CHEMISTRY 123 – LAB INTRODUCTION

LAB INFORMATION

Chemistry 123 laboratory consists of two introductory exercises and four experiments selected to demonstrate a variety of chemical concepts, analytical instruments, and laboratory techniques. The course differs in several ways from Chem 121 and 122 laboratories. First, most Chem 123 experiments involve analysis of unknowns, and a major part of your laboratory grade will be based on the precision and accuracy of your results. Second, you will work alone on about half of the experiments; the remaining experiments will be performed in pairs. Third, you will enter all of your lab work into a *laboratory notebook* and will generate a complete record of each experiment, as described below.

THE LABORATORY NOTEBOOK

An important part of any laboratory experience is learning to maintain very complete records of every experiment undertaken and every item of data obtained. Far too often, careless recording of data and observations has resulted in mistakes, frustration, and lost time due to needless repetition of experiments.

Your laboratory notebook is a complete record of your work in the Chemistry 123 lab. It is used to document experimental procedures and record observations and data as you conduct each experiment in the laboratory, to complete all calculations, and to report the results of each experiment. All written work connected with Chemistry 123 lab must be done in your notebook.

Notebook Requirements: The laboratory notebook must be permanently bound, not loose leaf or spiral. The pages must be lined (or graph) and preferably numbered. If you purchase a notebook with unnumbered pages, you must number each page.

Notebook Guidelines:

- All notebook entries must be done in ink, *not pencil*.
- Date procedure and data sections as you enter them in your notebook.
- Record data directly in your notebook, never on loose pieces of paper for later transcription.
- Place the report on the right hand pages only. *Do not* use the left hand pages as scratch paper. Blank left hand pages allow a convenient place for pre-lab calculations and for recording data if the experiment must be repeated.
- Do not crowd data in your notebook; entries should be well spaced and clearly labeled.
- Cross out incorrect entries with a *single line* which does not obliterate the entry, and make the correction next to or above the crossed out entry. Never write over a number; later it may be impossible to determine which number was correct.
- Pages should never be torn out of a scientific notebook; if you wish to have a page disregarded draw a single diagonal line across it.
- Spreadsheets and graphs generated for an experiment should be *taped* securely and *neatly* into your lab notebook in such a way that the edges of the page do not stick out of the notebook.

Save the first few pages of your notebook for a Table of Contents and keep it up to date. As you prepare your notebook, allow sufficient pages for data, calculations, and summary of results, including a blank page at the end of the experiment write-up to attach the graded Data and Results Summary Sheet.

Your lab instructor will review and grade your lab notebook throughout the quarter and will assign a final grade to your notebook at the last lab session of the quarter.

Each experiment in your notebook should have the six sections described below. The first three items **must** be completed prior to beginning work on your experiment.

Prior to Laboratory Period

1. *Title of the Experiment*
2. *Purpose* - This section should contain a brief statement of the objectives of the experiment.
3. *Overview of Procedures* - In this section, you should prepare a summary of experimental procedures. Many students find that an outline of the procedure helps them use their time more efficiently when conducting the experiment. Balanced equations and given terms should be included in this section for any chemical reactions carried out in the experiment.

During Laboratory Period

4. *Data & Observations* - As you conduct the experiment use this section to record a rough transcript of the experimental procedures, along with all data, observations, and the identifying number of any unknown. Whenever possible, data should be presented in tabular (tables) form. In all cases, data must be clearly labeled, *including units*. The information provided in this section of the notebook must contain sufficient detail so that someone could follow your notes to repeat the experiment.

During and Post Laboratory Period

5. *Calculations* - In this section, the final results are calculated from the experimental data. For experiments involving quantitative analyses, results for each individual determination as well as a mean and a relative standard deviation are computed. For each type of calculation, a sample calculation, using your measured data, should be given *in detail* including proper significant figures and units for all numerical quantities.
6. *Data & Results Summary* - Prepare the final page of the entry to attach your graded Data and Results Summary sheet, which will be handed out in lab. These sheets summarize selected data, sample calculations, and all experimental results. After grading, summary sheets are to be taped into your lab notebook in such a way that the edges of the page do not stick out of the notebook. Be sure to prepare a page between experiments for this purpose. Also, write a brief (2-3 sentences) summary of what was learned from the experiment.

On the following pages is a sample lab notebook write-up for the Chemistry 121 Analysis of Vinegar experiment. Please use this example as an aid in setting up and using your lab notebook for this course.

Sample Experiment Write-Up in Chem 123 Lab Notebook:

Analysis of Vinegar 9/12/2008

Purpose: To determine the molarity and weight percent of acetic acid in vinegar by titrating with standardized sodium hydroxide solution.

Overview of Procedures:

1. Obtain vinegar sample; cover with watchglass to prevent evaporation.
2. Rinse 25-mL pipet with water, then vinegar; pipet 25.00 mL vinegar into flask.
3. Add 40 mL DI H_2O and 3-4 drops phenolphthalein to vinegar sample.
4. Obtain standard NaOH solution; cover with watchglass.
5. Set-up buret:
   Rinse buret with water and check for leaks.
   Rinse buret with NaOH solution, clamp into ring stand, and fill with NaOH.
6. Titrate vinegar sample to faint pink endpoint.
7. Repeat titration with two additional samples of vinegar.
8. Check ppt difference between 3 titration volumes. If greater than 5 ppt, do another titration.
9. Calculate molarity and weight percent acetic acid for each trial.
10. Calculate the mean, standard deviation and relative standard deviation (ppt) for molarity and weight percent.

Relevant Equations and Terms:

$HC_2H_3O_2 + NaOH \rightarrow Na\,C_2H_3O_2 + H_2O$

Density of vinegar = 1.03 g/mL

Formula weight of acetic acid, FW = 60.05 g/mol

Data & Observations:

Got ~125 mL Fred Meyer brand vinegar in large beaker; covered beaker with watchglass.

Cleaned and rinsed 25-mL pipet 3 times with water and 3 times with vinegar.

Pipeted 25.00 mL vinegar into 250-mL Erlenmeyer flask.

Added 40 mL DI H_2O and 3 drops phenolphthalein indicator to vinegar in flask. Swirled to mix.

Got ~150 mL 0.5209 M sodium hydroxide standard solution.

Rinsed buret with DI H_2O and NaOH solution. Clamped buret to ringstand and filled with NaOH. Drained through tip into waste beaker. Positioned vinegar flask under the tip of the buret with white paper under flask.

Titration Data Table:

	1	2	3	4
Final Buret Reading (mL)	38.46	37.92	37.81	(not needed)
Initial Buret Reading (mL)	0.58	0.13	0.00	
Volume NaOH added (mL)	37.88	37.79	37.81	

Titrated with NaOH to faint pink endpoint. Added a fraction of a drop at a time near endpoint by quickly turning the stopcock 180°, swirling after each addition.

Repeated titration with two more vinegar samples, refilling buret between titrations.

Calculations:

ppt difference between volume NaOH from 3 trials:

average volume, V_{ave} = $\frac{37.88mL + 37.79mL + 37.81mL}{3} = 37.83mL$

ppt difference = $\frac{range}{V_{ave}} * 1000 = \frac{37.88mL - 37.79mL}{37.83mL} * 1000 = 2.379\,ppt$

Molarity of Acetic Acid:

$$Molarity = V_{NaOH} * \frac{1L}{1000mL} * M_{NaOH} * \frac{1\,mole\,HC_xH_3O_2}{1\,mole\,NaOH} * \frac{1}{0.02500\,L\,vinegar}$$

Trial 1:

M_1 = $37.88mL * \frac{1L}{1000mL} * 0.5209\frac{moles\,NaOH}{L} * \frac{1\,mole\,HC_xH_3O_2}{1\,mole\,NaOH} * \frac{1}{0.02500L}$ = 0.789268 M

Trial 2: M_2 = 0.787392 M

Trial 3: M_3 = 0.787809 M

Mean Molarity, $\bar{x} = \frac{0.789268M + 0.787392M + 0.787809M}{3}$ = 0.788156 M

Standard Deviation, s = $9.851*10^{-4}$ M (used calculator)

Rel. Standard Deviation, rsd = $\frac{s}{\bar{x}} * 10^3 = \frac{9.851*10^3 M}{0.788156M} * 10^3$ = 1.250 ppt

Weight Percent of Acetic Acid:

$$Weight\ percent = \frac{mass\ acetic\ acid\ (g)}{mass\ vinegar\ (g)} * 100 = \frac{moles\ acetic\ acid * FW_{acetic\ acid}}{25.00mL * vinegar\ density} * 100$$

Mass Vinegar = 25.00 mL x 1.03 g/mL = 25.75 g

Trial 1:

Mass $HC_2H_3O_2$ =

$$0.03788mL * 0.5209\frac{moles\ NaOH}{L} * \frac{1\ mole\ HC_2H_3O_2}{1\ mole\ NaOH} * \frac{60.05g}{mol\ HC_2H_3O_2} = 1.18489\ g$$

Weight % = $\frac{1.18489g}{25.75g} * 100$ = 4.6015%

Trial 2:

Mass $HC_2H_3O_2$ = 1.18207 g

Weight % = 4.5906%

Trial 3:

Mass $HC_2H_3O_2$ = 1.18270 g

Weight % = 4.5930%

Mean Weight % = $\bar{x} = \frac{4.6015\% + 4.5906\% + 4.5930\%}{3}$ = 4.5950%

Standard Deviation, s = $5.727 * 10^{-3}$% (used calculator)

Rel. Standard Deviation, rsd = 1.246 ppt

Data & Results Summary:

Leave a blank page after calculations to attach Data & Summary sheet after it has been graded and returned. Also, write a brief (2-3 sentences) summary of what was learned from the experiment. For example: "Using volumetric analysis (i.e., titrations with a standardized NaOH solution) we determined that Fred Meyer Brand vinegar has a molar concentration of acetic acid of 0.7882 M and is 4.60% acetic acid by weight. This compares favorably to the stated concentration (5%) found on the bottle. Our standard deviation for the titrations was $5.7 * 10^{-3}$M and the relative standard deviation (RSD) was 1.2 ppt, thus according to the grading scale we had a high degree of precision with our measurements (zero points taken off)."

LABORATORY GRADING

In general, the Chem 123 lab is worth 100 points, where the experiments are worth 85 points and the lab notebook is worth 15 points. The lecture instructor will determine the weight of the lab grade toward your final course grade.

The maximum points for each experiment are listed below:

1. 10 pts - The Nickel Exercise & The Volumetric Pipet Exercise
2. 20 pts - Analysis of a Nickel Complex
3. 20 pts - Volumetric Analysis: The Determination of a Weak Acid
4. 15 pts - Titration Curves and the Dissociation Constant of Acetic Acid
5. 20 pts - Wizards of the Winery

Experiments are graded based on results submitted on Data and Results Summary Sheets, Great care should be taken to prepare your Summary Sheets neatly and accurately. Results should not be entered until all calculations have been carried out completely in your lab notebook. As with the lab notebook, use pen to cross out errors with a single line and enter corrections next to or above the line. Never write over an entry; this forces your lab instructor to guess at your answer and could result in lost points.

Your lab instructor will consider a variety of factors in grading your Summary Sheets. In all experiments, the quality and completeness of the Summary Sheet as well as the results will be considered in grading. Points may be lost due to significant figure errors, missing or incorrect units, calculation errors, missing data, and poor precision and/or accuracy. Instructions regarding significant figures and rounding are given on the next page.

The standards used in grading precision and accuracy may vary between experiments. For example, the Volumetric Analysis of a Weak Acid experiment involves quantitative analysis of an unknown sample in which the precision and accuracy of the results are expected to be very high. This experiment will be graded primarily on the accuracy of your mean result and the precision of your set of individual results. The grading scale for that experiment as points deducted from the maximum of 20 points is given below. Although the other experiments involve quantitative measurements and calculated results, the accuracy and precision are not expected to be as great, and the grading scale will be adapted accordingly.

GRADING SCALE FOR CHEM 123

ACCURACY		PRECISION	
relative error (ppt)		relative standard deviation (ppt)	
0-2.9	-0 pt	0-2.4	-0 pt
3.0-5.9	-1 pt	2.5-3.9	-1 pt
6.0-8.9	-2 pt	4.0-6.9	-2 pt
9.0-11.9	-3 pt	7.0-9.9	-3 pt
12.0-14.9	-4 pt	over 10	-4 pt
over 15	-5 pt		

SIGNIFICANT FIGURES

Adding & Subtracting:

When adding or subtracting measured quantities, give the same number of decimal places in the answer as there are in the measurement with the *least* number of decimal places. Example:

$$\begin{array}{r} 0.001537 \\ -\ 0.001089 \\ \hline 0.000448 \end{array}$$

Even though the original measurement has 4 significant figures, the resulting calculation only warrants 3 significant figures due to the rules of adding and subtracting.

Multiplying & Dividing:

When multiplying or dividing measured quantities, give as many significant figures in the answer as there are in the measurement with the *least* number of significant figures.

Rounding:

If the first digit after your last significant figure is 5 or greater, round up.
If the first digit after your last significant figure is less than 5, round down.

Standard Deviation:

As a general rule, always report standard deviation, relative standard deviation, and relative error with two significant figures. Note that the units for standard deviation will be the same as the values for which you are calculating the standard deviation. For example, the standard deviation of three volume measurements made in milliliters, will have units of milliliters.

Things to Remember:

25-mL volumetric pipet is good to **25.00** mL
500-mL volumetric flask is good to **500.0** mL
50-mL buret should be read to **0.01** mL with every volume reading
Analytical balance: reads 4 decimal places, 0.0000 g (all are significant)
Top-loading balance: reads 2 decimal places, 0.00 g (all are significant)

When carrying out calculations, you should not round until you reach the final result. Keep track of significant figures by underlining the last significant digit in intermediate calculations and including at least one additional digit; i.e., 0.0013956 mol. Report the final result to the correct number of significant figures on your Summary Sheet and in your lab notebook.

CHEMISTRY LABORATORY RULES AND REGULATIONS

The rules and regulations outlined below are intended to provide maximum safety and efficiency in the laboratory for all students. For these reasons it is absolutely imperative that they be studied and observed.

SAFETY GOGGLES MUST BE WORN AT ALL TIMES

DO use only those chemicals that are on the shelf for your class or are specifically provided for your use.

ask your lab assistant if you cannot locate necessary chemicals or supplies.

take only the amount of chemicals needed. Waste is expensive and use of more chemicals than called for may be dangerous.

notify your lab instructor *immediately* of any spills. Any spillage on bench tops, floor, and especially on or around the balances must be cleaned up immediately.

clean all equipment for use. Glassware should be washed with soap and rinsed well with tap water, followed by deionized water.

return glassware and equipment obtained for specific experiments in the same condition you received it - clean and dry.

clean bench area and sinks at the end of each lab period and lock your drawer. You are responsible for the contents of your drawer.

know where the eyewash fountains, ceiling showers, and fire extinguishers are in the laboratory.

IMMEDIATELY REPORT *ANY* ACCIDENTS, REGARDLESS OF HOW MINOR, TO YOUR INSTRUCTOR.

DO NOT undertake any unauthorized experiments.

put anything into the reagent stock bottles, such as medicine droppers or stirring rods. Your instructor will demonstrate the proper way to dispense stock solids and liquids.

pour any reagent back into a stock bottle.

throw matches, litmus paper, or other solids into the sinks.

take reagent stock bottles to your desk from the side shelf or put equipment from the community lockers into your drawer.

weigh directly on the balance pans – use a piece of weighing paper, beaker or other container.

eat, drink, chew tobacco or smoke in the laboratory.

wear open-toed shoes, sandals, skirts, shorts, tank tops, or midriff-baring shirts. Protect yourself against chemical spills and other laboratory hazards.

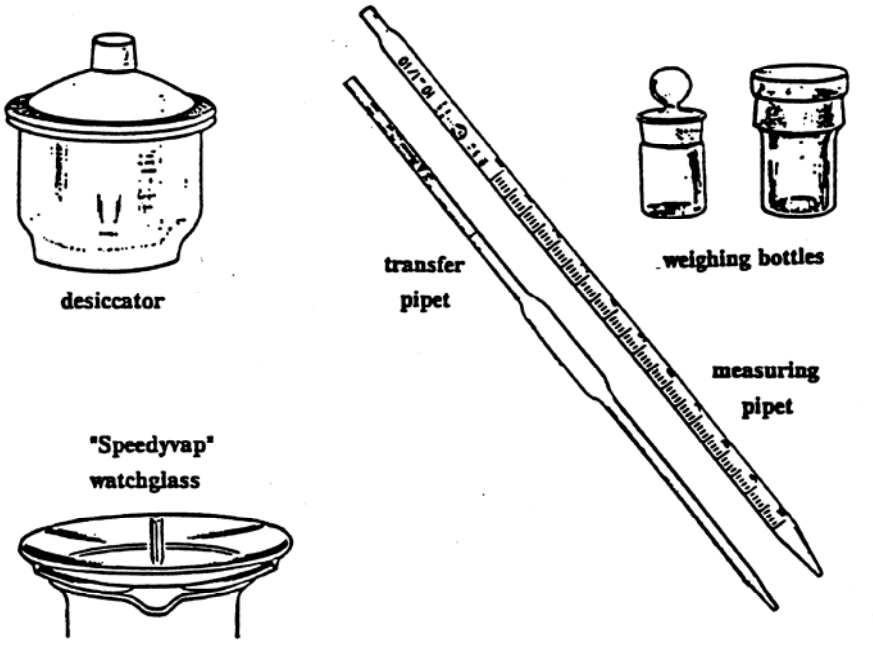
use cell phones in lab; phones should be turned off or silenced in lab.

CHEMISTRY 123 LAB EQUIPMENT

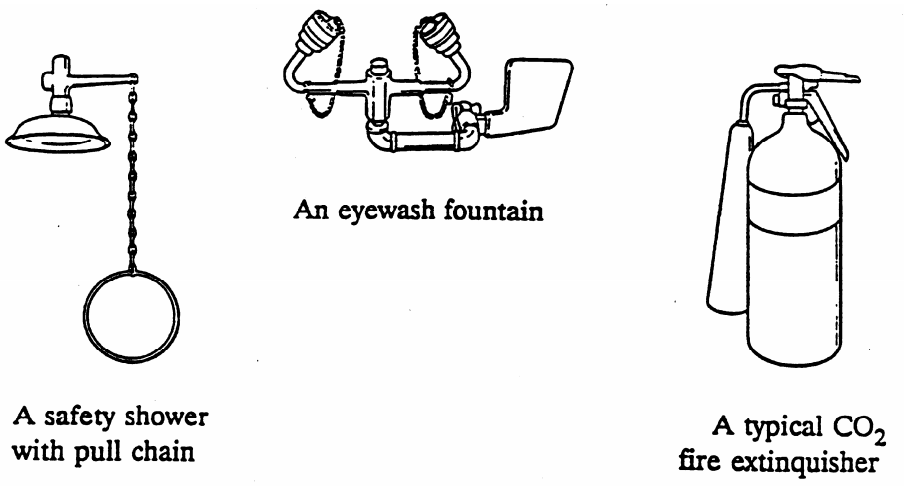
A few of the items in your lab drawer are shown below. After lab check-in, you will be responsible for the equipment in your drawer, so be sure to return all items to your drawer and lock it at the end of each lab period.

Hot plates, ring stands, pipette pumps and buret clamps are community equipment and are in cabinets. When you use these items, be sure to return them to the cabinets; do not put them in your drawer.

If you do not have safety goggles from a previous chemistry course, these must be purchased. Safety goggles must be worn at all times in the lab.



During the first lab period, your instructor will show you the safety equipment located throughout the lab. Some of these pieces of equipment are shown below. Make sure you are aware of where these items are located in the lab.



PERIODIC TABLE OF THE ELEMENTS

1 H 1.0079																	2 He 4.00260
3 Li 6.941	4 Be 9.01218											5 B 10.81	6 C 12.011	7 N 14.0067	8 O 15.9994	9 F 18.99840	10 Ne 20.179
11 Na 22.98977	12 Mg 24.305	Transition elements										13 Al 26.98154	14 Si 28.086	15 P 30.97376	16 S 32.06	17 Cl 35.453	18 Ar 39.948
19 K 39.098	20 Ca 40.08	21 Sc 44.9559	22 Ti 47.90	23 V 50.9414	24 Cr 51.996	25 Mn 54.9380	26 Fe 55.847	27 Co 58.9332	28 Ni 58.70	29 Cu 63.546	30 Zn 65.38	31 Ga 69.72	32 Ge 72.59	33 As 74.9216	34 Se 78.96	35 Br 79.904	36 Kr 83.80
37 Rb 85.4678	38 Sr 87.62	39 Y 88.9059	40 Zr 91.22	41 Nb 92.9064	42 Mo 95.94	43 Tc (97)	44 Ru 101.07	45 Rh 102.9055	46 Pd 106.4	47 Ag 107.868	48 Cd 112.40	49 In 114.82	50 Sn 118.69	51 Sb 121.75	52 Te 127.60	53 I 126.9045	54 Xe 131.30
55 Cs 132.9054	56 Ba 137.34	57 La 138.9055	72 Hf 178.49	73 Ta 180.9479	74 W 183.85	75 Re 186.207	76 Os 190.2	77 Ir 192.22	78 Pt 195.09	79 Au 196.9665	80 Hg 200.59	81 Tl 204.37	82 Pb 207.2	83 Bi 208.9804	84 Po (209)	85 At (210)	86 Rn (222)
87 Fr (223)	88 Ra 226.0254	89 Ac (227)	104 (Rf)	105 (Ha)	106												

Lanthanides	58 Ce 140.12	59 Pr 140.9077	60 Nd 144.24	61 Pm (145)	62 Sm 150.4	63 Eu 151.96	64 Gd 157.25	65 Tb 158.9254	66 Dy 162.50	67 Ho 164.9304	68 Er 167.26	69 Tm 168.9342	70 Yb 173.04	71 Lu 174.97
Actinides	90 Th 232.0381	91 Pa 231.0359	92 U 238.029	93 Np 237.0482	94 Pu (244)	95 Am (243)	96 Cm (247)	97 Bk (247)	98 Cf (251)	99 Es (254)	100 Fm (257)	101 Md (258)	102 No (255)	103 Lw (260)