \text{ mole HCl}}{1 \text{ mole NaOH}} \times \frac{1}{\text{Volume HCl (L)}}$$

$$= 0.1126 \text{ mol L}^{-1} \times 0.03611 \text{ L} \times \frac{1 \text{ mole HCl}}{1 \text{ mole NaOH}} \times \frac{1}{0.03546 \text{ L}} = 0.1147 \text{ M HCl}$$

Similarly, HCl Conc using flask 2 = _____ (M); HCl Conc using flask 3 = _____ (M)`

Average HCl Conc = _____ (M)

Results

NaOH Standardization	
Trial	**Conc NaOH** (mol L^{-1})
1	0.1126
2	...
3	...

HCl Determination	
Trial	**Conc HCl** (mol L^{-1})
1	0.1147
2	...
3	...

Avg NaOH Concentration

____ ± ____ M; %RSD = ____ ; 90% confidence interval = ____ ± ____ M

Avg HCl Concentration

____ ± ____ M; %RSD = ____ ; 90% confidence interval = ____ ± ____ M

Discussion Questions

Again, I'll leave these for you. Answer all the questions in the manual completely and coherently. Give me a break - I did the rest of the lab for you!

Conclusions

Standardization is a process used to establish accurate concentrations of solutions. Using KHP as a primary standard, a solution of NaOH has been standardized and compared to an HCl volumetric standard. The results obtained are (*what do you think of them*). My standardized NaOH concentration is (??) with a %RSD of (??) and a 90% confidence interval of ± (??). This means that the accuracy seems ?? and the precision is ?? (*Talk about YOUR results here*). Several sources of error affect these results. The largest error is associated with determining the equivalence point. The consistent reproduction of a very faint color change is difficult to do. There will be some variation in the color change due to the human limitation of observing colors consistently, and hence a variation in the endpoint will be observed. There is also a small amount of error associated with reading the burette, since the last digit of the reading is uncertain. The error associated with things like the indicator and reading burettes cannot be eliminated; they can only be minimized as much as possible through good technique. Running more replicates would increase precision and reduce random error. (*Any ideas you have can be put here*).

References

Don't forget to list references for any information used in your report, using footnote format as described in the lab report format section.

Notes on this sample lab report

- DO NOT plagiarize this sample report. It's meant as a guideline only. It is also a report from several years ago. Some parts of this experiment have changed since this report was written.
- DO NOT plagiarize right out of the lab manual. You all have the ability to put this stuff in your own words.
- Although it is not required to have the data portion of the lab laid out beforehand, it is highly recommended. It makes things go much faster pand easier.
- Always calculate and report the % RSD (% Relative Standard Deviation) and the 90% confidence interval using Student's t.
- This is just to give you an idea of what a good report looks like. Some sections may be longer or shorter for various labs, or may require different things. Please refer to the guidelines laid out in this lab manual for writing lab reports as you write your reports.

Preparation of an NaOH Standard Solution using Direct Titration

This experiment demonstrates the most common method for obtaining standard solutions for titrimetric analysis. It involves preparation of a solution that has the approximate concentration desired (usually within 10%), determination of the concentration by *direct titration* against a *primary standard*, and a test of the accuracy of your determined concentration by comparison with a known standard. It is important to standardize your solution carefully, as it will be used in later experiments. You should be able to determine your NaOH concentration to ± 0.5% of its actual value. You will be graded on your accuracy.

Required Reading

D.C. Harris, *Quantitative Chemical Analysis* (6th ed., W. H. Freeman, NY, 2003) pp. 128–31, 224–7, 239–44.
Electronic Balances (including Weighing by Difference), *Analytical Lab Manual*, pp. xxi–xxiii.
Burettes, *Analytical Lab Manual*, pp. xxiv–xxvii.

PreLab Quiz Topics

In addition to being able to explain the purpose of your experiment, the general procedure steps, the use of all chemicals in this experiment and any specific hazards, your prelab quiz may include explanations of any of the following terms: burette, titration, primary standard, equivalence point, endpoint, indicator, standardization. You should know all relevant chemical reactions for this experiment. Describe the chemical and/or physical processes that will occur when you reach the endpoint of your titration. What characteristics of KHP make it a good primary standard?

Chemicals and their Location

Balance Room

Potassium hydrogen phthalate

Above/on lab bench

Phenolphthalein, indicator soln
Sodium hydroxide, ~ 0.1 M aqueous soln
Hydrochloric acid, vol. std.

Equipment and its Location

Stockroom

Burette
Weighing bottle

Safety Issues and Chemical Hazard Information

	Physical Hazards	*Health Hazards*
Hydrochloric acid	water-reactive, corrosive	toxic
Phenolphthalein	none	irritant
Potassium hydrogen phthalate	none	irritant
Sodium hydroxide	water-reactive, corrosive	toxic, irritant

Procedure

There are various components to this procedure. If there is a delay at one step (e.g., all the balances are in use), perform another section of this procedure while you are waiting. Depending on the size of your lab section, your TA may suggest that half of the class work straight through the procedure, and the other half do Steps 2 and 4 before Steps 1 and 3, to reduce congestion in the lab.

Preparation of a Primary Standard (KHP)

1. KHP is a primary standard. It is predried and kept in desiccators in the balance room. To minimize contamination, use the spatula provided in the desiccator — do not use your own spatula. Using a clean dry weighing bottle, weigh accurately **by difference**, triplicate ~0.8 g samples of KHP, into labeled 250 mL Erlenmeyer flasks. To each flask, add 75 mL of distilled water (measure by graduated cylinder) and three drops of phenolphthalein indicator. Swirl gently. Cover the flasks and leave to dissolve, swirling periodically.

Preparation of Approximate Solution (0.1M NaOH)

2. The Stockroom has already made a set of solutions, all ~0.1M NaOH. Choose one of these solutions and **remember to record which one you used** (it will have an identifying letter or number). Fill a clean 1 L bottle with this solution. If you are using a glass bottle, use a rubber stopper (alkaline solutions tend to "freeze" ground glass joints). Clean your burette, and rinse with a small aliquot of your NaOH solution, before filling it with the NaOH solution. Make a note of the volume of liquid in your burette within 0.02 mL precision.

Standardization of NaOH with KHP

3. Begin to titrate your first KHP solution by adding NaOH rapidly until a pink colour is noticed. A piece of white paper under the titration flask will aid in observing the colour change. At this point, slow the addition of NaOH so that only a localized pink colour is observed in the stirred solution. Continue slowing the rate of addition to keep the amount of pink colour localized. Stop every so often to be sure that all colour disappears upon mixing. As the endpoint is approached, a single drop of NaOH may turn the solution pink for a moment. Proceed very slowly with drop by drop additions until the entire solution remains light pink for at least 60 seconds. At this point, the endpoint has been reached. Record the final volume of the burette within 0.02 mL precision (all burette readings must have two decimal places to attain any reasonable precision). Similarly, titrate your other two flasks of KHP.

Be careful not to let the fluid level in the burette drop below the 50 mL line. If more than 50 mL will be required for a titration, stop the titration before reaching the 50 mL line, record the exact volume, refill the burette, record the new initial volume, and continue with the titration. Upon completion, the two volumes can be **added** to obtain the **total** titrant volume.

Comparison of NaOH solution to HCl Volumetric standard

4. (If your TA has told you to perform this step before Step 3, read the titration instructions in Step 3 carefully before continuing.) To each of three clean labeled 250 mL Erlenmeyer flasks, add 50 mL of distilled water and 3 drops of phenolphthalein. Labeled burettes with volumetric standard HCl will be provided at the front of the lab. Choose one of these burettes. (Make sure that you use the same standard for all three flasks.) Record the initial reading of the HCl burette to the nearest 0.01 mL. Add approximately 35, 40 and 45 mL of the acid to flasks #1, #2 and #3, respectively, recording the initial and final burette readings each time, so that you can calculate **exactly** how much acid has been added to each flask. Titrate the HCl with your NaOH solution in the same way you titrated the KHP.

Shutdown procedures

IMPORTANT NOTE: Your NaOH solution **<u>must be saved</u>** upon completion of the experiment; you will use it again in future experiments.

5. Put all waste in the appropriate waste containers, as listed at the end of this procedure. Clean and dry all glassware. Clear up your work area. Return items to the Stockroom.
6. Make sure you have recorded information on the sample you studied, in this case, the number/letter identifying the HCl volumetric sample, and the number/letter identifying your NaOH starting solution.

Waste Disposal

Waste Chemicals	Waste Container	Location
Neutral solutions after titration	Neutral waste	Waste cabinet
HCl; unused KHP solution	Acid	Waste cabinet
KHP solid	Solid KHP	Balance room
NaOH	Base	Waste cabinet

Data Analysis

Read the ***Report Format: Data Analysis*** and the ***Useful Analysis Techniques: Data reporting in your lab reports*** sections of this manual before working on your first Data Analysis and make sure you follow them.

1. Using the weight of KHP in each flask, and the molecular weight of KHP, calculate the number of moles of KHP in each flask.

2. Calculate the molarity of your NaOH solution using the total volume of NaOH used to titrate each KHP flask to its endpoint. (Don't forget to convert your volume from mL to liters.) Report your standardized NaOH concentration as the average of your three replicates with appropriate statistical analysis (see ***Report Format: Results*** section).

3. Using your experimentally determined standardized NaOH concentration (use the average value), determine the molarity of your volumetric standard HCl. Report your determination of the HCl concentration as the average of your three replicates with appropriate statistical analysis (see ***Report Format: Results*** section).

Discussion Questions

1. Why can't we just dissolve a known mass of NaOH in a known volume of water and calculate the NaOH concentration? In other words, explain why you standardized your NaOH solution.

2. Ideally, when performing aqueous acid-base titrations, all distilled water should be boiled prior to use to remove dissolved carbon dioxide. All solutions should then be kept tightly closed when not in use; NaOH solutions are especially capable of adsorbing large amounts of CO_2 from the atmosphere. Why would we ideally want to eliminate CO_2 from solutions when performing acid-base titrations? You did not use boiled distilled water to make your KHP solution. What effect might this have on your standardization — would it raise or lower the concentration you determined for your NaOH solution? We do not take particular care storing your NaOH solution after it has been standardized. If you used it to determine an unknown acid at the end of the semester, what effect might the absorbed CO_2 have on your determination?

3. Describe how you would weigh your KHP samples, if you didn't know about weighing by differences. Mentioning any differences between your method and the method we made you use, explain why we use the technique of weighing by difference.

Fall 2004 CH455/6 Lab Report Title Page

Student Use	
Name:	**Class TA:**
Lab Partners (if any):	**Makeup TA** (if any):

Class TA Use	
Prelab Quiz Score (out of 3):	**Lab finished:** (TA signature required before student leaves lab)

Experiment Date:	**Report Due Date:**	**Due Time:** 9 / 9:30 am; 2 pm; 5 pm; 7 pm

Report Received		
Date:	**Time:**	**Initials:**

Grading		
Report Grade (/15):	**Late penalty** (/15):	**Report inc. penalty** (/15):
Regrade (/13.5):	**Final Grade** (/15) (includes penalty & regrade):	

Preparation of an NaOH Standard Solution using Direct Titration

Your report should follow the Lab Report format listed earlier in this manual. Show samples of all calculations, identify any unknowns, report the average with appropriate significant figures, standard deviation, %RSD and 90% confidence interval and answer the discussion questions. Don't forget to include your conclusion section.

Concentration of analyte in unknown sample	Average ± Std Dev	%RSD	Average ± 90% confidence interval
Standardized NaOH concentration	±		±

Identify your NaOH starting solution ________________________________

Unknown HCl concentration	±		±

Identify your HCl unknown ________________________________

Preparation of an NaOH Standard Solution using Direct Titration

Report Grading Breakdown

	Your Score	/	Out of a possible
Pre-lab Quiz		/	**3**
Calculations/Data Analysis		/	**2**
Results/Accuracy		/	**4**
Discussion & Conclusions		/	**3**
Presentation/Impression		/	**3**
Subtractions			
TOTAL		/	**15**