Chemistry 1110: Modern Chemistry I
Laboratory Manual

Fall 2014 Edition

Instructor: R.C. Mawhinney
Lab Instructor: Brad Miller
Department of Chemistry
Faculty of Science and Environmental Studies

DEPARTMENT OF
Chemistry

CHEMISTRY 1110 FA

LABORATORY MANUAL

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R.C. Mawhinney
M. Campbell, S.D. Kinrade
C.D. MacKinnon
G.D. Martin, B. Miller
G.J. Spivak
Department of Chemistry
Lakehead University
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LABORATORY SAFETY

The practice of safety in the chemical laboratory results from an attitude of mind, and therefore requires the desire on the part of the individual to protect him/herself and associates. All accidents are caused and therefore can be prevented. This can be accomplished if laboratory safety is made an integral part of every activity. It is the object of the lab course to do just that. It is acknowledged that beginning chemistry students are largely unfamiliar with the potentials for injury that surround them, so your instructor will make every effort to point out and explain them to you. **It is your responsibility as a student to prepare carefully for each experiment, and to understand and obey the safety rules listed below.**

SENDING/RECEIVING PHONE CALLS, TEXT MESSAGES AND EMAILS IS NOT PERMITTED IN THE LABORATORY. ANY STUDENT DOING SO WILL BE ASKED TO LEAVE THE LABORATORY AND WILL RECEIVE A MARK OF ZERO FOR THAT EXPERIMENT

PLEASE NOTE THAT BOTH PROPER EYE PROTECTION AND LABORATORY COATS ARE MANDATORY IN THE LAB.

STUDENTS NOT WORKING IN A SAFE MANNER WILL BE ASKED TO LEAVE AND WILL RECEIVE A MARK OF ZERO FOR THAT EXPERIMENT.

PRIOR TO ENTERING THE LAB, ALL STUDENTS MUST ADORN LAB SAFETY EQUIPMENT

1. **Adequate eye protection must be worn at all times** (*i.e.*, safety glasses). There is no exception to this rule. Prescription eye glasses are not sufficient; eye protection must be worn over prescription glasses. Safety glasses may be purchased at Chemistry Stores, CB2039, the university bookstore, or off-campus sites.
2. Proper clothing must be worn in the lab. Open-toed shoes, and any type of shoe that exposes the top of the foot (i.e. flats) **must not** be worn. Long hair must be **tied back**. Shorts and skirts are not to be worn to a laboratory period. Those doing so will not be permitted to complete the lab. **A lab coat must be worn**. Lab coats must be full length (to the knee) and buttoned up. Lab coats may be purchased through the Lakehead University Alumni Bookstore or from off-campus sources.
3. Eating, drinking, and smoking are not permitted in the lab.
4. Familiarize yourself with the positions and operation of the fire extinguishers, emergency shower and eye washes, and the location of the safety information poster and nearest exits.
5. All pipetting must be done with an appropriate device and **NOT BY MOUTH**.
6. Report **ALL INJURIES OR ACCIDENTS**, no matter how minor, to the instructor. Injuries may include, but are not limited to: cuts; burns; contact with corrosive liquids; inhalation of fumes; etc.

7. The eyewash areas (near the sinks) are to be kept clear of obstruction at all times.

8. Flammable solvents should not be flushed down the drains, with the exception of small volumes of certain solvents used in washing/drying small items of equipment.

9. Although you will not need them for CHEM1110 labs, gloves are available for purchase from Chemistry Stores (CB2039). If you should come in contact with any chemicals, solvents, etc., **WASH THE EXPOSED AREA IMMEDIATELY.** Regardless of whether or not you are wearing gloves, always wash your hands when leaving.

10. Material Safety Data Sheets (MSDS) for the chemicals used in Chem 1110 are available in the binders located near the fume hoods.

11. In case of **EMERGENCY**, call **SECURITY** at **ext. 8911**.

INTRODUCTION TO EXPERIMENTS

1. In order to obtain maximum benefit from your laboratory work, **be prepared before coming to the lab**. This preparation includes careful reading and studying of the experiment itself and the background theory, as well as completion of the pre-lab assignment.

2. Each pair of students will be responsible for a locker. The course will be considered complete only after the locker has been checked out at the end of term.

3. Read all labels before using any chemicals!

4. Broken glass and sharp objects must be discarded in the **BROKEN GLASS** bin. There is a separate sharps container for needles (which you won't use in CHEM 1110)

5. Try not to take more chemical than is required. In the event excess is taken, **DO NOT RETURN IT TO THE REAGENT CONTAINER**.

6. Closely follow the instructions given with each experiment regarding the handling of waste. **DO NOT DISPOSE OF ANY CHEMICALS AS INSTRUCTED.** Ask the lab technician or T.A. for chemical disposal instructions if you are uncertain of what to do!

7. When water is required in an experiment, always use distilled water that can be obtained from the stainless steel faucets at the sinks near the fume hoods.

8. Never put anything hot or wet on the pan of a balance.

9. The balances must be left clean and tidy. Clean up spills immediately. Clean up spills immediately – if uncertain on how, consult an instructor/demonstrator. Report any spills encountered, no matter how small.

10. Volumetric equipment, which is glassware designed to contain or deliver an accurate volume at a specific temperature, should never be heated or used for hot solutions. See **Appendix A** for an explanation of the care and use of volumetric equipment.

11. The laboratory must be left as it was found – clean and tidy. Students will be expected to clean up all spills and other messes as soon as they occur. Students must return all chemicals and equipment to their proper places before leaving the lab. Each student's tidiness will be assessed.

12. **DO NOT COME LATE**. You will miss the pre-lab discussion. Students who miss this discussion may not (at the instructor's discretion) be allowed to perform the experiment.

13. If you are unable to attend your scheduled lab section **and have a valid reason** (*e.g.* medical/family emergency) you **must** contact Mr. Brad Miller as soon as possible to make alternate arrangements.

LABORATORY CARDS†

All students enrolled in courses in the Department of Chemistry with laboratory components must obtain a **LABORATORY CARD** in order to cover the cost of broken equipment (*e.g.*, glassware). Students can obtain a laboratory card from Chemistry Stores (CB 2039) for a $20.00 deposit (Cash only); **THE CARD MUST BE OBTAINED BEFORE BEGINNING THE LAB**, and is valid for ALL chemistry courses that offer labs (*i.e.*, not just CHEM1110). When a piece of equipment is broken, a set amount is deducted from the value of the card (see below for a short list of items). Upon returning your laboratory card to Chemistry Stores, your $20.00 deposit will be refunded, **minus any deductions due to breakages**. If the full value of the card is consumed through equipment breakage, students must obtain a new card prior to proceeding with any further laboratory sessions. Please note that lost/stolen cards will **NOT BE REFUNDED**; in the event a card is lost and the student still has chemistry labs to complete, a new card must be obtained.

EQUIPMENT BREAKAGE PRICE LIST*

Size	Item	Cost ($)
150, 250, or 400 mL	beaker	2
600	beaker	3
800	beaker	4
50	burette	20
10	graduated cylinder	3
100	graduated cylinder	5
125	Erlenmeyer flask	3
250	Erlenmeyer flask	4
125	filter flask	15
100	volumetric flask	10
200	volumetric flask	16
10	volumetric pipette	14
20	volumetric pipette	15
25	volumetric pipette	16
15 × 125 or 20 × 150 mm	test tubes	1
90 mm	watch glass	3
-1° to 51°C	thermometer	12
5.5 cm	Buchner funnel, porcelain	20
75 mm	filter funnel, short stem	6

† The laboratory card can only be obtained by submitting a signed plagiarism certificate (see page vii for more details) along with your $20.00 cash deposit to the technician in Chemistry Stores.

* See the Chemistry Stores technician for a more complete list of equipment and corresponding breakage charges. Equipment breakage charges subject to change.

LABORATORY REPORTS

There are five experiments to perform in CHEM 1110. Experiment 4 must be written as a formal report (see 11, below). The remaining experiments are either hand-in sheets or informal reports, so you have some flexibility in format, noting the following points:

1. All results and observations must be recorded in a **LAB BOOK WITH BOUND PAPER**. Hard-back lab books or spiral-bound scribbler-style books are both acceptable. Lab reports often require sample calculations, but a full record of all calculations, even repetitive ones, should be kept in your lab book.
2. **PRE-LAB ASSIGNMENTS MUST BE HANDED IN UPON ENTERING THE LAB.** You may keep a copy for yourself, it will not be returned before your lab report is due.
3. Before leaving the lab, you must have your results sheet signed by the instructor or teaching assistant. **THIS SHEET MUST BE HANDED IN WITH YOUR LAB REPORT**; otherwise your report will not be graded.
4. In cases where students work in pairs on an experiment, **EACH** student must submit a separate results sheet and an **INDEPENDENTLY WRITTEN** lab report.
5. Lab reports must be stapled together, bound in a "duo-tang" cover, or submitted in a lab notebook (*i.e.*, not loose sheets of paper). The **title page** must contain the following:
    - title of the experiment
    - student name and number, and the lab section (F1, F2, etc.)
    - if working in pairs, your partner's name
    - date when the experiment was carried out
    - number of pages in the report
6. You will submit one of three types of lab reports, as indicated in the individual experiments:
    - A Hand-in sheet style report
    - An informal report (see point 10)
    - A formal report (see point 11)
7. Each report must begin with an **ABSTRACT** (see 11.II, below). Keep all answers and calculations well-organized. Calculations must be thorough, indicating the major steps.
8. Reports are due **BY 4:00 PM OF THE DAY ONE WEEK AFTER THE EXPERIMENT WAS COMPLETED.** Deposit in the appropriate slot in the cabinet outside of CB-2044. Late reports up to 24 hours after the original deadline will receive a -10% penalty; later reports receive a mark of 0. Students unable to hand in a report on time due to medical reasons must notify the instructor **IN PERSON** (not by phone or email) as soon as possible.
9. Plagiarized pre-labs or lab reports will be sent to the Dean of Science & Environmental Studies for review and penalty assessment under the Code of Student Behaviour. See point 12 for further information. Lab instructors and T.A.s have full authority to make judgements in all cases of academic misconduct in the lab.
10. The *informal* lab reports should follow this general format:
    - a title page (see 5, above) and an abstract (see 10(ii), below)
    - a section containing all of the results obtained and/or observations made (see 11.V, below), along with all necessary calculations

- a very brief discussion of your findings and any conclusions made
- use any questions that may accompany the experiment as points for leading your discussion
- the raw (signed) data sheet
- type-written informal reports are preferred, but not strictly necessary

11. The *formal* lab report is a **rationally organized, type-written** account of your findings and the conclusions that can be drawn from them. The following format is recommended:

I. **TITLE PAGE** (see 5, above)

II. **ABSTRACT**: a concise summary (about 2 or 3 sentences) of the experiment and significant results (*e.g.*,. important numbers, values, colours, etc.) and conclusions.

III. **INTRODUCTION**: a **brief** outline of the relevant theory associated with the experiment (including references). Do not copy theory directly from any source material.

IV. **EXPERIMENTAL METHOD**: only include modifications to (or clarification of) the procedure given in the lab manual which, in any case, must be referenced. This should be written in the past tense and use the passive voice.

V. **RESULTS**: all experimental observations should be included. Data must be clearly tabulated and include the appropriate units. Detailed sample calculations (which would clutter the main body of the report) should be included in an appendix and referenced appropriately (see 11.VII, below). Figures such as graphs must be neatly labeled and include a legend. Data points on graphs should be highlighted (*e.g.*, with open circles, squares, etc.).

VI. **DISCUSSION**: this section should include a clear presentation of the findings along with a critical evaluation (*i.e.*, compare your results with theoretical expectations or with values reported in the literature). In addition, you might suggest improvements to the procedure or note practical applications of the phenomenon under study. Use any questions that may accompany the experiment as points for leading your discussion.

VII. **REFERENCES**: in chemical literature, references are listed numerically in the order they have been cited. Any accepted format for presenting references may be employed, but be consistent. Citations within the text are made using superscripted or bracketed numbers (*e.g.*, "...Smith and Wesson[4] have reported that..."; "...Ben and Jerry[24] have shown..."). If the citation occurs at the end of a paragraph or next to punctuation, make sure it comes after the punctuation. We encourage students to include the full references on a separate page at the end of the report, rather than as footnotes. Acceptable formats would be:

For a text, *e.g.*: Zumdahl, S. and Zumdahl, S. Chemistry (9^{th} edition). New York Houghton Mifflin, 2010, pp. 175-177.

For a journal article, *e.g.*: Doe, J. *Journal of Chemistry* **2005**, *34*, 123-456.

(*i.e.*, author(s), then the journal name in italics, followed by the year in bold, followed by the volume in italics, followed by the page numbers).

Wikipedia **(and similar web sites) is NOT a reliable reference source and should NEVER be used as a primary source.** (they can be used to find primary information, however)

VIII. **APPENDICES**: include, for example, sample calculations and the raw (signed) data sheet.

For a more complete guide to undergraduate science writing, see:

https://ugr.ue.ucsc.edu/sites/default/files/jyi_guide_to_scientific_writing.pdf

In particular, see part II, which gives advice on writing an abstract, introduction, discussion, and the presentation of Tables, Figures and Equations, especially on how to correctly prepare a caption.

12. Plagiarism is a serious academic offense. It may be understandable that, in the context of writing and referencing works for a lab report, inadvertent plagiarism may occur; however, ignorance is not a valid defence! In order to prevent misunderstandings about what constitutes plagiarism, the Department of Chemistry has prepared a document that clearly defines plagiarism, giving several examples. Students will have to sign a Plagiarism Certification that verifies that they have read this material before they are allowed to start any experiments. See: http://www.chemistry.lakeheadu.ca

EXPERIMENT #1

ANALYTICAL TECHNIQUES: ACCURACY AND PRECISION VIA THE STANDARDIZATION OF A HYDROCHLORIC ACID SOLUTION

Objectives

A. To become familiar with the proper techniques for using the analytic balance, burette, pipette and graduated cylinder.

B. To be able to distinguish between accuracy and precision in experimental results, and to determine the propagation of experimental uncertainty in calculations.

Introduction *(Zumdahl and Zumdahl, pp. 8-27 and pp. A10-A13)*

Uncertainty and Precision* vs. *Accuracy

You will encounter two types of numbers in scientific research. **Exact numbers** are countable or have specifically defined values, *e.g.*, this carton has 8 eggs, or there are exactly 1000 mg in a gram. These numbers will always be the same, regardless of the conditions. In contrast, numbers obtained through or derived from experimentation are sensitive towards the limitations in the equipment or instrumentation used (as well as the individual who collected the data!). In other words, *experimentally measured or derived quantities always have some degree of uncertainty*; they are **inexact numbers.** Thus, quantities measured using any measuring device must be reported along with its uncertainty. Generally, the last reported digit in the measured number has some degree of uncertainty associated with it. For example, the analytical balances used in this lab can be read to 0.0001 g, so all masses must be reported as, *e.g.*, 1.2345 ± 0.0001 g. Similarly, 50 mL burettes can be read to 0.02 mL, so all burette readings must be reported as, *e.g.*, 12.34 ± 0.02 mL. In doing so, you are indicating the uncertainty of the last digit in the measured quantity. The terms "precision" and "accuracy" are often used: **precision** is a measure of how closely individual measurements agree with one another, while **accuracy** refers to the degree of agreement between measured quantities and a "true" or "accepted" quantity (we'll discuss this more later).

Significant Figures

A calculation using measured quantities is governed by the least accurate measurement used in the calculation (that is, it is the limiting measurement). For this reason, it is important to know the number of **significant figures** or digits of a measured quantity (an inexact number). Note: exact numbers are regarded as having an infinite number of significant figures (and so can be ignored when determining the number of significant figures in a calculation). In general, *the greater the number of significant figures, the greater is the certainty implied for the measurement.* Zeros are special digits because they may or may not be significant depending on how they appear in the number. More specifically, *zeros may be used as part of the measured quantity, or they may be used to locate the position of a decimal point.* In the former instance, the zero is significant, while in the latter, not significant. Consider the following rules:

1. Zeros between nonzero digits are significant, *e.g.*, 401 g and 4.01 g (both have 3 significant figures)

2. Zeros at the beginning of a number are never significant, and just provide the position of the decimal point, *e.g.*, 0.0012 g (2 significant figures) and 0.0134 mL (3 significant figures)

3. Trailing zeros are always significant for digits after the decimal point in a number, *e.g.*, 0.1200 g (4 significant figures; the two trailing zeros are significant)

4. Zeros that fall at the end of a number and after a decimal point are always significant, *e.g.*, 7.00 g (3 significant figures)

5. Zeros that fall after a nonzero digit and before the decimal point, may or may not be significant. In general, they are not unless specified as such, *e.g.*, 310 mL has 2 significant figures) but 310. mL (note the decimal!) would have 3 significant figures

In order to eliminate all confusion, it is best to write the number using base 10 exponential notation (also referred to as scientific notation). *All digits (including zeros) are significant for a number written in exponential notation*, *e.g.*, 5.01×10^4 mL (3 significant figures) *vs.* 5.0100×10^4 mL (5 significant figures).

When performing calculations, attention must always be paid towards significant figures. In working problems on a calculator, *perform the calculation using all of the digits allowed by the calculator and round off at the end of the problem*. The calculation rules for simple operations are:

1. When adding/subtracting, the number of decimal places in the answer must equal the number of decimal places in the number with the *fewest* places, *e.g.*, 0.21 + 4.9 + 11.1124 = 16.2224 on a calculator, but should be reported as 16.2 (4.9 has only one decimal place)

2. When multiplying/dividing, the number of significant figures in the answer should be the same as that in the number with the *fewest* significant figures, *e.g.*, $15.50 \times 27.3 \times 5.4 =$ 2285.01 on a calculator, but should be reported as 2300 (2.3×10^3) (5.4 has only 2 significant figures)

Keep in mind that the line of numbers which appears on your calculator's display after hitting the "=" button is a correct but un-rounded arithmetical calculation. For example, for the calculation:

$$0.1003\ g\ Na_2C_2O_4 \times \left(\frac{1\ mol\ Na_2C_2O_4}{134.00\ g\ Na_2C_2O_4}\right) = 7.708955224 \times 10^{-4}\ mol$$

The answer contains far more figures than are justified. The answer should be given to 4 significant figures, specifically 7.709×10^{-4} mol.

Propagation of Experimental Uncertainty

When experimental measurements are used in calculations, there is a propagation of experimental uncertainty that must be determined such that the final result of the calculation also includes the overall experimental uncertainty. *Except for standard deviation calculations* (see below), all experimental values and calculations based on them must include uncertainty limits. Sample calculations for the propagation of experimental uncertainty are given in Appendix B.

The **accuracy** of a measurement refers to the agreement between the measured value and the true value. To determine accuracy, we calculate the difference between the true value (assuming it is known) and the average value from a series of measurements. The **average** (arithmetic mean) is given by:

$$Average = \bar{x} = \frac{\sum_{0}^{n} x_i}{n} \qquad (1)$$

where Σ is the operation of summation (*i.e.*, the sum of the individual measurements), $\bar{x}$ is the average value, x_i is the individual measurements, and n is the number of measurements. The accuracy would then be defined as:

$$\text{Accuracy} = \bar{x} - \text{true value} \qquad (2)$$

An accurate result is one that agrees closely with the true value (the smaller the difference, the greater the accuracy). This is sometimes called the mean error, and is reported in the same units as the measurement, *e.g.* if the true value is 2.62 g and $\bar{x}$ = 2.52 g, the accuracy would be -0.10 g. Many times errors are expressed in terms of a percentage. The percent error (or "relative error") is the absolute value of the difference of the true value and the experimental (measured or mean) value, divided by the true value, and multiplied by 100:

$$\text{Percent error} = \frac{|\text{true value - experimental value}|}{\text{true value}} \times 100 \qquad (3)$$

Precision refers to the agreement between several measurements of the same quantity, *i.e.*, the reproducibility of a given type of measurement, and is usually described in terms of the **standard deviation**, *s*, which is defined as follows:

$$s = \sqrt{\frac{\sum_{i=0}^{n}(x_i - \bar{x})^2}{(n-1)}} \qquad (4)$$

where $(x_i - \bar{x})$ is the deviation of each measurement from the average. (Actually this formula only gives an estimate of the standard deviation; an exact one would require an infinite number of measurements. Therefore, our confidence in our mean is low if we only take a few measurements.)

As an example of this type of calculation, consider the following problem. In a titration experiment using a burette, you obtain the following results: 36.78 mL, 36.80 mL and 36.75 mL. You'd like to know how well these volumes agree with one another, so you proceed to calculate the standard deviation. First, calculate the mean volume:

$$= (36.78 + 36.80 + 36.75) \div 3$$
$$= 36.78 \text{ mL}$$

Now:

x_i (mL)	$(x_i - \bar{x})$ (mL)	$(x_i - \bar{x})^2$ $(mL)^2$
36.78	0.00	0.0000
36.80	0.02	0.0004
36.75	0.03	0.0009
		S = 0.0013

So:

$$s = \sqrt{\frac{0.0013}{(3\text{-}1)}} = 0.03$$

The mean volume would have a standard deviation of 0.03 mL, so we would write the answer as 36.78 mL ± 0.03 mL. The standard deviation calculation is an estimate of the random uncertainty in any given measurement. Note that this is **not** the same as the experimental uncertainty explained above (that is, uncertainties due to inherent limitations of the instrument or user, often referred to as systematic uncertainty). Generally speaking, for a large number of measurements, the probability that a given measurement will fall within ± *s* of the average value is 68% and there is a 99.7% probability that it will fall within ± 3*s*.

Titration

Titration is a technique used to determine the concentration of an unknown solution by reacting it completely with a solution of known concentration. Since the measurements consist of finding the volumes of solutions containing the same number of moles, it is commonly known as volumetric analysis. In this technique, a known volume of one solution (A), with a known concentration, is reacted with a known volume of another solution (B) of unknown concentration. If the reaction stoichiometry between A and B is known, and there is some means of establishing when the reaction is complete (normally by means of an indicator – a substance that changes colour when enough of A has been added to react with all of B; in an acid-base reaction this occurs at a specific pH), the concentration of solution B can be determined.

The most common unit of concentration is molarity (M), which is defined as the number of moles of solute per litre of solution. Therefore, if the molarity is known, a certain volume (V) of the solution is equivalent to certain number of moles of solute:

$$(\text{M})(\text{V}) = \left(\frac{\text{moles of solute}}{\text{litre of solution}}\right)(\text{litres of solution}) = \text{moles of solute}$$

For example, the concentration of an acid solution can be determined by neutralizing the hydronium ion (H_3O^+) with the required volume of a base solution of a known molarity according to the reaction:

$$H_3O^+ (aq) + OH^- (aq) \rightarrow 2\ H_2O\ (l) \qquad (5)$$

In this experiment, you will determine the molarity of an HCl solution using a standardized (known molarity) NaOH solution. The HCl solution concentration will be determined two ways to compare the accuracy and precision of a graduated cylinder and a pipette.

Let's work out an example. **Consult Appendix B and C at this point!** You have a standardized solution of NaOH (*aq*) which has a concentration of 0.1000 ± 0.0002 M. Using a graduated cylinder, you measure out 10.2 ± 0.1 mL (the error associated with a graduated cylinder is ± 0.1 mL) of the HCl solution, and titrate it using the standard NaOH solution. Your initial burette reading was 10.88 ± 0.01 mL (for the purpose of this example, burettes are ± 0.01 mL) and after reaching the end point, the final burette reading was 20.97 ± 0.01 mL. The total volume of

NaOH solution used would be:

$$(20.97 \pm 0.01) - (10.88 \pm 0.01) = \mathbf{10.09 \pm \underline{0.02}\ mL}$$

We **<u>add</u>** the **absolute** uncertainties here (see Appendix B). The number of moles of NaOH (and thus OH^-) would be calculated using equation (6) (note that **relative** uncertainties are added in the case of multiplication or division and then converted back to absolute uncertainties in the final result).

$$A \times B \pm \left[A \times B \left(\frac{a}{A} + \frac{b}{B}\right)\right] \qquad (6)$$

Where:

A = molarity of the NaOH solution
a = the uncertainty associated with the NaOH solution
B = volume of NaOH solution used to reach the endpoint
b = the uncertainty associated with the volume of NaOH solution

$$\text{moles of } OH^- = (0.1000)(0.01009) \pm \left[(0.1000)(0.01009)\left(\frac{0.0002}{0.1000} + \frac{0.02}{10.09}\right)\right]$$
$$= (1.009 \pm 0.004) \times 10^{-3} \text{ mols } OH^-$$

Since equation (5) tells us H^+ and OH^- reacts 1:1, there are $(1.009 \pm 0.004) \times 10^{-3}$ mols of H^+ in the solution we just titrated (note the uncertainty in the moles of OH^- corresponds to the last decimal place reported). We now know the number of moles of H^+ that was in our sample; we just need volume (remember, molarity = moles/volume). We know the volume of HCl solution titrated was 10.2 ± 0.1 mL. So, the $(1.009 \pm 0.004) \times 10^{-3}$ mols of H^+ must have been in the 10.2 ± 0.1 mL of HCl solution. We have moles and volume, so molarity is (we need to divide here):

$$\frac{A}{B} \pm \left[\frac{A}{B}\left(\frac{a}{A} + \frac{b}{B}\right)\right]$$

In this case:

A = moles of H^+ (or HCl)
a = uncertainty associated with the moles of H^+ (or HCl)
B = volume of HCl solution
b = uncertainty associated with the HCl solution

$$\text{Molarity of HCl} = \frac{1.009 \times 10^{-3}}{0.0102} \pm \left[\frac{1.009 \times 10^{-3}}{0.0102}\left(\frac{0.004 \times 10^{-3}}{1.009 \times 10^{-3}} + \frac{0.1}{10.2}\right)\right]$$
$$= 0.100 \pm 0.001 \text{ M HCl}$$

So, our HCl solution has a concentration of 0.100 ± 0.001 M. Notice that our answer is uncertain beginning at the third decimal place, hence we have rounded to this precision (this also happens to be the correct number of significant figures in this example). Compare this with our NaOH solution concentration, which was uncertain beginning at the fourth decimal place. This shouldn't be

surprising since all of the numbers we used to calculate our acid concentration were uncertain, and these uncertainties were propagated to our final answer.

Pre-Lab Assignment

READ Appendix A, B, and C PRIOR TO STARTING THIS PRE-LAB

1. A group of students read the same burette prior to starting a titration and could not agree amongst themselves what the burette read. Their burette readings were as follows (±0.02 mL): 22.38 mL, 22.37 mL, 22.35 mL and 22.36 mL. A lab demonstrator overheard their discussion and offered to read the burette as well. The lab demonstrator obtained a burette reading of 27.43 mL. Determine the mean and standard deviation, and comment on the accuracy and precision of the student readings using the demonstrators' value as the "true" reading.

2. A student titrated 10.00 ± 0.02 mL of an HCl solution with a standardized NaOH solution (M = 0.1284 mol/L ± 0.0002 mol/L) using a phenolphthalein indicator. It required 13.78 ± 0.04 mL of NaOH to reach the endpoint. Calculate the molarity of the HCl solution and the associated uncertainty.

3. Prepare Tables in your lab book like the ones given in the Lab Report section (pages 9 – 13) for recording data throughout the lab session

All waste solutions can be poured down the sink

Experimental Procedure

A. Proper Technique to Read a Burette (done in pairs)

On each bench are four sealed burettes containing water. Record the burette number and read and record the liquid level in each burette, such that there are 4 burette readings per group (see Appendix A for the correct method for reading a burette and meniscus). Ask the lab demonstrators to check your burette readings.

YOU CANNOT START PART B WITHOUT HAVING YOUR READINGS CHECKED.

B. Standardization of a Hydrochloric Acid Solution (done individually)

(a) Obtain ~100 mL of both the standardized NaOH solution and the unknown molarity HCl solution from the stock solutions provided (DO NOT RETURN EXCESS SOLUTIONS TO THE STOCK CONTAINERS, IT WILL CONTAMINATE THE STOCK SOLUTION).

(b) Fill the burette at your station with the NaOH solution. Open the stopcock fully to evacuate any air pockets in the burette. It may be necessary to tap the burette lightly to aid in the removal of trapped air.

(c) Using a 10 mL graduated cylinder, transfer approximately exactly 10.0 mL (it need not be exactly 10.0 mL [***it can't be greater*** than 10.0 mL] hence the phrase approximately, as long as you record the "exact" value) of the HCl solution to an Erlenmeyer flask and record the volume in your data table. Add 3 drops of the phenolphthalein indicator. In acidic media this indicator is colourless and in basic media it is pink.

(d) Prior to beginning this step, confer with a TA to ensure that your burette does not have any air pockets in the jet. Record the initial burette reading (**to X.XX mL ± 0.02 mL**) in your data table. Begin the titration by adding the standardized NaOH solution to the flask until the solution turns a faint but permanent pink colour. This is the endpoint. Record the final burette reading to determine the amount of NaOH required to reach the endpoint. Empty the flask and repeat so that two titrations using the graduated cylinder are completed. For the second trial, obtain a volume of HCl that is as close as possible to the volume of HCl used in the first graduated cylinder trial.

(e) Repeat steps (c) and (d) using a 10.00 mL pipette (see appendix A, or a demonstrator, for the proper technique) to deliver the 10.00 mL of HCl solution to the flask.

C. Assessment of "true" concentration of Hydrochloric Acid (done individually)

Weigh a 125 mL Erlenmeyer flask on the analytical balance and record its mass. **Make sure you use a 125 mL Erlenmeyer flask; the 250 mL Erlenmeyer flask is too heavy for the analytical balance**. Pipette 10 mL of the HCl solution into the flask and reweigh to determine the amount of

HCl added. Add phenolphthalein indicator and reweigh to determine the amount of indicator added. Titrate with NaOH until the first trace of permanent pink colour appears. Weigh the flask again to determine the amount of NaOH required to reach the endpoint. You will use this data for question 4 of the report section to calculate your "true" value.

Experiment 1 Lab Report Hand-in Sheets

Abstract:

Part A:

Burette Number	Burette Reading (±0.02 mL)
Average Reading	
Standard Deviation	

1. Calculate the average reading and determine the standard deviation in the space provided. Report the final values in the table above (be aware of significant figures). [if you have insufficient room write on back of page]

Be sure to include uncertainties in your calculations

Part B:

Concentration of NaOH solution:

	Graduated Cylinder		Pipette	
Trial	1	2	1	2
Vol. of HCl (mL)				
NaOH V_f (±0.02 mL)				
NaOH V_i (±0.02 mL)				
NaOH ΔV (±0.04 mL)				
Molarity of HCl				
Average Molarity				

2. Use the total volume of NaOH used to reach the endpoint and the amount of HCl added to determine the molarity of the unknown HCl solution in each of the 4 trials. Show your work for one of the trials in the space below. Report the average molarity for the graduated cylinder and pipette trials.

Part C:

Mass of empty flask:	
Mass of flask with 10 mL of HCl:	
Mass of flask with indicator:	
Mass of flask with NaOH:	
True Molarity of HCl	

3. Assuming a density of exactly 1.00 g/mL for both NaOH and HCl solutions, calculate the "true" molarity of HCl solution and report it in the table above. Show your work in the space below.

4. Using the molarity calculated in **Part C** as your true value, and the average molarities from the graduated cylinder and pipette trials as your experimental values, determine the percent error associated with each device. Which one was more accurate? Is this expected?

EXPERIMENT #2

TYPES OF CHEMICAL REACTIONS

Objectives

A. To perform and carefully observe several chemical reactions.

B. To write balanced chemical equations for the chemical reactions observed.

C. To identity the contents of unknown solutions.

Introduction *(Zumdahl and Zumdahl, pp. 97-102; pp. 144-169)*

It is not easy to classify all chemical reactions precisely. Nevertheless, in this experiment you will study three basic reaction types: *precipitation*, *reduction-oxidation (redox)*, and *acid-base*. A *precipitation* reaction is one kind of a double-replacement reaction whereby atoms or ions exchange partners and an insoluble product is formed. A *redox* reaction involves the transfer of electrons when one substance is reduced and another is oxidized. An *acid-base* reaction usually involves the transfer of protons from one molecule or ion to another.

Pre-Lab Assignment

1. Predict the products (if any) and write the balanced chemical equation (includes all species), the complete ionic equation (includes all ions), and the net ionic equation (omitting species that appear on both sides of the complete ionic equation) for each of the following:

   (a) K_2SO_4 (*aq*) + $Ba(NO_3)_2$ (*aq*) →
   (b) HNO_3 (*aq*) + NaOH (*aq*) →
   (c) Adding solid silver nitrate to aqueous hydrochloric acid yields...

2. A sample solution is given to you that may contain any or all of the following ions: Na^+, Pb^{2+}, and Cr^{3+}. In an attempt to determine which cation(s) are present, you add the following solutions. You found that no precipitate formed when either an *aqueous* solution of NaCl or of Na_2SO_4 was added to the sample solution. However, a precipitate did form when you treated the sample with a basic solution of NaOH. Which cation or cations are present in the sample? Give your reasoning. (Hint: see Table 4.1 of Zumdahl and Zumdahl).

3. Balance the following *redox* reactions using the half-reaction method. Identify the oxidizing and reducing agents, and the substances oxidized and reduced. (See: Section 18.1 of your Zumdahl and Zumdahl textbook).

   (a) N_2 (*g*) + MnO_4^{2-} (*aq*) → NO_3^{2-} (*aq*) + MnO_2 (*s*) in <u>basic</u> solution
   (b) PbO_2 (*s*) + U^{3+} (*aq*) → Pb^{2+} (*aq*) + UO_2^+ (*aq*) in <u>acidic</u> solution

4. Prepare data tables in your lab book similar to the ones given in the Lab Report section (pages 17 – 22) for recording data and observations throughout the lab session

Procedure ***(in pairs)***

Note: keep in mind that you will be marked on your observations so *record them carefully*.

- All waste solutions are to be poured into the containers provided.
- All metals are to be placed in the container provided, **NOT** in the garbage.

A. Precipitation Reactions

Dispensers are set up containing 0.1 *M* aqueous solutions of Al(III), Ni(II), Ca(II), Cu(II), Fe(II), and Fe(III) ions, as well as a dispenser containing an aqueous solution of NaOH. Take about 2 mL of each cation solution in separate test tubes and, to each, add about the same amount of sodium hydroxide solution. Record the colours of the original solutions and record the overall appearance of each precipitate immediately after adding the base. Leave the mixtures for about half an hour and record your final observations such that you have three sets of observations for each mixture.

B. Redox

Record your observations for the following reactions:

(a) Place some silver(I) nitrate ($AgNO_3$) solution in a test tube to a depth of about 2-3 cm and then add a clean piece of copper wire in the solution. Allow to stand *at least* 30 min. before recording your final observation. What has happened to the wire? Hint: the copper is not "rusting" (copper roofs turn green when they react with the air). What about the Ag(I) solution?

(b) Place 1 mL of 0.1 *M* sodium oxalate ($Na_2C_2O_4$) solution in a test tube. Next, add 10 drops of 6 *M* sulfuric acid followed by 2 drops of 0.1 *M* potassium permanganate solution. Using a test tube clamp, gently warm the solution by moving it in and out of a Bunsen burner flame. As soon as you note any change in colour quickly remove from the flame and examine the solution (NOTE: the solution did not boil). The final solution will be colourless with manganese in the 2+ oxidation state.

C. Acid-Base Reactions

Record your observations for the following reactions:

(a) Add 1 mL of 0.5 *M* Na_2CO_3 to a clean test tube, and then carefully and slowly add dropwise 1 mL of 6 *M* HCl. What happens? This is a general property of metal carbonates in the presence of acids.

(b) Add a small amount of magnesium oxide (MgO) (no more than the size of a pea) to a test tube and add 1 mL of water. Check the pH of the solution using pH paper. Is the solution acidic or basic? This is a general property of ionic metal oxides.

D. Identification of Unknowns

You will be given a set of five test tubes that are labeled with only a number. The object is to determine the identity of the contents of each tube. Each tube will contain one of the following: 0.1 *M* solutions of: HCl, $AgNO_3$, NaOH, Na_2CO_3 and one tube containing only H_2O. Test small amounts of the contents of one tube with a few drops from each of the other four tubes in **SEPARATE** reactions, *i.e.*, **DO NOT MIX MORE THAN TWO OF THE SOLUTIONS IN ANY ONE TEST**. Repeat with the other solutions so that you have an accurate record of the 10 different possible combinations.

Measure the pH of each of the original tubes with universal indicator paper.

Experiment 2 Lab Report Hand-in Sheets

Abstract:

Part A

Report your observations and write net ionic equations (NIE) for each precipitation reaction.

Cation	Solution appearance	Appearance of precipitate	
		Immediately	**After 1 hour**
Al(III)			
	NIE:		
Ni(II)			
	NIE:		
Ca(II)			
	NIE:		
Cu(II)			
	NIE:		
Fe(II)			
	NIE:		
Fe(III)			
	NIE:		

Part B

Record your observations. Indicate the species oxidized and reduced (with oxidation states) in each, and give the overall balanced equation for each reaction.

(a)

Appearance of:	Initially	After 30 minutes
Silver(I) nitrate		
Copper wire		
Species	**Oxidized**	**Reduced**
Overall Balanced Reaction		

(b)

Appearance of solution		
after adding sodium oxalate		
after adding sulfuric acid		
after adding potassium permanganate		
after warming		
Species	**Oxidized**	**Reduced**
Overall Balanced Reaction		

Part C

Record your observations and write net ionic equations for each reaction.

(a) Addition of HCl to Na_2CO_3

Observations:

NIE:

(b) Addition of H_2O to MgO

Observations:

pH of magnesium oxide solution:

NIE:

Part D

Report your observations for mixing the various solutions together and deduce the identity of the contents of each of the numbered tubes. Clearly show your reasoning.

Data:

Test Tube #	Test Tube #	Observations

Identity and Reasoning:

Test Tube #	pH	Identity	Reasoning

EXPERIMENT #3

VOLUMETRIC DETERMINATION OF CHLORINE IN BLEACH: THE IODOMETRIC METHOD

Objectives

A. To apply your knowledge of limiting reagents, stoichiometry and redox chemistry to a genuine chemistry problem.

B. Specifically, to determine the grams of available chlorine per litre of two commercial brands of bleach. Also, to determine the cost per gram of available chlorine in the bleach samples and to compare the cost effectiveness of the two bleach brands.

Introduction *(review Zumdahl and Zumdahl, pp. 102-113 and pp. 157-168)*

You will determine the "available chlorine" in two brands of household bleach. The active ingredient responsible for the bleaching action in commercial bleaches is OCl^-, the *hypochlorite ion*, usually in the form of NaOCl. There are also "chlorine-free" bleaches, which usually have hydrogen peroxide, H_2O_2, as the active ingredient. When the strengths of various bleaches are compared, the standard oxidant for bleaching is assumed to be Cl_2. In other words, the bleaching agent is rated according to an equivalent mass of Cl_2 per unit volume of solution. This rating is called the *available chlorine* (it also applies to chlorine-free bleaches, as a way to rank all bleaches on a common scale).

Colour depends on visible light: when light comes in contact with the coloured substance (fabric dye, stain, etc.), the energy from the light may be absorbed by electrons located in the molecules. This causes a temporary rearrangement of electrons to a higher, less stable, energy level. Colour is produced when only certain parts of the visible light spectrum are absorbed by the particular molecule, the rest of the visible spectrum photons that are not absorbed are scattered or reflected away from the fabric. The summed effect produces the colour that we perceive.

Bleaches are compounds that remove unwanted colour from fabrics by removing "the loosely bound electrons" that are responsible for the production of colour. When electrons are drawn away from a substance, that substance is oxidized (loses electrons). The substance drawing the electrons away is the oxidizing agent (which gets reduced); in the case of a standard bleach it is the hypochlorite ion. The relevant half reaction is:

$$OCl^-\,(aq) + H_2O\,(l) + 2e^- \rightarrow Cl^-\,(aq) + 2\,OH^-\,(aq) \qquad (1)$$

For a "chlorine-free" bleach containing peroxide, the reaction is:

$$H_2O_2\,(aq) + 2\,H^+\,(aq) + 2e^- \rightarrow 2\,H_2O\,(l) \qquad (1a)$$

Removal of electrons from the absorbing energy levels means the whole visible spectrum interacts with the fabric the same way, and the fabric appears white. As you can see, both oxidizing agents accept 2 electrons, so the stoichiometry is the same for peroxide bleaches as it is for hypochlorite bleaches. For the rest of this introduction, we will use the hypochlorite bleach as an example but keep in mind the stoichiometry is the same for peroxide based bleach.

In this experiment the analysis of a bleach involves the redox reaction of the hypochlorite

ion with an excess of iodide ion:

$$OCl^- (aq) + 3\ I^- (aq) + H_2O (l) \rightarrow I_3^- (aq) + Cl^- (aq) + 2\ OH^- (aq) \quad (2)$$

The solution is acidified and the triiodide ion, I_3^-, is titrated with sodium thiosulfate, $Na_2S_2O_3$, solution until the yellow colour of I_3^- (actually $[I_2 \bullet I]^-$) nearly disappears just before the endpoint:

$$I_3^- (aq) + 2\ S_2O_3^{2-} (aq) \rightarrow 3\ I^- (aq) + S_4O_6^{2-} (aq) \quad (3)$$

Starch indicator solution is then added to form a soluble, deep-blue complex ion with the triiodide ion:

$$I_3^- (aq) + \text{starch} (aq) \rightarrow [I_3 \bullet \text{starch}]^- (aq) \text{ (deep blue)} \quad (4)$$

The titration is then continued and at the stoichiometric endpoint, the remaining I_3^- in the complex ion is reduced by the thiosulfate ion (as it is in equation 3) and the blue colour disappears. (This titration is done in two stages because the blue colour is easier to see than a pale yellow!)

$$2e^- + [I_3 \bullet \text{starch}]^- (aq) \rightarrow 3\ I^- (aq) + \text{starch} (aq) \quad (5)$$

To summarize: $Na_2S_2O_3$ reacts with I_3^- that was generated from the OCl^- in the bleach. Since the moles of $Na_2S_2O_3$ that react are known, the moles of OCl^- in the bleach can be determined. By the stoichiometries of equations 2 and 3, **2 moles of thiosulfate ion are equivalent to 1 mole of I_3^- and thus to 1 mole of OCl^-**:

$$OCl^- \rightarrow I_3^- \text{ (from equation 2) and } 2\ S_2O_3^{2-} \rightarrow I_3^- \text{ (from equation 3)}$$

$$\therefore\ OCl^- \rightarrow 2\ S_2O_3^{2-}$$

As stated earlier, since the stoichiometry is the same for a peroxide based bleach:

$$H_2O_2 \rightarrow 2\ S_2O_3^{2-}$$

The "available chlorine" is calculated as if free Cl_2 per unit volume of bleach is actually present and reacts with I^-. If chlorine *was* actually present, it would react as follows:

$$Cl_2 (aq) + 3\ I^- (aq) \rightarrow 2\ Cl^- (aq) + I_3^- (aq) \quad (6)$$

$$I_3^- (aq) + 2\ S_2O_3^{2-} (aq) \rightarrow 3\ I^- (aq) + S_4O_6^{2-} (aq) \quad (7)$$

These equations serve as the basis for determining the strengths of bleaches in this experiment. Notice that both 1 mole OCl^- (equations 2 and 3) and 1 mole Cl_2 (equations 6 and 7) react *indirectly* with 2 moles of $S_2O_3^{2-}$ and **hence 1 mole of OCl^- is equivalent to 1 mole of available chlorine**:

$2\ S_2O_3^{2-} \rightarrow OCl^-$ (from equations 2 and 3) and $2\ S_2O_3^{2-} \rightarrow Cl_2$ (from equations 6 and 7)

$\therefore\ OCl^- \rightarrow Cl_2$
&
$H_2O_2 \rightarrow Cl_2$

In other words, your experimental result will yield moles of OCl^- and H_2O_2 which are actually in the bleach and you will then express them in terms of moles of available chlorine.

This method of creating I_3^- and titrating with thiosulfate is called the **iodometric method.**

Pre-Lab Assignment

1. Prepare a raw data sheet for today's experiment. Consider reviewing the raw data sheet for experiment #1. Allow room for collecting data for 6 titrations

2. A mineral sample containing copper(II) is analyzed using the iodometric method by the reaction:

$$2\ Cu^{2+} + 5\ I^- \rightarrow 2\ CuI + I_3^-$$

The liberated I_3^- is titrated with 0.100 *M* $S_2O_3^{2-}$, requiring 88.6 mL. Using this reaction and equation (3), determine the amount of Cu(II) (in g) in the sample. Show your calculations.

3. You pipette 10.00 mL of commercial bleach (from the bottle) into a 250.00 mL volumetric flask and dilute to the mark with distilled water. Next, you add a 10.00 mL portion of this diluted solution (from the volumetric flask) to an Erlenmeyer flask, followed by NaI. Titrating this solution (using starch indicator) requires 9.52 mL of a standardized solution of $Na_2S_2O_3$ (0.0500 *M*). Assuming OCl^- is the active ingredient, determine:

   (a) the number of moles OCl^- in the 10.00 mL sample of **diluted** bleach

   (b) the number of moles of OCl^- in the 200.00 mL volumetric flask

   (c) the number of moles of OCl^- in the 10.00 mL sample of **undiluted** bleach

   Show your calculations.

Procedure

*(Each partner will analyze one type of bleach and exchange information at the end of the lab. It is your responsibility to ensure your data is exchanged, **NOT** the lab instructor or TA!!)*

All waste solutions are to be placed in the container provided. Nothing is to go down the drains.

It is a very good idea to do the first titration quickly to get an idea of the approximate volume of thiosulfate solution required to reach an endpoint.

A. Hypochlorite Bleach

Take approximately 30 mL of one of the given commercial bleaches in a clean, dry beaker.

Note the brand, cost, and volume of the original container. Pipette 10.00 mL of the bleach into a 200 mL volumetric flask (*see Appendix A*) and then make up to exactly 200.00 mL with distilled water. It is a good idea to switch to a medicine dropper as you approach the etched line on the neck of the volumetric flask to ensure you don't over fill your flask. Stopper the flask and **invert the flask** a few times to ensure effective mixing.

Clean a burette, rinse it with small portions of the standard $Na_2S_2O_3$ solution and fill it with the same. **Note the exact molarity of the thiosulfate solution which is marked on the container.** Proper titration technique is shown in *Appendix A*.

Pipette 10.00 mL of the diluted bleach solution to a 250 mL Erlenmeyer flask. Add 25 mL of distilled water and 1 g of KI. Next, add 5 mL of glacial acetic acid (CARE!) and *immediately begin the titration* by titrating the liberated iodine (triiodide ion, red-brown colour) with the standard $Na_2S_2O_3$ solution until the red-brown colour fades to light yellow. **IF YOU DO NOT ADD THE ACID BEFORE TITRATING YOU WILL REACH A (FALSE) ENDPOINT WITH ONLY A FEW DROPS OF THE THIOSULPHATE SOLUTION.** Once the red-brown colour fades to light yellow, add 2 mL of the starch solution to form the deep-blue complex. Continue this titration **SLOWLY** (since you are very close to the endpoint and the reaction with the complex is not instantaneous) until the blue colour just disappears.

> REPEAT WITH FURTHER 10.00 mL PORTIONS OF THE DILUTED BLEACH. **YOU MUST GET TWO TITRES THAT ARE WITHIN 0.04 mL OF ONE ANOTHER.** TAKE THE AVERAGE OF THE TWO TITRES THAT ARE WITHIN "0.04 mL.

B. Peroxide Bleach

As above, take approximately 30 mL of one of the "chlorine-free" bleaches in a clean, dry beaker. Note the brand, cost, and volume of the original container. Pipette 10.00 mL of the bleach into a 200 mL volumetric flask and make up to exactly 200.00 mL with distilled water, again using a medicine dropper as you approach the etched line on the volumetric flask. Mix well.

Pipette 10.00 mL of the diluted bleach solution to a 250 mL Erlenmeyer flask. Add 25 mL of distilled water and 1 g of KI. Next, add 5 mL of glacial acetic acid (CARE!) and **2 TO 3 DROPS OF THE "MOLYBDENUM REAGENT" (make sure all of the KI has dissolved before adding the molybdenum reagent)**. Titrate with the standard $Na_2S_2O_3$ solution. The titration will be the same as that above for Part A (go to straw yellow, add starch, then to colourless endpoint).

Report *(Informal – see pages vi - vii)*

1. Note the types of bleach used and the molarity of the thiosulfate solution.

2. Report **ALL** titration readings, *i.e.*, initial/final burette readings and total volumes.

3. Calculate for each bleach sample:

   (a) the number of grams of available chlorine per litre of undiluted bleach (see the relationships between the various reagents, outlined in the **Introduction**, specifically those which appear in the text boxes).

(b) cost per gram of available chlorine (note the volumes/prices for each bleach).

(c) for your hypochlorite bleach, calculate the percent by mass of NaOCl [(g of NaOCl per g bleach) × 100%] in the undiluted bleach, assuming the density of the bleach to be 1.084 g/mL (*i.e.,* 1 mL of bleach weighs 1.084 g). Note: you have already found mol OCl^- (which equals the number of mols of NaOCl) in 3(a), above.

(d) for the peroxide bleach, repeat the percent mass calculation as in 3(c), except use a density of 1.006 g/mL

4. Is KI or the active ingredient (NaOCl or H_2O_2) the limiting reagent in each titration? How do you know? (Note: a calculation is required to answer this question)

5. Comment on the cost effectiveness of hypochlorite versus peroxide products by comparing your results for the two brands of bleach.

EXPERIMENT #4

SPECTROSCOPY AND QUALITATIVE ANALYSIS IN THE DETERMINATION OF AN UNKNOWN ZINC SALT

Objectives

There are several objectives to this experiment. You will learn about Beer's Law and how to apply it using data collected from a spectrophotometer. You will also learn about qualitative analysis for anion determination. Using this information you will determine the identity of a zinc salt, its molecular weight and the water of hydration.

Introduction (Zumdahl & Zumdahl, pp. 153-158, pp. 296-304, pp. 973-975, and pp. A16-A19)

A. Spectrophotometry

The electromagnetic (EM) spectrum covers an enormous range of wavelengths (or energies). The major divisions are depicted in Figure 7.2 of your textbook. You will notice that the region that can be perceived by the human eye (the visible spectrum, 400 – 700 nm) is only a small part of this spectrum. When a substance absorbs energy in any region of the EM spectrum it will undergo a change. This change can be bond vibration, molecular rotation, electron excitation etc. The amount of energy required to promote this change determines the region in which the change is observed. A graph of the amount of energy absorbed by a species at different wavelengths is called an absorption spectrum and it can be used to identify the species or determine its concentration (Figure 1). Normally, single wavelength analysis is used for concentration determinations (as long as the wavelength of maximum absorbance, λ_{MAX}, is known).

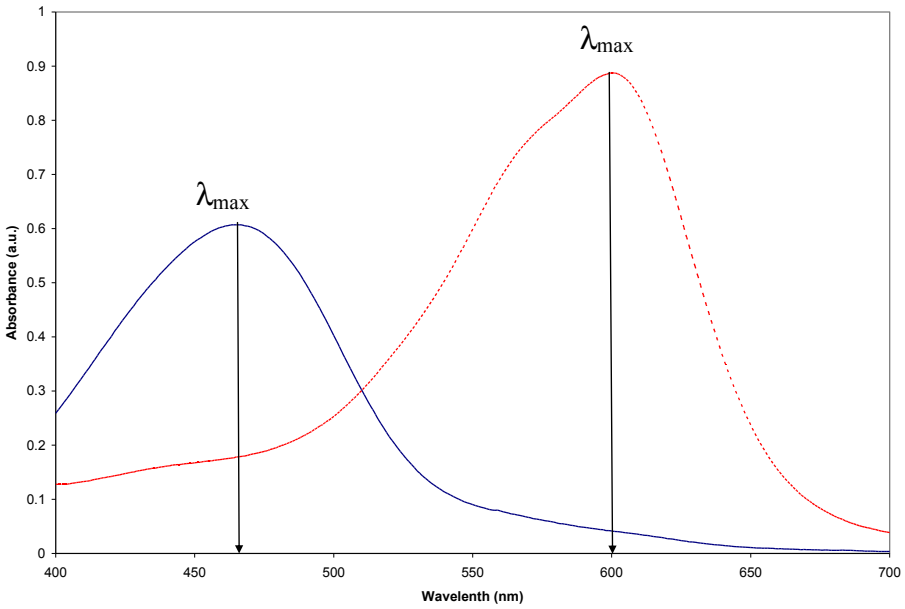


Figure 1: Absorption spectrum of Zincon reagent (blue line) and Zincon-zinc complex (red dots). Note λ_{MAX} for each species.

Instruments that measure absorbance spectra are called spectrophotometers. In these instruments, a beam of energy (light) passes through a sample to a detector. The absorbance (A) is the logarithm

of the ratio of the original amount of energy (I_o) relative to the amount of energy reaching the detector (I):

$$A = \log(I_o/I)$$

The spectrophotometer is set at a fixed wavelength where the absorbance of the sample is at a maximum (λ_{MAX}). At fixed wavelengths, the amount of energy absorbed is proportional to two parameters: (i) the concentration of the absorbing species, and (ii) the distance travelled through the solution containing the absorbing species. The dependence of absorbance on these two factors is summarized by Beer's Law:

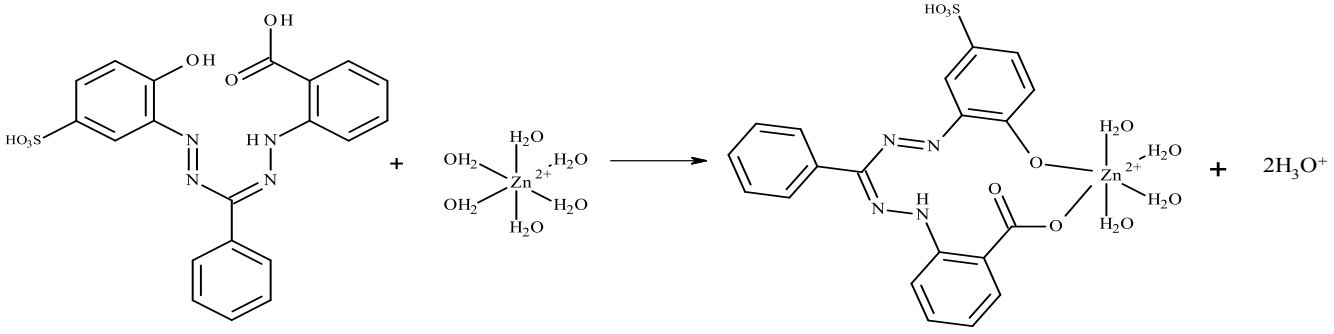
$$A = \epsilon \bullet l \bullet c$$

where A is the amount of light absorbed (absorbance), ϵ is the molar absorptivity coefficient (a proportionality constant), l is the path length (distance travelled through the solution) and c is the concentration of the solution. By operating at a fixed wavelength, ϵ remains constant and if all of measurements are recorded using the same cell l remains constant. Under these conditions, absorbance becomes directly proportional to concentration.

The first part of a quantitative analysis experiment involves determining the relationship between the amount of energy absorbed and the compound of interest. This is accomplished by preparing a series of standards containing the compound of interest, with known concentrations. One of the standards will be a blank solution. This solution will contain everything used to prepare standards and your unknown except the compound of interest. This will remove any influence from the other components of the solution on the observed absorbance. This is especially important in this experiment since the Zincon reagent, which is used to complex the zinc, absorbs slightly at the fixed wavelength being used (see Fig. 1).

B. Zincon and Zn(II)

Aqueous solutions of the Zn(II) salts being analyzed do not absorb in the visible region of the EM spectrum and thus cannot be analyzed by traditional visible spectrophotometers. However, there are a variety of reagents that can form complexes with Zn(II) ions do absorb in the visible region. One such reagent is *o*-{2-[α-(2-hydroxy-5-sulfophenylazo)-benzylidene]hydrazino} benzoic acid (hence the short form Zincon). Zincon reacts with zinc(II) ions in a one to one reagent to metal ratio:



The complex is intensely coloured and obeys Beer's law at concentration ranges of 0.1 to 2.4 parts per million (ppm), where 1 ppm equals 1 mg/L.

C. Water of Hydration

Quite often, salts will crystallize with a certain quantity of water present, known as water of hydration. The inclusion of water into the crystalline solid can take several forms, including but not limited to: formation of a complex ion where water is coordinated to the metal, water molecules existing between the cation and anion or as lattice water (where water molecules occupy lattice positions but are not associated directly with either anion or cation). The quantity, or moles, of water present in a salt can be estimated by comparing the experimentally determined molecular weight to that of the anhydrous form of the salt.

D. Qualitative Analysis

Qualitative analysis is the study of the way and manner in which substances can be identified. Inorganic qualitative analysis focuses on determining which cations and anions are present in substances and mixtures of substances. A complete system of analysis would involve methods for determining all possible cations and anions which is outside the scope of this experiment. Instead, you will focus on a few simple tests to identify which anion is in your sample. Since this type of analysis normally involves procedures based on the reactions of ions, a solution of the sample is required. Depending on the compound being investigated, creating a solution can be challenging since strong acids and bases may be required to dissolve the compound. The procedure listed for the determination of the anion in your sample is applicable only in the absence of other anions that could interfere with your result. Therefore, it is imperative that all glassware used in this portion of the lab be clean and dry. Errors frequently occur from the use of beakers, flasks and test tubes that contain trace interfering ions or ions included in the analysis scheme but are not present in your sample. SO TAKE CARE!!

Pre-Lab Assignment

1. Prepare a raw data sheet for today's experiment. You may wish to consult the raw data sheets for experiments 1 and 2. (NOTE: If unprepared, student will be unable to continue with lab session!)

2. What is a Beer's Law plot and what information will be needed to generate it? What information will you be able to get from this plot?

3. In what manner will the waste generated in this experiment be disposed?

4. In step 2 of the procedure why is it important to thoroughly rinse the 10 mL beaker into the 100 mL volumetric flask?

5. A 111.4 mg ore sample containing iron is digested with acid, filtered and diluted to 200.0 mL in a volumetric flask. A 10.00 mL aliquot is transferred to a 100.00 mL volumetric flask containing hydroxylamine hydrochloride, sodium acetate and 1,10-phenanthroline and diluted to the mark. The resulting orange-red solution had an absorbance of 0.486 at 508 nm in a 1 cm cell. If the molar absorptivity of the iron-phenanthroline complex at this wavelength is 1.89×10^3 L/mol•cm, determine the percentage of iron in the ore sample.

Procedure ***(in pairs)***

- All Zincon-containing solutions can be poured down the drain.
- The stock zinc solution is to be poured in the container provided.
- All waste from the qualitative analysis section is to be placed in the container provided.

Preparation of zinc standards and unknown zinc salt

1. Obtain one of the kits provided as well as one of the unknown zinc salts. All of the zinc salts (which may or may not be hydrated) are in the +2 oxidation state and will have one of the following anions: Cl^-, Br^-, I^-, SO_4^{2-} and CH_3COO^- (acetate). Also, obtain 150 mL of the pH 9 buffer.

2. Using the 10 mL beaker weigh out ~ 0.03 to 0.035 g (±0.0001 g) of your salt. Transfer the salt to a 100.00 mL volumetric flask using a small amount of water. Rinse the beaker 3 to 4 times and transfer the washing to the flask. Allow the Zn salt to completely dissolve and then **dilute to the mark**.

3. Transfer 1 mL of the solution from step 2 (using the provided pipette) into a 100 mL volumetric flask. To the flask add 20 mL of the pH 9 buffer (pipette) and, from the burette provided, 2 mL of the Zincon reagent. Dilute to the mark and mix thoroughly.

4. Prepare the standards as follows: into individual 100 volumetric flasks add 1, 2, 3 and 4 ml of the standard Zn^{2+} solution from the burettes provided (record the concentration of the Zn^{2+} solution). **It need not be exactly 1.00, 2.00, etc. mL. However, the actual volume added must be known. Therefore, initial and final burette readings are required (±0.02 mL).** To the individual flask also add 20 mL of the pH 9 buffer and 2 mL of the Zincon reagent. Dilute to the mark and mix.

5. Using a beaker, prepare the blank by adding 20 mL of the pH 9 buffer and 2 mL of the Zincon reagent and dilute to 100 mL with distilled water. **NOTE: A VOLUMETRIC FLASK IS *NOT* NEEDED FOR THIS STEP, SO *DON'T* USE ONE!!**

6. To analyze your samples ask the lab demonstrators for instruction on the proper use of the spectrophotometer. Obtain the absorbance of each of the standards and the unknown at 600 nm.

Quantitative Analysis

Transfer some of your salt (tip of a scoopula) into a beaker and dissolve it in ~ 30 mL distilled water. Transfer equal portions of this solution into three test tubes (about 2-3 cm in height).

1. **Chloride and Bromide.** Add 3 drops of 6 *M* HNO_3 to the solution in the first test tube and shake well. Add 5 drops of 0.1 *M* $AgNO_3$ and mix well. If a precipitate has formed upon

the addition of silver nitrate a positive test for chloride or bromide has occurred.

To determine if you have the chloride **or** bromide anion add 10 drops of 6 M NH_4OH to the mixture (do not mix). If there are two distinct layers then the chloride anion is present. If the solution did not change then the bromide anion is present.

2. **Sulfate.** Add 3 drops of 6 *M* HNO_3 to the solution in the second test tube and shake well. Add 5 drops of 0.1 *M* $BaCl_2$. The formation of a white precipitate indicates the presence of SO_4^{2-}.

3. **Iodide.** To the final test tube add 3 drops of 6 M HNO_3 and shake well. Add 10 drops of the sodium hypochlorite (NaOCl) solution. The appearance of a dark brown/black precipitate indicates the presence of the iodide anion.

4. **Acetate.** Place a small amount of the solid salt (~ the size of a pea) into a test tube. Add 1 mL of concentrated H_2SO_4 (CARE!!!) and 2 mL of amyl alcohol (1-pentanol) from the burettes in the fume hood and mix with a glass stirring rod. Place the test tube in the water bath provided for about 5 minutes. Remove the test tube from the bath and check the odour of the solution by bringing your nose slowly to the test tube and waving your hand over the top of the test tube towards your nose. You can also pour it onto a watch glass to make it easier to pick up the odour. If a fruity odour is present (it often reminds people of fake banana smell), it indicates the presence of the acetate ion.

Report *(Formal – see page vii)*

1. Using the concentration of the stock Zn solution and the volumes delivered, calculate the concentration of your standard Zn solutions in ppm.

2. Construct a Beer's Law plot using mm graph paper (absorbance vs. Zn concentration, in ppm). Draw a line of best fit through your data points. Don't forget that it must go through (0,0) to account for the blank. Clearly mark the absorbance of the unknown sample and determine its concentration.

3. Using the mass of the sample analyzed, the dilutions you performed and the positive anion test determine: the identity of your unknown salt, its molecular weight and the water of hydration.

4. Propose net ionic equations for all observed reactions. Include anion tests that gave negative results but give equations as if the reactions gave positive results.

EXPERIMENT #5

ORGANIC CHEMISTRY
PREPARATION AND PURIFICATION OF AN ESTER

Objectives:

1. Develop basic techniques used in an Organic Chemistry Laboratory

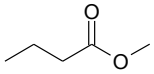
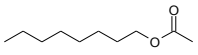
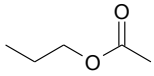
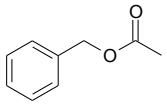
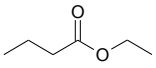
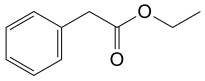
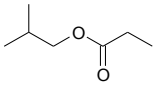
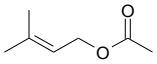
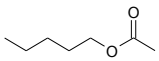
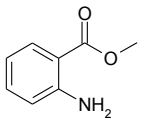
2. Prepare an ester from the parent alcohol and acetic anhydride

Introduction

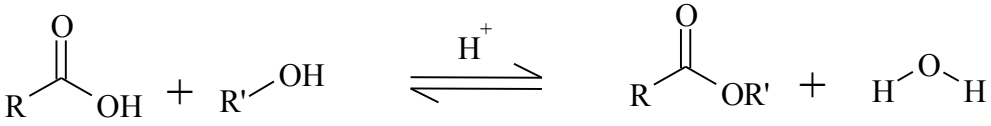
A. Theoretical Aspects

Although many organic compounds have traditionally been isolated from plant and animal sources, most organic compounds used today are synthesized. They are sometimes synthesized from inorganic salts like carbonates and cyanides, but more commonly from other organic compounds (i.e. using small organic compounds to make larger ones).

Esters are an important class of organic molecules that are formally derived from alcohols and carboxylic acids. Esters are very prevalent in nature and are important components of many flavour and fragrance industries. Simple structural variations of either the alcohol or carboxylic acid can lead to markedly different fragrances:

Ester	Structure	Fragrance	Ester	Structure	Fragrance
Methyl butyrate		Apple	Octyl acetate		Orange
n-propyl acetate		Pear	Benzyl acetate		Peach
Ethyl butyrate		Pineapple	Ethyl phenylacetate		Honey
Isobutyl propionate		Rum	Isopentenyl acetate		Juicy fruit
Amyl acetate		Banana	Methyl anthranilate		Grape

The formation of an ester from an alcohol and a carboxylic acid can be catalyzed by the addition of a strong acid such as sulfuric acid (denoted as H^+ over the arrows in the reaction scheme below). However, the water produced during the reaction can hydrolyze the ester. Therefore esterification of an alcohol is a reversible equilibrium process:

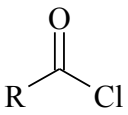


with the following equilibrium expression:

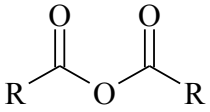
$$K_{eq} = \frac{[\mathrm{RCOOR'}][\mathrm{H_2O}]}{[\mathrm{RCOOH}][\mathrm{R'OH}]}$$

where R and R′ represent unspecified, but different, carbon containing substituents. This equilibrium can be shifted (Le Châtelier's principle) to favour ester formation by either increasing the amount of reactants (carboxylic acid or alcohol) or reducing the amount of products (typically water through distillation) in the reaction mixture. Unfortunately, purification of the product can become more difficult in the former case and in the latter, high temperatures may destroy the product.

An alternative method is to use a more reactive derivative of the carboxylic acid. The acid chloride or acid anhydride are the most common derivatives:



Acid Chloride



Acid Anhydride

Reactions involving the chloride or anhydride are typically highly exothermic (much more reactive) and favour the formation of the ester. In this experiment you will prepare an ester by using acetic anhydride and one of the following parent alcohols:

1-propanol

1-pentanol

phenyl methanol

B. Practical Aspects

Inevitably, once the organic reaction is complete, the reaction mixture will contain the target molecule as well as side products and any catalyst used. In addition, a reaction always has a limiting reagent, resulting in starting material. Therefore, the target molecule has to be separated and purified from the reaction mixture. The most common methods of separation are distillation, filtration, and extraction (which will be used in this experiment). Prior to extraction, the reaction mixture should be neutralized (worked up). This accomplishes two things: safety (*e.g.*, eliminates strongly acidic/basic solutions that can be spilled on clothing), and it changes the solubility of the desired product, making it easier to separate from the reaction mixture.

Extraction

A common method of extraction is liquid/liquid extraction (using a separatory funnel, Figure 1); one phase is *aqueous* and the other a suitable organic solvent. The densities of the two phases should be suitably different such that a distinct phase boundary is created. Proper use of a separatory funnel will be shown during the pre-lab talk.

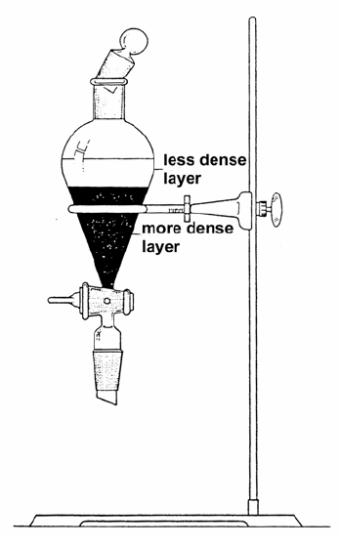


Figure 1: Separatory funnel showing two distinct phases, one aqueous and one organic.

The mixture is shaken and if the right solvent has been selected most of the organic substance will be transferred from the aqueous phase to the organic phase. However, most organic materials (including esters) have an equilibrium balance of material in both phases, so all of the organic substance is not transferred to the organic phase in the first mixing. Therefore, multiple extractions are required. A good solvent for extraction should be negligibly soluble in the liquid phase holding the desired product, it should be volatile with a boiling point substantially lower than that of the organic substance so that it can be removed readily through evaporation, and most importantly, the desired organic substance should be very soluble in the extracting solvent.

Drying agents

In any extraction, each phase becomes saturated with respect to the other phase (*i.e.* some organic solvent will interact with the *aqueous* phase and some of the *aqueous* material will interact with the organic phase). Therefore, it is necessary to remove all dissolved water from the organic phase prior to evaporation. This is accomplished using a two-step process. First, the organic phase is washed with a saturated sodium chloride solution. The salt water works to pull the water from the organic phase into the water phase (since the salt solution wants to become more dilute and it has a stronger affinity for water than the organic phase). The second step involves adding an anhydrous inorganic salt (*e.g.* Na_2SO_4) to the organic phase to remove any remaining water by the reaction:

$$Na_2SO_4 + 10\ H_2O \rightarrow Na_2SO_4 \bullet 10H_2O$$

Calculation of Yield

Quite often there is some confusion for students between product yield and percent error calculations (see introduction of Experiment 1). In a percent error calculation the primary concern is the deviation of the experimental value from that of the true value. Conversely, in a percent yield calculation the chief concern is the amount of product produced based on the theoretical amount of product attainable:

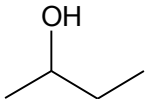
$$\text{Percent Yield} = \frac{\text{mass of product}}{\text{theoretical mass of product}} \text{x}100$$

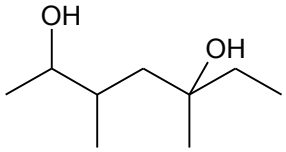
The mass of product is simply the actual mass of the product obtained in your reaction (following any purification steps). The theoretical mass of product is determined based on the limiting reagent. The mass of the limiting reagent is converted to moles and, based on the balanced chemical equation, equated to the number of moles of product that could be theoretically produced. The number of moles is then converted back to a mass (using the molecular weight of the product) and used to determine the percent yield.

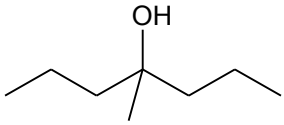
Pre-Lab assignment

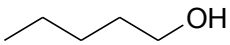
1. Prepare a raw data sheet (NOTE: If unprepared, student will be unable to continue with lab session!).

2. Define the hydroxyl groups in the following alcohols as primary, secondary, or tertiary.

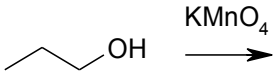
(a) 

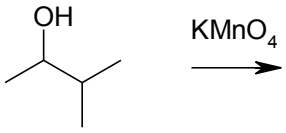
(b) 

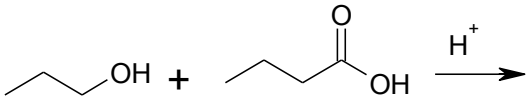
(c) 

(d) 

3. Give the IUPAC names of the starting materials and predict the products (as well as their IUPAC names) for the following reactions:

(a) 

(b) 

(c) 

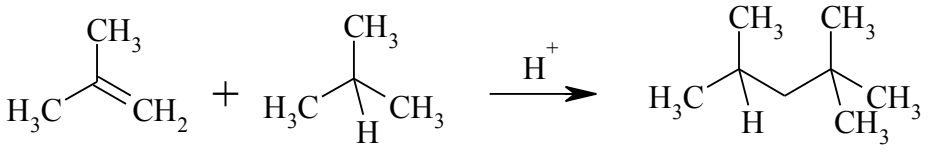
4. A separatory funnel contains a water phase and the following organic solvents as the organic phase.
   (a) Ether (also known as ethyl ether or diethyl ether)
   (b) Dichloromethane (also known as methylene chloride)

(c) Ethyl acetate (also known as acetic ether)
(d) Chloroform (also known as trichloromethane)

In each case, where is the water phase expected to settle; on the top or bottom? (Hint: density is an important factor) You may wish to consult the CRC handbook or a Merck index for assistance.

5. Extraction of an organic substance from a water mixture is required. The organic substance is soluble in both ethanol and ether. Which solvent should be used to fully extract the organic substance? Why?

6. A reaction vessel contains 10.13 g of isobutylene, 12.00 g of isobutane and an acid catalysis. They react according to the following balanced reaction scheme:



Isobutylene Isobutane 2,2,4-Trimethylpentane

After working up the reaction mixture, 17.35 g of product was obtained. Determine:

(a) The limiting reagent
(b) The theoretical mass of product (assume 100% yield)
(c) The percent yield of the reaction

Procedure (Work in Pairs)

All waste solutions are to be poured into the container provided.

ETHER IS HIGHLY VOLITILE AND EXTREMELY FLAMMABLE. IT CAN FORM EXPLOSIVE MIXTURES WITH AIR. OPEN FLAMES OR SOURCES OF HEAT ARE NOT PERMITTED DURING THE LABORATORY PERIOD.

1. Weigh a dry 125 mL Erlenmeyer flask. Add 5 mL of your alcohol weigh and record the mass. Add 7 mL of acetic anhydride (CARE!! Acetic anhydride is a severe skin irritant) and record the total mass. Swirl to mix. ***If, at this point, you have selected 1-propanol as your alcohol consult your lab instructor for assistance.*** Add 3 drops of concentrated H_2SO_4 (CAUTION!!). The reaction mixture will become quite warm, mix the solution periodically until the temperature of the solution returns to ambient temperature. Note the appearance of the mixture.

2. Once the reaction mixture has cooled, add cold 20% NaOH to the reaction flask in 10 mL increments (measuring the pH after each addition with universal litmus paper) until the pH

of the solution is basic. Again, allow the solution to cool and note the appearance of the mixture.

3. Transfer the solution to a separatory funnel and add 10 mL of ether to the reaction flask. Swirl and transfer the ether to the separatory funnel. Mix the two phases as shown during the pre-lab talk (MAKE SURE TO VENT THE FUNNEL PERIODICALLY TO RELIEVE THE BUILT UP PRESSURE!!). Let the funnel sit for a few minutes to allow the two phases to separate. Draw off the bottom phase into the reaction flask and transfer the top phase (containing ether and product) into a clean, dry flask/beaker. Repeat this step two more times such that about 30 mL of ether has been used.

4. Discard the water phase and transfer the organic phase back to the separatory funnel. Add 25 mL of saturated NaCl solution to the funnel and mix as in step 3. Draw off the bottom phase and discard. Transfer the top phase to a flask or beaker.

5. Add drying agent, in moderate amounts, to the ether/ester mixture until the drying agent no longer clumps together (indicating all of the water has been removed from the ether).

6. To a flask add a few boiling chips, weigh and record the mass. Decant the now dry ether mixture into the flask with the boiling chips. To the residual drying agent add another 10 mL of fresh ether, mix and decant again. Place the flask on a steam bath and boil to remove the ether from the mixture. Once the solution stops boiling allow the flask to cool and weigh to determine the amount of product obtained.

Report *(Informal – see pages vi - vii)*

1. Propose a balanced chemical reaction for the formation of your ester.

2. Based on the appearance of the mixtures after steps 1 and 2, comment on the solubility of your ester in acidic and basic media. Would more ether be required to extract your ester from an acidic or basic environment? Why?

3. Why would ether be added to the residual drying agent in step 6?

4. (a) Is the alcohol or acetic anhydride the limiting reagent in this reaction? Why?

   (b) Based on your answer for (a), what is the theoretical mass of your ester that could be produced (assume 100 % yield).

   (c) Based on your answer for (b), determine the percent yield for your ester.

5. Explain why you did not obtain a 100 % yield. **NOTE**: "Human error" cannot be used as an explanation, we are looking for a systematic answer that would always apply.

6. Give two common commercial uses of your ester.

APPENDIX A

WEIGHING AND VOLUMETRIC EQUIPMENT AND TECHNIQUES

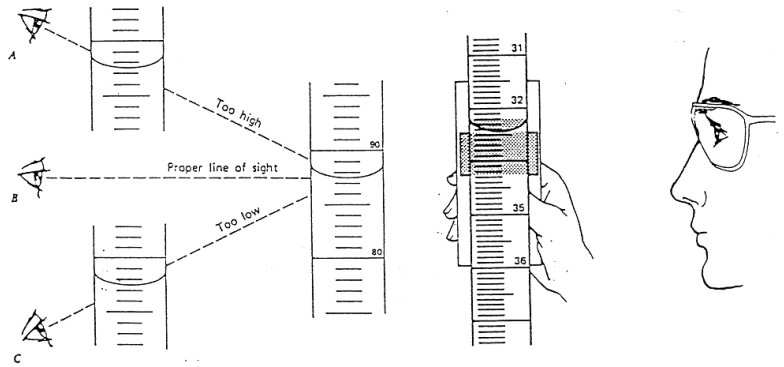
Analytical Balance

The analytical balances in the lab can be read to four decimal places (*i.e.,* to the nearest 0.0001 g or 0.1 mg), with uncertainties of ±0.0001 g. **All analytical weighings are made by difference** (never add your reagent to the weighing container while it is on the pan), so the effect of a possible systematic error in the balance (*e.g.,* always providing readings 0.01 g higher) is not important, **provided that the two readings are made on the same balance!** Be sure not to rest your arm or apply any pressure on the balance table, as the balance is quite sensitive to slight changes in position. When performing a weighing or zeroing the balance, make sure the sliding doors are closed, as even small wind currents will frustrate balance readings. Make sure you are aware of the maximum allowable mass the balance can sustain before placing your weighing container (*e.g.,* boat, small beaker, etc.) on the pan, or measuring out a specific quantity. Most four-place balances cannot be used for masses greater than about 150 g. If some of your reagent is spilled, **clean it up immediately.**

Volumetric Equipment and Techniques

A. Reading a Meniscus

For exacting measurements of clear or transparent liquids in graduated cylinders, pipettes, burettes and volumetric flasks, the volume of a solution is read at the bottom of the meniscus. Steady the eye horizontal to the surface of the liquid [Figure A1(a)]; position the top edge of a black mark (made on a white card) just below the meniscus. The black reflection in the meniscus, viewed against the white card, precisely defines the liquid level [Figure A1(b)]. Substituting a finger for the black mark on a white card is not as effective, but it helps.



(a) **(b)**

Figure A1. (a) Reading the meniscus, and (b) using a "meniscus reader".

B. Cleaning of Volumetric Equipment

Wash the glassware with a small amount (it is concentrated) of the detergent solution. There are brushes available in the lab designed to wash the various pieces of equipment. Rinse the glassware well with tap water and then finally with distilled water. No water droplets will adhere to the inner walls of the glassware if it is cleaned properly.

C. Volumetric Flasks

Volumetric flasks are pear-shaped to facilitate drainage, and have a narrow neck to increase the precision of the volume reading at the single graduation mark. They are calibrated to contain a specified volume. A weighed amount (or measured volume) of substance is added to the flask, and solvent is added precisely to the graduation mark. The flask is capped and inverted a number of times to ensure thorough mixing. The temperature of the solution should be close to the calibrated value (a 5°C variation will cause an error of about 1 mL in 1000 mL – that is a 0.02% error per degree).

D. Pipettes

Transfer pipettes are designed to deliver a single, fixed volume of liquid with good precision. Transfer the solution that you intend to pipette from the reagent bottle to a beaker. To avoid contamination, **never** pipette directly from a reagent bottle. Dry the tip of the pipette with a tissue. Using suction from a rubber bulb (**Caution: NEVER use your mouth**), draw 2-3 small portions into the pipette as a rinse (Figure A2). Roll each rinse around in the pipette to make sure the solution washes the entire inner surface (but not into the bulb!). Deliver each rinse to a waste beaker.

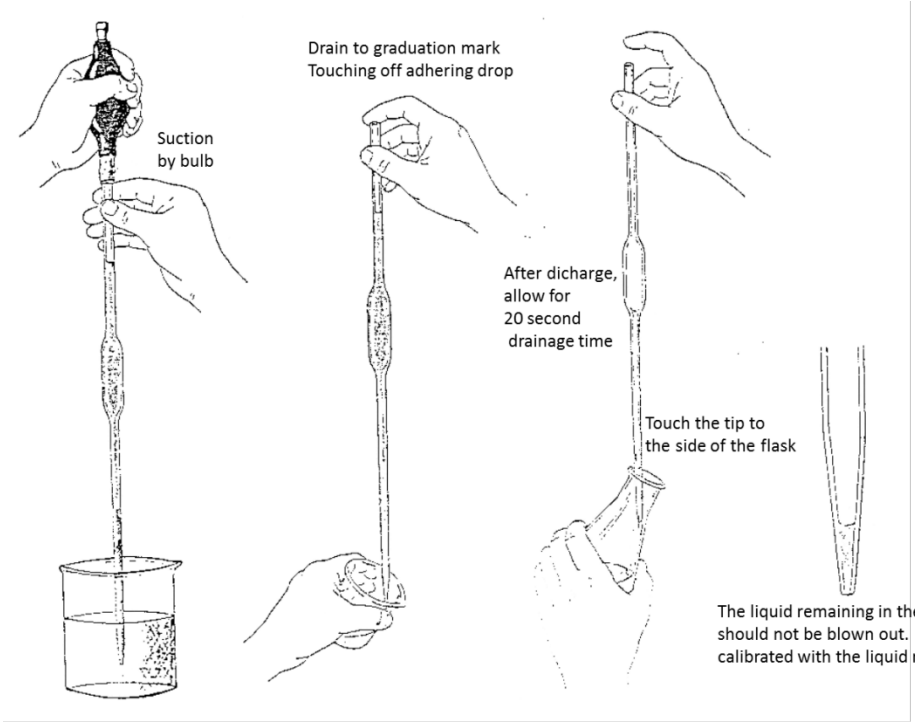


Figure A2. Technique for measuring liquids with a pipette for a right-handed person.

Use the suction bulb to fill the pipette so that the liquid level is above the graduation mark. Remove the bulb and quickly put your index finger over the end of the pipette. Roll your finger

over to the side (so that air can enter the pipette slowly) and allow the liquid to drain until the bottom of the meniscus is exactly on the graduation mark.

The tip of a pipette should be small enough to require 20 seconds to empty a 10 mL pipette, 30 seconds for a 50 mL pipette, etc. If the outflow rate is faster, this much time should be allowed for drainage. The pipette is maintained in a vertical position during discharge, with the contacting wall of the receiving vessel at about 45^{o}. Finally, the adhering drop at the tip is touched off inside the receiving vessel, **but the small portion of liquid in the tip is left in place**, *i.e.* DO NOT BLOW THE LAST DROP OF LIQUID OUT WITH THE PIPETTE BULB.

E. Burettes

Burettes are long cylindrical tubes of very uniform bore, calibrated along their length, with some device to control the flow of liquid - a glass stopcock or a Teflon valve. Burettes are calibrated to deliver the measured volume of liquid. If you are using a particular burette for the first time, examine its markings before you fill it. The lines that span the entire circumference occur for each mL, starting with zero at the top and reaching the maximum volume at the bottom. As a result, the burette will show the volume of a liquid that has been delivered rather than the volume that remains. On a 50 mL burette, the smaller lines indicate each tenth of a mL. The spacings between these lines will allow you to estimate the volume **to the nearest 0.02 mL**. Thus typical burette readings would be 9.34 mL and 17.60 mL. Readings such as 9.3 mL and 17.6 mL are not acceptable.

As with a pipette, the burette must be rinsed with small amounts of the solution to be measured. Then fill the burette to above the zero mark with the stopcock closed. Open the stopcock fully once or twice so that the liquid drains rapidly to flush out any air bubbles in the tip of the burette. Drain the burette until the meniscus rests between the zero and the 1 mL mark. Do not waste time trying to align the bottom of the meniscus with the zero or any other specific mark. Read the burette with your eye on the same level as the meniscus. To obtain the volume of the liquid that you delivered, subtract this reading from the final reading.

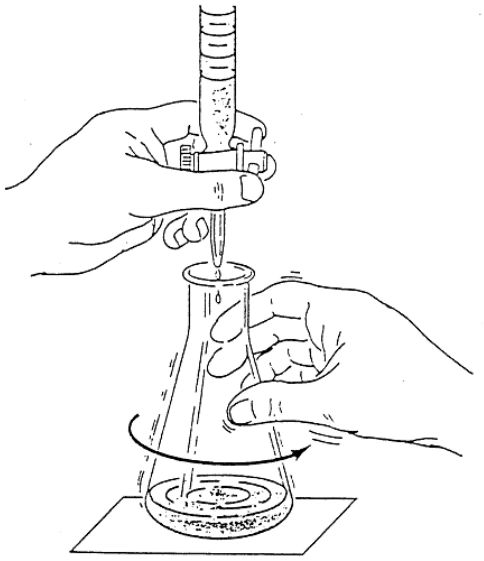


Figure A3. Titration technique for a right-handed person.

The tip must be small enough that the delivery time is sufficient to allow for drainage (about 40 seconds for 25 mL or 90 seconds for the full 50 mL). If the outflow rate is faster, this much time must be allowed before reading the final volume. Figure A3 shows the best technique for a titration by a right-handed student. Note that the left hand is used to open and close the stopcock. With a bit of practice, you will be able to adjust the stopcock so that as little as half a drop will form on the tip. This drop then is touched off onto the inside neck of the receiving flask and washed down with distilled water (unless conducting a potentiometric titration, *i.e.*, using a pH meter). The right hand is used to swirl the flask as the titrant is added. (A left-handed student would turn the burette 180^{o}, control the stopcock with the right hand and swirl with the left hand.) Coloured endpoints are most easily seen if a white background, such as a white tile, is used. As the endpoint is neared the colour of the indicator persists longer in the solution and the addition of titrant is slowed down. Note that, as shown in Figure A3, the tip of the burette is kept

close to the mouth of the flask to avoid errors due to splattering. Experienced chemists usually perform a quick, preliminary titration. Although the endpoint may be overshot, it gives the chemist an idea of the size of the titre to be expected and is preferable to doing the first titration very slowly and carefully when the size of the titre is unknown. This procedure, of course, depends on the amount of solution that is available for analysis.

APPENDIX B

THE PROPAGATION OF EXPERIMENTAL UNCERTAINTY IN CALCULATIONS

Significant Figures and Calculations

For this course we will be using the definitions and rules for significant figures that are given in the textbook by Zumdahl and Zumdahl in Appendix A1.

Experimental Uncertainty

There are several methods for determining uncertainty when measurements are combined in calculations. Below are procedures for estimating the maximum experimental uncertainty.

Addition and Subtraction. When measured quantities are added or subtracted, the maximum absolute uncertainty in the result is the sum of the uncertainty in each measurement.

i.e.: for two numbers (**A** and **B**) and their respective uncertainties (**± a** and ± **b**):

the **sum** (lets call it **C**) and its uncertainty (call it ± **c**) would be:

(A ± a) + (B ± b) = (C ± c) where **C = A + B** and **c = a + b**

the **difference** (subtraction now) between **A** and **B** would be:

(A ± a) - (B ± b) = (C ± c) where **C = A - B** and **c = a + b**

For example, in weighing, you may obtain data such as these:

mass of weighing bottle:	12.2835 " 0.0001 g
mass of bottle + water:	22.2708 " 0.0001 g
mass of water:	9.9873 " **0.0002** g

Here we took the difference (subtraction!) between the two masses to get the mass of the water, yet we still added the uncertainties together. This result can be verified by using data which yield the largest and smallest mass of water:

22.2709-12.2834 = 9.9875 g and 22.2707-12.2836 = 9.9871 g

The mean value is indeed 9.9873, with uncertainty ±0.0002 g.

Multiplication and Division. When measured quantities undergo the operations of multiplication or division, the derived result (in this case **C**) has a maximum relative (*i.e.*, fractional or percentage) uncertainty (in this case **c**) which is the sum of the relative uncertainties in each measurement.

i.e.: for two numbers (**A** and **B**) and their respective uncertainties (**± a** and ± **b**):

the **product** of **A** and **B** (lets call it **C**) and its uncertainty (call it ± **c**) would be

$$A \times B \pm \left[A \times B \left(\frac{a}{A} + \frac{b}{B}\right)\right]$$ (everything within the square brackets is small **c**)

the **<u>quotient</u>** of **A** and **B** (lets call it **C**) and its uncertainty (call it ± **c**) would be

$$A \div B \pm \left[A \div B \left(\frac{a}{A} + \frac{b}{B}\right)\right]$$ (everything within the square brackets is small **c**)

For example, *what is the mass of 20.2 ± 0.1 mL of mercury which has a density, at 20°C, of 13.4562 ± 0.0001 gmL^{-1}?*

This would be an example of <u>multiplying</u> (mass = density × volume) where **A = 20.2** (**a = 0.1)**, and **B = 13.4562** (**b** = 0.0001). So:

$$\text{Mass of Hg} = (13.5462\ \text{g}\bullet\text{mL}^{-1})(20.2\ \text{mL}) \pm \left[(13.5462)(20.2)\left(\frac{0.0001}{13.5462} + \frac{0.1}{20.2}\right)\right]$$
$$= 274\ \text{g} \pm 1\ \text{g}$$

APPENDIX C

EQUIPMENT UNCERTAINTIES

Equipment	Volume (mL)	Uncertainty (± mL)
Volumetric Flask	100	0.08
	200	0.10
	250	0.12
	500	0.20
	1000	0.30
	2000	0.50
Pipette	1	0.006
	2	0.006
	10	0.02
	20	0.03
	25	0.03
	50	0.05
Burette	50	0.02
	100	0.03

	Mass (g)	Uncertainty (± g)
Balance - Analytical	0 to 120	0.0001
Balance – Top loader	0 to 200	0.03
Balance – Top loader	0 to 400	0.03

pH meters:	0.00 to 14.00 pH, ± 0.01 pH
Spectrophotometer:	0 to 2.500 A, ± 0.003 A

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Department of Chemistry

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Please read the following statement. If you accept this statement, please sign and date it in the space provided and give your signed statement to your instructor. NOTE: This statement may not be required by your instructor, but if it is then you MUST complete and return this form before turning in any material for grading.

I certify that ALL of the following are true:

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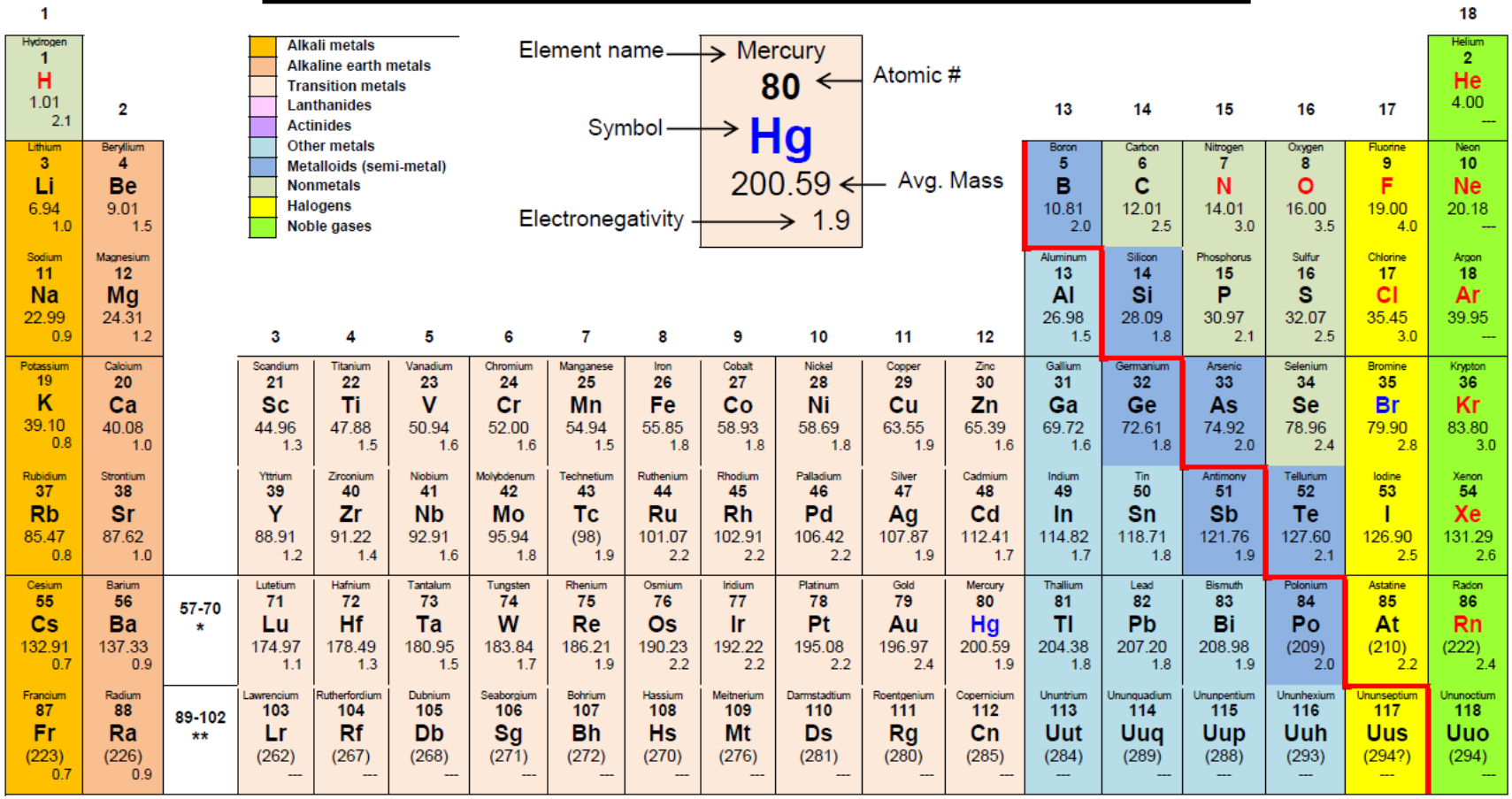
2. I fully understand the definition of plagiarism and recognize specifically that it includes copying of assignments, paraphrasing, reusing old lab reports and related acts.
3. I recognize that the minimum penalty for plagiarism is a zero for the work.
4. If I am unsure about whether something constitutes plagiarism I will consult my instructor before I turn in the assignment.
5. I have given correct information on this form.

Name (Print):	Student ID #:
Signature:	Date:

The Periodic Table of the Elements (with Electronegativities)



	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
	Hydrogen 1 H 1.01 2.1																	Helium 2 He 4.00 ---
	Lithium 3 Li 6.94 1.0	Beryllium 4 Be 9.01 1.5											Boron 5 B 10.81 2.0	Carbon 6 C 12.01 2.5	Nitrogen 7 N 14.01 3.0	Oxygen 8 O 16.00 3.5	Fluorine 9 F 19.00 4.0	Neon 10 Ne 20.18 ---
	Sodium 11 Na 22.99 0.9	Magnesium 12 Mg 24.31 1.2											Aluminum 13 Al 26.98 1.5	Silicon 14 Si 28.09 1.8	Phosphorus 15 P 30.97 2.1	Sulfur 16 S 32.07 2.5	Chlorine 17 Cl 35.45 3.0	Argon 18 Ar 39.95 ---
	Potassium 19 K 39.10 0.8	Calcium 20 Ca 40.08 1.0	Scandium 21 Sc 44.96 1.3	Titanium 22 Ti 47.88 1.5	Vanadium 23 V 50.94 1.6	Chromium 24 Cr 52.00 1.6	Manganese 25 Mn 54.94 1.5	Iron 26 Fe 55.85 1.8	Cobalt 27 Co 58.93 1.8	Nickel 28 Ni 58.69 1.8	Copper 29 Cu 63.55 1.9	Zinc 30 Zn 65.39 1.6	Gallium 31 Ga 69.72 1.6	Germanium 32 Ge 72.61 1.8	Arsenic 33 As 74.92 2.0	Selenium 34 Se 78.96 2.4	Bromine 35 Br 79.90 2.8	Krypton 36 Kr 83.80 3.0
	Rubidium 37 Rb 85.47 0.8	Strontium 38 Sr 87.62 1.0	Yttrium 39 Y 88.91 1.2	Zirconium 40 Zr 91.22 1.4	Niobium 41 Nb 92.91 1.6	Molybdenum 42 Mo 95.94 1.8	Technetium 43 Tc (98) 1.9	Ruthenium 44 Ru 101.07 2.2	Rhodium 45 Rh 102.91 2.2	Palladium 46 Pd 106.42 2.2	Silver 47 Ag 107.87 1.9	Cadmium 48 Cd 112.41 1.7	Indium 49 In 114.82 1.7	Tin 50 Sn 118.71 1.8	Antimony 51 Sb 121.76 1.9	Tellurium 52 Te 127.60 2.1	Iodine 53 I 126.90 2.5	Xenon 54 Xe 131.29 2.6
	Cesium 55 Cs 132.91 0.7	Barium 56 Ba 137.33 0.9	Lutetium 71 Lu 174.97 1.1	Hafnium 72 Hf 178.49 1.3	Tantalum 73 Ta 180.95 1.5	Tungsten 74 W 183.84 1.7	Rhenium 75 Re 186.21 1.9	Osmium 76 Os 190.23 2.2	Iridium 77 Ir 192.22 2.2	Platinum 78 Pt 195.08 2.2	Gold 79 Au 196.97 2.4	Mercury 80 Hg 200.59 1.9	Thallium 81 Tl 204.38 1.8	Lead 82 Pb 207.20 1.8	Bismuth 83 Bi 208.98 1.9	Polonium 84 Po (209) 2.0	Astatine 85 At (210) 2.2	Radon 86 Rn (222) 2.4
	Francium 87 Fr (223) 0.7	Radium 88 Ra (226) 0.9	Lawrencium 103 Lr (262) ---	Rutherfordium 104 Rf (267) ---	Dubnium 105 Db (268) ---	Seaborgium 106 Sg (271) ---	Bohrium 107 Bh (272) ---	Hassium 108 Hs (270) ---	Meitnerium 109 Mt (276) ---	Darmstadtium 110 Ds (281) ---	Roentgenium 111 Rg (280) ---	Copernicium 112 Cn (285) ---	Ununtrium 113 Uut (284) ---	Ununquadium 114 Uuq (289) ---	Ununpentium 115 Uup (288) ---	Ununhexium 116 Uuh (293) ---	Ununseptium 117 Uus (294?) ---	Ununoctium 118 Uuo (294) ---
*lanthanides			Lanthanum 57 La 138.91 1.1	Cerium 58 Ce 140.12 1.1	Praseodymium 59 Pr 140.91 1.1	Neodymium 60 Nd 144.24 1.1	Promethium 61 Pm (145) 1.1	Samarium 62 Sm 150.36 1.2	Europium 63 Eu 151.97 1.1	Gadolinium 64 Gd 157.25 1.2	Terbium 65 Tb 158.93 1.1	Dysprosium 66 Dy 162.50 1.2	Holmium 67 Ho 164.93 1.2	Erbium 68 Er 167.26 1.2	Thulium 69 Tm 168.93 1.3	Ytterbium 70 Yb 173.04 1.1		
**actinides			Actinium 89 Ac (227) 1.1	Thorium 90 Th 232.04 1.3	Protactinium 91 Pa 231.04 1.5	Uranium 92 U 238.03 1.4	Neptunium 93 Np (237) 1.4	Plutonium 94 Pu (244) 1.3	Americium 95 Am (243) 1.3	Curium 96 Cm (247) 1.3	Berkelium 97 Bk (247) 1.3	Californium 98 Cf (251) 1.3	Einsteinium 99 Es (252) 1.3	Fermium 100 Fm (257) 1.3	Mendelevium 101 Md (258) 1.3	Nobelium 102 No (259) 1.3		