Geology 203
Environmental Geochemisty and Analysis
Syllabus

Instructor:	Karen Harpp
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Office Hours:	M, W 2:45-4:30 PM and almost any other time by appointment (email or call me to make one)

The best way to contact me is by email. If you are not familiar with the system, or do not have an active account on the Colgate system, you need to do so immediately. Frequently, we will communicate as a class by electronic mail, including changes in assignments and class schedule, so it is *essential* that you become comfortable with this system as soon as possible and that you check it DAILY for announcements about the class. I will assume that you can get info within 24 hours of my sending it.

Meeting times and places

This is a bit complex and we'll be doing some arranging the first time we meet.

Officially: Tuesdays and Thursdays 9:55-11:10 AM in 404 Lathrop. This will be our base but we'll move around quite a bit depending on what we're up to in lab at the time.

Lab will meet either Tuesdays from 1:20 PM-4:00 PM *and (usually)* from 6:00-9:00 PM. Meet in our classroom (Lathrop 404) unless announced otherwise, but we'll be moving around a lot for a variety of logistical reasons, so stay tuned to the plans each day. We'll spend some time in B14 Winn and a lot of time in Lathrop. *We'll announce plans in class or by email at least 24 hours in advance.*

Because this course is effectively an integrated lab/class experience, there will be no conceptual separation between the "class" sessions and the lab sessions, they'll all merge together. **The bottom line is that you should come to class promptly at 9:55 AM and assume that you will be in class/lab through 9:30 that night with breaks in the middle around lunch and dinner.** There will be some weeks where we do less, as well, but assume this is going to be the case to be on the safe side.

Class and lab will be integrated in this course, and we will go back and forth between group activities and individual lab work frequently. This means you are required to attend the entire class and laboratory period every week without fail. If you are miraculously done with the assigned lab work, I will help you work on other aspects of your projects. This isn't the type of class where the lab is a self-contained task, it's a series of projects that you design that are extensive and require your full attention for the full class and lab periods.

Catalog Description

Effective examination of environmental problems necessitates an understanding of the geological, chemical, biological, and physical parameters that interact within each system. The focus of this interdisciplinary laboratory and field-based course is the development of practical skills essential to the study of natural systems. Through a series of local projects, students will learn how to address environmental questions, including initial experimental design, collection and analysis of samples, statistical and graphical treatment of data, interpretation of results, and presentation of conclusions. Specific skills will include a variety of instrumental and laboratory-based techniques for the chemical analysis of natural materials including rock, soil, and water (e.g., atomic, ultraviolet, and x-ray spectroscopy, microscopy, mass spectrometry, wet chemistry techniques), the use of spreadsheets and graphing programs in data analysis, sample collection and handling techniques, experimental design, and methods of data presentation. Fundamental aspects of environmental geology and chemistry will be examined in the context of the specific projects.

Course Philosophy

In a nutshell, this course is ultimately applicable to many aspects of a scientific career. The lab skills that you will learn and refine are some of the most marketable parts of your undergraduate degree. The precision and care that you should learn to apply to analytical procedures will serve you in any scientific investigation you may encounter in the future. The basic data processing and presentation techniques you learn will be useful in any scientific endeavor, regardless of your particular discipline. The informed skepticism that you should learn to apply to the data you obtain is essential to carrying out high quality scientific work. In other words, the tools you will be developing in this course will serve you throughout your careers as scientists, be that as biologists, chemists, geologists, environmentalists, physicists, astronauts, dog trainers, pastry chefs, whatever....

It is essential that you understand what our goals are prior to embarking on this course. You will be learning how to apply analytical techniques to answer a variety of questions. It is important to understand fully what the capabilities and limitations of a given technique are, so that you can use it appropriately and with maximum efficiency.

Analytical science is a field that in isolation is hard to grasp. Learning how to determine iron contents is not particularly informative or interesting unless you have a reason to find out those concentrations. In other words, you employ analytical techniques as a *tool* to address problems of significance. We will be emphasizing the use of analytical techniques to examine questions about the environment. Environmental science is a field that relies heavily upon high quality analytical information about a wide variety of materials, ranging from water, to food, to soils.

The role of the analytical environmental scientist is multi-faceted; to address a problem, the scientist must:

-- formulate the question of interest;

-- choose the appropriate analytical technique to address the question;

-- determine which samples, when analyzed, will most efficiently and completely help to answer the question;

-- carry out the analysis efficiently and precisely;

-- analyze the data and present it in a coherent, clear manner;

-- draw sound conclusions based on the data.

The course will culminate with a final project that you will design in full, from the question to be asked right down to the analytical details and sampling procedures. I expect you to work hard and do your very best in all aspects of this course. I hold you to the highest standards and know that you will achieve them, but it will take patience, time, and diligence on your part. I will help in every aspect of this course and you will emerge from it proud of your accomplishments and with a rigorous grounding in analytical science.

Required Materials

1. Textbook: There isn't one appropriate for this course, so we have compiled a set of chapters from various books for your reference. Some provide important information about analytical techniques and ways to handle data, others describe the instruments we will be using in details, and still others provide background on environmental geochemistry. See the schedule for readings. We'll put all the readings in a binder for you (what a deal). Use these copied chapters as reference and as primary reading, they will provide an enormous amount of essential information for you during the term. I also recommend that you explore the references on environmental science in the library (Cooley), there's a ton of useful information there, as well as on the web (but make sure your sites are reliable and not "Fred's Toxic Environmental Site" or something).

2. Lab book: Bound notebook; spiral books are *not acceptable*. They fall apart too easily, and the metal reacts with acid. You should number the pages as you use them. There is no need to purchase fancy overpriced brown lab books unless you have an overwhelming desire to do so. Feel free, of course, but it's no problem either way as long as you can read what you write and the book holds up throughout the term. And write in pen, not pencil.

3. Computer file storage: We will be doing work on computers during class/lab time, which means you will want to take those files with you once we're done. You can certainly leave them on the computers in Lathrop 401, or email them to yourselves, but you should have your own copies at least as backup (particularly for the more extensive write-ups). I recommend either zip disks or CDs, but please avoid floppy disks, which shouldn't be used for anything other than coasters (zips either, really). Most of the computers we'll use have CD-writers. All final lab write-ups will be done on word processors, and will include illustrations and graphs that also must be constructed electronically. Unless we run into some weird technological snag, all reports must be handed in via the digital drop-box on Blackboard on the web (http://bb6.colgate.edu); I'll show you how this works in class. Problem sets may be handwritten or word-processed or any combination thereof, unless a graph is required, in which case it must be done on the computer.

The Laboratory

This is primarily a laboratory class. We will merge class and lab activities that are traditionally separate because, in reality, they are intimately intertwined. We will often collect samples for the projects, so we'll work in many different locations at various times. We will have to organize sample collection expeditions, and we will usually use official "class" time for lab work and sample processing. So always come to class ready for lab. I will be doing the minimum of lecturing, we will be doing the maximum amount of hands-on work. We will never be doing an experiment to which I (or anyone else for that matter) know the answer; we are discovering new information about the area with every new analysis we perform, and you are learning important techniques in the process.

Analytical geochemistry is measurement science. You are interested in answering a specific question about an environmental issue; you need analytical tools to help you determine the real story. High precision results are the goal of most lab work. As a result, the quality of your lab results and technique is extremely important. Use the lab time to learn and refine basic techniques; your goal should be to learn how to produce high quality environmental data.

The lab work is a *tool* that helps you determine answers to real environmental questions. It will require skills and careful work, so be patient in the lab…you will skilled excellent at analytical work and will learn many incredibly useful tools that will make it possible for you to explore and answer many environmental questions. We will practice techniques and basic laboratory skills that you will need to carry out the more extensive experiments. You will be evaluated on your ability to perform these tasks, so use the lab time to practice those skills (in other words, exams may include laboratory skills).

You must adopt an approach to labs different from that which was sufficient for many other labs. You cannot come to lab and say "Here I am, what do I do now?". I am more likely to ask *you* what you are doing, and expect an informed and organized reply.

You will find that detailed instructions for experiments are limited to the specific techniques. You need to develop a proper approach to the lab; it will be the one you will use for future scientific research. In general, read the procedure before you come to lab, at least twice. The first time, determine what question you are trying to answer with the lab work. In a "real world" situation, as well as for your independent projects, you determine the question ahead of time according to your needs and/or interests and choose the technique accordingly. It's not usually handed to you in the write-up...The second time, read in greater detail to figure out exactly *how* you are going to answer that question in lab, summarizing your plans in your lab book before you come to work.

Establish a plan of approach to the experiment as a whole, in significant detail. Such a plan will consist of each step to be taken in the order you feel is the most efficient. Oftentimes, the order in the experimental handout is not the optimal one. Your plan must include the weights and volumes of reagents and solutions to be used at each step in the procedure, as well as tables for the data you will be taking. This will lead to better organization of your notebook and more efficient work in the lab. **I will check to see that the plan has been prepared during the lab period. Your grade will suffer if there is not any evidence of preparation prior to coming to lab.**

Try to understand the background information behind the environmental question you are trying to answer; do extensive research in the library and on the web before getting involved in the project (but watch out for environmental websites, they often are subject to some fairly extreme biases, so be sure that your site is objective). Know what contaminants you need to analyze, what the levels of contaminants are that are considered dangerous or important, and then make connections between your research and the analytical techniques. Will you be able to detect the relevant levels of the contaminants, or is the technique too crude? Be aware of every step in the analytical procedure, and the basic principles behind them. In other words, understand the geochemistry behind what you are going to do in lab. Changes in procedure are much more easily managed when you understand what is really going on in each analytical process. Such changes may occur for a variety of reasons, usually with a goal to improve the technique. Don't panic! As you will see, even procedures printed in the scientific literature do not always work for everyone, sometimes not for anyone. This is where you must learn to be skeptical and think about what you are doing in detail. If it doesn't work, figure out why it isn't working and FIX IT.

Do not hesitate to ask for help at any point in the process. If you don't understand what you are doing before lab, make a point of asking questions in class before and during lab or find me before lab. This will ensure that you are ready for the work we plan to do. When you ask for help before lab, you are my top priority; should you come to lab unprepared, you will take priority behind those who are fully prepared. We will work out problems collaboratively.

Course Requirements

You will be responsible for the following assignments:

Radon project (individual)	10%
Lead in soil project (small groups)	15%
Onondaga Lake project (TBA)	20%
Independent project (individual or small groups)	25%
Midterm exam (individual)	10%
Final exam (individual)	10%
Optional problem sets (individual)	up to 10% extra credit
Participation (individual; includes short assignments)	10%
Total	**100%**

The approximate grading scale for the course will be:

A	= 90%
A/B	= 85%
B	= 80%
B/C	= 75%
C	= 70%
C/D	= 65%
D	= 60%.

The letter grade awarded to those who fall in an intermediate range (e.g., A/B or 85 - 90%) will depend upon total points, as well as my perception of that student's effort, participation, reliability, and aptitude. As a reminder, a grade of C means your work is acceptable; it just means you have room to improve. Do not get distressed at a grade of C, just crank up the effort. Always feel free to come discuss with me how you can improve your work. The grade of A requires exceptional work, in all aspects of the assignment.

Exams

There will be one midterm and one final exam in this course, each worth 10% of your final grade. They will be timed exams, and you must do them all in one sitting, but you may choose the time you take the exam (within reason, like a self-scheduled exam at finals, for instance). There may be a lab component to the exams, potentially.

Final Project

The independent project is worth 25% of your final grade. The project will be of your own design, and must incorporate the skills and processes you will have learned over the course of the term, choice of an important and relevant environmental question (usually of local interest but it could be related to an issue in your hometown or some other place where you can collect samples), sample collection, experimental design, proper use of instrumentation, appropriate choice of instruments for the question at hand, sample preparation, data processing, data presentation, literature research and references, and oral communication. You are responsible for a formal presentation of your project on the last day of class, which must use Microsoft PowerPoint (or a similar multi-media application) for the visual aids. In other words, you do this project from start to finish, as a opportunity to apply what you've learned and to learn something useful about our environment.

Collaboration

A few comments on cooperation between scientists are in order. No person in science works in a vacuum (ok, figuratively speaking). The reason for scientific meetings and the journal literature is to provide a means for the sharing of ideas and results. Out of this interaction, new ideas are born. The same can be said for your work in this course. The interactions you have with other students can be extremely valuable for learning the material, and cooperation is strongly encouraged. You will be working in various group sizes on the different labs and projects. Some experiments will require individual work, others will require small teams, while we will address some issues as an entire class. One of the projects requires you to work as a single team, which is difficult, but can be an amazing experience if done carefully, with patience, and in an organized manner.

The same rules apply to our class interactions that apply to those of any group of scientists. **All scientific interactions must be properly referenced**. If you work with someone as a lab partner on an experiment, you are **both** expected to keep a lab notebook detailing what you did individually. The notebooks will not be identical as you will certainly divide the work and will have different styles of presenting what you have done. All work not done by you must be referenced to indicate who did it or who thought of the ideas. When you write a report, if you use someone else's ideas as a part of the report, give them credit for them. Most importantly, never copy anything that is not properly referenced. I hold you to the highest level of honesty and integrity and expect no less from you.

Late Policy

Work that is up to 24 hours late will be penalized by 20%. Work that is more than 24 hours late will not be accepted unless a postponement was arranged a minimum of 48 hours in advance of the deadline. No exceptions.

Class Attendance and Preparation

Your regular attendance in class/lab is required. There will be a significant amount of course credit given for in-class participation, in the form of presentations, group discussions, and group analysis of data. Part of the responsibility of a scientist is learning how to communicate your ideas and results effectively, and how to participate efficiently in group projects; discussions and presentations are essential in refining communication skills. Your final grade will be *strongly* affected by your participation, preparedness for class, and consistent attendance.

It is best to have the reading done *before* coming to the class where we consider that topic; see the syllabus for a tentative schedule. You will also have to do background research in the library, in scientific journals, textbooks, reference books, and on the web. In addition, I will have reference textbooks in my office, you are welcome to use those as well.

Because this class is an integrated lab/class program, your attendance is mandatory *at all class/lab periods* and *for the entire time*. Only very rarely will you be able to leave lab before its official ending time, so adjust your schedules accordingly.

If you are drowsy or inattentive in class, or if you are late to or absent from the class/laboratory, your grade will be adversely affected:

- Students with one unexcused absence from the required class/lab day will be penalized by a lowering of their course grade by one letter (e.g. A will become B; B will become C, etc.). Partial attendance of a lab period will count as an absence.

- Students with two or more unexcused absences from class/lab will receive an F in the course.

- Students who habitually come to class late or are drowsy or inattentive in class will be penalized by a lowering of their course grade by up to a full letter grade.

Notify me in person, by email, or by phone if you will need to miss a class due to illness or personal calamity. Leaving me a message is not enough; you need a response from me for the absence to be excused. You will generally need some sort of official sanction (note from health center or dean of students office) in order to be excused from class/laboratory. You will need to bring written notification **in advance** signed by your coach/professor for any class that you will miss for athletic or other academic reasons.

Bear in mind that given the flexible, integrated nature of this class, we can adapt to a reasonable degree to accommodate exceptional circumstances, so be sure to talk to me about any impending crises or conflicts, and we'll do our very best to work around them. Should you have to miss a class, you are responsible for obtaining the relevant information yourself.

And finally, a reminder....

HOW TO DO HIGH QUALITY WORK

The grades you receive for your work depends only in part on 'getting the right answer'. In fact, in Chemistry 21, I don't know *any* of the answers; often, we are looking at natural systems that change on a daily basis. It is also very important that you communicate what you know clearly and effectively, and so your grade will depend on the form of your work as well as its content. Heed the following, terribly simple advice:

D o h i g h q u a l i t y w o r k !

This may seem obvious! But, what does it mean? The best advice I can give you is to avoid producing work in this or any course that looks like you are just going through the motions of something without knowing why except that you were told to do it, or hastily getting something done in time, or complying grudgingly with something that you are being made to do. Craft your work well. Plan and think before you write. Make your work both complete and precise: avoid vague generalizations and, whenever appropriate, include relevant details and show your logic and rationale. Make sure your tone and language are worthy of the occasion: scholarly and professional. Find a way to get into the spirit of things that is compatible with your basic nature. There are many ways to shine. Nevertheless, excellent work LOOKS excellent -- mediocre work LOOKS mediocre. Some guidelines:

FORM: 1) Correctness. A basic issue is always the correctness of your work -- punctuation, grammar, spelling. Make sure your handwriting is neat and legible. If I can't read it, how can I give you credit for it?

2) Accuracy and precision of language. A big problem many students have is the use of inaccurate and imprecise language. Avoid vague, cryptic and colloquial language. It reflects both inadequate thought formulation and inadequate facility with vocabulary. Time and care can fix this problem.

CONTENT: 3) Focus and relevance. Did you stay on one well-defined subject or fly off on tangents? Did you have a point or did you wander and ramble, as though lost?

4) Verisimilitude. Was you interpretation of the problem or issue reasonable or did it indicate a probable misunderstanding?

5) Preparation. Did your answer reflect adequate familiarity with the material we have studied, or did it look like you hadn't studied very much or paid attention in class?

If your work is weak in any of those ways, then it is hard to think of it as more than fair to mediocre in quality, and to give you more than about a C.

SOME QUALITIES OF EXCELLENCE: To get an honest and heartfelt B or higher for your work, it needs in addition to display at least some of the following qualities:

1) A sense of mission. Did you get the point of the exercise? Or did you seem confused?

2) Deftness. Was the tail wagging the dog, or vise versa? Did you seem as if you didn't have a clue about what you were doing or why, or did you have things under control?

3) Insight. Did you see deeply into the issue? Did you have an original thought about it?

4) Awareness of context and significance. Did you indicate when and how the problem called for a larger understanding of the material as well as the various contexts in which it could be usefully viewed?

5) Subtlety. Did you seem to appreciate the depth and complexity of the issue? Or were your thoughts facile, superficial, poorly formulated, hasty, incomplete, etc.?

Laboratory Records

For each lab experiment that you perform, you will need to produce TWO documents.

1) **The lab notebook**: this is a record of what happened in lab, during the actual lab period. You should be able to completely reconstruct the lab based on these notes, so they should be thorough, neat, and organized. All measurements are recorded directly into the notebook.

2) **The lab report**: this is a summary of the results from your lab work, including all graphs and data tables, as well as a concise conclusion.

The Lab Notebook

I am not going to check your lab book, not formally at least (I'll check informally and give suggestions, though). It is YOUR set of records for the experiments. If you keep a good, organized record of your work, you will be able to produce thorough, concise laboratory reports once the experiments are complete. If you do not keep a thorough record of your work during lab, your reports will obviously suffer. Feel free to ask for suggestions about how to keep your lab records, or design your own system...just be thorough, and everything must be reproducible. A few guidelines you should follow are described below.

A scientific notebook should hold a permanent record of the experimental work. Consequently, the pages should be securely bound (not a spiral binding) and entries made in ink at the time work is done. The pages are numbered and the entries are dated. The format of the entries must be such that the book could be read by any scientist who is familiar with quantitative analytical work. Readability of the original data is essential. Neatness is convenient but not central.

It is common practice to record data on the right-hand page and to use the left-hand page for preliminary readings, notes and calculations. Any evidence of recording data on scratch paper, paper towels, etc. will adversely affect your participation/lab organization grade.

Errors are a part of scientific work. Data is frequently invalid; experimental conditions may not be adequately controlled, reagents may be contaminated, instruments may not be operating correctly, scales of instruments may be incorrectly read, etc. If results have more than the anticipated variation, the first reaction may be to discard the whole thing and start over. Later, more significance may be read into the data. All data, including data that is known to be invalid, remains a part of the laboratory record. To correct an entry draw a single line through it and enter the correct value above the original value that remains readable. If the reason for the change is not obvious, an explanation should be recorded. If a large section of the work is considered invalid, a single line is drawn diagonally across the page and the reason for discarding the work stated. All entries remain readable. Under no circumstances is a page removed from the book. To erase or block out data, or to remove a page from a notebook is considered a violation of scientific integrity. In research, valid notebooks are the basis of determining priority of scientific discoveries and the granting of patent rights.

At the end of a determination, the work is summarized in a table which includes the data and calculated values for all trials. The pages on which the data, the calculations and the summary occur should be cross-referenced if the pages are not consecutive. With experience, it is possible to record essentially all data directly in this summary table. These tables, particularly if they show intermediate values in the calculations, are helpful in discovering trends and locating errors in calculations. One of the chief sources of errors in quantitative determinations is the incorrect treatment of the data. Calculations are as much a part of a determination as any measurement.

The correct use of significant figures requires alertness and judgment. The most common error is the copying of all of the digits on the computer or calculator printout. The use of computers and calculators require careful attention to the correct entering of data followed by careful consideration of the number of figures to be retained in the final answer. One additional figure should be retained in all intermediate values of a calculation and the final answer reduced to the correct significance. It takes hard work to get good data. Don't invalidate the results with sloppy calculations.

Anyone can keep an accurate notebook if they put their mind to it. Here are some suggestions which may help organize the notebook so it is easier to read.

1. Plan what you are going to do before you start writing.
2. Don't try to put too much on one page.
3. Use one end of your bench space for your notebook and the other for wet work.
4. When not in use, keep the notebook closed and off of the desk top. If a disaster occurs, data may be transferred to another page. Draw a single line across the ruined page and cross-reference the two pages.
5. Run through calculations on scrap paper first. This is quite permissible since calculations can always be repeated as long as the data are available. Show only one example calculation in your notebook to allow later verification of the method used.
6. Units MUST be clearly recorded with all measurements. This is a crucial practice worth pursuing, it will save confusion and often help you figure out how to do a calculation if you keep the units clearly marked at all times.
7. Data and calculated values should be presented in tables. Even though instrument recording and computer printouts are attached to the reports, significant values should be included in the tables.

Tables of results are possibly the most crucial part of the notebook. A well-organized student can often prepare the table prior to beginning the experiment. This saves laboratory time and indicates a thoughtful, orderly approach to the experiment. A well thought out table will simplify recording and subsequent calculation of results.

The importance of establishing a pattern of careful notebook keeping is difficult to overemphasize. Most industrial laboratories engaged in analytical chemistry are required to follow Good Laboratory Practice standards (GLPs) established by professional groups or government agencies. In some laboratories, for example, every weighing, buret reading, etc., must be witnessed and countersigned. This would be a little extreme in our course. An almost universal practice is to have each page witnessed at the end of each day's work. Obviously, we will not bother with such practice. Bear in mind, however, that **complete documentation of all procedures and data exactly as done and when done is the goal of the lab notebook.**

In some cases, the labs are assigned to teams of two or three. Each team member is expected to be prepared and to contribute equally to the work in the laboratory. Tasks may be divided at the discretion of the team members, but each should get a broad, balanced exposure to the experiment. Data recorded by one partner should be transferred to the notebook of the others as soon as is practical. Xeroxed copies of the data are perfectly acceptable *as long as they are secured in the notebook*.

Suggested Format for the Lab Notebook

1) **Title** and **Date**

2) **Objective**: This is a short statement of the *technical* goals of the experiment. Include any major reactions that are known to occur in the experiment; see the lab instructions or me for help with these if you are unfamiliar or uncomfortable with them.

3) **Reagent Table**: Include the name and/or chemical formula, the concentration, the approximate amount needed, and physical properties (such as melting point, boiling point, and solubility) which are relevant to the experimental manipulations or data analysis. Such information can be obtained from the Handbook of Chemistry and Physics, also known as the CRC, located in the science library.

4) **Procedure**: An operational outline of the planned experimental procedure such that it could be reliably followed by one of your peers. Be sure to state the original procedure reference clearly.

5) **Data** and **Observations**: This includes raw data tables, qualitative observations, and working conditions, parameters, and instrument settings which pertain. Tables should be prepared in advance of the lab period when possible. Be sure to record the uncertainties in measured values.

6) Your **conclusion** will be drawn in your final report, after data analysis. You may include a tentative conclusion in the notebook, based purely on laboratory work.

A well-kept notebook is an important and invaluable record for preparation of the report.

The Lab Report

Due dates for the lab reports are shown in the syllabus. Generally, they are due at least one week after completion of the last day of the lab work. There are, however, a few labs you will be doing in pieces over the course of the term. **It is imperative that you keep**

good records of your lab work, because you will have to go back and recall what happened a few weeks ago, in order to write your final reports.

The lab report is the summary of all of your work, background information on the project, all data and observations, precisions, and the conclusions which you draw from the data. The report must include tables of data, graphs, maps if appropriate, a report of or reference to experimental procedure, statistical treatment of the data, discussion of results and a conclusion. The report should be kept to an absolute minimum, relaying the data, data analysis, error, and conclusions as concisely as possible. Brevity is a major advantage. It means you are sufficiently organized and understand the lab well enough to get your conclusions across efficiently.

For some experiments, each individual will be required to produce a report regardless of the size of the group involved in the project; for others, group reports will be required (see syllabus for details). It is perfectly acceptable, and in fact, encouraged, for you to work together on producing the laboratory reports but you absolutely positively **must** acknowledge your partners during any part of the lab or field work, *and* anyone who works with you during the write-up and data analysis, for any fraction of it.

Report format

Always check the instructions given with each lab handout for suggestions of issues to address. You can always ask me for help and suggestions as well.

1) Lab reports must be written up using Microsoft Word. No handwritten reports will be accepted. All tables must be produced using a spreadsheet; all graphs must be produced using a graphing program. I recommend using Microsoft Excel for spreadsheet calculations, and Excel or Deltagraph for plotting/graphing applications. Use Microsoft Word for text production. Deltagraph will be available on the Lathrop 401 computers, probably. Do not hand in numerous files and charts that are disconnected; everything needed for the report must be in a single Word document, or you will lose points for disorganization. You can import charts and spreadsheets from other applications as necessary.

2) You must submit the report *via* the Digital Dropbox in Blackboard (http://bb5.colgate.edu). The report must be submitted *no later* than the due date and time listed on the syllabus.

3) **Cover page:** Begin the report with a single cover page that summarizes the lab objectives and final results. This is a one paragraph summary or abstract of your work. It's easiest to write this after you've finished the report itself.

4) **Introduction:** This should be a brief paragraph explicitly describing what question you are trying to address with the analytical work, and how you did the analyses.

5) **Procedures:** Do not write a background or procedure section unless you deviated from the prescribed method. Do include the complete reference for the procedure details, with indications everywhere you changed the procedure. **Make sure somebody could reproduce your work exactly from your instructions and references to existing instructions (such as the lab handouts).**

6) **Data:** Include an organized summary of measured data, with experimental uncertainties, preferably in tabular form. This is only information pertaining to the data, what it is, what the errors are for all calculations and measurements, averages, standard deviations, replicate analyses, relevant calculations, formulae used and clearly define all parameters and variables, balanced equations to indicate relevant stoichiometric relationships where

appropriate, and any other relevant information, a table of final results (if applicable). There is no interpretation of data here. Any discussion should be limited to the quality of your data, comparison with known values where possible, and observed patterns. The accuracy and precision of your final results will play a non-trivial role in the final report grade.

7) **Discussion:** Here you interpret your data. What does it mean? Did you answer your original question? Can you prove that you did? Use statistical analysis, charts, figures, graphs, illustrations, and text to back up your conclusions. What are the ramifications of your results? What do they mean? Only conclusions based on the data are acceptable. Do not hypothesize or speculate unless you can back up your arguments with literature information at the minimum. Include outside references to support your arguments here. **Charts, graphs, figures:** Attach supporting charts, graphs, and figures to your report with a stapler. All charts/graphs must be labeled with a title, sample name, date, axis labels, scales, and any relevant experimental parameters. And don't forget that if you hand in your report by email, the entire report should be in a single Word document, not a series of separate files and charts and graphs which I will have trouble reconstructing into something coherent. I guarantee that if you hand in a paper copy and there are loose graphs/tables/text I will take off points. No question. Get a stapler.

8) **Conclusion:** Include a concise conclusion that summarizes your arguments in the discussion.

9) **References and Acknowledgements:** Acknowledge any help that is received from others outside of your group and from other bibliographic sources. I encourage you to find relevant background information to support the reasons behind doing the work, background details, allowable levels of contaminants (e.g., from the Environmental Protection Agency) helpful information, and anything that assists you in coming to your final conclusions. Any form of citation is acceptable as long as I can find the original source easily and you are consistent. I encourage you to use the library and journals and public information to support your conclusions in your discussion. Incidentally, the Web is a perfectly acceptable source for information, as long as it is cited as a reference; just be sure it's a legitimate website and not unreliable in some way. Be absolutely certain that you acknowledge every single individual with whom you worked in any way, from the field to lab to discussions and data processing.

10) Finally, a word to the wise: **PROOFREAD**. **SPELLCHECK**. Read the syllabus again, where it describes how to do high quality work. I guarantee that you will lose points for sloppy, careless writing, grammar, spelling, presentation, and organization. Do the best that you can, I expect no less from you. Obviously the occasional typographical error is understandable, but anything beyond the level you find in this document, for example, is thoroughly unacceptable. Seriously. Do your best work and present it so that you are proud of it.

Laboratory Techniques

There are essentially two criteria for judging a technique. Does it work? Is there a less laborious technique that works equally well? This section and the text provide some guidance to help you develop habits of work that are both simple and effective.

Reagents

In so far as practical, reagents will be supplied in the manufacturer's bottles. Note the analysis reported on the label. Record the manufacturer and grade of the reagent used in your lab notebook.

The purity of the reagents used is a determining factor in the results obtainable in analytical work. Consequently, every effort must be made to keep stock bottles free from contamination. **Under no circumstances should material be returned to a stock bottle in a general laboratory**. A large stainless steel scoopula with a handle or a porcelain spatula may be used to break up caked solids. Use some judgement as you would not want to introduce the stainless steel spatula into a reagent used for trace metal analysis. An example from recent history was a geological analysis that was ruined because of contamination from a platinum ring worn by the technician doing the sample preparation. This error received worldwide publicity due to the nature and importance of the erroneous findings.

In general, solids are best poured from bottles. Rotating the bottle back and forth helps to control the rate of flow. Droppers and pipets should never be dipped into stock bottles. Droppers from dropper bottles should not come in contact with any surface outside of the dropper bottle itself.

Drying at Elevated Temperatures

Drying ovens are usually operated at 120-125°C. As the name implies they are used for drying at an elevated temperature. The efficiency of the drying process also depends upon the pressure of water vapor in the immediate atmosphere. Consequently a very wet object introduced into an oven may actually cause another object in the same oven to pick up water. Consequently, take care when using ovens that you do not put something extremely wet (such as glassware) into one containing delicate samples. Before using any ovens, note the temperature at which they are operating. Do not change the setting on an oven without consulting the other people working in the lab.

Space is always at a premium in a drying oven. Distribute objects around the sides and back of the shelves so that all objects can be reached with tongs. Crucibles and weighing bottles should be dried in small labeled beakers covered with small ribbed watch glasses. Any object left in an oven for several days without a note explaining how long it should be left there could be removed during cleaning of the lab, so make sure that it is clear what you are doing with your equipment/samples.

Microwave ovens are replacing conventional ovens in the laboratory just as they are in the kitchen; unfortunately, we do not have one dedicated to the lab at this point. But just for your edification, typically, samples can be dried in about 25% of the time it would require in a conventional oven. Nevertheless, there are some significant exceptions. Some samples melt, burn, or decompose when placed in a microwave oven for an extended time. It is always advisable to experiment with a small quantity of the material before intrusting your entire sample to the microwave.

CAUTION: MATERIALS REMOVED FROM BOTH MICROWAVE OVENS AND CONVENTIONAL OVENS ARE HOT. USE TONGS OR GLOVES WHEN HANDLING THESE OBJECTS TO PREVENT BURNS. I realize you know that already, but it's like the caution note on McDonald's coffee cups...prevents law suits, I guess.

Cleaning Glassware

The cleaning process should be as simple as possible. Rinsing with tap water and several small portions of distilled water may be adequate. For more dirty glassware, scrubbing with detergent and water should precede the rinsing. From a chemical point of view soap, detergents, etc. are dirt. Rinse very thoroughly with tap water and then at least three times with small volumes of distilled water. In a few cases special chemicals may be needed to dissolve solids or oils. The inside of a container that may come in contact with chemicals is not dried with a towel because this introduces lint. In many cases it is not necessary to dry glassware. Simply rinse with the solution to be used unless this would invalidate measurements. Beware of drying equipment with compressed air. This may introduce oil vapor from the pump.

Chromic Acid Cleaning Solutions

A solution of potassium dichromate dissolved in concentrated sulfuric acid was traditionally used for difficult cleaning tasks.

THIS SOLUTION WILL NOT BE USED IN THIS LAB UNDER ANY CIRCUMSTANCES. Chromium compounds are Class I carcinogens and their use is severely restricted. Mmmm.

Glassware that resists cleaning with lab soap and water may be cleaned with Citranox or other special cleaning solutions. Ask me if you need any extra cleaning power.

Cleaning Volumetric Glassware

Volumetric glassware must be clean so that water drains from the surface without leaving droplets. It will not do this if there is the least bit of oil on the surface. Once "clean" glassware becomes dry it will usually not drain properly when water is added again. Volumetric glassware is cleaned just before use or cleaned and stored full of distilled water or other solvent. The 50-mL burets can be scrubbed with lab soap (1 teaspoon in a large beaker of warm water) and a buret brush. In doing this, scrub the buret in sections, about 10 cm at a time. The 10-mL buret is fragile and difficult to clean. It is too small for a brush and is cleaned in the same fashion as the pipet. The tip is very small and the cleaning solution should not be taken through it.

Working Surfaces

The bench tops should be protected from acid solutions and organic solvents. All other surfaces should be protected from all reagents. Use paper towels to wipe up all spilled materials. Repeatedly wash the surface with a wet towel to remove water soluble materials including acids and bases. The working surface should be kept clean and dry. Spilled material is then quite evident and contamination can be kept at a minimum. If working materials are arranged in an organized manner, there are fewer opportunities for confusion and there is a higher probability that a determination will be carried through to completion without error. Our lab in the chemistry department must be immaculately clean every time we are done working there; this means everything is put away in its original location, all messes and spills cleaned up, no garbage lying around whatsoever. We may jeopardize our permission to use this space otherwise, then we'd be homeless and that would just not be a very good situation.

Quantitative Transfer

Quantitative transfer is the complete transfer of a sample without loss of any kind. The techniques used are a matter of common sense: do not spill, splash, drool, or abandon. Dry solids are poured or transferred with a spatula. If the surface tension of a liquid is high it should be transferred by pouring it down a stirring rod. This prevents the liquid from running down the outside of the original container and also prevents splashing as the liquid enters the second container. Last traces are transferred by washing the original container and transfer equipment such as spatula, stirring rod and funnel with a miscible liquid, usually water. This can be done by a batch method using repeated small volumes of the wash liquid or by a continuous flow method using a stream of wash liquid from a wash bottle. A rubber policeman (a narrow rubber spatula) is used to facilitate the process and minimize the amount of wash solution necessary.

Reading Instrument Scales

The advent of digital readouts has reduced the opportunities to read analog and vernier scales. Therefore, when you encounter the need to read instrument scales or calibrations on burets and pipets, it is especially important that you pay close attention to this process. Even with digital readouts, it is possible to make serious errors of the instrument has several modes of display (e.g., Transmittance and Absorbance). Become familiar with the scale. Does it read from left to right or right to left? Top to bottom or bottom to top? Determine the significance in both units and magnitude of the largest divisions then determine the significance of the smallest divisions. The number and magnitude of the small divisions may not be the same in all ranges of the scale. With most analog scales the orientation of the operator's eye and the instrument determine the reading. If all readings occur at a fixed position on the instrument, it is only necessary that the position of the eye be the same for a set of measurements. To read a variety of positions on a horizontal scale, the most reproducible orientation is to have the eye directly above the pointer. For a variety of positions on a vertical scale the most reproducible orientation is to have the eye at the same level as the pointer. In both cases the line of vision is at right angles to the surface bearing the scale. If the surface of a liquid is to be read in place of a pointer, a reproducible position on the surface must be chosen. This is usually taken to be at the bottom of the meniscus if the liquid wets the glass and at the top if the liquid does not wet the glass. The line of vision is tangent to the bottom or the top of the meniscus. Calibrations that cover at least half of the circumference of the tube serve as a check on the correct eye level. The angle of reflection of light makes a significant difference in the appearance of the meniscus. The lighting can be controlled by holding a card against the back of the tube: for clear liquids a white card containing a broad dash line serves as the best means of reading the meniscus. The dark line is raised until it just touches the bottom of the meniscus. The top of the line is then compared with the graduations on the device to determine the value.

Readings are in general made to 0.1 of the smallest calibration division; for example, the readings with a scale calibrated to 0.10 cm are estimated to the nearest 0.01cm. Because calibration lines have width, some convention must be established for the use of the line. If the line width itself is 0.02 cm., the top of the 3.10 line could be read as 3.09, the center as 3.10 and the bottom as 3.11. A pointer also has width. Choose a point of reference such as the center, one edge, some irregularity on the pointer, etc. To record a reading as 3.1 cm states that the value is thought to be closer to 3.1 cm than it is to 3.0 cm or 3.2 cm. To record a reading as 3.10 cm states that the value is thought to be closer to 3.10 than to 3.09 or 3.11 cm. To record 3.1 cm implies one of two things. It is impossible to determine the value more carefully or the operator simply chose not to read the value more carefully. In the first case, 3.1 is the correct reading. In the second, 3.1 is an approximate reading. To make an approximate reading is lame when the more exact value is needed. To record 3.1 when the value 3.10 has been read is unscientific and, well, sloppy.

Weighing Out Samples

Weighing bottles should be completely closed after the bottle has come to room temperature in the dessicator, if you use a weighing bottle. Two methods of weighing out samples are commonly used. One is to weigh the container, add the sample and reweigh. The other, known as weighing by difference, is to weigh the closed weighing bottle, transfer the sample, and reweigh the closed weighing bottle. This last weight is also the first weight for a second sample. If the weighing bottle stands for several hours in the desiccator before taking the next sample, its weight should be checked. The first method is direct and is free from errors due to loss of sample during transfer. It does, however, leave the sample exposed to air during the weighing process. It also requires a dry receiving container that is within the weight range of the balance. The second method requires a minimum number of weighings to obtain samples. It also allows samples to be weighed into wet flasks for titration. One limitation is that if one of the middle weighings is in error, two determinations are involved. It is awkward to handle the weighing bottle with tongs, try using a band of dry paper or cloth pulled firmly around the bottle. Do not use your fingers directly on the weighing bottle as the moisture from your fingers will affect the weight.

Rules for Use of Analytical Balances

Weighing performed on the analytical balances shall never be done on weighing paper (or filter paper, paper towels, etc.). If sufficient precision is demanded to require the analytical balance it also requires the use of procedures which do not involve such high risk of loss during transfer. The second problem with use of paper is that it leads to dirty and subsequently to damaged balances. Acceptable weighing containers are weighing bottles, plastic weighing boats, and glassware with sides to contain the material.

No reagent shall be added to or subtracted from a container while in the analytical balance. Remove the container to the bench top, make the addition and return to the balance. This is the most fundamental rule of the use of the balance.

Heating and Concentrating Solutions

Aqueous solutions may be heated either on a Bunsen burner with wire gauze or on the hot plates. The burner is often a quicker method for rapid heating while the hot plate will provide a constant level of heat for a long time. Solutions other than water or dilute aqueous salts should be heated in the hoods. A watch glass should always be used during the heating of solutions both to prevent entry of extraneous material from your neighbors sample, the paint on the ceiling, etc., and to prevent loss of sample due to splattering. Ordinary watch glasses restrict the loss of vapor and are used to maintain the value of the solution. When evaporation is desirable the watch glass may be supported with three glass hooks or ribbed watch glasses may be used. These allow escape of vapors. In either case, some sample will collect on the watch glass and must be rinsed back into the container with a small volume of the solvent whenever the watch glass is removed. Boiling is generally to be avoided with samples due to the high risk of mechanical loss. When boiling is desired, boiling chips should be used whenever possible. Selection of an appropriate boiling chip requires knowledge of what may be added without causing contamination of the material to be boiled. Glass beads or chips, marble chips, silicon carbide, and many other substances have been used as boiling chips.

Handling Stock Solutions

A uniformly mixed solution may develop a concentration gradient on standing even in a closed bottle. Evaporation occurs from the surface of the liquid. Condensation takes place on the wall of the container above the surface and the condensate flows down the wall into the solution again. Shake or mix stock solutions before use. And **NEVER** put a pipet directly into a stock solution; pour what seems like sufficient (but not excessive) solution into one of your beakers, then take from that solution. Never pour any solution back into a stock bottle. The possibilities of contamination from such careless actions, and the subsequent demise and ruin of your and your fellow classmate's work are endless....

Volumetric Glassware

Flasks, Burets and Pipets

The specifications used by most manufacturers of volumetric glassware meet and usually exceed the National Bureau of Standards recommendations. Measurements of volumes larger than l0 mL can easily be made with an accuracy of 1 or 2 parts per thousand. Smaller volumes, which are so much a part of present day chemistry, may present special problems.

Because the volume of a container depends upon the temperature, volumetric equipment is calibrated for a specified temperature - usually 20°C. The coefficient of expansion of glass is so small that calibrations for 20°C are valid over the usual range of laboratory temperatures. Even at 30°C the error is less than 0.3 ppt. Tolerances for various pieces of equipment have been recommended by NBS. For example, the tolerance for a 50-mL flask or a 50-mL transfer pipet is 0.05 mL. Properly used the volume is 50.00 +/– 0.05 mL. This is a maximum error of 1 ppt. The tolerance for a l0-mL transfer pipet is 0.02 mL, 2 ppt. Note that for the smaller volume the tolerance is proportionally larger although smaller in absolute magnitude. The calibration of all volumetric glassware should be checked before it is used for work in which high accuracy is sought. Such recalibrations are also a check on the skill of the operator in the use of volumetric glassware.

Graduated Cylinders

Graduate cylinders should not be used for any quantitative measurement which is part of an analytical determination in this course. They are useful for preparing stock solutions, HPLC mobile phases, and other places where the accuracy of the measurement does not have a direct contribution to the quantitative calculation. Graduated Cylinders are very crude pieces of volumetric equipment. Nonuniformity of glass at the base of the cylinder makes the measurement of small volumes of liquid in the bottom of the cylinder particularly unreliable. Small volumes can be measured with more precision as the difference between two larger volumes.

Graduated Measuring Pipets

Measuring pipets look like a buret without a stopcock. They are frequently convenient to use but should in no sense be considered as precision equipment, largely due to the difficulty in controlling the level of the liquid. In normal use they are no better than graduated cylinders and should not be used quantitatively unless errors greater than 1% relative are expected in the results.

Volumetric Transfer Pipets

A transfer pipet has a single calibration line on tubing of small diameter and is capable of high precision. It is, however, frequently used incorrectly and becomes a serious source of error. For this reason an operator should run enough calibration checks to attain self-confidence in their technique as well as confidence in the equipment.

The proper use of these pipets is as follows:

1. Fill the pipet above the calibration line using a bulb. Mouth pipeting is not allowed. No joke.
2. If you are using a bulb, replace it with your index finger. You can leave the syringes on the pipet, if that is what you are using. Tip the pipet to an angle to prevent leakage.
3. Wipe excess liquid from the outer surface of the pipet with a clean towel or wipe. If it is not a clean tissue, you risk contaminating your solution, of course.
4. Drain the pipet until the liquid level reaches the calibration line.
5. Touch the tip of the pipet to a glass surface to remove the attached drop which is probably present.
6. Tilt the pipet to carry it to the receiving vessel. This will prevent loss of sample.
7. Drain the contents into the receiving vessel. Do not force the liquid out. Let it take its time.
8. After the pipet has stopped draining for 10-20 seconds during which time the film of liquid in the pipet will continue to drain down, touch the tip of the pipet to the edge of the receiving vessel. Do not blow out the liquid remaining in the pipet after this procedure.

You will rarely encounter a blow out pipet in a chemistry lab, but they are still common in biology labs. They are sometimes marked with two stripes near the top.

Finally, it is never proper to place your mouth on a pipet. Many solvents and chemicals are toxic or carcinogenic. In a biology lab one must also contend with pathogenic substances. Always imagine you are pipetting a sample of some horrible nasty virus or a terrible toxin. Yum.

Absolute Calibration of a Pipet

Determine the weight of distilled water of known temperature transferred by a pipet. The degree to which the weights obtained in several trials agree is a check of your technique in the use of the pipet. Using the average of the weights and the temperature of the water calculate the absolute volume of the pipet. The volume delivered depends both on the surface tension and the viscosity of the liquid. Consequently the calibration value is only valid for water or dilute aqueous solutions.

Use of a Volumetric Flask for Solution Preparation

The interior wall of the flask, particularly the neck, should be checked for uniform drainage with a small volume of the solvent. If necessary, reclean the flask. A funnel is usually used to obtain quantitative transfer of the sample. If the space between the stem of the funnel and the neck of the flask is small, air may be trapped in the flask when liquid is added. The trapped air may in turn force liquid back up the neck of the flask. This can be avoided by using a funnel with a stem that extends into the bulb of the flask or by tipping a shorter stemmed funnel so that the end of the stem touches the wall of the flask at one point. A small piece of paper between the funnel and the top of the flask will hold the funnel in this cocked position. Solids that dissolve readily in the solvent at room temperature may be added through the funnel if care is taken to wash the solid down with solvent and removed. Solvent is added until the bulb of the flask is about 9/10 filled and a uniform solution is obtained by swinging the flask in a small circle to promote swirling of the liquid without bringing it into the neck. Mixing at this time allows volume changes that accompany dilution to take place before the solution is made up to volume. Solvent is now added to bring the solution to the calibration mark. The last few drops may be added with a dropper. The stoppered flask is repeatedly inverted to obtain uniform mixing: at least l5 inversions, more if the solution is viscous. Volume changes that accompany this mixing are included in experimental error. Because a uniform solution has been prepared, solution that is now removed on the stopper in no way changes the concentration of the solution.

Volumetric flasks should never be placed on a flame or hot plate to dissolve a difficult solute. This can cause permanent changes in the volume of the flask. If a solute is difficult to dissolve, carry out the dissolution in a beaker or flask and then quantitatively transfer the solution to the volumetric flask and bring to the final volume. Ultrasonic bath cleaners can be used to dissolve solutes in volumetric flasks.

The solution is prepared at some temperature, usually room temperature. The volume of the solution and consequently the concentration of the solution is dependent on the coefficient of expansion of the solution. (If all volumes are measured in glass equipment, then it is the difference between the coefficient for the solution and the coefficient for glass that is significant.) Around room temperature the coefficient of expansion for dilute aqueous solutions is about 0.024% per °C. A change of 4°C, therefore, corresponds to a change in concentration of about 1 ppt, usually negligible by our analytical standards, but something to be aware of for future use.

Relative Calibration of a Pipet and Volumetric Flask

Relative calibrations are frequently more to the point than absolute calibrations. This is particularly true of a pipet used to remove a known fraction of a solution prepared in a volumetric flask. The flask should be thoroughly drained so that not more than an invisible film of water remains in the flask. To dry the flask would invalidate the calibration if the condition of the glass is such that droplets of water cling to the neck of the flask above the calibration mark. Being careful to handle both the flask and the pipet so that the temperature of the water is not changed, transfer the appropriate number of pipets of water to the flask. Use a label to mark the position of the meniscus. Note that the mass of the water transferred is dependent upon the temperature but that the volume transferred at constant temperature depends only on the coefficient of expansion of the glass. The coefficient of expansion of the glass is so small that for any reasonable room temperature the volume of the glass equipment is essentially constant. (The mass of water is dependent on the temperature and the coefficient of expansion of water.)

Burets

The calibration lines on a 50-mL buret are at 0.10-mL intervals. To obtain maximum precision, volumes are estimated to 0.01 mL. The calibrations on the 10-mL microburets are at 0.020-mL intervals and volumes are estimated to 0.002 mL. When using a 50 mL buret, the standard deviation of a single buret reading is assumed to be ± 0.02 mL. The two readings required in measuring the volume of reagent transferred introduces an uncertainty of $[(0.02)^2 + (0.02)^2]^{1/2} = 0.03$ mL. Thus, volumes smaller than 20 mL and 4 mL, even under ideal conditions, would have an uncertainty greater than 1 ppt. Most operators prefer to work in the 40 and 8.0 mL range respectively.

Use of a Buret to Carry Out a Titration

Teflon and ground glass stopcocks are commonly used in burets. The teflon stopcock require no lubricant. Tension on the stopcock is simply increased until the fit is sufficiently secure to prevent leakage but not interfere with the easy rotation of the stopcock. A Teflon stopcock should be stored free from tension: loosen the nut. This applies to those on burets and on other glassware such as separatory funnels. The correct assembly of the teflon stopcock has the white teflon ring next to the glass and the black O-ring next to the teflon nut which tightens the assembly. Check that assembly is correct before you attempt to use a teflon stopcock.

All stopcock burets are designed to be operated with the left hand so that the right hand is free to agitate the reaction mixture. With the scale of the buret facing the operator, the handle of the stopcock is on the operator's right. With the base of the left hand to the left of the buret, the thumb and first two fingers encircle the buret to control the handle of the plug, the last two fingers against the left of the tip. This braced position of the hand leads to maximum control of the stopcock. It also makes it possible to keep constant pull on the plug into a secure position in the seat. This is essential with glass stopcocks to avoid leakage. If initially the position of the left hand seems awkward, make a conscious effort to develop this skill. I will be glad to give advice and commiserate with your difficulties.

To fill the 50-mL buret rinse three times with 3-4 mL portions of the liquid to be used. Use a buret funnel so this liquid can be directed to flow over the entire interior surface. Do this in the buret stand to prevent any titrant that may spill from running down to your hand. Allow time for each portion to drain from the buret before the next is added. Fill the buret, including the tip, and replace the buret funnel with a buret cap. Just exactly what you do with the buret funnel is a problem. The next time it is used it can be a source of contamination due to evaporation or reaction of the solution remaining on it or to the solvent remaining in it if it has been washed. One procedure that is adequate for most reagents is to hang the funnel in the neck of the appropriate reagent bottle and cover the funnel with a watch glass. To fill the tip of the buret fully open the stopcock to allow the liquid to flow rapidly through the tip. If necessary, use a rubber bulb to exert pressure on the liquid and increase the rate of flow. Filling the 10-mL buret presents special problems because the diameter of the barrel is too small to allow liquids to be poured into the top. Consequently liquids are brought into the buret from the bottom. These burets may also be filled through the tip in exactly the same manner that a pipet is filled. This is the procedure most frequently used unless a large number of titrations are to be carried out. The tips of these burets are extremely fine and care should be taken not to pull solid particles into them. CAUTION: If titrant is spilled on the outside of the buret, it must be cleaned up and the waste neutralized and disposed of in a proper manner.

Between laboratory periods, the burets are filled to the top with the solution being used or with distilled water and left in position on the buret stand in your lab bench.

Logically one should proceed to carry out an absolute calibration of the buret. In practice the quality of the burets manufactured today is so high and the technique is so straightforward this is not necessary unless unusual precision is necessary. But it's really good practice...

To proceed with the titration bring the level of the liquid of the filled buret onto the scale and read the position after allowing a short time for the film of liquid to drain down the walls. The reading is more objective if an effort is not made to set the level at zero on the scale. You do not need to have any initial buret readings of 0.00 mL. Excess liquid on the tip of the buret is removed by washing with a stream of solvent from the wash bottle and touching the tip to a glass surface.

Using one hand to swirl the flask and the other hand to control the stopcock, add liquid at a rapid and uniform rate. Reaction in the localized region of mixing produces an indicator change. The addition of the titrant is periodically stopped and the rapidity with which the indicator returns to its color in the first solution is observed. Using this as a guide, the addition of the titrant is continued at a gradually decreasing rate. The intent is to stop when the end point has been exceeded by one drop. The tip of the buret and the walls of the flask are washed down with a small volume of solvent from the wash bottle. The process of addition and rinsing is continued until the end point decided upon in the preliminary investigation has been located within a drop or within a fraction of a drop. After a suitable drainage period both burets are read.

Fractional drops are obtained by stopping the addition before a full drop has formed. This fractional drop is then washed into the reaction mixture. The solvent added in the rinsing process increases the volume of the reaction mixture. This makes the end point more difficult to locate since the larger the volume of the reaction mixture the smaller the concentration change produced by one drop of titrant. The volume of solvent added must be adequate to bring all of the reagents into the reaction mixture at the end point. Premature washing with large volumes of solvent may reduce the precision of the work. This is particularly true when the concentration of the titrant is small. "Small" cannot be specified because it depends upon the properties of the reactants and the indicator. The time that should be allowed for drainage depends upon the volume of liquid withdrawn, the rate of withdrawal and the dimensions of the buret. The fine tip has been designed to place a maximum limit on the second. The micro burets are particularly troublesome since the surface area is large in comparison to the volume. A check on the reading after a short time indicates whether adequate drainage time had been allowed.

An overrun end point is an overrun end point. In spite of this the initial addition should be continuous and reasonably rapid. There is a limit to how long attention can be focused on drop by drop addition. If the effort invested in a sample is not large, it is well to use one sample to find the approximate volume of titrant per unit of sample taken. The estimated volume of titrant for the following samples can then be approached rapidly with confidence. Very close to the end point it is well to keep a running record of buret readings. This locates the end point between two successive readings and places a limit on the maximum error that could be involved in locating the end point.

Visual Indicators

The selection of an indicator may be a tricky business which depends upon knowing a great deal about the chemical reactions involved. Once an indicator has been selected some familiarity with that indicator should be acquired before making a serious attempt to carry out a titration. This can be done in a very qualitative manner, even to adding the titrant with a dropper to an approximate small sample in a beaker. Aknowledge of the colors involved leads to confidence and greatly reduces the time consumed in the first titrations. If a back titration is feasible, the preliminary investigation provides an opportunity to decide which color change is preferred: color A to color B or color B to color A. It is easier to titrate to a definite color change but in some cases a suitable indicator is not available and it is necessary to match a color standard. The preliminary investigation sets up this color standard and gives experience in judging how rapidly the color changes. Work with an indicator until you are confident you can judge its behavior.

Determination of an Indicator Blank

The indicator gives the "end point," the point at which the titration is ended. Ideally this would be at the "equivalence point," the point at which chemically equivalent quantities of reagents have been brought together. In practice the end point and the equivalence point may not coincide. The determination of an indicator blank gives some information on this point. Even when the indicator has been correctly chosen, a significant quantity of the titrant may be required to produce the indicator change or to react with contaminants in the reagents. In some cases it is possible to determine the quantity so used by running a blank, a titration that is equivalent in every way with the exception that the substance to be determined is not included in the reaction mixture. In order to determine an indicator blank that has meaning, the operator must be secure in their knowledge of the behavior of the indicator.