STUDENT PACKET - CHEM 14
Summer 2006

PENN STATE UNIVERSITY
DEPARTMENT OF CHEMISTRY

SYLLABUS

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Additional administrative information may be found on the Chem 14 web page at http://courses.chem.psu.edu/chem14 or the ANGEL website at https://cms.psu.edu.

GETTING STARTED

<table>
<thead>
<tr>
<th>Course-Section</th>
<th>1st Lab Meeting</th>
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<td>Chem 14-001</td>
<td>Friday, June 9, 2006</td>
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LOCATION: All of the General Chemistry Lab's are located on the first floor of the Whitmore Laboratory Building. On the first day you should go there 10 minutes before the start of your lab period and look for the posted lists which will indicate where to go for your introductory lecture. This will last for about an hour. Afterwards, we will proceed to the lab to complete the "Check-In/Orientation" experiment which is contained in this packet.
GETTING STARTED

You MUST have the following 4 items before you can be checked in to lab:

1. Lab Manual - "PSU Chemtrek" – This will be used for both Chem 14 and 15. The current edition is Aug. 2005. The Aug. 2004 version may also be used, provided that you write in the updated information (this will be provided upon request).

2. Chem 14 Student Packet, Summer 2006 - This is what you have in your hand. This contains the course syllabus and other handouts necessary for the course.

3. Eye Protection - There are 4 or 5 approved styles sold in the Penn State Bookstore. Choose a style you like. More detailed information on eye protection is given elsewhere in this packet.

4. Chem 14 Lab Kit (In a big zip lock bag, sold only in the Penn State Bookstore).

You may supply your own eye protection if you already have it, but it will need to be approved by your Teaching Assistant (Note: regular eyeglasses are not sufficient).

It would also be a good idea to buy a scientific calculator if you do not already own one. It is not important for it to be programmable, but it should be able to handle logs, exponentials, trigonometric functions and scientific notation.

WEAR APPROPRIATE CLOTHING - Lab is potentially a messy experience. A T-shirt, blue jeans and shoes (or sneakers) would be reasonable. (No sandals allowed). We reserve the right to send you home to change if you are not dressed appropriately.
THE COURSE VISION

Chem 14 is an introductory chemistry laboratory experience that gives students the opportunity to put into practice the essential principles utilized in a professional chemistry laboratory regarding topics relevant to Chem 12.

Essential Principles:

- attention to detail
- free form writing
- proper use of a lab notebook
- use of the literature
- experimental design
- interpretation of data/statistics
- an awareness of safety issues
- an awareness of environmental issues
- appreciation for what instruments can and cannot do
Dear Chem 14 Students,

Welcome to our General Chemistry Laboratory program. In the fall of 1993 we revamped our program using the "Small Scale" approach. **The philosophy behind small scale chemistry is to use the smallest possible scale that teaches the desired points.** This approach has many advantages over the traditional experiments. These are discussed in Chapter 3, "Small Scale Techniques ...".

We are one of the first major universities to adopt this approach in both our General Chemistry and our Organic Chemistry Laboratories. But, there is evidence that this is the wave of the future. For example:

- The *Journal of Chemical Education* now has an entire section of each issue devoted to small scale chemistry.

- The American Chemical Society (ACS) devoted an entire issue of one of its periodicals (ChemUnity) to small scale chemistry. One of ACS's regional directors wrote a strong editorial in favor of small scale chemistry as part of this article (a copy is attached). The entire issue has been sent to 20,000 chemistry teachers’ nation wide.

- The State of Massachusetts and the Environmental Protection Agency (EPA) have both funded the National Microscale Chemistry Center at Merrimack College. The goal of this Center is to train teachers in small scale techniques. (An article on this Center is attached.)

- *Science* published an article entitled, "Innovations in Teaching". One of the people featured in this article was Steve Thompson (author of Chemtrek). A copy of this article is attached.

I would also like to point out that this small scale trend in education parallels current developments in research and industry. The same technology which has enabled the computer chip to shrink, is enabling the analytical lab to shrink. In fact, scientists are already developing the "chemistry lab on a chip" (two articles are enclosed).

**The day is coming in which you will be able to have an analytical lab in your wrist watch!** And, as a result, chemical analyses will be faster, safer, cheaper, and involve less waste .... i.e., smaller is better for the 21st Century.

Please feel free to talk to me if you have any questions or comments regarding these developments, or any other matters regarding this course.

Sincerely,

Joseph T. Keiser
Director of the General Chemistry Laboratories
Chemistry is a laboratory science. Although it has a firm theoretical component that complements and organizes the discipline, the accepted principles are the result of more than two centuries of experimentation. Currently, at the research and development frontiers, exploration is proceeding at a feverish pace. Nonetheless, powerful forces are impeding the teaching of chemistry as a laboratory science, and some feel that these impediments may become insurmountable barriers that ultimately will regulate chemistry to something absorbed from books and not experienced firsthand by the learner-effectively turning chemistry into history.

What are these developments that are inimical to teaching chemistry by doing chemistry? They are easy to list: the growing potential of liability claims, the concern over disposal and pollution of the environment, new regulations on chemical storage and safety and, not to be underestimated, the scheduling requirements that often stultify good laboratory investigations and trivialize the laboratory experience.

Fortunately, a comparatively recent development has great promise for alleviating the pressure to deemphasize or even eliminate labs (replacement by computers is urged by a few). Small-scale chemistry is not merely a downsizing of macroscale experiments to smaller dimensions and amounts; it is proving to be the catalyst for new and exciting approaches to teaching chemistry in the laboratory. Not only does the introduction of small-scale chemistry save time, money, and space, and virtually eliminate disposal problems, but it also allows open-ended experimentation by the student and enables the teacher to make creative assignments in the wider universe of chemical transformations.

The use of small amounts of reagents considerably expands the horizon of what can be done in the lab. Experiments that were once too costly, generated too many toxic fumes, led to explosions, or took too long to accomplish can now, by imaginative use of simple equipment and the adaptation of inexpensive plastic ware, be carried out safely and expeditiously by every student-and on a Spartan budget!

Indeed, small-scale chemistry can provide the means to restore the laboratory to its proper central place in the chemistry curriculum. In addition, small-scale chemistry promotes better assessment and facilitates teaching by inquiry. Thus, it will help align the curriculum with the forthcoming National Science Education Standards.

Small-scale chemistry also can be quantitative. Simply by counting drops one can calibrate, titrate, measure gas volumes by displacement, and perform analysis with a precision that is astonishing and an accuracy that is often better than that achieved by macroscale methods. As one pursues the articles in this issue of Chemunity News, the conclusion is inescapable- the potential of small-scale chemistry to revitalize the school laboratory program is enormous, limited only by the imagination of the experimenter.

Another trend is the inclusion of more consumer chemicals and other consumer products in the laboratory procedures. Students will view chemistry differently when they are exposed in the most direct way, to the use of household materials in their laboratory experiments, Chemistry and chemicals will not longer be viewed as something foreign, far more removed from everyday life and inherently sinister. Chemicals will take their place in the minds of the students as they have already taken place in their homes-familiar everyday substances that we eat, use to clean our homes, put on our bodies, and pump into our cars, Augmenting small-scale chemistry with a substantial component of consumer materials can supply the antidote to the prevalence of chemophobia in our society, a condition that can be mitigated only by bringing chemicals and chemistry into the sphere of the ordinary in the minds of citizens. This is the ultimate benefit of incorporating small-scale chemistry and consumer products into the curriculum.

In this issue of Chemunity News exponents of the exciting developments associated with small-scale chemistry and the use of everyday materials in the teaching laboratory provide a glimpse of this rapidly evolving methodology.

The articles convey the excitement of these practitioners. They know that they are on the leading edge of an important advance in chemical education. Read what they have to say. I believe you will agree that small-scale chemistry is an idea whose time has come.

New Center Promotes Microscale Chemistry to cut wastes

The National Microscale Chemistry center (NMC²) at Merrimack College, North Andover, Mass., is up and running, and business is booming. Its first session of workshops ended last weekend. All workshops were filled to capacity, and participants—from academia and industry—have indicated that they are eager to implement microscale chemistry at their home labs.

The workshops promote laboratory waste reduction at the source and advocate improvements in the laboratory environment through application of microscale chemistry. According to the center’s executive director, Ronald M. Pike, “We plan to have an impact on the entire span of education—elementary, high school, community college, and college—as well as on industrial and governmental laboratories.”

NMC² was formally established in January 1993, although workshops were conducted informally over the past five years. The center is funded by the Environmental Protection Agency, the Toxics Use Reduction Institute of the Commonwealth of Massachusetts, and the National Science Foundation.

Microscale chemistry is a way of carrying out chemical processes by using sharply reduced amounts of chemicals. Laboratory methods, glassware, and chemical analytical techniques must be modified to carry out chemical operations on this scale. In microscale chemistry, the amounts of chemicals used are reduced from the traditional 10 to 50 g for solids or 100 to 500 mL for liquids to 25 to 100 mg for solids or 100 to 2000 µL for liquids.

The goal of the center is ambitious: It seeks to effect a cultural change in the way that chemists use chemicals. NMC² is attempting to integrate attention to chemical use reduction, air quality improvement, exposure limitation, recycling, and waste reduction into the thinking of every student. It intends to do this by providing a network for development of new microscale techniques, offering training in the techniques, and introducing microscale chemistry and methodology throughout science curricula.

With the microscale approach, waste is reduced at the source because it is never produced. Pike says that without using microscale techniques, 30,000 educational institutions currently generate more than 4000 metric tons of hazardous waste per year in the U.S. Conversion to microscale chemistry, says Pike, would markedly reduce the amount of and cost of chemical waste disposal. “In colleges alone, the conversion to microscale levels would amount to the elimination of 3960 metric tons of toxic waste annually, with a cost savings of hundreds of millions of dollars per year.”

Chemistry professor Zvi Szafrań, associate director of NMC², says: “Students who learn in microscale laboratories enjoy a more healthful (lab) environment because the air quality is better and their exposure to chemicals is reduced. They also learn to minimize their laboratory waste and to think of the impact of the chemistry they do on the environment. Since they work with reduced quantities of chemicals, they are more meticulous chemists.”

If a microscale mind-set were adopted by industry, says Mono M. Singh, and associate professor of chemistry and associate director of the center, even a 5% reduction in hazardous waste could effect a cost savings of nearly $1 billion per year. “In simple terms, microscale chemistry amounts to a total quality management approach to the handling of chemical wastes.”

The center is seeking grant funding to set up a collaborative program with elementary and high school teachers to establish standard curricula in chemistry involving microscale laboratories.

Plans for the next year include establishment of a network of demonstration projects. Currently, the only demonstration site is Roxbury Community College in Boston. The center wants to set up sites in each state for high school and elementary schools. An Internet mailing list for the network has been established. The address for subscription is microscale-1@merrimack.edu.

Plans are also under way to offer workshops specifically designed for industrial personnel next year. The long-range goal for these workshops is to impact waste disposal through a number of initiatives—for example, by establishing microscale procedures in R&D labs, by effecting waste minimization at production sites, and by microscaling the EPA standard methods. Information on NMC² is available by phoning (508) 837-5000, ext. 4384.

-Linda Ross

Stephen Thompson: Call Him Czar of Small-Scale Chemistry

Copyright American Association for the Advancement of Science Nov 4, 1994

Bulky beakers, rusty ring clamps, test tubes, and Bunsen burners aren't part of Stephen Thompson's lab. Instead, his chemistry students use "tubular integrated containers," "environmental chambers," and "sci-plexes." But don't think these tools are the latest in high-tech equipment. Tubular integrated containers, which serve as everything from clamps to filtration columns to ring stands, are really soda straws, and the environmental chambers in which chemical reactions occur are Petri dishes. Magnifying glasses are microscopes. Styrofoam cups are integral parts of a gas thermometer, and trays with microtiter wells, the mainstay of molecular biologists, hold chemicals for spectroscopic analysis.

Crude tools, yes, but each year 2800 students at Colorado State University in Fort Collins use them to analyze vitamin C concentrations, develop a chemical test for intoxication, perform redox reactions, and study acid-base equilibria. For Thompson, who directs the undergraduate chemistry laboratories at Colorado State, the methods in his madness began a few decades ago and are spelled out in his textbook, Chemtrek. And they serve several purposes.

Small-scale chemistry is cheaper, for one: Indeed, Colorado State's costs for "consumables" in student labs has dropped, Thompson says, from more than $50,000 a year to less than $5000. "Molecules are extremely small, and 50 billion of them do the same as 5000 billion," he says. Then there's the waste disposal advantage: Small amounts of chemicals mean there's less to be rid of. And best of all, according to Thompson, students forced to work with small-scale equipment draw large-scale lessons they won't forget.

Thompson is convinced the small-is-beautiful approach moves students away from following recipes and toward thinking about how chemists know what they know. It "allows people to play," he says, which, in turn, allows people to learn. Moreover, in Thompson's small world, students must build most of the instrumentation they use. "I want students to see the relationship between form and function," Thompson says. "There's a tremendous difference between the tools and instruments needed for research and the tools and instruments needed for teaching research."

For example, Thompson's students analyze a chemical not with a $15,000 gas chromatograph but with a 25-cent contraption that they build themselves using such low-tech gear as clothes pins and syringes. "Steve is trying to take the black-box mentality out of science and show that it's what's inside the black box that matters," says Frederick Stein, a physical chemist who directs Colorado State's Center for Science, Mathematics, and Technology Education. The center carries the small-scale gospel to middle school and high school teachers throughout Colorado.

In fact, Stein believes so strongly in the concept that he has helped raise money for Thompson to purchase "sci-plexes" for his undergraduates. These are computer workstations, shaped like boomerangs, where small-scale experiments are carried out on screen. The two advantages: Students can load their lab notebooks on the machines and directly enter data, and the light from the screens can be used in experiments to, say, illuminate the scattering of gas molecules that are in a Petri dish.

An impish man with bushy white eyebrows and a lilting British accent, Stephen Thompson is a born entertainer: He put himself through graduate school by working as a fire eater in the circus. Ever since, Thompson has been breathing fire into the sometimes all-too-tepid traditions of undergraduate chemistry.

-Jon Cohen

Chemistry in microreactors has been touted as faster, safer, and cheaper, so it’s not surprising that interest in such systems continues to intensify. Microreactors typically consist of plates containing microstructures that define a very small reaction volume, as well as pumping accessories for continuous operation. Now that microreactors are available off the shelf at modest cost, more and more companies engaged in fine chemicals production are testing the technology.

Recently, Sigma-Aldrich took the plunge. Last February, it installed a standard Cytos Lab System microreactor at its R&D facilities in Buchs, Switzerland. Developed by CPC (Cellular Process Chemistry Systems GmbH, Frankfurt), the system has a list price of about $187,000. Sigma-Aldrich is in good company: Sixteen other pharmaceutical and fine chemicals producers are interested in CPC’s technology, according to Sigma-Aldrich’s Fabian Wahl, manager of R&D for Europe. Wahl’s group is spearheading the firm’s technology evaluation.

The key feature of microchemical systems is the high ratio of surface area to volume. That ratio is 200 for a microreactor with a reaction volume of 1.5 mL, such as that of CPC’s, and only 0.6 for a 1-m² reactor. Mass transfer, heat transfer, and mixing are vastly more efficient in a microreactor, allowing far more precise reaction control and far better product quality control than can be achieved with conventional reactors, Wahl says.

In addition, an optimized process can be run at any scale without further R&D by simply running in parallel as many reactors as are required. Such a mode of operation is easily executed because microreactor systems typically are modular.

The technology would reduce the cost of scale-up and resources deployed to develop safe and stable processes, Wahl concludes. It is useful and practical for Sigma-Aldrich’s businesses, particularly for developing and making catalog products.

Many of Sigma-Aldrich’s catalog products are produced under typical lab conditions in flasks of up to 20 L. Of the more than 2,000 compounds in this portfolio, about 800 could be produced in microreactors with little or no process modification, Wahl says. For such cases, microreactors would reduce reaction time and cost.

For example, the condensation of 2-trimethylsilyl ethanol and p-nitrophenyl chloroformate to produce 2-(trimethylsilyl)ethyl 4-nitrophenyl carbonate requires 14 hours to complete in a conventional setup but only 18.4 minutes in a microreactor. Because the time is so brief, the possibility that the product will degrade or that by-products will form is vastly reduced, Wahl says.

Even more attractive is the opportunity microreactors offer to run problematic chemistries, such as those that are highly exothermic, produce unstable products, or form difficult-to-separate side products. Wahl’s team has explored use of microreactors for two such reactions.

The first is ester hydrolysis to produce an alcohol that readily degrades. Wahl declines to identify the ester or the alcohol. He says only that Sigma-Aldrich could not keep up with the demand for the alcohol because yield deteriorates as the reaction is scaled up: 70% at 5 L, 35% at 20 L, and 10% at 100 L.

Because Sigma-Aldrich’s original process requires an insoluble component, it could not be carried out in a microreactor. “We had to change the chemistry, but we did not know in which way,” Wahl says. With microreactors, they found out quickly.

It took less than a day to run a model reaction under 12 different conditions. A substrate that yields a stable alcohol was used so as not to complicate process development. After the best conditions were identified for the model system, it took only two hours to optimize the reaction of the actual substrate.

The second problematic chemistry tested by Wahl’s team is preparation of methylene cyclopentane from a substrate that Wahl does not wish to disclose. The reaction is highly exothermic, and process control is not good under conventional conditions. Furthermore, up to 30% of the yield consists of the more thermodynamically stable product, 1-methylecyclopentene, which is difficult to separate. For these reasons, production of methylene cyclopentane had been discontinued.

Using the CPC microreactor, Wahl’s team devised a reaction that gives 70% conversion, no by-product, and a throughput of 300 g per hour. A 70% conversion with no by-product is better than a higher conversion with some by-product because separating the product from the precursor is easier than separating it from the by-product, he explains.

Wahl expects microreactor technology also to have a positive impact on Sigma-Aldrich’s custom synthesis business, in which rapid process development is a competitive advantage. His expectation that microreactors would reduce development time by 40% has been met so far. Thus, Sigma-Aldrich is inclined to adopt microreactor technology more widely, he says.

The major drawback is the inability to work with gases and insoluble reagents. A minor one is the fact that the CPC microreactors are fabricated from steel, which is chemically sensitive. CPC is now testing microreactors made with Hastelloy, a chemically resistant material.

The CPC microreactors are easy to work with. Wahl says. The most common problem is blockage, which often is solved with one call to CPC’s hotline. Changeover from one process to another requires only a five-minute rinse with solvent.

Last month, Wahl visited Massachusetts Institute of Technology for a close look at the microchemical system being developed by Klavs Jensen, a professor of chemical engineering. The MIT system is different from CPC’s in three major ways. First, the reaction volume is in the microliter range, making lots of experiments possible with minuscule amounts of materials. Second, the reactor is made of silicon, which can be oxidized to form glass surfaces familiar to chemists. Because silicon and silicon dioxide (glass) surfaces are easy to modify, it is possible to fabricate reactors that are preloaded with catalysts, Wahl says. Third, the structured surfaces of the MIT system are covered with glass, so reactions can be observed as they take place.

The MIT system is not yet available commercially. But when it becomes available, Sigma-Aldrich will be considering it for possible adoption into its business operations, Wahl says. Meanwhile, Sigma-Aldrich will be closely following the further development of MIT’s technology, hoping for a chance to test a prototype.
GRADING

Lab Reports
For most experiments your lab report will consist of your "on the fly" notes, data, and your answers to the questions associated with the experiment. One experiment, "The Chemistry of Natural Waters" (Expt. 10) will require a formal report, which will count as the equivalent of two lab reports.
Lab reports should be handed in on time. If they are not handed in on time, substantial penalties will apply (-25 points for material which is 1 week late). Material which is more than one week late will not be accepted.

Quizzes/Test
There will be short quizzes given at the beginning of most lab periods according to the schedule. Questions may cover details of the particular experiment, general laboratory procedures, and safety. Students who arrive late for lab (i.e., after the quiz has been collected) will receive a zero on that quiz. There will also be a cumulative lab test given on the last day of laboratory. Enclosed in this packet are outlines of what will be covered on each quiz and on the lab test.

Instructor Evaluation
The instructor will assign a grade to each student based on his/her perception of each student's performance in the lab. This will include the use of the laboratory notebook, attitude, independence and technique.

Lab Monitoring
Each student will be assigned one day for which they are responsible for lab monitoring. This will normally entail an end of the period clean up of the shared areas such as the sinks, balances and the chemical supply area.

THE FINAL GRADE:

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Teaching Assistants
Most of the time you will be working with a Graduate Teaching Assistant (TA). Your TA will supervise your work in the laboratory and grade your reports and quizzes. The final grading decisions, however, will be made by Dr. Keiser.
Misc. Policies

Attendance  Each student is expected to be present in the laboratory at the beginning of each session and to remain in the laboratory for all the scheduled time unless explicit instructions to the contrary are given. A grade of zero will be assigned for any laboratory missed unless permission for a make-up has been given.

Make-up Requests  If a student has a legitimate excuse for missing a lab (e.g., family emergencies, a note from your Doctor), then they should complete a Make-up Request Form (one is on the next page) and drop it off in the mail slot on the cubicle on the 1st floor of Whitmore. If possible, you should try to attend one of the other sections that same week. If not, then most likely you will be asked to attend a special make-up experiment which will be held during your normal laboratory period on the week before the lab test. In this latter case, you will still be held responsible for the material covered on the missed experiment for the lab test.

Conflict Exams  (Usually this only effects the evening lab sections) Many Departments now schedule exams in the evenings. Sometimes these may conflict with a scheduled laboratory meeting. In these cases you are expected to come to lab. The Department which is giving the exam is expected to offer you a suitable make-up opportunity. This will normally involve giving you the same exam at a different time. (Note: most Departments have a cut off date for the scheduling of conflict exams. Be sure to check through your entire semester’s schedule for lab/exam conflicts.)

The Lab Notebook  You are expected to keep a detailed and legible laboratory notebook in this course. We will use the notebook as a cross between a professional laboratory notebook, and a lab journal (similar to a diary). Some guidelines on how to do this are given in the section of this packet entitled, "How to Keep a Laboratory Notebook”. At the end of each day, you must have the lab instructor sign and date your lab notebook. All original data and observations should be recorded in your lab notebook. Your notebook will typically be turned in at the end of each laboratory period.

Lab Neighbors  You should feel free to discuss the experiments with your neighbors, but, reports must be written up individually. If information in your notebook comes from someone else, then it must be referenced. The presentation of someone else’s work under your name (i.e., without a reference) is plagiarism. There are serious penalties for plagiarism, potentially including an “F” in the course, and an academic dishonesty “flag” on your transcript. Note: the facilitation of plagiarism, for example by posting your lab reports to the web, is also considered academically dishonest. A full listing of all Penn State policies on ethics and honorable behavior that apply to this course is given at http://www.psu.edu/ufs/policies/.

Clever Chemist Awards  We are interested in identifying and acknowledging students who go "above and beyond" the normal expectations for their lab work. This could be a clever experimental idea, or an especially impressive lab write up, or the discovery of a significant mistake in the lab manual. TA’s will make recommendations to the Lab Director. If selected, the student will receive a "Clever Chemist” award. This consists of a unique pair of safety glasses, and a gift certificate to a local store.

Office Hours  “Open” office hours are held in room 211 Whitmore- i.e., any Chem 14 student may go to any Chem 14 TA’s office hours. But, if possible, you should try to attend your own TA’s office hours, since he/she will be more familiar with your work.
CHEM 14
Make-Up Request
If this form is incomplete or illegible it may be rejected

This should be dropped off in the mail slot on the door to the Cubicle ASAP, but no later than one week after your absence.

Today's Date___________________ Your Name__________________________________________
Phone ________________________ Email Address_______________________________________
Student ID______________________________________________________________
Your TA's Name _________________________________
Your Normal Lab Day and Time _____________________
Date you missed/will miss lab________________________

Reason (attach any pertinent documentation):

If an assignment was due on the date you missed, what is its current status? i.e., is it completed? Has it been handed in? /When do you plan to hand it in?

====================================================================
Makeup Request Approved?     Yes            No
Plan to attend the make-up experiment scheduled during your normal lab period, on the week before the Lab Test.

Note: This will be a different experiment than any that were done during the summer session. The grade you earn on this experiment will replace the grade for the missed experiment. But, you will still be held responsible for the material covered in the missed experiment for the Lab Test. There are only provisions for one make-up during summer session.

Comments:

Approved by ________________ Date ________________