

A N A L Y T I C A L C H E M I S T R Y 2 5 6
R O L E - P L A Y I N G L A B

1998-1999 Academic Year

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St. Olaf College

Northfield, MN 55057

Introduction to Role-Playing and Laboratory Computing

Defining the Kinds of Role-Playing Responsibilities

Round-Robin Certification of Laboratory Glassware

Production Quality Control Lead Analysis

Statistical/Chemical Evaluation of Lead Data

Semi-Automated Weak Acid Titration

Graphical Analysis of Weak Acid Titration

Designing a Mock Robot Experiment

The Incredible Edible Easter Egg Grass Advertising Dilemma

The Downsizing Dilemma

The Broken Pill Coating Machine Assembly Line Shutdown Dilemma

The Instrument Purchase Payment Release Dilemma

Closure

ROLES:

Manager: Teaches all about Laboratory Safety and organization
Chemist: Teaches all about **MSDS** forms, solution preparation, and disposal
Software: Teaches all how to use LabVIEW to acquire data and do file transfers.
Hardware: Teaches all how to make a cable to connect between balance and computers.

OBJECTIVES:

Starting the role-playing laboratory and exploring how the roles can work is a major task, along with communicating the essential steps of lab operation. Following a 45 minute "staff meeting" and "check-in", **Manager** organizes the Company to accomplish the above tasks before the end of the day. Of special importance is teaching safety and issue of the safety "sign-off". **Hardware** instructs in soldering cables, and **Software** explains how to use LabVIEW to read the analytical balance. **Chemist** explains how to get a **MSDS** to accompany safe chemical handling and solution preparation. **Software** also shows how to use the appletalk network and the Analytical Chemistry Server to transfer data and how to avoid launching over the network. The Company members assume teaching liability for good lab practices and explore their future roles together.

MANAGEMENT INTERVIEW:

Manager does not have to schedule an interview for this session. Instead, s/he submits the **MSDS** sheet, the "safety sign-off" sheet, and a general progress report (using e-mail) of how things went to **Staff**, with a copy to **Upper Management**. The results of the balance calibrations are part of this progress report. **Manager** may also want to report on **Chemist's** difficulty or lack thereof both in locating information on waste disposal, and how to handle the labels on the local reagent bottles, and preparing a solution, and how well **Hardware** tested and used his/her cable for reading the analytical balance. In the event there are three people in the Company, instead of four, then **Hardware** and **Software** roles are combined, and **Manager** will report how she/he balanced out the work load.

Introduction

Objectives and Responsibilities

This is the first formal experiment of the "Role-Playing" Analytical Chemistry laboratory. It also is the first role-playing experiment that many of you will have done. It also deals with some very important organizational aspects that will be with us for the rest of the semester. So, it warrants a little extra introductory material compared to our other experiments.

The **objectives** of the experiment are to set up and prepare technically what I call the "**Company Consultants**". These are the people who will take responsibility for teaching others the **laboratory procedures** for the next immediate experiment. These are critical responsibilities, and have to be approached maturely, even if they are only first time stereotypes.

There are four responsibilities. The first, which is **Manager's** to teach, is **Laboratory Safety**. The second, which is **Chemist's** to teach, is the **MSDS, Reagent Handling, Solution Preparation and Waste Disposal**. The third, which is **Software's** to teach, is **Laboratory Information Management**. The last, which is **Hardware's** to teach, is beginning **Laboratory Equipment Interfacing**. This latter responsibility has to be limited to such devices as are immediately needed, since it would be very difficult to do all of this now at the beginning of the course. The Instrumental Analysis course, Chemistry 382/378, deals more with instrument interfacing.

These objectives are the responsibilities of the people playing the roles defined above. What are the resources available? What would be a good plan for **Manager** to use when organizing the time and work of others in the company? What kind of a report should **Manager** prepare to signify the quality of the effort? These questions require some guidelines from **Upper Management** to help at the start of the course.

Responsibilities

Manager

At the start, **Manager** is responsible for making sure that all of the instruction in this experiment is accomplished for all of the people in his/her company, and especially, in delivering the instruction and demonstrations on **Laboratory Safety** that are included in the experiment.

Chemist

Chemist is responsible for instructing others in the company how to handle the hazardous chemicals in the lab, how to use a solution of alcoholic KOH to clean glassware, how to use the lab resources to identify degrees of hazard, how to dispose of the small amounts of waste that will be generated during the next experiment that uses hazardous chemicals, and, of most importance, how to use a beaker and volumetric flask to prepare a solution of known composition..

Software

There are several computers that we will use during the semester. There are two at each company. **Software** is responsible for instructing those in the Company, and especially **Chemist** and **Manager**, in the operation of the Macintosh lab computer to acquire an **MSDS** set of information, and in showing **Manager** how to edit the resulting data via Microsoft Word so that it can be included in his/her progress report.

Additionally **Software** is responsible for showing **Manager**, and any other members of the Company that **Manager** feels need to know, how to use the Mac computers to access the **Analytical Chemistry Server** to transfer an experiment to the bench so that it can be read (and/or printed on the course laser) using Microsoft Word. Since there are no paper copies of the experiments distributed at the start of the course, this is an especially important task for **Software** and **Manager**.

Staff will need to show **Software** how to launch a LabVIEW “Virtual Instrument”, so **Software** can instruct others in the Company. Furthermore, **Software** will have the responsibility of showing others how to launch Microsoft Excel. **Manager** will need to do some beginning tutorial work on it as a way of keeping electronic laboratory records.

Hardware

The Company balances (one electronic analytical and one electronic top loading), the Company pH meter, and digital voltmeter will be on the bench, but they will be unconnected, unlevelled, and uncalibrated. **Hardware** will read the manuals to learn how to connect, calibrate, and operate these devices and teach this to the others in the Company. **Hardware** then follows diagrams to solder a cable together to link the analytical balance to the Mac 7200 computer so that they can be operated via a LabVIEW program.

Methods and Resources

This first lab is complex in its administration. For this reason, **Upper Management** and **Staff** will call and hold a pre-lab organizational staff meeting with the local Company **Managers** to help them get organized. While not wishing to usurp **Manager's** authority and initiative, **Upper Management** will communicate the following points. You may assume that something relating to these will happen in the lab period.

Safety

No one can describe a set of rules or regulations that will prevent all accidents. Even if it were possible to do this in general, there would be so many local circumstances that differed according to the people involved that the collection would probably be more trouble than it was worth. Instead, what **Manager** has to do here is to make sure that everyone in the Company **knows what conditions and hazards exist** in our labs that **could** cause an injury if someone did not know about them and was doing their lab work in a normal, mature manner.

To help with this, **Upper Management** has assembled a set of "safety rules" that he has learned "the hard way" in teaching this lab in the university environment since 1961. Some of them relate to days of political unrest at the University of Wisconsin, Madison, from 1968-1972. Some are from earlier times at the University of Illinois. Some were taken from situations at St. Olaf. All of them apply to just this course! **Manager** can assume that by teaching others in the Company these "rules", s/he will cover all the situations that **Upper Management** knows have happened at one time or another in this, or a similar lab.

Chemicals

Manager will explain that there is a consultant for handling corrosive and hazardous chemicals. That person is the one who plays the role of **Chemist** in today's experiment. We are fortunate in having very few chemicals that are difficult to handle. But, **Chemist** will demonstrate how, and how not to:

- pour acids,
- dispense concentrated NaOH,
- dispose of acid/water mixtures,
- handle the heavy metal wastes,
- digest solids on a hot plate without bumping.

Computers

Our first organized period together was devoted to an introduction to laboratory computing. Thus, additional written instructions on the use of the Excel spreadsheet and Microsoft Word word processor are not appended to this experiment write-up.

What is needed most, is a tutorial introduction to how to read and translate an **MSDS** form for **Chemist** to distribute to others on the bench. **Software** will learn how to do this in collaboration with **Chemist**.

Instruments

There are a limited number of instruments that we will use, and some of them can be set up and learned "on the fly". However, there are some that are used so often that they must be learned by **Hardware** at the very start of the course, and then taught by round-robin methods to others.

In addition, some instruments require constant calibration and certification before each lab period. These are the two balances (analytical and top loading), the pH meter, and (perhaps!) the spectrophotometer. **Hardware** will learn and teach these using the Company instruction manuals.

Organization

This role-playing lab is different than your other labs in that it is **Manager** who handles all of the organization of the tasks to be done during the period. It is also different in that it is **Manager** who keeps the electronic laboratory records and from them prepares the report for the entire experiment (even if it is just a progress report), and submits it electronically either to **Staff** or **Upper Management** via electronic mail and in the Management Interview.

Thus, from the start, all who will be **Manager** must anticipate how to get his/her staff coordinated, working interdependently, aware of what each other is doing, and interested in doing good work. To help with this, **Manager** needs some information on management. To this end, there are some books that can be profitably read during this first period:

Initial Responsibilities for Manager

The initial teaching responsibilities for **Manager** are to instruct the group in, and assume accountability for the instruction in, lab bench safety, organization, and record keeping. In addition, each person should be made aware of the resources available to **Manager**, and how to use them to develop skills in small group dynamics. **Manager** also has to coordinate the next experiment on certification of analytical glassware and reagents.

The primary responsibility of **Manager** in this experiment is to make sure that everyone in the group has read and understood the **Safety Policy and Procedures** called for in the daily operation of the lab.

Safety instruction is a serious responsibility. To help make it more manageable to a novice, **Upper Management** has assembled the **Safety Policy and Procedures** material that follows based on 30+ years of teaching experience in analytical chemistry laboratories in three academic environments. Additional input came from the American Chemical Society booklet called Safety in Academic Chemistry Laboratories. **Manager** has to understand "safety" at the conceptual level before this teaching responsibility can be accepted well. The common misconception is that laboratory safety implies giving directions that prevent accidents. This is wrong. The only person who can prevent an accident is the person actually doing the work that could produce an accident if done incorrectly. It follows then that **Manager's** responsibility for laboratory safety is the collective process of **informing and motivating**.

Informing means telling your people what the **known safety hazards of the laboratory environment are**. That means, from the start, that anyone who is managing must themselves have read this, looked at the lab environment with the hazards in mind, and taken the time to develop a plan for communicating them to his/her staff in a way that they will understand them.

Motivating means developing ways that enable your staff to read and understand the information about the known safety hazards of lab operation without feeling manipulated, controlled, or in other ways treated in a condescending or degrading manner. In other words, **motivating** means empowering your staff to be aware of laboratory safety on a personal basis. So done, each staff person feels comfortable and secure in a personal way. Each person feels they are aware of what can be problem situations. Each person feels knowledgeable and informed. Motivating does not mean controlling or intimidating.

It takes good management to handle laboratory safety!

What are the tools available to **Manager** to tackle a problem this challenging? I have assembled some "**Tools for the Manager Role**". They are as follows:

Safety_Policy During the "war at home" that took place at the University of Wisconsin, Madison, in the late 60's, four professors in the chemistry department (Don Gaines (inorganic), John Walters (analytical), Claude Woods (physical), and Marion O'Leary (organic)), began what has turned into a life-long effort in safety instruction for new teaching assistants and undergraduates.

The result of that effort was the precursor to the document included at the end of this write-up called **Safety Policy and Procedures**. The UW document underwent many revisions, including four that were done after Professor Walters moved to St. Olaf in 1982. It represents the best document he can give you that delineates what can happen in an analytical chemistry laboratory that could produce an unsafe condition or contribute to an accident.

The_Lab_Map It is hard to get a facilities overview, especially with regard to potential hazards, from just being in a lab room. You are almost always too close to the detail to see the way that **reverberations** from simple actions can compound into situations. To help with this overview, Professor Walters has prepared a "**lab map**". This "lab map" is a diagram of the SC321 room when it is not packed with equipment, stools, and other apparatus that we will be using this semester. **Manager** should mark the location of known safety devices, hazards, potentially troublesome conditions, and human "hot spots". The map can then be used to show others in the Company where hazardous spots are, and where safety devices, such as the fire extinguishers and eye fountain are.

The_ACS_Book The American Chemical Society is concerned with laboratory safety. They take it very seriously. The issue is one of both protection and potential malpractice litigation. To help all people in academic laboratories understand what has been learned about what can go wrong, in general, in academic labs, they have published the booklet *Safety in Academic Chemistry Laboratories*. Not only have they done an excellent job in writing and publishing this, they have signaled how important it is to have each student read it by selling it to us for only \$1.50 each. The ACS takes laboratory safety seriously enough to pay for it!

Human_Skills People do science, not the converse.

The best equipment in the world just rusts unless a human decides to use it (robots included!). You, as **Manager** need more than anyone else in the Company to have access to simple documents that describe in clear terms what human skills you can cultivate to communicate with and motivate your staff. The tools for effective management are the three books on management that I have provided for each Company. These books are too expensive to give to each person. Instead, there is one copy for each Company, labeled with the Company name. These books may be borrowed for reading, but should be available for each person who wants to read them before they do a **Manager** role play without hassle. The books are:

The One-Minute Manager, Blanchard & Johnson

Empowering Leadership, Roberts and Thorsheim

The Giving Tree, Shel Silverstein













In addition to these, there are available copies of M. Scott Peck's marvelous book on interdependence, The Different Drum, which **Upper Management** can loan out to any who want to read it. It is also available in the library, and can be found using SAGE. The book is longer than the above, and deals with many concepts, so it is a developmental resource for long term growth for planning out the **Manager** role. For some, it is well worth detailed reading, especially those who are interested in combining a career in economics with any of the physical sciences.

What are some ways you should manage this first **Manager's** responsibilities lab session? Here are **Upper Management's** recommendations, based on work done before the role-playing labs were fully implemented at St. Olaf.

Read the ACS Booklet "Safety in Academic Chemistry Laboratories" and highlight in yellow those passages in the handout "Safety Policy and Procedures + The Map" that are also mentioned in the ACS booklet.

Read the Handout "Safety Policy and Procedures + The Map" to your group out loud (I have done this for years!). As you come upon certain points that need illustration in the lab, mark them on "The Map" and then observe them at the same time in the lab room.

Walk around the lab with your staff and **annotate the map** by labeling the proper location with the proper name of the following equipment:

The safety showers (both of them):	-----	
The yellow fire extinguishers:	-----	
The red fire extinguishers:	-----	
The fire blanket:	-----	
Exits to use in case of fire:	-----	
The hoods for exhausting noxious fumes:	-----	
Closest running water for washing eyes:	-----	
The "Approved" eye wash fountain:	-----	
The first aid kit:	-----	
The closest telephone to use to call 911:	-----	
The person who drives you to the hospital:	-----	
The stockroom:	-----	

Sign (and have each person on your staff sign) the last page of this handout indicating that they have indeed read all of the handout called "Safety Policy and Procedures", and do in fact understand what it says and how what it says applies to the specific conditions in the SC321 lab room, as shown on "The Map".

Show **Upper Management** the signed sheets for your group, and ask and answer any questions that result from this.

Hand In to **Upper Management** the signed page from the handout on "Safety Policy and Procedures" indicating that the material has been read and understood one for each person on your staff, including yourself, before leaving the lab that afternoon.

SAFETY POLICY AND PROCEDURES

(1.) REPORT ANY ACCIDENT, NO MATTER HOW TRIVIAL, IMMEDIATELY TO THE INSTRUCTOR, WITHOUT FEAR OF CRITICISM OR PENALTY.

(2.) A person may do chemical work in the laboratory **ONLY** when an instructor or assistant who is involved in the course management is **physically present**. The lab will be instructed regularly and open from 1-5 p.m. as scheduled in the College calendar; more time will not be needed to do the work in the spirit intended. Do not plan on coming in "after hours" to do chemical work alone.

(a.) Work, in the above chemical sense, includes **any and all chemically based operations associated with execution of the experiment**, such as washing dishes, weighing samples, using the ovens, hoods, sinks, wet benches, handling or using chemicals or chemical glassware, causing reactions, and developing chemical methods with others. It is assumed that the person doing chemical work in the lab will have the explicit permission of the instructor or assistant to be doing so, at that time.

(b.) In short, no one may do chemical work in the lab alone.

(c.) The lab computers may be used for analytical chemistry coursework at any time during regular stockroom hours, but, if chemical work is going on while they are being used, the instructor or assistant must be present.

(3.) No work, in any form, may be done in the laboratory WITHOUT THE WEARING OF PROFESSIONALLY APPROVED SAFETY GLASSES when ANY CHEMICAL WORK of ANY KIND is going on in the lab at the same time.

(a.) This **does** include work in the instrument room. If you will be going in and out of the instrument room on a periodic basis, it will also be necessary to be putting on and taking off your safety glasses on a periodic basis. It might be simpler to leave them on.

(b.) It is important that only approved safety glasses be worn in the laboratory. Sun glasses, plastic wind glasses or their equivalents are not approved. Tempered and hardened prescription glasses are also not approved. Conventional, hardened prescription glasses may be worn in a laboratory only if they are covered with laboratory type plastic goggles.

(c.) **CONTACT LENSES ARE VERY DANGEROUS IN A LABORATORY. THEY SHOULD NOT BE WORN IN THIS LAB.**

It is not clear how different kinds of contact lenses will resist lab fumes. If a spill were to occur, it is not clear how you could be effected. Wear conventional glasses under approved safety glasses as a replacement for contact lenses. Buy conventional glasses if you do not have them, and wear them to lab as soon as you have them.

Any student doing chemical work in the laboratory for any reason without approved safety glasses will be asked to leave the room. **There are NO EXCEPTIONS to this policy.**

If a person does not do this, then they will be asked to drop the course.

- (4.) A laboratory coat or apron or smock should be worn in the lab. Shorts should not be worn. It is not wise to wear good clothes to the lab. A lab coat, smock, or apron should be used to protect your clothing from spills. One lab coat will be hanging by each hood. There is only one provided for each hood. Personal garments are not provided. When doing harsh work this coat may be used.
- (5.) Awareness and care should be exercised to avoid coming in contact with **inconspicuous residues** left or occurring on the laboratory benches. Sponges are provided to clean the benches. Your immediate work area should be cleaned with a wet sponge and dried with a scrap towel both before starting any work and after any work is done. Your work area should be kept clean, preferably dry, and well organized while you are using it.
- (6.) Clothing with loose portions, such as open sweaters, baggy cuffs, hanging scarves, and so forth should not be worn in the laboratory. Loose clothing can catch on standing glassware, (knocking it over) can **drag through spills**, and can inhibit skillful handling of small lab ware. Loose or cuffed sleeves in a wet unknown residue on the bench may become highly hazardous.
- (7.) Loose, long hair should be tied up and/or back such that it will not inhibit your ability to concentrate or to focus on small motion motor skills. Loose hair is similar to loose clothing in causing laboratory accidents by distracting you when you are trying to manipulate, pour, or transfer solutions. It should be bound in a manner to prevent its interference with careful manipulation of reagents or glassware or apparatus.

(8.) Your feet should be protected against spilled chemicals, both on top and bottom. **It is an absolute RULE that SHOES MUST BE WORN IN THE LABORATORY, even if only passing through.**

Under no circumstances may anyone be in the laboratory in bare feet. Conventional shoes, with soles and tops in good condition and of material with sensible resistance to chemical attack should be worn.

- (9.) Eating or drinking are not allowed in the laboratory. Smoking is prohibited throughout the building. Many of the chemicals used in the lab could prove toxic in small amounts if ingested. You should not put anything in your mouth when working with chemicals. You should wash your hands thoroughly after handling chemicals. You may adopt the attitude that no chemical should be touched by human hands. Refer to the **MSDS** on all chemicals before handling them with unprotected hands.
- (10.) Radios, personal stereos with earphones ("walkman" devices), televisions, tape recorders, "boom boxes", and similar devices that would distract either your concentration or that of those around you are not desired in the laboratory.

Your laboratory work, and that of those around you, requires calm, careful concentration with studied attention to interrelated chemical and instrumental details. Demonstrations, public speeches, music, loud noise, unexpected disturbances, and similar sounds are sources of distraction to many people, and are not appropriate in the laboratory. So too is any device that inhibits your ability to hear normal background noises and safety related comments from others nearby.

- (11.) Persons not enrolled in the course, or for any other reason not **formally** aware of **THESE** laboratory policies and procedures, should **not visit with you IN the laboratory room**.

Visits, problem sessions, collaborations on the course, and informal conversations with people who are not actually involved in the execution of any experiment, and/or not present during the initial teaching and discussion of these policies, are best held nearby, such as in the hall, but not in the laboratory room.

- (12.) **Any action commonly accepted as "horseplay", "fooling around", and/or of a personally harassing, disruptive, or aggressive or uncontrolled nature is SPECIFICALLY FORBIDDEN in the laboratory.**

- (a.) Safe laboratory work requires careful, methodical, and deliberately planned action and manipulation. Potentially hazardous chemicals or reagents must be carried and handled in containers as part of the execution of many experiments. **Any action that could unexpectedly startle or disrupt the control of another person is STRICTLY PROHIBITED in the laboratory.**
- (b.) Public speaking, group rallies, demonstrations (passive or active), sit-ins, picketing, striking, or other group based activities that are not part of the scheduled laboratory work and **cause disruption of the safe execution of that work** should not be caused to occur in the laboratory room. Persons causing such to occur will be asked to leave.
- (c.) Harassment of any nature can produce stress, which can lead to tension and unsafe laboratory actions. Harassment is not to occur. If a person perceives that they are being harassed, then they are, regardless of the intent of the person perceived to be harassing. Any harassment of a personal, ethical, or sexual nature should be reported to **Upper Management**, without fear and with complete surety of whatever confidentiality desired.

- (13.) Laboratory waste and residues are to be disposed of in an advised and approved manner. Waste containers will be provided, and explicitly pointed out at the start of and during the lab period. Separate waste containers will be provided for liquid and solid chemical wastes. Some experiments will require creation of waste disposal methods as part of the chemical manipulations of the experiment, and will make use of waste containers provided. Put chemical liquids and solids only in such designated containers. Never put liquids into the sink without first checking the **MSDS**, or with the lab professor or assistant.

Potentially unstable combinations of strong oxidants and/or reductants should not be disposed of in any container without first checking the **MSDS** or with the lab professor. In any case where you either are unsure of proper and safe waste disposal, or do not wish to be responsible for judging correct procedure, feel encouraged to ask the lab professor for advice before acting. The **MSDS** will be descriptive, but also may need interpretation. Waste disposal books are in the library for guidance, and can be consulted.

- (14.) **Label all containers** (bottles, beakers, jars, flasks, and so forth) with a readable and complete description of their contents. Many of the reagents we use have to be stored for as long as two weeks between uses. It is always difficult, and usually impossible, to recall with safe surety what is in an unlabeled bottle, even if only a few minutes have passed since filling. It also can be dangerous when the bottle is emptied. Proper labels will always be available from **Staff** or **Upper Management**. Do not assume that you are so pressed for time that the few minutes it takes to label a bottle is unavailable. **LABEL FIRST, THEN FILL!**

- (15.) Locate all of the following objects in our laboratory before beginning any work:

Safety Shower	_____
Fire Extinguisher	_____
Fire Blanket	_____
Room and Fire Exits	_____
Fume Hoods	_____
Eye Fountain	_____
Closest Running Water	_____
First Aid Kit	_____
Closest Telephone	_____
Stockroom	_____

Be SURE that you know how to get to these resources BY MEMORY. Do not assume that your vision will always be clear; consider how you would get to any of these devices were the room darkened, due to a power failure in a storm or fire, or smoke filled. It is the same problem you would face in a hotel fire.

- (16.) Occasionally it seems necessary to smell the products of a chemical reaction. Do so only if it actually is necessary (color often is better than smell for signaling reaction progress), and then only with the greatest of caution. **NEVER sniff the fumes coming from a container at close distance.** Rather, lightly waft a bit of contaminated air by your nose at arm's length.

NEVER put your nose directly over a container and inhale. You may severely damage your nose and throat.

- (17.) There is only one correct way to fill a pipette. That is by using a pipette bulb. **Under no circumstances should a pipette be filled by mouth, no matter what you have seen or been told in other courses or under other circumstances.**

Small amounts of liquids to be pipetted should be transferred carefully from master reagent bottles into labeled beakers for use at your bench. Then the pipette should be filled from these, as opposed to from the large, and often heavy, reagent bottles.

- (18.) Heating and boiling untended and unclamped beakers and flasks is very dangerous. Take the time to knowledgeably assemble professionally proper apparatus before heating or boiling liquids in either beakers or flasks. If you are unsure, please ask for advice.

Heating a volumetric flask on a hot plate is unwise. These flasks are designed to contain exact amounts of liquid at room temperature, and are poorly shaped for direct dissolution. Avoid heating in volumetric flasks altogether. Use an Erlenmeyer flask instead.

Heating a STOPPERED volumetric or other stoppered flask on a hot plate is acutely dangerous, and may cause violent sprays of the liquid being heated to be ejected out of the top of the flask.

- (19.) Reagent bottles generally are placed on fiberglas™ trays, and have plastic or ground glass stoppers that may be gripped while dispensing. Take the time to touch the stopper to the inside of the bottle before pouring so that it does not drip onto the outside of the bottle.

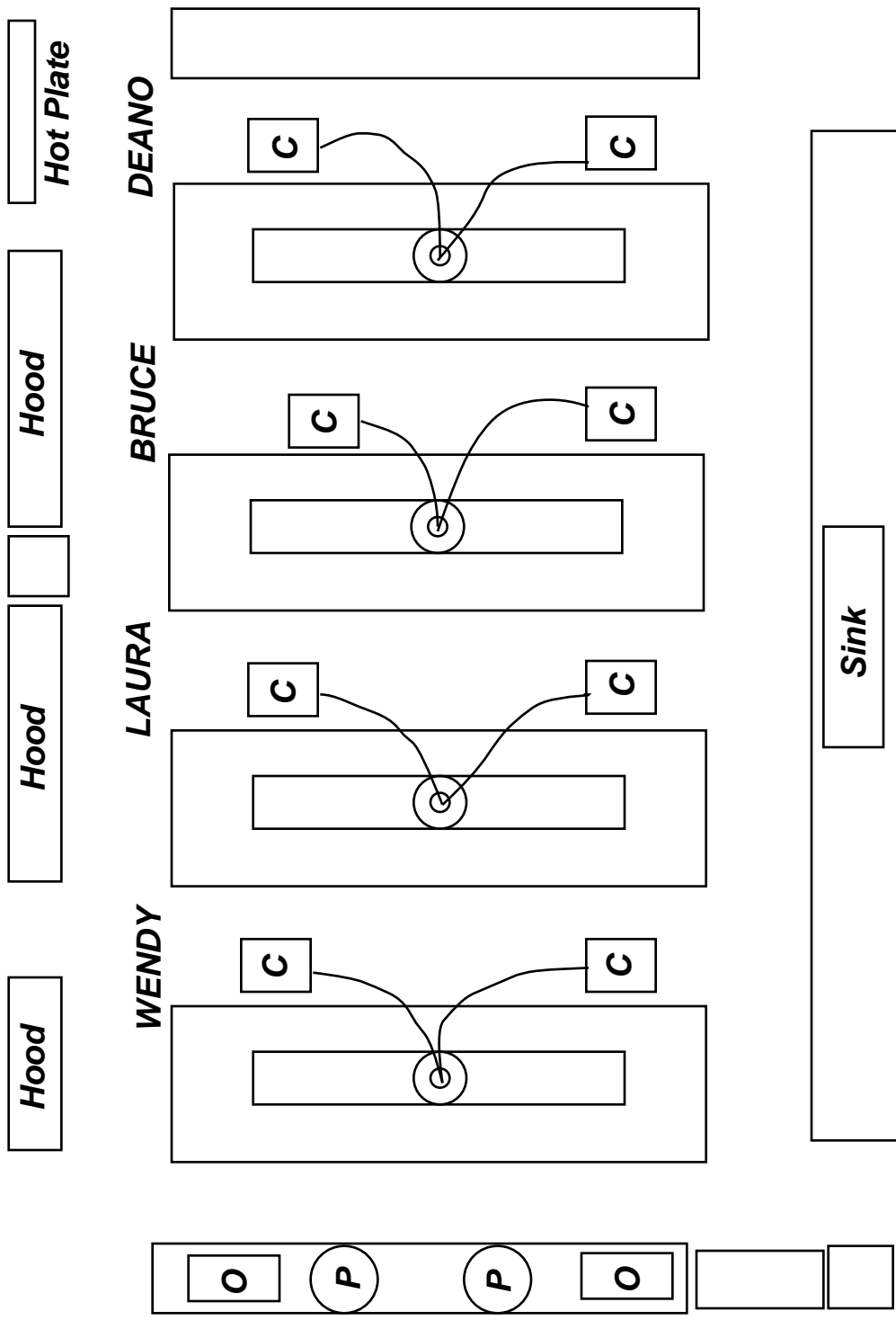
A damp sponge should be used to wipe off the residues on reagent bottles so others will not get them on their hands. Then use a towel to dry the bottle. This will take time! Then carefully place the sponge in the sink under running water to clean it, taking particular care not to touch the places where the chemical is.

- (20.) There will be few, if any, opportunities to work with glass rods or tubing during the semester. Should it be necessary to bend or break glass rods or tubing, or to insert them through holes in a rubber stopper, first consult your lab professor or assistant. Do not improvise. Do not invent new ways to do this. Ask for tutorial help in all cases.
- (21.) Do not attempt to clean glassware with corrosive or strongly oxidizing acidic solutions. Many cleaning solutions are so corrosive as to be hazardous in the hands of a novice. Some are mutagenic. Some are carcinogenic. If there is a need to clean volumetric or other glassware with any cleaning solution other than soap and deionized water, then either have it done by your lab professor or assistant, or have them demonstrate what solutions to use. Do not use corrosive cleaning solutions on your own without professional assistance. Always check the **MSDS** first for toxicity.
- (22.) Do not dispense concentrated acids in anything other than a slow, deliberate, and totally aware manner. Do not carry beakers or graduated cylinders of concentrated acid from the dispensing location to your bench with anything other than the utmost caution.
- (a.) In a few experiments, it will be necessary to obtain **a few milliliters** of concentrated acid or base from a central dispenser or from the stockroom. This is to be done only when the lab professor or assistant is in the immediate vicinity, and then only in a calm, unhurried, and alert manner.
- (b.) If, **for any reason**, personal or professional, you should feel **even slightly uncomfortable** with this task, please feel completely at ease in asking that it be done for you by either your lab professor or assistant. There is absolutely no educational necessity to having you transport reagents, and it is **NO PROBLEM** to have it done should you feel even just a bit uncomfortable about doing it. Peer response to asking for help is of absolutely no professional relevance.
- (23.) Should you spill a chemical onto yourself or your clothing, it is essential to flood the area involved with water as soon as possible. Contaminated clothing should be removed as soon as possible so that your skin may be flooded with water. **Large amounts of flowing tap water are needed, applied without hesitation and IMMEDIATELY AFTER the spill.** Safety showers are available in the lab for this. Again, ask for help!
- (24.) No work of a chemical nature should be done until **all people in the group doing the work** have seen and acknowledged the meaning of the **MSDS** associated with all of the chemical reagents and expected reaction products involved in the work.

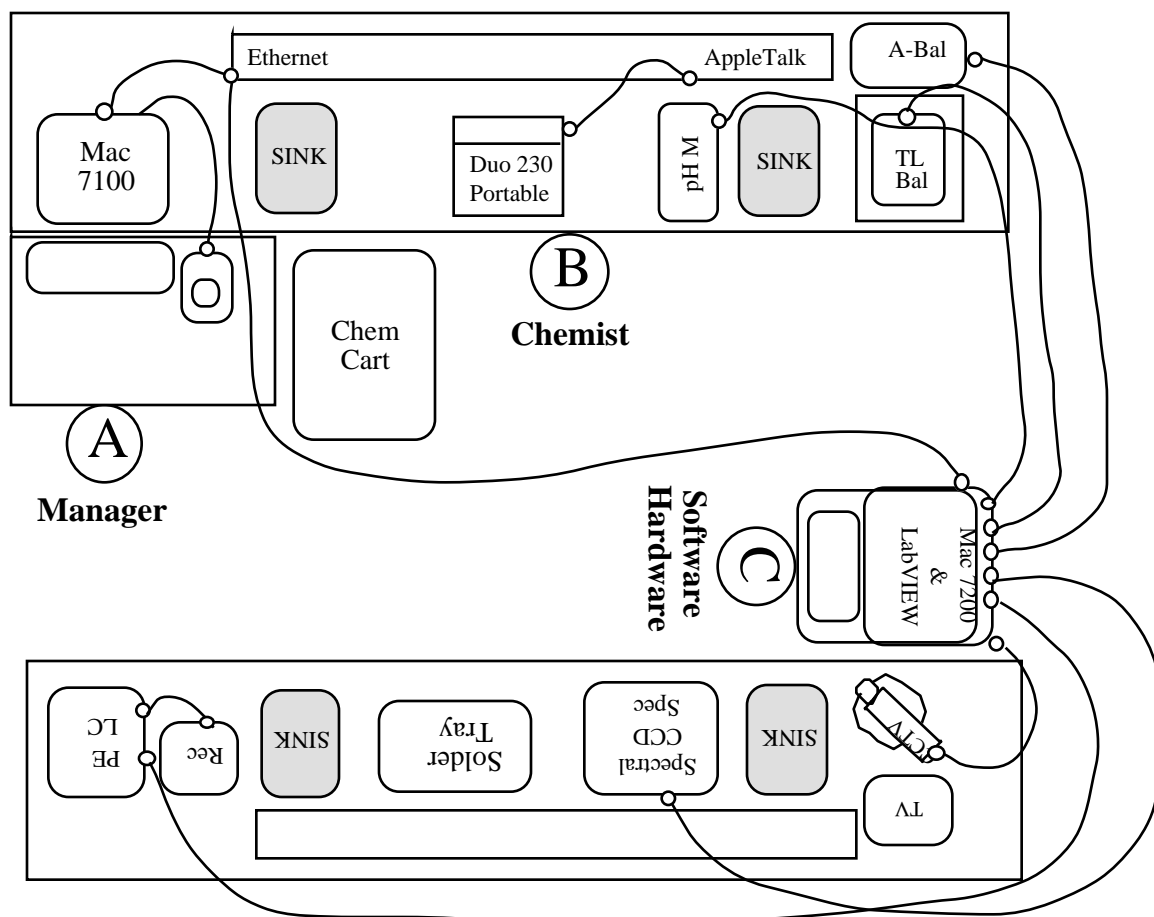
The **MSDS** is a legitimate and valuable piece of information regarding handling, using, and disposing of laboratory reagents.

**NO MATTER HOW TRIVIAL IT SEEMS,
REPORT ANY ACCIDENT
IMMEDIATELY
TO UPPER MANAGEMENT OR
STAFF.**

The Map



The Company Work Area



There may be small differences in the instrument layout on the benches from the time this figure was prepared, and the time you come to lab. Class rearrangements often cause this, as do recent equipment updates and new instrument purchases. In the event that you have a Company of four people, the **Hardware** and **Software** roles will be played by two different people. There then will be two seats on the lower bench. Otherwise the Company bay areas are essentially as shown above.

Safety Policy and Procedures Signature Sheet

I have read **all** of the material in the Safety Policy and Procedures + The Map handout for the Spring or Fall Semester offering of Chemistry 256 laboratory, and **understand how it applies** to the lab room SC321 in which I will be working. I have asked for clarification of those points I did not understand. I have either been given a copy of the book Safety in Academic Chemistry Laboratories, as published by the American Chemical Society, or read a copy available for this purpose, and understand that it is to my professional benefit to read it in relation to my work in Chemistry 256 lab.

Signed: _____ Date: _____

Print Name: _____

Company: _____, on: _____, Manager: _____

Accepted: _____ Date: _____

Remarks:

INITIAL RESPONSIBILITIES FOR CHEMIST

The initial responsibilities for **Chemist** concern reagent handling, solution preparation, waste disposal, and providing "right-to-know" safety specifications to others in the Company for possible substance toxicity.

By far the biggest challenge that **Chemist** faces in initial responsibilities is determining the safety, handling, and disposal procedures for the chemicals that are going to be used in the next few weeks of lab work. In addition, proper bottling and labeling of these chemicals according to their hazards to people has to be done.

The hazards of just handling chemicals, even those that we often consider "harmless" are finally coming to public attention. Started in the 60's as a movement of social awareness, environmentalism has stretched into concern for all environments in which people have to work, and concern for what they work with. Chemists, as a professional class, are "on the point" in this concern, if for no other reason than the fact that they themselves have to work in an environment that is daily altered by the wide variety of substances that can be toxic, or hazardous, if mishandled. In the last few years, new information has become commonly available that indicates better what these hazards can be, and even goes so far as to suggest what to do about them.

In our lab, chemicals are considered necessary tools of the trade. But, at the same time, they may also be considered as necessary evils when it comes to handling with human hands. For this reason, each Company is equipped with a box of disposable, lightweight gloves. It is expected that **Chemist** will want to wear some of these when handling open bottles of stock chemicals, and even, on occasion, when working with diluted solutions of stock chemicals. What viewpoint prevails depends on the situation.

Chemist has the responsibilities of finding out the hazards of each chemical that will be used in an experiment. This consists of the following steps:

- (1.) Going to the Science Library or to the WWW or to the lab collection with a copy of the **Local Material Safety Data Sheet** (as appended here), looking in the yellow "Material Safety Data Sheet Handbooks" in the appropriate place, extracting all of the data needed to fill out the sheet, and making it available when completed to read by each member of the Company. For MSDS information see the following URL:
<http://www.chem.utah.edu/MSDS/MSDS.html>
- (2.) Alternately, **Chemist** may collaborate with **Software** to use the CD-ROM facility available for use on any of the Mac computers in the lab to view and print from the Aldrich Safety Database and extract a **Material Safety Data Sheet** on any of the chemicals that will be used by the Company in that lab session. The value to this is in practicing a professional procedure, as the Aldrich database is used in many professional industrial situations that are firewall insulated from connection with the internet for proprietary protection.
- (3.) Fill out a set of **NFPA Diamond Labels** to use on any chemicals that will be used in the coming experiment. These labels will be available to each Company, and the bottles used to hold the chemicals when taking from the stock shelf will be the ones to be so labeled.

These steps are conceptually simple, but may take some skill to get going when **Chemist** is unfamiliar with them. For this reason, **Upper Management** will consult with both **Chemist** and **Software** during this initial experiment to help get the necessary information from whatever source of information is chosen.

During the first period, **Chemist** should learn how to fill out the **Local Material Safety Data Sheet** for the chemicals that will be used in **Production Quality Control Lead Analysis Experiment**. These chemicals are available in moderately dilute form on the reagent shelf between the hoods. They are:

Potassium Dichromate $K_2Cr_2O_7$

Potassium Bromate $KBrO_3$

Chromic Nitrate $Cr(NO_3)_3$

Lead Nitrate $Pb(NO_3)_2$

Sodium Acetate NaAc

Nitric Acid HNO_3

When these sheets are filled out, they should be taken to **Manager** for approval, and then copies made for each member of the group. Use the copy machine in the departmental office for this.

Following this, **Chemist** should prepare **NFPA Labels** for the **Intermediate Storage Bottles**, where it will be necessary to bring any of the above reagents to the lab bench. These bottles will be available from **Upper Management**, and the NFPA labels also will be available from him. A "Sharpie" **permanent** marker will be available for each Company. The NFPA codes are shown on the following page, and the values to use for each chemical solution or solid can be located in the primary MSDS source or from the chemistry stockroom.

Chemist should then determine if the **Production Quality Control Lead Analysis** experiment will require the disposal of any solid chemicals generated at the Company bench. If so, **Chemist** must transport the dry chemical waste from the bench to the waste disposal area in the orange laboratory hood.

When the **NFPA Labels** have been prepared and placed on the reagents, **Chemist** must gather the Company staff, explain the labels and the chemicals to each person, give each person a filled out **Local Material Safety Data Sheet**, and discuss with them the hazardous nature of what they will be using. For any substance that is classed with a **NFPA code** of **2** or higher in the **BLUE DIAMOND** Health Hazard Code, **Chemist** must also make handling recommendations for the Company. See **Upper Management** for tutoring on this.

National Fire Protection Agency DIAMOND HAZARD CODES**Special Hazard Code WHITE DIAMOND**Hazard Code

Oxidizer	OXY
Acid	ACID
Alkali	ALK
Corrosive	COR
Use No Water	W
Radiation	RAD

Reactivity Hazard Code YELLOW DIAMONDHazard Code

Stable	0
Unstable if Heated	1
Violent Chemical Change	2
Shock/Heat may Detonate	3
May Detonate	4

Fire Hazard Code RED DIAMONDHazard Code

Will not Burn	0
Burns Above 200 °F	1
Burns Below 200 °F	2
Burns Below 100 °F	3
Burns Below 73 °F	4

Health Hazard Code BLUE DIAMONDHazard Code

Normal Material	0
Slightly Hazardous	1
Hazardous	2
Extreme Danger	3
Deadly	4

The following material is an example of a **MSDS**

POTASSIUM DICHROMATE

Material Safety Data Sheet

Emergency Telephone Number

Mallinckrodt Inc. 314-982-5000
Science Products Division
P.O. Box M
Paris, Kentucky 40361

Effective Date: 04-06-89 Supersedes 08-08-85

PRODUCT IDENTIFICATION:

Synonyms: Dichromic Acid, Dipotassium Salt; Potassium Bichromate; Dipotassium Dichromate

Formula CAS No.: 7778-50-9 Molecular Weight: 294.18
Hazardous Ingredients: Chemical Formula: K₂Cr₂O₇
Potassium dichromate

PRECAUTIONARY MEASURES

DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE. MAY BE FATAL IF SWALLOWED. CAUSES BURNS. CONTAINS HEXAVALENT CHROMIUM. EXPOSURE MAY CREATE A CANCER RISK. HARMFUL IF INHALED.

Keep from contact with clothing and other combustible materials.
Store in a tightly closed container.
Remove and wash contaminated clothing promptly.
Do not breathe dust.
Use only with adequate ventilation.
Avoid contact with eyes, skin and clothing.
Wash thoroughly after handling.

EMERGENCY FIRST AID

If swallowed, induce vomiting immediately by giving two glasses of water and sticking finger down throat. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. In case of contact, immediately flush skin or eyes with plenty of water for at least 15 minutes.
In all cases call a physician.
SEE SECTION 5.

DOT Hazard Class: ORM-A

Physical Data

Appearance:	Orange-red crystals or powder
Odor:	Odorless.
Solubility:	6.5% @ 103C (503F) in water.
Boiling Point:	Decomposes @ 500 C (932
Vapor Density (Air=1):	No info. found.
Melting Point:	398 C (784 F)
Vapor Pressure (mm Hg):	No info. found.
Specific Gravity:	2.69
Evaporation Rate:	No info. found.
NFPA Ratings:	Health: 1
	Flammability: 0
	Reactivity: 1
	Other: Oxidizer

Fire and Explosion

SECTION 2

Fire: Not combustible, but substance is a strong oxidizer and its heat of reaction with reducing agents or combustibles may cause ignition.

Releases oxygen upon decomposition which enhances combustion.

Explosion: Contact with oxidizable substances may cause extremely violent combustion

Fire Extinguishing Media: Flood with large amounts of water. Water spray may be used to keep fire exposed containers cool. Do not allow water run off to enter sewer.

Special Information: In the event of a fire, wear full protective clothing and NIOSH-approved self-contained breathing apparatus with full face piece operated in the pressure demand or other positive pressure mode.

Reactivity Data

SECTION 3

Stability: Stable under ordinary conditions of use and storage.

Hazardous Decomposition Products: Emits oxygen and toxic fumes of chromium trioxide when heated to decomposition.

Hazardous Polymerization: This substance does not polymeric.

Incompatibilities: Reducing agents, acetone plus sulfuric acid, hydrazine, and hydroxylamine.

Leak/Spill Disposal Information

SECTION 4

Ventilate area of leak or spill. Clean-up personnel require protective clothing and respiratory protection from dust. Spills: Pick up and place in a suitable container for reclamation or disposal in a method that does not generate dust. Disposal: Whatever cannot be saved for reclamation may be disposed in a RCRA approved hazardous waste facility. Do not flush to the sewer.

Reportable Quantity (RQ)(CWA/CERCLA) : 1000 lbs.
Ensure compliance with local, state and federal regulations.

Health Hazard Information

SECTION 5

A. Exposure/Health Effects

Inhalation: May cause irritation to the respiratory tract, coughing, wheezing, fever, headache, and labored breathing. May produce pulmonary sensitization.

Ingestion: May cause abdominal pain, vomiting, dizziness, intense thirst, fever, coma, and liver damage. Death may occur from circulatory collapse or renal failure. Estimated lethal dose 5 grams.

Skin Contact: Corrosive. May cause irritation, ulceration, and scarring. Skin may become sensitized. Absorption through the skin may cause systemic poisoning; symptoms may parallel ingestion.

Eye Contact: Corrosive. May cause severe irritation, redness, pain, and conjunctivitis.

Chronic Exposure: Repeated or prolonged skin contact may cause eczematous dermatitis. Repeated or prolonged inhalation of dust may cause ulceration, bleeding, and perforation of the nasal septum.

Aggravation of Pre-existing Conditions: Persons with pre-existing skin disorders may be more susceptible to the effects of this material.

B. FIRST AID

Inhalation: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

Ingestion: If swallowed, induce vomiting immediately by giving two glasses of water and sticking finger down throat. Never give anything by mouth to an unconscious person. Call physician immediately.

Skin Exposure: In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Call a physician.

Eye Exposure: Wash eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

C. TOXICITY (RTECS, 1982)

Mutation References Cited; Reproductive Data Cited. Hexavalent Chromium compounds are listed as carcinogens by the National Toxicology Program (NTP) and by the International agency for Research on Cancer (IARC). Carcinogenic Determination Animal / Inadequate Data (IARC 23,302 80) Hexavalent Chromium Compounds: Sufficient evidence for carcinogenicity in short term testing (IARC Suppl.4, 1982).

Occupational Control Measures SECTION 6

Airborne Exposure Limits: -OSHA Permissible Exposure Limit (PEL): 0.1 mg/m³ Ceiling for chromates -ACGIH Threshold Limit Value (TLV): 0.05 mg/m³ for CrVI compounds NIOSH

Criteria Document Recommendation: Occupational
Exposure to Cr(VI) : 25 ug/m³ (TWA) Ceiling limit:50 ug/m³/15M.

Ventilation System: A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, "Industrial Ventilation, A Manual of Recommended Practices", most recent edition, for details.

Personal Respirators: If the TLV is exceeded, a dust/mist respirator (NIOSH Approved) with chemical goggles may be worn, in general, up to ten times the TLV. Consult respirator supplier for limitations. Alternatively, a supplied air full face piece respirator or airlined hood may be worn.

Skin Protection: Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls to prevent skin contact.

Eye Protection: Use chemical safety goggles and/or full face shield where dusting or splashing of solutions is possible. Contact lenses should not be worn when working with this material. Maintain eye wash fountain and quick-drench facilities in work area.

Storage and Special Information SECTION 7

Protect against physical damage. Remove and dispose of any spilled dichromates; do not return to original containers. Wash hands, face, forearms and neck when exiting restricted areas. Shower, dispose of outer clothing, change to clean garments at the end of the day. Avoid cross-contamination of street clothes. Wash hands before eating and do not eat, drink, or smoke in work-place.

How to Prepare a Solution

Chemist has the responsibility of showing others in the Company how to make a solution that has components of known molarity. While this task should be straightforward, it has been done so seldom in courses offered prior to this class that many people have virtually no experience in doing it. Mistakes are unfortunately all too common, and usually demoralizing to **Manager** and others in the Company, as they can lead to who experiment failure. Thus, this is an especially important part of this first lab.

For example, later in the semester, **Chemist** will have to dissolve a metallic sample in a particularly nasty hot mixture of hydrochloric and nitric acids known as aqua regia. After the sample has dissolved, and before any of the solution is removed from the beaker it is in, **Chemist** has to transfer absolutely all of the solution to a volumetric flask, dilute the result to the mark with deionized water, and mix thoroughly by repeated inversions for several minutes. Then an aliquot can be taken with a pipette from the volumetric flask to make subsequent dilution's.

Every semester, about half of the **Chemists** fail to do this properly, taking the aliquot from the beaker and not the filled volumetric, causing **Manager** to fail the lab for everyone in the Company.

In what follows, the correct way to make a standard solution, from which other sub-standards can be prepared by aliquot/dilution, is described. This is the correct way. In this class, there is no other way.

There are three tools that are needed to correctly prepare a standard solution from a primary standard solid substance. The first is the analytical balance, and a weighing boat. The second is an Erlenmeyer flask and hot plate combination in which to dissolve the weighed solid. The third is a volumetric flask into which the dissolved solid is quantitatively transferred and diluted to a known volume to bring it to a known concentration.

The solution that **Chemist** will prepare is 100 parts per million (ppm) of a ferric iron standard that can be used later in the semester for calibration of the new Charge Coupled Device ("CCD") spectrophotometer. The solution will be made from primary standard iron wire, dissolved in hydrochloric acid, and diluted with deionized water in a one liter volumetric flask. The following detailed directions are appropriate.

- 1.) Obtain a sample of cut strands of primary standard grade iron wire. Using plastic tweezers to hold the wire, cut several pieces into about 5 mm lengths with a scissors, allowing the pieces to fall into a plastic weighing boat.
- 2.) Using the tweezers, weigh by difference from the plastic boat into a 250 ml Erlenmeyer flask **approximately** 0.1 g of wire to **0.000x** significance. Assure that your weighing is recorded to 0.000x significance regardless of the specific weight obtained. A correct example would be 0.0985 g. An incorrect example would be 0.099 g.
- 3.) Add, in the hood, from a graduated cylinder, about 25 mL of deionized water to the Erlenmeyer flask. Then slowly add about 25 mL of concentrated HCl to the water. (Recall - **Add Acid to Water**). The wire may froth as the acid strikes it. Note that you do not know the exact amount of solution in the flask, so you do not know the concentration of the iron in it either. You cannot take an aliquot of this solution and expect to get a known amount of iron.
- 4.) When the frothing (if any) stops, cover the top of the flask with a small beaker turned upside down. Then, place the covered flask on a hot plate in the back of the hood. Adjust the hot plate so that the acid mixture is refluxing, but not boiling. There should be bubbles of hydrogen clearly visible on the surface of the iron wire as it dissolves. Complete dissolution should take place within an hour. Periodically adjust the hot plate setting so the solution does not boil or "bump".

- 5.) Inspect the flask containing the dissolved iron wire carefully. If the wire has completely dissolved, there should be no residue. The solution should be transparent, and have a faint straw yellow color. If there are black specks floating around or on the bottom of the flask, ask for professional advice.
- 6.) Using a glass funnel, a wash bottle filled with deionized water, and a stirring rod, quantitatively transfer the iron solution to a clean (but not necessarily dry) 1000 mL volumetric flask. This is a critical step. If you spill, stop here and begin again with a new sample of iron wire. A quantitative transfer is a transfer where absolutely all of the dissolved solute is transferred from the Erlenmeyer flask to the volumetric flask.
- 7.) Rinse both the funnel and the Erlenmeyer flask completely several times with deionized water from your wash bottle. Each time, quantitatively transfer the rinsing to the above one liter volumetric flask.
- 8.) Completely rinse the transfer funnel and stirring rod with deionized water, making sure that the washings all end up in the one liter volumetric flask.
- 9.) Fill the above one liter volumetric flask to the mark with deionized water, thoroughly rinsing down all of the insides of the neck in the process. Use the proper precautions to avoid overfilling. If the flask is accidentally overfilled, do not attempt to remove liquid, but instead note where the meniscus falls. A later attempt may be made to determine the actual volume present by weight. It is far better not to overfill!
- 10.) Stopper the flask and repeatedly invert it to ensure very thorough mixing. As much as 5 minutes of inverting may be required to completely mix the contents of the flask. The mixed solution is the 100 ppm standard.
- 11.) Unless exactly 0.1000 g of primary standard iron wire were weighed out, the solution will not be exactly 100 ppm. Calculate what the ppm iron is, transfer the contents of the flask to a one liter glass storage bottle, and label it appropriately.

INITIAL RESPONSIBILITIES FOR SOFTWARE

The initial teaching responsibilities that **Software** has concern **word processing** and **data processing**.

A major function for **Software** is handling the four aspects of computing that make up **Laboratory Information Management**. These are:

- | | |
|------------------------|---|
| Word_Processing | The preparation of electronic reports and instructions, the annotation of data taken from instruments, layout of data sheets, and all of those aspects keeping lab records electronically constitute technical word processing. |
| Data_Processing | The preparation of spreadsheets to hold data, portray trends graphically, do calculations on sets of data ("objects"), and print out results tables to be included in reports and such constitute technical data processing. Also included are tasks related to the use of LabVIEW. |
| Data_Storing | The preparation of data base files that will carry on over the years as archived locations for results, entering data into existing data base files, and doing selected recalls of data meeting certain criteria constitutes technical data storing. This also includes use of the Analytical Chemistry server. |

Data_Telemetry Sending data (including numbers, charts, pictures, spreadsheets, data base files, and textual reports) from one location to another, in a form where they can be used electronically, constitutes technical data telemetry. This also includes use of the Analytical Chemistry server.

Data Telemetry is quite manageable at our level. **Software will be accomplished in Data Telemetry** at the level needed in the lab by simply knowing how to drag and drop files between lab machines and the Analytical Chemistry Server.

Data Processing

Our **data processing** needs are well met without any programming! The reason for this is the spreadsheet (Excel) for the Mac. Excel is the top selling corporate program today, and has been for many years now. Whatever you are able to learn about Excel can only help you when it comes time to interview for jobs.

Additional data acquisition and processing will be done using the LabVIEW programming environment. A few LabVIEW programs may be set up from scratch. Most will be available in core form, and adjusted for appropriate display of the information acquired, and storage in a file that can be transferred to Excel.

A particularly important job for **Software** and **Chemist** will be to make out an **MSDS** sheet for every chemical that is to be used in the experiment at hand. These data can be obtained on the WWW. **Software** can use the WWW and capture the data directly from its source. I recommend that all of the **Software** people spend some time with **Staff** during this introductory lab period to learn how to do this.

When an **MSDS** sheet has been captured, it can be edited using Word, dumped to the printer, and then **Chemist** can adapt it to the local version that **Manager** needs to have everyone sign. It takes about an hour at the start of the period to pull all of this off, so **Manager** and **Software** will want to schedule accordingly! **Software** will need to know the following, and keep it recorded in a place where others can get to it when they are the **Software** rotation.

INITIAL RESPONSIBILITIES FOR HARDWARE

The initial teaching responsibilities for **Hardware** concern the calibration and operation of the electronic analytical balance and the electronic top loading balance, and the serial connection schemes between the electronic balances and the appropriate Mac computer.

Hardware has some basic, yet critical, responsibilities in this first week of lab. Later, there will be two key pieces of analytical equipment that will be needed by the other staff in the Company to make the experiment work. One is the electronic top loading balance, which will be used in place of volumetric glassware. A companion to this is the electronic analytical balance, which forms the only true primary standard for accurate weights that we have.

Hardware also will see that there is a **tray of parts available for Hardware to solder together 25 conductor cable** (none are already made up for you!) to connect to the analytical balance. The ends mate one with the balance and the other with one of the terminals near the balance. **Upper Management** or **Staff** will give a soldering tutorial to all of the **Hardware's** to show you how to make up cables.

In the case a Company has only 3 people in it, and the **Hardware** and **Software** roles are played out by one person, a commercial cable may be used. **Hardware** and **Manager** should consult on this. In the past, many people have wanted to invest the time in learning how to solder as a vocational skill that they have not had the opportunity to learn in other classes.

Hardware may also want to read the manual on, and explore the use of, the Orion, model 920, portable pH meter that will be the main sensor **transducer** that we use in the first part of the lab. All of these pieces of equipment need to be calibrated, and it is **Hardware's** responsibility to see that this happens. **Hardware** will also have to communicate the results of the calibrations to others in the Company. **Manager** will have to decide if the person playing **Hardware** has enough time in this lab period to take on this task, or if it should be deferred to a later date when another person has the role.

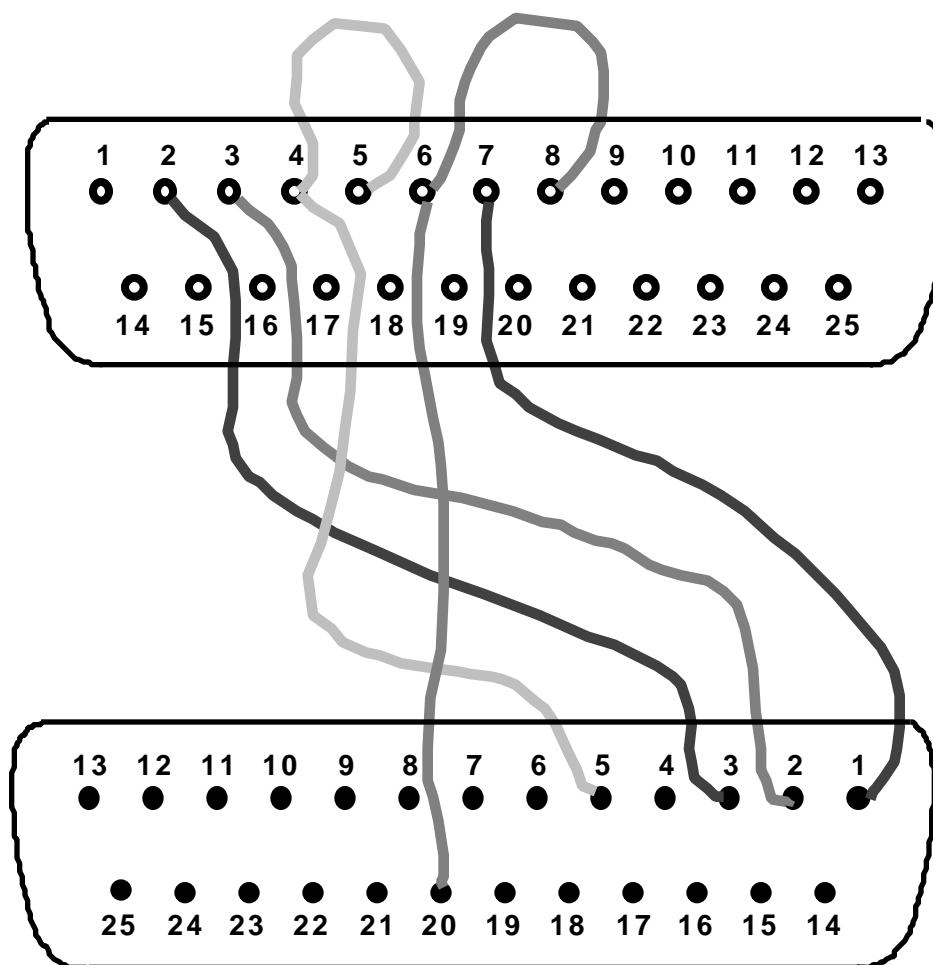
Certifying the Calibration of the Analytical Balance

To begin the analytical balance calibration certification, it first is necessary to locate a set of weights. **Upper Management** has these and will show you how to handle them so they do not change weight while they are being used. Let's begin with the top loading balance, since it is not so delicate as the analytical balance.

Next, it is necessary to make a cable to interface the analytical balance to the Mac computer serial port. Staff will tutor on how to solder the connections for this cable, using the diagram that follows.

Viewed looking into the gold solder pins

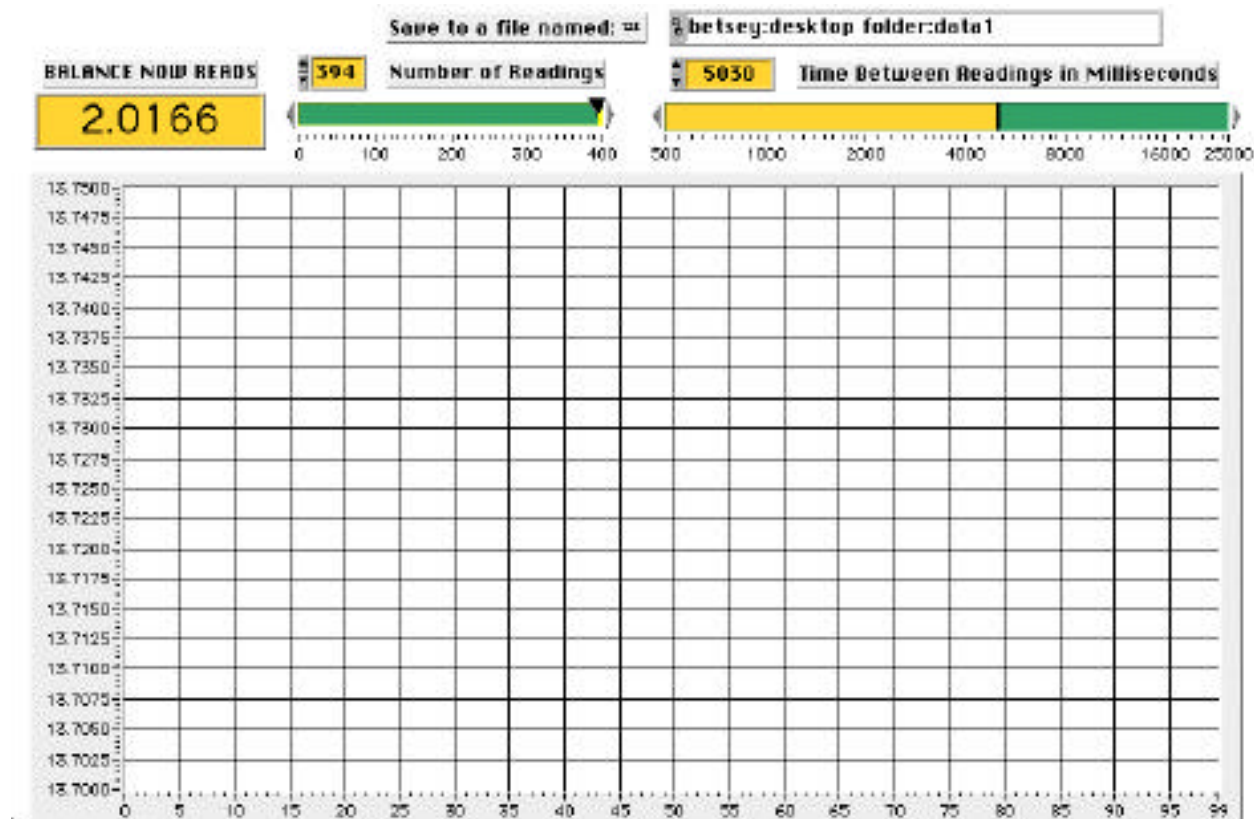
SOCKET end of cable



PIN end of cable

When wiring this cable, pay attention to the numbers on the pins. These are embossed on the connectors themselves near the pins. It is easy to make a mistake. After the cable has been wired, use the continuity tester to determine that the connections are electrically correct. Then put the “hoods” on each cable.

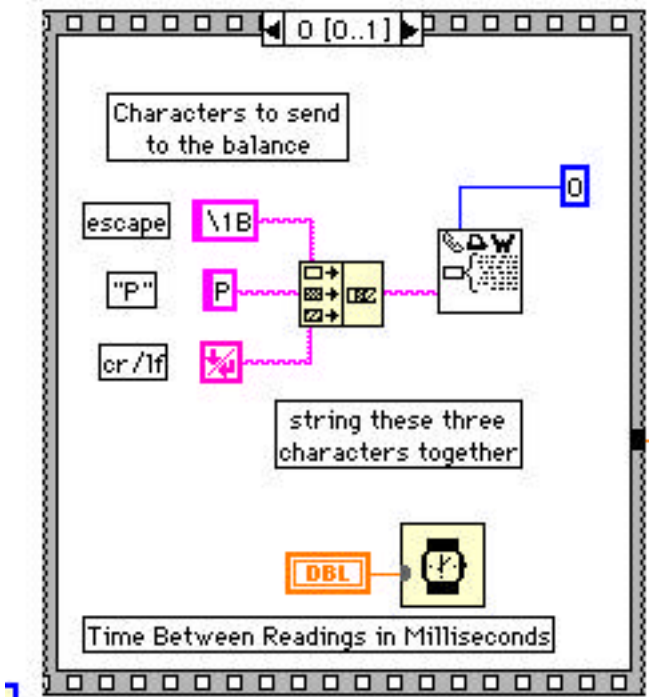
When the connections have been made between the balance and the terminal, **Software** can check their integrity by issuing some commands to the balance. This will be done using the LabVIEW “balance reader” virtual instrument shown in diagram form below:



Explore the properties of this virtual instrument to learn how often to take readings, how many readings to take, and how to adjust the ordinate limits so that you can easily see what is happening as the balance is calibrated.

When you are confident that you have the balance on line, make a set of weighings using a assortment of the standard weights **Upper Management** will provide. You will have to design an approach here. What is wanted is an equation describing the relation between the weight placed on the balance pan and the reading it reports. This is called its **transfer function**. You will have to take enough readings to find out what the transfer function is over the range 0 to 150 grams, since this is about the range it will use when weighing liquid volumes (say up to 150 mL of water).

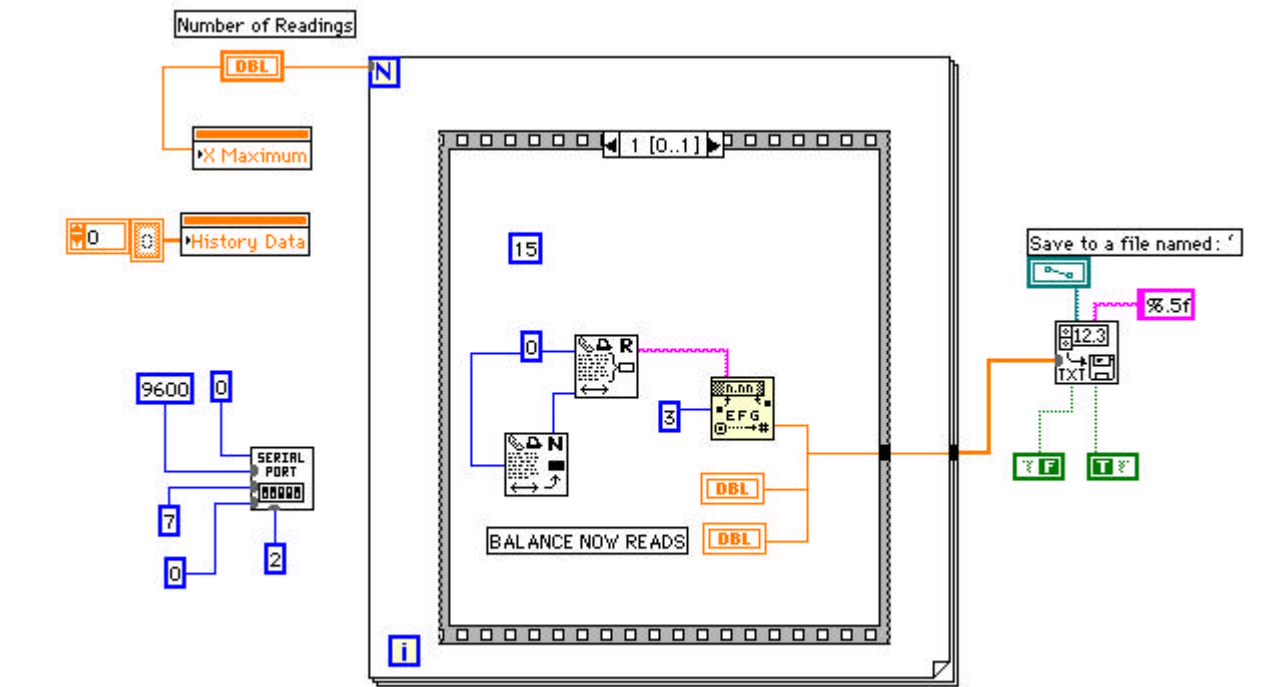
After a set of calibration readings have been obtained, and the data transferred to Excel, an interesting additional experiment can be done to see the sensitivity of the analytical balance. Obtain an aluminum tag from **Staff**. Place it on the balance pan, but leave the top and side balance doors open. Air currents in the room, which should be random, will then cause the observed weight to fluctuate. Use the above LabVIEW VI to record several hundred readings, transfer them to a spreadsheet, and use the histogram function of the spreadsheet to see if the distribution does have a random shape.



The way in which the balance reader LabVIEW virtual instrument works is shown in these two diagrams. It is unlikely that they will be sensible to you at this time. But, **Staff** can explain to you what functions the various icons perform, and how they are sequenced together to send a command to the analytical balance and get a reading back.

The diagram at the left shows what has to be “written” to the balance to activate its circuitry and cause it to send back a reading. It is the character string, “escape, capital letter P, carriage return, and line feed”.

The diagram below shows how the data that are sent back are read, “parsed”, and ultimately written to a file whose name you specify from the front panel of the instrument. Also shown are the serial parameters that have to be used to communicate with the balance, and some attributes for the front panel graphical display.



The interval between weights, and the number of points taken, will have to be enough to allow you to determine the transfer function via the technique of **least squares** numerical fitting.

BALANCE CALIBRATION			
DATE: 2-15-93			
TIME: 13:32:30			
ANALYTICAL BALANCE		Regression Output:	
WEIGHT REAL WT.(g)	MEASURED 1	Constant-	0.00015
A 50	50.0011	R Squared	0.999993
B 30	30.0012	No. of Observations	8
C 20	20.001	Degrees of Freedom	6
D 10	9.9999	X Coefficient(s)	0.99997
E 5	5.0001		
F 3	3.0003		
G 2	2.0002		
H 1	1.0003		
TOP-LOADING		Regression Output:	
	REAL	MEASURED	Constant
A	50	50.01	-0.00144
B	30	30.01	R Squared
C	20	20.01	0.999999
D	10	10.01	No. of Observations
E	5	5	8
F	3	3	Degrees of Freedom
G	2	2	6
H	1	1	X Coefficient(s)
			0.99976

Take the weights and let **Manager** know what the transfer function for the balance is. The same task has to be performed on the electronic analytical balance. This too can be interfaced to a terminal; check with **Upper Management**. The analytical balance need only be calibrated between 0 and 50 grams. The results however need to be reported to the full sensitivity of the readout that is, all figures are significant. And, if the transfer function is not an almost perfect straight line with slope of 1.000x, **Upper Management** needs to know about it right away! This is important, so have **Manager** aware of the condition of the balance right way.

A possible additional task **Manager** may want **Hardware** to assume responsibility for is **standardization of** the portable pH meter. The pH meter manual is not hard to read. Today, we will only want to do the **three point automatic calibration**, using the pH 4.001, 7.000, and 10.001 buffers provided for you. To test the calibration you may read the pH of the pH lab unknown provided.

Hardware should stay with this procedure with the manual until it works. Working from the manual is all you could do when professionally employed. **Upper Management** will consult, but this is definitely billable if it just a matter of interpreting the manual. If **Hardware** suspects an equipment malfunction, then **Upper Management** will consult for free.

If **Manager** wants to interface to the less precise top loading balance, there are a set of commands that can be used to cause many things to happen at the balance. These are described in the instruction manual under the heading General Information Regarding Instruction Set. As these commands are reviewed, note that the balance will not wake up unless it receives, at the end of the command, the sequence <cr> (the enter key) followed by a <lf> (the line feed key). That is part of the nature of the beast! Try waking it up with the **ID** command and see if it tells you who it is.

The electronic top-loading balance will respond to commands from the terminal in the following interesting manner (the electronic analytical balance is much simpler!):

<u>Command</u>	<u>Response</u>
S	SEND the next stable reading, and then stop sending
SI	SEND the next immediate reading, and then stop
SR	SEND the next stable reading, and another when ...
SNR	SEND the next stable reading, and another when ...
SIR	SEND the next immediate reading, and another, and ...
T	TARE yourself! (Set to zero)
B n	BASE (subtract) the number n from each reading
U n	UNIT of measurement will be n (see manual)
D nnn	DISPLAY as text the nnn message on the balance panel

A LabVIEW virtual instrument will be available to **Software** to use in sending one or another of these commands to the balance.

The Local Material Safety Data Sheet, MSDS

MATERIAL SAFETY DATA SHEET
 St. Olaf College - Analytical
 Chem 255/256 - Spring, 1994
 © Prof. John P. Walters

SUBSTANCE

Manager: _____ **Software:** _____ **Approved:** _____
Chemist: _____ **Hardware:** _____ **Date:** ___/___/___

Health Hazards

Inhalation _____
Skin Contact _____
Ingestion _____
Signs of Exposure _____
First Aid _____
Protective Clothing _____

DESCRIPTIVE CHEMISTRY

Physical State _____ **Appearance** _____
Boiling Point _____
Flash Point _____
Melting Point _____
Specific Gravity (H₂O = 1) _____
Vapor Density (H₂O = 1) _____
Vapor Pressure _____ **Odor** _____
Fire/Explosion Hazards _____
Fire Extinguisher Media _____ **Color** _____

Evaporation Rate
(Butyl Acetate = 1)

NFPA LABEL

Health (BLUE)
Flammability (RED)
Reactivity (YELLOW)
Special (WHITE)

Solution Properties (for Mixtures)

	Name	Formula	Oxidation State	Concentration
Cations:	_____	_____	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____
Anions:	_____	_____	_____	_____
	_____	_____	_____	_____
	_____	_____	_____	_____

Waste Disposal Procedures and Precautions

Steps to be Taken if Material is Spilled:

Waste Disposal Methods and Chemical Reactions:

Precautions to be Taken in Handling:
