Dakota State University

Organic Chemistry Laboratory Manual

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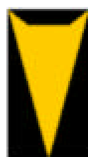

Introduction

Organic chemistry is a fascinating field. Think about it, of roughly 110 elements on the periodic chart, organic chemistry is a discipline that deals primarily with just one of these elements; carbon. And that element is so versatile and important, that organic compounds are the largest group of compounds, far outnumbering compounds made from every other element on the periodic chart. In your study of organic chemistry, remember that you are starting on the ground floor of the chemistry of life. This is where the term “organic” comes from; there was a belief that organic compounds could only come from the action of living organisms (the “vitalism theory,” one of the more famous failures to survive the test of time). The earliest chemists would categorize compounds into “organic” and “other” (or, more appropriately, “inorganic”). Now, even though we know that organic compounds can be made from inorganic compounds synthetically, the chemistry of carbon is so closely related to metabolic processes that many of these same mechanisms and reactions are used by your body.

Special thanks to Amanda Miller for her help as my photographer.

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Laboratory Safety Guidelines

Dakota State University

Chemistry Laboratories

Legal Notice: These safety guidelines are just that; guidelines. To the best of the author's knowledge, this is as complete a document as can be reasonably expected, however, following these guidelines does not guarantee that an individual may not be harmed in a lab, and because situations can arise that are not expected and there may be guidelines that have been overlooked by simple mistake, the College of Arts and Sciences, Dakota State University, and the author claim no responsibility for any reason whatsoever by those who choose to use this document. This document is provided to the general public as a courtesy; any individual, institution, or organization who chooses to use this document, either in its original or in a modified form, do so at their own risk, assume all responsibilities and, by use of this document, implicitly agree that they shall not hold the aforementioned College of Arts and Sciences, Dakota State University or Dr. Richard E. Bleil liable for injuries or accidents that occur in any lab.

Introduction

Very rarely will an injury or accident occur in a well-supervised laboratory. When an injury or accident *does* occur, it is generally brought about by complacency. In this laboratory, you will hear a LOT about lab safety; you will be given safety instructions at the start of each lab, you will be told of the major hazards of each chemical you will be using, and you will be quizzed on safety. Sometimes, such an emphasis makes a student nervous about what may be a new learning environment for them. This is an unfortunate and unintentional side effect, but it is important to give such emphasis on safety to reduce the odds of injuries in the lab by being sure that students know what hazards exist, how to avoid them and how to respond if something does go wrong. Knowledge is the best defense against injury in a chemistry lab.

The best way to prevent accidents is for you to know the possible hazards of the laboratory. Any experiment, no matter how often it has been performed in the past, has the potential to fail with hazardous results. By knowing the hazards, you will develop a healthy respect for what is happening around you, and with this respect, heightened levels of observation are sure to follow. This implies that potential accidents can be spotted *before* they can occur. If there is ever anything that does not seem right to *you*, it is not only your right, but also your obligation to point them out to *me*, your instructor. I will do my part to keep you safe, but I will need your help.

The following sections present some general guidelines. These are *not* arbitrary rules set down to make your life less enjoyable. Each and every one of them has a specific purpose, which will hopefully be made clear to you. If not, ask! There will be a safety exam which you will be required to pass (90% or greater) before the fourth lab day. Even though this grade will not be a part of your final grade, you must pass this exam to continue in the lab, so take it seriously. On the other hand, it is not designed to trick you or to be particularly difficult. If you understand the following guidelines and the reasons behind each point, you will pass the exam.

Most of laboratory safety is common sense. Remember that this is a general guideline, and therefore may be incomplete. If you are ever unsure about safety, ***please*** ask.

Laboratory Apparel

(1) Safety goggles are required in the laboratory AT ALL TIMES! Splash hazards are perhaps the most significant danger present in the lab, and eyes are extremely sensitive.

(2) Contact lenses are not permitted in the lab. Your goggles will protect your eyes from spill hazards, but do nothing to protect you from fumes, which can dry your contacts out and may result in the necessity of an operation for their removal. Contact lenses can also absorb chemicals from the air (especially the new "breathable" lenses), concentrate and hold them against the eye, and prevent proper flushing of the eye should a chemical be splashed into the eyes.

(3) Laboratory aprons must be donned at all times. In the event of a spill, these aprons are chemical and flame resistant, and could save you from scar tissue!

(4) Sandals, open-toed shoes and high heels are not permitted in the lab. This is to protect your feet from splashes and spills. The restriction on high heels is for balance.

(5) Shorts or skirts cut above the knee are not permitted in the lab. Again, should a spill occur, it will be your clothing that will be your protection from direct exposure of the skin to that chemical. The idea is to put as many layers of clothing as possible between you and a chemical spill. The more clothing, the more diffuse the chemical will be by the time it reaches the skin, and the greater the chance to remove the chemical *before* it reaches your skin.

(6) Careful consideration should be given before wearing any jewelry into the lab. Some chemicals evaporate very quickly and therefore pose relatively little danger should they get onto your skin. However, if they get beneath a ring, watch or some other form of jewelry, they can be prevented from evaporating, held against the skin longer and greatly increase the risk of injury. Should you decide to wear jewelry to the lab (as I will be wearing my watch), be particularly mindful of itching, burning or any other irritation under or around your jewelry. (By the way, NEVER wear opals, pearls, or other "soft gems" in the lab. The harsh laboratory environment may dry them out or otherwise damage them, and neither your instructor nor DSU will replace or repair such items.)

(7) Never wear clothes that hang, such as loose sleeves. Be sure ties and scarves are tucked well inside your laboratory apron. These pose fire hazards (if you are reaching or bending down near an open flame) as well as chemical hazards (if they accidentally get dragged through a chemical, they can transport that chemical directly to your skin). In fact, you may want to give very serious consideration to wearing only very old clothes. Some of my students have, in the

past, brought old clothes with them in a gym bag and changed right before and after lab. Be especially careful of sleeves around open flames..

(8) Long hair is to be constrained. Like hanging clothes, long hair is subject to fire and contact with chemicals. A rubber band *will* be used to constrain particularly long hair if necessary.

(9) No radios, tape players, CD players or any other devices of this type will be permitted in the laboratory at any time. Loud music is distracting, and headphones prevent you from hearing announcements or verbal warnings given in the lab.

Safety Equipment

(1) Take the time to identify all of the laboratory safety equipment, and keep their location in your mind at all times. You should be able to close your eyes any time during a lab and point to such safety equipment as the fire extinguisher, the emergency eyewash stations, the fire blankets, the safety shower, etc. If you were to splash a chemical in your eyes, you'd better be able to find that eyewash station without your eyes well before permanent damage can occur (which can be seconds depending on the nature of the chemical).

(2) Check all safety equipment. I'll keep as close an eye on it as possible, but I need your help as well. Is the fire extinguisher charged? Does it have the plastic "seal"? Is there enough sodium bicarbonate in case there is a chemical spill? If anything does not look right to you, report it to your lab instructor IMMEDIATELY!

(3) Material Data Safety Sheets (MSDS's) are available to you *on request only*. Basic safety information will be given during the safety lecture before each lab. Yow can also find links for MSDS's on my homepage at http://www.homepages.dsu.edu/bleilr/ if you are interested.

General Behavior

(1) ABSOLUTELY NO HORSEPLAY WILL BE TOLERATED IN THE LABORATORY! Offenders of this one will be unceremoniously cast out with a zero resulting for that day's work. I realize that at times it is awfully tempting to grab that water bottle and squirt your friends, but many hazardous chemicals look like water. The humor will be lost if something other than water is in that bottle.

(2) Always read the upcoming experiments carefully and thoroughly, being sure to understand all of the directions *before* entering the lab. This will help you to be prepared to handle any hazards of the experiment, and will also help you to perform the experiment more quickly resulting in less "fumbling around" and reckless work as you rush to finish on time. To ensure that you have read the upcoming experiment, you are required to complete the pre-lab

assignment before entering the lab. **If your fail to complete the pre-lab assignment on time, you will not be allowed to perform the experiment.**

(3) Be in the lab and ready promptly when the lab begins. The safety lecture (specific to that day's experiment) will be the first item of business each day. **If you are not present to get this important information, you will *not* be allowed to do the experiment.**

(4) Absolutely no food or beverages will be permitted inside the lab. They can absorb chemicals from the air (and concentrate them), or can pick them up from the bench, causing ingestion of these chemicals. Everything possible will be done to be sure the laboratory air is safe for working in without the use of special respiratory equipment. Please don't complicate the issue by eating these chemicals as well!

(5) WASH YOUR HANDS! Wash your hands frequently during lab, and definitely wash you hands twice at the end of the lab, once in the lab itself, and again outside of lab (as in a public rest room), ESPECIALLY before eating. Once you get home, you should wash your face as well. You don't want to drag too many chemicals around with you on your skin.

(6) Do NOT apply makeup (including Chapstick and other lip balms) in the lab. In fact, you may want to seriously consider not wearing makeup to the lab at all. Makeup can also pick up and concentrate fumes from the air, and hold them against the skin causing irritation. Perfumes, colognes or other fragrances may also interfere with the olfactory senses when an experiment calls for "smelling" something.

(7) Should an injury occur, regardless of how minor it is, report it IMMEDIATELY to the lab supervisor. The smallest puncture wound allows for chemicals to enter the blood stream directly. By notifying your supervisor, even if no action is taken, the incident will be reported to the student health center. In the event that this wound should become infected later, having this information on file may prove to be of extreme importance for prompt treatment.

(8) Never pick up broken glassware with your bare hands, regardless of the size of the pieces. Typically, puncture wounds occur with the largest pieces in such a situation, because they look to be the most harmless. A brush and dust pan is provided for broken glassware. Please place all broken glassware in the appropriate broken glassware container, and never put caps, paper or other waste in this same container. Very small bits of broken glassware (as in the bottom of a drawer) can be picked up with a damp paper towel.

(9) NEVER put broken glass in a regular garbage can. A container is provided that is especially designed for broken glassware.

(10) Always read the labels to reagents (chemicals used in an experiment) **twice**! Many chemicals look identical on first glance, and may differ only slightly in their spelling or

concentration. Sodium sulfate may look similar to sodium sulfite, but they are most certainly different and confusing them in the lab may result in dire consequences. Therefore, read the label as you grab the bottle, and holding it in your hand, look carefully at the label a second time and verify that it is exactly what you want.

(11) Never make unauthorized substitutions. If you are wondering what would happen if you used this instead of that, ask me. If it's safe, I may let you try it. If not, I'll let you know what would have happened if you tried it.

(12) Never use reagents from an unmarked bottle. All reagent bottles will have proper labels, so if a reagent bottle is unlabeled, it is the incorrect reagent.

(13) In any emergency, the fastest way to get the lab supervisors attention is to ***SCREAM***!

(14) If you are not feeling well, report it to the laboratory supervisor immediately. If your supervisor should lose consciousness during a lab period, it may be due to chemical fumes. Evacuate the lab immediately and seek another professor for help. Should anybody else lose consciousness in the lab, the lab supervisor will determine whether or not evacuation of the lab is warranted (it probably will not be).

(15) Avoid bringing excess coats, books, backpacks or other personal items to the laboratory. There is always the danger of spilling chemicals on them, and they create a fire hazard if left in the isles. In the general chemistry lab, you may use the small cabinets underneath each drawer to store personal items during an experiment

(16) Close your lab drawer! Once you have retrieved the equipment you need from your equipment drawer, be sure to close it again. Open drawers can pose tripping hazards (especially bottom drawers) and obstruction of walkways. Thump! OUCH!! The reason we do not have stools in the lab is to avoid similar obstruction.

(17) Never smell a chemical straight out of a container. Some chemicals are extremely caustic (fumes severely irritate delicate tissue) and the fumes should be avoided. To safely smell a chemical, hold it two to three feet from your nose, and with your other hand cupped, waft the fumes towards you. You may slowly move the chemical closer to your nose if you cannot smell it all the while taking only small sniffs.

Fire

(1) In the event of a fire, *DON'T PANIC*! This is probably good advice for a lot of sections of this outline.

(2) If a small portion of your clothes catches fire, the fire may be extinguished by patting it out.

(3) If a larger portion of your clothes should catch fire, there are three options for putting the flames out. (1) Drop to the ground and roll. (2) Use the safety shower. (3) Use the fire blanket.

(4) NEVER use a fire extinguisher on a person. Carbon dioxide fire extinguishers (distinguishable by their flared out nozzles) are extremely cold and may cause shock to the person or frostbite of the eyes. Chemical fire extinguishers cause excessive scarring by mixing of the chemical in the extinguisher with the damaged skin. All fire extinguishers have the potential of causing asphyxiation.

(5) If a fire should occur in a beaker or some other container, cover it with a glass dish or other flame-retardant item.

(6) NEVER move ANY object that is burning. If you try to pick up a beaker that is on fire, should you drop it, the burning chemical will spill making the situation even worse.

(7) Never use water to extinguish a chemical fire. Many flammable liquids float on water, meaning that the water will have no effect but to spread the fire. Other chemicals may even react explosively with water!

(8) If a fire is large enough to warrant the use of a fire extinguisher, the proper use of the extinguisher is as follows; (1) Be sure there is an exit behind you in case you cannot get the fire under control; (2) pull out the restraining pin (which requires breaking the plastic seal); (3) point the extinguisher hose at the base of the fire; (4) holding the extinguisher UPRIGHT, squeeze the handle to release the extinguishing media; (5) sweep the spray back and forth at the front of the fire. There are two important things to remember when using a fire extinguisher. (1) You may only have about a 30 second blast of extinguishing media, so extinguishers are only for use on relatively small fires. (2) Some fires may be inappropriate for a fire extinguisher. Be sure you have the right rating of the extinguisher, and never try to extinguish a fire on a vertical surface!

Chemicals and Chemical Spills

(1) Report all chemical spills IMMEDIATELY to your lab supervisor. The chemicals you will be handling are NOT "scaled down" chemicals-they are exactly the same chemicals any professional chemist would order and use. Keep a healthy respect for them, or they may bite you!

(2) Should a chemical spill on your person, immediate remove all affected clothing (tops from the back forward to avoid dragging the chemical across your face) and wash the affected body area with copious amounts of water. Unfortunately, chemicals have no respect for modesty

and will cause permanent damage if not treated immediately. If a large portion of your clothing is affected, immediately get to the safety shower and remove the contaminated clothing while the water is running.

(3) Small spills on the bench or floor must be cleaned up immediately. Sodium Bicarbonate and vinegar are included as part of the safety equipment for neutralization of acids and alkaline (basic) solutions respectively. Neutralize all acid and alkaline spills before cleaning. If you are not sure how to clean a spill, let your lab supervisor know immediately.

(4) Be especially careful of spills around the balances. These electronic devices are EXTREMELY sensitive to corrosion. A brush is kept near the balances so you can brush the balances thoroughly after EACH use (even a single grain of a reagent can cause irreversible damage). Clean up ANY spill near the balance IMMEDIATELY, and report it to your laboratory supervisor.

(5) Mercury, lead, and other heavy metals pose a particular health hazard in that the human body cannot get rid of heavy metals. Any heavy metals you've ever been exposed to are still with you today (including mercury if you ever played with it, or lead if you've ever eaten lead paint, a favorite activities of children as it tends to have a sweet taste). As a result, although most heavy metal poisons are not *particularly* toxic, the effects of heavy metal poisoning are typically only seen long-term, and can include uncontrolled trembling, insanity and death. The only way to combat these effects is through minimization of exposure to heavy metal poisoning. Mercury poses a particular hazard as vapors from the liquid accumulate in a room and quickly are at dangerous concentrations in the air. As a result, report ANY spills of mercury, as, for example, from a broken thermometer, as quickly as possible so it can be cleaned up immediately.

Laboratory Equipment

(1) Never heat a piece of glassware (beakers, flasks, etc.) that is chipped or cracked unless otherwise told to do so by your lab supervisor. Heating defective glassware can cause that glassware to break (or explode!), resulting in a spill.

(2) If you have chipped or cracked glassware, or glassware with sharp or jagged edges, inform your lab supervisor immediately. The equipment will probably be replaced, or you may simply be given special instructions on using that bit of equipment.

General Guidelines

(1) Epilepsy, pregnancy, dyslexia as well as other medical conditions can be hazardous in the laboratory. Every effort will be made to keep you safe, but I will need some help. *IF YOU HAVE ANY MEDICAL CONDITION WHICH YOU THINK MAY ADVERSELY AFFECT YOUR ABILITY TO SAFELY PERFORM IN THE LABORATORY, OR THAT MAKES YOU*

PARTICULARLY AT RISK TO BE IN THE LABORATORY, ***PLEASE*** *INFORM ME AS SOON AS POSSIBLE!* Many such conditions may be deemed personal, but the chemicals themselves cannot tell the difference. Therefore, please feel free to stop in my office as soon as possible so you can to tell me in private, and, of course, anything you do tell me will be kept in the strictest confidence.

(2) To turn on a Bunsen burner, first turn the nozzle on the bottom of the burner all the way off, then turn it back on about 2 turns. With a LIT MATCH in one hand, slowly turn on the gas at the spigot. Hold the match near the edge of the burner as you do so the air being pushed out by the propane does not blow it out. Such a procedure will avoid "explosions" when lighting the burner.

(3) Before using a burner, be sure nobody else on the bench has any organic solvents. Organic solvents are flammable, and heavier than air, meaning that as they evaporate, they creep down the edge of their container to the bench top, whereupon they spread out horizontally. Once these fumes reach an open flame, they can ignite causing "flashback", thereby causing the beaker of solvent to catch fire from four feet or more away!

(4) Before getting any organic solvent, be sure nobody on your entire lab bench has an open flame.

(5) Never take more of a reagent than you need. This means that if you need about 5 mL of a solvent, use your 10 mL beaker to get it, NOT your 600 mL beaker.

(6) NEVER return an unused portion of a reagent to its original container. See if anybody else at your bench, or in the lab, needs it. If not, give it to your instructor, who will look at you in a forlorn and sullen manner but will appreciate that you did not put it back in the original container. Returning unused portions of reagent greatly increase the odds of cross contamination, that is, getting the reagent contaminated with an unwanted chemical.

(7) NEVER pour a waste chemical in the drain, or put it in the garbage, unless otherwise instructed to do so by your lab supervisor. Waste bottles will be provided. Always pour waste into the appropriate and labeled waste bottle (reading the waste bottle label twice).

(8) If you have glass stirring rods or glass tubes with sharp or jagged edges, fire polish them. This means holding the sharp end in a Bunsen burner flame and rotating the rod or tube until a bright orange flame begins to show on the end being heated. Continue to heat while rotating another minute or so, effectively melting that end a little bit. Be SURE to let it cool COMPLETELY before attempting to fire polish the other end.

(9) Many items (glass, metal, etc) look exactly the same HOT they do cool. Be VERY careful whenever working with flames that ALL of your equipment (beakers, flasks, ring stands, etc.) are cool before handling them.

(10) If you are inserting glass tubing into a rubber stopper, use the following technique to avoid jamming a jagged piece of glass through your hand; (1) use glycerol or water to lubricate either the end of the glass tubing being inserted, the hole in the stopper the tubing will be inserted into, or both; (2) protect your hands by using a paper towel to hold both the glass tubing as well as the rubber stopper; (3) hold the rubber stopper in such a way that the tubing cannot go through the hole and into your palm (your fingers should actually curve, holding the edge of the stopper, as if to make the letter "C"); (4) hold the glass tubing, also with your palm away from the end, near the end being inserted into the rubber stopper; (5) insert the glass tubing with a twisting motion; (6) clean up any excess glycerol; and (7) live your life free from scar tissue on your palms that everybody for the rest of your life will ask about by saying "how did that happen?", to which you will have to reply that you didn't listen to your dedicated and caring chemistry professor.

(11) Improper heating of a test tube can result in the chemicals within the test tube shooting out, possibly resulting in injury to anybody in the path. When heating a test tube, use the following procedure; (1) unless directed otherwise, always place a few (five or six) boiling chips in the test tube; (2) use a test tube clamp to hold the test tube; (3) hold the test tube at about a 45° angle; (4) be sure the opening of the test tube is pointing *away* from anybody else (preferably towards a wall in a low-traffic area of the lab); (5) NEVER heat the bottom of the test tube (unless otherwise directed); instead heat the middle of the test tube just at the level of the liquid in the test tube; (6) move the test tube horizontally back and forth across the flame to prevent the liquid from heating too quickly; (7) should the liquid begin to overheat (heat too rapidly), remove the test tube from the flame and allow the contents to cool for a minute or so.

(12) NEVER look down the opening of ANY container, including beakers, flasks, and test tubes (as well as any other piece of equipment). Should something happen to cause the chemicals to "blast out" of the container, they will go directly into your face if you are looking down the opening at the time.

(13) Do not use graduated cylinders for any purpose other than to measure a volume of as liquid. Graduated cylinders should vat he used to get reagent for an experiment (use a beaker for this) or to run reactions (use a test tube for this).

(14) Never put a dropper into a reagent bottle. Instead, put the reagent in a beaker so you can bring it back to your desk and use a dropper there.

I hope you see that these guidelines are for YOUR benefit, and follow them faithfully; they will become habit more quickly than you can imagine. Most importantly, if you have ANY questions

or comments, *please* tell me as quickly as possible. I will be more than happy to clarify any questions you may have.

Chemistry Laboratory Name and Section Number: ______________________________

Date: ____________________________________

Name:______________________________

I, the undersigned student, have received safety training, understood it and agree to abide by the safety guidelines. I understand the importance of proper eye protection in the laboratory at all times. I have been warned about the dangers of wearing contact lenses in the laboratory and understand that I should not wear contacts in the laboratory. I also understand that if I do wear contacts in the lab or fail to abide by the safety rules, I am doing so at my own risk and will not hold Dakota State University or Dr. Richard Bleil liable for any injuries that result.

Signature of Student:DO NOT SIGN-FOR YOUR RECORDS Date:_____________

Using the Pasco System

Your first question ought to be "What is Pasco and why do I need it?" To answer that question, we need to discuss analog and digital devices (starting with the latter). Your computer is a digital device, which means it only can think in terms of "Ones" and "zeros", or, if you prefer, "on" or "off". For example, take your plain old-fashioned light switch: it only bas two settings, it is on, or it is off.

Analog devices, on the other hand, can take any valve we set. When I was in high school, my best friend was (and still is) Mitch. Now, Mitch's parents had a cleaning woman stop by once a week, who had a child of her own. She would often bring her child with her as she came to clean their house. The child took great delight in going into Mitch's room and turning the volume of his stereo all the way up. When Mitch would turn the stereo on, then, it blasted like you cannot believe. Now, if the stereo was digital, he would not have been able to turn it down; his only choice would be to turn it on or off. Fortunately, it was an analog stereo, so be could set the volume to any value be wanted between the stereos lower and upper limits.

"But wait," some of you are surely thinking, "I have a stereo at home that is digital, and I can set the volume on that stereo as well." Ours has become a digital society; digital signals are cleaner and more reliable than analog signals, so they are used for all kinds of things, like television, radio and even telephone signals. What makes these devices digital is that they "think" in terms of ones and zeros. The problem is that, while we might not care how the device works internally, we do care how devices present their output to us. If all we got out of our stereo was a stream of ones and zeros, it would not be of much use to us. We need an analog output to make sense to us (since, after all, we are analog creatures). To accomplish this, our digital devices have "digital to analog converters", or "DAC's". These convert the streams of ones and zeros into an analog signal that sounds like music to us, and even allows us to choose the volume we want.

As you might well imagine, if we can convert a digital signal to an analog signal, then we should be able to turn it around and convert analog signals to digital. We can, and, not surprisingly, to do so we need an "analog to digital converter" or "ADC". Your mobile phone has one of these (as well as a DAC) which it uses to convert your spoken (and analog) words into a digital stream of ones and zeros that it can transmit.

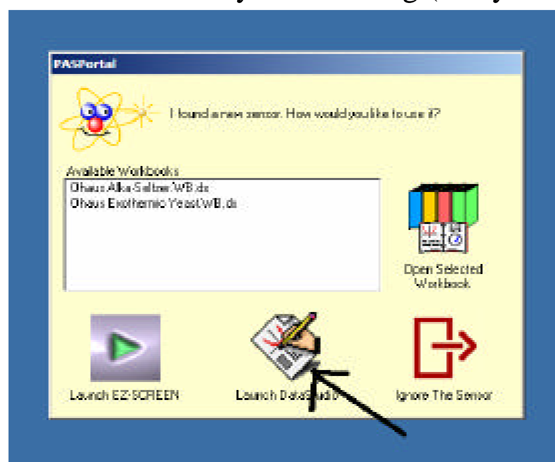
Essentially, this is what the Pasco system is: the black box (literally) is nothing more than an analog to digital converter, albeit somewhat larger than the one in your cell phone. The Pasco probes are really just devices that convert certain measurement into voltages; for example, the "temperature probes" give off higher voltages as the temperature increases. These voltages, just like temperature, are analog in nature. When you plug this probe into the Pasco box, the box convents this voltage into a digital signal, that your computer can interpret, store and manipulate.

Naturally, your computer has to know how to deal with this data, so, of course, yow will need the appropriate Pasco software. This is to introduce you to the Pasco software and provide you with the basic process for using your Pasco system to collect data on your computer.

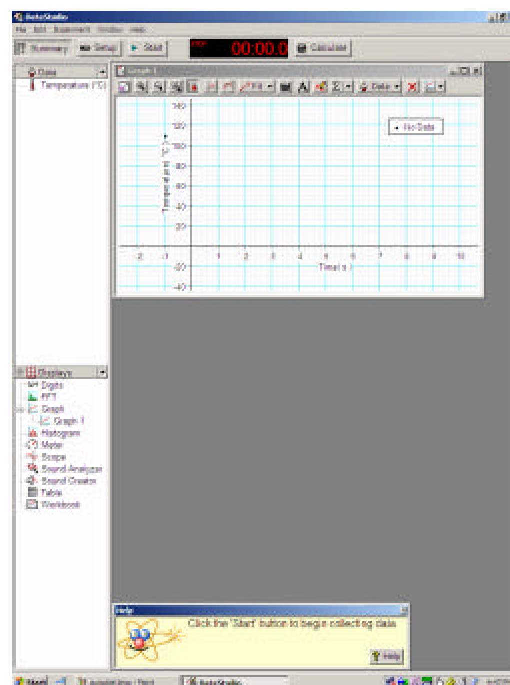
Starting Pasco

Of course, we will begin by assuming that you have already installed the Data Studio software on your computer. Make sure that you have installed both the Data Studio software as well as the PasPort hardware driver. If this is the first time you've used PasPort sensors, get the CD and install Data Studio. Keep the CD in the drive as you plug in the PasPort interface and Windows will automatically install the software. When the question is asked regarding "Windows Signing" answer "proceed anyways." Once Data Studio and the driver are installed, unplug the PasPort interface, and reboot the system.

With the system running (and you logged in), plug the PasPort interface, WITHOUT a sensor in it, into a USB port. Make sure the green light is on on the Pasport sensor front. Next, plug the probe you want into the PasPort interface; make sure the writing on the probe and the interface are in the same direction, and it should plug in smoothly. You system will recognize the sensor, and bring up a dialog screen asking you what you would like to do; choose "Launch DataStudio."



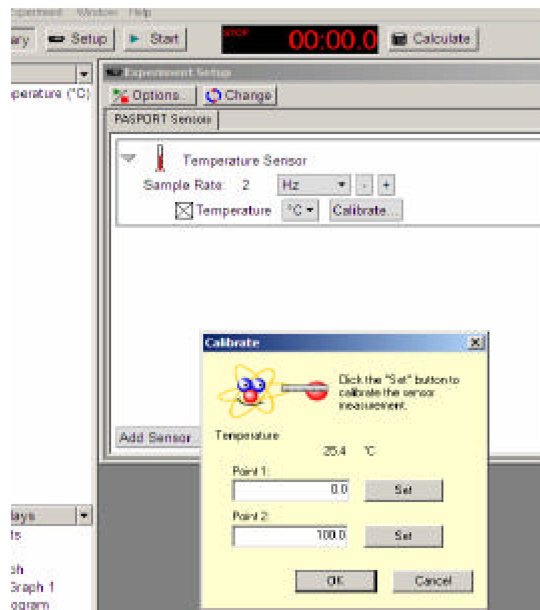
Once DataStudio is launched, it looks as if you should just be able to hit "start" and it will begin collecting data. Indeed, it would, but it might not be what you want it to do, so we must customize the software so it knows what we want it to do.



Calibration of the System

Many of the probes are pre-calibrated, however, there are times that we will want to calibrate them ourselves. The probes work by voltage; whatever they are measuring is converted into a voltage, which is read by the interface and fed to the computer. For example, the temperature probe has some given voltage that corresponds to a given temperature, and the factory calibration for this is relatively good. However, differences in manufacturing from one probe to the next means that there are small differences that might throw the sensor off a bit. For many experiments, these differences are not important and will usually cancel each other out; however, if we are doing a highly precise experiment where we need the absolute temperature (instead of the relative temperature), then we will want to calibrate the probe.

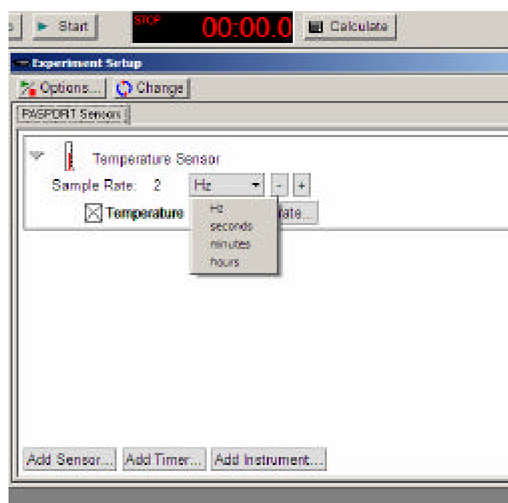
To calibrate the probe, choose the "set-up" icon near the top of the display. There you will see a variety of choices in the new dialog screen; to calibrate the probe, choose "calibrate." To perform a proper calibration, Pasco will typically ask you for two set points, a high set point and a low one. They assume that the system will act linearly; as a chemist, this kind of bothers me. I've learned a long time ago that a minimum of three points is necessary to assure linearity, but we will discuss this, as well.



The set points can be done in one of two ways; either you can measure the value relative to a source that you trust more, or you can measure a fixed point. For example, for the temperature probe, we can use a high precision mercury thermometer in the lab to compare the values with Pasco, or we can use a well-known phenomenon like melting or boiling water. For the former, simply place the Pasco temperature probe and the thermometer into the same material (perhaps a beaker of water). Give both the probe and the thermometer a minute or so to equilibrate, and read the temperature as indicated by the thermometer. Type this value in for point 1 and click "set." Notice that you will have to do this for two different temperatures; you can repeat the procedure for point 2 using a warmed or cooled beaker.

To use melting and boiling points, we take advantage of the fact that water freezes at 0°C, and boils at 100°C. For point 1, put the probe in an ice-water bath, and after it has a minute or so to equilibrate, type "0" in for the point and click "set." Do the same for point 2 in the boiling water, only type in "100" before clicking "set." This method is not as accurate as the former, because for these values to be true, the water must be absolutely pure, and the pressure must be exactly 760 torr; any deviation will result in slightly lower melting and slightly higher boiling points.

Once calibration is complete, be sure to press "OK" rather than "cancel."



Automatic Data Collection

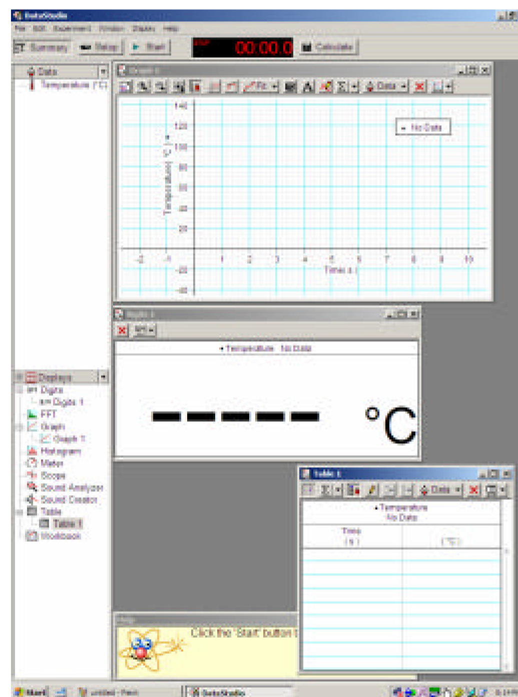
If you have not opened it yet (or you have closed it), open the "Setup" dialog box. For most probes, you will see "Sample Rate" followed by a number and a pull-down menu. This is for automated data collection. If "Hz" is in the pull-down menu, this means "per second." For example, the temperature probe defaults to 2 Hz; this means that Pasco will collect 2 data points every second, or one data point

every 0.5 seconds. If it were at 10 Hz (which you can change by pressing the "+" and "-" icons after the pull-down menu), then there would be 10 data points per second, or one data point every 1/10th of a second.

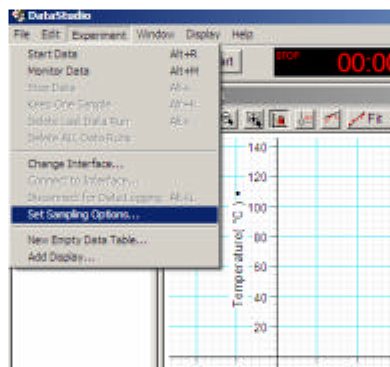
Sometimes you want this kind of rapid data collection, but usually it serves to do nothing but sop up valuable hard drive space and slow down your computer. Think about what it is you are measuring, and decide on how rapidly you would like the data points to be taken. For example, if I wanted to measure the temperature under my armpit, I don't need a temperature update every 0.5 seconds; instead, maybe I'll choose 10 seconds instead, that is, one new date point every 10 seconds. So, I will go to the pull-down menu, and choose "seconds" rather than "Hz." Then I will click "+" until I get to 10. Once I close the window, Pasco will remember my choices.

Choosing displays

Now, there are a variety of ways we can view the data as we are collecting it. The default is usually to bring up a graph, which I usually like to keep. Other options include Digits (my other usual choice), FFT (for "Fast Fourier Transform"; we usually will not use this), Histogram, Meter, Scope, Sound Analyzer, Sound Creator, Table (another common favorite) and Workbook. You will see these to the left of the screen near the bottom (if not, click on the "Displays" tab on the left near the bottom). For our armpit experiment, I want to see the digits, and keep a table of the data, so I will click and drag the digits icon and the table icon onto the view screen. For each of these displays, I recommend playing with the options so you can see what they can do. At this point, if you press "Start" you will see that Pasco begins taking data at the rate of 1 point every 10 seconds.

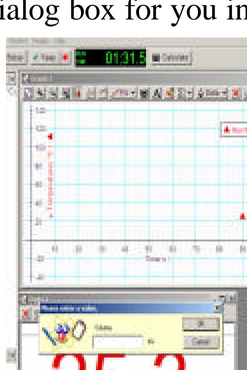


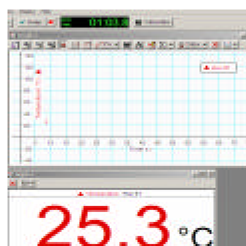
Manual Data Collection



The default data collection is time-based, but there are experiments in which you do not want the data to be collected automatically. For example, in a titration, you might want to measure pH as a function of the volume of base that you have added. In this case, at each data point, you want to tell the system exactly what the volume is according to the buret, and have it record pH as a function of that specific volume.

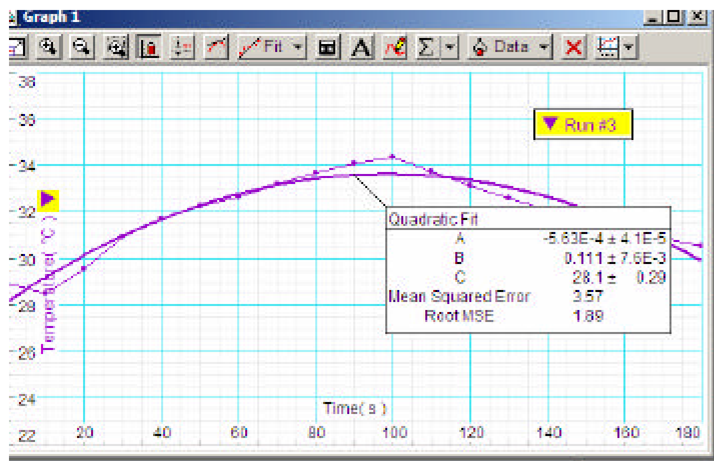
To do this, go to "Experiment" and "Set Sampling Options." This will open up a new dialog box for you in which you can be more precise in telling Pasco what you want it to do. In this dialog box, click on "Keep data values only when commanded." This will automatically choose "Enter a keyboard value…" and "prompt for a value." These are typically what you would want; the first tells Pasco to take the values from the keyboard (in our example, volume) and to prompt for this value. In "Name" put down the name that makes sense to you (such as "Volume of Base Added"), and do the same for Units (for this example, probably "mL"). Click "OK". Now, when you click "record," instead of automatically starting to collect data points, the system will begin collecting data, but not recording it. When you click on "Keep", a new dialog box will open asking you what value to associate that reading with. Type in the value you want associated with this reading, and click "OK."





Manipulating Data

Data Studio does have some ability to manipulate data. You will notice, on the right side of the screen, that each data set has been automatically stored. To delete one of these sets, just click on the data set once to highlight the specific run you want deleted, and press "del." Notice that each screen (in this case, the graph and the table) allows you to manipulate the data. For example, look at the graph. Suppose we want to expand the scale so we can see it better; to do this, simply double click on one of the scales in the graph, and choose the values you want. You can also go to "Data" and choose which data sets to display, or not. There is even a curve fitting tool that can be used. The manipulation of data depends on what you are taking and what you need to do with the data.



Exporting data

Finally, suppose you want to manipulate the data using Excel, so you can pull the graphs directly into a lab report. To do so, highlight the table so it is the active screen, and go to "File" and "Export". Choose the run you want exported, and click "OK". Save it as a "txt" file in an

easy location to find. In Excel, go to “Data” and “Import external data”, and import the file you just saved.

Using Hyper Chem

See? It's an old game now. Yow are already expecting me to start with "So what is Hyper Chem?" Well, I can't let you down. Hyper Chem is a molecular visualization and calculation package. Do you know how you wished that you could see things like molecules and orbital so they were not so abstract? Well, this is what Hype them does. At its simplest level, Hyper Chem allows you to visualize molecules in three dimensions, including zooming in or out, or even rotating the molecule so you can see it from different perspectives. Hyper Chem allows you to import molecular structures or build your own, and change the way it looks, but it can do much more.

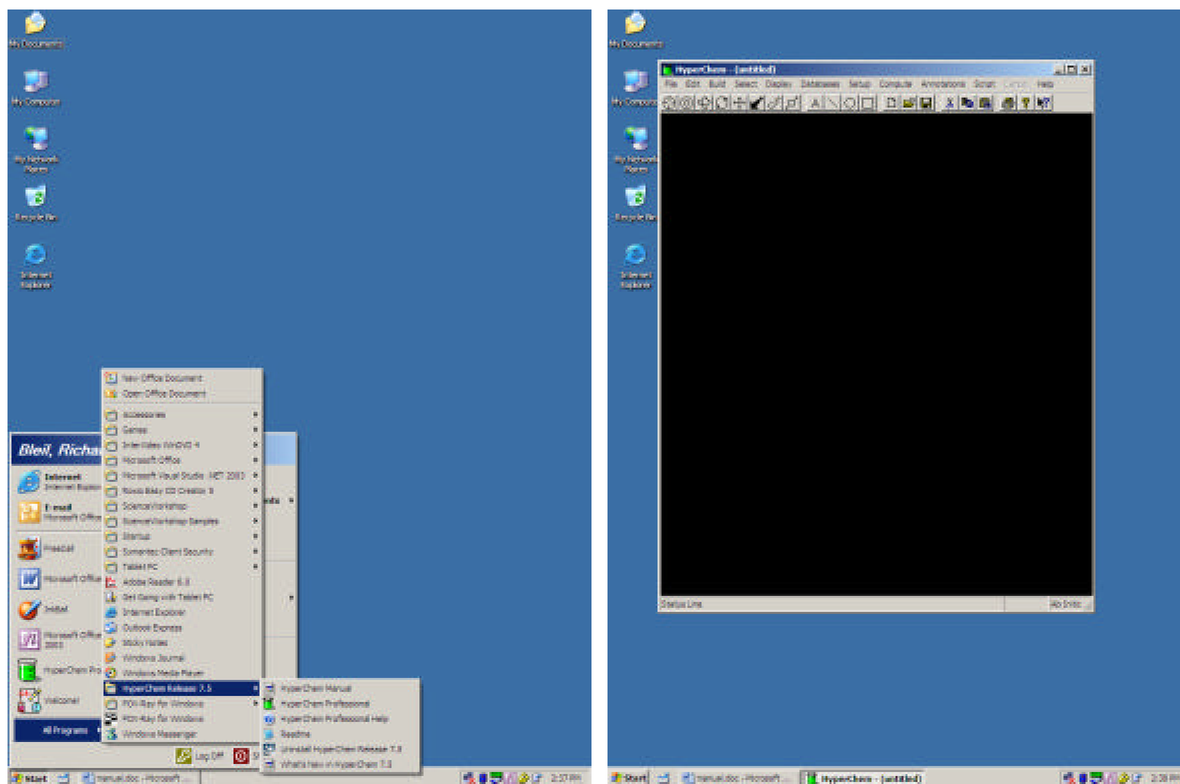
In addition to simple visualization, Hype Chem is a powerful tool for calculating the properties of molecules. At this point, we will be using these tools blindly; that is, l will not be giving you a lot of details on how it is working, but we can still get a lot out of it. Some of these are more or less automatic. For example, when you build your own molecule, Hyper Chem automatically calcula tes the most likely bond angles and lengths. In addition, Hyper Chem can do things like simulate molecular motion, or calculate and display electron orbitals. Using these tools help make chemistry feel less abstract.

Starting Hyper Chem

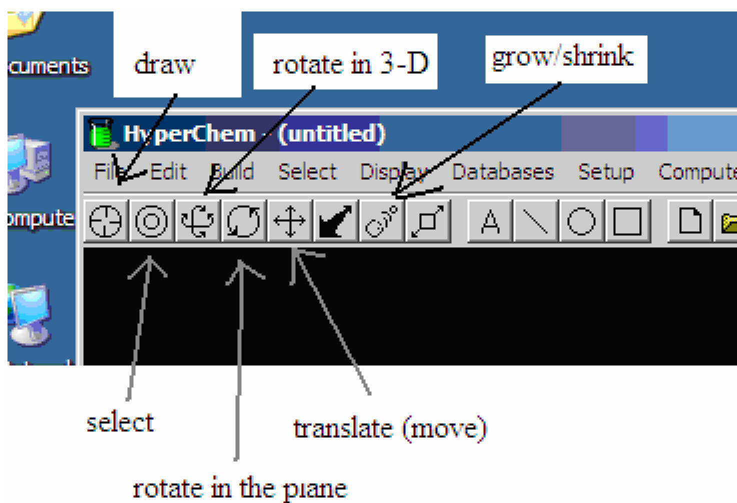
I will assume you hove already bought your copy of Hyper Chem If not, it is available for download at http://www.hyper.com (go to the student version) and can be downloaded and used for free for 30 days. However, yow will want to purchase the program for this course. In this introduction, I will show you the basics of Hyper Chem, but more specific instructions will be included with those experiments that utilize it.

Basic Hyper Chem Building Tools

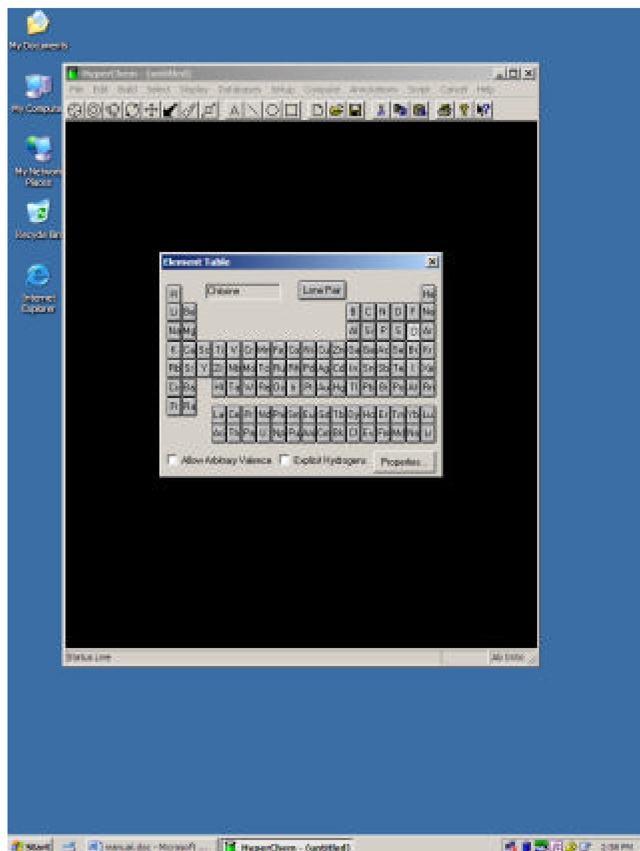
If you have purchased and installed Hyper Chem, it should be in your program files. Just start it up normally. By default, it starts with a black screen, which can be intimidating, but don't let it be.



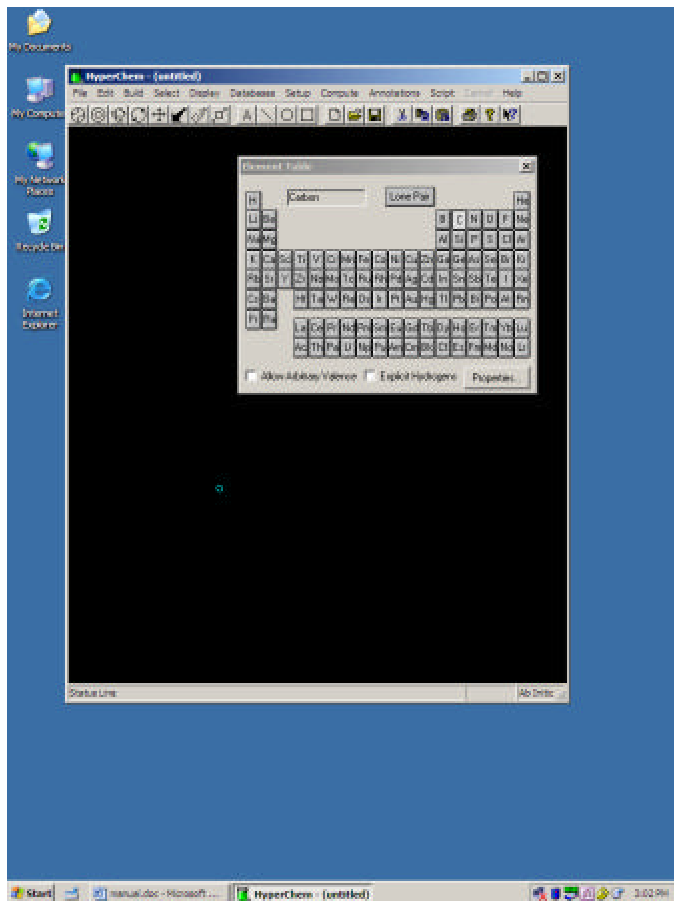
The most important keys are on the bar; although hovering your mouse above them will bring up an explanation, you will find that you quickly learn what they are and their function.



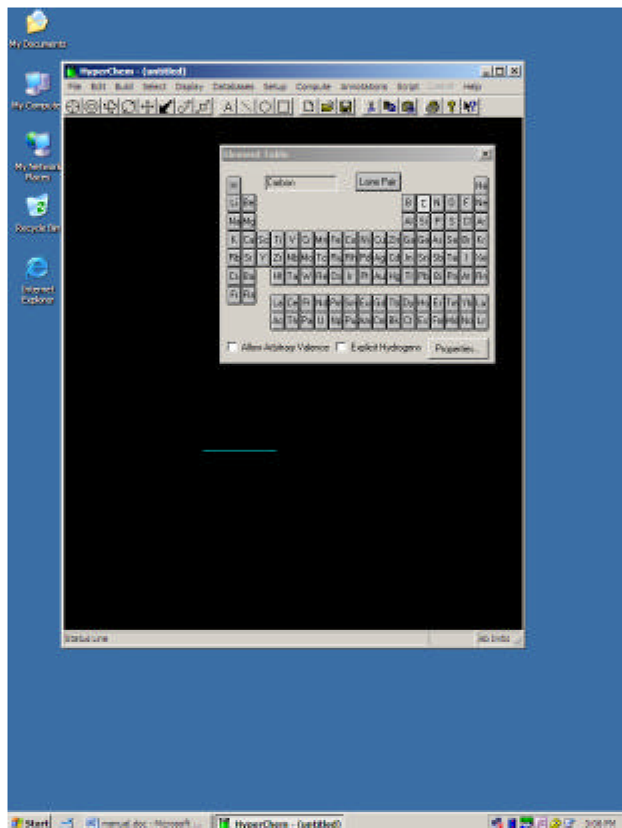
To build a molecule from scratch (say, for example, isopropanal), begin by double clicking on the draw key. This will open up a periodic chart for you (to the best of my knowledge, this is the ONLY way to get the periodic chart to open).



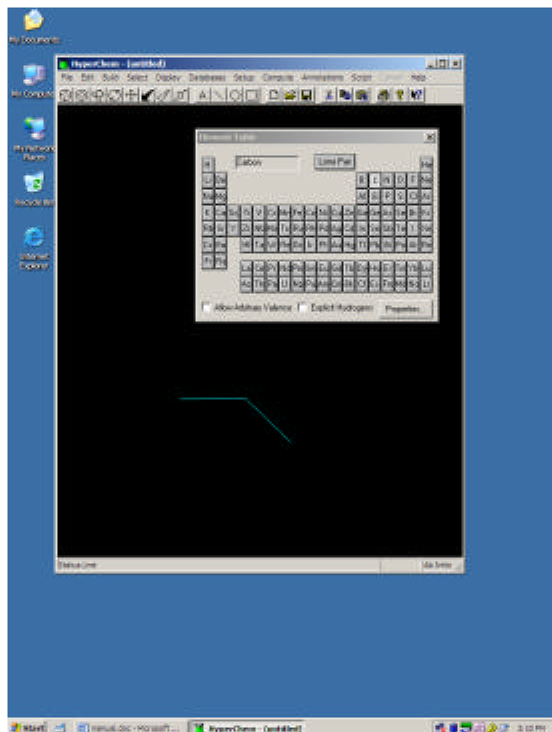
Notice that every element is here (as is on ANY periodic chart). To build our molecule, select carbon (C), and "place" a carbon somewhere in the black area. Notice that you might have to click twice to get the carbon to show (it will appear as a small blue circle); this is because when you click on the periodic chart, that becomes the active window. The first time you click on the black background, it makes the Hyper Chem window the active screen, so the second click is required to place the carbon.



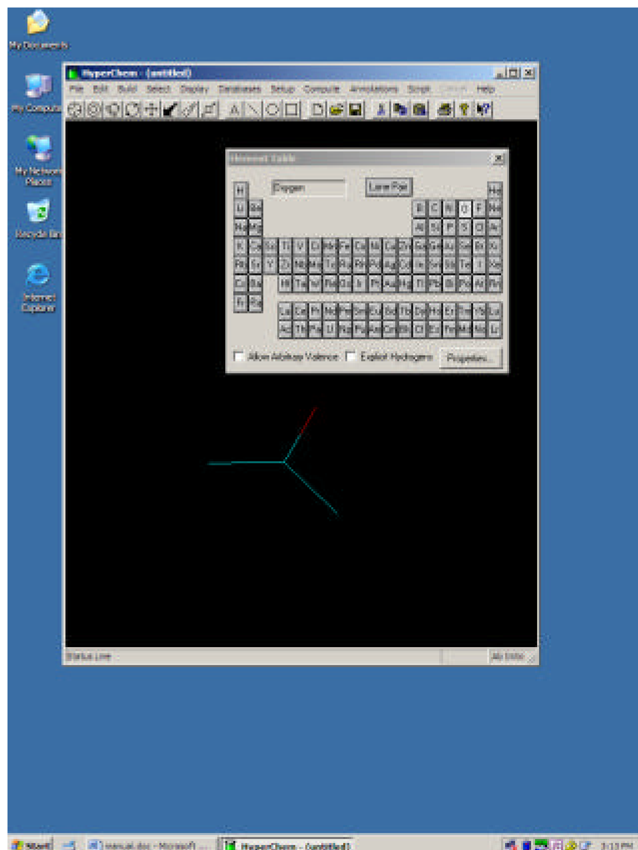
Now, we want to attach a second carbon to our first, so begin by clicking on the carbon already there, and drag a line a little ways to represent the bond. You will find that the circle disappears, and all that remains is the line. This is OK; the default rendering (that is, how the program displays molecules) is a short-hand "stick" form. This line represents the single line between the two carbons, with a carbon on each end. If the line does not draw the first time, try it again (again, if the Hyper Chem window is not the active display, the first time you click on it all it does is activate the window).



Now, we need to add another carbon to our chain of two, so, simply click on one end of the line, and draw another line. Here’s a hint, though; make the line at a slight angle, so you can easily see each atom. By doing this, there is a carbon at each end, and one carbon at each bend (in this case, only one bend).

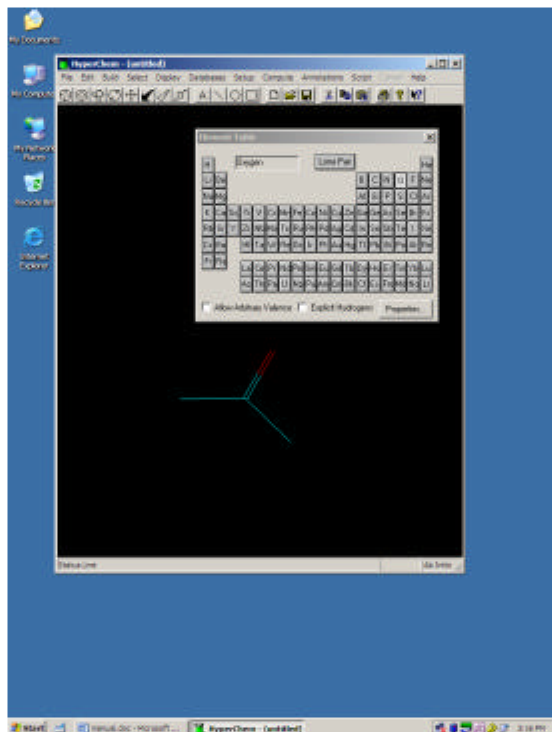


Now, isopropanal (which is present in trace amounts in isopropyl alcohol) has a double bonded oxygen to the central carbon (here, the one in the bend). So, select oxygen in the periodic chart (O), click the bend in our line (the central carbon), and draw a line up for the oxygen. Notice again that we will only get a line, but THIS time, the line is blue on one side (where the carbon is) and red on the other (to represent oxygen on the other side).

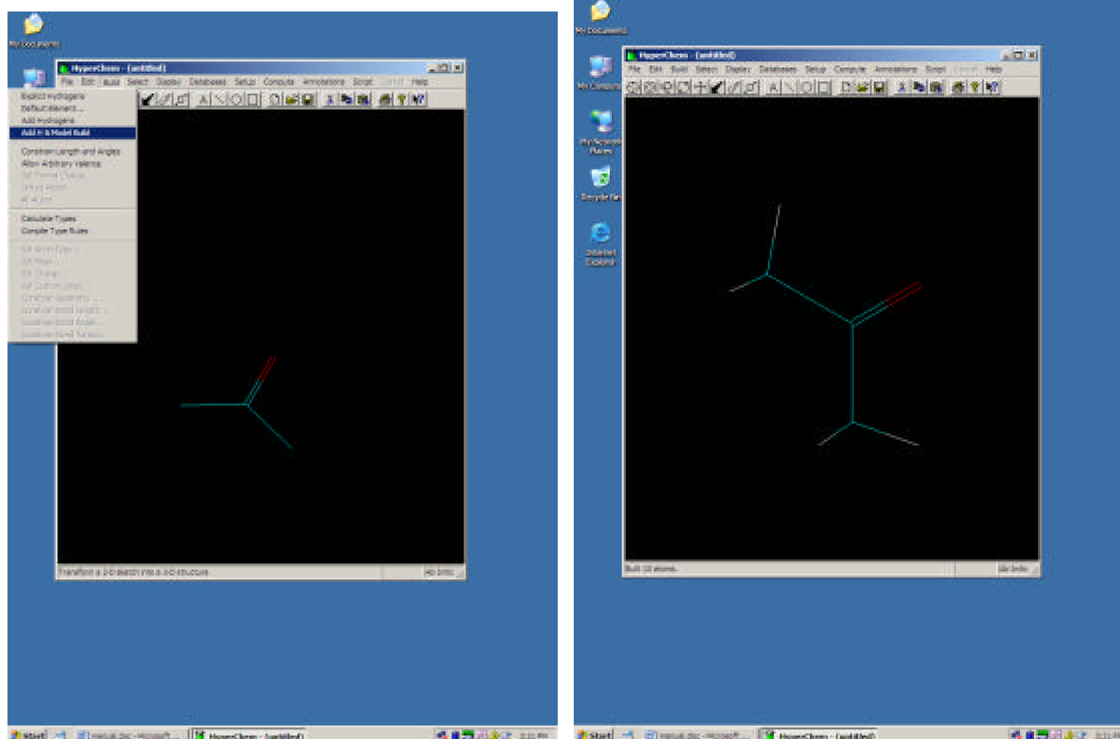


If you accidentally clicked on the middle carbon twice, you will notice that it changed from blue to red. Hyper Chem assumed you wanted to change the identity of that atom (which you did not). No problem, just choose Carbon in the periodic chart and double click the red bend to turn it back to blue; then click on Oxygen, click and drag the line from the middle carbon to put the oxygen on just as before.

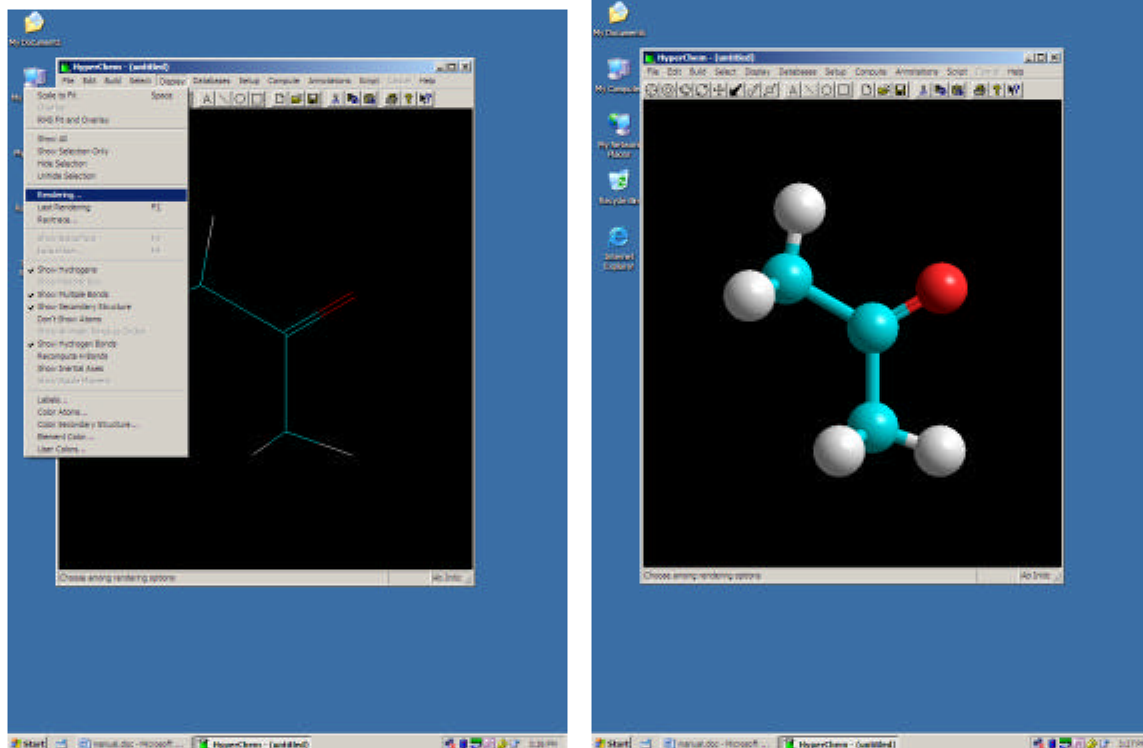
Now, we have a problem. We don't want a single bond between the carbon and the oxygen, but rather, a double bond. Well, this really is not a problem; with oxygen still selected in the periodic chart, just click once in the middle of the line between the carbon and the oxygen. You will see it change from a single line to two lines, to represent a double bond.



Now, isopropanal actually has six hydrogens, but we are not going to add them manually. Hyper Chem actually has a very nice feature to do this automatically. Close the periodic chart, and under "Build" choose "Add H & Model Build". This feature automatically completes your molecule by adding hydrogen to any "open valences" (that is, location where it is expecting another bond but there is not one) and selecting the best bond lengths and angles for all of the atoms in the molecule.



Now, I am very comfortable with this shorthand notation since I have been through organic chemistry, but maybe you are not. If you want to change the way the program displays our molecule, simply go to “display” and choose “rendering…”. I recommend “balls and cylinders” which will make the molecule look like it would if you built it out of small plastic balls (like when I was taking chemistry).



If you are following along on these instructions, I recommend now that you choose some of the other tools (begin with rotate in 3D, then rotate in the plane and translate) to see what these do. For example, choose "rotate in 3D", click and hold the molecule while moving the stylus and you will see it rotate as you do in any direction you like.

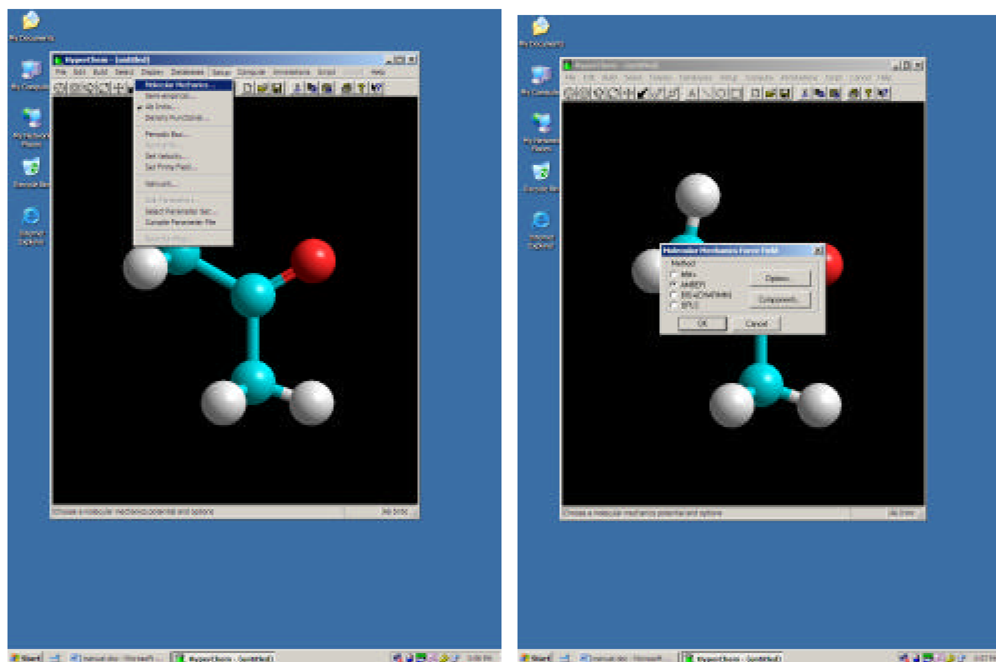
Now, obviously, this technique is suitable only for very simple molecules. If you have a much more complicated molecule, such as DNA, RNA, proteins or crystals, Hyper Chem comes with some very powerful tools built into the program to help you with these. They are found under "Databases". We will not cover these here because you probably will not need them for this course, however, I invite you to play with them. You will find they are fairly self-explanatory; it is a very simple thing to very quickly build a double-stranded DNA of any sequence that you choose.

Introduction to Hyper Chem calculations

Hyper Chem calculations are basically broken up into three basic steps; (1) define the calculation, (2) run the calculation, and (3) display the results. We will not go into this in great detail here as there are a LOT of possible calculations (an exceptionally impressive amount) that come with this package. However, we'll run through one simple one so you can see the theory; molecular dynamics. Molecular dynamics (or MD) simulates the motions of molecules by taking into account attraction and repulsion of every atom in the system with every other atom. While it must be remembered that these calculations are only as good as the programmers who developed

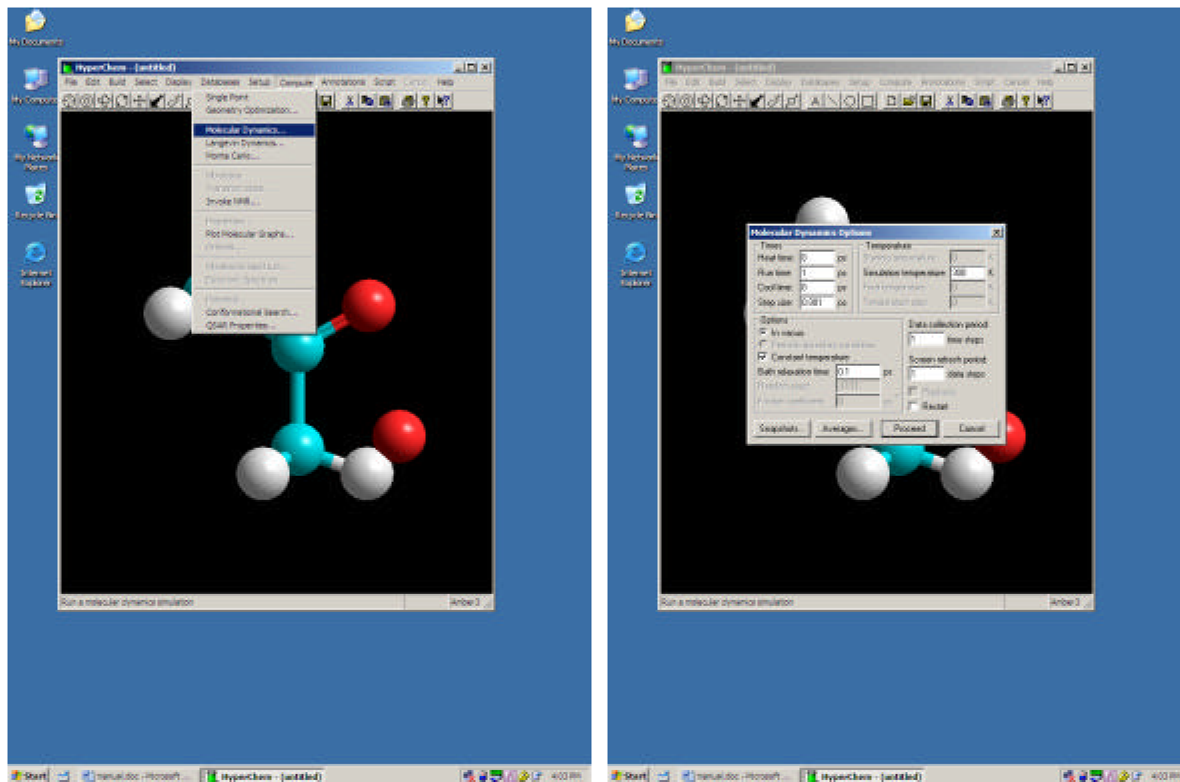
them, through the years they have become so accurate at reproducing experimental outcomes that some scientists today consider MD calculations to be legitimate experimental result in and of themselves (I disagree with this philosophy as I recognize that there will always be factors that we, as human beings, will simply not recognize, regardless of how accurate we try to get the program).

To run MD simulations on isopropanal, begin by going to "set-up", and choosing "Molecular Mechanics…". Once in there, select one of the options (I usually choose AMBER, since I am familiar with this potential function).



There are two basic ways to run MD calculations, "In Vacuo" (as I'm sure you guessed, Latin for "In a Vacuum"), or in water. If you want to add water, under "Set Up" choose "periodic box"; I will run this one in vacuo, however, since it often becomes difficult to see the molecule with a lot of water molecules around it.

Step one is complete; the calculation has been set up. Now, step 2; run the calculation. Go to "Compute" and "Molecular Dynamics"; Hyper Chem will remember your choices from the previous step. For example, my MD will run using the AMBER force field. You will notice there are more choices here, such as step size and time of simulation. Typically, the smaller the step size and the longer the run, the more accurate the results, but the slower it will be. For our purposes, I recommend simply choosing the results. You will see the isopropanal begin to dance on your screen; this is a simulation of how the molecule can be expected to actually move, if we could see it so clearly, in reality.



Now, aside from the really cool ways the molecule moves, there is not much more that we need here today. However, while this simulation was running, there were certain properties that were calculated as it ran. Although we don't need them now, if you want to display these properties (the third and final step of the sequence), go to "compute" and you will see several options that were not available before (such as "properties" and "plot molecular graphs"). Feel free to take a look at what is in these if you are curious.

Basic Laboratory Procedures

Laboratory Equipment:

Most basic laboratory equipment is made of glass so one can easily see what is happening inside. When I think of the most common equipment, the beaker and the flask come to mind.

Beakers are probably most commonly used; their wide mouth and spouts make it very easy to transfer solutions from one beaker to another. The flask (or, to be more precise, the "Erlenmeyer flask") is an excellent choice to run reactions if you do not plan to transfer solutions frequently. The tapered neck of the flask makes it very easy to grab, hold, swirl and manipulate.

Other pieces of common equipment includes the scoopula (for manipulating moderate sized amounts of solids), the spatula (for smaller amounts of solid), The stirring rod and the rubber policeman (on the end of a stirring rod, for scraping crystals out of beakers). For this lab, we will replace the thermometer with a Pasco temperature probe. Rather than droppers, we will use disposable pipettes.

Balance:

The balance is used to determine the mass of an object. Like so many other things today, modern balances just continue to get easier to use. At DSU, we use digital balances. The basic operation of these balances is trivial; place your object on the pan, and the mass appears on the display. However, also like so many modern devices, there are advanced features that may not be so obvious. In this section, I will not only

describe these features, but also the basics of maintenance that must be observed by all users and, finally, how to correctly read the display to avoid errors.

Let's begin with maintenance. Careful use of the balance is critical because of three factors that cross to make the balance, perhaps, the single most critical piece of equipment in the chemistry laboratory today. First, you will learn that there is a close relationship between the mass of a substance, and the number of molecules present. Chemists think in terms of molecules (or, more precisely, moles of molecules), but there is no instrument capable of counting the exact number of atoms or molecules. The next best option is to measure mass, which can easily be converted to and from the number of molecules. This in and of itself makes a good balance, to a good extent, the life blood of a chemist. The second factor is one of simple economics. A good quality balance easily will cost several thousand dollars, while high end ("analytical") balances will cost tens of thousands of dollars. Finally, balances are extremely precise instruments. This means that balances are very easily damaged, susceptible to both mechanical and chemical damage.

Some forms of damage are obvious, such as mechanical damage. If you drop or hit the balance, you can quickly and easily damage the mechanical components that do the work in a balance, especially the "knife edge". Modern electronics balances are also designed to word on a level, draft free surface. These high tech devices still rely on the good old-fashioned low-tech "bubble" leveling device. It is good practice to check the level bubble to ensure that the balance is level before you begin.

Balances are also very susceptible to corrosion. For the reason, you should never weigh any reagent directly on the weighing pan, even if it is a solid. Always use a piece of weighing paper, or a piece of laboratory glassware such as a beaker, flask or a watch glass. If you should inadvertently spill something on the balance, clean it up as soon as possible. Liquids must be prevented from getting inside the balance (paper towels will he near the balances) and solids must be removed as well (a brush works well for this, and will be located near the balance).

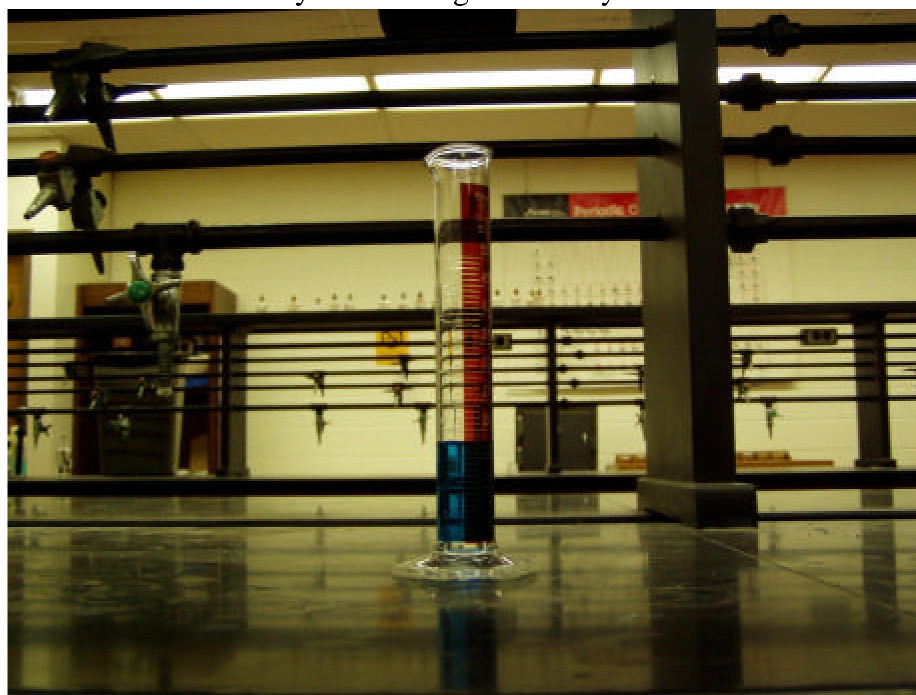
To use the balance, first you must decide what the plan is. This may sound odd, but there are a couple of ways that the balance can be used. Always cheek the bubble to be sure the balance is level. If you have both the reagent and a container ready that you want to measure the reagent in, then put the container on the balance and press the "tare" button. This will set the balance to "zero", even with the container on the weighing pan. Now, pour your reagent into the container; the mass shown on the scale is the mass of the reagent alone. Remember to add the reagent slowly, so you don't have to remove excess reagent (remember, if you do have to take some of the reagent out, do not put it back into the original container).

Sometimes, you will need the mass of the container so you can take it back to your bench, put something in it, and weigh it again. You can then get the mass of the contents by taking the mass of the beaker and contents and subtracting the mass of the beaker. This is known as "mass by difference." In this case, start by checking to see that the balance is level, and press the "tare" button to set the balance to zero. Place your container on the balance pan, and record the mass of the container. Once the container has the material in it, repeat the procedure (check to see that the balance is level, press the "tare" button to set the balance to zero, place your container on the balance pan, and record the mass of the container and material.)

Finally, just a couple of hints to improve your results when using a balance. First always use the same balance. If the balance is off slightly, thee errors will usually cancel themselves out if you are always using the same balance. Secondly, avoid drafts, vibrations or anything the that might give you an erroneous reading. The best way to do this it avoid motion near the balance when it is in use, avoid weighing objects when they are hot, and do not lean on the counter when the balance is in use.

Graduated Cylinders:

Graduated cylinders are used to measure volume. They are the most commonly used devices for volume measurement in the lab because of their accuracy, speed and ease of use. NEVER use the graduations on a beaker or flask for volume measurement; their accuracy is not sufficient for laboratory use. Most graduated cylinders are accurate to three significant figures (as opposed of flasks and beakers that are accurate to only two significant figures, and burettes and pipettes that are accurate to four significant figures).



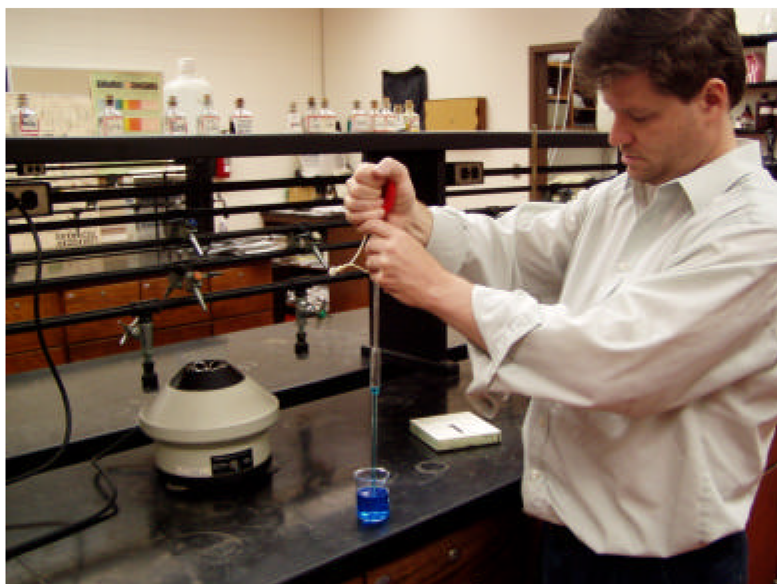
Before we begin, it is important to note that graduated cylinders are to be used for measuring volume **only**. NEVER use the graduated cylinder to mix reagents or to heat a substance!

With a liquid in the graduated cylinder, always read the bottom (or top) of the meniscus. A meniscus is a curvature to the liquid caused by intermolecular forces between the liquid and the glass. If you have attractive forces between the glass and the liquid, such as water, the liquid will "creep up" the sides of the glass slightly to cause the normal downward curvature. If these forces are repulsive, then the liquid will not move up along the walls as far as the liquid, creating an inverted meniscus. Always look past the wall of the glass, and read the volume at the center of the liquid.

Remember to estimate the last significant figure when reading the volume. This means that you simply guess how far in between the two closest graduation lines the top of the meniscus is. If it looks to you like the top of the meniscus is right on one of the graduation lines, then record an extra "0" it the end of the recorded value so the reader knows that this is the case.

<u>Pipette:</u>

Like the graduated cylinder, the pipette is used to measure volume. Unlike the graduated cylinder however the pipette is designed to measure one, and only one, volume, as indicated on the pipette. Never case a pipette that has a chipped or cracked tip, as these are no longer properly calibrated.



Begin by cleaning the pipette according to standard methods. Be especially careful to avoid bumping the tip against the sink or other surfaces. If the pipette is clean, liquid should flow out of it smoothly without leaving spots.

To use a pipette, begin by verifying that it is the correct type of pipette. Read the volume on the side (and record it in your notebook), and verify that it says "TD", not "TC". "TD" stands for "To Deliver", which means that the volume that comes OUT of the pipette is exactly the amount that the pipette is calibrated for (NOT the amount the pipette will hold). Although "TC" (or "To Contain"; that is, it is calibrated so the amount of liquid actually IN the pipette is the recorded volume) pipettes are rare, they do exist. We will ignore the "TC" procedure, and focus only on the "TD" procedure, as these are the commonly used pipettes today.

The bulbs we use in the chemistry lab have a hard plastic base attached to a rubber bulb. These give the user more finesse and better reproducibility than a mechanically designed device or a "three-port" bulb. Notice that the bulbs we use are NOT designed to fit onto the top of the pipette; they are designed to be placed there and quickly and easily removed.

Begin by holding the pipette vertically. Do not hold the pipette by the "fat" part of the glass; warmth from your finger will cause it to expand, and the pipette will lose it's calibration. Instead, hold the pipette near the top (above the calibration mark) so it is easy to get your finger over the top. Squeeze the air out of the bulb (NOT on the buret) and place the bulb on the buret top. Place the buret into the liquid to be drawn up and slowly release the pressure on the bulb, allowing the vacuum created to suck up the liquid. If your bulb completely expands before the liquid is above the calibration mark at the top of the pipette, quickly remove the bulb and cap the top of your pipette with your INDEX finger (not thumb). Squeeze the air out of the bulb, put it back on the pipette, and continue to draw up the liquid.

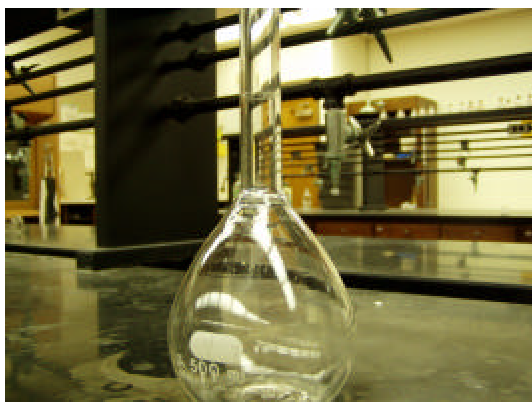
Once the liquid is above the calibration mark, take the pipette bulb off and cap the pipette with your index finger (again, not your thumb; you will have better control and get better results with your index finger). Put the bulb down, and slowly allow the liquid to flow out of the pipette until the bottom of the meniscus is right at the calibration mark. (If you are having trouble controlling the flow, try these few tricks; if you cannot hold the liquid in the pipette, moisten your finger slightly to get a better seal; instead of trying to lift your finger up to get the fluid to flow out, try rolling it slightly to one side instead; if the fluid still is too rapid, CAREFULLY put the tip of the pipette direction onto the bottom of the container with the fluid.)

Once the fluid is at the correct level, lift the pipette out of the fluid, and touch the tip to the side of the container to get any excess drops off. Put the pipette over the container you want the liquid in, and take your finger off of the top of the pipette. Holding the pipette vertically, allow the fluid to flow out on its own WITHOUT trying to force the liquid out. Once the flow stops, touch the tip of the pipette to the side of the container to get any last drops off and remove the pipette. You will notice that a small amount of liquid remains in the pipette; do NOT try to "blow" this last drop out. The pipette is calibrated to keep this amount of liquid in the pipette, so if you blow this last bit out, you have ruined the calibration and do not know precisely how much liquid you have.

If necessary, wash the reagent down the side of the container with a distilled water bottle.

Volumetric Flask:

The volumetric flask is a piece of volumetric glassware (calibrated to four significant figures) designed to contain the volume on the flask. Notice on the neck that there is a single calibration mark; when the flask is filled to this mark (with the bottom of the meniscus), it contains the volume indicated.



There are a couple of tricks that are necessary to make it easier to work with a volumetric flask. First, make sure you have the correct size lid. Plastic tops are probably better than ground glass for general purpose since ground glass tops can easily dry out and get stuck or damaged. The T/S number on the flask should be identical to the one on the stopper, so if the flask reads T/S 19, get a T/S 19 stopper.

If you are dissolving materials in the flask, once you have placed in your reagent, do not fill the flask to the graduation mark initially. Instead, fill the bulb about half full; this will allow you to swirl more vigorously to get the solid to dissolve. If you want to shake the flask, put the stopper on it first. If the solid does not dissolve immediately, add a little more water and continue. Once the solid has dissolved completely, fill the flask to the graduation mark.

Notice that if you stop here, the solution in the neck is not mixed thoroughly with the solution in the bulb. The next step is to put the stopper on it, and hold the flask such that your

palm is holding the stopper relatively firmly on the flask. Invert the flask, and swirl it relatively vigorously. Return the flask to it's upright position, allow the fluid to flow out of the neck of the flask, and repeat the procedure. Typically you will want to invert and swirl a minimum of three times.

Finally, avoid handling the volumetric flask by the bulb at the bottom. It is best to handle it with the neck above the graduation mark for the same reason that you want to handle a pipette in an analogous fashion; if you hold the bulb, the glass will warm and expand, thereby throwing off the actual volume.

NEVER heat anything in a volumetric flask, and NEVER run reactions in a volumetric flask. Only store solutions in a volumetric flask on rare occasion and for short periods of time. If you overshoot the calibration mark, do NOT try to backtrack by removing some of the fluid from the neck. While this fluid may not be well mixed yet, it does have some solute in it, and this maneuver will decrease the accuracy of the concentration. Instead, discard the solution, and start over again.

Gravity Filtration:

In chemistry, even filtration is more sophisticated than it might seem. Take gravity filtration; all you do is stick a piece of filter paper in a funnel and let it go, right? Wrong. The reason it is called "gravity filtration" is because we employ gravity to help us out, if we are careful enough.

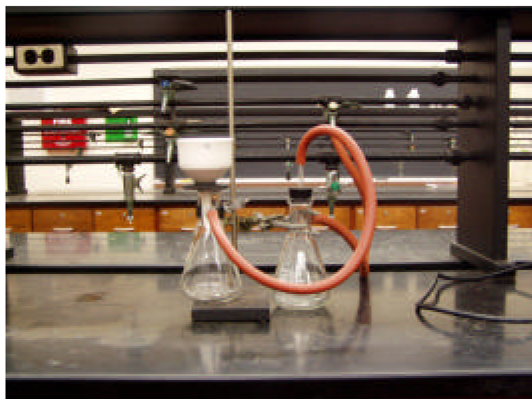
Begin with a clean long-stem funnel. Place it in an iron ring. Take an appropriate piece of filter paper (on the back of Whatmann boxes, you will find a table of types of filter paper; the slower the paper, the finer the porosity, so the longer it will take, and the smaller the particles it will catch). Fold the filter paper in half, and fold it in half again, but not perfectly; there should be a little angle, about 5°, made from the corner of the second fold when you compare the back of the folded paper with the front. Tear a small corner off of the front fold; this will help the filter paper to lie more smoothly next to the glass of the funnel so there are no bubbles between the funnel and the paper.

Moisten the filter paper completely with a bit of distilled water and carefully press the filter paper against the funnel. Be very careful to avoid tearing the paper, but you want to be sure there are no bubbles between the filter paper and the funnel. Place a clean receiving vessel underneath the funnel. Add your solution, and allow the solution to filter through the funnel naturally.

If you have set the filtration up correctly, you should see a "plug" of liquid forming in the stem of the funnel. If there are no bubbles between the funnel and the filter paper, this will create a little vacuum that will help the filtration proceed more rapidly.

Vacuum Filtration:

Choose a clean, dry side-armed flask, and secure it to a ring stand so it will not tip over. Attach a piece of vacuum hosing from the side arm, using water for lubrication if necessary, to a water trap. Attach a second piece of vacuum tubing from the water trap to an aspirator or vacuum line. The water trap prevents both liquid drawn from the faucet in an aspirator into the filtrate, and keeps filtrate from accidentally being pulled into a vacuum line. This is an important step even if you do not plan on using the filtrate because, if you need to re-filter, your filtrate will not be contaminated.



Place a collar on the top of the side-armed flask, and a Buchner funnel onto the collar. This collar is not intended to fit snuggly; it will be quite loose, but the vacuum, once applied, will draw it in tightly. Be sure that the vacuum works by turning it on and testing to see that a vacuum is created. Turn off the vacuum, and place a piece of filter paper into the Buchner funnel; the paper must not be larger than the funnel, but it must be large enough to cover all of the holes in the bottom of the Buchner funnel. Moisten the filter paper completely with a little distilled water.

Add your solution to be filtered, and turn on the vacuum. If your crystals are to be washed, first, turn off and break the vacuum by lifting the Buchner funnel slightly. Add the wash liquid to the original container, and pour it onto the crystals in the Buchner funnel. Using a rubber policeman, VERY carefully stir the crystals to break them up and wash them thoroughly, but do NOT tear the filter paper. Re-apply the vacuum. This step is usually repeated three times.

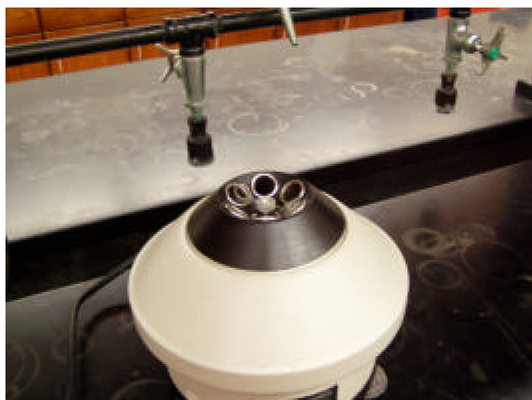
Decanting:

There are times that you would like to separate a mixture, but it is not necessary to do so with extreme care. Decanting is a method in which one can separate liquids from solids in a mixture rapidly, but relatively sloppily. Typically, one begins by centrifuging the mixture; this forces all of the solid to the bottom of the test tube (although if you are decanting from a larger container, say, a beaker, this step is obviously impossible with standard laboratory equipment). Place a stirring rod across the top of the container, not to stop the solid from flowing out, but rather to help the liquid flow out more easily because it breaks the surface tension which can form without it. Slowly and carefully pour the liquid from the mixture into another container; stop when the solid is about ready to pour out as well.

It must be kept in mind that this is not a good separation technique; it is designed to be fast and crude when this is all that is required. The solid will still have a considerable amount of liquid left on it, and the liquid will have some of the solid in it as well. Of course, do not use this as an excuse to be sloppy. It is really the analysts call as to when the separation is complete; if you try to get too precise with it, you lose the speed (which is the only real advantage), but if you are not precise enough, you may as well not be decanting at all. The only real hint I can give you is to pour slowly and try to avoid agitating the solution.

Centrifuging:

Centrifugation is a process which uses centrifugal force to separate mixtures by density; the more dense material will be on the bottom (typically solids) while the less dense will be on top (typically liquid, although a similar technique is used to separate proteins by biochemists). The most important thing about centrifuging is to balance the centrifuge; put a test tube of the same size and design opposite the test tube to be centrifuged. The test tube can be filled with tap water if necessary (do NOT dilute another solution with tap water; instead, place it in its own slot and balance the centrifuge). If the centrifuge is not properly balanced, it is very easy to severely damage the centrifuge. If you understand the concept of vectors, you can balance the centrifuge with three test tubes as well; ask your instructor for more information.



Once the centrifuge is balanced, turn it on. Allow it to run for one or two minutes. If the centrifuge begins making a lot of noise, turn it off and check the balance. You must stay with the centrifuge the entire time it is running, since minor vibrations can cause a centrifuge to “walk” off of the

bench. When you turn it off, allow it to come to a stop itself; NEVER put your hand (or anything else) above the centrifuge.

Ice baths:

Although there are times that one would like to simply pack ice or ice chips around the outside of a container to keep it cool, it is far more common to use an ice bath. Ice baths are easier to work with and tend to be more efficient, since they will make more thorough contact with the container. Start with a bath that is ice, and half or less of water; if too much ice melts (if the ice does not touch the bottom of the ice bath), pour some of the water out and add more ice, since this can create isotherms that are warmer than you would like. If you need the ice bath colder, add salt (usually low grade since it will not be used for a chemical reaction).

Litmus Paper:

Litmus paper will be blue in a basic solution and pink in an acidic solution. Because it is easier to see color changes, use blue litmus paper to test for acidic solutions (it will turn from blue to pink) and red litmus paper to test for alkaline (or basic) solutions (it will turn from pink to blue). Do NOT place the litmus paper into the solution to be tested; instead, put a piece of litmus paper on the desk, and use a clean glass stirring rod to transfer a drop from the container to be tested onto the paper. In this fashion, the same piece of litmus paper can be used many times. Read the litmus paper BEFORE it dries, and never try to use the same spot of a piece of litmus paper twice.

Glowing Splint:

The oldest trick in the book for testing gas is the "glowing splint" test. Depending on your reactants, there are a variety of gases that can possibly be given off; this simple little test helps to distinguish four of these gases: oxygen, hydrogen, carbon dioxide and nitrogen. You perform it by taking a glowing splint, lighting it on fire, extinguishing the fire such that the splint continues to glow, and using the splint to test the gases coming off from the reaction. One of three things can happen.

If the splint…	*The gas is likely to be…*
Goes out completely	Carbon dioxide or nitrogen
Re-ignites or glows brighter sometimes accompanied by a "pop"	oxygen
Explodes (in the form of a loud "pop") but does not glow brighter	hydrogen

To perform this test, there are only a few things to keep in mind. **Be sure the test tube or container is set in a holder; not held by you**. If you are holding it and it "pops," you could be

startled into dropping it. Simply take a glowing splint (looks like a tongue depresser), and start it on fire in a Bunsen burner. Allow it to burn for a few moments, so when you gently blow or shake it out, the wood is hot enough to continue to glow. Put the glowing part of the splint into the top of the test tube or container, but do NOT drop it or allow it to touch the liquid (remember you are testing the gas, not the solution). Note the IMMEDIATE reaction of the splint; if you wait too long, there will not be enough oxygen to support the glow, and the splint will give a false-positive for carbon dioxide or nitrogen.

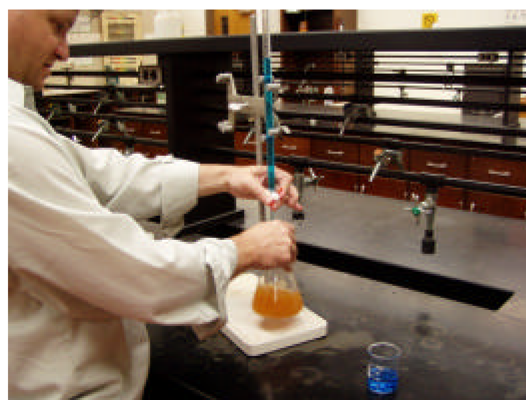
Remember that the hydrogen test is an explosion; we must keep it contained. **Never use any container other than a test tube unless otherwise instructed to do so.**

Buret:

Like the pipette, the buret is a precision instrument for measuring volumes of liquid. However, the buret is different in two major differences; first, the volume it measures is variable. Secondly, even though the buret is often read to four significant figures as well, it is not quite as accurate as the pipette. Because of human error, there tends to be larger variance in the last significant figure.

Careful inspection of the buret reveals that the volume measurements appear to be "backwards", with 0 at the top, and the maximum volume (we will use mainly 50 mL burets) at the bottom. This is because the buret is designed to show how much volume has been delivered, rather than how much it contains (like a graduated cylinder, for example).

There is a special clamp and ring stand for use with burets. Because burets are primarily used for titrations, and titrations usually require the ability to see a color change (indicating the endpoint), when you use a buret, you want to take a ring stand with a white ceramic base. Avoid using these ring stands for anything else, because the more stained they become, the harder they are to use. The clamp has two positions for burets, and is designed to hold the buret(s), and to be very easy to take the burets out of the clamp. This is for safety reasons; you must never fill a buret while it is in the clamp.



Notice the valve at the bottom. The valve is open when it is parallel with the buret (vertical), and closed when it is perpendicular (horizontal). Begin by cleaning the buret by standard methods. To fill the buret, take the buret out of the clamp. Check the valve to be sure it is closed. Pour the reagent from a beaker into the top of the buret. A funnel may be used if necessary, but is discouraged

because of mechanical difficulties this tends to produce. As you are filling the buret, at some point, pause and look at the tip to be sure the liquid is not pouring out of the bottom. If it is, take necessary action immediately to contain the reagent and clean up the spill. Fill the buret slightly above the "zero" mark and return it to the buret clamp.

The buret is still not quite ready to use, because the tip of the buret is probably still filled with air. If you try to measure the volume of liquid now, you will think you've added more reagent than you actually have because this volume of air will show in the reading. Put either the reagent beaker, or, better still, a waste beaker, under the buret tip and open the buret tip fully to expel the air and fill the tip with reagent. Sometimes it helps to "flick" the buret tip to dislodge any stuck bubbles. When the tip is full of reagent (no air remaining), close the stopcock, and check to be sure that the volume in the buret is now BELOW the zero mark. Do not waste time trying to get the volume EXACTLY to zero; it does not matter what volume you actually have, as long as you can read it (which is why it must be below zero).

Record the starting value; remember to estimate the volume to the nearest 0.01 mL (one more significant digit than the graduations on the buret). Follow the titration procedure. If you are right handed, the correct way to use the buret is as follows: use an Erlenmeyer flask (rather than a beaker) for the titration. Once you have added the titrant (the chemical to be titrated) to the flask, you can add additional distilled water as needed since this will not change the amount of the chemical already in the flask. This is convenient for washing down droplets as they splash onto the sides of the flask.

There is a proper, and an improper, way to add reagent from a buret. The proper way feels a little bit cumbersome at first, but will give you better results. Begin by noting if you are right or left handed; I will refer to them as your "dominant" and "secondary" hand. Position the buret such that the stopcock control is on the same side as your dominant hand, and the scale is facing you. The burets we use all have stopcocks that can be twisted around to accommodate your dominant hand; be careful to do this before filling the buret, though, so the stopcock does not fall out causing a chemical spill.

Now comes the part that throws most students. Even though the buret now looks like it is set up to be controlled by your dominant hand, you will actually use your secondary hand to handle the stopcock; your primary hand will be used to swirl the flask. Reach your secondary hand around the barrel of the buret and the stopcock to control the flow from the buret, and use your primary hand to swirl the flask. We do this so the tendency is to pull the stopcock in tighter, rather than looser, so we don't have to contend with a leak halfway through a titration. This is not a two-person operation; the same individual who is controlling the flask also controls the buret, and reads the volumes on the buret.

Once the endpoint is reached, read the final volume in the buret. The volume added to the flask is this volume minus the initial volume. You need not refill the buret after each run; if you still have enough liquid in the buret for a second run (which should have the same volume as the first run), you can just use the liquid that is already in the buret. Only refill the buret if necessary (and remember; do NOT refill the buret in the buret clamp!). If you underestimate the volume in the buret, do not try to "save" the run by letting the liquid run below 50 mL in the buret, or by adding liquid mid-titration. These are designed to be exceptionally high precision

experiments; either of these techniques will introduce undue error into your calculations. Just refill the buret, and do another run.

Cleaning laboratory Equipment:

Finally, we come to the general process of cleaning laboratory equipment. This is critical; contaminants can have a profound influence on an experiment, and since equipment is shared, you will want to be sure the glassware is clean from the other groups. Heck, you don't know what they did! So how does one go about cleaning laboratory glassware? Easy; you make your lab partner do it.

But what if YOU are the lab partner? OK, well, it's really not that bad. The first thing you will want to recognize, however, is the simple fact that glassware is easiest to clean before the gunk has the opportunity to dry into it. However, the same basic rules apply even if it is old. Begin by rinsing very well with tap water (surprised?). You will find an assortment of brushes near each sink; rinse out as much of the bulk of the contaminants as possible. Then, still using tap water, add Alconox. Alconox is a white cleaning agent at each sink; it is a detergent made BY chemists FOR chemists. This is good stuff; it's like Lava soap with an attitude. Use the brush to clean the glassware thoroughly; if you are cleaning something that cannot be cleaned with a brush (like a pipette); dissolve a little Alconox in a beaker of water and run it through as best you can.

After cleaning with Alconox, rinse the glassware VERY THOROUGHLY with TAP water to get rid of all of the excess soap. If you are not happy with how clean it is, repeat the step with Alconox. Finally, rinse it THREE TIMES with SMALL amounts of distilled water (in the large carboys near each sink). Do NOT allow the distilled water to run the entire time during the rinsing process; open the spicket just long enough to allow a small volume (maybe 5 mL) out, and close it again. Rinse thoroughly and repeat two more times. It does take time to make distilled water, so if we run out during a lab, there is nothing I can do about it. If you are working with something like a pipette, run distilled water through it using a wash bottle.

If the glassware is clean, the water should form a smooth sheath as it runs off of the sides. If there are any spots where a "bubble" forms, you might want to re-clean the glassware. At this point, decide if you need to dry the glassware or not. If you are washing it for next week, it will dry without spots after the distilled water rinse. If you are going to put water based solutions into it, you might not have to dry it. If it is a qualitative lab and the exact concentration of the reagent is not so important, you can pour the reagent directly into the wet container. If, on the other hand, it is a quantitative lab, you might want to repeat the rinse cycle with even smaller volumes (say about 1 mL portions) of the reagent you will use. For example, draw a little of the reagent up into the pipette, wet the entire inside of the pipette, and repeat three times, discarding each wash. On the other hand, if you are working with something like and organic solvent, you will want to dry the container with paper towels before proceeding.

One of the easiest way to dry a test tube is to begin with a paper towel twisted small enough to fit inside. This in and of itself may not sound terribly insightful; however, once you put the paper towel inside the test tube, twist the paper towel in the opposite direction of the twist

you used on the paper towel itself. This will force the paper towel to open up, and, therefore expand, to dry the test tube.

Organic Laboratory Equipment and Procedures

Introduction:

Organic chemistry lab is different from general chemistry. Organic compounds are delicate, and require a delicate touch for success. In organic chemistry, you will have to learn to be more careful, clean and observant than you were in general chemistry.

The organic chemistry lab will introduce you to several new techniques and pieces of equipment. Here, we will show you both the standard scale, and the microscale equipment and how to use it, along with the goal of the equipment and/or procedure.

Product Formation:

Stirring Hotplates:

Organic reactions can take quite a while to complete, and often need constant stirring, heat or both. This is where "stirring hotplates" come into play. Standard stirring hotplates have a heating element with built in temperature controls. In addition, it has a magnet on a motor inside. By placing a stir bar, which is a Teflon coated magnet, in the container on the hotplate, the stir bar is made to rotate by aligning itself with the magnet in the stirring hotplate as that magnet rotates via the motor. This way, one can heat a container without worrying about burning the contents.

There are other forms of heating sleeves; sometimes they have built in control devices and sometimes they do not. If no control device is apparent, you might need a "variac", a variable AC power device. Do not plug these directly into the power, since this will cause them to automatically heat to full power.

Sand, Oil and Water Baths:

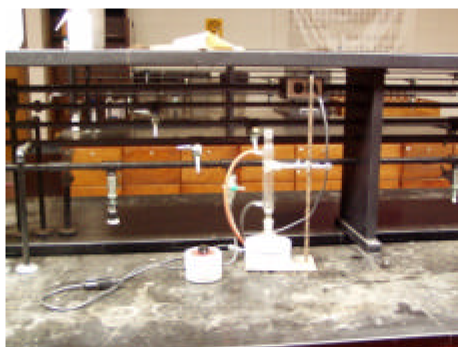
One of the shortcomings of stirring hot plates is uneven heat distribution. Often, it is important to surround the reaction vessel with heat, rather than simply have heat at the point of contact. When this occurs, one might consider using a sand or water bath. These are just as they sound; containers filled with sand or water that the reaction vessel goes into.

Naturally, you want to be sure there is enough sand, oil or water in the bath to cover an appropriate amount of the reaction vessel. The most important thing to remember is that, while baths provide more consistent and uniform heating, you must be patient with them. They take longer to heat up than most people are used to, and because they take a longer time to heat up, they also take longer to cool down. Thus, if you try to heat them up more rapidly by increasing the temperature of the hotplate, you can easily overshoot your mark. It's always much easier to heat them up slowly rather than trying to cool them down later.

The difference between the three baths depends on your needs. Water baths have the lowest limit to heating (only 100°C; why?), but you do want to be careful not to allow the volume of water to get too low. Also be careful, because even though there is an upper limit to this temperature, remember that water boils at a much higher temperature than most of the organic solvents.

Oil and sand baths can become much hotter. Be careful with oil, though, as it is flammable, and oil can begin to smoke if they are heated too hot.

Reflux Condensers:



Reactions often take a long time to run, as mentioned previously, but you might wonder what a "long time" means. There are reactions that can take days or even weeks to complete; in this case, you want to be sure that the solvent does not go dry. There are two ways to do this; one is to stay with it the entire time the reaction is occurring so you can add solvent manually as needed. The easier method, however, is with a reflux condenser.

A reflux condenser is designed to capture solvent vapor and return the solvent to the reaction flask. Typically, one would use a round-bottom flask. Make sure the condenser fits the flask correctly. Always use a ring stand and clamps to secure the reflux condenser. Be sure the water goes in at the bottom and out through the top. The top of the condenser is kept open to the atmosphere; if it is closed off, pressure can build up and cause a problem. However, since the vapor will condense and drip back into the reaction vessel, keeping the reflux condenser open to the atmosphere will not cause a problem. More than one reflux condenser can be hooked up in series if necessary. If you are letting it run overnight, be sure the water is on as little more than a trickle. Often, building water pressure increases in the night due to less water usage, which can cause the hose to pop off.

Inert Atmospheres:

Here is something that sounds exceptionally redundant; oxygen is a strong oxidizing agent. The problem is that organic compounds are typically very easy to oxidize. So, frequently, one must keep oxygen away from the reaction. To do so, we need an "inert atmosphere."

This is typically a noble gas, such as helium, neon or argon, or nitrogen. Often, the gas in pumped into a rubber injection port with a syringe tip. Keeping the atmosphere inert, however,

is the real trick. This is done with a simple rubber balloon. If the pressure increases, the balloon will expand, but if pressure drops, the balloon will allow more gas in.

Product Identification/Verification:

Melting Point Determination:

Melting and boiling points are critical not only for product identification, but also for verification of purity. Recall from colligative properties that solutions always have a lower melting point and a higher boiling point than pure solvent. Thus, if your product is not pure, your boiling point and melting points won't be exactly where they should be.

OK, I can hear your cry, "then how can it help with identification?" Well, there is a hint that the material is or is not pure. See, if the sample is impure, the melting and boiling points will not only be off, but will also have a broader range. Very pure substances melt very sharply at exactly one temperature, while impure substances will begin to melt at one temperature and finish at another (nearby) temperature. This is why, rather than simply determining the melting or boiling point, you need to get a range from where melting point begins to where it ends. If the melting point is not sharp, then you can assume it will be a bit too low; this is a qualitative statement, but it is why melting points are first steps in product identification, rather than definitive.

For melting point determination, most organic chemists use a device called a "Mel-Temp." This is actually a brand name, but it has become synonymous with the device, much as "Band-Aid" is a brand name but is used generally to describe adhesive bandages. All melting point apparatuses have the same basic features. They have a thermometer (or temperature output device), and a magnifier positioned to observe several capillary tube holders. They also have variable voltage devices in the front. To obtain the best results, if you are determining an organic compound with a relatively low melting point, use a low setting so the mineral oil inside the device heats up slowly. For high melting point devices, use higher settings.

Load a capillary tube (a small glass tube open on one end and closed on the other) by dipping the open end gently into your crystal sample to get a little bit inside the tube. Don't try to get too much as it will be difficult to tap down. Turn the capillary tube over, and tap the closed end on a bench to force the crystals to fall to the bottom. You only need enough crystals to be able to see them; remember they will be magnified, so if you can see them with the naked eye, you will be able to see them in the melting point apparatus. If you have too much sample, surface area effects will cause problems; the smaller the sample, the better.

Before placing the capillary into the melting point apparatus, check the thermometer to be sure it is not still hot from a previous run. If the melting point is above, or close to, your expected melting point, allow the apparatus to cool *before* inserting the capillary tube. Be sure the device is off or it will not cool. If you let the sample melt and recrystallize, the results will not be accurate. If capillary tubes have been left in the apparatus from previous runs, remove them and discard them in the broken waste disposal container.

Once the apparatus is appropriately cool, place the capillary tube(s) into the capillary tube holders on the device. Set the voltage to the appropriate range, and turn on the device. Watch the tubes very carefully, and be ready to quickly switch from watching the tubes to reading the temperature. As soon as it appears your crystals are melting (they will look "wet"), read the temperature (remember to estimate to one more significant figure than the divisions on the thermometer). This is your "initial melting temperature." Take this reading *quickly* and look back at the crystal through the magnifier. As soon as the sample has completely melted, read the temperature one more time for the "final melting temperature." These two temperatures are your range; if the sample melted so rapidly that you could not get a range, make a note of this as it is a good indication that your sample is quite pure.

If a melting point determination device is not available, you can make your own with an appropriate bath. Choose a water or mineral oil bath (depending on the expected melting point), and get enough water or mineral oil so it is at least 10 cm deep.

Load a capillary tube as before. Strap it on to the most precise thermometer available (that is, the greatest number of significant figures) using a thin clipping from a rubber hose. Set up a clamp to hold the thermometer in the temperature bath, but be sure the rubber clipping does not get into the bath (if it is mineral oil). Be sure that the crystals are centered as closely as possible with the bulb of the thermometer. Insert the thermometer and capillary tube into the bath, and begin heating the oil. Take the temperature range as before.

Boiling Point Determination:

There are two ways to determine the boiling point of a sample, depending on the amount (and toxicity) of your sample. If you have a great deal of the sample, and it is not too toxic (or you can do it in an operational fume hood), the easiest way is to simply boil it. Set up a small flask with enough of the liquid in it such that it will be able to boil without going dry on a hot plate. Clamp a thermometer to a ring stand such that the bulb is just above the level of the liquid. Heat the liquid until it begins to boil; read the boiling point on the thermometer.

For micro or particularly hazardous samples, we will set up an apparatus very similar to that for melting point. Use a thin wire to transfer a bit of the liquid sample into the bottom of a capillary tube (it might be easier to use a syringe). Attach the capillary tube to the thermometer as you did for the solid, but this time, attach it upside down. Use a water bath (NOT mineral oil, as the band will have to be submerged) to heat the sample. You are looking for a steady stream of bubbles coming out from the opening in the capillary tube; at this temperature, the liquid is boiling. Read the temperature quickly, as the water bath will continue to heat.

Index of Refraction:

As light passes through various media, it will slow down or speed up (it slows down as it goes into increasing density material). This causes a bend in its path, which is referred to as "refraction." The index of refraction is a constant for any given liquid and is often used for identification purposes.

You'll notice that the refractometer has a "hidden container;" the name plate on the front of the base swings. Inside, you will find a little piece of glass. This is a critical component of the refractometer; take special precautions to ensure that you do not damage or lose it! You'll also notice a clip on the eyepiece. Careful inspection of the glass will yield an observation that one edge is cut at an angle rather than straight; the piece of glass goes into this clip with that angularly cut side up, such that a skewed "v" is formed between the glass of the eyepiece and this piece of glass.

Once installed, place ONE DROP of your liquid on top of the glass (the "v" is more or less a holder for the liquid). Press the button in the back to turn on the light and look through the eyepiece. You will notice two lines on the scale; the particularly bright one is caused by the glass itself; ignore it. The lighter line is caused by the liquid in the holder. Read the index of refraction from the scale.

FT/IR:

The FTIR is an expensive device and challenging to use; your instructor will either demonstrate its use, or run your samples for you. However, it is important to note here that the FTIR is a critical tool in molecular identification. It works in the infra-red portion of the spectrum, which is the portion of the electromagnetic spectrum that is absorbed by various functional groups. Thus, the FTIR will tell you if you have, for example, an aldehyde, a ketone, an alcohol or a carboxylic acid. This machine will be used frequently in product identification.

Molecular Mass Determination:

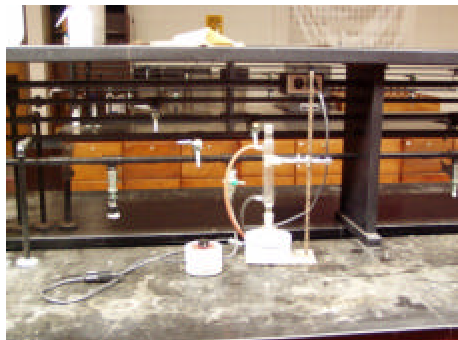
The easiest way (short of owning a mass spectrograph) to estimate molecular mass is through freezing point depression or boiling point elevation. For solids, I recommend mixing them with naphthalene, and for liquids, I recommend mixing with hexane or methanol. Look up "freezing point depression", "boiling point elevation" and/or "colligative properties" in your general chemistry textbook for a thorough discussion of how to calculate molecular mass.

Product Purification:

Distillation:

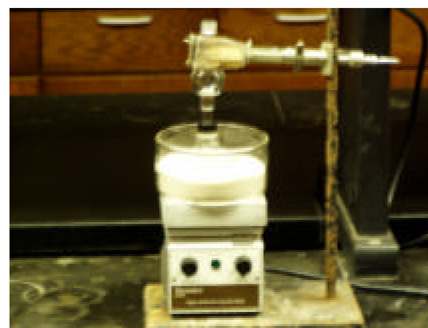
Distillation (or "fractional distillation") is the most commonly used means to separate liquids from other substances. With fractional distillation, one can separate multiple components from a mixture (for example, in gasoline, fractional distillation can be used to separate out the individual hydrocarbons.

The principle is quite simple; liquids in solutions will boil out at different temperatures. Thus, by hooking up a condenser to a heat source (typically a heating manifold, similar to a hot plate, and a round bottom flask), it's just a matter of collecting the liquids as they come out of the condenser. Remember to have the water for cooling enter the condenser at the bottom, and exit out the top. Do not turn the water on too strongly; a trickle is sufficient. Having too much pressure can cause a hose to "pop" off. When I was in graduate school, this happened in a lab above the one I was working in; there was over an inch of water on the floor in their lab, plus it leaked through, damaging the ceiling on our lab. We were fortunate; the water actually fell on a bench right inbetween two very expensive pieces of equipment that were on opposite sides of that bench.



Typically, one includes a thermometer attachment. This is a glass "elbow" with a rubber thermometer holder on the top. By placing a thermometer in here such that the bulb is just below the elbow, one can get the boiling points of each fraction as they come out of the mixture. As each individual component begins vaporizing, the temperature will reach a "plateau." That is, the temperature of the vapors will continue to increase until the first liquid begins to vaporize, at which point the temperature will remain stable until that particular liquid is removed from the mixture. At this time, the temperature will begin increasing again. For fractional distillation, simply be sure to replace the collection vessel between each of these plateaus, and you will have a different chemical in each of the collection vessels.

A microscale technique for distillation has been developed as well. The "condenser" is really just an attachment with a small flat area to catch small drops of the product. Be very careful not to heat too quickly if you are using a microdistillation technique to avoid post-distillation contamination.



Recrystallization:

You can almost think of recrystallization as the solid-form alternative to distillation. When crystals form, the atoms align themselves in a highly ordered fashion that minimizes their energy. This alignment is not possible if there are impurities, which results in a loss of energy (you get a higher energy crystal). As such, when crystals form, they *want* to form pure. So, to purify a solid, you simply cause it to dissolve, and force it to reform crystals. Each time you do this, you remove more impurities, but, of course,

the more you do it, the more you lose product as well. It's a careful tradeoff chemists have to decide on between how pure they want their final product to be and how much of it they want.

There are two basic forms of recrystallization; the most common relies on solubility variation as temperature changes. Thus, you look for a solvent in which your product has a very high solubility at high temperatures, but a very low solubility at low temperatures. The greater this difference, the less product that will be lost. Simply stated, dissolve your crude product in as little solvent as possible at a high temperature (the more solvent you use, the more product you lose). Once dissolved, place the solution in an ice bath, and allow the crystals to reform. If recrystallization doe not occur on its own, try scratching the bottom of the beaker; you may have a super-saturated solution. Once the crystals have formed, filter and dry the crystals. Repeat as often as you think necessary to get the purity you desire.

The second method of recrystallization is called "solvent exchange." To use this technique, you need two solvents that are miscible with each other, and in which the product is soluble in one but insoluble in the other. Dissolve the crude product in the solvent in which it is soluble, using as little solvent as possible. Once dissolved, if some of the impurities are still in solid form, you can filter at this time. Then, simply mix in the other solvent, until the crystals reform. In essence, you have replaced one solvent with another.

Organic Library Resources

A colleague of mine told me that he had gotten into a discussion with somebody regarding the shape of orbitals in the absence of other atoms. "All orbitals are spherical if there is no other external field" (such as a second nucleus) he argued. So, off he went, to run some calculations in hopes to prove his point and, he tells me (I have not verified this), he did in fact find the solution. So, he wrote up a little article and sent it off to be published, only to discover that somebody had already published these results. They were not results of particular interest; how often are we interested in one atom that has no others nearby? NEVER, I tell you! Well, rarely anyway.

The point is this, if he had taken the time to do a literature search, he would have found the work had already been done, and he could have saved himself the time and effort of repeating somebody else's experiment. This is one key reason for utilizing library resources in organic chemistry, but there is another: the need to know where to begin.

See, the other thing is that, even if you are indeed working on a unique compound never before synthesized (as one of my pet projects), there is still a good chance that somebody has been working on a similar compound. This is often an excellent place to find a great starting point for your own project. You can see what somebody else has done, compare their target compound with yours, and if it is similar, and that person had success, you can base your synthesis strategy on their work; just don't forget to give them credit in your publication!!!

Print Reference Resources:

Of course, on the library home page (http://www.departments.dsu.edu/library/) you will find their on-line catalog (http://www.departments.dsu.edu/library/libcat.htm). Here you will find a variety of old organic text books, specialized organic topics, all kinds of wild things. Don't forget you can also get materials from other institutional libraries by using interlibrary loan. Probably the two most commonly used print references by chemists these days is the CRC handbook of chemistry and physics, and the Merck Index, both of which are available in the library (and several copies of the CRC reside in the science building).

The **CRC Handbook of Chemistry and Physics** is basically a table that lists many properties of many compounds. The basic tables include things like melting points, boiling points, solubilities, and so forth, but there are a LOT of very useful tables beyond these. One of the most useful sets of tables is the boiling and melting point tables; if you have an unknown compound, it is easy to ascertain the boiling or melting point. The CRC lists compounds BY boiling or melting point in the back, which is often a great way to begin when trying to identify an unknown. While the CRC is a very powerful tool for chemists, it has one very major drawback: you must know the correct IUPAC name of the compound you are looking up, or it is very difficult to use.

My personal favorite handbook is the **Merck Index.** This, too, has basic information on chemicals, although not in a table format. Instead, each chemical has its own entry, which discusses several things such as chemical and physical hazards, therapeutic categories, and more. The thing I really like about this index is in the back, they list compounds by their correct

IUPAC names AND most synonyms. Even if this book does not have the information you need, it is often a convenient place to begin since it does list the correct name once you find it.

On-Line Resources:

DSU is very fortunate that the American Chemical Society (www.acs.org) has elected to put ALL of its journals online, and at a cost that (with a little annual grant to DSU from yours truly) we can afford. Just go to http://pubs.acs.org/about.html and you will find a lot of very interesting journals, since the ACS is the principle publisher of chemical journals in America. Of particular interest to this course is the Journal of the American Chemical Society, the Journal of Organic Chemistry, Organic Chemistry Letters, Organic Process Research and Development, and several others. The nice thing about this service is the search function, which allows you to search all journals, past and present, based on keyword, title, author, etc.

A number of years ago, there was a series of journals called "Organic Synthesis." The concept was quite simple; the editors wanted a create a compendium of methods for synthesizing, well, just about anything you could want. As you might imagine, the series became large and unruly, and, yes, I have a complete set, but no, I WON'T share. But that's because they are now online at www.orgsyn.org. This is typically a FIRST reference for organic chemists, with a very nice search engine as well. It is written in a cookbook style, so if you find what you are seeking, usually about the only thing you have to do is scale (since this is designed for industrial scale syntheses).

If you want your own chemistry handbook online, you might want to take a look at http://www.chemfinder.com/. This provides the kind of information you would find in the CRC, but with less detail.

Experiment 1: Organic Techniques

Purpose: To practice some of the more common methods of purification and product identification that will be used in the organic chemistry lab

Background:

See "Organic Laboratory Equipment and Procedures"

Introduction:

There's an old joke that goes, "How do you get to Carnegie Hall? Practice, practice, practice." And then everybody would laugh. I guess. I don't know, I don't get it either. But, I do know that practice is important to be successful at any endeavor, and today's experiment is designed to provide you with practice with some of the more common tasks that you will perform in the chemistry lab.

As you perform the tasks in this lab, remember to take your time. Remember that in organic chemistry, if you want to be successful, you will have to be subtle. In general chemistry, you can be relatively sloppy and still get reasonable results, so if you approached general chemistry with the attitude of getting out quickly, you were probably still successful (you must have been; otherwise, you would not be here now). To be successful in organic chemistry, now you have to have an attitude of succeeding in the experiment. Time to forget the clock, and pay attention to the experiment!

Procedure:

You need not perform this experiment in the order it is presented. Follow the instructions of your lab instructor.

Distillation:

Your lab instructor will provide you with a mixture of two organic compounds. Set up a classical distillation system and perform a fractional distillation to separate and collect the two pure liquids from 20 g of the mixture. Determine the boiling points of these liquids.

Recrystallization by Temperature:

You will be provided with a compound for purification. Determine the melting point of the crude product using a Mel-Temp. Measure out 1 g of the crude material, and recrystallize the in the solvent provided using the difference in solubility by temperature. Determine the percent product recovered, and the melting point of the recrystallized product with the Mel-Temp. (HINT! You can do both melting point determinations simultaneously.)

Microdistillation:

You will be provided a mixture of an organic liquid with a solid contaminant. Measure out 1 g of the mixture. Set up a distillation using a microdistillation technique. Calculate the percent of the organic liquid in the original mixture. Determine the boiling point of the liquid using the microorganic technique (inverted capillary tube).

Recrystallization by Solvent Exchange:

Your lab instructor will provide you with a crude compound for purification that may or may not be the same as that used before. Determine the melting point of this material without using the Mel-Temp. Using solvent exchange, recrystallize 0.1 g of the product. Determine the mass of the recovered material. Calculate the percent recovery, and determine the melting point of the recrystallized product without using the Mel-Temp. (HINT! You can do both melting point determinations simultaneously.)

Determination of Molecular Mass by Melting Point Depression:

You will either be provided with a solid compound, or asked to use one of the solids you have already crystallized by your lab instructor, who will also provide an appropriate "solvent" (in this case a solid) and the necessary information (melting point and freezing point depression constant for the solvent) to perform this experiment. Decide on appropriate amounts of solvent and unknown to perform this procedure. Determine the molecular mass of the unknown solid.

Calculations:

Distillation:

Report the boiling point of each of the liquids, and the percent by mass of each liquid in the mixture.

Recrystallization by Temperature:

Report the percent recovery and the melting point of both the crude and recrystallized product.

Microdistillation:

Report the boiling point of the liquid recovered, and the percent recovered.

Recrystallization by Solvent Exchange:

Report the percent product recovered, and the melting point of the crude and recrystallized product.

Molecular Mass:

Report the molecular mass of the unknown solid.

Pre-Lab Questions:

1. If you have a mixture of two compounds, one liquid and one solid, why won't distillation alone be sufficient to extract each of the pure compounds?

2. Using a CRC handbook of chemistry and physics, determine what two solvents, and the order you would use them, to recrystallize sucrose via solvent exchange.

3. What masses will you use for the molecular mass determination of the unknown solid?

Post-Lab Questions:

1. After the separation by distillation of the mixture of liquids, did the masses add up to 100% of the total volume? What do you supposed happened with any missing volume?

2. For the two recrystallization parts, how did the melting points compare before and after the recrystallization? What does this tell you?

3. Do you suppose melting point depression can be used to determine percent purity? Explain.

Experiment 2: Classification of Organic Functional Groups

Purpose: To understand how functional groups can be classified by chemical and instrumental means

Background:

See “Organic Laboratory Equipment and Procedures”

Introduction:

I struggled with organic chemistry. I really did, until, one day, I realized that it may be organic, but it is still chemistry. As it is still chemistry, that implies that all of the principles that I learned in general chemistry are equally applicable to organic chemistry, including simple little ones such as “different compounds will have different chemical and physical properties.”

Well, this is all we are really doing in today’s experiment; we are playing around with a variety of organic compounds with a variety of functional groups. We will be performing tests to see how they can be classified, and maybe, if you’re really good, we’ll even through in the IR spectra of these compounds.

We find that similar functional groups tend to have similar chemical properties.

Functional Group	Polarity	Ionic nature	Intermolecular forces
Alkanes, alkenes, alkynes	Non-polar	Non-ionic	Very weak
Alcohols	Polar	Non-ionic	Strong
Aldehydes/ketones	Polar	Non-ionic	Moderate
Amines	Polar	Alkaline	Strong
Carboxylic acids	Polar	Acidic	Strong
Phenols	Polar	Acidic	Strong
Amides	Polar	Acidic	Intermediate
Esters	Polar	Non-ionic	intermediate

Experimental Procedure:

Your laboratory instructor will have a series of unknown organic compounds available to you. For each of them, determine the following:

(1) water solubility (polar solvent)
(2) acidic or basic (only for those compounds that are water soluble)
(3) hexane solubility (non-polar solvent)
(4) melting or boiling point

Based on the above table, classify each compound's potential functional group(s).

Pre-Lab Questions:

1. How does solubility in water and hexane give clues about polarity?
2. What functional groups do you suppose would be hardest to separate from one another?
3. Look up the functional groups you put in part 2 in your textbook (you should have listed at least 3 of them). Based on the properties of these functional groups, propose additional tests you could perform to further classify them.

Post-Lab Question:

1. An IR spectrograph will be provided for each of the unknowns. Using a table of IR peaks, identify the locations of the peaks that identify each functional group. Which were you wrong on guessing based on the chemical properties? Why do you suppose you were wrong?
2. Looking at the IR peaks and your pre-lab question 2, how could you use IR to distinguish those functional groups that are difficult based on chemical and physical properties?

Experiment 3: Homologous Series

Purpose: To understand homologous series

Background:

See "Organic Laboratory Equipment and Procedures"

Introduction:

This summer, I went to an interesting meeting on forensic science. The people who were there were looking for all kinds of wild things, such as trying to trace common sources for drugs based on contaminants, seeking hidden messages or images in otherwise innocuous looking pictures, and determining means of detecting the presence of illegal substances. Here is an interesting question, though; if the compounds we want to study are illegal, then how can we study them? After all, methamphetamine, for example, is just as illegal for me to have possession of as it is for you.

There are three possible solutions to this problem. One is to become DEA certified to have these narcotics in your lab. Now, this is not an easy procedure. There are exceptionally strict guidelines on safely storing these chemicals, and you cannot dispose of them because extremely tight accounting practices are employed to track each and every milligram of each of these substances. This is not terribly practical unless you are already a federal agency and do that kind of stuff already. The second method (and the most common one used here at DSU) is to use DEA approved "standard samples" for labs without the certification. These samples are such low concentrations, and sold in such small quantities, that you could never get enough of them extracted to be of any "recreational" or resale use. Couple this with the fact that the cost, on a per gram basis, is astronomical, and they are safe to sell to anybody intelligent enough to realize the futility of trying to get a buzz from them. Of course, the major problems here is that the amounts you end up with are so miniscule that they are only detectable by the most sensitive of instruments, and there are still some narcotics that are so dangerous that you cannot get them even in this fashion.

So what do you do if you need cheap drugs in large quantities? If you said "just ask me," please don't let us know! No, as chemists, we are more clever than that; we often rely on what is referred to as "homologous series." These are compounds that are VERY much related to the compounds of interest to us, but with slight enough differences that they are legal (and ineffective for "recreational use"). For example, there is an "N+1" analogue of methamphetamine, in which one more carbon is added to the main chain of the compound. This analog does not show the psychedelic side effects of the "real" stuff, and is therefore quite legal. However, because all of the functional groups are the same, and because it can be prepared in exactly the same way as MDMA, it is very useful for testing in a non-certified lab.

In this experiment, you are going to be playing around with a homologous series. No, not of a drug. You will be asked to keep track of certain chemical and physical properties. We hope

you will see how they are related, and yet, how they differ from one another; after all, they are NOT the same compounds, so we should not expect them to have identical properties!

Procedure:

Physical Properties:

Determine the melting or boiling point of each compound (whichever is more appropriate) and density. Plot these properties.

Chemical Properties:

Check the pH of each of these compounds and make a plot.

You will be provided with potassium permanganate. This is a deep purple compound that, when sufficiently diluted, looks pink. On reaction, it forms colorless Mn^{+2} or brown MnO. It is a very strong oxidizing agent and will oxidize most organic compounds. Take approximately equal volumes of each of the compounds in the series. To each, add 1 drop of potassium permanganate solution and measure the amount of time it takes for the pink color to disappear. Make a plot for these times.

Pre-Lab Questions:

1. Beginning with Methanol, CH_3OH, write the formulas for the next four alcohols in the homologous series.
2. Why would you expect the compounds in a homologous series to have similar chemical and physical properties? Why won't these properties be exact?

Post-Lab Questions:

1. Look at each of the plots you created. What trends to you notice? Are they as you would expect? Why?
2. What observations stand out as unexpected in these plots? Propose a hypothesis on why these might occur.

Experiment 4: Organic Titration: Determination of Saturation

Purpose: To determine the degree of unsaturation through a halogen titration

Background:

See "Basic Laboratory Techniques" and "Organic Laboratory Equipment and Procedures"

Introduction:

Kekule had an interesting problem. He had an organic compound, that he knew was comprised only of 6 carbons and 6 hydrogens, and he knew that there were three double bonds. He struggled to determine the structure of this odd compound. When he finally figured it out, he became the first chemist to propose an organic ring structure; his unknown compound was benzene.

We hear of degrees of unsaturation in everyday life. That fat is saturated, or it is unsaturated, or even polyunsaturated. Ever wonder how this can be determined? How did Kekule know there were three pi bonds in benzene? How do we know if a fat is mono-, di-, tri- or even polyunsaturated? That is the goal of the experiment today. It is not common to find a quantitative experiment in chemistry, but today we will be performing one.

Procedure:

<u>Perform this experiment in a fume hood; halogen vapors are highly corrosive and toxic.</u>

<u>Standardization of Iodine Solution:</u>

Fill a 50 mL buret with the iodine solution. Using the analytical balance, measure out 0.02 g of sodium bisulfite standard solution. Dissolve in water, and add a few drops of starch indicator. Titrate to the endpoint. Repeat for three trials.

<u>Titration of the Unknown:</u>

You will be provided with a water-soluble unknown and the molecular mass of the compound. Measure out about 0.02 g of the unknown compound, and dissolve it in water. Add a few drops of the starch indicator and titrate it with the iodine solution. Repeat three times.

Calculations:

<u>Standardization:</u> The titration is between Iodine, I_2, and bisulfite, $S_2O_3^{-2}$. When the reaction is complete, you form iodide, I^-, and sulfate, SO_4^{-2}. If you balance this redox reaction, you will

find that you have a 1:5 molar ratio between iodine and sodium bisulfite. Find the average concentration of iodine in the solution.

Titration: For each trial, find the moles of the unknown compound using the molecular mass of the compound. From the titration and the concentration of the iodine you found in the standardization step, calculate the moles of iodine used for each titration. Divide the moles of iodine by the moles of compound; this will be the number of pi bonds per molecule. Find the average.

Pre-Lab Questions:

1. Balance the redox reaction between iodine and sodium bisulfite.
2. One of your students does not remember stoichiometry. After measuring out 0.0112 g of sodium bisulfite, it took 21.25 mL to reach the endpoint. What is the concentration of iodine in the solution?
3. It requires 39.28 mL of the standard solution from question 2 to reach the endpoint. How many moles of iodine were required?

Post-Lab Questions:

1. Report the number of pi bonds per molecule.
2. If you could throw out any 1 run from the titration, how much would the affect the results? Why did you choose the run you did to discard? Discarding this run, what is the average number of pi bonds per molecule.
3. Explain any sources of error you can think of for this experiment.

Experiment 5: Resonance Conundrum: Phenol

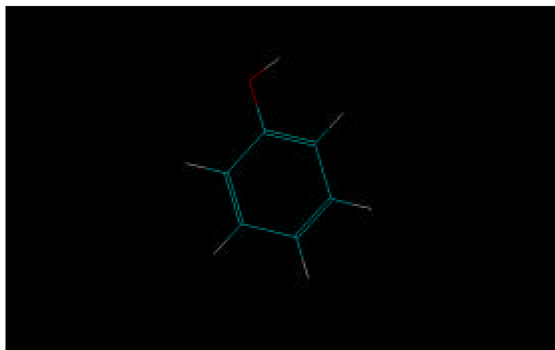
Purpose: To gain insight into resonance.

Background:

See "Basic Laboratory Techniques" and "Organic Laboratory Equipment and Procedures"

Introduction:

Yeah, I was there in Cincinnati, the year of the "Benzene Horror." I dunno, maybe it was in the early to mid '80's, a company upriver from Cincinnati (in Cleveland, I think) "accidentally" spilled about 50 gallons of benzene into the Ohio river. After the initial report, somebody decided to do some checking and found out that benzene is *slightly* carcinogenic. Oh my GOD; you would have thought it was the apocalypse. Every day, the news would report on the location of the deadly and devastating Benzene, as it drew ever closer, and closer to Cincinnati. Never mind the fact that most of it would have evaporated in the first day, leaving only minute traces in the Ohio river, and never mind that if you took a BATH in the benzene, the odds of getting cancer were exceptionally minute (only from prolonged exposure did there seem to be a slight risk, and note I said slight; chemists used to wash their hands in benzene every day before that tragic event). Lo and behold, the benzene finally reached, and passed, Cincinnati without incident.



Now, the reason I bring that up is because Cincinnati is a major enough metropolitan that now we are not ALLOWED to use benzene in chemistry labs, myeh myeh, myeh MYEH myeh myeh. Tell you what, give ME a glass of benzene, and I'll dump it in my hair for ya. But, for today's experiment, we are not allowed to use benzene, so instead, we'll use a benzene like compound known as phenol.

Now, phenol has a very well known structure; it is like benzene with an oxygen. It's formula is C_6H_6O, and is used as the active ingredient in some cough suppressants. It has three pi bonds, so it should react with three equivalents of iodine. The molecular mass of phenol is 94.00 g/mol.

Procedure:

<u>Perform this experiment in a fume hood; halogen vapors are highly corrosive and toxic.</u>

<u>Standardization of Iodine Solution:</u>

Fill a 50 mL buret with the iodine solution. Using the analytical balance, measure out 0.02 g of sodium bisulfite standard solution. Dissolve in water, and add a few drops of starch indicator. Titrate to the endpoint. Repeat for three trials.

Titration of Phenol:

You will be provided with water-soluble phenol. Measure out about 0.02 g of the unknown compound, and dissolve it in water. Add a few drops of the starch indicator and titrate it with the iodine solution. Repeat three times.

Calculations:

Standardization: The titration is between Iodine, I_2, and bisulfite, $S_2O_3^{-2}$. When the reaction is complete, you form iodide, I^-, and sulfate, SO_4^{-2}. If you balance this redox reaction, you will find that you have a 1:5 molar ratio between iodine and sodium bisulfite. Find the average concentration of iodine in the solution.

Titration: For each trial, find the moles of phenol using the molecular mass of the compound. From the titration and the concentration of the iodine you found in the standardization step, calculate the moles of iodine used for each titration. Divide the moles of iodine by the moles of compound; this will be the number of pi bonds per molecule. Find the average.

Pre-Lab Questions:

1. Knowing that phenol has three pi bonds per molecule, what volume of iodine should be required to reach the endpoint of 0.02 g of phenol if the concentration of the iodine solution is 0.28 g/100cc?

2. Most of the procedure was "written" by cutting and pasting the procedure before it. Why?

Post-Lab Questions:

1. Report the average number of pi bonds per phenol found from experiment. How does this compare with the known number?

2. Why doesn't a phenyl group behave like a standard compound with three pi bonds?

Experiment 6: Organic Synthesis: Formation of an Ester

Purpose: To perform your first Organic Synthesis

Background:

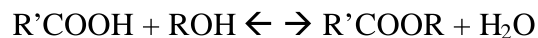
See “Organic Laboratory Equipment and Techniques:

Introduction:

There is an old Hindu saying; “The deer will spend her entire life seeking the pleasing fragrance in the woods, never once looking inside of herself.” I love this saying, but its roots, I’m afraid, has a rather sad origin. See, there is (apparently) a gland in the belly of a deer that secretes a very good smelling compound, and this compound, in fact, has been the essence of several perfumes throughout history. Well, despite this sad fact, it is still a lovely sentiment.

Fortunately, today we don’t have to kill deer to get the smells we use for fragrances. Instead, we have artificial means to create our own aromatic compounds (and here, I mean aromatic as in “pleasing aroma”; not phenyl-containing groups). You may not be making the same compounds as other groups; part of the fun of this experiment is to allow try to form various aromas. Oh, man, that is NOT the kind of aroma I meant; next time keep your farts to yourself.

This reaction belongs to a VERY important class of reactions, especially physiologically, referred to as a “dehydration” reaction. It is similar to the reactions used to create proteins, DNA, and RNA. The opposite reaction, “hydration,” us used to break these same molecules down, for example as in the digestive process. Here, the reaction is between an alcohol, ROH, and a carboxyilic acid R’COOH. In the reaction, we have:



$$R'COOH + ROH \leftarrow \rightarrow R'COOR + H_2O$$

Depending on the choices of R’ and R, we have a variety of properties of the final ester, R’COOR. Small chain side groups tend to give very aromatic compounds, while long-chain side groups form waxy compounds (in fact, this is the original definition of a wax, long-chain esters, and it is the same type of compound that forms ear wax).

Procedure:

You will be presented with a variety of acids and alcohols, and you may or may not be assigned a specific acid/alcohol pair. At your instructor’s discretion, you might be given or asked to obtain the FTIR for your starting alcohol and acid.

Set up a microscale reflux system with a sand bath and stirring hot plate. You will want at least a 5 mL conical container. Measure out sufficient acid for 10 mmol of acid, and sufficient

alcohol for 20 mmol of the alcohol. Heat the reaction to between 60 and 80 ^{o}C and allow to react with stirring for a minimum of 30 minutes.

Purify the product through fractional distillation. You should obtain at least two, and perhaps three fractions (alcohol, perhaps acid, and product).

Determine the mass of the purified product. Obtain an FTIR of your product and characterize its smell. Determine the boiling point of this product.

Calculations:

From the mass of the acid, determine the percent yield of your final product.

Pre-Lab Questions:

1. Now, "wax" refers to any substance with waxy-like properties. What was the original definition of a wax?
2. Explain how Le Chatliere's principle figures into this reaction. How are we using it to tip the balance in our favor?

Post-Lab Questions:

1. What is the percent yield? How can this be improved?
2. How does the boiling point of your final product compare to what is expected? What does this tell you about the purity of your final product?
3. Interpret the FTIR of this compound. Identify the principle peaks. Does the FTIR show any contaminant from the initial reactants? Explain.

Experiment 7: Retrosynthesis I: cis-3-hexene

Purpose: To design your first synthesis and try it in the lab

Background:

See "Organic Equipment and Techniques"

Introduction:

A number of years ago, when I was an undergraduate at the University of Cincinnati, I got to know one of the graduate students fairly well who was working in the field of Organic Chemistry. He was trying to synthesize a compound that, hopefully, would provide the basis for "artificial blood;" a generic mixture that could be used for anybody, regardless of their blood type, until their own bodies could restore the original blood lost. Sadly, he did not succeed in making this compound.

Well, how sad is it, though? After all, in the process, he managed to form, and patent, over 150 original organic compounds. Yep, every time he'd try, he did not get what he wanted, but if he did get a new compound, he'd patent the process. Maybe most of these had no value at all, but if a purpose was ever found for one of them, HE would be getting the royalties. Plus, he did successfully complete his Ph.D. even without finding the target compound; there comes a time when he's had enough tangential successes (the 150 patents) that he earned the degree.

For each new compound, he had to devise a new synthesis. How do you suppose he did this? Yes, we had the internet (thanks to Al Gore), but these are compounds that have never been made before, which means nobody had any idea of how to make them. For each new trial, he would have to develop a new pathway using the retrosynthesis technique. This is the technique that is still used today by industrial organic chemists every day to try to come up with newer, cheaper, better synthesis routes for drugs, pesticides, herbicides, artificial flavors, and just about anything else you can think of.

There are different levels of retrosyntheses. We will be starting with the absolute simplest; we will tell you what the target compound is (necessary in ANY retrosynthesis) and even give you the starting reagents. Later on, we might get to the type of retrosynthesis that my friend was working on, with a final product, but no starting points at all. These often do have guidelines, such as maximum cost for starting materials, or avoiding certain starting materials (for example, if we are working on a pharmaceutical, we'd be better off avoiding any toxic starting materials, don't you think?).

Procedure:

Get your procedure approved in advance by your laboratory instructor.

Follow your own procedure. Purify the product as you see fit. Obtain the FTIR, boiling point and molecular mass of your product.

Pre-Lab Questions:

Determine a synthetic pathway to form cis-3-hexene from bromoethane and 1-butyne. *Your procedure must be approved by the laboratory instructor prior to the experiment.*

Post-Lab Questions:

1. What was your percent yield? How could it be improved if you were to repeat this experiment?
2. How does the boiling point compare to what you expected? What does this tell you?
3. Interpret the FTIR; were you successful in obtaining the compound you want? Does the molecular mass determination corroborate this success?

Experiment 8: Isomerization and Nomenclature

Purpose: To gain experience with nomenclature and the various types of isomerization

Background:

See "Using HyperChem"

Introduction:

There really is a need for vocabulary. If we did not have a common vocabulary, then we would have no grounds to hold a discussion. At the most basic level, we are using the common vocabulary of English to convey our discussion in this lab manual. But, when we speak of vocabularies, each field, either in science, the arts, or any other field you speak of, has its own vocabulary.

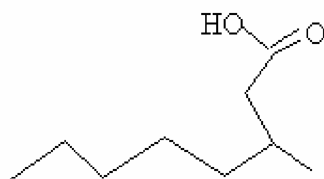
The vocabulary of a chemist is fraught with nomenclature. This is because chemical compounds are very much at the heart of the field. Thus, we have a little exercise for you to try your hand at nomenclature. This will use a variety of nomenclature styles for you to practice.

Procedure:

For each of the following, create any structures with stars in HyperChem.

Name each of the following compounds:

1.



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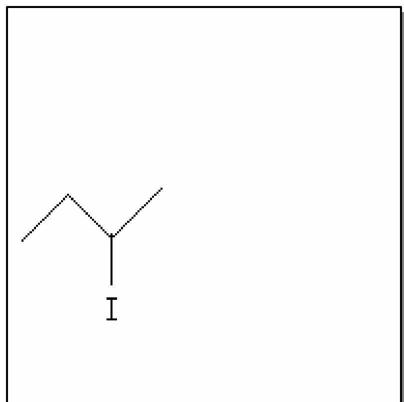

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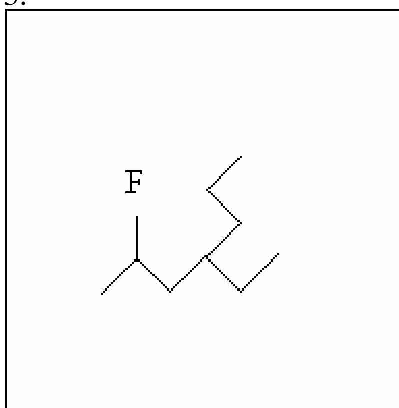

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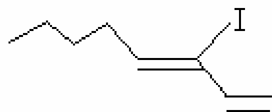

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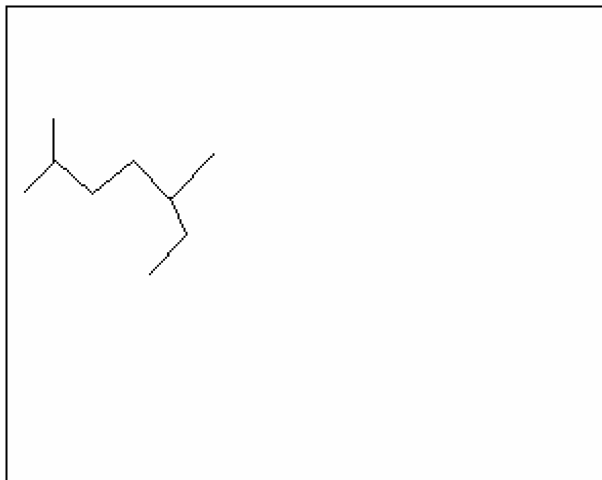

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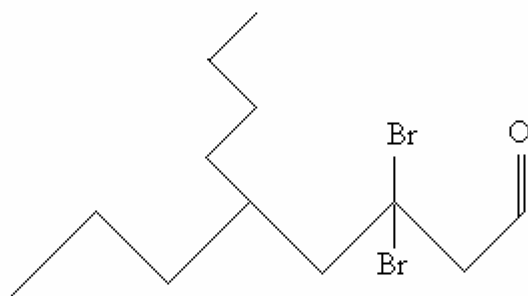

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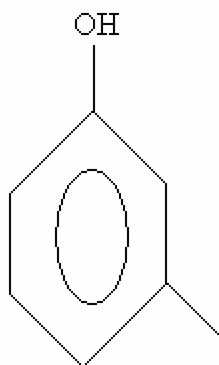
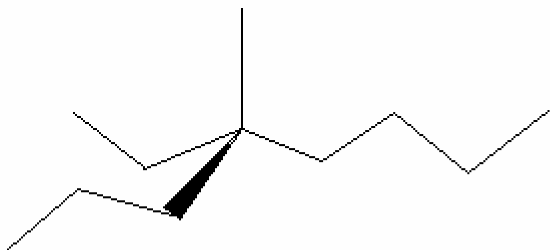
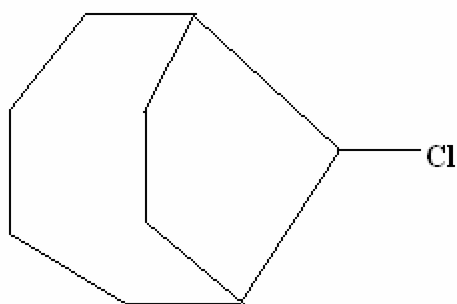


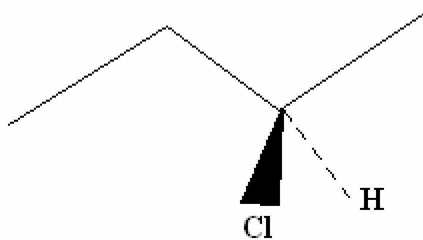
8.



9.



10.



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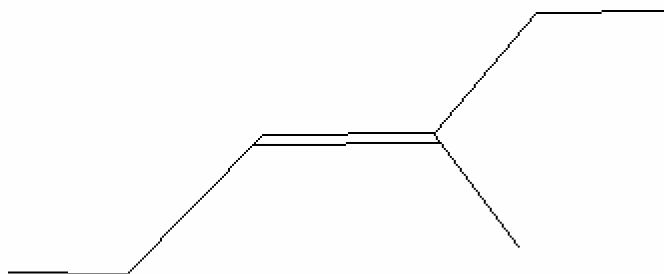

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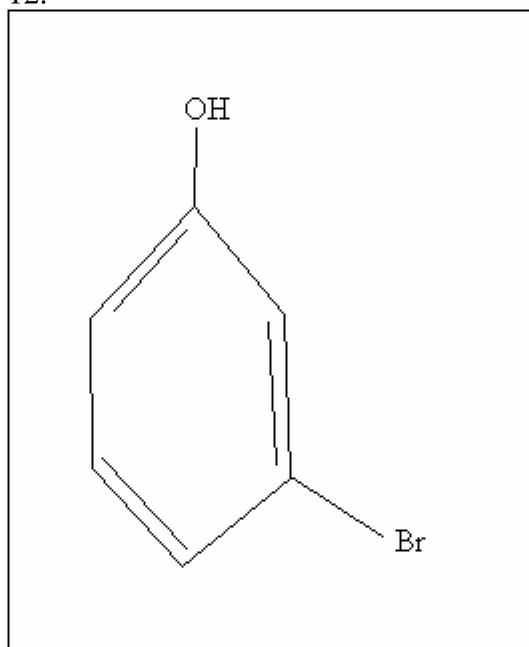

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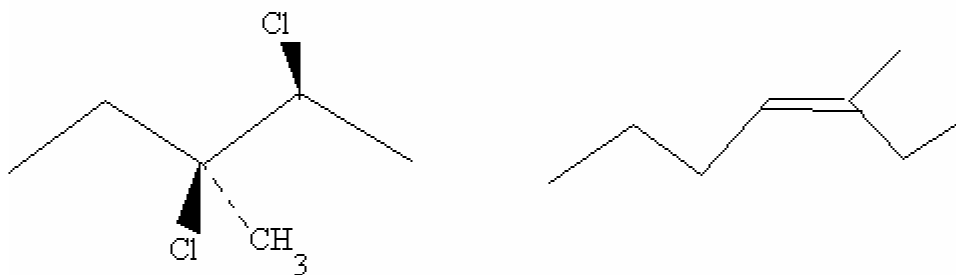

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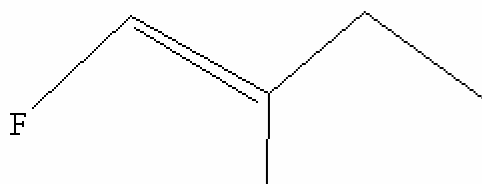

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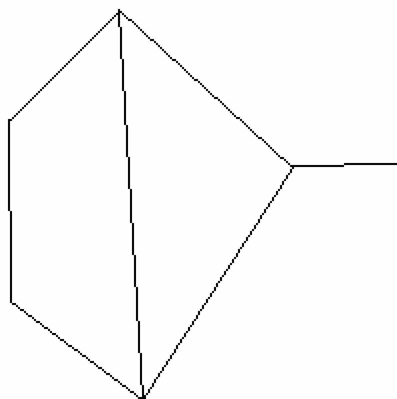

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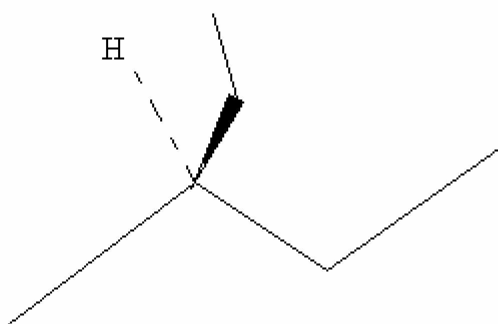

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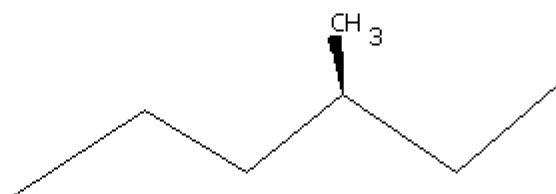


Draw the structure of each of the following compounds:

1. 1,1,1-trichloroethane
2. 3-cyclopentyl isohexane
3. 4,5 dimethylheptanoic
4. (E) 3,5-dimethyl-3-hexene
5. N-methylcyclohexylamine
6. trans-2-pentene
7. 3-methyl-3,7-dichloro bicycle [4.3.1] decane
8. and 9. propanal and propanone

10. and 11. 1 propanol and 2 propanol

12. and 13. 1-chloropropane and 1-bromoethane

14. pentane

15. propene

16. 17. and 18. 3,3-dimethyl-2-butanol; 2,3-dimethyl-2-butene; 3,3-dimethyl-1-butene

19. methyl bromide

20. ethyne

21. 2-methyl pentane

23. propyne

24. (E)-4-octene

25. 26. and 27. 2,5-dimethyl hexane; 3-methyl butyne; 2-chloropropane

28. 1,2 dibromoethane

Pre-Lab Questions:

None

Post-Lab Questions:

None

Experiment 9: Polymerization Reactions

Purpose: To understand polymerization reactions

Background:

See "Basic Laboratory Techniques"

Introduction:

Polymers have become an integral part of our lives. All plastics are polymeric in nature; even on the tablet I am using to type this has polymers for each of the keys, the rest pads, the buttons, indeed, the entire case. Even the parts that are designed to look metallic are clearly plastics. And let's not forget fashion; polyester was the fashionable fabric of the '70's!

Polymers all start off as monomers. These are simple molecules, smaller, in fact, than many organic molecules. What differentiates monomers from standard organic molecules, however, is the ability to not only bind to other monomers of the same type, but to propagate the bond for a considerable length. Two monomers will bond together to form a dimer, but with a "sticky end." That is, the end of the molecule is typically a free radical, which will quickly bond with another monomer, to turn the dimer to a trimer. The trimer will also have this sticky end, and will bond to form a quatramer, and another sticky end. This process continues, hundreds, thousands of times or more to create very long chain-like molecules.

Procedure:

Your instructor may or may not elect to have you perform all parts of this procedure.

<u>Polymethyl Methacrylate:</u>

Heat up a water bath to between 80 – 90°C. Before a polymerization reaction can begin, inhibitor (added before shipping to prevent premature polymerization) must be removed by placing roughly 10 mL of Methyl Methacrylate in with 2 grams of Alumina. Mix to a slurry, and filter out the alumina. Put 10 mL of this inhibiter-free methyl methacrylate with 0.05g benzoyl peroxide into a test tube. Immerse the test tube into the hot water bath. Remove when hardened (approximately an hour).

To remove the polymer from the test tube, wrap the test tube up in several layers of paper towel, and carefully break the glass with a hammer. Use tongs to remove the polymer from the glass shards and rinse it off carefully to remove all of the glass.

<u>Polystyrene:</u>

Heat up a water bath to between 80 – 90°C. Before a polymerization reaction can begin, inhibitor must be removed by placing roughly 10 mL of styrene in with 2 grams of Alumina. Mix to a slurry, and filter out the alumina. Put 10 mL of this inhibiter-free methyl styrene with 0.05g benzoyl peroxide into a test tube. Immerse the test tube into the hot water bath. Remove when hardened (ten to twenty minutes).

To remove the polymer from the test tube, pour the viscous but still liquid polystyrene out into a mold (another container) before it cools. If you would like to imbed an object into the polystyrene, you can do so while it is still in this state. The polymer will harden on cooling.

Nylon:

This is a fascinating reaction for two reasons. First, it is an example of a heterogeneous polymer (two reagents, rather than just one), and second, it is an example of an "interfacial reaction" (that is, there are two immiscible liquids placed in a beaker, the nylon will form at the interface of these liquids).

Into a small beaker, pour 10 mL of 0.5 M hexamethylenediamine (in 0.5 M NaOH) solution. CAREFULLY (so as to avoid mixing) pour 10 mL of 0.2 M sebacoyl chloride solution (in hexane). Wearing gloves, pluck the nylon from the center of the interface. Pull it smoothly out starting at one end of the bench. Pull it as far as you can before it breaks.

Post lab analysis:

Take a sample of all polymers you made and determine the average molecular mass of the polymeric product. Compare this result with the molecular mass of the monomer and determine the average number of monomers per polymeric chain in the sample.

Obtain the FTIR of the product.

Calculations:

None.

Pre-Lab Questions:

1. Look up and report the molecular structure for the monomeric units of methyl methacrylate and styrene.

2. Why is it necessary for chemical manufacturers to add an inhibitor to the styrene monomer before shipping?

Post-Lab Questions:

1. Look at the FTIR of the polymers you produced, and compare them with the monomeric molecules. For each polymer formed, what functional groups changed? What remained the same?

2. Determine the structure of each polymer you produced based on the above information.

Experiment 10: Aromaticity

Purpose: To gain an understanding of aromaticity and "directors".

Background:

See "Basic Laboratory Techniques" and "Organic Laboratory Equipment and Procedures"

Introduction:

Aromatic compounds caused quite a stir. After all, on first glance, they look like simple double bonded compounds, but they don't behave like double bonded compounds. Addition reactions, for example, preserve the pi bonds, rather than simply adding across and destroying them. And imagine the surprise when o/p and m directors were discovered. Simply amazing.

In this experiment, we will be examining the bromination reaction of two organic aromatic compounds, nitrobenzene and aniline.

Procedure:

Set up a stirring hotplate in the fume hood. Put a stirbar in a small beaker, along with 2 mL of nitrobenzene. Stir the solution; turn on the heat, but do not allow the solution to get so hot that the reaction becomes uncontrollable (start with no heat first and add heat slowly). Add one drop of bromine; note how long it takes for the color to dissipate. Continue adding bromine, drop at a time, until the color no longer dissipates. Keep track of the number of drops of bromine that you add. Repeat this procedure for aniline

Add a small amount of zinc to the solution to remove any excess bromine. Once the color is completely dissipated, filter out the zinc. Analyze the product by determining the molecular mass of the product and by FTIR. Determine the products formed.

Calculations:

Calculate the number of moles of nitrobenzene and aniline used in this experiment based on the density and molecular mass of these compounds. Determine the number of drops per mole of bromine added to each of these. Which absorbed more bromine? Does this observation match what you would expect now that you have an idea of which should be the O/P and which should be the M director?

Pre-lab Questions:

1. What is an o/p director? What is a m director?
2. Which reaction is usually faster, an o/p or a m director?
3. What is meant by o/p? What is meant by m? Draw a figure to demonstrate the difference.

Post-lab Questions:

1. Was aniline an o/p director or a m director? What about nitrobenzene?
2. Both aniline and nitrobenzene are nitrogen-containing aromatic compounds. Why is one o/p directing and the other m?

Experiment 11: Reaction Mechanisms

Purpose: To gain experience with how products can help determine reaction mechanisms

Background:

See "Basic Laboratory Techniques" and "Organic Laboratory Equipment and Procedures"

Introduction:

You wanna know why I love old chemistry textbooks? It's because they had to figure everything out manually. Nowadays, what we can't "google" (and get out of this habit, by the way; we have a fantastic library and resources through the library, so get in the habit of using them first) there is usually some fancy-schmancy hoity toity snooty machine to tell us everything we need. In fact, we are using one of these new-fangled machines (the FTIR) to help us out today. Still, though, although we will use this to analyze our products, we leave it up to YOU to analyze the results.

Procedure:

Perform this experiment in the fume hood.

Place 12 mmol of cyclohexene into an appropriately sized conical flask. Add 10 mmol of bromine to the reaction flask. Set it up in a sand bath with a stirbar and a reflux condenser. Heat the reaction to 60°C with stirring and allow it to react for at least one hour. The solution should be colorless when the reaction is complete.

Remove the reaction flask from the hot plate and allow it to cool. Perform a fractional distillation, determining the boiling point of each component as it comes out of the solution. Find the FTIR of the product.

Cis-1,2-dibromohexane has a boiling point of about 115°C, while trans-1,2-dibromohexane has a boiling point of about 145°C.

Calculations:

None.

Pre-lab Questions:

1. If the density of cyclohexene is 0.6796 g/mL, what volume is needed to get 12 mmol of this compound?
2. If the density of bromine is 3.1023 g/mL, what volume is needed to get 10 mmol of Br_2?

Post-lab Questions:

1. What product did you form, cis, trans or both?
2. Based on your product, what was the reaction mechanism (E1 or E2)?
3. Explain how you came to the conclusion of which reaction mechanism is correct.

Experiment 12: Retrosynthesis II: 4-bromononane

Purpose: To gain more experience with retrosyntheses with restrictions

Background:

See "Basic Laboratory Techniques" and "Organic Laboratory Equipment and Procedures"

Introduction:

Last time we did a retrosynthesis, we left it pretty much up to you how you wanted to start. It is not uncommon for a chemist to see a similarity of one compound, and work it into a strategy to build another. This is our goal today; somebody somewhere has a method to produce propanol quickly, cheaply and in abundance, but YOU need 4-bromononane. How can you make 4-bromononane from propanol?

Procedure:

Perform your approved synthesis to produce 1.0 g of 4-bromononane. Analyze the product to determine its purity, the percent yield, and prove that you did obtain the target compound.

Calculations:

Determine the percent yield.

Pre-Lab Questions:

1. Determine a retrosynthesis for 4-bromononane from propanol and get it approved by your laboratory instructor. Be sure to include a plan for purification of the final product.

Post-Lab Questions:

1. What was your percent yield?
2. Provide proof that you achieved your target compound, or identify the compound you isolated if it was not your target compound.

Experiment 13: The Diels-Alder Reaction

Purpose: To gain experience with the Diels-Alder Reaction

Background:

See "Basic Laboratory Techniques" and "Organic Laboratory Equipment and Procedures"

Introduction:

The Diels-Alder reaction is VERY important in synthetic organic chemistry, because it is a ring-closure reaction. OK, enough said.

Procedure:

Place 2.0 mmol of propenoic acid and 2.0 mmol of 1,3 butadiene-1,4-diol. Attach a reflux condenser, and heat it while stirring; do not allow the temperature to exceed 90°C. Purify the products by fractional distillation. Use boiling points and FTIR to identify the products.

Calculations:

None

Pre-Lab Questions:

1. If the density of propenoic acid is 1.0511 g/mL, what volume do we need to get 2.0 mmol of this reactant?
2. If the density of 1,3-butadiene-1,4-diol is 1.064 g/mL, what volume do we need?

Post-Lab Question:

1. What product did you produce?
2. What was the percent yield?

Experiment 14: Instrumental Analysis

Purpose: To understand how one can manually identify compounds through spectrometry

Background:

None

Introduction:

There are two principle ways to use instruments in the identification of unknown compounds. The first we have used repeatedly throughout this year; comparison with known spectra. This technique requires that a "library" be built, either by the user or an outside source (which can be quite expensive). Simply, known compounds are run, and the spectra are stored. Once an unknown compound is run, then the computer compares the unknown spectra with the library of spectra to find the best possible match. But what happens if the unknown is not a compound that is already in the library?

Then it becomes much more complicated. The chemist must piece together clues from the various possible spectra and try to determine the compound. This is what you will be doing today.

Procedure:

You will be provided with a variety of spectra. Each compound may or may not include a mass spectrograph, FTIR or IR spectrograph, H-NMR and ^{13}C-NMR. From each spectra given, find the clues you need to determine the structure of each unknown compound.

Calculations:

None

Pre-Lab Questions:

None

Post-Lab Questions:

1. What were each of the unknowns?

Experiment 15: Wet Chemical Analysis

Purpose: To identify a compound using classical wet chemical techniques

Background:

See "Basic Laboratory Techniques" and "Organic Laboratory Equipment and Procedures"

Introduction:

To identify a compound using wet chemical techniques, you will be using all of the information you've been learning throughout general chemistry. Physical properties such as solubility will be used to help initially categorize the unknown compound, while chemical properties will be used to identify specific functional groups.

In the following procedure, you won't follow all of the steps. Instead, follow the flow chart and run the procedures you need to identify your unknown compound. Remember, your only goal is to identify your compound (we will assume your unknown is already pure), so think logically and run the tests you need. For example, you would not run a cholesterol test on a patient who has an ulcer.

Procedure:

You will be provided a small amount of unknown; additional unknown will cost you points.

Initial categorization tests

All unknowns will be small enough that we can neglect length issues (for example, polar head group on a molecule long enough to "masquerade" as a non-polar group).

Flammability and explosivity:

Put a very small portion of your unknown onto a ceramic spatula, and put it into a Bunsen burner flame. If the compound is explosive, it will "pop." If the compound is explosive, be very careful not to run any tests that require heat.

Physical Properties:

Determine the melting or boiling point (whichever is applicable), molecular mass, and index of refraction for liquids.

Solubility tests:

Test some water for pH before adding your unknown. Place a *small* amount of your unknown in water and note its solubility. Test the water for pH and compare the results with the pH of the water. Use the table below to categorize your unknown.

	Soluble	**Insoluble**
Low pH	Carboxylic acid	NA
No pH change	Ketone, aldehyde, alcohol	Alkane, alkene, alkyne
High pH	amine	NA

Specific functional group tests

Alcohol:

Ammonium Hexanitratocerium (IV) Oxidation: Add about 4 drops or 0.1 g of your unknown to 1 mL of ammonium hexanitratocerate reagent. If the reagent turns from yellow to red, an alcohol is present. Once the red color forms, note how long it takes for the solution to turn colorless. The longer it takes, the higher the order alcohol (that is, primary alcohols take less time to turn colorless than secondary and so on).

Alkene or Alkyne: If your compound is insoluble, to test for saturation, add a drop of bromine water to a drop of your unknown. If the color fades, you have an alkene or an alkyne.

Amine:

Acetyl Chloride: Add a few drops of acetyl chloride to 0.5 mL of your sample. Pour the mixture into a little bit of water. The presence of a precipitate is a positive indication for amines.

Carboxylic acid:

Phenylhydrazide salts: Dissole a little bit of your unknown into distilled water, and neutralize the solution with 10% sodium hydroxide. Add phenylhydrazine, and boil the resulting mixture for 30 minutes. The presence of a precipitate is a positive indication of a carboxylic acid.

Aldehyde or ketone:

Benedict's Reagent: Add 2 mL of distilled water and 2 mL of Benedict's reagent to 0.1 g of your sample. Heat to boiling. A red precipitate is a positive indication of an aldehyde or ketone.

Fuchsin-Aldehyde Reagent: If Benedict's reagent was positive, add 1 mL of the Fuchsin-Aldehyde reagent in to 1 drop or 0.1 g of your sample. Shake the tube gently (be careful not to get any on your skin). If the reagent turns from pink to colorless, this is a positive

indication of an aldehyde. If it remains pink (and Benedict's reagent was positive), then you have a ketone.

Determination of the Unknown:

Based on the information gathered, predict what the compound might be. In the back of the organic chemistry table in the CRC, you will find a list of compounds based on melting or boiling points; this is often an excellent place to start to narrow down the possibilities (don't forget to factor in potential experimental error). Comparison of the physical properties and functional groups from this list should help you to pinpoint the unknown to one, or at least no more than three possibilities.

Verification:

The simplest way to verify your unknown is to use colligative properties. Ask for a sample of each of your suspect unknowns. In separate test tubes, mix a little bit of your unknown with each of these potential compounds (about a 50/50 mixture). Determine the melting or boiling point. If the potential compound is different from the unknown, colligative properties tell us that the melting point will decrease and the boiling point will increase. If the potential compound is indeed the unknown compound, the melting point should not change.

Calculations:

None

Pre-Lab Questions:

1. Write a flow chart for the above tests.

Post-Lab Questions:

1. Identify your unknown.
2. Discuss your confidence.

Experiment 16: Retrosynthesis III: N,N-dipropanal amine

Purpose: To gain more experience with retrosyntheses with restrictions

Background:

See "Basic Laboratory Techniques" and "Organic Laboratory Equipment and Procedures"

Introduction:

Here it is, the big final retrosynthesis project. This assignment will require more than one step, and probably at least two or three weeks to complete. Good luck.

Procedure:

Perform your approved synthesis to produce 1.0 g of N,N-dipropanal amine. Analyze the product to determine its purity, the percent yield, and prove that you did obtain the target compound.

Calculations:

Determine the percent yield.

Pre-Lab Questions:

1. Determine a retrosynthesis for N,N-dipropanal amine and get it approved by your laboratory instructor. Be sure to include a plan for purification of the final product.

Post-Lab Questions:

1. What was your percent yield?
2. Provide proof that you achieved your target compound, or identify the compound you isolated if it was not your target compound.

Chemistry Laboratory Notebooks

Introduction: For an experimental scientist, there is no tool more important than the laboratory notebook. In the real world, the laboratory notebook is a legal document; they are often subpoenaed and used in court cases. A poorly kept notebook ultimately could result in millions of dollars lost to a company.

For this course, you will be required to keep and maintain a laboratory notebook, and graded on how well you keep it. Although there can be variances in style, there are several headings that should be common to all laboratory notebooks. Following is a guide of what I expect in the notebooks for this class and some helpful hints on keeping a good one. Unless otherwise noted, all headings are required in the order presented.

Experiment title and date: Start each experiment by putting the title of the experiment at the top of the page and the date.

Purpose: One or two sentences on exactly what we hope to achieve in the experiment. Often it is too easy to perform the steps in an experiment while losing sight of what it is we are trying to accomplish. The purpose is the "big picture", the brass ring to keep your eye on. Keep it very brief; for example, "To gain experience with a variety of chemical reactions."

Introduction (optional): The purpose of the introduction is to show the relevance of the experiment. Two or three paragraphs should be devoted here, which can include, for example, how the experiment relates to class, or how it has implications on some other aspect of life that you might be interested in. Ultimately, a few years down the road, this should remind you of why we were doing the experiment in the first place. Keep this brief if you choose to include it.

Data and Observations: Write down all raw data and observations. Data should be in tables whenever possible; use the data sheet in the lab manual as a guide but do not write the date in the lab manual as this is not your lab notebook, and you want all of your raw data in your notebook. Observations should be plentiful. Well kept observations could be important in tracking down problems if the experiment does not come out with the results you are expecting. You will be graded on the number and quality of observations taken.

Calculations: Write down ALL calculations involved in the experiment, including separate ones if you were asked to repeat an experiment several times. If there were no calculations for a given experiment, simply write "none" for this section.

Results: SUMMARIZE your findings. This should correspond with your purpose and be very brief. If we are asked, for example, to determine the percent acetic acid in vinegar, then here just write the individual calculated values for each run and the final average concentration.

Discussion: In two or three paragraphs, discuss your findings, conclusions, sources of error and thoughts on the experiment. How can it be improved? Do you trust the results? Why or why not? Think of any interesting applications of how this might be used elsewhere?

Questions: Answer all post-lab questions. As with the pre-lab questions, you need not write the questions here if you don't want to, but be sure that it is easy to ascertain which answer corresponds with which question.

Do's and Don'ts of Laboratory Notebooks:

DO write the page number on top of EACH page;
DO include an index on the first page or two of the notebook and keep it current;
DO write in non-erasable medium;
DO write only on the right side of the page; this saves the left side for scratch paper in case you need to perform a calculation during the experiment;
DO cross out any errors with a single line, keeping it legible, then date and initialize near the cross-out;
DO get my initials before you leave lab (this is proof that you performed the experiment in lab; lab reports without my initials will receive a "0" grade);
DON'T tear ANY pages out;
DON'T write in pencil or erasable ink
DON'T write ANYTHING outside of the notebook; write everything directly into the notebook;
DON'T use "White-Out" or blot out errors completely

Lab Preview Questions: This is MY proof that YOU are ready for the experiment. You must turn in your pre-lab questions on a separate sheet of paper *before* the experiment begins. You need not re-write the questions if you don't want to; however, this section should be written such that it is clear which answer corresponds with each question (typically simply by using the same numbering scheme as in the book).

Plotting Experimental Data

Prepared By: Richard E. Bleil, Ph.D.

Introduction:

Often in lab experiments it becomes necessary to make a graph, but unfortunately, many of us have not been given procedures for making a proper graph. As a result, several common mistakes tend to occur; improper scales are chosen resulting in graphs that do not utilize the entire piece of graph paper; straight lines are drawn through only two points; axes and titles are not correctly labeled. If you have never been taught how to make a graph, it is nothing to be embarrassed about. In fact, perhaps it is the educational institutions that you have attended that ought to be embarrassed for having not taught you. In an attempt to ensure that no students go through chemistry without having had this instruction, I've written this little guideline for you broken up into individual sections for (hopefully) ease of understanding. If you have any questions, comments, or suggestions, please do not hesitate to call (298-3399 x-5658) or email (rbleil@hotmail.com) or stop by (G-12A) to see me.

Determining Data Ranges:

One of the most common mistakes made is choosing a scale that utilizes only a very small corner of the graph paper rather than the entire piece. This results in graphs that can be difficult to see and larger than necessary experimental error should it be necessary to find the slope, intercept and/or use in predictions. Therefore, it is important to be able to choose a scale for the graph that is appropriate.

To begin, we have to decide what kind of graph we are making. There are three general categories; (1) graphs to cover only the experimental points; (2) graphs that must extend to some smaller number well beyond the smallest experimental point (often zero); and (3) graphs that must extend to some greater number well beyond the largest experimental point. Regardless of the type of graph that one must make, begin by examining your data. You'll have a set of data that includes both "x" and "y" data. Generally, "x" is taken to be exact, while "y" is the measured quantity, that is, the quantity that has experimental error. For instance, suppose I were to make a graph of boiling points versus molecular weight. You'll find a table of such data below in the appendix. The boiling points I measure experimentally. This implies that there could (and will) be some experimental error associated with this measurement. The molecular weight, however, can be easily calculated with very little error (much much smaller error than the boiling point). Therefore, I make my measurement with the greatest error (boiling point) the "y" axis, and the measurement with the smallest error (molecular weight) the "x" axis.

For both the x and y axis, determine your largest and smallest value. If your graph must extend to some range other than that covered by the experimental data, use these as your limits instead. For instance, perhaps my graph of boiling points versus molecular weight must cover very small molecular weights, say down to the molecular weight of methane CH_4, which has a molecular weight of 16 g/mol. However, my

experimental data has molecular weights ranging from 86 g/mol to 142 g/mol. Then I will make a plot that runs from at most 16 g/mol through at least 142 g/mol. For ease of calculation, I may choose numbers close to our limits, such as, say, 10 g/mol to 150 g/mol as my limits. Doing this tends to make it easier to figure out the scale.

As for the y axis, it is a little more difficult. We want to choose a minimum value that we expect will be low enough include the boiling point of a chemical with molecular weight of 10 g/mol. It would be much easier if we knew we wanted the graph to go to some finite amount. Looking at the experimental data, it looks to me as if the boiling point for 10 g/mol should not be less than, say, 50°C, so this is the value I will choose for the minimum value for the boiling point. If it turns out that this guess is far too small or far too large, I'll choose a new minimum value and replot.

Choosing a Graph Orientation:

Once I have examined my experimental point ranges (both x and y axis), I'll choose an orientation for my graph. Since graph paper is *usually* longer than it is wide (this may not be true if a space for graphing is provided), then I am free to choose if I want the x axis to be along the width or length of the paper. The axis with the widest range of values I will make the length of the paper. In our case, the boiling points, that is, the "y" axis, has the greater differences between minimum and maximum values. Thus, I'll make this the length of the paper, and the molecular weights the width.

Setting Up the Axes:

Now look at the graph paper carefully, examining the number of divisions along both the length and the width of the paper. The particular piece of graph paper that I have chosen has 39 divisions along the width and 52 divisions along the length. I'll need a little space to draw in the axes, so I will leave 4 divisions along the bottom and 2 divisions from the right, making my effective graph paper 50x35. I'll draw in the graph axes now and label them. Notice that my lables include *both* the label title *and* the units in parentheses.

We'll set the minimum values for the lower left-hand corner of the axes I've just drawn. Now I have to choose the appropriate value for each division. This is as much an art as anything else, but to get some idea, with 50 divisions spanning 50 to 200 ^{o}C, I expect each division along the y axis to be about (200-50)/50, or about 3 ^{o}C for each division. Before drawing anything, I'll check this by hand. I notice that this will take my scale up to 200 ^{o}C, so I will draw them in. First, I'll draw a mark every fifth line. Typically this can be every fifth or tenth line for ease. Then, I'll write in the temperature at each of these new divisions, and add "1 div = 3 ^{o}C" to the axis title.

Similarly, along the x axis, I expect each division to have a value (150-10)/35, or 4 g/mol per division. Checking this by hand, I see that it is exactly right, so, as with the y axis, I'll draw in the value every fifth division and add the line "1 div = 4 g/mol" to the x axis title. Now my axes are set, and the graph will take the entire sheet of graph paper.

Plotting the Data and Drawing Experimental Lines:

Now I plot the points. Each point I'll plot as carefully as possible, estimating the distance between divisions when necessary. Each experimental point I'll circle once they're plotted. You may notice that in our example, the points do not line up as nicely as perhaps we would like. This makes the task of drawing a straight line all the more challenging.

If we simply draw a line from the first point to the last point, then we have drawn a two-point curve and there was no reason to take more experimental measurements than just these two points. Instead, we want a line that best fits as much of the experimental data as possible. To do so, place a straight edge amongst the experimental data. If the curve *must* intercept at zero (or some other point), fix this side of the straight edge. Typically, however, there are no requirements for the intercept. Now move the straight edge such that when you draw the line, it looks as if the error above the line is about equal to the error below the line.

At this point, you're probably wondering what I mean. Well, if we assume that the error is uniformly distributed amongst all of the data. That means that I should have as much error that is too high as error that is too low. Recall that we assumed the "x" values are exact, and the only error is in the "y" values. So, if we were to measure the distance from each point to the line drawn for those points above the line, this sum should be equal to the sum of the distances from the points below the line to the line. Now, we needn't be so exact as to actually take these measurements and do the summations, but try to draw a line so that if we were to do this, the two errors would be as close as possible.

In some graphs, this is easier to do than in others. Typically (but not always), we'll have about as many points above the line as below (in our example, it is two above and three below). However, really we are drawing a line so the points look as randomly distributed about that line as possible. There are mathematically exact ways to determine the line that best fits data, but we shall not go into this here. If you would like to learn how to do this (it is called "Linear Regression"), please consult a statistics textbook or stop by my office and ask. I'll be happy to teach this technique to any who would like to learn it on an individual by-request basis.

Discarding Data:

Most of the time, we cannot discard data. In our present example, the points are so greatly spread out that I cannot assume that any data can be discarded. Typically, the only time it is acceptable to discard *one* experimental point is if it is clearly far off from the line that could be drawn without it. That is, if all of the points lie very very close to a line and one point is extremely far off, then most likely that one point is an exception and can be discarded. However, typically too few points are taken to be able to do this. If you feel that you have a point that *should be* discarded for any reason, talk with your professor and ask his/her opinion before discarding.

Reading a Point:

So now we want to find a point. We want to use our results to estimate the boiling point of methane (16 g/mol). Now we draw a line from the "x" axis at 16 g/mol)

up to the line and read the corresponding boiling point. This turns out to be about 55 ^{o}C. This is actually a *terrible* result, but it is what we found. Always be true to your data. If a result is far from correct, write a little paragraph on what you think may be the reason behind the poor result.

Title:

Always include a title with your graph, as well as your name and the date. The title is always something along the lines of "Graph of y versus x:. When we use "versus", by convention, it is always y versus x. You may include a sub-title if you like, which is often helpful for similar plots from different sets of data.

REMEMBER: (1) ALWAYS use the ENTIRE piece of graph paper.

(2) ALWAYS label your axes including units and division sizes.

(3) ALWAYS draw the line through as many experimental points as possible.

(4) ALWAYS include a title, name and date.

These few rules will help to ensure that you make the best possible graphs.

Appendix: Example of graphing; boiling points versus molecular weight.

Experimental data:

Molecular Formula/Name	Molecular Weight in g/mol	Boiling Point in ^oC [1]
C_6H_{14} / Hexane	86.18	123
C_7H_{16} / Heptane	100.21	104
C_8H_{18} / Octane	114.23	124
C_9H_{20} / Nonane	128.26	145
$C_{10}H_{22}$ / Decane	142.29	198

[1] In order to simulate expermimental data, this column was obtained by taking the published boiling points for these liquids and randomly adding +- 15% error.

Graph Ranges:

x axis range (molecular weight): 10 - 150 g/mol (this will be the width of the graph paper)
y axis range (boiling point): 50 - 200 ^oC (this will be the length of the graph paper)

Graph Paper Divisions:

x (width): 39 before axis; 35 after axis; (150-10)/35 = 4 g/mol per division
y (length): 52 before axis; 50 after axis; (200-50)/50 = 3 ^oC per division

Boiling point of methane (16 g/mol) from the line: approx. 55 ^oC.

Factor Label Method

What is it? The factor label method is an algebra intensive method of problem solving utilizing units.

Where would I use it? The factor label method is generally applicable to most problem solving situations.

What is the benefit? The factor label method has two main benefits making it a powerful technique well worth the time and effort to learn it. First, utilizing the factor label method can often predict when an answer is wrong. Second, utilization of the factor label method often removes the necessity of memorization of many formulas and equations.

What are the drawbacks? Being a problem solving system, it is not for everybody. It seems cumbersome at first, difficult to get a handle on, and other problem solving techniques exist that work well also. However, many students have never been taught *any* problem solving techniques, and would find this beneficial. However, it will take some work before the student is comfortable with the method.

The Factor Label Method Foundation:

The factor label method is based on a very simple idea; the idea of unity. Yep, unity, the number one.

One is the loneliest number that you'll ever do. Two can be as bad as one, it's the loneliest number since the number one. At least this is what "Three Dog Night" would have us believe, but in fact, one is the most powerful number that you'll ever do. It has some wonderfully mystical and powerful properties, although these properties are so well known and so common that most have never even really considered them. If you multiply a number by one, the number is not changed. If you divide a number by one, the number is not changed. And the way that we write the number one can be varied, twisted and bizarre.

Sometimes one is assumed. Take the number 12. The number 12 implies 12*1, 1*12, or even 12/1. It's this last representation that we'll dwell on. We like the representation 12/1, because it allows us to write division by 12 as a multiplication. For instance, 3/12=3*(1/12). So dividing by a number is identical to multiplying by the fraction 1 divided by that number. You'll see how this idea becomes significant a little later, but first I would like to continue with another way in which we can represent the number one.

As we know, twelve is a number. It is a number, in fact, equal to 12. But what is 12 divided by one dozen? Well, since 1 dozen=12, then

$$\frac{12}{1dozen} = \frac{12}{12} = 1$$

Then, isn't writing 12/dozen a form of writing 1? And since there are 2.54 cm in each inch, then isn't 2.54 cm / 1 in another form of writing 1? Or 5,280 ft / mile, or 1 yd /3 ft all ways of writing the number one? And aren't these constants all equal to dozen/12, 1 in/2.54 cm, 1 mile/5280 ft, or even 3 ft/ yd, respectively, since, after all, 1/1=1? We call these quantities "conversion factors."

A conversion factor is a fractional representation of the number 1. Since they are representations of 1, we are free to change the numerator and denominator *at will*, which you will eventually see is a very important characteristic in the factor label method. Another concept that is important is that of *units* being *algebraic quantities.*

If we consider units to be algebraic quantities, then we can "cancel out" equivalent units in numerators and denominators. For instance, everybody knows that if we have 3 feet, and we want to find out how many inches that is, than we simply multiply 3 by 12. However, in the factor label, we multiply 3 feet by 12 inches/foot. Recognizing the equivalents of feet and foot (feet is just plural foots!), then we can write

$$3foot * \frac{12inch}{1foot} = 36inch$$

Notice how "foot" cancels, since it is in both the numerator and denominator, just as an algebraic quantity "x" would cancel if it were both in the numerator and denominator, as in

$$x * \frac{y}{x} = y$$

The factor label method in action:

So now we've got it. Conversion factors are equivalent regardless of what is in the numerator and what is in the denominator, and units cancel algebraically. In the final analysis, all we really need to do is make sure that the units all cancel such that the units of the final answer match the units we seek.

All we really need to do is make sure that the units all cancel such that the units of the final answer match the units we seek!

I felt that that needed reiteration. It's the heart of the factor label method, however, so it warrants this reiteration.

To be sure that the units cancel, there are a few simple points to keep in mind.

(1) ALWAYS use consistent units! (For instance, feet and cm are length units, but feet don't cancel out cm!!)

(2) ALWAYS write the units down AVOIDING superfluous units (made up units that don't belong)!!
(3) ALWAYS be sure that the units cancel out by having one in the denominator and the other in the numerator or vice versa.
(4) ALWAYS be sure the units of the final answer match the units you are seeking.

Beware, however, of one point. The factor label method will tell you if an answer is wrong if the units are not the units you want. However, if a unitless number is needed (such as π), then the units will match, but the answer will still be wrong.

A few examples:

Example 1: Recently, I have been putting together an electric race car track (remember the electric slot cars? No? Am I showing my age?). The track occupies what would be my bedroom were I married, but since I'm a bachelor, it is more important to me to have an electric race car track than a bed. Anyway, I have 120 track pieces, an average of 9 inches each, on a scale of 1:87. What is the scale length of my track in miles?

GIVEN: 120 track pieces, 9 inches/piece, scale 1:87
FIND: miles of scale track
CONVERSION FACTORS: We will find we need 12 in/ft, and 5,280 ft/mile. In a problem, you typically will not know what conversion factors you will need ahead of time, but as you perform the problem, you will see how they are required. For instance, if you know the conversion factor 5,280 ft/mile, and you are at "inches" in the problem, then you will know you need to convert from inches to feet, or 12 in/ft.

Let's set it up and see what we get:

$$120 piece * \frac{9 inch}{piece} * \frac{87 inch}{inch} * \frac{foot}{12 inch} * \frac{mile}{5{,}280 foot} = 1.48 mile$$

Notice how all units cancel, piece with piece, inch by inch, foot cancels foot. The term "87 inch / 1 inch" is our scale factor. By convention, scale factors have really no units. I just included "inch" terms to make it easier to see how I got that term, but even so, the inches in this conversion factor would cancel with themselves, making the scaling factor unitless. You may also notice that the "grammar" is not quite correct, since foot is singular but there are 5,280 of them. However, using "feet" rather than "foot" can lead to confusion as to whether or not a unit can cancel, so I always just use singular units and try to avoid plurals.

Example 2: Some calculations are made much easier by not requiring the memorization of some formula, which sometimes means you can even perform a calculation without requiring that you have covered that subject in class! For instance, suppose you have a gold bar that measures about 9 inches by 5 inches by 2 inches. Given the density of gold is 19.3 g/mL, how many pounds will this bar weigh?

GIVEN: gold bar 9"x5"x2", 19.3 g/mL
FIND: pounds
CONVERSION FACTORS: We will need 1 mL = 1 cm^3, 2.54 cm/in,. 2.2046 lb/kg, 1,000 g/kg. In a problem, you would have a table of conversion factors that you can choose from.

First, let's find the volume independently of the factor label method. We know we need volume, because mL, milliliter, is a volume. We have a height, width and length, and the volume of the gold bar is height times width times length, or

$$9inch * 5inch * 2inch = 90inch^3$$

Notice that even here I used labels. Now that we have our volume, let's solve this problem using the factor label method.

$$90inch^3 * \left(\frac{2.54cm}{inch}\right)^3 * \frac{mL}{cm^3} * \frac{19.3g}{mL} * \frac{kg}{1000g} * \frac{2.2046pound}{kg} = 62.8pound$$

A gold bar weighs over 60 pounds! Consider THAT the next time you see a movie where they are handling gold bars single handedly!!

Notice above that we needed to convert from in^3 to cm^3, but all we had was the conversion factor 2.54 cm/in. If we had multiplied 90 in^3 * (2.54 cm/in), we would have seen that only ONE of the "inch" units would have canceled, leaving us with 228.6 in^2cm, a meaningless number. To cancel all three inches in the in^3 term, we had to cube our conversion factor 2.54 cm/in, as shown above. Another approach would have been to cube this number before hand and use the conversion factor 16.387 cm^3/in^3. *Carry out this calculation and be sure that your answer matches that shown in order to be sure that you are "cubing" correctly!*

<u>*Example 3:*</u> The nearest star is about 4.5 light years away (one light year is the distance light will travel in a vacuum in one year). The Apollo spacecraft travelled about as fast as a bullet, which we can approximate as 1,200 ft/second. Suppose that we stock a craft which travels at 1,200 ft/sec with a crew of men and women who are to create a new generation to take control of the ship every 20 years. How many new generations will be required before reaching Alpha-Centauri?

GIVEN: 4.5 light years, 1,200 ft/sec, 1 generation/20 years
FIND: generations
CONVERSION FACTORS: we will need $3.0x10^{10}$ cm/s (speed of light), 365.25 days/year, 24 hr/day, 60 min/hr, 60 sec/min, 2.54 cm/in, 12 in/ft
OK, first, let's see what distance we are talking, thereby converting this into a standard distance/speed problem.

$$45 year * \frac{365.25 day}{year} * \frac{24 hour}{day} * \frac{60 \min}{hour} * \frac{60 \sec}{\min} * \frac{3.0x10^{10} cm}{\sec} = 4.3x10^{18} cm$$

Now, let's finish up the problem.

$$4.3x10^{18} cm * \frac{inch}{2.54 cm} * \frac{ft}{12 inch} * \frac{\sec}{1{,}200 ft} * \frac{\min}{60 \sec} * \frac{hr}{60 \min} * \frac{day}{24 hour} * \frac{year}{365.25 day} * \frac{generation}{20 year} =$$

$$186{,}000 generations$$

This problem was inspired by an old episode of Star Trek.

Example 4: In the last few problems, the starting point has always been the number with one single unit (as opposed to this *per* that), so here's a problem that is slightly different. The velocity of light (as we've seen) is $3.0x10^{10}$ cm / sec. What is this speed in miles / hour?

GIVEN: $3.0x10^{10}$ cm/sec
FIND: velocity in miles / hour
CONVERSION FACTORS: We'll need 2.54 cm / in, 12 in / ft, 5280 ft / mile, 60 sec / min and 60 min / hour

OK, in this case the starting point is pretty obvious, but this won't always be the case. If you're not sure where to start, start anywhere and just work it out. This *can* lead to the problem of having the correct units but in the inverse arrangement. This problem can be solved by simply inverting the answer, as will be seen in the following example. As for the present example, let's work it out.

$$\frac{3.0x10^{10} cm}{\sec} * \frac{inch}{2.54 cm} * \frac{foot}{12 inch} * \frac{mile}{5{,}280 foot} * \frac{60 \sec}{\min} * \frac{60 \min}{hour} = 6.7x10^{8}\ mile/hour$$

Notice in this one that we let the unit "sec" slide along until we first converted from cm to mile.

Example 5: Finally, not all problems will go along seamlessly. One problem that shows up periodically is when you get the correct units, but in the inverse order (that is the numerator and denominator are switched). There are two ways to solve this difficulty, one is to go back and start over again with the starting point put in the inverse way that you started before (for instance, if you started with feet / sec, then we would use sec / feet). The other is to notice that taking 1. / the answer will put the units in the right way, which is simply one divided by the answer. You may want to initially try both ways, one to verify the other, until you are comfortable with this little "factor label trick". Here's an example problem. If we add 150,000 cal to 100. g of water with a specific heat of 1.0 cal / g ^{o}C, what will be the temperature change of the water in degrees Celsius?

GIVEN: 150,000 cal, 100. g, 1.0 cal / g ^{0}C
FIND: Temperature change in ^{o}C

CONVERSION FACTORS: no additional conversion factors needed

Notice that we have not one, but *two* single unit numbers (150,000 cal and 100. g). With which do we start? Well, without any further information, it's not clear, so we'll start with one of them, say 100 g, and we'll just keep going. So, let's set it up.

$$100.g * \frac{1.0cal}{g^oC} * \frac{1}{150{,}000cal} = 6.67x10^{-4}\,{}^oC^{-1}$$

There are a couple of things to note about this problem. The first is that the last term is written as 1/150,000 cal. I knew I needed calories in the denominator to cancel calories in the numerator, but 150,000 calories is a single unit number. Thus, I recognize that 150,000 cal is equivalent to 150,000 cal / 1, so to get calories in the numerator, I just "flip" this number around. I did NOT add any superfluous or artificial units to the "1" in the numerator. When we write 150,000 cal in this fashion, this means that we *divide* by 150,000 cal.

Finally, we note that our final units here are $^oC^{-1}$. This is NOT the unit we wanted, which was simply ^oC, so to get our final answer, we take 1. / our answer above, or $1./6.67x10^{-4}\ {}^oC^{-1} = 1{,}500\ {}^oC$. The unit $^oC^{-1}$ becomes simply ^oC on inversion of the answer, and we get the answer we seek. The very first time I worked out this problem, I chose mass to start with and my answer was the inverse of that which I sought. Now I know that I *should have* started with calories, which would have given the correct units. However, I chose to start with mass again here to demonstrate that when problems arise, it is not (typically) the end of reality as we know it.

Practice Problems:

Ready to try a few problems on your own? If so, let's go; following you will find a series of problems for you to practice on your own. Answers are provided, but YOU get to get there on your own!

Problem 1: Here's a classic style of problem used to practice Factor-Label. Suppose we have a farmer who has four cows ready to go to market. He trades the cow for chicken at the rate of 20 chicken per cow. He trades the chickens for pig at a rate of 15 chicken per 2 pig. On average, each pig weighs 140 lb. The pig he trades for corn, 3 bushels of corn per pound of pig. The corn is then traded for wheat at 1 bushel of wheat per 2 bushels of corn. Wheat is premium, so he trades it for rye, 5 bushels of rye per bushel of wheat. The rye he then gives to a baker, who makes 500 loaves of bread for each bushel. (I'm making these numbers up, so I have no idea how realistic they are.) The loaves of bread are given to a pet store who can feed 3 birds per loaf of bread. The birds are used to feed snakes, and each snake eats an average of 9 birds before being sold for $32.00 each. How much money did the pet store make from the four cows?'

Answer: Wow, what a horse trader, as it were. Turns out the pet store made $59,733,333.33. I'm in the *wrong* business!

Problem 2: OK...how about a simpler one, but one that you will need to be able to do eventually in chemistry? First a one-step calculation: The density of ethyl xanthogenic ester is 1.085 g/mL. What volume in mL would you need to get 100 g?

Answer: Even if you don't know what ethyl xanthogenic ester is, or even what density is, you can solve this problem using the factor label method (which turns out to be 92.2 mL).

Problem 3: Another one-step problem: If the molecular weight of the rather long-named compound 5β-Pregnan-20α-ol-3-one is 318.50 g/mol, then how many moles (abbreviated mol) would you have if you had 15.00 g?

Answer: Just what *is* this stuff? Dunno...don't care, at least not for this problem. Factor label tells us the answer is 0.04710 mol.

Problem 4: OK, now let's combine the two types of problems, and see how we do. If we need 0.5000 moles of 3-hexyne, which has a molecular weight of 82.15 g/mol and density of 0.7231 g/mL, what volume in mL do we need to get?

Answer: Two step problem here, and one of the types of problems that tends to give students much grief. The answer is 56.80 mL...how'd you do?

Basic Laboratory Statistics

In chemistry, we work very hard to control the environment to ensure that our results are well understood. We make sure that our equipment is clean to avoid extraneous reactions, we control temperature, heck, sometimes, we even control the amount of light in the room. It is not uncommon to find organic laboratories that happen to have windows to have aluminum foil over those windows to block any extraneous light, since this is typically an un-controllable and un-accounted for factor. Most importantly, we do many repetitions of the same experiment, to ensure that the results we get are not one-time flukes. To be a valuable piece of information, all experiments must be reproducible.

As you get further ahead in your studies of chemistry, you will undoubtedly learn more about probability and statistics, but this is a good time to start with some basic concepts. Although many of the formulas I will show you here are often standard in software packages such as Excel, it is important to understand the principle behind them to be able to interpret them correctly. Before we begin, though, we need to understand the two major categories of errors that can arise, and the results these will have on our findings.

Types of Errors

There are two broad categories of errors that arise, referred to as "random" or "systematic." Any specific error you can name should fit into one of these two categories, and each has a unique impact on the results.

Random error is just that, random. These are often referred to as "human error," although in truth these errors are far more common than that. For example, if you fail to get all of the reagent off of a piece of weighing paper, or if you accidentally get an unnoticed piece of dust in your reagent, these errors are random. They are unpredictable, and can have a variety of consequences, including either overestimating the value that you seek, or underestimating it.

Systematic errors are typically associated with the instruments that we use. For example, if your balance is not calibrated correctly, all of it's readings may be off by some fixed amount, say, for example, 0.11 g. These errors will always result in errors in one direction only; for example, all of our readings may be 0.11 g too high, or 0.11 g too low.

These errors impact "accuracy" and/or "precision." These terms are often used synonymously, but in fact, they each have very special and unique definitions.

"Precision" is a measure of how close all of the measurements are with one another. That is, in our misaligned balance, we may get a series of readings that are all very close to one another, and yet they are not very close to what the true value ought to be. For example, five masses of 100.00 mL of water might read 100.09, 100.11, 100.10, 100.12 and 100.10. These readings are all close to one another, but they ought to be reading closer to 100.00; they are precise, but not very accurate. Sometimes, these errors can be corrected for by using the same equipment. For example, if we are using mass difference, and get the mass of a beaker first, it's mass will also be off by 0.11 g. Thus, when we subtract the mass of the beaker from the mass of the liquid and the beaker to get

just the mass of the liquid, this systematic error will automatically cancel itself. However, it is important to note that *this error cancellation will only occur if we are consistent with the instruments that we use* since it is highly unlikely that a second instrument, in this case a balance, will be off by the same amount as the other. Thus, if we switch balances in the middle of the experiment, these systematic errors will not cancel out.

"Accuracy" is how close the mean is to the true value (which may or may not be known). For example, because of the density of water, 100.00 mL of water should have a mass of 100.00 g. You can have high accuracy with low precision. For example, suppose our balance is outside on a gusty day; we might get five readings of 95.02, 107.80, 96.42, 101.33 and 99.46. The mean of this data (see below) is 100.01 g, which is very close to what it ought to be, but the values themselves are all over the place.

Naturally, we want results that are both accurate and precise. If we cannot have both, however, which would you rather have yourself, high accuracy, or high precision?

Mean, Median and Mode

The term "average" is thrown around a lot, but has very low precision in its meaning (see how I did that?). People usually mean "mean" when speaking of average, but it can also refer to median or mode. From here on out, get in the habit of using the terms "mean," "median" or "mode" instead of "average," as this will tell people precisely what you mean.

There are three basic ways to discuss the "middle of the value" or "average" for a set of data. The "mean" is the most common, this is simply determined by adding up the individual data points, and dividing by the total number of data points:

$$mean = \bar{x} = \frac{\sum_{i=1}^{N} x_i}{N}$$

Thus, for our set of data 95.02, 107.80, 96.42, 101.33 and 99.46, we have

$$\bar{x} = \frac{95.02 + 107.80 + 96.42 + 101.33 + 99.46}{5} = 100.01$$

as our mean (see "significant figures" below).

The "median" is the middle value of the data. If we re-arrange this data in increasing (or decreasing) order, we have 95.02, 96.42, 99.46, 101.33, and 107.80. Since we have five data points, the middle data point will be the third data point, or, in this case, 99.46. This is our median. If we have an even number of data points, then the median is the mean of the two center data points. Notice that this is close to, but distinctly different from our mean.

Finally, the "mode" refers to the number that is most frequently seen. We have no mode in this data set, since none of the data values repeat. Suppose, however, that we actually have six data values, where two of them were both 101.33; then the mode would

be 101.33, and the median would be (99.46+101.33)/2=100.40. Notice that this would also change the mean to 100.27; why?

The closer the mean, the median and the mode are to one another, the more "normalized" the data is (that is, the closer the data would fit to a curve created with no systematic errors at all).

Variance and Standard Deviation

We would like to have a measure of how close our data points are to one another, or, better still, how close they are to the mean. For any given data point, we can simply do a subtraction, $\bar{x} - x_i$, but the problem is, if we try to take the average distance from the mean, or $\frac{\sum_{i=1}^{N}(\bar{x} - x_i)}{N}$, we will find that this gives us a value of zero, because of the way that the mean is defined. In other words, we will have the same error above the mean value as we have below the mean value. How can we get around this problem? Well, instead of taking the absolute difference between each data point and the mean, let's square this value instead. That is, let's find $(\bar{x} - x_i)^2$. Since the square value of any negative number is positive, NOW we can add these points together, and get a legitimate non-zero value. We call this the "Variance":

$$Variance = V = \frac{\sum_{i=1}^{N}(\bar{x} - x_i)^2}{N}$$

Notice that this does not give us the mean value of each data point from the mean, but rather the square of this value. We are really not interested in the square distance from the mean, so we define the standard deviation as

$$Stddev = \boldsymbol{s} = \sqrt{V}$$

The smaller our standard deviation is, the less spread out our data is.

Significant Figures

Possibly one of the most confusing subject for students is the concept of "significant figures." One reason that this is so might be that students tend to not understand why significant figures are, well, significant. The reason is simply this; it is the quickest and easiest way for a scientist to show to the reader how reliable their data is. An older method used to be using the "±" symbol. For example, suppose I want to tell you the temperature is 106.234456756316846541684±0.01°C. Two things immediately come to mind; first, why did I bother to write out all of those digits if I can only trust the result to ±0.01? The second is, why bother writing all of this out, including "±0.01", when 106.23 means *exactly* the same thing.

THIS is what significant figures are. The assumption is an error of ±1 in the last significant decimal point. This is why your instructor will, much to your dismay, insist that you write down "0" from time to time to show the significant figures; if we write 101.2, we are implying an accuracy of only ±0.1; however, 101.20 implies and accuracy of ±0.01, ten times the accuracy of the previous number. The "0" will not figure into any of the calculations, but it is telling the reader that you were very careful in your measurements (only 10 times more careful than if you write 101.2).

So, what are the rules for significant figures? What is significant, and what is not? Let's separate this into two different categories:

Numbers Less than One:

(1) Any non-zero number is significant;
(2) Any zero between non-zero numbers is significant;
(3) Any zero after the last non-zero number is significant;
(4) Any zero before or after the decimal point, but proceeding the first non-zero number, is NOT significant.

I know, that last one has you confused. We say "it is not significant because it merely places the decimal point," to which students typically scream "knowing where the decimal point is pretty %*$(@ significant!!!" No, actually, it's pretty %*$(@ important, but it is not significant. Let me explain a bit further; the number of zeros before the first non-zero number can change by simply changing the units. For example, there are 4 significant figures in the number 0.2330 mm, but if we convert from millimeters to meters, this number becomes 0.0002330 m. We should not be able to change the number of significant figures simply by changing the units, so "0.000" are not significant zeros, although they are very important.

Numbers Greater than One:

(1) Any non-zero number is significant
(2) Any zero between non-zero numbers is significant;
(4) Any zero after a decimal point is significant;
(5) *if there is no decimal point shown,* then any zeros after the last non-zero number is NOT significant, however;

(6) *if there is a decimal point shown,* then any zeros after the last non-zero number IS significant

I know your head hurts; have you tried aspirin? This seems like an odd thing, so why should it matter if I wrote down a simple little "." or not? I think of it this way, the author did not HAVE to write down that decimal point, since the zeros place it; thus, if (s)he does go through the effort of writing it, then the author is trying to tell us something. Thus, for example, if you ask how much money I have, I might write $100 as my answer. This means it's somewhere around $100, but I'm not giving you an exact number. Maybe it's as low as $90, or as high as $110; all you have is a ballpark figure. However, if I write $100. as my answer, notice that I did not *have* to write that decimal point. I am telling you that, ±$1, I have 100. Maybe it's only $99, or as high as $101, but you know much more precisely how much I have, and the say I tell you that I am giving you a more exact figure is with the decimal point. Notice that I can get even more precise by telling you I have $99.32; now you know, to the penny, exactly how much I have.

Mathematical Functions and Significant Figures

Often, math is required using numbers of various significant figures. We'll cover only two basic classes of mathematical functions:

(1) *Addition and Subtraction:* When adding or subtracting two or more numbers, do not worry so much about the number of significant figures. Instead, keep only the smallest number of digits after the decimal point that are significant in any of the numbers. For instance, when adding 101.2336 and 207.44, the answer will have only two places past the decimal point (308.67).

(2) *Multiplication and Division:* When multiplying or dividing, keep only the maximum number of significant figures as is in any of the digits involved. For example, 101.2336 has 7 significant figures, but 207.44 has only 5 significant figures, so our product can have only 5 significant figures (21,000.).

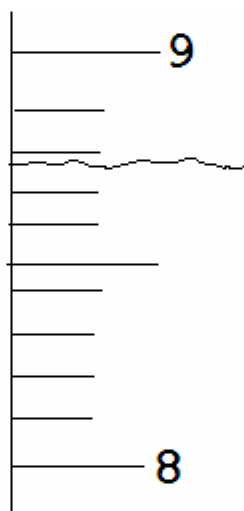
Be careful not to confuse exact numbers for inexact. For example, we know there are 12 inches in 1 foot, so if we are using this conversion factor to convert 3.0252 ft to inches, we might think we can only have two significant figures because of the factor "12." However, this is an exact definition, and as such, it can have as many significant figures as we want. In other words, even though we don't bother writing the zeros, there are really "12.000000000000000…" inches in 1 foot because this is an exact definition. Thus, our answer will be 36.302 inches (5 significant figures).

Reading Instruments

There are two types of instruments; analog and digital. Digital instruments are easy, just write down every number they give you, including zeros. This automatically

gives you the number of significant figures. However, analog devices are a little more tricky.

Analogue devices have some form of scale, with an indicator. In an old-fashioned thermometer, the scale is on the side, with the indicator being the level of the liquid. Other instruments, like voltmeters, for example, had a scale (usually with a portion mirrored so you always look at it from the same angle by lining up the pointer so you cannot see the image) with a pointer. Whenever you have an instrument like this, you can always estimate one significant figure more than the scale on the instrument.



Take the following example; suppose you are measuring the liquid in a graduated cylinder, with markings every 0.1 mL, as shown in the figure to the left. We know that the liquid level is above 8.7, but less than 8.8; so what is it? (Forgive the squiggly line; it was drawn by hand.) Well, how far up does it look to you? Maybe 70% of the way? OK, so you record 8.77 in your records. Don't worry that the last number is a guess, this is what a significant figure means! The reader knows that last 7 is not significant, but if you don't record it, then the reader will assume that the first 7 was a guess. This is also why you need to record zeros; if the line were exactly on the 8.7 line, record it as 8.70, so the reader knows that your estimate is to 0.01, not just 0.1.

Rounding

In the significant figures section, I intentionally left out how to round numbers. The rules for rounding are usually a little more involved than most people realize (not much, just a little). Remember that the purpose of rounding is to try to minimize errors in the final results. If we always round down, we know our answer will always be too low; if we always round up, the answer will always be too high. Through proper rounding techniques, we will get a mix of answers that are either too low or too high, and the errors have at least a fighting chance of canceling each other out so our final answer is close to the actual answer.

(1) Numbers greater than 5 always round up. Thus, to 4 significant figures, 22.347 becomes 22.35. Keep in mind that any number greater than five will round up; thus 654.235000000004 rounds up to 654.24 to 5 significant figures, because 0.005000000004 is greater than 0.005, even though it is not by much.

(2) Numbers less than 5 always round down. Thus, to 3 significant figures, 22.94432 rounds to 22.9. Again, though, remember that it is any number less than 5, so to six significant figures, 345.31249999999999993 rounds to 345.312, since 0.00049999999999993 is less than 0.0005, again, not by much, but it is.

(3) Numbers exactly equal to zero depend on a convention. Some people will tell you if it is exactly 5, you always round up, or always round down, but this introduces a systematic error. Instead, use this: *by convention*, if it is exactly equal to 5, always round up IF the number proceeding the 5 is odd, and always round down if the number proceeding the five is even. Thus, to 3 significant figures, 23.45 rounds to 23.4, but 63.75 rounds to 63.8. This may sound very arbitrary to you, but that is only because it is; however, it is less arbitrary than always rounding the same way. The underlying assumption is that half the time you will round up, and half the time you will round down; thus, the errors, over a long period of time, will cancel out.