LABORATORY MANUAL
GENERAL CHEMISTRY - CHM 152L

INTRODUCTION

CHM 152L is the laboratory course that should be taken concurrently with CHM 152, the second semester of general chemistry. It is assumed that the key techniques, concepts, and calculations covered in CHM 151L have been mastered. CHM 152L will explore more complex chemical concepts and problem solving. The topics of thermodynamics, kinetics, equilibrium, spectroscopy, and redox chemistry will be explored using various chemical reactions. Each student will do laboratory work, data collection and record keeping in a lab notebook, calculations, error analysis, and pre and post lab questions for each experiment. Chemical analysis, data interpretation, and the keeping of a laboratory notebook will be the focus of CHM 152L.

This course will be tied together by a cooperative project conducted by a team of students. The project will focus on the synthesis, purification, and characterization of emerald green crystals (iron salt). The synthesis and purification of the iron salt will be done in experiment C and then analyzed during subsequent experiments. The analysis or characterization will be done as extensions or applications of concepts and techniques introduced in later experiments. Student teams will have to coordinate individual analysis work to discover the empirical formula of the emerald green iron salt crystals and then write individual reports on their results.

Table of Contents:

Revised 6/2016

COURSE OBJECTIVES:

Laboratory manipulations and operations including the use of a computer interfaced spectrophotometer, pH electrode, and temperature probe.
Chemical concepts and calculations (using dimensional analysis)
- Including chemical synthesis, thermodynamics, kinetics, equilibrium, acids/bases, and redox chemistry
- Computer data collection and analysis
- Construction of graphs and plots

Record keeping with emphasis placed on correct data collection and recording in a permanent notebook
Critical analysis and interpretation of data

GRADING:

Grades will be assigned based on the grading scale and point assignments listed in the course syllabus. The point distribution is based primarily upon evaluation of the laboratory notebook, pre-lab work and quizzes, post-lab work, reported values for unknowns, and a final examination (practical and/or multiple choice). In addition, two short mini-reports will be required one for experiment B and another for the synthesis and purification (experiment C) and analysis of the iron salt (D, E, F).

The recording of procedures, data, observations, and calculations in a laboratory notebook in the proper format is a key aspect of this course. The details on how the notebook is kept and graded are provided starting on the next page. The laboratory notebook will be checked each lab period and it must be signed and dated after the last entry at the end of each lab period by the TA or instructor. Error analysis will be an important theme of post lab work done in the lab notebook. The notebook will be formally evaluated twice during the semester.

Pre-lab work begins by reading the experiment in the lab manual and concisely outlining key aspects of the experimental procedure. Pre-lab questions must then be completed and a pre-lab quiz taken before starting the experiment. Many of the questions on the pre-lab quiz will be similar to the pre-lab questions. The post-lab work will involve calculation and analysis of data and completing post lab work.

Students will also submit results for unknowns that must be within given tolerance limits to receive full credit. Points for unknowns will be assigned on how close the student's answer is to the correct answer. Students may repeat any unknown (using a new unknown sample checked out from the TA) with the best score contributing to their course points for that unknown. Point will be taken off for incomplete report sheets and repeats of unknowns.

Be aware that the CHM 152 lecture and CHM 152L are different courses with separate grades assigned for each. **Each course must be added or dropped separately.** The grade of incomplete may be given only when a course is unfinished because of illness or other conditions beyond the control of the student. The deadline to drop is published in the Class Schedule.

THE LABORATORY NOTEBOOK

Introduction:

The laboratory notebook serves several purposes; the most important of which is to be the permanent, understandable record of data and observations taken during an experiment. You should be able to look at your notebook a year from now and be able repeat the experiment or calculations. Calculations should also be shown in the lab notebook using dimensional analysis or unit cancellation. If the same calculation is repeated several times, the calculation can be shown once, and the rest of the results can be listed in a table. If a spreadsheet is used to do the calculations, a copy of it can be taped into the lab notebook.

Before you start an experiment you may wish to note key aspects of the laboratory procedure in the notebook. Any procedures not in the lab manual including changes to procedures listed in the manual must be noted in the lab notebook. The laboratory notebook used for this course should be bound and have at least 50 pages (not spiral).

At the end of each class period the TA will put a line across the notebook page under the last entry, initial by the line and put the date and time. They will do a quick check of the notebook at this time and give you suggestions on how to improve your notebook. Points will be taken off if the notebook is not checked and initialed by a TA or instructor at the end of each lab period.

The notebook will be graded twice during the course of the semester. One grading will be done at random during the course of the semester and the second will be done after the last experiment has been completed. The notebook will be evaluated based on adherence to proper format as explained below and will include observations, data, calculations, and post lab work for experiments completed. Computer generated graphs should be trimmed to size and taped into the lab notebook. A copy of the grading sheet for the notebook can be found later in the notebook information.

Format of the Notebook:

Write your name, class number and section letter, and the semester on the front of the notebook. For example:

Joe Smith CHM 152L-J Fall 2012

Also be sure to reserve the first two pages in the lab notebook for a Table of Contents. All pages used in the notebook must be numbered. Numbering the first 50 pages before you start using the notebook would be a good idea. Certain rules need to be followed when keeping a scientific laboratory notebook:

1. <u>Record all data and observations directly into the notebook.</u> This is by far the most important rule in keeping a good laboratory notebook. Do not transcribe from other pieces of paper, i.e., **DO NOT record data on scraps of paper or in this manual and then recopy the data into the notebook!** Write down exactly what you are doing and your observations as you are doing it. Errors in your procedure can be caught this way. In

addition all computer generated graphs must be printed and taped into the notebook on the same day the data is collected. Points may be taken off for writing data outside the lab notebook or not taping graphs into your notebook on the same day they are collected.

2. Clearly identify all data, graphs, and axes, and use correct units.

3. Use a black or blue ball point pen for all entries. Do not white out, erase, or obliterate any entry; simply cross out mistakes with a single line (the mistake should still be readable) and give a short note to explain the nature of the mistake, e.g., "misread." (Sometimes you will find later that the entry was not a mistake after all and you may want to retrieve the data!)

4. All the work in the notebook should be sequential going from oldest to newest entries (don't skip spaces). You may, however, start a new lab period or a new experiment on a new, unused page. If blank spaces are left in the notebook put an X through them.

5. Begin each page and new section of work with the date and title of the experiment (i.e., sections or parts of an experiment). If, when you stop work for the day, there is space left on the page for more entries enter the new date at that place when you resume work unless you are starting a new experiment.

6. As mentioned above, reserve the first two pages in the lab notebook for a Table of Contents. One convenient form for the Table of Contents is:

Experiment A – Calorimetry	pp. 4-11	9/6/12
Changes to Experimental Procedure	pp. 4-5	9/6/12
Data and Observations	pp. 5-6, 9	9/6/12
Calculations	pp. 7, 8, 10	9/9/12
Data and Calculations	pp. 11	9/13/12

Note how the data and observations are intermingled with the calculations. This student was completing the calculations each week instead of waiting to finish the whole experiment. This will be important since some of the experiments may take up to three weeks to complete.

7. Before an experiment is started, the entire experimental write up must be read. As you read, it would be a good idea to note the objectives and key points of the experimental procedure in your laboratory notebook. This will prepare you for the experiment before you come to lab.

8. Another important facet of scientific experiments involves the propagation of uncertainty in measurements and calculations. Use the correct number of significant figures, as outlined later in the introduction, in collection of data and calculations, to communicate the uncertainty or precision of your final results.

An example notebook page showing data collection and calculations can be found on page I-5. Note how data at the bottom of the page was lined through and not obliterated. Also note how unit cancellation was used when doing the calculation in the middle of the page. Be sure to use unit cancellation whenever possible!

Example Page from Laboratory Notebook

50

Caron Smith

Volumetric Chloride 152L – A

9/26/90

Preparation of $AgNO_3$ solution

Dry $AgNO_3$ 110°C In 1:00 pm out 2:15 pm (1 hr 15 min)

$AgNO_3$ + vial 12.584 g
Vial 8.059 g
$AgNO_3$ ~~4.625 g~~ 4.525 g (miscalc)

Dissolved in DW, transferred to 250-mL vol flask, diluted to mark with DW.

$$M_{AgNO_3} = 4.525\,g\,AgNO_3 \times \frac{1\,mol\,AgNO_3}{169.9\,g\,AgNO_3} \times \frac{1}{0.2500\,L\,sol'n} = 0.1065\,M$$

10/3/90

Unknown sample.

Dry at 110°C In 1:00 pm out 2:00 pm (1 hour)

Trial #1

UNK + vial ~~23.062 g~~ (bal. not zeroed)
22.743 g
Remove UNK – 22.589 g
UNK sple 0.154 g

Titration: Final reading ~~25.88~~ 15.88 mL (misread) Buret filled with $AgNO_3$ solution
Initial 0.23 mL
Delivered 15.65

CHM 152L NOTEBOOK GRADING SHEET

Name_______________________________ Dana ID____________ Section Letter____

Locker #_____ Instructor_____________________ TA_________________________

Note: Each section will be worth a certain amount of points. TAs will provide exact point distributions. For certain experiments the point distribution may be changed or other requirements added.

POINTS EARNED — AREA GRADED – check indicates area is ok.

_____/30 PTS – General Mechanics of Notebook Entry

____/3pts - data was recorded directly into notebook (very important)

____/3pts - chronological order was use from older to new work (no blank areas)

____/3pts - student's name, class #, and section letter appears on outside of notebook

____/3pts - all pages of notebook currently in use are numbered

____/3pts - current and complete table of contents

____/3pts - notebook understandable, each page and each day's lab work started with title and date, also each new section of work, readable

____/3pts - did not obliterate, use pencil, or white out but "X" or line out blank space

____/3pts - all data, calculations (use dimensional analysis), and graphs are identified and have correct units

____/3pts - reasonable significant figures were used

____/3pts - notebook initialed and dated after each lab period's work

_____PTS – All data, observations, procedures, and calculation present in notebook

	First Notebook Grading			Second Grading		
Experiment Letter	A	B	C	D	E	F
Procedure Outline/Changes	___/3	___/3	___/2	___/3	___/3	___/3
Risk Assessment	___/1	___/1	___/1	___/1	___/1	___/1
Data & Graphs	___/2	___/2	___/1	___/2	___/2	___/2
Observations	___/1	___/1	___/3	___/1	___/1	___/1
Calculations	___/3	___/1		___/3	___/3	___/3
_____PTS – Post Lab Question(s)/Work	___/5			___/5	___/5	

_____TOTAL POINTS FOR THIS GRADING (60 1st and 70pts 2nd Grading)

Special Notebook Instructions for Individual Experiments:

Experiment A

Experimental Data: Record all masses, volumes, and concentrations used.
Calculations: All time-temperature data will be collected and plotted on a computer. Tape computer generated graphs into your notebook. Record the initial and final temperatures and the temperature change determined from each graph or trial in your lab notebook by or on the graphs.

Experiment B

Experimental data: You will be doing the experimental work in groups, but each of you should maintain a copy of the data in your notebook and tape all graphs generated into your notebook before leaving lab. Make copies if needed.
Calculations: All calculations are to be done individually.

Experiment C

Student teams formed from each lab bench will acquire the data. The observations and data for the synthesis of the iron salt should be copied into each student's notebook.

Experiment D and E

Calculations: Plot graphs on a computer. Tape all graphs into your lab notebook during the lab period they are made. Record key information such as slope and correlation coefficients for Exp. D and buret readings, equivalence point, etc. for Exp. E close to or on the graphs taped into the notebook.

Experiments F (No separate instructions)

Scientific Integrity:

Scientific advances are based firmly on experimental observations and depend on the accuracy and honesty of the experimental data. The laboratory notebook must preserve the "sanctity" of data and observations--once the measurement is taken and recorded, it cannot be changed. "Dry-labbing" (using fake data) or using data from past semesters is totally unacceptable because it negates the very basis of the scientific method and is considered academic dishonesty. Proven cases will result in serious consequences such as failing the course.

SAFETY IN THE CHEMISTRY LABORATORY

Laboratory safety is a core consideration before doing experimental work and involves the prevention of and response to laboratory emergencies. Good prevention is far better than someone getting hurt. This begins with always being aware of chemical and laboratory hazards. Hazard codes, chemical labels, and safety data sheets (SDS) or material safety data sheets (MSDS) are key sources of information that help us prepare to work safely in a laboratory. This information can be used to do a **risk assessment** on the experiment you are about to do. Certain rules need to be followed to keep you safe, and you must know what to do in case of an emergency. Chemical waste management is another important aspect of a safe laboratory and a key regulatory compliance issue.

<u>Risk Assessment</u>

A risk assessment analyzes what hazards will be encountered during an experiment or lab procedure, how to mitigate them using precautions such as goggles, gloves, or a fume hood, and what should be done if something goes wrong. There may be physical or chemical hazards present that will be indicated in the experiment write up using hazard codes and other information. Labeling for reagent chemical bottles will always include hazard codes and/or special warning stickers. If a chemical has a higher hazard more information can be obtained by referring to the safety data sheet (SDS) and noting hazards and what precautions to use in response to these hazards. For every experiment you must outline the experimental procedure and write a risk assessment that includes hazards and precautions before you start lab work.

<u>Becoming Informed: Chemical Labels, Hazard Codes, and Safety Data Sheets (SDS)</u>

Label on Chemical Bottle: The first source of information is the label on a chemical bottle. Read the label carefully before using a chemical. A commercial chemical bottle will have extensive information on the label such as the chemical name and formula, physical properties, purity, molar mass, hazards, safety precautions, suggested protective equipment, hazard codes, and other information. **Chemical labels <u>must</u> include the chemical name and hazard(s).**

Baker Hazard Code: This course uses the "Baker" hazard code classification system and other systems to inform users of potentially hazardous chemicals. This system is designed to provide information to people who handle chemicals in laboratories and classifies chemical hazards according to four types: health (toxic), flammability (fire), reactivity (explosive or reactive), and contact (corrosive). The intensity of the hazard is indicated by using a number from "0" (no hazard) to "4" (extreme hazard). This information is conveyed using either a four-colored label found on "J.T.Baker" chemical products or as a series of four digits. The label on chemical bottles may look like this:

Health	Flammability	Reactivity	Contact
1	3	2	1
Blue	Red	Yellow	White

The four-digit hazard code used in the lab manual or on our reagent bottles will look like this:

1321

For example, the code listed previously for acetone indicates a slight health hazard (1), a high flammability hazard (3), a moderate reactivity hazard (2), and a slight contact hazard (1). Hazard codes will be listed after the chemical inside parentheses: (1321). The "Baker Codes" for each of the four hazards are defined according to the following scheme:

HEALTH (BLUE): Toxic effects of a substance if inhaled, ingested, or absorbed.

0. No Hazard
1. Slight hazard
2. Moderate hazard
3. Severe danger
4. Deadly, Life Threatening

FLAMMABILITY (RED): Tendency of a substance to burn.

0. Will not burn
1. Flash point above 200°F
2. Flash point between 100-200°F
3. Flash point between 73-100°F
4. Flash point below 73°F

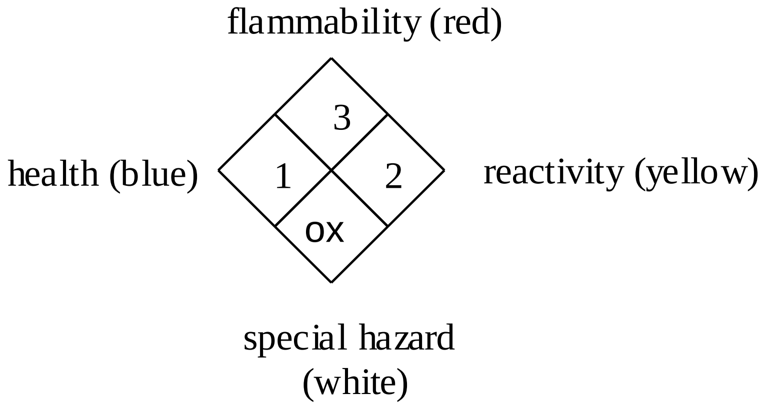
REACTIVITY (YELLOW): Potential of a substance to react violently with air, water or other substances.

0. Stable
1. Reacts under elevated temperature or when in contact with other substances under abnormal working conditions
2. Reacts violently but will probably not explode under normal working conditions
3. Reacts violently or explodes under normal working conditions when in contact with air, water or other substances
4. May react violently or detonate spontaneously under normal working conditions

CONTACT (WHITE): The danger a substance presents when it comes in contact with skin, eyes or mucous membranes.

0. No contact hazard to normal, healthy tissues
1. Slight hazard; irritant to sensitive tissues, avoid contact with eyes and mucous membranes
2. Moderate hazard; irritant to sensitive tissues, damages tissues.
3. Severe danger; destroys tissues, including skin
4. Extreme danger; life threatening

NFPA Code: The National Fire Protection Association (NFPA) uses a hazard code system that was adopted in 1975 to communicate hazards to emergency responders. This system uses a label that you may be familiar with since it appears on entrances to stores containing hazardous chemicals and on some chemical containers. The NFPA may differ from the “Baker” code since it provides information to firefighters while the “Baker” code provides hazard information in a laboratory situation. The codes are very similar except the white section in the NFPA code refers to special or specific hazards of importance to firefighters such as “ox” which stands for oxidizing agent.



The positions on the NFPA diamond are defined as follows:

Health Hazard (Blue): Degree of hazard for short-term protection.

0. Ordinary combustible hazards in a fire
1. Slightly hazardous
2. Hazardous
3. Extreme danger
4. Deadly

Flammability (Red): Susceptibility to burning.

0. Will not burn
1. Will ignite if preheated
2. Will ignite if moderately heated
3. Will ignite at most ambient conditions
4. Burns readily at ambient conditions

Reactivity, Instability (Yellow): Energy released if burned, decomposed, or mixed.

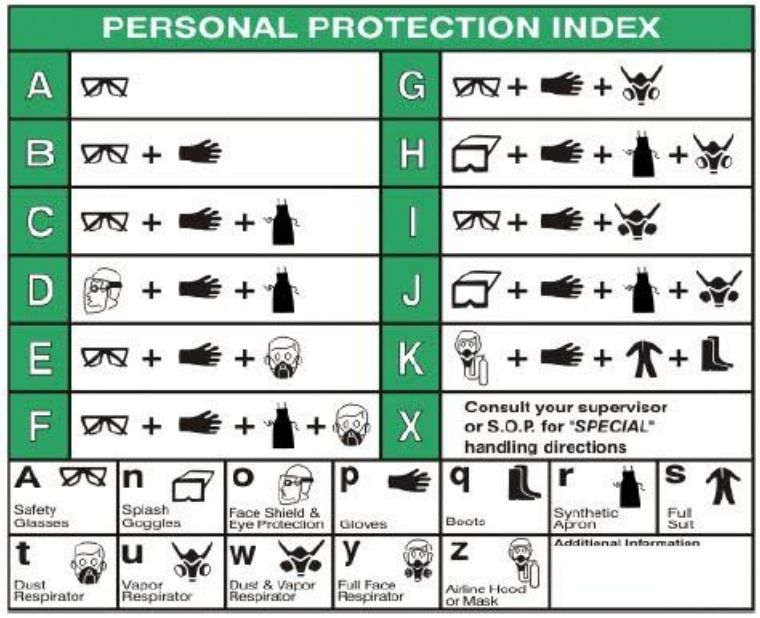
0. Stable and not reactive with water
1. Unstable if heated
2. Violent chemical change
3. Shock and heat may detonate
4. May detonate

Special Hazard (White position on diamond):

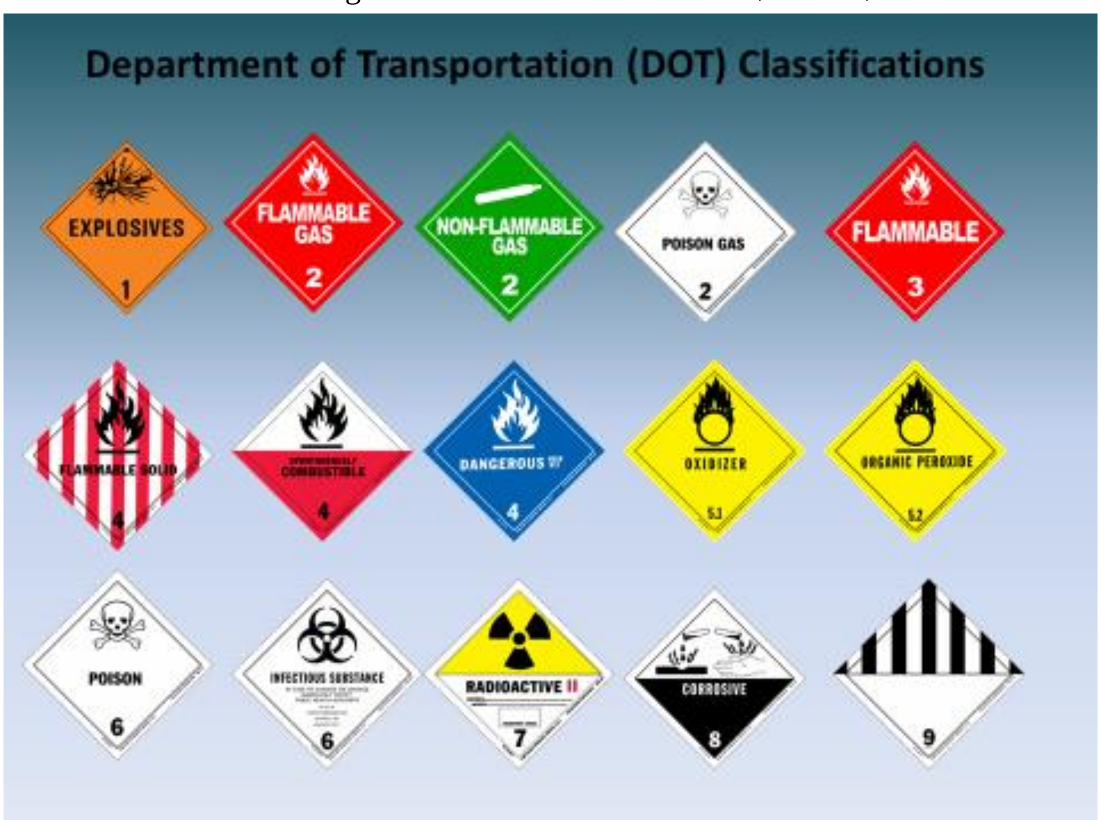
OX Oxidizer
~~W~~ Use no water, reacts!

HMIS Code System: The American Coatings Association uses the Hazardous Materials Identification System (HMIS) that is similar to the Baker and NFPA systems but includes a code for precautions to take for using a product or chemical. It also includes suggested personal protection for using chemicals or products. The code is often seen on paint and other products that might be found in a hardware store. The "*" indicates a chronic health exposure.

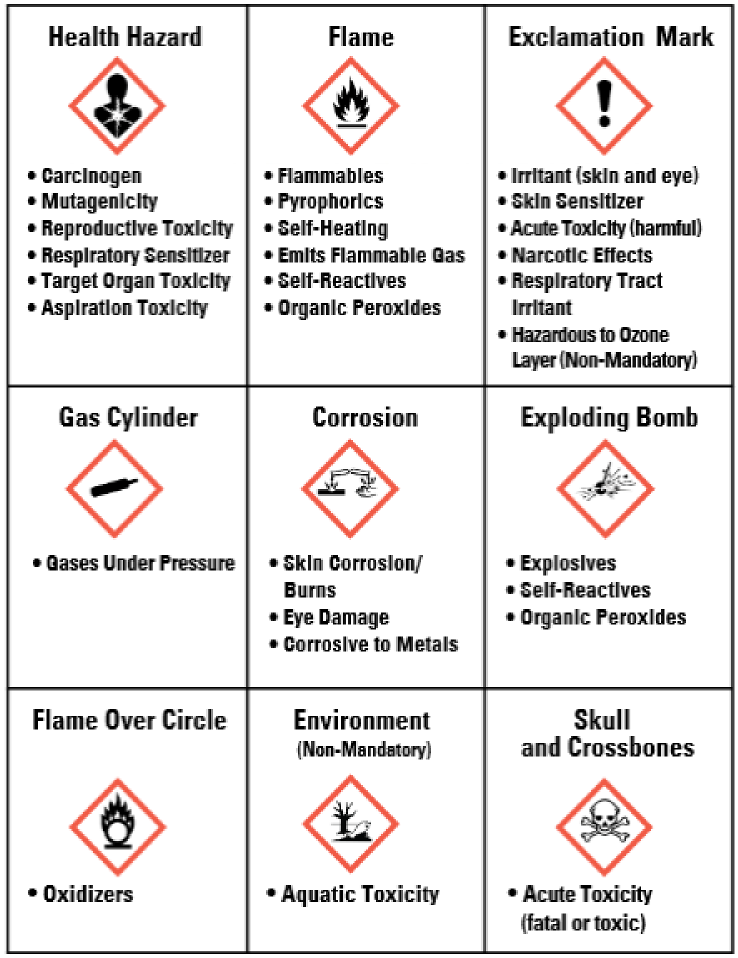
HMIS Code Label System:



Department of Transportation (DOT) Classifications: The DOT uses fifteen placards to communicate hazards for labeling containers or boxes and trucks, railcars, etc.



Globally Harmonized System (GHS): The Federal Government recently adopted the new Globally Harmonized System (GHS) hazard communication system. The GHS uses the nine hazard pictograms shown to the right to communicate chemical and product hazards. These are similar to DOT placards on the previous page. In this lab, course chemicals with the corrosive (Corrosion), oxidizers (Flame Over Circle), flammables (Flame), and toxins (Health Hazard and/or Exclamation Mark) may be encountered in lab. This system has three health hazard categories, 2-3 reactive categories, and two new additional areas: environment and gas cylinders. The SDS also uses an in-depth hazard coding system that is currently being implemented nationally and internationally.



The Safety Data Sheet (SDS)

The SDS is part of the GHS and should be read to obtain additional safety information before using hazardous chemicals. The SDS is required by OSHA for any workplace chemical hazards. If you are an employee, it is your right to have access to an SDS for any chemical product that you will likely encounter in the future. Every sheet is required to have 16 sections:

1. Identification (chemical or product)
2. Hazard Identification
3. Composition (chemical or product)
4. First-Aid Measures
5. Fire-fighting Measures
6. Accidental Release Measures
7. Handling and Storage
8. Exposure Controls and Personal Protection
9. Physical and Chemical Properties
10. Stability and Reactivity
11. Toxicological Information
12. Ecological Information
13. Disposal Considerations
14. Transport Information
15. Regulatory Information
16. Other Information

The signal words "Danger" (higher risk) and "Warning" (less risk than danger) are used in the SDS and labels. These sheets are available for all chemicals used in this course in the chemical hygiene plan notebook located in the "Right to Know Hazard Communication Station". These are to be kept in the lab at all times for reference. The Material Safety Data Sheet (MSDS, replaced by the SDS) has fewer sections and sometimes much less information and has no set format. The internet is a great resource for SDS/MSDS and other safety information. To get an SDS search the internet using the chemical name and SDS (or MSDS if SDS is not productive).

Precautions - How to Protect Yourself

1. **Eye Protection** MUST BE WORN IN THE LABORATORY AT ALL TIMES unless otherwise noted by the instructor or TA. Avoid rubbing your eyes in lab unless you wash your hands first. Use extra caution when using corrosive chemicals. Indirectly vented or nonvented goggles are the required eye protection for this lab course. Safety glasses or directly vented goggles are not acceptable. Do not modify or remove the vents on goggles. Write your name, course number, and section letter on your goggles.
2. **Skin protection** should be employed where appropriate; you may be required to wear long pants. Avoid wearing shorts. The use of a lab coat or plastic apron is recommended, but optional. Closed toed shoes must be worn at all times in the laboratory for protection against broken glass and spilled chemicals. Open-toed shoes or sandals are not appropriate footwear in lab areas. Disposable gloves are available for the handling of hazardous chemicals. Avoid touching your face or personal items while wearing gloves. <u>Always remove them before exiting the lab.</u> After completing lab work for the day, wipe down your entire work area (or any area used including the balance, fume hoods, or reagent areas) with a clean damp sponge to clean up any spilled chemicals and other material. Rinse out the sponge several times and wring it out. Wash your hands as you exit the lab.
3. **Protection from fumes or fine powders:** Never allow hazardous chemical fumes or dust to escape into the open room; use fume hoods when necessary or specified. Be sure to use the fume hoods correctly, following the instructions provided by your TA or instructor. Avoid putting your head inside the fume hood, and close the sash or fume hood window when it is not in use.
4. **Protection from internal poisoning:** Never "pipet by mouth", eat, drink, or smoke in the laboratory. These activities are prohibited. Do not keep food, beverage or personal items at your workstation. They must be kept in the designated area at the entrance to your lab. Wash your hands after you have completed lab work or leave the lab room.
5 **Protection from hot surfaces:** Use the appropriate types of tongs to handle hot objects. Test tube holders are too weak for carrying flasks.
6. **Protection from fire and explosion:** Never allow flammable vapors to escape into the open room (see No. 3). Diethyl ether is especially dangerous in this respect. Never use an open flame while flammable liquids are being used in the room. Hot plates/magnetic stirrers are an ignition source so keep away from flammables. Flammable liquids should be used in fume hoods and stored in solvent cabinets when possible. Long hair should be tied back to keep it away from open flames.
7. **Protection from cuts**: When manipulating glassware or ceramic ware, protect your hands with a cloth towel or gloves. Clean up broken glass immediately. Do not pick

up broken glass with bare hands. Use a broom and dustpan to dispose of glass in the "Broken Glass Container".

8. **Protection from the unexpected:** Always read all labels noting the chemical name, formula, concentration, and warnings (including hazard codes) carefully, and double check to make sure you have the correct chemical and concentration. Follow directions in the experimental procedure exactly. Remove obstacles by keeping lockers closed, lab chairs pushed in, and backpacks and coats stored on coat rack. For unassigned lab work, you must have the approval of the instructor. Carefully follow hazardous waste disposal instructions given later.
9. **Safety Violations:** Any student who does not follow the above guidelines will be given one warning and will then be removed from the lab for the day for any subsequent violations. There may also be grade deductions or permanent removal from the lab for serious or repeated violations.

<u>What to Do in Case of Accident</u>

1. During your first lab period, locate the position of the fire extinguishers, eyewashes, safety shower, first aid kit, phone, fire alarm pull stations, exits, hallway showers, safety data sheets, and any other safety equipment.
2. In all cases of accident or injury, notify the TA and the instructor immediately.
3. For any serious fire or injury: Call the POLICE DEPARTMENT (3-3000) from any campus phone or 523-3000 on a cell phone. Campus security is in the best position to summon fire or ambulance service. Call the Flagstaff Fire Department (8-774-1414) or dial 8-911 if Security cannot be reached. Use the FIRE ALARM PULL STATIONS (red box by every stairwell entrance) to clear the building of personnel. THE LOCAL FIRE ALARM IN THE LAB BUILDING WILL SUMMON HELP, BUT ALWAYS CONTACT CAMPUS SECURITY FROM A SAFE LOCATION TO PROVIDE DETAILS AS TO THE NATURE OF THE EMERGENCY. Students must evacuate and stay with their lab TA if it is safe to do so.
4. In case of a small fire: Immediately get help from your TA or instructor. Fire extinguishers are rated for ABC type fires in chemistry where A is combustible (paper, etc.), B is flammable liquids, C is electrical, and D is combustible metals. Use dry sand for D type fires or a "D" extinguisher. To use an extinguisher remember **"PASS": Pull the pin, Aim the hose, Squeeze the handle, and Sweep the base of the flames**. If a person's clothing is on fire, they should immediately stop-drop-roll, use the safety shower if it is close, or smother the fire with a lab coat or fire blanket. Cover beaker fires with a watch glass or larger beaker to remove oxygen and put out the fire. Cool minor burns in cold water immediately.
5. In case of chemical contact: If the area of contact is small, flush it under the nearest water tap for 15 minutes. Eyes must be flushed immediately using the eyewash at one of the sinks or the eyewash by the safety shower, keeping the contaminated eye(s) open. In case of large areas of contact, start rinsing the person using the safety shower and remove contaminated clothing. After decontamination, the person may be taken to

a shower room by an employee where rinsing will continue for at least 15 minutes or until EMS arrives, if called. Immediately inform the instructor or TA in any case.

6. In the unlikely case of a mercury (Hg) spill: Notify your TA and he or she will collect the Hg using a special spill kit. This occurs most commonly in broken mercury thermometers. This is rare now since we now use alcohol thermometers.
7. Chemical spill: If only a few drops of chemical are spilled, immediately clean up the material with a damp sponge, rinse out the sponge well at a sink, and wipe down the area a second time with the rinsed out sponge, and rinse the sponge again. In case of a larger chemical spill, immediately notify your TA and ask for help. Sodium bicarbonate (baking soda) can be used to neutralize acid spills. If the substance spilled is flammable, turn off all burners, hot plates, or electrical devices and get help from your TA. For large spills notify the instructor, staff, or faculty. Clean-up materials are available in the lab or preparation stock.

Hazardous Waste Disposal

The Resource Conservation and Recovery Act (RCRA) mandates the proper disposal of hazardous waste. Disposal of many waste chemicals by putting them down the sink is illegal. Regardless of regulations, the proper management of hazardous waste is of particular importance to the people of Arizona where the contamination of groundwater by hazardous waste could have grave consequences. Please carefully follow the instructions below to protect our groundwater and keep your lab safe. Hazardous waste is determined by four properties:

TOXIC: A poisonous substance, potentially harmful to human health, can cause cancer, birth defects, or can contaminate, harm or kill wildlife.

FLAMMABLE: A substance which can explode, ignite, or emit toxic gases or fumes if exposed to a source of ignition.

REACTIVE: An unstable substance which can react spontaneously if exposed to heat, shock, air, or water. Reactions may include fires or explosions. The research director or instructor for the lab must neutralize any reactive substance before it can be accepted for disposal.

CORROSIVE: A substance that could corrode storage containers or damage human tissue upon contact. (For example, acids and bases, pH <5 or >10)

Used chemicals in this lab that are only acidic or basic (pH <5 or >10) will be collected in the “Corrosive Liquids” bucket and will be neutralized by staff. Used chemicals with other hazardous properties (such as toxic metals) cannot be added to the corrosive liquids bucket, or it will become a mixed hazardous waste and must be disposed of following EPA rules which is much more costly. Used chemicals that do not fit into the above categories may be flushed down the drain with **large** amounts of water, but check with the instructor or TA if you are uncertain regarding disposal.

All hazardous chemical waste bottles are labeled and may be color-coded with tape. The label will include an experiment letter and a hazardous waste description that will help you decide which bottles to put your waste into. Find the correct waste bottle for your experiment number and for the type of chemical waste you have; make sure the description of the composition fits the waste you are adding to the bottle. Using the wrong waste bottle could create a safety hazard and will be treated as a safety violation. The following table should help.

Blue	-	health hazard (poisons, etc.)
Red	-	flammable hazard (organic solvents, etc.)
Yellow	-	reactivity hazard (strong oxidizers, etc.)
White	-	contact hazard (corrosives such as acids or bases, etc.)
Green	-	low hazard materials with hazard codes of 2 or less

Class	Exp.	Colors	Description of Waste	Comment
152L	C	red	Waste Acetone	Trace potassium oxalate
152L	A,D,E,F	white	Corrosive Liquids	Neutralize
152L	F	yellow,white	Waste Aqueous 0.1%$KMnO_4$	Reactive, Unused $KMnO_4$
152L	C,D,E,F	blue	Iron Salt Crystals Waste	Used or Extra, Neutralize
152L	C,F	blue	Waste Oxalates (K,Na,Unk)	Extra, Neutralize
152L	G	blue	Waste Ag,Cu,Pb,Mg,Zn	As metals and nitrates

Handling Reagents and Standard Procedures

The liquids, solids, and solutions used in a laboratory are called reagents. You must become well acquainted with these reagents, their containers, and their proper use. The reagents are kept on a separate bench or hood away from your work area. Some reagents must be kept in the fume hood because they generate flammable or toxic fumes. The reagents are grouped according to experiment, starting with Experiment A and ending with Experiment F. When you need a reagent please follow these rules:

1. Be sure to use the correct reagent. Before using the reagent, carefully check the chemical name, formula, and concentration and double check to be sure you have the right one. Note the hazard code and warnings and take necessary precautions.
2. Do not take reagent containers to your work area, and take only what you need. Conserve!
3. Do not contaminate the reagents. Always use a clean spatula for solids and clean glassware for liquids. Never put a pipet or pipettor into a liquid reagent, instead pour what is needed into a clean, dry container and take it to your work area to pipet from there.
4. Put lids back on the reagent containers snugly and put them back in the correct locations. Clean up any reagents you spill with a wet sponge, rinse out the sponge at the sink, and then wash your hands.
5. NEVER return unused reagents, liquid or solid, to the reagent bottles. Discard or share any excess. Label any container you use to store a reagent with the chemical name and hazard or hazard code. The concentration and chemical formula along with your name, section, and date would also be good information to add to the label.
6. Use great care with corrosive chemicals (strongly acidic or basic solutions). Always wear safety goggles! Rinse your hands with tap water after using corrosive chemicals, especially if you feel a burning or slimy sensation on your skin. Wear the gloves provided in the laboratory if called for. Most strong acids and bases will be disposed of in the “Corrosive Liquids bucket”, as noted in experimental procedures unless the used chemical has other hazardous properties.
7. Dispose of nonhazardous chemicals in the large sinks available in the lab. Be sure to follow the instructions in the experiments with regard to the disposal of chemicals.

8. **Pure water (PW)** is made using activated carbon filtration, reverse osmosis (RO), and ion exchange or distillation followed by UV treatment to remove any salts or organic compounds and kill any microbes that could contaminate your solutions. All pure water taps will be labeled with PW. When washing glassware, often all that is needed is to rinse well with hot tap water 4 or 5 times followed by one rinse with PW inside and out. If the glassware is really dirty use detergent or simple green, then rinse hot tap water. Then, rinse all glassware with PW from a wash bottle or carboy filled with PW before use or storage. Fill your plastic wash bottle with PW for this purpose. You do not need to dry the inside of glassware. Never store dirty glassware!
9. Hot objects can damage the lab bench surface. Never put hot objects on the bench top; instead, place hot objects on white hot pads that are provided first and then they can be placed on the counter top as long as they are clean and dry.
10. At the end of every lab period you must clean your workstation bench space and any area you used by wiping it down with a clean, damp sponge. Rinse out and wring out the sponge when you are done. Your workstation drawer must be neat and complete with clean glassware and equipment for the next student. If you break glassware during lab, be sure to obtain a replacement from your TA before you leave. Do not store your goggles, solutions, or unknowns in your workstation. Instead place them in your student storage bin.

DATA RECORDING, SIGNIFICANT FIGURES, AND ERROR ANALYSIS

Recording Experimental Data Using Correct Significant Figures

It is important to take data and report answers such that both the one doing the experiment and the reader of the reported results know how precise the results are. The simplest way of expressing this precision is by using the concept of significant figures. A **significant figure** is any digit that contributes to the accuracy of an experimentally measured number or to a number calculated from experimentally measured numbers. Please refer to the chemistry lecture textbook for a discussion pertaining to the use of significant figures.

In this laboratory course, mass, volume, time, and temperature are experimentally measured and used to calculate density, concentration, percent by mass, and other values of interest. In CHM152L, mass in grams (g) is usually measured using a top loading electronic balance with a precision of ±0.001g. Most mass measurements should be recorded to this precision even though the last digit may vary somewhat. For example, if the mass of an object on a balance reads 25.001, 25.000, 24.999 and moves between these values, 25.000 should be recorded. Recording 25, 25.0, or 25.00 would be wrong since these would not communicate the true precision of the number. If values on the balance change randomly from 25.000, 25.001, to 25.002 then 25.001g should be recorded. . For very precise mass measurements an analytical balance is used to ±0.0001g.

Time in seconds (s) is measured using a timer, stopwatch, or perhaps a clock so the precision of the measurement might vary from ±1 to ±0.01 seconds. Always record the number to the maximum precision. Temperature will be measured using an alcohol thermometer that can be read to a precision of ±0.2 °C so estimate to the tenth of a degree (i.e. 21.3 ℃).

Measuring volume in mL is a tradeoff between speed and the precision of the measurement and requires skill in choosing the right glassware for the task. When an approximate volume is needed, a beaker, Erlenmeyer flask, or graduated cylinder can be used, but when an accurate volume is needed, a pipet, pipettor, buret, or volumetric flask will be specified for use. Recognizing when to make an accurate measurement and when to be satisfied with an approximate measurement can save much time.

Frequently, the written directions will give clues to the needed precision by using the words "approximately" or "about" when the precision is not important and "exactly" or "precisely" when the precision is important. Another clue would be the number of significant figures used to write a number. For example, a volume of 5.00 mL would require the use of a 5 mL volumetric pipet or pipettor to measure the desired volume. On the other hand, a volume expressed as “about 5 mL” would require only a small beaker or graduated cylinder to measure the volume. It is also important to note that glassware used for accurate measurements is calibrated at a specific temperature, which is noted on the glassware. The precision of various types of glassware is shown in the following table:

Precision of Glassware for Volume Measurement

Equipment	Precision	Purpose of Glassware/Equipment
250 mL Beaker	±10 mL	Solution preparation, storage, reactions
125 mL Erlenmeyer flask	±6 mL	Solution preparation, storage, reactions
250 mL graduated cylinder	±1 mL	Volume transfer – moderate precision
25 mL graduated cylinder	±0.2 mL	Volume transfer – moderate precision
5 mL bottle top dispenser	±0.1 mL	Volume transfer – moderate precision
100 mL volumetric flask (class A)	±0.08 mL	Precise final volume for dilutions
10 mL measuring pipet (Mohr)	±0.05 mL	Volume transfer – good precision
5 mL pipettor	±0.025 mL	Volume transfer – very precise
25 mL buret	±0.02 mL	Precise volume delivery for titration
5, 10 mL volumetric pipet	±0.01 mL	Volume transfer – very precise

When a measurement is made, the question arises: "How many digits or figures should be recorded?" The answer is straightforward: **For a measured number record all digits, which are known with certainty, and the last digit, which is estimated.** Many of the measurements in this course involves estimation to the nearest one-fifth or one-tenth of a scale marking. For example, in Experiment 1 a 25 mL graduated cylinder, which has scale markings every 0.5 mL, should be read to the nearest 0.1 mL, estimation to the nearest one-fifth of a division. The graduated cylinder does not need to be used to this accuracy at all times; for example, if the instruction say “add about 25 mL of water” being within 1-2 mL of 25 would be ok.

NOTE: Whenever estimation between markings is being done and the reading is "on the mark," the last digit should be included to convey the idea of accuracy to the reader. For example, with a buret, which has markings every 0.1 mL, a reading on the mark of 11.3 mL would be recorded as 11.30 mL; otherwise, the reader will not know that the buret was really read to the nearest 0.01 mL. (You must estimate the last digit by looking carefully between the markings).

Another factor to take into account when measuring volume is the level of hazard for the chemical being measured. Bottle top dispensers will often be used to dispense more hazardous liquids. Pump dispensers reduce the amount of transfers from one container to another and can be used with good precision. Be sure to familiarize yourself with the use of each type of pump dispenser. Slow, deliberate use of the dispenser will help insure that the right volume is delivered.

Sometimes approximate small amounts of liquid are needed. In this case instructions may indicate to measure out drops from a dropper bottle or eye dropper. One drop of water or a dilute solution on average is about 0.05 mL. This can also be a safer method because it does not involve pouring the liquid from one container to another.

Generally speaking, all the glassware in the table on the previous page is for transferring known volumes of liquid from one container to another except for the beaker and flasks. Beakers along with erlenmeyer flasks are generally used for conducting chemical reactions or other lab manipulations. The volumetric flask is used for preparing precise solutions or dilutions.

Calculated Values and Tracking Uncertainty Using Significant Figures

Recorded data is then used to calculate some value of interest in one or more steps. You will need to know how precise or how many significant figures an answer should have depending on the precision of the data or calculated values used in the calculation and the type of math operation done. If you are unsure how many significant figures to use for calculated values it is better to use too many than too few. Using too few significant figures will introduce rounding errors into final answers! Usually keeping at least four significant figures for calculated values will prevent this.

Reporting Answers in Addition and Subtraction

When experimental data has been recorded correctly, the uncertain or estimated digit is the last digit. The calculated sum or difference of experimental measurements must be carried out only to the place where the first digit of uncertainty enters the calculation. Example: Add 14.75, 1.475, and .001475 (all of which are experimental numbers). The digits of uncertainty are underlined.

$$\begin{array}{r} 14.7\underline{5} \\ 1.47\underline{5} \\ 0.00147\underline{5} \\ \hline 16.2\underline{26}47\underline{5} \end{array}$$

but report to 16.23

Since the answer may include only the first digit of uncertainty, it should be rounded off to that digit and reported as 16.23. It helps to line the numbers up by the decimal point.

Reporting Answers in Multiplication and/or Division

1. All measurements should be recorded to the appropriate number of digits as discussed in the section on recording experimental data.
2. The position of the decimal point is ignored in counting the number of significant figures.
3. All digits except zero are always significant.
4. Zeros may or may not be significant. Leading zeros are never significant (0.02562 has 4 significant figures because neither zero is significant). Using exponential form, 2.562×10^{-2}, clarifies this issue because only the numbers before the exponent multiplier count.

a. Any zero to the right of the first non-zero digit is always significant if there is a decimal point (2.5070 has 5 significant figures since both zeros are significant).

b. If there is no decimal point, zeros to the right of non-zero digits are ambiguous. For example, if all the zeros are significant in 25000 (five significant figures) it would be much better to write the number in exponential form as 2.5000×10^4 to convey the precision. If the number is known to less precision, say three significant figures, it should be written as 2.50×10^4 to remove any question about the precision.

4. In multiplication and/or division, the answer should be reported to the same number of significant figures as the value in the computation with the least number of significant figures

Example: Find the answer to the following multiplication/division problem to the correct number of significant figures.

$$\frac{(0.085)(.08206)(366)}{(0.782)(0.14200)}$$

0.085 has 2 significant figures; 0.08206 has 4; 366 has 3; 0.782 has 3; and 0.14200 has 5. A calculator shows the answer to be 22.989865, so the answer should be reported as 23 since the number with the fewest number of significant figures, 0.085, has 2 and dictates the precision of the result.

Just Remember: When adding and subtracting, look at the fewest number of decimal places when reporting the final answer. When multiplying and dividing, look at the fewest number of significant figures when reporting the final answer.

Interpretation of Data

Significant figures are excellent to express the precision of raw data but not as good to express the precision of calculated values. **As a general rule in this laboratory course you should always use at least four significant figures for calculated values to avoid rounding errors.** In order to interpret quality of your results, certain terms are useful. You will need to understand the following definitions.

1. Accuracy: The term "accuracy" describes the nearness of a measurement to its accepted or true value.
2. Precision: The term "precision" describes the "reproducibility" of results. It can be defined as the agreement between the numerical values of two or more measurements that have been made in an identical fashion. Good precision does not necessarily mean that a result is accurate.
3. Range: The "range" is one of several ways of describing the precision of a series of measurements. The range is simply the difference between the lowest (or lower) and the highest (or higher) of the values reported. As the range becomes smaller, the precision becomes better.

   Example: Find the range of 10.06, 10.38, 10.08, and 10.12.

   Range = 10.38 – 10.06 = 0.32
4. Mean: The "mean" or "average" is the numerical value obtained by dividing the sum of a set of repeated measurements by the number of individual results in the set.

Example: Find the mean of 10.06, 10.38, 10.08, 10.12

$$\frac{10.06+10.38+10.08+10.12}{4}=10.16$$

(Note that the value 10.38, which is far greater than the other values, has a large influence on the mean, which is larger than three out of the 4 individual values.)

5. Median: The "median" of a set is that value about which all others are equally distributed, half being numerically greater and half being numerically smaller.

   If the data set has an odd number of measurements, selection of the median may be made directly.

   Example: the median of 7.9, 8.6, 7.7, 8.0 and 7.8 is 7.9, the "middle" of the five.

   For an even number of data, the average of the central pair is taken as the median.

   Example: the median of 10.06, 10.38, 10.08, and 10.12 is 10.10 which is the average of the middle pair of 10.08 and 10.12.

   Notice in the example that the median is not influenced much by the value 10.38, which differs greatly from the other three values as in the example for the mean above. For this reason, the **median is usually better** to use in reporting results than the mean for small data sets.

6. Error: The absolute error of an experimental value is the difference between it and the true value. For example if the experimental value is 30.9 and the true value is 26.5, the error would be

   30.9–26.5 or 4.4.

7. Relative percent error would be the error divided by the true value times 100:

   (4.4/26.5)x100%=16.6% or 17%.

8. Relative percent range is one way to estimate the relative percent error when we don't know what the true value is. To calculate this value divide the range by the median value and multiply by 100%. Using 10.06, 10.38, 10.08, and 10.12 from the previous items 3 and 5:

$$\frac{0.32}{10.10}x100\% = 3.2\%$$

Graphing: You will also be graphing data using a program called Graphical Analysis and doing a linear fit or regression to examine the linearity of data sets. The correlation coefficient from doing the linear regression indicates how linear the data is where 1.0000 would indicate perfectly linear data and smaller numbers such as 0.6000 a much poorer fit. Instructions will be provided for using this program. In some cases you may use excel or other software for graphing data.

Unit Analysis: Use unit conversion (unit analysis, unit cancellation, or dimensional analysis) for all calculations where appropriate. This method will help solve problems and help verify the correct solution. See the lecture text or the Instructional Labs Homepage for information.

Equipment List CHM152L: Rooms 414, 415, 416

Contents of Workstation and Student Material Bin:

Workstation Glassware and Equipment:

02 0202500 1 ( ) BEAKER 10 mL
02 0204500 1 ( ) BEAKER 50 mL
02 0205000 1 ( ) BEAKER 100 mL
02 0205500 1 ( ) BEAKER 150 mL
02 0206000 1 ( ) BEAKER 250 mL
02 0206500 1 ( ) BEAKER 400 mL
02 0207000 1 ( ) BEAKER 600 mL
02 0220100 5 ( ) FRENCH SQUARE BOTTLE
01 0207000 1 ( ) PLASTIC WASH BOTTLE-250 mL
01 0211400 1 ( ) BRUSH TEST TUBE SMALL
01 0214500 1 ( ) BULB PIPET 2 OZ
01 0328000 1 ( ) CLAMP, TEST TUBE
01 0315500 1 ( ) CLAMP EXT. 2 PRONG 9.5"
01 0317000 1 ( ) CLAMP EXT. 3 PRONG 9"
01 0318000 2 ( ) CLAMP HOLDERS FIXED
1 ( ) DROPPER, PLASTIC
02 0622500 3 ( ) ERLENMEYER FLASK 125 mL
02 0306000 1 ( ) GRADUATED CYLINDER 25 mL
02 0306500 1 ( ) GRADUATED CYLINDER 50 mL
02 1613000 1 ( ) PIPET VOLUMETRIC 5 mL
02 1613500 1 ( ) PIPET VOLUMETRIC 10 mL
00 0000000 1 ( ) Pipettor 1-5 mL
01 1622300 1 ( ) Pipettor Tip 1-5mL
01 1802500 1 ( ) RACK, TEST TUBE
02 1802500 1 ( ) ROD STIR GLASS 6"
01 1913500 1 ( ) SPATULA SCOOP ONLY
01 1915000 1 ( ) SPONGE
01 1917000 1 ( ) STIR BAR, MAGNETIC 1"
01 1956500 3 ( ) STOPPER NEOPRENE #5
01 2010500 1 ( ) THERMOMETER 110ºC
01 2013500 1 ( ) TONG CRUCIBLE
02 2015500 2 ( ) TUBE TEST 4"
02 0634500 1 ( ) Volumetric Flask 25Ml&Stopper
02 2303000 1 ( ) WATCH GLASS 4"
79 1000000 1 ( ) BIN/LOCKER CLEANUP FEE ($30)

Student Material Bin (in Section Drawer, see TA to access)

02 0216000 1 ( ) AMBER BOTTLE 16 OZ
02 0638000 1 ( ) VOLUMETRIC FLASK 100 mL
00 0000000 1 ( ) GOGGLES (student's)
01 1953500 1 ( ) STOPPER NEOPRENE #00
01 0351300 1 ( ) TWO STYROFOAM CUPS & LID
02 2201200 2 ( ) VIAL W/CAP 3 DRAM (short)

CONTRACT: I have completed a lab performance contract and assignment 1 (safety exercise) and understand my responsibilities. I will follow the safety rules at all times while in lab. I have checked the contents of my workstation locker and student materials bin and all items are present and in good condition. I understand that I am responsible from this moment for any items I break or lose in lab. Broken glassware will be replaced but you may be billed for it on your Louie account. I also understand that all workstation equipment and glassware must be present and that all glassware and my workstation must be clean at the end of each lab period or I may lose lab performance points or be charged for missing equipment

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