CHEM 2204 - Chemical Laboratory Techniques
Laboratory Manual

CHEM 2204 Laboratory Manual Table of Contents

BCIT Chemistry Department Laboratory Safety Policy

1. Eye Protection

Appropriate, WCB approved, safety glasses must be worn in the laboratory at all times. Persons wearing prescription glasses must cover them with approved eye protection. Each student must purchase his/her own safety glasses or goggles.[1]

2. Contact Lenses

Contact lenses may not be worn in the laboratory. Students who normally wear contact lenses must inform their lab instructor. Prescription glasses and WCB approved eye protection must be worn in place of contact lenses. Each student must purchase his/her own safety glasses or goggles.[1]

3. Laboratory Coat

A lab coat must be worn in the laboratory at all times. Each student must purchase his/her own lab coat.[1]

4. Safety Equipment / Fume Hoods

The instructor will indicate the location and describe the operation of fire extinguishers, the eye wash station, and the safety shower in the laboratory. Always work in the fume hood when using volatile chemicals.

5. Disposal of Waste Materials

Separate waste containers are provided for:

a. broken glassware (container is labelled)
b. waste paper towels and other trash
c. waste chemicals and solvents (containers will be labelled)

6. Personal Hygiene

The consumption of food or beverages is not allowed in the laboratory. Long hair should be tied back. Sandals or open-toed shoes should not be worn. Wash your hands before leaving the laboratory.

7. Cell Phones / Music

Cellular phones must be turned off prior to entering the laboratory. The playing of music or the use of headphones for listening to music is not permitted.

8. Chemicals

Never use chemicals from an unlabelled container. Material safety data sheets are available in the laboratory.

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[1] Marks will be deducted if a student arrives for the lab without a lab coat and/or safety glasses /goggles.

9. Unauthorized Experiments

Do not conduct any experiments other than those you have been assigned.

10. Laboratory Conduct

No practical joking, running, pushing, or jumping is allowed.

11. Unscheduled Laboratory Periods

Under no circumstances should you work in the laboratory alone.

12. Conditions of Your Work Area

Maintain a work area that is free of books, coats, purses, chemical spills, excess chemicals, unnecessary equipment, and trash. All chemical spills should be cleaned up immediately. Consult your lab instructor if necessary.

13. Accidents

Report all accidents to your lab instructor.

Lab Report Format

1. COVER PAGE

- Experiment number and title of experiment
- Student's name and partner's name(s)
- Date of the Experiment

2. DATA

- **Original** data must be included in the lab report
- Enter data into data sheet directly **IN INK**, not on a piece of scrap paper, nor on paper towels
- Neatly tabulated (watch significant figures, units ...)
- Data should be checked and **SIGNED** by instructor before leaving laboratory

3. GRAPH (when required)

- If graphed by hand, **PROPER** graph paper must be used. (ie - Do not use papers with squares, engineering papers ...)
- The graph should nearly fill a 8-1/2" x 11" page
- Computer generated graphs are acceptable
- Each graph must contain the following information:

1. Title of experiment, Experiment #, Graph # (if more than one graph)
2. Descriptive Title of Graph (by convention "Y versus X")
3. Chemical Equation(s)
4. Label for the axes with appropriate units
5. Show: (i) the points used for slope calculations
   (ii) slope calculation on graph
   (iii) units, if any

4. CALCULATIONS - show sample calculations

- Calculations are done in the space provided on the data sheets
- Report all numbers with proper number of **SIGNIFICANT FIGURES**

Remember to sign your lab report

	Main groups												Main groups					
	1A[a] 1																	8A 18
1	1 H 1.00794	2A 2											3A 13	4A 14	5A 15	6A 16	7A 17	2 He 4.002602
2	3 Li 6.941	4 Be 9.012182											5 B 10.811	6 C 12.0107	7 N 14.0067	8 O 15.9994	9 F 18.998403	10 Ne 20.1797
3	11 Na 22.989770	12 Mg 24.3050	3B 3	4B 4	5B 5	6B 6	7B 7	8B 8	8B 9	8B 10	1B 11	2B 12	13 Al 26.981538	14 Si 28.0855	15 P 30.973761	16 S 32.065	17 Cl 35.453	18 Ar 39.948
4	19 K 39.0983	20 Ca 40.078	21 Sc 44.955910	22 Ti 47.867	23 V 50.9415	24 Cr 51.9961	25 Mn 54.938049	26 Fe 55.845	27 Co 58.933200	28 Ni 58.6934	29 Cu 63.546	30 Zn 65.39	31 Ga 69.723	32 Ge 72.64	33 As 74.92160	34 Se 78.96	35 Br 79.904	36 Kr 83.80
5	37 Rb 85.4678	38 Sr 87.62	39 Y 88.90585	40 Zr 91.224	41 Nb 92.90638	42 Mo 95.94	43 Tc [98]	44 Ru 101.07	45 Rh 102.90550	46 Pd 106.42	47 Ag 107.8682	48 Cd 112.411	49 In 114.818	50 Sn 118.710	51 Sb 121.760	52 Te 127.60	53 I 126.90447	54 Xe 131.293
6	55 Cs 132.90545	56 Ba 137.327	71 Lu 174.967	72 Hf 178.49	73 Ta 180.9479	74 W 183.84	75 Re 186.207	76 Os 190.23	77 Ir 192.217	78 Pt 195.078	79 Au 196.96655	80 Hg 200.59	81 Tl 204.3833	82 Pb 207.2	83 Bi 208.98038	84 Po [208.98]	85 At [209.99]	86 Rn [222.02]
7	87 Fr [223.02]	88 Ra [226.03]	103 Lr [262.11]	104 Rf [261.11]	105 Db [262.11]	106 Sg [266.12]	107 Bh [264.12]	108 Hs [269.13]	109 Mt [268.14]	110 [271.15]	111 [272.15]	112 [277]	113 [284]	114 [289]	115 [288]	116 [292]		

Transition metals

*Lanthanide series	57 *La 138.9055	58 Ce 140.116	59 Pr 140.90765	60 Nd 144.24	61 Pm [145]	62 Sm 150.36	63 Eu 151.964	64 Gd 157.25	65 Tb 158.92534	66 Dy 162.50	67 Ho 164.93032	68 Er 167.259	69 Tm 168.93421	70 Yb 173.04
†Actinide series	89 †Ac [227.03]	90 Th 232.0381	91 Pa 231.03588	92 U 238.02891	93 Np [237.05]	94 Pu [244.06]	95 Am [243.06]	96 Cm [247.07]	97 Bk [247.07]	98 Cf [251.08]	99 Es [252.08]	100 Fm [257.10]	101 Md [258.10]	102 No [259.10]

[a]The labels on top (1A, 2A, etc.) are common American usage. The labels below these (1, 2, etc.) are those recommended by the International Union of Pure and Applied Chemistry.

The names and symbols for elements 110 and above have not yet been decided.

Atomic weights in brackets are the masses of the longest-lived or most important isotope of radioactive elements.

Further information is available at http://www.webelements.com

The production of element 116 was reported in May 1999 by scientists at Lawrence Berkeley National Laboratory.

Techniques and Practice 1:
Weighing, Bottle-Top Dispenser, Graphing

Objectives

At the end of this lab, the student should be able to:

1. Weigh by difference
2. Weigh by taring
3. Set up and operate a bottle-top dispenser
4. Use proper weighing techniques to determine the mass of a sample of sand
5. Use proper weighing techniques to study the relationship between related variables

Reading

- Demonstration of the Eight Practical Lab Techniques
    - Technique 4: Weighing
    - Technique 5: Use of a Bottle-Top Dispenser

Part A: Weighing

1. Your instructor will demonstrate the two methods of weighing:
    - Weighing by difference
    - Weighing by taring

2. Task 1: Weigh a sample of sand that is approximately 3 – 3.2 g using the two methods of weighing.

    Note:
    The final sand samples should be placed in a small beaker.
    - Use the datasheet provided to enter your mass measurements.
    - When you are finished, return the sand to the waste bottle provided.

3. Task 2: You will be given a vial of sand. Record the label on the vial in the datasheet. Determine the mass of the sand in the vial.

Datasheet:

Task 1:

1. Weighing by taring

1. Mass of weigh boat and sample _____

2. Mass of empty weigh boat 0.0000 g

3. Mass of sample _____

2. Weighing by difference

1. Mass of weigh boat and sample _____

2. Mass of emptied weigh boat _____

3. Mass of sample transferred _____

Task 2:

Vial Label: _____

Record data for Task 2

Mass of the sand in the vial: _____

Assessment Scheme: Maximum score = 15 points

Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory; (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Technique 4 - Weighing	U	S	G	E	Comment
Knowledge of the two different methods of weighing					
Check balance is zeroed and the balance chamber is clean					
Record mass to proper number of significant figures					
Removal of weigh boat from the balance before adding sample					
Set the weigh boat on a clean surface (on Kimwipes)					
Close doors before reading mass					
Leave balance with doors closed and balance turned off					
Labeling of glassware					

Common mistakes:

- Mass of sand is *slightly* not within the specified range – S
- Clean/sweep the balance chamber while balance is 'ON' – S
- Forget to replace the cap on the reagent bottle – S
- Zero balance with door open – U
- Do not wait for the balance to stabilize before taking a reading – U
- Excess sand/compound is scooped back into the bottle – U

Part B: Use of a Bottle-Top Dispenser

1. Your instructor will demonstrate the set up of a bottle-top dispenser:
2. Your task: Set up a bottle-top dispenser to dispense a pre-determined volume.

    - Reach your hand into the box and draw a volume. Preset the bottle-top dispenser to this volume.
    - Use a graduated cylinder and measure the volume dispensed by the bottle-top dispenser. The volume collected should be within 2% of the preset volume.
    - If the volume being dispensed is outside the preset volume, adjust the dispenser and repeat.
    - Enter the preset volume and the volume dispensed in the datasheet.

Datasheet

1. Preset dispenser volume (First try) _____

2. Volume dispensed by the bottle-top dispenser Trial 1: _____

Trial 2: _____

Trial 3: _____

1. Preset dispenser volume (Second try) _____

2. Volume dispensed by the bottle-top dispenser Trial 1: _____

Trial 2: _____

Trial 3: _____

Assessment Scheme: Maximum score = 15 points

Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Technique 5 – Bottle-Top Dispenser	U	S	G	E	Comment
Knowledge of keywords: bottle-top dispenser, accuracy of a bottle-top dispenser					
Ability to set the dispenser to the approximate volume specified					
Ensure the dispenser is free of air bubbles					
Use graduated cylinder to check preset volume					
Proper use of dispenser pump					
Record volume to proper number of significant figures.					

Common mistakes:

- Dispense into beaker then transfer to the graduated cylinder – S
- Lift graduated cylinder off the surface to take a reading – S
- Press on the plunger while dispensing – S

Part C – Graph the relationship between the volume of water and its mass

1. Weigh a 10 mL graduated cylinder on the balance.
2. Use a Pasteur pipette and place 1 mL of water in the graduated cylinder. Record the mass of the water and graduated cylinder.
3. Add water, 1 mL at a time, until you have added 10 mL. Record the mass of the graduated cylinder and water for each milliliter of water added.

Datasheet:

Volume of water (mL)	**Mass of graduated cylinder and water (g)**	**Mass of water (g)**

Analysis of data:

1. Plot the ordered pairs of data on a graph. Always plot the independent variable on the horizontal, x-axis, and the dependent variable on the vertical, y-axis. You may generate the graph by hand or with Excel.
   Note:
   - Use the whole sheet.
   - Label the axes.
   - Give a title to your graph. (“Y versus X”)
   - Draw the best straight line through the points. Do not connect the dots.
   - Determine the equation of the line. (i.e. $y = mx + b$)
   - Indicate the room temperature

Hand in Part C datasheet and the graph the following week. On the graph, indicate:

1. What is the average increase in mass per milliliter of water?

2. Predict the mass of 100 mL of water.

Experiment 1: Moisture and Ashing **(**View photos of equipment on website**)**

Part A Introduction | Part B Introduction | Part A Procedure | Part B Procedure | Part A Data Sheet | Part B Data Sheet

Objectives:

The student should be able to carry out moisture determinations and analysis including calculations on wet, air-dried and oven-dried basis. Analysis of the ash content of coal using a muffle furnace will also be carried out. in addition, the student should be able to assess the different types of moisture present, and be able to operate different types of moisture removal (drying) apparatus.

Part A - Determination of Moisture Content

Introduction

Because the amount of moisture present will vary with the temperature, humidity and particle size, it is important for the analyst to know the moisture content of the sample in question. Such a determination is usually done and quoted in the following ways:

Oven dried basis (OD basis) - none, or very little percent moisture in the sample.

Air dried basis (air dried basis) - low percent moisture in the sample.

As received or wet basis (wet basis) - percentage moisture can go up to 100% or higher.

Stable, hygroscopic substances are usually heated for 1-3 hours at 100-110^{o}C to remove surface moisture. This standardizes the moisture conditions of the sample, which is then said to be analyzed on an **oven dried basis (OD weight)** as "in" wood products. Substances which are not appreciably hygroscopic may be **air dried** (metals, alloys, many ores and minerals, animal or vegetable tissue).
Analytical results are then reported on an air dried basis, where free water has evaporated and an equilibrium is attained. Substances which contain water of constitution (chemically bound) such as potassium hydrogen sulfate or water of crystallization such as calcium chloride dihydrate cannot usually be dried by heating, nor can substances which undergo oxidation or decomposition when heated. These must be dried in other ways. One such way is to use a **dessicator** (a device for storing chemicals in contact with a drying agent such as a salt hydrate, a substance of definite known vapour pressure). For example, borax if dried over hydrated sodium bromide will contain 10 molecules of water of crystallization, whereas if air dried it will partially decompose to form the pentahydrate. Desiccators are also used as a place of storage for dried materials until the analyst is ready to use them further.

Wood is a hygroscopic substance and therefore always contains water (except under unusual conditions). In fact, the affinity of wood for water is so great that it is never absolutely dry unless subjected to desiccation so drastic (may take weeks) that it is **chemically** changed.

Analysis reports on **wood** and wood products are calculated on an **oven dry basis**. The standard dryness for wood is its oven dry weight, which is arbitrary as tests have shown that it still contains approximately 1% of water after it has been subjected to oven drying at a temperature of 100 - 105^{o} C for a length of time, **where no further loss in weight has been recorded by repeated weighings**. (In arriving at the OD weight, the wood should remain in the drying oven **slightly longer** (approximately 1/2 hour) than necessary to ensure weight consistency; prolonged drying may result in the loss of volatilizable components, in addition to the water that is still present). TAPPI (Technical Association of the Pulp and Paper Industry) methods call for 3-4 hours drying with the proper sampling technique at 100 - 110^{o} C (see

TAPPI-T11 and T608). The amount of water contained in a given sample of wood can be determined if its initial moisture and its dry weight are known. (The unit of measurement is irrelevant, provided that it is the same in each instance). Consequently the following relationship holds

Weight of wood at initial moisture = OD weight of wood + Weight of water present

It is customary to perform analysis on the initial sample (moist) and to determine separately the moisture on another portion of the original sample. Both results are then used to compute the analysis on an OD basis.

Since the weight of the included water is expressed as a percentage, symbols can be used and the equation expressed as:

$$M_{(\text{wet basis})} = \frac{100\ (W - D)}{W}$$

where
W = the weight of initial sample (moisture and solids)
D = the oven dry weight
$M_{(\text{wet basis})}$ = the percentage moisture in the sample based on oven dry weight

If the moisture on an air-dried basis is required then the following equation may be used

$$M_{(\text{air dried})} = \frac{100\ (W - A)}{W}$$

where
A = the air-dried weight
$M_{(\text{air dried})}$ = the percentage moisture in the sample based on air-dried weight

In **wood analysis (and wood products)**, it is customary to express the percentage moisture on the oven dry weight instead of the wet weight (see TAPPI); therefore, the formula for the percent moisture is:

$$M_{(\text{OD basis})} = \frac{100\ (W - D)}{D}$$

Example:

A wood sample is cut from a green board and its weight when green is found to be 25 grams. It is dried in a constant temperature oven at 100^{o} C until repeated weighings show no further loss in weight. The constant weight (OD weight) is 16.4 grams. What is the moisture content?

$$M_{(\text{OD basis})} = \frac{100\ (25 - 16.4)}{16.4} = 52.4\ \%\ (\text{OD basis wood})$$

This sample of wood was then analyzed for iron, which was determined gravimetrically and found to be 1.25 mg. What is the % Fe?

$$\%Fe = \frac{100 \times 1.25\ \text{mg}}{16.4\ \text{g} \times 1000\ \text{mg/g}} = 0.00762\ \%\ (\text{OD basis})$$

Experiment 1: Moisture and Ashing

Part A Introduction | Part B Introduction | Part A Procedure | Part B Procedure | Part A Data Sheet | Part B Data Sheet

Part B - Determination of Percent Ash

Introduction

The percentage of ash in a material such as a coal or pulp sample can be determined by heating a portion of the sample up to around 800-900^{o} C for a period in excess of 1 hour. The percent ash is then calculated from

$$\% \text{ ash} = \frac{\text{Weight of residue}}{\text{Weight of starting material}} \times 100\%$$

The importance of determining the ash content of a material cannot be underestimated. Materials for which ASTM (American Society for Testing and Materials) methods detailing the determination of ash content are listed include coal, wood, paper, coal tar, engine coolants, petroleum products, adhesives, thermoplastics, fats/oils and leather. For some of these materials the % ash measurement can be regarded strictly as a quality control measurement while for some of these methods the level of % ash may have a more theoretical meaning. For example, the level of % ash in engine coolants is directly related to the level of inorganic inhibitors present in the coolant.

In a subsequent laboratory exercise involving the determination of Ca based on the formation of $BaSO_{4,}$the final precipitate collected on filter paper is heated at a very high temperature in a muffle furnace. During this process the filter paper is heated sufficiently to burn the paper completely and produce CO_2 (g) and H_2O (g) alone. For this and similar analytical procedures, the filter paper used is of the **ASHLESS** type. This refers to the fact that on burning this particular type of filter paper the amount of residue left by the paper is very low, thus not influencing the weight of the precipitate significantly.

Equipment Used for Drying

Dessicator

- Used for cooling and drying samples, storing pure samples and tared crucibles
- Contains a desiccant - a hygroscopic material such as $CaSO_4$ (Drierite) or CaO that removes moisture
- Do not put a very hot vessel in a dessicator

Vacuum Dessicator

- Dessicator is connected to a vacuum so that the content is subjected to a vacuum
- Provides additional moisture removal
- Removal of organic solvents from a sample

Vacuum Oven

- A vacuum pump draws air through drying towers containing H_2SO_4 and a moisture trap that ensures that the air is dry and clean. From here it enters the vacuum oven which is used to dry samples. The constant airflow helps to remove solvents from the sample.
- The reduced pressure in the oven lowers the temperatures at which solvents are removed and thus helps to minimize the amount of heat used.

Freeze-dryer

- Used for heat-sensitive materials, (coffee, tissue samples) and also to dehydrate materials prior to storage.
    1. Place the sample in the chamber
    2. Freeze the sample and reduce the pressure of the system to below 4.58 torr. Below this pressure water cannot exist as a liquid.
    3. Slowly raise the temperature, which causes the frozen water to sublime (solid $\longrightarrow$ vapour). The water is driven off leaving the sample dehydrated.

IR Moisture Balance

- A simple method for determining the moisture content of a liquid (eg - black liquor).
    1. Set the scale on the balance to 100% moisture (zero gram) with the Al dish in place.
    2. Weigh 10 gram of the sample onto the dish. The balance reads 0% moisture (10 g).
    3. Position the IR source over the sample.
    4. Set the desired power and time of illumination. The % moisture will be displayed at the end of this time.
- Time of illumination, power and height of the IR source above the sample all affect the process. These parameters should be optimized prior to the analysis or suitable values found from the standard methods.

Drying Oven

- Electrical heating coils inside a metallic frame used for heating and drying of samples and equipment.
- Temperatures of 100 - 150^{o} C are used.
- Large sample capacity
- Temperature readings not very accurate

Muffle Furnace

- A brick lined unit using an electrical heating coil system.
- Very high temperatures of operation (> 1000^{o} C).
- Used to ash samples or carry out high temperature reactions such as fusions.
- Leave the door open initially to prevent pressure build-up in furnace.
- Always practice extreme safety/care when using.

Experiment 1: Moisture and Ashing

Part A Introduction | Part B Introduction | Part A Procedure | Part B Procedure | Part A Data Sheet | Part B Data Sheet

Part A - Determine the Moisture Content

Procedure

1. Desiccators are to be used at several times during this lab. Before using, ensure that your desiccator contains fresh or usable desiccant.
2. Carefully remove two moisture dishes with lids (record reference numbers) from the oven using suitable tongs. Place the dishes and lids in the desiccator. **DO NOT MIX UP THE LIDS.**
3. When the dishes are at room temperature (after about 15 minutes), weigh the moisture dishes and lids. Add about 2 grams of sawdust to the dishes and then reweigh the dishes and lids. Place the dishes in the oven for 2 hours at 110^{o} C. At the end of this time carefully remove the dishes using tongs and place them in the desiccator. After cooling to room temperature reweigh the dishes.

Treatment of Data

1. Calculate the % moisture on a wet basis and the % moisture on an oven dried basis.(Read Part A Introduction for theoretical details).
2. In the space provided on the data sheet, show your calculations for both samples and calculate the average value for the sample.

Part B - Ash Determination

Procedure

1. Prior to this lab, the technician will have heated 2 porcelain crucibles per student in the furnace to constant weight at a temperature of 1000º C. On removal from the furnace the crucibles are allowed to cool partially in air for 1 minute prior to be placed in desiccators. In addition, the furnace temperature is reduced to 500º C.
2. Carefully weigh each empty porcelain crucible and ensure that each crucible is uniquely identified.
3. Add around 0.5 g of dry coal sample to each and accurately reweigh.
4. Each crucible will be placed in the muffle furnace using the procedure demonstrated by your instructor. Raise the furnace temperature to 800º C.
5. Heat the coal sample until only a grey ash remains. This will typically take around 75 to 90 minutes.
6. Once the sample has been fully ashed, carefully remove the crucibles from the furnace using the procedure demonstrated by your instructor.
7. Cool them partially in air and then place them in a desiccator.
8. Cool to room temperature and reweigh the crucibles.

Treatment of Data

1. Calculate the % ash in the coal sample. (Read Part B Introduction for theoretical details).
2. In the space provided on the data sheet, show your calculations for both samples and calculate the average value for the sample.

Experiment 1: Moisture and Ashing

Part A Introduction | Part B Introduction | Part A Procedure | Part B Procedure | Part A Data Sheet | Part B Data Sheet

Part A - Determine the Moisture Content

Data Sheet

	Dish # _____	Dish # _____
1. Weight of dish	_____	_____
2. Weight of wet sawdust	_____	_____
3. Weight of dish + dry sawdust	_____	_____
4. Weight of dry sawdust (item 3 - item 1)	_____	_____

Calculation:

% Moisture (Wet Basis)

Results: **Trial 1: _____Trial 2: _____**

Average = _____

% Moisture (Oven dry)

Results: **Trial 1: _____Trial 2: _____**

Average = _____

Part B - Ash Determination

Data Sheet

	Crucible # _____	Crucible # _____
1. Weight of crucible	_____	_____
2. Weight of coal	_____	_____
3. Weight of crucible + ash	_____	_____
4. Weight of ash (item 3 - item 1)	_____	_____

Calculation:

% Ash

Results: **Trial 1: _____Trial 2: _____**

Average = _____

Techniques and Practice 2:
Rotary Evaporator, Precipitation, Gravity Filtration, Graphing

Objectives

At the end of this lab, the student should be able to:

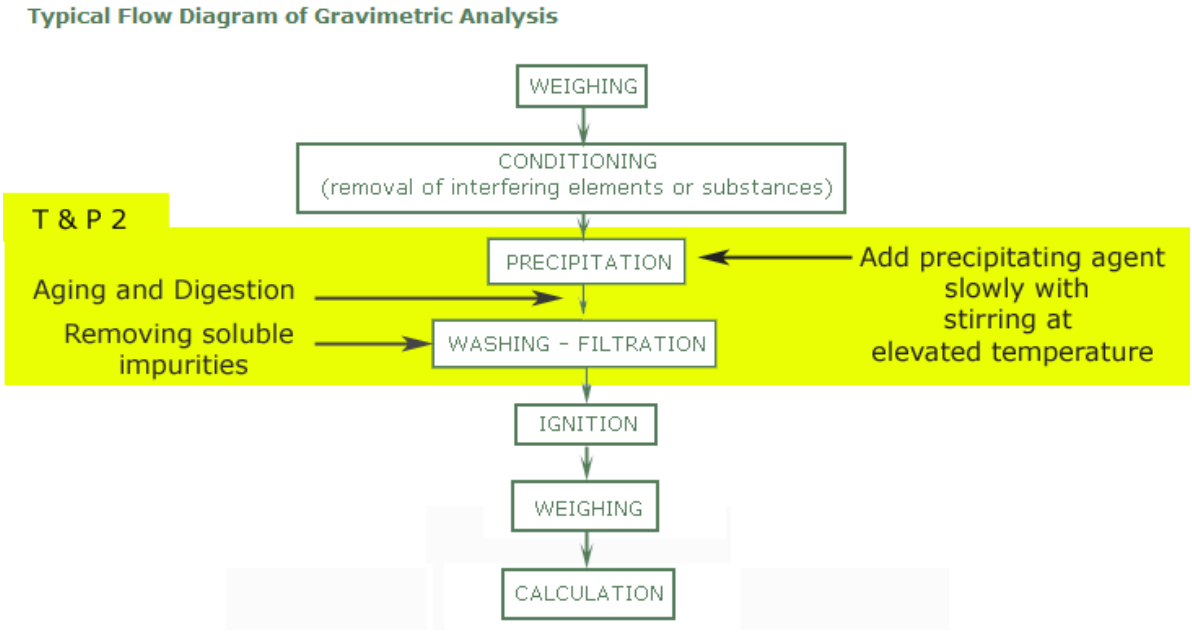
1. Use proper precipitation techniques to precipitate a sample of $BaSO_4$.
2. Set up the apparatus for gravity filtration.
3. Operate the rotary evaporator.
4. Use graphing techniques to determine a physical property of water.

Reading

- Demonstration of the Eight Practical Lab Techniques
  - Technique 6: Gravity Filtration

Part A: Precipitation and Gravity Filtration

Precipitation and gravity filtration are steps in the flow diagram of gravimetric analysis. In this lab, we will focus on the Precipitation and the Washing – Filtration steps. Your instructor will demonstrate these steps.



Task 1: Precipitate a Sulfur containing compound as $BaSO_4$

1. Heat 200 mL of distilled water in a beaker. This is wash water for use in step 9.

2. Assemble the gravity filtration apparatus and select a piece of fast flow filter paper for the funnel provided.

3. Dispense 25.0 mL sample and place in a 600 mL beaker.

4. Add 10 drops of 12 N HCl to the sample and dilute to approximately 225 mL. Cover with a speedy vap watch glass.

Precipitation step:

5. Heat the solution to almost boiling. Keep the beaker on low heat and, while stirring with the glass end of the rubber policeman, add 15 mL 5% $BaCl_2$ solution. This should take 10 – 15 minutes.

Aging step:

6. Leave the rubber policeman in the beaker, cover the solution with a speedy vap watch glass and keep the solution to almost boiling for 10 minutes.

7. Let cool and allow the precipitate to settle.

Washing and Filtration:

8. Filter by decantation through a fast flow filter paper.

9. Wash the precipitate 2 to 3 times with 5 mL portions of hot distilled water. Do not over wash. (Solubility of $BaSO_4$ is 3 mg/L at 20^oC.)

10. Collect some of the filtrate of the third washing with a small beaker. Test the filtrate for chloride by acidifying the filtrate with dilute HNO_3, followed by 1 to 2 drops of $AgNO_3$. If a white precipitate forms or if the filtrate becomes white and cloudy, then the test for chloride is positive. Clean the collection flask and repeat step 9.

11. Repeat the test for chloride. If the chloride test results with the filtrate faintly milky or clear, then you may stop washing the precipitate.

12. Transfer the precipitate quantitatively to the funnel.

Assessment Scheme: Maximum score = 15 points

Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory; (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Technique 6 – Gravity Filtration	U	S	G	E	Comment
Knowledge of keywords: name different types of filter papers, decantation, washing, purpose of tearing a small corner of the filter paper, filtrate					
Selection of proper filter paper for the funnel provided					
Folding of filter paper					
Apparatus setup – height of filter rack, position of the funnel and the beaker					
Wetting of the filter paper – should fit snugly into the funnel, should have good flow					
Decant technique					
Wash technique					
Transfer technique – proper use of rubber policeman and rinsing					

Common mistakes:

- Washing with a constant stream of water - S
- Place filter paper on bench to fold - S
- Decanting - Does not direct rod over thick part of the filter paper - G
- Does not use the rubber part of the rubber policeman during scraping and transferring – S
- Place rubber policeman down without rinsing - U

Part B: Rotary Evaporator

A sample of sand which has been contaminated with oil was collected. A solid-liquid extraction analysis procedure was carried out, where the contaminated sand (solid) was left stirring in dichloromethane (liquid), the extracting solvent, for several hours. Since oil is a nonpolar substance, over time, the oil in the sand will be extracted into the dicholoromethane layer. You will be given a sample of this sand and dichlormethane mixture. Use the rotary evaporator to remove the dichloromethane from the extracted oil.

The rotary evaporator is an apparatus that is designed to distill a liquid under reduced pressure. At the lower pressure, the liquid can be distilled at a lower temperature than it would at atmospheric pressure. This is a safe and fast method for distilling a flammable solvent like dichloromethane. Dichloromethane boils at around 40^{o}C at 1 atm.

Your instructor will demonstrate how to operate the rotary evaporator.

Task 2: Recover the oil extracted from contaminated sand

DISPOSAL OF SOLVENT:

- **Dispose all waste dichloromethane into the chlorinated waste container.**
- **Dispose all waste acetone into the non-chlorinated waste container.**

1. You will be given a sample of contaminated sand and dichloromethane mixture in a small beaker. The sand has been stirring in the dichloromethane for several hours. Set up the filtration apparatus in the fume hood and filter the sample by gravity filtration using a fast flow filter paper.

2. Wet down the filter paper with a small amount of dichloromethane. **Do NOT use water!** [Note: Dichloromethane evaporates quickly. In order to keep the filter paper in place, start the filtration quickly.] Collect the dichloromethane in a round bottom flask.

3. Begin filtering by pouring and scraping the contaminated sand mixture into the funnel with a rubber policeman. Collect the filtrate in a 100 mL round bottom flask.

4. Rinse the beaker with two 5 mL portions of dichloromethane. Rinse the filter paper with two 5 mL portions of dichloromethane

   NOTE: After all the washings, the volume of solvent in the round bottom flask should not be more than one-third full.

5. Remove the dichloromethane solvent using a rotary evaporator. What remains in the round bottom flask is the extracted oil.

To clean up, pour some dichloromethane into the round bottom flask to dissolve the oil. Discard the dichloromethane into the chlorinated waste container. Use acetone to rinse the round bottom flask. The waste acetone should be disposed in the non-chlorinated waste container.

Assessment Scheme: Maximum score = 15 points

Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory; (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Technique 9 – Rotary Evaporator	U	S	G	E	Comment
Knowledge of the purpose of using the equipment					
Use of the circular clip in the correct orientation					
Close the air inlet valve					
Water to the aspirator on full strength					
Water to the condenser enough for circulation					
Assembly of the rotary evaporator					
Disassembly of the rotary evaporator					

Part C: Graphing – Determine the Heat of Vapourization of Water

The purpose of graphing is to examine the relationship between variables. Often a theory predicts how the two variables are related, and a graph of the experimental data can confirm or disprove the theory. A set of data was collected for the boiling point of water at various pressures.

T(°C)	P(kPa)
0	0.612
10	1.227
20	2.536
30	4.242
40	7.37
50	12.33
60	19.9
70	31.15
75.7	46.12
89.7	70.1
100	101.32
120	198.5
200	1554.3

The relationship that describes the relationship between pressure and temperature is given by the equation,

$$\ln P = -\frac{\Delta H_{vap}}{R}\frac{1}{T} + C$$

where

- P is pressure (kPascal),
- R is the gas constant (8.314 joules/moleK),
- T is temperature (Kelvin), and
- *C* is a constant.

The quantity of interest is ΔH_{vap}. It is known as the heat of vapourization. In this case, this is the energy required to convert a mole of liquid water into its vapour.

Recognize that the above equation is in the form of a straight line, $y=mx + b$, where

y	ln P
m	$-\frac{\Delta H_{vap}}{R}$
x	$\frac{1}{T}$
b	*C*

Plot a graph using the data given and determine the ΔH_{vap} of water. Be sure to:

1. Title the graph "Y vs. X for the Vapourization of Water".
2. Label the axes.
   Note: ln P does not have units. The axis should be labeled "ln P (P in kPa)".
   It is important to show the unit of pressure.
3. Fit the data with a trendline. Add the equation of the trendline and the R^2 value on the graph.
4. On the graph, write the slope = _____ unit.
5. Show your work on the graph and determine the ΔH_{vap} of water.

Experiment 2: Determination of Calcium by Gravimetric Analysis

Introduction | Flow diagram of Gravimetric Analysis | Procedure | Data Sheet #1 | Data Sheet #2 | Data Sheet #3 | Data Sheet #4

Objectives:

The student should be able to master all the fundamental operations of gravimetric analysis:

- from **sample preparation** (involving weighing, conditioning - removal or masking of interfering materials),
- through **separation** (involving precipitation, digestion, washing, isolation and ignition),
- to **measurement** (usually by weighing), and
- **calculation** (by obtaining the desired result by the use of gravimetric factors).

The student will adopt and use recognized quantitative techniques inherent in these procedures as applied to the analysis of 'natural' samples, namely **calcium sulfate** in sulfite cook acid.

Introduction

Raw cook acid is produced by passing water or liquor together with SO_2 over a bed of limestone ($CaCO_3$) and providing heat.

$$CaCO_3\,(s) + 2\,H_2O\,(l) + 3\,SO_2\,(g) \longrightarrow H_2SO_3\,(aq) + Ca(HSO_3)_2\,(aq) + CO_2\,(g)$$

This liquor is then used in digestors to separate out the lignin, salts, sugars etc. from the cellulose contained in wood chips.

After reaction, the "cook acid" contains chemicals that have been used and are left over. These chemicals may be recovered via a furnace and then mixed up with make up chemicals to produce more fresh liquor. During the furnace operation the unwanted lignin, sugars etc. are volatilized. In addition, digestor scale, which consists of $CaSO_4$, $CaSO_4 \cdot 5\,H_2O$ and sulfites etc. is formed. To remove this scale, the steam/chemical channels are switched such that the steam now dissolves the digestor scale giving ions such as Ca^{2+}, SO_4^{2}, HSO_3^{-}, SO_3^{2-} and the gas SO_2. This liquor can be reused but it is important to know the composition of the liquor as the wood chip digestion chemistry is greatly affected by the composition of the liquor. In addition, digestor scale can build up in the furnace leading to a clogging of pipes and potential pressure build-up.

A sample of cook acid is to be analyzed gravimetrically for the amount of Ca in the sample, which can also be related to the amount of SO_4^{2-} and S. Initially the sample is conditioned by the addition of conc. HCl that destroys any sulfites present in the sample.

$$HCl\,(aq) + HSO_3^{-}\,(aq) \longrightarrow SO_2\,(g) + H_2O\,(l) + Cl^{-}\,(aq)$$

$$2\,HCl\,(aq) + SO_3^{2-}\,(aq) \longrightarrow SO_2\,(g) + H_2O\,(l) + 2\,Cl^{-}\,(aq)$$

Any Ca^{2+} (and SO_4^{2-}) present in the sample is unaffected by the addition of HCl. Precipitation is carried out by the addition of $BaCl_2$ to the sample.

$$Ca^{2+}\ (aq) + SO_4^{2-}\ (aq) + Ba^{2+}\ (aq)\ (\text{slightly excess}) \longrightarrow BaSO_4\ (s) + Ca^{2+}\ (aq)$$

The valid assumption made is that $Ca^{2+} = SO_4^{2-}$. After filtering, the $BaSO_4$ (s) is collected by igniting the filter paper in a furnace at 800°C. The filter paper (represented by the formula $(C_6H_{10}O_5)_n$) is converted into CO_2 and water provided that sufficient oxygen is present.

$$(C_6H_{10}O_5)_n\ (s) + O_2\ (g) \longrightarrow CO_2\ (g) + H_2O\ (l)$$

Although $BaSO_4$ (s) is stable up to 1400° C, it may be reduced to BaS at temperatures above 600° C by carbon from the filter paper

$$BaSO_4\ (s) + 4\ C\ (s) \longrightarrow BaS\ (s) + 4\ CO\ (g)$$

This reaction is avoided by charring the filter paper and then burning off slowly at a low temperature with an excess of air. Final ignition is done at 600 - 800° C.

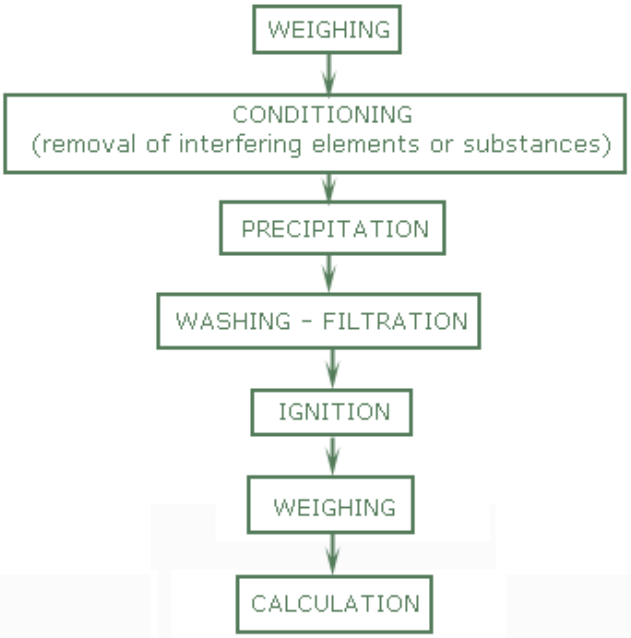
Final weighing of the sample is as $BaSO_4$ (s). From the weight of $BaSO_4$, the %S, $\%SO_4^{2-}$, and % Ca may be calculated using the appropriate gravimetric factors.

This sample is a liquid sample and thus is measured as a volume initially. In order to convert the volume of sample to the mass of sample, the density (or specific gravity) of the sample is used. The specific gravity of the sample will be measured during the lab using a hydrometer.

Experiment 2: Determination of Calcium by Gravimetric Analysis

Introduction | Flow diagram of Gravimetric Analysis | Procedure | Data Sheet #1 | Data Sheet #2 | Data Sheet #3 | Data Sheet #4

Typical Flow Diagram of Gravimetric Analysis



Experiment 2: Determination of Calcium by Gravimetric Analysis

Procedure

WEEK 1:

1. Carefully remove **two** porcelain crucibles from the muffle furnace (preheated to constant weight at 1000º C by your instructor. Cool partially on asbestos. Reduce furnace to 500º C. Transfer the crucibles to the desiccator and let cool to room temperature before weighing the empty crucibles.

THE FOLLOWING PROCEDURE APPLIES TO DUPLICATE SAMPLES - ENSURE BEAKERS ARE PROPERLY LABELLED

2. In the fumehood, add approximately 50 mL distilled water in a 400 mL beaker. Add 100 mL liquid sample using a graduated cylinder.

   Density of the sample measured is = 1.0112 g/mL

3. Carefully add 15 mL conc. HCl. Cover with a speedyvap watch glass. Boil gently for 1/2 hour to drive off the SO_2.
4. With a pasteur pipette, slowly add 25 mL of 10% $BaCl_2$. Boil gently for 10 minutes. Let cool and settle.
5. Filter by decantation through an ashless filter paper (Whatman #40).
6. Transfer the precipitate quantitatively. Wash the precipitate 2 to 3 times with 5 mL portions of distilled water. **Do not over wash.**
7. Test filtrate for chloride by acidifying the filtrate with dilute HNO_3, followed by 2 to 3 drops of $AgNO_3$.
8. Place the folded filter paper in the empty preweighed crucible. Put everything in a covered beaker. Label and store for next week.

WEEK 2:

9. Place the crucible carefully in the muffle funace at 500º C with excess O_2 (g) for approximately 15 minutes to char the paper. Leave the door partially open.
10. Close the furnace door. Raise the temperature to 750 - 800º C for approximately 30 minutes. The total time to complete steps 9 and 10 is approximately 45 minutes.
11. Remove the crucible carefully. Cool partially on asbestos. Transfer the crucible to the desiccator and let cool to room temperature.
12. Weigh the crucible and residue.

Treatment of Data

1. Calculate the % S, % SO_4^{2-} and % $CaSO_4$ on a w/w basis, knowing that for one equivalent of $BaSO_4$ produced, one equivalent of $CaSO_4$ is used.

   $$Ba^{2+}\ (aq) + CaSO_4\ (s) \longrightarrow BaSO_4\ (s) + Ca^{2+}\ (aq)$$

Experiment 2: Determination of Calcium by Gravimetric Analysis

Introduction | Flow diagram of Gravimetric Analysis | Procedure | Data Sheet #1 | Data Sheet #2 | Data Sheet #3 | Data Sheet #4

Data Sheet

	Crucible # _____	Crucible # _____
1. Weight of empty crucible	_____	_____
2. Weight of crucible + residue	_____	_____
3. Weight of $BaSO_4$ (s) (item 2 - item 1)	_____	_____
4. specific gravity of the cook acid sample	_____	_____
5. Weight of cook acid sample	_____	_____

Observation of filtrate:

Record Observations of:	**Filtrate**
appearance of filtrate	
after addition of 2 to 3 drops of HNO_3 solution	
after addition of 2-3 drops of $AgNO_3$ solution	

Experiment 2: Determination of Calcium by Gravimetric Analysis

Introduction | Flow diagram of Gravimetric Analysis | Procedure | Data Sheet #1 | Data Sheet #2 | Data Sheet #3 | Data Sheet #4

Calculation of % w/w S :

Gravimetric Factor for sulfur (show calculation and units):
_____

Calculation of % S: _____

% w/w S = _____ **% w/w S =_____**

Average = _____

Experiment 2: Determination of Calcium by Gravimetric Analysis

Introduction | Flow diagram of Gravimetric Analysis | Procedure | Data Sheet #1 | Data Sheet #2 | Data Sheet #3 | Data Sheet #4

Calculation of % w/w SO_4^{2-}:

Gravimetric Factor for SO_4^{2-} (show calculation and units):
_____

Calculation of % SO_4^{2-}: _____

Results: **% w/w SO_4^{2-} =** _____ **% w/w SO_4^{2-} =** _____

Average = _____

Experiment 2: Determination of Calcium by Gravimetric Analysis

Introduction | Flow diagram of Gravimetric Analysis | Procedure | Data Sheet #1 | Data Sheet #2 | Data Sheet #3 | Data Sheet #4

Calculation of % w/w $CaSO_4$:

Gravimetric Factor for calcium sulfate (show calculation and units):_____

Calculation of % $CaSO_4$: _____

Results: **% w/w $CaSO_4$ =** _____ **% w/w $CaSO_4$=**_____

Average = _____

Techniques and Practice 3:
Volumetric flask, Pipette, Standard solution, Dilution, Graphing

Objectives

At the end of this lab, the student should be able to:

1. Recognize basic volumetric glassware and lab equipment and know how to select the appropriate glassware for the tasks at hand.
2. Describe glassware classifications, specifications and tolerances of volumetric flask, pipettes and burettes.
3. Describe the terms 'To Contain' and 'To Deliver'.
4. Describe the factors affecting the accuracy of volumetric glassware.
5. Explain the concept of a primary standard and its properties.
6. Demonstrate the correct manner of using a variety of pipette bulbs (single bulb).
7. Use proper transfer techniques with measuring pipettes and volumetric pipettes.
8. Choose a primary standards and preparing primary standard solutions from solid reagents and by dilution.
9. Prepare a set of standard solutions by dilution.

Reading

- Demonstration of the Eight Practical Lab Techniques

  Technique #2 – Use of a Pipette

  Technique #3 – Use of a Volumetric Flask

  Technique #7 - Preparation of a Standard Solution

COMPLETE ALL THE CALCULATIONS AND FILL IN THE HIGHLIGHTED FIELDS ON PAGE TP3-8, 9 AND 11 BEFORE COMING TO THE LAB.

YOU WANT TO MAXIMIZE PRACTICE TIME ON THE GLASSWARE.

Introduction

Volumetric laboratory glassware includes volumetric flasks, pipettes and burettes. They are used for the precise measurement of volume. Volumetric equipment is marked with letters for the type of calibration (usually TD for "to deliver" or TC for "to contain") and the calibration temperature. Class A volumetric glassware provides the highest accuracy. Often ambient laboratory conditions are not identical to standard conditions; therefore, the volumes dispensed or contained in volumetric glassware are not exactly the same as the manufacturer's specifications, but within some acceptable limits or tolerance determined by the manufacturer. The tolerance of a Class A 100.00 mL volumetric flask is ±0.08 mL. This is accurate to 8 parts per 10, 000 or ±0.08%. The tolerance of a 50 mL Class A buret is in the order of 0.1%. This slight variation in volume will introduce a systematic error when solutions are made using these volumetric glassware.

Dilution

We define a ***stock solution*** as a concentrated solution from which we can dilute to some lower concentration for actual use. The solution that we end up using to prepare calibration standards is the ***working solution***. Dilution is the process of preparing a set of calibration standard solutions.

For example, if we need to create 5 standard solutions of the following concentrations:

- 1.00×10^{-4} M $NaNO_3$
- 3.00×10^{-4} M $NaNO_3$
- 5.00×10^{-4} M $NaNO_3$
- 7.00×10^{-4} M $NaNO_3$
- 1.00×10^{-3} M $NaNO_3$

we can start from a working solution of 1.00×10^{-2} M $NaNO_3$ which is made from a 1.000 M $NaNO_3$ stock solution.

The calculations involved when determining how to prepare your dilution is

$$C_i V_i = C_f V_f \qquad \text{Equation (1)}$$

where

C_i = concentration of the stock solution
V_i = volume of the stock solution
C_f = final concentration of the dilute solution
V_f = final volume of the dilute solution

Using Equation (1), one viable method to carry out the dilutions would be:

Prepare the working solution: 1.00×10^{-2} M $NaNO_3$

- 5.00 mL of the 1.000 M $NaNO_3$ stock solution into a 500-mL volumetric flask, and fill to the mark.

Prepare the 1.00×10^{-4} M $NaNO_3$

- 1.00 mL of 1.00×10^{-2} M $NaNO_3$ into a 100-mL volumetric flask, and fill to the mark.

Prepare the 3.00×10^{-4} M $NaNO_3$

- 3.00 mL of 1.00×10^{-2} M $NaNO_3$ into a 100-mL volumetric flask, and fill to the mark.

Prepare the 5.00×10^{-4} M $NaNO_3$

- 5.00 mL of 1.00×10^{-2} M $NaNO_3$ into a 100-mL volumetric flask, and fill to the mark.

Prepare the 7.00×10^{-4} M $NaNO_3$

- 7.00 mL of 1.00×10^{-2} M $NaNO_3$ into a 100-mL volumetric flask, and fill to the mark.

Prepare the 1.00×10^{-3} M $NaNO_3$

- 10.00 mL of 1.00×10^{-2} M $NaNO_3$ into a 100-mL volumetric flask, and fill to the mark.

Part A: Use of a Pipette

Task:

1. Choose a volumetric pipette from the selection of volumetric pipettes available.
2. Determine the volume that the volumetric pipette can hold.
3. Using the pipette chosen, transfer the volume of a solution provided into an Erlenmeyer flask.
4. Enter the volume of the solution that has been transferred into the Erlenmeyer flask in the datasheet.

Datasheet:

Choose from the following volumes and transfer **TWO** volumes. Record the transferred volumes.

1 mL: _____

3 mL: _____

5 mL: _____

10 mL: _____

20 mL: _____

50 mL: _____

Assessment Scheme: Maximum score = 15 points
Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory; (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Use of Volumetric Pipette	U	S	G	E	Comment
Knowledge of keywords: acclimatization, meniscus, volumetric pipette, measuring pipette					
Use of pipette bulb					
Acclimatize technique					
Wiping the outside of the pipette before adjusting to the mark					
Control of pipette while adjusting the liquid level to the mark – Bring to eye level					
Dispensing technique – draining with tip touching wall of containing and waiting for 10 seconds					

Common mistakes:

- Bring solution to mark then wipe pipette tip – G
- Drain the pipette with tip touching the side of receiving vessel:
  for 5 sec or less – U
  between 5 to 8 sec – S
  between 8-9 sec – G
  for 10 sec – E
- Does not touch tip to the side of the receiving vessel for 10 sec – U
- Do not replace cap on reagent bottle – S
- Drain with tip of pipette immersed in solution – U
- Level of solution fall below or over mark – S
- Level of solution at the mark but there is a drop that hangs outside the tip of the pipette. Does not remove the hanging drop. – S
- Use paper towel to wipe – S
- Dispense into a beaker too small – S
- Use thumb instead of index finger to control the solution – S
- Use incorrect size of beaker – S

- Record to improper significant figures – U
- Pipette from reagent bottle – U

Acclimatization:

- Acclimatize once – S
- Acclimatize twice – G
- Contamination - Solution level goes up and down back into the beaker – S
- Pipette from contaminated solution – U
- Not using enough solution in acclimatization. Solution should be drawn to at least 1/3 in the bulb – S

Part B: Use of a Volumetric Flask

Solutions are often prepared by dilutions of a stock solution. The concentration of the stock solution, C_i, is given on the reagent bottle. The dilution equation is given in Equation (1). The dilution factor, D is

$$D = C_i / C_f . \qquad \text{Equation (2)}$$

where C_i is the initial concentration and C_f is the final concentration of the solution.

Example: Prepare a 100.0 mL of a solution by diluting the stock solution provided by a dilution factor of 30. The concentration of the stock solution is 2.010 M.

To calculate the final concentration of the dilute solution, let C_i=2.010 M and let D= 30. Substitute C_i and D into Equation (2) and solve for C_f, the concentration of the dilute solution.

$$30 = 2.010\ M / C_f$$

$$C_f = 2.010\ M / 30 = 0.06700\ M$$

The concentration of the dilute solution is 0.06700 M.

Use Equation (1) to determine the volume of the stock solution, V_i, needed.

$$(2.010\ M)\ V_i = (0.06700\ M)\ (100.0\ mL)$$

$$V_i = 3.333\ mL$$

Planned action: To prepare a 0.06700 M solution, pipet 3.333 mL from the 2.010 M stock solution and transfer this quantity into a 100 mL volumetric flask. Make up to the mark with distilled water and mix thoroughly.

Task 1: Dilution

1. You are asked to dilute the stock solution, Solution A, by the dilution factors given in the table below. Each diluted solution has a final volume of 100.0 mL.
   The Stock solution, Solution A, is 2.010 M.

   Using Equations (1) and (2), calculate C_f and V_i. Fill out the table below.

Dilution Factor	**C_i (M)**	**C_f (M)**	**V_i (mL)**	**V_f (mL)**
2	2.010	_____	_____	100.0
5	2.010	_____	_____	100.0
10	2.010	_____	_____	100.0

2. Reach your hand into the box and draw a dilution factor.
3. Use the calculated values, prepare 100.0 mL of the dilute solution.

Your Planned action:

To prepare a _____ M (C_f) solution, withdraw _____ mL (V_i) from the 2.010 M stock solution and transfer this quantity into a 100 mL volumetric flask. Make up to the mark with distilled water and mix thoroughly.

Task 2: Prepare a set of standard solutions by dilution (in pairs)

You will be given a stock solution of 1.000 M. From this solution prepare a 1.00×10^{-2} M working solution. From the working solution, prepare the following standards.

- 1.00×10^{-4} M
- 3.00×10^{-4} M
- 5.00×10^{-4} M
- 7.00×10^{-4} M
- 1.00×10^{-3} M

Datasheet:

Preparation of Working Solution: 1.00×10^{-2} M

Stock Solution: _____ M

Solution	**Volume from the stock solution (mL)**	**Total Volume (mL)**
1.00×10^{-2} M	_____	_____

Preparation of Standards:

Working Solution: _____ M

Solution	**Volume from the working solution (mL)**	**Total Volume (mL)**
Standard 1: 1.00×10^{-4} M	_____	_____
Standard 2: 3.00×10^{-4} M	_____	_____
Standard 3: 5.00×10^{-4} M	_____	_____
Standard 4: 7.00×10^{-4} M	_____	_____
Standard 5: 1.00×10^{-3} M	_____	_____

Assessment Scheme: Maximum score = 15 points
Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory; (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Use of Volumetric Flask	U	S	G	E	Comment
Knowledge of keywords: meniscus, Pasteur pipette, quantitative transfer					
Check dilution calculation					
Pipette of stock solution using small beaker					
Observe filling the flask 2/3 full and mix.					
Techniques in bringing level to mark – use of Pasteur pipette, bring mark to eye level					
Uniform mixing of final solution - invert flask repeatedly (~15 times)					

Common mistakes:

- Over or under the mark – U
- Use water bottle to bring to mark – S
- Forget to replace cap on reagent bottle – S
- Improper mixing (not inverting 15 times) – S
- Use Pasteur pipette directly from water bottle – S
- Record to improper sig figs – U
- Pipette directly from reagent bottle - U

Part C: Preparation of a Standard Solution

Task:

You will be asked to prepare a 50.00 mL standard solution of copper (II) sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$.

1. Reach your hand into the box and draw a concentration. This is the concentration of the $CuSO_4 \cdot 5H_2O$ standard solution you are preparing.
2. Calculate the mass of $CuSO_4 \cdot 5H_2O$ needed using this equation.

$$\text{concentration of } CuSO_4 \cdot 5H_2O \text{ (M)} = \frac{\dfrac{\text{Mass } CuSO_4 \cdot 5H_2O \text{ (g)}}{\text{Molar Mass } CuSO_4 \cdot 5H_2O \text{ (g/mole)}}}{\text{Total volume of solution (L)}}$$

3. a. Concentration of $CuSO_4 \cdot 5H_2O$ standard solution _____

   b. Molar mass of $CuSO_4 \cdot 5H_2O$ _____

   c. Total volume of solution 50.00 mL

   d. Mass of $CuSO_4 \cdot 5H_2O$ to be weighed _____

4. Weigh the $CuSO_4 \cdot 5H_2O$ sample.

Weigh by taring

1. Mass of weigh boat and $CuSO_4 \cdot 5H_2O$ _____

2. Mass of empty weigh boat 0.0000 g

3. Mass of $CuSO_4 \cdot 5H_2O$ _____

Assessment Scheme: Maximum score = 15 points
Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory; (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Prepare a 50-mL Standard Solution	**U**	**S**	**G**	**E**	**Comment**
Knowledge of keywords: standard solution, quantitative transfer, primary standard, three properties of a primary standard					
Correct calculation of $CuSO_4 \bullet 5H_2O$ required for the targeted concentration					
Technique in weighing of $CuSO_4 \bullet 5H_2O$ **(Technique 4)**					
(i) Knowledge of the two different methods of weighing					
(ii) Check balance is zeroed and the balance chamber is clean					
(iii) Record mass to proper number of significant figures					
(iv) Removal of weigh boat from the balance before adding sample					
(v) Set the weigh boat on a clean surface (on Kimwipes)					
(vi) Close doors before reading mass					
(vii) Leave balance with doors closed and balance turned off					
Transfer technique – weigh boat to beaker					
Dissolving $CuSO_4 \bullet 5H_2O$ in a beaker with ~2/3					
Technique in quantitative transfer – rinsing at					

least 3 times					
Transfer technique – beaker to volumetric flask					
Technique in making the solution in the volumetric flask **(Technique 3)**					
(i) Observe filling the flask 2/3 full and mix.					
(ii) Techniques in bringing level to mark – use of Pasteur pipette, bring mark to eye level					
(iii) Uniform mixing of final solution - invert flask repeatedly (~15 times)					

Common mistakes:

- Review common mistakes for Techniques 3, 4
- Quantitative transfer:
  Rinse once – S
  Rinse twice – G
- Does not dissolve crystal in the beaker before transferring to flask – S
- Record to improper significant figures – U

Graphing – Determine the Heat of Vapourization of Water

Last week, you were given data for the boiling point of water at various pressures. The mathematical relationship between pressure and temperature is given in Equation (3).

$$\ln P = -\frac{\Delta H_{vap}}{R}\frac{1}{T} + C \qquad \text{Equation (3)}$$

where

- P is pressure (kPascal),
- R is the gas constant (8.314 joules/moleK),
- T is temperature (Kelvin), and
- *C* is a constant.

The graph, "**ln P Vs 1/T**", showed a linear relationship between ln Pressure (P in kPa) and 1/Temperature (K^{-1}). We applied statistical error analysis using LINEST in Excel and determined the uncertainties in the slope and the intercept of the best fit line. From the slope of the line, ΔH_{vap}, the vapourization of water, was determined.

This week, we are given another of data for the boiling point of water at various pressures. Note that the temperature readings are recorded with significant figures in mind.

T(°C)	P(kPa)
0.1	0.612
10.0	1.227
20.0	2.536
30.1	4.242
40.2	7.37
50.0	12.33
59.8	19.9
70.2	31.15
75.7	46.12
89.7	70.1
100.1	101.32
120.2	198.5
200.2	1554.3

The quantity of interest is ΔH_{vap}. It is known as the heat of vapourization. In this case, this is the energy required to convert a mole of liquid water into its vapour.

Plot a graph of "**log P Vs 1/T**" using the data given and determine the ΔH_{vap} of water. The conversion between natural logarithm (ln) and logarithm to the base 10 (log) is given in Equation (4).

$$\ln y = 2.303 \times log\ y \qquad \text{Equation (4)}$$

Apply the Equation (4) to Equation (3) and determine the ΔH_{vap} of water from your graph of "log P Vs 1/T".

Be sure to:

1. Title the graph "Y vs. X for the Vapourization of Water".
2. Label the axes.
   Note: log P does not have units. The axis should be labeled "log P (P in kPa)".
   It is important to show the unit of pressure.
3. Fit the data with a trendline. Add the equation of the trendline and the R^2 value on the graph.
4. Use LINEST to find the uncertainty in the slope and intercept. Include the numbers generated by LINEST on your graph, or on a separate piece of paper.
5. On the graph, write the slope = _____ unit.
6. Show your work on the graph and determine the ΔH_{vap} of water.

Experiment 3: Practical Extractions / Introduction to Statistical Methods (**View photos of equipment on website**)

Introduction: Practical Extraction | Mean, σ, RSD | Example: Mean, σ, RSD | Outliers & Dixon Q Test
Procedure | Datasheet #1 | Datasheet #2

Objectives:

Part A: The student will be introduced to a simple practical extraction technique. The importance of such extractions, including the practical considerations involved, will be emphasized.

Part B: The student will be introduced to some simple statistical methods that are frequently used in chemical analysis. Practical situations will be used to demonstrate the usefulness of these methods.

Part A: Practical Extraction

Introduction:

Solvent extraction is a very useful process for the separation and isolation of compounds from mixtures. It is generally applied to organic materials. Some of many applications of solvent extraction are:

- extraction of pesticides from food or soils,
- lignins from wood,
- alkaloids from leaves and bark, and
- perfume essences from flowers.

Extraction is where a liquid phase, the extractant, removes a desired substance from a second phase. This second phase may be a solid (**solid-liquid extraction)**, or liquid (**liquid-liquid extraction**).

Theory of Liquid-Liquid Extraction

When a solution of a solute in one solvent is shaken with a second solvent, which is **immiscible** with the first, the solute distributes itself between the two solvents in proportion to its solubility in the two pure solvents. Thus a constant ratio is set up between the concentrations of the solute in the two phases or immiscible solvent layers. This ratio or equilibrium can be represented by the equation:

$$K = \frac{C_1}{C_2}$$

K is called the distribution or partition coefficient. C_1 and C_2 are the concentrations of the solute in the two solvents. These concentrations are expressed in grams/mL.

K is approximately constant for all concentrations of C_1 and C_2. However, K will vary with temperature as solubility is temperature dependent.

Properties of a Good Extracting Solvent

Properties desirable for a good extracting solvent are:

1. It should be immiscible, or very sparingly soluble in the liquid from which the solute is to be extracted.
2. It should readily dissolve the substance to be extracted.
3. It should be capable of being easily separated from the solute after the extraction. This is usually by distillation.
4. It should ideally be cheap and non-flammable.

Choice of Solvents

The choice of immiscible solvent pair can be generalized as follows. One solvent should be polar and the other solvent should be considerably less polar. The polar solvent is usually water and the low polarity solvent is diethyl ether or a volatile hydrocarbon.

A useful generalization in predicting solubility and hence partitioning between solvents is the following:

> **Salts and other compounds having an ionic structure are generally soluble in water and insoluble in ether and hydrocarbons. Conversely, organic compounds, including the unionized forms of organic acids and bases are generally insoluble in water and soluble in ether and organic solvents. However it must be remembered that most compounds have some solubility in both types of solvents.**

Solid-Liquid Extraction

When the analyte needs to be extracted from a solid material rather than a liquid, it is refrred to as a solid-liquid extraction. In this case, a solid sample is placed in the same container as the liquid and the analyte is separated from the solid because it dissolves in the liquid while the other sample components do not.

The extraction process can be carried out in two ways:

1. The sample and the liquid are shaken together in the same container. The resultant mixture is filtered, and the filtrate, which then contains the analyte, is collected.
2. The fresh extracting solvent is continously cycled over a period of hours through the solid sample via a continuous evaporation-condensation process. This maximizes the transfer of the analyte to the liquid phase and the filtrate is collected. This technique is known as a **Soxhlet extraction**. The advantages of this technique are:
    1. fresh solvent is continuously in contact with the sample without having to introduce more solvent, which could dilute the extract.
    2. the process is automated so that the extraction can be conveniently set to occur overnight if desired.

In Part A of this experiment, the first method will be used while the second method will be demonstrated in a later experiment.

Experiment 3: Practical Extractions / Introduction to Statistical Methods

Introduction: Practical Extraction | Mean, σ, RSD | Example: Mean, σ, RSD | Outliers & Dixon Q Test
Procedure | Datasheet #1 | Datasheet #2

Part B: Statistical Methods
Introduction:

1. Mean or Average

In the chemical laboratory it is usually not a good practice to analyze one portion of a sample and use the results of the single analysis to make general statements about the properties, such as concentrations, of the sample. Many repeat analyses should be performed on the same sample. As the results of more analyses are pooled together then the level of confidence in the result obtained is increased to a satisfactory conclusion. The most common way to represent the overall result from a series of analyses performed on the same sample is to use the **mean value** of the data. It is simply the average of all the available results and may be calculated with the following equation

$$\text{mean} = \frac{x + y + z}{n}$$

where x, y and z are the readings while n is the number of readings.

2. Standard Deviation

Calculation of the mean provides the chemical analyst with a way to represent the overall value or average value for the data. However it does not provide any information about the range of results that were obtained. This can be critical in many situations. For example, consider a sawdust sample where the moisture level (wet basis) was found to be 18%, 118%, 95%, 250% and 10% for several analyzed portions of the same sample. The mean moisture level (wet basis) is 98.2% (i.e. (18 + 118 + 95 + 250 + 10)/5 = 98.2%) . However it is readily apparent from the results that the sample is heterogeneous in composition; something that cannot be indicated by the mean value. What is required is a measure of the range or spread of values that were obtained.

The variance is a measure of how spread out a distribution is. It is computed as the average square deviation of each number from its mean. For example, for the numbers 1, 2, and 3, the mean is 2 (i.e. - (1 + 2 + 3)/3 = 2) and the variance is:

$$\sigma^2 = \frac{(1 - 2)^2 + (2 - 2)^2 + (3 - 2)^2}{3} = 0.667$$

The formula for the variance in a population, where a population consists of an entire set of objects, observations, or scores that have something in common. For example, a population might be defined as all female between the ages of 12 and 14, is:

$$\sigma^2 = \frac{\sum_{i=1}^{N} (x_i - \mu)^2}{N}$$

where μ is the mean and N is the number of observations or scores.

When the variance is computed in a sample, where a sample is a subset of a population, the statistic

$$S^2 = \frac{\sum_{i=1}^{N} (x_i - M)^2}{N}$$

where M is the mean of the sample and S^2 is a biased estimate of σ^2.

Note: Since it is usually impractical to test every member of a population, a sample from the population is typically the best approach available. A statistic is biased if, in the long run, it consistently over or underestimates the parameter it is estimating.

The most common formula for computing variance in a sample is:

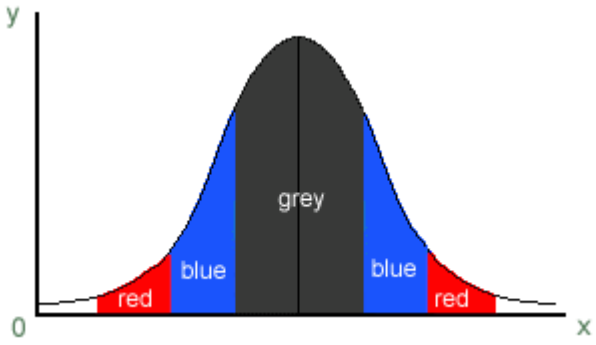
$$S^2 = \frac{\sum_{i=1}^{N} (x_i - M)^2}{N - 1}$$

The formula for the standard deviation is very simple. It is the square root of the variance. It is the most commonly used measure of spread, or how precise the average is. That is, how well the individual numbers agree with each other.

$$S = \sqrt{\frac{\sum_{i=1}^{N} (x_i - M)^2}{N - 1}}$$

For normal distributions, the distributions are symmetric with scores more concentrated in the middle than in the tails.

Within one standard deviation, or 1 σ from the mean in either direction on the horizontal axis (the grey area on the graph below) accounts for somewhere around 68 percent of the scores. Within two standard deviations, or 2σ away from the mean (the grey and blue areas) account for roughly 95 percent of the scores. Within three standard deviations, or 3 σ (the grey, blue and red areas) account for about 99 percent of the scores.



The **relative standard deviation** (RSD) is sometimes more convenient. It is expressed in percent and is obtained by multiplying the standard deviation by 100 and by dividing this product by the average.

$$\text{relative standard deviation, RSD} = \frac{100 \cdot S}{M}$$

Experiment 3: Practical Extractions / Introduction to Statistical Methods

Introduction: Practical Extraction | Mean, σ, RSD | Example: Mean, σ, RSD | Outliers & Dixon Q Test
Procedure | Datasheet #1 | Datasheet #2

Part B: Statistical Methods
Introduction:

An example of calculations of: (i) mean or average, (ii) standard deviation, and (iii) relative standard deviation.

Four measurements of lengths were taken: 51.3 cm, 55.6 cm, 49.9 cm and 52.0 cm.

(i) mean or average:

$$\text{mean} = \frac{51.3 + 55.6 + 49.9 + 52.0}{4} = \frac{208.8}{4} = 52.2 \text{ cm}$$

(ii) standard deviation:

$$S = \sqrt{\frac{(51.3-52.2)^2 + (55.6-52.2)^2 + (49.9-52.2)^2 + (52.0-52.2)^2}{4 - 1}}$$

$$= \sqrt{\frac{(-0.9)^2 + (3.4)^2 + (-2.3)^2 + (-0.2)^2}{3}}$$

$$= \sqrt{\frac{0.81 + 11.56 + 5.29 + 0.04}{3}}$$

$$= \sqrt{5.9}$$

$$= 2.4$$

(iii) relative standard deviation, RSD = (S / mean) x 100 = 2.4 / 52.2 x 100 = 4.6 %

Therefore, for our measurement, we can write 52.2 ± 2.4 cm or 52.2 cm ± 4.6 %.

Part B: Statistical Methods
Introduction:

3. Outliers and the Dixon Q Test

The requirements for having more than a single analysis on which to make general statements regarding the composition of a sample also introduce other considerations. If we have several results, how can we decide scientifically if the results should all be retained. Perhaps we suspect that one of the results is erroneous, **an outlier**.

Several methods exist to test sets of results to see if any potential outliers can be identified. The most commonly used is the **Dixon Q Test** (Reference: p 178).

The data set containing N values is sorted either in an ascending or descending order, with x_1 being the suspect value. Then the test statistic Q is calculated using the equation

$$Q = \frac{| x_1 - x_2 |}{| x_1 - x_N |}$$

The decision whether x_1 is an outlier is performed by comparing the value Q to the critical values listed in the following table:

Q(P, *N*)			
N	**P = 0.90***	**P = 0.95****	**P = 0.99*****
3	0.89	0.94	0.99
4	0.68	0.76	0.89
5	0.56	0.64	0.76
6	0.48	0.56	0.70
7	0.43	0.51	0.64
8	0.40	0.47	0.58
9	-	0.44	-
10	-	0.41	-

* P = 0.90 means that the data is valid to 90% confident level.
** P = 0.95 means that the data is valid to 95% confident level.
*** P = 0.99 means that the data is valid to 99% confident level.

If the Q value is greater than the tabulated value for the same number of results then the result can be rejected.

Example:

Consider the following set of data:

Trials	**#1**	**#2**	**#3**	**#4**	**#5**	**#6**
Data (mg/L)	203	204	205	206	207	214

Is the data point 214 mg/L in Trial #6 an outlier for a 95% confident level?

Calculate the Q value:

$$Q = \frac{|\ 214 - 207\ |}{|\ 214 - 203\ |} = 0.64$$

Compare the calculated Q value with *N* = 6 in the Dixon Q Test table.

P = 0.90 ⟶ Q = 0.48
P = 0.95 ⟶ Q = 0.56
P = 0.99 ⟶ Q = 0.70

Our calculated Q value = 0.64 **is greater** than the Q value at 95% confidence level. Therefore, Trial #6 is removed from the data set.

Experiment 3: Practical Extractions / Introduction to Statistical Methods

Introduction: Practical Extraction | Mean, σ, RSD | Example: Mean, σ, RSD | Outliers & Dixon Q Test
Procedure | Datasheet #1 | Datasheet #2

Procedure:

Students will work in pairs. Data generated by all students will be pooled for use in subsequent calculations to be submitted as part of the lab report.

1. A large bulk sample of contaminated sand will be provided. In pairs, cone and quarter the sand three times. Transfer the portion of sample selected onto wax paper. You will have about **60 g** of sample at this stage.
2. Table and then quarter the sample on the wax paper. At the end of this step you should have about **30 g** of sample (lab sample).

Work individually from this step onward.

3. Clean the provided Erlenmeyer flask with acetone and then ensure that it is dry.
4. Weigh accurately 10 g of the contaminated sand sample in the flask.
5. In the fume hood, add 25 mL of dichloromethane, cover with a watch glass and then swirl gently. Place the flask on a magnetic stirrer and stir for about 60 to 70 minutes.
6. In the fume hood, filter the sample using Whatman #1 filter paper which has been washed with a small amount of dicloromethane. Discard the washing.
7. Filter the contaminated sand mixture by gravity filtration. The filtrate should be collected in a **preweighed** round bottom flask that can be used with the rotary evaporator.
8. Rinse the Erlenmeyer flask with small portions of dichloromethane, transferring the washings to the filter. Finally rinse the filter paper with two 5 mL portions of dichloromethane.
9. With the aid of your instructor, remove the dichloromethane solvent using a rotary evaporator. Once all the solvent is removed, dry the outside of the flask and reweigh it. The weight of the extracted material is the difference between the two weighs.
10. The recovered dichloromethane solvent should be placed in a suitable container as it can be used again for subsequent extractions.
11. Calculate the % oil in the sand. Remember to subtract the blank value provided by your instructor. The blank value is the weight of the dichloromethane extracted material if no oil has been added to the sand.
12. Assess the % recovery of the oil from the sand using the provided reference value for the amount of oil added to the sand.
13. Tabulate the % oil values for all the set. Calculate the mean, standard deviation and relative standard deviation of the results. Remember to test any suspect values using Dixon's Test.

Experiment 3: Practical Extractions / Introduction to Statistical Methods

Introduction: Practical Extraction | Mean, σ, RSD | Example: Mean, σ, RSD | Outliers & Dixon Q Test
Procedure | Datasheet #1 | Datasheet #2

Practical Extractions

Data Sheet:

Flask # _____

1. Weight of sand (oven dried weight) _____

2. Weight of round bottom flask _____

3. Weight of round bottom flask + extracted material _____

4. Weight of extracted material (item 3 - item 2) _____

Calculations:

Weight of extracted material for blank = **0.03000 g for 10 grams of material**

Weight of extracted material for blank in Flask # =

_____ x weight of sand/10 g = _____

1. % oil in the sand: (Show calculation in the space provided below. Remember to account for the blank.) Give this number to your instructor.

2. % recovery of the oil from the sand for Flask # _____
(Show calculation in the space provided below.)

Reference value for weight of oil spike = 0.5000 g.

Experiment 3: Practical Extractions / Introduction to Statistical Methods

Introduction: Practical Extraction | Mean, σ, RSD | Example: Mean, σ, RSD | Outliers & Dixon Q Test Procedure | Datasheet #1 | Datasheet #2

Practical Extractions

Data Sheet:

Tabulate values for the whole set of results:

Site 1	Site 2	Site 3

In the space provided below, show your work on performing Dixon Tests. If you need to eliminate any data as a result of performing the Dixon Q Test, put a neat line through any data listed in the

above table.

Calculate mean, standard deviation and relative standard deviation for the whole data set:

Mean	Standard Deviation	Relative Standard Deviation

Techniques and Practice 4: Burette, Titration, Graphing

Objectives

At the end of this lab, the student should be able to perform a titration with a burette.

Reading

- Demonstration of the Nine Practical Lab Techniques

  Technique #1 – Use of a Burette

  Technique #8 - Titration

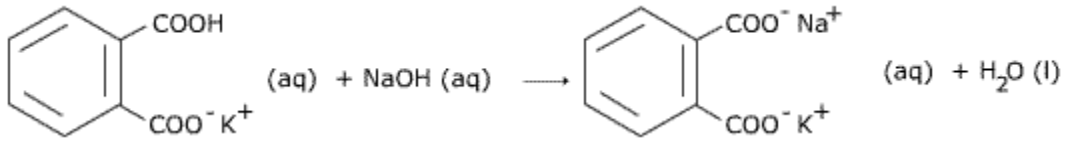
Introduction

Alkaline solutions (eg 0.1 M NaOH) should be protected from the atmosphere because it absorbs carbon dioxide and reacts with it:

$$OH^-(aq) + CO_2(g) \longrightarrow HCO_3^-(aq)$$

CO_2 absorption changes the concentration of a strong base over a period of time. It also decreases the extent of reaction near the endpoint in the titration of weak acids. If the alkaline solutions are stored in tightly capped polyethylene bottles, they may be used for about a week with little change in concentration.

Reagent grade sodium hydroxide may also contain sodium carbonate (from reaction with atmospheric CO_2 and water) and adsorbed water. Solutions of sodium hydroxide must be standardized against a primary standard. **Potassium hydrogen phthalate** (**KHP**) is among the most convenient primary standards for this purpose. It is a white, crystalline, nonhygroscopic, acidic solid with a high degree of purity. To determine the molar concentration of the NaOH solution, a measured mass of potassium hydrogen phthalate is dissolved in distilled water and the NaOH solution is added from a buret until the endpoint is reached. Sodium hydroxide reacts with potassium hydrogen phthalate according to the equation:



The amount (in moles) of potassium hydrogen phthalate titrated in the analysis can be calculated from the initial measured mass and the molecular weight (molar mass) of KHP.

$$\text{mass of KHP (g)} \times \frac{\text{1 mole KHP}}{\text{204.23 g KHP}} = \text{moles of KHP}$$

At the equivalence point, equal moles of KHP and NaOH have reacted.

$$\text{moles of NaOH} = \text{moles of KHP}$$

The molar concentration of the prepared NaOH solution is calculated from the moles of NaOH needed to react with the KHP and the volume of NaOH added from the buret.

$$\text{molarity of NaOH, M} = \frac{\text{moles of NaOH}}{\text{Volume of NaOH (liter)}}$$

Part A - Technique #1 – Use of a Burette

Task:

1. Prepare and fill the burette with the solution provided.
2. Record the initial burette reading.
3. Place an Erlenmeyer flask under the burette to receive the solution from the burette.
4. Using the timer provided, open the stopcock for approximately 15 seconds.
5. Record the final burette reading.
6. Determine the volume of the solution delivered into the Erlenmeyer flask.

Datasheet:

1. Final Burette Reading _____

2. Initial Burette Reading _____

3. Volume of solution delivered _____

Instructor initial: _____

Assessment Scheme: Maximum score = 15 points

Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory; (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Use of Burette	U	S	G	E	Comment
Knowledge of keywords: acclimatization, meniscus					
Acclimatize technique					
Check for bubble and filling technique					
Relative position of burette to receiving flask					
Reading burette – eye level, check agreement with student					
Recording data to proper significant figures					

Common mistakes:

- Angled on the burette stand – S
- Burette tip not well inside the neck of the Erlenmeyer flask – G
- Take burette off the clamp to read – G
- Bubble remains even after trying to dislodge it – S
- Wet outside – S
- Does not know bubble is there or check for bubble – U
- Disagreement with reading – U
- Record to improper sig figs (not from calculations) – U

Part B - Preparation of 0.1 M NaOH Solution

1. Dissolve 3.0 g +/- 0.1 g of sodium hydroxide pellets in 15 mL of distilled water in a 250 mL rubber-stoppered Erlenmeyer flask. Thoroughly mix and allow the solution to stand briefly for the precipitation of any sodium carbonate. Na_2CO_3 has a low solubility in a concentrated NaOH solution.
2. With a graduated cylinder, transfer 10 mL of the concentrated NaOH solution into a 500.0 mL volumetric flask. Do not transfer any of the Na_2CO_3 precipitate. Add distilled water to the mark and thoroughly mix the dilute NaOH solution.

Part C - Standardization of NaOH Solution

1. Clean a buret for titration. Acclimatize the buret by rinsing with three 5 mL portions of the prepared NaOH solution, making sure that the solution wets its entire inner surface including the tip. Fill the buret with the NaOH solution.
2. Measure 3 portions of 0.3 +/- 0.05 g of dried potassium hydrogen phthalate (KHP) accurately to 4 decimal places in a clean, dry 125 mL Erlenmeyer flask. The KHP has been dried at 105^oC for several hours and allowed to cool in a desiccator.
3. Dissolve the KHP in 30 mL of distilled water and add 3 drops of phenolphthalein solution. The phenolphthalein solution has been prepared by dissolving 0.5 g of phenolphthalein in a 50 mL of ethanol and then adding 50 mL of water.
4. Titrate the KHP solution with the NaOH solution from the buret until the endpoint is reached. This occurs when a single half-drop causes the pink colour of the phenolphthalein indicator to persist for 30 seconds. Record the volume of NaOH solution added to an accuracy of 0.02 mL. Repeat two more times.
5. Repeat steps 5 and 6 at least two more times with accurately known amounts of potassium hydrogen pthlalate until the molar concentration of NaOH solution is within 0.001 M.

Data Sheet:

Part B - Preparation of 0.1 M NaOH Solution

Mass of sodium hydroxide pellets _____ g

Part C - Standardization of NaOH Solution

	Trial 1	**Trial 2**	**Trial 3**	**Trial 4**
Mass of KHP (g)				
Initial buret reading (mL)				
Final buret reading (mL)				
Volume of NaOH solution (mL)				
Concentration of NaOH solution (M)				

Average concentration of NaOH Solution =

_____

Aim: molar concentration of NaOH to be within 0.001 M.

Show sample calculation of the concentration of NaOH solution for Trial #1.

Assessment Scheme: Maximum score = 15 points
Marking Scheme: (marks in brackets)
U = unsatisfactory (0); S = satisfactory; (1); G = Good (2); E = Excellent (3)

General:

- Does not wear goggles – minus 1 point
- Does not write data directly on the datasheet in pen – minus 1 point
- Does not label glassware – minus 1 point
- You must obtain a minimum of 5 checks. Less than 5 checks means an incomplete technique – minus 1 point

Titration	U	S	G	E	Comment
Knowledge of keywords: burette, volumetric pipette, titrant, indicator, phenolpthalein, colour change expected					
Burette height relative to the Erlenmeyer flask					
Obtain acid using a beaker and pipette from the beaker					
Technique in pipetting **(Technique 2)**					
(i) Use of pipette bulb					
(ii) Acclimatize technique					
(iii) Wiping the outside of the pipette before adjusting to the mark					
(iv) Control of pipette while adjusting the liquid level to the mark – Bring to eye level					
(v) Dispensing technique – draining with tip touching wall of containing and waiting for 10 seconds					
Remember to add phenolpthalein					
Technique in swirling and rinsing					
Control of the stopcock to dispense titrant at various various rate (ie – a stream to drop-wise)					
Ability to hold a partial drop at the tip of the burette and wash the drop into the Erlenmeyer flask					
Acceptable final colour change					

Record of data to proper number of significant figures					
Final burette volume is +/- 0.10 mL					

Common mistakes:

- Does not choose to use the small beaker to pour acid – S
- Within +/- 0.20 mL of the true titration volume – G
- Within +/- 0.30 mL of the true titration volume - S
- Greater than +/- 0.30 mL of the true titration volume - U
- On 2nd and subsequent titration, does not quickly get close to the 1st titration volume – S
- Half-drop technique – drop by 1 for each missed try
- No white paper under the Erlenmeyer flask – S
- Disagreement with reading – U
- Record to improper sig figs (not from calculations) – U
- Pipette from reagent bottle - U

Graphing – Determine the relationship between concentration and time

For a reaction

$$A \rightarrow B + C$$

The concentration of A, [A], in unit of molarity, was measured over time in seconds, and the following data was obtained.

Time (s)	[A] (M)
192	5.755
270	5.3
499	4.624
596	4.244
788	3.787
955	3.289
1069	2.932
1382	2.45
1697	2.013
1992	1.635
2167	1.403
2772	0.945
3399	0.622

(a) Plot "Concentration of species A versus Time" and comment on the relationship between the concentration of species A and time.
(b) Plot "ln (Concentration of species A) versus Time" and comment on the relationship between ln (Concentration of species A) and time.
(c) Run linest on the graph that exhibits a linear relationship. The slope of the graph is the negative rate constant, k .

$$\text{slope} = -k$$

(d) Determine the rate constant, k.

Experiment 4 - Acid-Base Titration

Objective

To determine the concentration of a NaOH solution.

Apparatus:

1. 50 mL burette
2. Burette clamp
3. 25 mL pipette
4. Pipette bulb
5. Small funnel
6. Weighing boat
7. Wash bottle
8. 250 mL volumetric flask
9. 100 mL beaker
10. 125 mL Erlenmeyer flask
11. 250 mL beaker

Chemical:

1. Solid Potassium Hydrogen Phthalate

Solutions:

1. NaOH solution of unknown concentration
2. Phenolphthalein indicator

Introduction

In this experiment, a standard solution of potassium hydrogen phthalate, $HKC_8H_4O_4$ will be prepared. Given the molar mass for $HKC_8H_4O_4$ is 204.2 g/mole, the concentration of the standard solution can be determined using the following equation.

$$\text{Concentration of } HKC_8H_4O_4\,(M) = \frac{\left[\dfrac{\text{mass of } HKC_8H_4O_4\,(g)}{\text{molar mass of } HKC_8H_4O_4\,(g/mole)}\right]}{\text{Volume of solution (L)}}$$

The balanced chemical reaction involving potassium hydrogen phthalate and NaOH is given below.

$$HKC_8H_4O_{4(aq)} + NaOH_{(aq)} \rightarrow NaKC_8H_4O_{4(aq)} + H_2O_{(l)}$$

Using phenolphthalein as the acid-base indicator, the concentration of the NaOH solution can be determined.

$$\text{Concentration of NaOH (M)} = \frac{\text{Volume of } HKC_8H_4O_4 \text{ used (L)} \cdot \text{Concentration of } HKC_8H_4O_4\,(M)}{\text{Average titration volume (L)}}$$

Procedure:

Part A - Preparation of Standard Potassium Hydrogen Phthalate, $HKC_8H_4O_4$, Solution

1. Weigh 3.0 - 3.2 grams of Potassium Hydrogen Phthalate, $HKC_8H_4O_4$.
2. Dissolve the acid in about 150 mL of distilled water.
3. Transfer the acid solution quantitatively into a 250.0 mL volumetric flask.
4. Fill the flask to the mark with distilled water, and mix thoroughly.

Procedure:

Part B - Acid-Base Titration

1. Obtain about 100 mL of the NaOH solution in a clean and dry beaker.
2. Acclimatize the buret and then fill the buret with the base solution
3. Pipet 25.00 mL of the acid solution into an Erlenmeyer flask. Add about 3 drops of phenolphthalein indicator solution to the acid in the flask.
4. Titrate the potassium hydrogen phthalate, $HKC_8H_4O_4$, solution with the NaOH solution from the buret until a pink end point is reached.

5. Record the volume of the NaOH solution added to an accuracy of +/- 0.02 mL.
6. Repeat steps 3 to 5 until the titration volumes agree within +/- 0.10 mL.
7. Enter your data clearly on the datasheet and present calculations to show how you determined the concentration of the NaOH solution in moles/L.

Datasheet:

Part A - Preparation of Standard Potassium Hydrogen Phthalate, $HKC_8H_4O_4$, Solution

1. Mass of empty weighing boat	
2. Mass of empty weighing boat and Potassium Hydrogen Phthalate, $HKC_8H_4O_4$.	
3. Mass of Potassium Hydrogen Phthalate, $HKC_8H_4O_4$.	
4. Volume of solution prepared	

Datasheet:

Part B - Acid-Base Titration

1. Concentration of standard $HKC_8H_4O_4$ solution				
2. Volume of $HKC_8H_4O_4$ solution used (Part B - step 4)				
3. Volume of NaOH solution added to reach the end point.				
	Trial 1:	**Trial 2:**	**Trial 3:**	**Trial 4:**
1. Final burette reading (mL)				
2. Initial burette reading (mL)				
3. Volume of titrant used in titration (mL)				

Calculations:

Part A - Preparation of Standard Potassium Hydrogen Phthalate, $HKC_8H_4O_4$, Solution

Show all work in calculating the concentration of the potassium hydrogen phthalate, $HKC_8H_4O_4$, solution in moles/L.

Part B - Acid-Base Titration

Show all work in calculating the concentration of the NaOH solution in moles/L

Experiment 5: Water Analysis (Colorimetry/Spectrophotometry)

Introduction: Colorimetry | Spectrophotometry | Lab Introduction
Procedures: Standards & Sample Preparation | Determination of λ_{max} | Analysis of Sample
Datasheet #1 | Datasheet #2

Objectives:

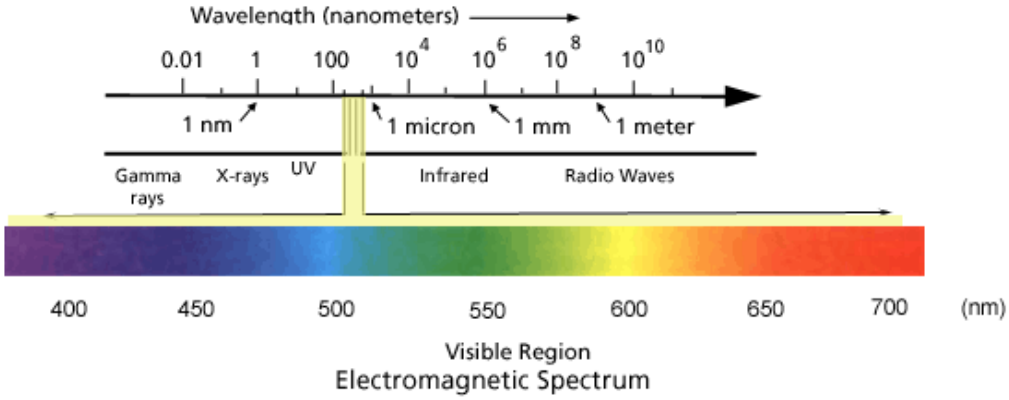
The student will be able to carry out the analysis of a river water sample for iron using the method of Nessler (colour comparison) and by spectrophotometry using a modern solid state spectrocphotometer. Inherent in the analysis will be the ability to choose the correct wavelength by scientific means, make up standards accurately, carry out chemical conversion to the final colour form and assess potential errors in both procedures.

Introduction:

Colorimetry is the chemical analysis of a **coloured** species in solution through the measurement of absorption of radiation. Human eyes are usually used to detect the colour. It is used in **quantitative** determination of any soluble coloured material.

When a **spectrophotometer** is used in the determination of colour by employing an **electronic detector** instead of the human eyes, the method is called **spectrophotometry**. Thus it eliminates the human error due to our eyes.

Visible light is electromagnetic radiation with wavelengths between 400 nm and 700 nm. The colour of light depends on the wavelength as shown below.



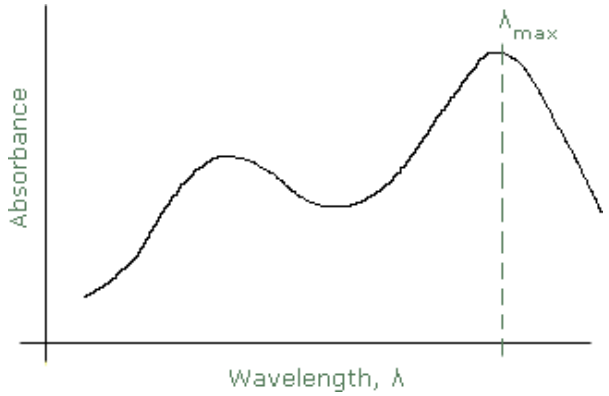
Electromagnetic Spectrum

The electromagnetic spectrum, which encompasses the visible region of light, extends from gamma rays with wavelengths of one hundredth of a nanometer to radio waves with wavelengths of one meter or greater.

The solutions of many substances can selectively absorb light of certain wavelengths and transmit the remainder. For example, methylene blue is blue because it absorbs red and orange light and transmits light of other wavelengths.

For every absorbing substance there is a wavelength where the absorption is a maximum; this wavelength is called the λ_{max}. Spectrophotometric analyses are always carried out at the λ_{max} since these readings are the most accurate.

Absorption Spectrum of a Hypothetical Compound



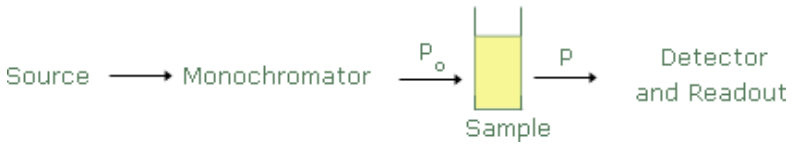
Experiment 5: Water Analysis (Colorimetry/Spectrophotometry)

Introduction: Colorimetry | Spectrophotometry | Lab Introduction
Procedures: Standards & Sample Preparation | Determination of λ_{max} | Analysis of Sample
Datasheet #1 | Datasheet #2

Spectrophotometry:

The absorbance at various wavelengths is measured with a spectrophotometer which consists of:

1. Light Source
2. Monochromator (wavelength selector)
3. Sample container
4. Detector and readout



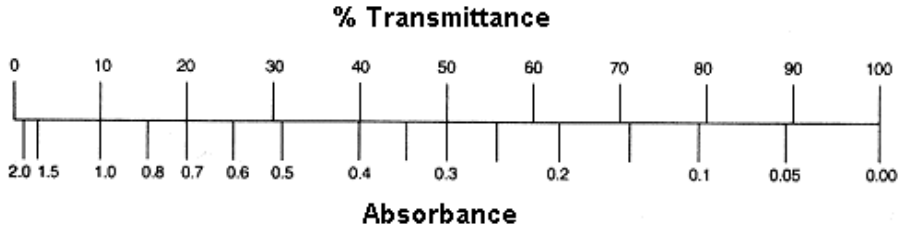
Light of appropriate wavelength is selected by the monochromator. This light, P_o passes through the sample and some of it is absorbed. The remaining light P is transmitted. It strikes the detector and is measured.

Transmittance, T, is the ratio of the radiant power, P, transmitted by the sample to the radiant power, P_o entering the sample and percent transmittance is this ratio multiplied by 100.

$$T = \frac{P}{P_o}$$

$$\%T = \frac{P}{P_o} \times 100$$

The relationship between absorbance and transmittance is illustrated in the following diagram.



So if all the light passes through a solution without any absorption, then absorbance is zero and %T is 100 %. If all the light is absorbed, then %T is zero and absorbance is infinite.

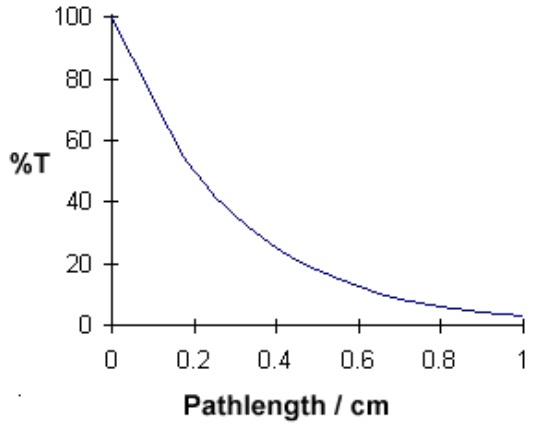
Absorbance, A, is the logarithm to the base 10 of the reciprocal of transmittance.

$$A = \log_{10} \frac{P_o}{P}$$

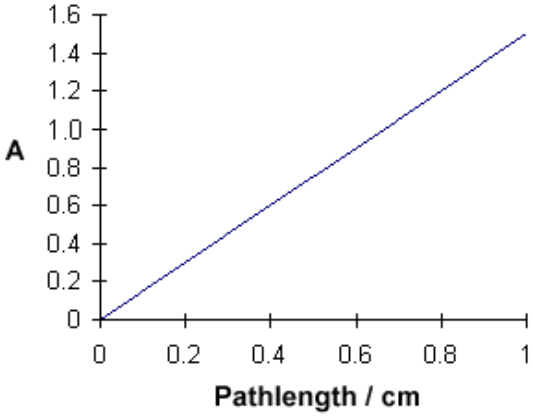
$$A = \log_{10} \frac{1}{T}$$

$$A = \log_{10} \frac{100}{\%T}$$

It was found by Lambert that the intensity of the transmitted light decreases exponentially as the length of the light path through the sample increases. Subsequently it was found by Beer that the intensity of transmitted light decreases exponentially as the concentration of the sample increases. These findings are illustrated below.



If the same data were plotted using absorbance instead of transmittance, a linear function is obtained.



These ideas are combined in the Beer-Lambert law which is expressed as:

$$A = abc$$

where

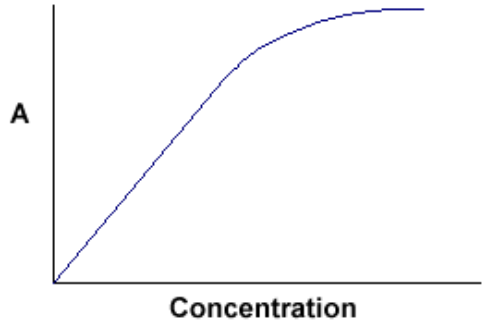
A = absorbance

a = absorptivity; this is a constant for a given substance. A compound with a high molar absorptivity is very effective at absorbing light of an appropriate wavelength. Hence low concentrations of such a compound can be easily detected.

b = length of the light path through the sample

c = concentration

Generally, the light path, b, is kept constant (ie: b = 1 cm) and thus Absorbance, A, is directly proportional to concentration, c. Hence, a graph of "Absorbance versus concentration", which is known as a standard curve, should yield a straight line passing through the origin (0,0).



Note: At high concentrations, Beer-Lambert law deviates from linearity. The deviation from linearity will not be dealt with in this course.

Experiment 5: Water Analysis (Colorimetry/Spectrophotometry)

Introduction: Colorimetry | Spectrophotometry | Lab Introduction
Procedures: Standards & Sample Preparation | Determination of λ_{max} | Analysis of Sample
Datasheet #1 | Datasheet #2

The reaction between ferrous ion and 1, 10-phenanthroline to form a red complex $(C_{12}H_8N_2)Fe^{2+}$ serves as a good sensitive method for determining iron. The intensity of the colour is independent of pH in the range of 2 to 9. The complex is very stable and the colour intensity does not change appreciably over very long periods of time. Beer's Law is obeyed.

The iron must be in the ferrous state, and hence a reducing agent is added before the colour is developed. Hydroxylamine, as its hydrochloride, can be used, the reaction is

$$2\ Fe^{3+}\ (aq) + 2\ NH_2OH\ (aq) + 2\ OH^- \rightleftharpoons 2\ Fe^{2+}\ (aq) + N_2\ (g) + 4\ H_2O\ (l)$$

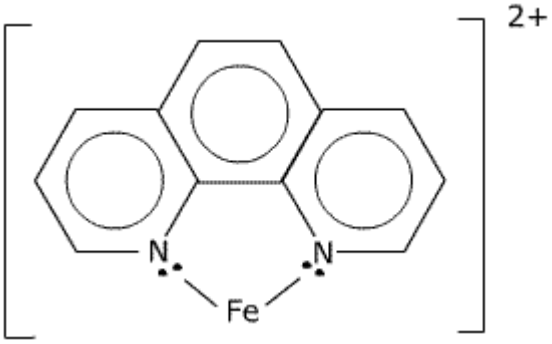
The pH is adjusted to a value between 6 and 9 by addition of ammonia or sodium acetate.

Then all Fe^{2+} is converted to an orange-red complex ion by the reaction with 1,10-phenanthroline. Note that 1,10-phenanthroline has two pairs of unshared electrons that can be used to form coordinate covalent bonds to the Fe^{2+} .

$$Fe^{2+}\ (aq) + 3\ Phen \longrightarrow [Fe(Phen)_3]^{2+}$$

orange red complex

The structure of $[Fe(Phen)_3]^{2+}$ is:



Experiment 5: Water Analysis (Colorimetry/Spectrophotometry)

Introduction: Colorimetry | Spectrophotometry | Lab Introduction
Procedures: Standards & Sample Preparation | Determination of λ_{max} | Analysis of Sample
Datasheet #1 | Datasheet #2

Procedure:

Preparation of Standards and Sample:

1. Prepare 5 standards, and label them Blank, Solutions A, B, C and D.
2. Using the following table as a guide, into five 100 mL Nessler tubes, pipet the follow solutions.Then make up to the 100 mL marks and let stand for 10 minutes.
3. Prepare an unknown river water sample using the following table as a guide. Then make up to the 100 mL mark and let stand for 10 minutes.

		Volume of standard iron solution (mL)	Volume of hydroxylamine solution (mL)	Volume of 1,10-phenanthroline solution	Volume of sodium acetate solution
Blank	-	0.00	1.00	10.00	8.00
Solution A	-	1.00	1.00	10.00	8.00
Solution B	-	5.00	1.00	10.00	8.00
Solution C	-	10.00	1.00	10.00	8.00
Solution D	-	25.00	1.00	10.00	8.00
Unknown River Water sample	10.00 unknown	-	1.00	10.00	8.00

4. Using the Nessler tube rack and the colour 'box', estimate the concentration of the unknown in ppm and g/L Fe^{2+} .

Procedure:

Determination of λ_{max} for the Analysis of Iron:

1. After the colour development of your standards, obtain the 'blank' and Solution C. Collect two clean cuvettes.
2. Adjust the wavelength control on the spectrophotometer to 400 nm.
3. Fill the first cuvette with the blank solution. Fill it to at least 3/4 full. Place the cuvette in the spectrophotometer and adjust absorbance to zero.
4. Acclimatize the second cuvette with Solution C and fill the cuvette with Solution C. Fill it to at least 3/4 full. Record the % Transmittance and Absorbance at this wavelength.

Rough scan:

5. Adjust the wavelength control to 425 nm. Zero the spectrophotometer with the blank and measure the % Transmittance and Absorbance with Solution C at this wavelength.
6. Repeat Step 5 by increasing the wavelength by 25 nm. Continue until λ = 575 nm.
7. Take a look at the data obtained from Step 5 and 6 and note this wavelength, we'll call this λ_1, where the maximum absorbance occurred. You will now perform a fine scan to zeroing in on λ_{max}.

Fine scan:

8. Adjust the wavelength control to (λ_1 - 25) nm. Zero the spectrophotometer with the blank and measure the % Transmittance and Absorbance with Solution C at this wavelength.
9. Repeat Step 8 by increasing the wavelength by 5 nm. Continue until λ = (λ_1 + 25) nm.
10. Plot a graph showing the absorbance as a function of the wavelength and a graph showing the %T as a function of the wavelength to the data recorded in Steps 8 and 9 to determine λ_{max}.

Procedure:

Analysis of the Sample for Iron Content:

1. With the wavelength set to the λ_{max} determined, use the blank solution and adjust the absorbance to zero.
2. Acclimatize the cuvette with Solution A. Fill the cuvette with this standard solution. Record the absorbance of the standard solution.
3. Repeat Step 2 with Solutions B, C and D.
4. Acclimatize the cuvette with the sample solution. Fill the cuvette with the sample solution. Record the absorbance of the sample solution.
5. Plot Absorbance versus Concentration and find the concentration of Fe^{2+} in the unknown river water sample.

Experiment 5: Water Analysis (Colorimetry/Spectrophotometry)

Introduction: Colorimetry | Spectrophotometry | Lab Introduction
Procedures: Standards & Sample Preparation | Determination of λ_{max} | Analysis of Sample
Datasheet #1 | Datasheet #2

Data Sheet:

Colour Comparison using Nessler Tubes

Estimated iron concentration of diluted unknown river water sample _____ ppm

Iron concentration of original river water sample _____ ppm

_____ g/L

Determination of λ_{max}:

ROUGH SCAN			FINE SCAN		
Wavelength λ (nm)	**Absorbance**	**% Transmittance**	**Wavelength λ (nm)**	**Absorbance**	**% Transmittance**
400					
425					
450					

475					
500					
525					
550					
575					

λ_{max} = _____

(Attach graph to support the determination of λ_{max}.)

Experiment 5: Water Analysis (Colorimetry/Spectrophotometry)

Introduction: Colorimetry | Spectrophotometry | Lab Introduction
Procedures: Standards & Sample Preparation | Determination of λ_{max} | Analysis of Sample
Datasheet #1 | Datasheet #2

Data Sheet:

Analysis of the Sample for Iron Content

Concentration of initial standard _____ g/mL

_____ ppm

Volume of standard used (mL)	Nessler Volume (mL)	Concentration of Fe in Nessler tube (ppm)	Absorbance
0.00	100.0		
1.00	100.0		
5.00	100.0		
10.00	100.0		
25.00	100.0		
Dilute river water sample	100.0		

Attach graph for the above tabulated data

Concentration of iron in diluted river water sample (from graph) _____ ppm

Dilution factor used in preparing the diluted sample _____

Concentration of iron in original river water sample _____ ppm

Concentration of iron in original river water sample _____ g/L

T&P 5: Ion Exchange and Complexometric Analysis Techniques

~~ALL TITRATIONS ARE DONE INDIVIDUALLY~~

Procedure: (For this lab, all pipets provided are clean - do not acclimatize)

Prepare the ion exchange column (Work in a group of 2)

1. Put a plug of cotton wool at the bottom of the column.
2. Add a suspension of resin in distilled water to the column.
   Note: The resin has been previously regenerated using 4 N HCl.
   Add resin with a graduated cylinder to get a height equivalent of 10 mL in the column.
3. Wash the column with about 50 mL of distilled water. Collect the **effluent** and use pH paper to check that the effluent is the same pH as the distilled water that you are using.
   Note: **Never let the resin go dry - always maintain a minimum of 0.5 to 1 cm head of liquid above the top of the resin.**
   Check the pH of the distilled water.

Prepare the sample (Work in a group of 2)

4. Pipet 50 mL of the bark extract solution in a beaker.
   NOTE: The solution is reddish-brown.
5. Place a large beaker below the column. Transfer the bark extract sample quantitatively to the column adjusting the flow so that the sample will go through the column and into the effluent beaker at about **1 drop per second**. This will allow time for the Mg^{2+} ions to exchange for H^{+} ions at the resin active sites.
6. Continue until the last of the sample sits about 0.5 cm on top of the resin. Discard the effluent.

   During this time, the standardization of the EDTA solution should be carried out (steps 14 -16) .
7. Rinse the beaker with two 10 mL portions of distilled water, allow to drip through the resin and finally add another 50 to 100 mL distilled water to the column. The final water flush can proceed at a faster rate (2 drops per second). Discard the effluent.
8. Continue washing until the effluent is clear.
9. Place a 250 mL **volumetric flask** under the column outlet. Add 150 mL of 4 N HCl to the separatory funnel. Drip the acid into the resin at about **1 drop per second for the first 20 mL**, gradually increasing the rate of elution until the full volume has passed into the resin.
10. Add about 50 mL of distilled water to the funnel and flush through the resin. All of the eluent is collected in the volumetric flask (approx. 200 mL collected). Dilute to the mark. Mix well.

11. **TITRATION (Steps 11 and 12 to be done INDIVIDUALLY)**:
    Place about 125 mL distilled water into a 500 mL Erlenmeyer flask. Pipet 25 mL of eluent the Erlenmeyer flask. In the fume hood, carefully add 15 mL of conc. ammonium hydroxide, then 2 drops of ammonium sulfide solution and mix gently. Record the initial volume and add about 3 mL of EDTA from the buret to the sample.
12. Add a small pinch of Manver-type indicator (Eriochrome black T/methyl red) and titrate with the EDTA solution to a pure blue colour. The titration must be finished as quickly as possible after the addition of the ammonia. Repeat the titration and aim for the same colour change.

Standardization of the EDTA Solution (Steps 13 to 17 to be done INDIVIDUALLY)

13. Acclimatize and fill the buret with the EDTA solution.

14. Place 125 mL distilled water in a 500 mL Erlenmeyer flask. From the microburet add 5.00 mL of

standard Mg solution (2.000 g/L), add 15 mL 4 N HCl and mix. In the fume hood, carefully add 15 mL conc. NH_4OH, add 2 drops ammonium sulfide, $(NH_4)_2S$, solution.

15. Mix gently. Add 3 mL of EDTA from the buret before adding the indicator.
16. Add a small pinch Manver-type indicator (Eriochrome black T/methyl red) and titrate with the EDTA solution to a pure blue colour. Do duplicates until you get good agreement.

    Aim for a light pink solution with the indicator to start (not an opaque solution, otherwiase it may be difficult to see the colour change) If the pink is too light, then the titration colour does not change to blue and stays green.

17. The titration must be finished as quickly as possible after the addition of the ammonia .

Experiment 6: Determination of Magnesium by Ion Exchange and Complexometric Analysis

Introduction | Procedure | Datasheet #1 | Datasheet #2

Objectives:

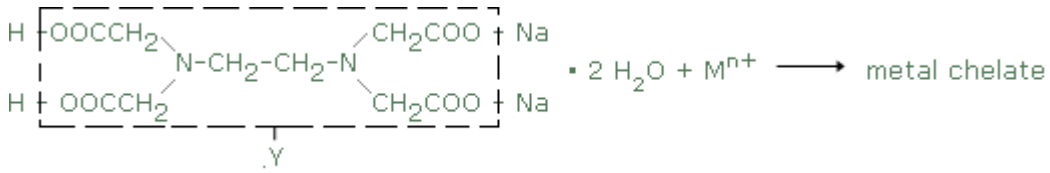
The student should be able to determine Magnesium in a natural sample using the ion exchange method where the Mg^{2+} ions adhere to a strong cation exchange resin while "organics" pass through and are removed. With this technique, the interfering elements are masked and the pH is controlled for selective complexometric titration with ethylene diamine tetra-acetic acid, EDTA. Skills which will be mastered by the student are: initial column preparation, the use of ion exchange column including removal of the **effluent** and quantitative control of the **eluent**, and the final indicator-colour selection.

Introduction:

In this method the metallic constituents are separated from the organic material with an ion exchanger. An aliquot of the eluent is titrated with a standard solution of ethylene diamine tetra-acetic acid, EDTA. Since a proprietary Eriochrome black T type indicator is used, calcium is also titrated. Iron is made unreactive with ammonia and metals soluble in this ammonical solution, but forming insoluble sulfides are made inert with ammonium sulfide.

The molecule known as ethylene diamine tetra-acetic acid, EDTA, forms a stable complex, chelate with metal cations. This method for Mg determination proves to be accurate and extremely fast in contrast to the usually time-consuming gravimetric analysis.

Iron, copper, manganese interfere with the titration in proportion to their concentrations. Abnormal amounts of these metals can be removed by sodium hydroxide precipitation and filtration.

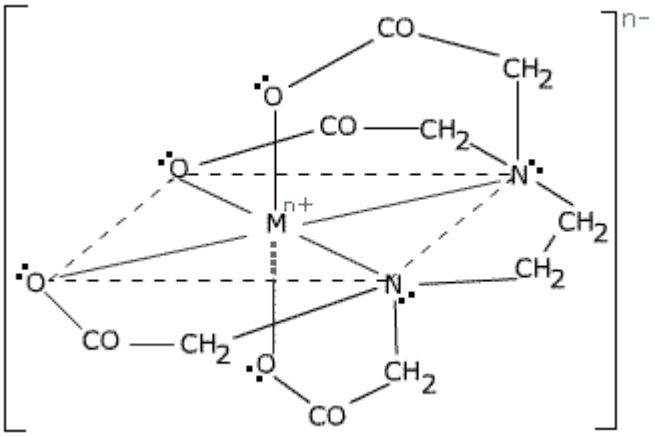


If we give the formula Na_2H_2Y (aq) to the above formula for EDTA disodium salt in aqueous solution containing the cations M^{n+}, the principle reactions taking place will be:

$$Na_2H_2Y\ (aq) \longrightarrow 2\ Na^+\ (aq) + H_2Y^{2-}\ (aq)$$

$$H_2Y^{2-}\ (aq) + M^{n+}\ (aq) \longrightarrow MY^{(n-4)+}\ (aq) + 2\ H^+\ (aq)\ n-$$

One mole of Na_2H_2Y is stoichiometrically equivalent to one mole of metallic cation irrespective of the charge on the cation. The structure of the resulting complex is probably as follows:



Hexadentate chelate of EDTA
(Probable structure of metal-EDTA complex)

The resin chosen (**REXYN 101H** or equivalent), a strong cation exchange resin, must be regenerated after previous use. Cations formerly adhering to the active sites are eluted by flushing with a high concentration of H^+ solution (4 N HCl), then removing any excess HCl with distilled water.

For the standardization of the ~ 0.02 M EDTA solution, which is provided, a standard Magesium solution (2.000 g Mg/liter) is provided.

Experiment 6: Determination of Magnesium by Ion Exchange and Complexometric Analysis

Introduction | Procedure | Datasheet #1 | Datasheet #2

Procedure:

Prepare the ion exchange column

1. Put a plug of cotton wool at the bottom of the column.
2. Add a suspension of resin in distilled water to the column.
   Note: The resin has been previously regenerated using 4 N HCl.
   Add resin with a graduated cylinder to get a height equivalent of 8 to 9 mL in the column.
3. Wash the column with about 50 mL of distilled water. Collect the **effluent** and use pH paper to check that the effluent is the same pH as the distilled water that you are using.
   Note: **Never let the resin go dry - always maintain a minimum of 0.5 to 1 cm head of liquid above the top of the resin.**
   Check the pH of the distilled water.

Prepare the sample

4. Weigh accurately close to 0.5 g of bark extract and dissolve in about 50 mL of distilled water in a beaker. The solution is reddish-brown.
5. Place a large beaker below the column. Transfer the bark extract sample quantitatively to the column adjusting the flow so that the sample will go through the column and into the effluent beaker at about **1 drop per second**. This will allow time for the Mg^{2+} ions to exchange for H^{+} ions at the resin active sites.
6. Continue until the last of the sample sits about 0.5 cm on top of the resin. Discard the effluent.

During this time, the standardization of the EDTA solution should be carried out (steps 14 -16) .

7. Rinse the beaker with two 10 mL portions of distilled water, allow to drip through the resin and finally add another 50 to 100 mL distilled water to the column. The final water flush can proceed at a faster rate (2 drops per second). Discard the effluent.
8. Continue washing until the effluent is clear.
9. Place a 250 mL **volumetric flask** under the column outlet. Add 150 mL of 4 N HCl to the separatory funnel. Drip the acid into the resin at about **1 drop per second for the first 20 mL**, gradually increasing the rate of elution until the full volume has passed into the resin.
10. Add about 50 mL of distilled water to the funnel and flush through the resin. All of the eluent is collected in the volumetric flask (approx. 200 mL collected). Dilute to the mark. Mix well.
11. Place about 125 mL distilled water into a 500 mL erlenmeyer flask. Pipet 25 mL of eluent into the Erlenmeyer flask. In the fume hood, carefully add 15 mL of conc. NH_4OH, then 2 drops of ammonium sulfide, $(NH_4)_2S$, solution and mix gently. Record the initial volume and add about 3 mL of EDTA from the buret to the sample.
12. Add a small pinch of Manver-type indicator (**Eriochrome black T/methyl red**) and titrate with the EDTA solution to a pure blue colour. The titration must be finished as quickly as possible after the addition of the ammonia. Repeat the titration and aim for the same colour change.
13. Using your results for this analysis and the subsequent results for the standardization of the EDTA solution, calculate the % w/w Mg on an oven-dried basis for the Bark Extract sample.

Standardization of the EDTA Solution - Acclimatize and fill buret with the EDTA soltuion

14. Place 125 mL distilled water in a 500 mL Erlenmeyer flask. From the microburet add 5.00 mL of

standard Mg solution (2.000 g/L), add 15 mL 4 N HCl and mix. In the fume hood, carefully add 15 mL conc. NH_4OH, add 2 drops ammonium sulfide, $(NH_4)_2S$, solution. Mix gently. Add 3 mL of EDTA from the buret before adding the indicator.

15. Add a small pinch Manver-type indicator (**Eriochrome black T/methyl red**) and titrate with the EDTA solution to a pure blue colour. Do duplicates until you get good agreement. The titration must be finished as quickly as possible after the addition of the ammonia.

    Aim for a light pink solution with the indicator to start (not an opaque solution, otherwiase it may be difficult to see the colour change) If the pink is too light, then the titration colour does not change to blue and stays green.

16. Calculate the value of the EDTA in terms of grams of magnesium per mL of EDTA.

Experiment 6: Determination of Magnesium by Ion Exchange and Complexometric Analysis

Introduction | Procedure | Datasheet #1 | Datasheet #2

Standardization of EDTA with Standard Mg Solution

Data Sheet

1. Concentration of standard Mg Solution _____

2. Volume from the microburet _____

3. Grams of Mg used per standardization _____

Volume of EDTA used:

Trial 1: **Trial 2:** **Trial 3:**

_____ _____ _____

Average: _____

Calculation:

Concentration of EDTA solution - grams of Mg / mL EDTA

Answer: _____ g Mg / mL EDTA

Experiment 6: Determination of Magnesium by Ion Exchange and Complexometric Analysis

Introduction | Procedure | Datasheet #1 | Datasheet #2

Determination of % Magnesium in Sample

Data Sheet

1. Weight of bark sample _____

2. % Moisture (wet basis) _____

Volume of EDTA used:

Trial 1: _____ **Trial 2:** _____ **Trial 3:** _____

Average: _____

Calculations:

1. Weight of oven-dried sample given the % moisture (wet basis)

Weight of oven-dried sample: _____ g (oven dried)

2. % Mg w/w (Remember to take into account the dilution you performed)

% Mg w/w: _____

Demonstrations of

Nine Practical Lab Techniques

Technique #1 – Use of a Burette

Technique #2 – Use of a Pipette

Technique #3 – Use of a Volumetric Flask

Technique #4 – Weighing

Technique #5 – Use of a Bottle-Top Dispenser

Technique #6 - Gravity Filtration, Vacuum Filtration

Technique #7 – Preparation of a Standard Solution

Technique #8 – Titration

Technique #9 – Rotary Evaporator

#1 - Techniques on the Use of a Burette

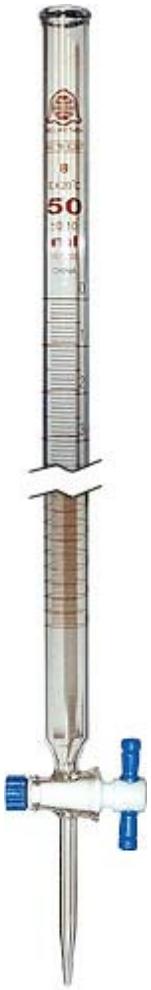


Figure 1: A 50 mL burette

A burette is used to deliver variable volumes of solution precisely and accurately (Figure 1). It is use for titrations, where the solution in the burette is called the ***titrant***. When the stopcock valve is aligned with the burette, as shown in Figure 1, the valve is in the open position and the titrant flows from the burette into the collecting vessel. Rotate the stopcock valve 90 degrees to close the valve. A commonly used burette in our lab delivers a total volume of 50 mL and is calibrated in 0.1 mL increments. This means that the finest division on the burette is 0.1 mL (Figure 2).

When reading the burette, the last digit is the digit where you estimate to within the finest division. Therefore, a burette reading of the 50 mL burette is recorded to two decimal places.

Steps to prepare and use a burette:

1. A burette is usually stored with distilled water and capped.

2. Empty the burette of distilled water from the top and check that the stopcock is liquid-tight. If the stopcock feels loose when it is turned, hold onto the stopcock handle and rotate the screw clockwise to tighten. The stopcock should rotate smoothly. If it doesn't, you should disassemble the stopcock to clean the stopcock and where it touches the glass surface of the burette. Reassemble the stopcock with all its parts as laid out in Figure 3.

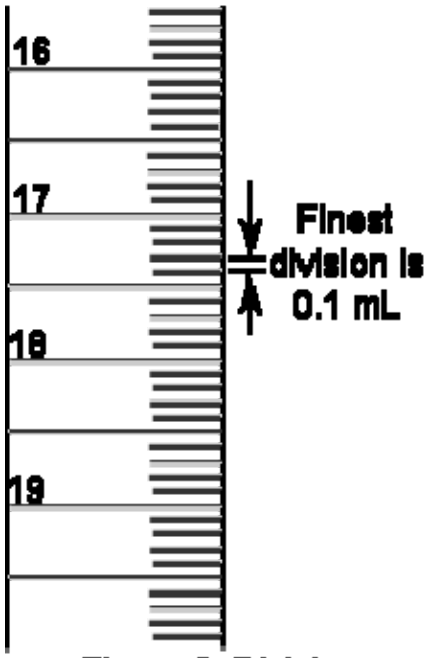


Figure 2: Division markings on a 50 mL burette

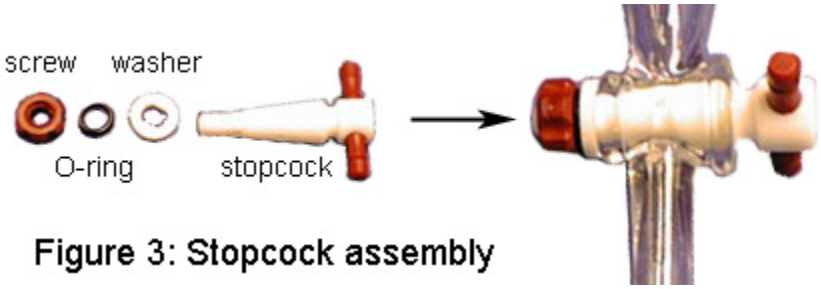


Figure 3: Stopcock assembly

3. Close the stopcock and using a small beaker, add approximate 10 mL of the titrant from the top. Hold the burette horizontally, rotate the burette to ensure the titrant touches the interior surface of the burette. Open the stopcock to allow the titrant to drain through the tip. Drain the rest of the titrant from the top of the burette. This step known as ***acclimatization*** is done at least twice.

4. Fill the burette well above the zero mark. Rotate the stopcock to let some solution flow through the tip.

5. Look for any trapped air bubble below the stopcock (Figure 4). To dislodge the air bubble, take your burette over to the sink, rapidly rotate the stopcock and allow small quantities of titrant to pass. Close the stopcock and check if the bubble is gone. If the bubble remains, open the stop stopcock and tap the burette near the stopcock, or give the burette with a quick up-and-down jerk. A combination of the above procedures should dislodge the trapped bubble (Figure 5).

Figure 4: Trapped air bubble

Figure 5: Air bubble removed

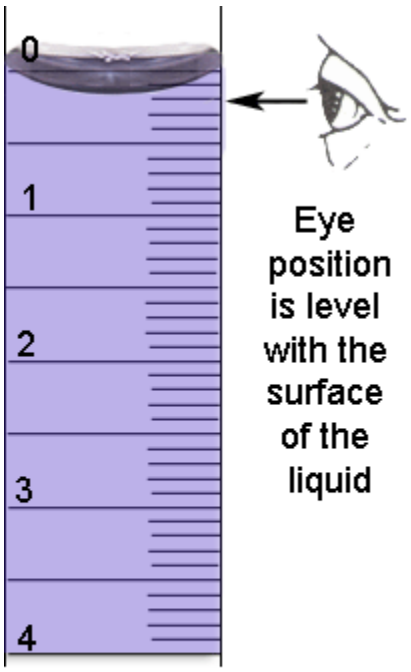


Figure 6: Initial reading of the burette. (eg - V_i = 0.20 mL)

6. Finally, refill the burette to above the zero mark. Lower the level of the titrant below the zero mark. The eye should be level with the meniscus for a proper reading. The correct reading is where the bottom of the meniscus touches the scale. Record the initial volume (V_i) to 2 decimal places (Figure 6).

7. Place a receiving flask (usually an Erlenmeyer flask) under the tip of the burette. Adjust the burette height so that the tip of the burette is inside the neck of the Erlenmeyer flask (Figure 25).

8. Dispense the titrant through the burette until a desired volume is reached or until a chemical analysis is complete. **Do not let the solution drain past the last mark on the burette**.

9. Record the final volume (V_f). The volume of solution dispensed is found by subtracting V_i from V_f.

Final Burette Reading, V_f (mL)	23.45
Initial Burette Reading, V_i (mL)	0.20
Volume delivered (mL)	23.25

10. To clean up, drain the solution from the top of the burette into a proper waste container. Rinse the burette 2-3 times with distilled water. Each time, open the stopcock to drain some of the rinse water through the tip and then empty the rest through the top of the burette.

11. Store the burette with distilled water.

#2 - Techniques on the Use of a Pipette

Pipettes are for the transfer of known volumes of liquid from one container to another. Pipettes that deliver a fixed volume are called volumetric or transfer pipettes (Figure 7a).

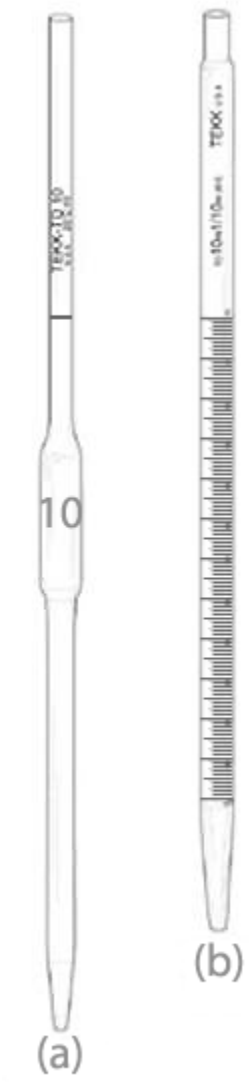


Figure 7: Pipettes

Other pipettes are known as measuring pipettes. They are calibrated with graduated markings along the side so that any volume up to the maximum capacity can be delivered (Figure 7b).

Liquids are drawn into the pipette through a slight vaacuum by using a pipette bulb (Figure 8). ***Never use your mouth to pipette***.

The volume of a volumetric pipette is recorded to two decimal places (i.e. – 10.00 mL). To read the volume of a measuring pipette, estimate the last digit to within the finest division on the pipette.

Figure 8: Different types of pipette bulbs

Steps to prepare and use a volumetric pipette:

1. Inspect the pipette to make sure that the tip is not chipped and the top end of the pipette is smooth and flat. If the top end of the pipette is chipped, or not smooth, it is very difficult to pipette.

2. To fill a pipette, a rubber bulb (Figure 8) is used to provide suction to pull up the solution. Keep the tip of the pipette below the surface of the liquid. It is easier to draw up solution from a smaller, narrower container (i.e. 50 mL beaker) to avoid sucking up air. ***Avoid sucking liquid into the pipette bulb***.
(**Note:** Never dip a pipette into the reagent bottle. Obtain the solution with a small clean and dry beaker. Pipette the solution out of the beaker.)

3. Acclimatize the pipette with the solution to be used in the analysis by drawing up a small amount of the solution. Remove the bulb and tilt the pipette horizontally to rinse the inner walls of the pipette. Drain completely. Repeat this step three times.

4. Fill the pipette with solution past the calibration mark. Remove the bulb and quickly place the index finger of the hand holding the pipette over the exposed end of the pipette.

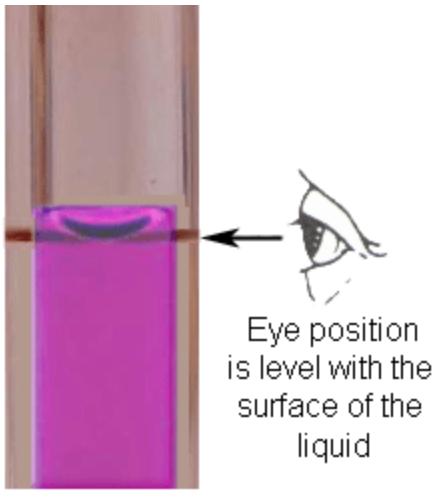


Figure 9: The bottom of the meniscus sits at the calibration mark

5. Tilt the pipette slightly and wipe away any liquid on the outside surface. Slowly release pressure on the index finger so that the bottom of meniscus approaches the calibration mark at eye level. At the mark, apply pressure on the index finger to stop the level of the liquid.

6. Touch the tip of the pipette on the wall of the container. This will drain any drop that remains on the pipette tip.

7. Transfer the pipette to the receiving container and release the pressure on the index finger. Drain the solution into the receiving container with the pipette tip touching the wall of the container. After draining, wait 10 seconds before removing the pipette. At this point, the calibrated amount of liquid has been transferred. Look closely at the tip of the pipette. A small portion of solution remains in the tip (Figure 10). ***Do not blow out the pipette***.

8. After use, clean the pipette by rinsing with distilled water.

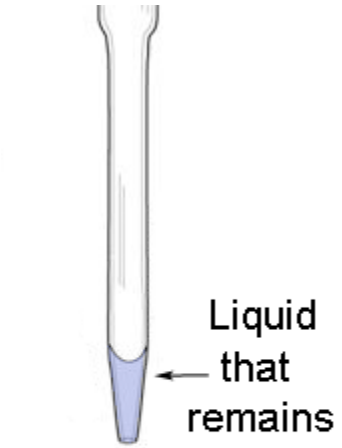


Figure 10: A small portion of the liquid remains.

#3 - Techniques on the Use of a Volumetric Flask

Figure 11: A 100.0 mL Volumetric flask

A volumetric flask is calibrated to contain one specified volume. It is used to prepare standard solutions and for the dilution of solutions. Volumetric flasks are cleaned and thoroughly rinsed but rarely do they need to be dried. When the meniscus touches the mark that is etched on the neck of the flask, the calibrated volume is contained in the volumetric flask.

When preparing a solution with a solid solute, the solid should not be emptied directly into the volumetric flask. First, dissolve the solid in a beaker using about half to two-thirds of the volume of the final solution. Second, quantitatively transfer the solution to the volumetric flask. Details of preparing a standard solution and quantitative transfer are covered in *Technique #7*.

The volume measured in a volumetric flask is recorded to four significant figures (i.e. – 100.0 mL).

Steps to make a Dilution using a Volumetric Flask

1. Pipet the solution to be diluted into the volumetric flask directly.
2. Add distilled water to fill the flask about half to two-thirds full.
3. Swirl the flask to mix the solution.
4. Bring the level close to the mark and allow time for drainage. Then use a Pasteur pipette to make the final addition (Figure 12). The eye should be level with the meniscus and the mark to make a correct reading for the volume (Figure 13).

Figure 12: Pasteur pipette

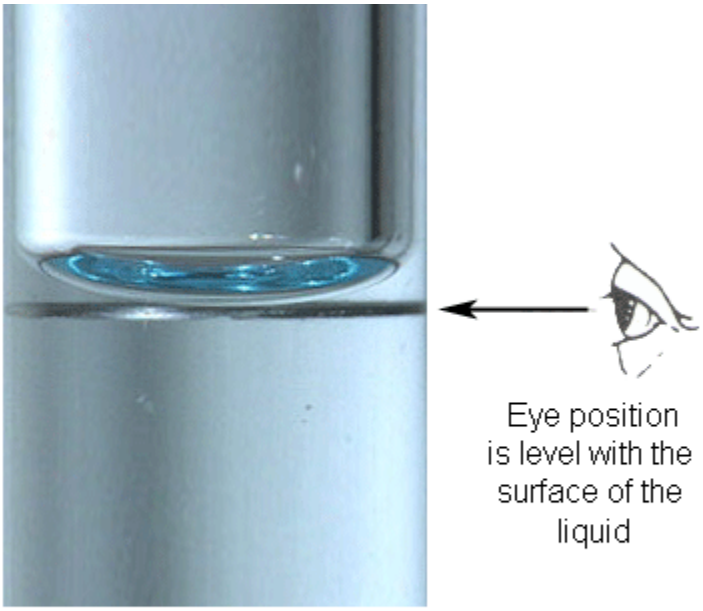


Figure 13: Fill the volumetric flask such that the bottom of the meniscus touches the mark on the neck of the flask

6. Firmly stopper the flask and invert repeatedly (at least 15 times) to assure uniform mixing.

#4 – Techniques on Weighing

Figure 14: An electronic analytical balance

There are many different types of balances or scales available to measure the mass of a sample. The selection of the balance depends on the mass of the object or sample and the precision needed for the measurement. A top-loading balance is used to determine the approximate mass of the sample needed. In this course, we will be mostly using electronic analytical balances (Figure 14). These balances are easy to operate and are capable of measuring to 0.0001 g. Models of these balances vary in the labs. Consult your instructor and operating manual specific to the model of the balance.

There are two methods of weighing:

1. Weighing by difference – This is a technique which is used when it is important to know the precise amount of the sample that has been transferred to a reaction mixture.
2. Weighing by taring – This technique is used when the mass of the empty container is not important. The empty container is 'zeroed' or ***tared*** on the balance.

These two methods will be described in detail below.

The Balance Room

Figure 15: The balance room

Balances are sensitive to drafts, changes in temperature, or the vibrations caused by moving people. The balances are stored in a separate room to minimize these variables and are placed on concrete tables.

Balances are very expensive and are sensitive to attack by corrosive chemicals. ***Do not take liquid into the balance room.*** When possible, chemicals should be added to the weighing container outside of the balance chamber. During the weighing process, the

weighing container should be placed on a clean surface, such as a kimwipe, so that the bottom of the container does not pick up any dust. It is important that you clean up all chemical spills. If in doubt consult your instructor.

Weigh boats (Figure 16) are disposable containers used for weighing. They are made of polypropylene plastic and are inexpensive. A used weigh boat should be discarded in the waste container. All chemicals and spatulas that are used should be returned to their proper places. Depending on the experiment, other types of weighing container could be a porcelain crucible (Figure 17), an aluminum plate (Figure 18) or a small beaker.

Figure 16: Weigh boat

Figure 17: Crucible with lid

Figure 18: Aluminum containers

The balance room must be kept tidy. Materials taken into the balance room include datasheets, pen and the sample to be weighed. Enter mass measurements directly on the datasheet with your pen. Before you leave your balance, make sure:

1. The balance and the area around it is clean. Spills inside the balance should be brushed off using the brush on top of the balance. Spills on the concrete table should be cleaned using Kimwipes.
2. Close all the doors of the balance.
3. Turn off the balance.

Weighing by difference

1. Pre-weigh an approximate quantity of the sample into a weigh boat using a top-loading balance.
2. Record all the masses for each step directly on the datasheet in pen.
3. Turn on the balance by pressing on the control bar. After a few seconds, the display will read 0.0000.
4. Open the sliding glass door (on the side) and place the sample/weigh boat on the balance pan. Close the sliding glass door and wait until the reading is stable. Record the value.
5. Transfer the sample into a beaker.
6. Weigh the emptied weigh boat on the analytical balance. Do not brush off any sample particles from the emptied weigh boat. Record the mass of the emptied weigh boat.

7. The difference between the two weighings is the mass of the sample transferred into the beaker.

Datasheet for Weighing by difference

1. Mass of weigh boat and sample _____ g

2. Mass of emptied weigh boat _____ g

3. Mass of sample transferred _____ g

Weighing by taring

1. Place a weigh boat on the balance pan. Close the doors and wait for the reading to stabilize. Press briefly on the control bar or the tare button and the display changes to 0.0000 g. The weight of the weighing boat is now tared.
2. Remove the weigh boat from the balance and set it on a piece of kimwipe. With a spatula, carefully add the sample to the weigh boat. Place the weigh boat back on the balance pan. Close the doors and wait for the reading to stabilize. Record the mass of the sample.

Datasheet for Weighing by taring

1. Mass of weigh boat and sample _____ g

2. Mass of empty weigh boat 0.0000 g

3. Mass of sample _____ g

#5 – Techniques on the Use of a Bottle-Top Dispenser

Figure 19:
A Bottle-Top Dispenser

A bottle-top dispenser is a hand operated pump that screw on to a reagent bottle. These dispensers are surrounded by a clear plastic sleeve, which protects the glass cylinder from breaking, and eliminates the risk of hazardous spills. The volume is accurate to about 1-2%. The volume to be dispensed is set by adjusting the volume knob on the plunger.

Steps to Use a Bottle-Top Dispenser

1. Prior to use, check the dispenser to see whether the dispenser volume is set correctly.
2. Make sure that the reagent bottle has enough solution before mounting the bottle-top dispenser on the reagent bottle.
3. Place a graduated cylinder at the outlet.
4. Pull the top of the dispenser up to as far as it will go. Let go of the dispenser and let the plunger fall by gravity. The liquid should be collected in a graduated cylinder.
5. While the liquid is dispensing into the graduated cylinder, observe that there is no air bubble in the liquid. If air bubbles are present, it could be that the container is near empty or the dispenser is malfunctioning. Consult your instructor to refill the solution and check the mounting of the dispenser.
6. Read the graduated cylinder and make sure that the volume collected is within 2% of the preset volume.
7. Place a clean, empty container at the outlet.
8. Repeat steps 3 and 4 to dispense the solution into the container.

#6 – Techniques on Gravity Filtration

There are two general methods of filtration: gravity and vacuum. In gravity filtration, the filtrate passes through the filter medium under the force of gravity and the capillary action between the liquid and the funnel stem.

There are several varieties of filter paper. Good filtration depends on the retention of the filter paper and the speed of the filter paper. Usually, fast papers will retain coarse particles. Slow papers will retain fine particles. The optimum choice is a paper, which is as fast as possible, yet retains all visible particles, and thus giving a clear filtrate.

Low-ash or ashless quantitative-grade papers can be ignited without leaving an ash. The residue left by an 11-cm circle of a low-ash paper may be as low as 0.06 mg. An ashless-grade paper typically leaves 0.05 mg or less from an 11-cm circle. This small mass is considered negligible in most analytical procedures.

Decantation is a process used to separate the liquid from the mixture to be filtered. To decant a liquid from a solid, in one hand hold the beaker that has the mixture in it. Hold a glass-stirring rod in the other hand. Touch the lip of the beaker to the glass rod and pour the mixture to be filtered using the glass rod as a guide to pour slowly to ensure that the solid is not carried along. This also prevents the liquid from running back along the outside of the beaker.

Washing of the solid to remove soluble impurities follows the decantation of the supernatant liquid. Use a small amount of wash liquid and mix it thoroughly with the solid. Allow the solid to settle and decant the wash liquid through the filter. Repeat this procedure several times. Several washings with small volumes of liquid are more effective in removing soluble contaminants.

Transfer the washed solid in the filter funnel is the final step. The bulk of the solid is transferred to the funnel by a squirting a stream of wash liquid from a wash bottle. The last traces of the solid are removed from the walls of the beaker by scrubbing with a rubber policeman. Rinse the beaker and rubber policeman and transfer the rinse liquid to the funnel. Repeat this at least two times.

Step to prepare a gravity filtration

1. It is important to use the correct size filter paper. Properly sized filter paper should stop just below the rim of the glass funnel. As a guide, use filter paper whose diameter is about 1 cm less than twice the diameter of the funnel, for example a six-centimeter diameter funnel uses a filter paper of eleven-centimeter diameter. The filter paper should sit a few millimeters from the rim of the funnel (Figure 20).

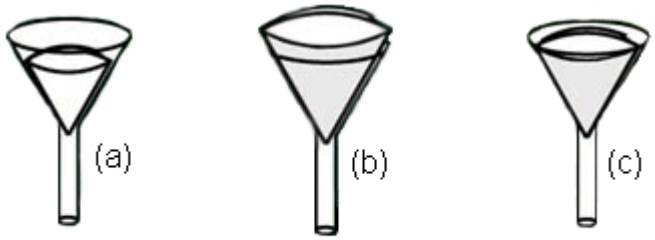


Figure 20 - Correct size of filter paper is (c)

2. Fold the filter paper by referring to Figure 21.

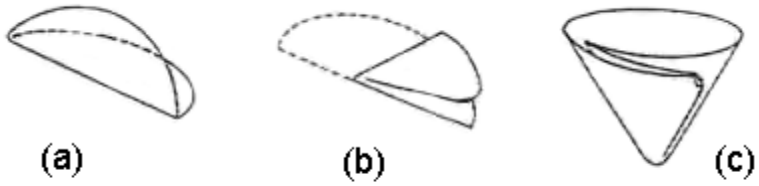


Figure 21 - Folding a filter paper

   (a) Fold the filter paper in half.
   (b) Fold the filter again to within about 10^{o} of a 90^{o} fold. The second fold is not exactly at a right angle. Tear off the corner of top fold.
   (c) Open the filter paper so that the torn corner is on the outside of the cone. The tear enables the paper to stick better to the funnel.

3. Place the folded filter paper snugly into the funnel by moistening the filter paper with the solvent of the mixture to be filtered. This should ressemble Figure 20 (c).

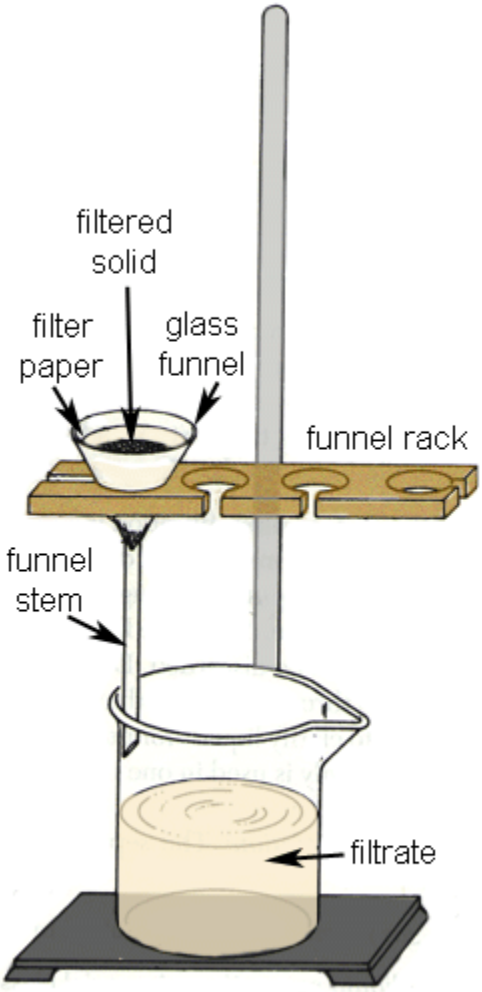


Figure 22 - A gravity filtration setup

4. Press the filter paper against the top wall of the funnel to form a seal. Support the funnel with a funnel rack.

5. Set up the gravity filtration apparatus as per Figure 22. Ensure that the funnel rack is positioned so that the funnel stem is inside the beaker. Position the beaker so that the funnel stem is touching the side of the beaker to avoid splashing.

6. Allow the mixture to settle and then ***decant*** the liquid from the solid. ***Wash*** the solid which remains in the beaker several times. Finally, ***transfer the washed solid*** to the funnel.

Techniques on Vacuum Filtration

This type of filtration is used with water or high-boiling organic solvents and is much faster than gravity filtration. For the set-up (Figure 22a), a filter flask must be clamped in position before attaching the rubber tubing, rubber stopper (adapter) and Büchner funnel (or crucible). This prevents the top-heavy apparatus from toppling over and spilling material. A medium- or slow-speed filter paper is used that is wetted with the solvent before the vacuum is applied with the water aspirator. Use a large beaker under the aspirator to minimize splashing. Check that there is a good seal between the apparatus when vacuum is applied before filtering the sample.

During the filtration, the mixture should be poured at a rate that the bottom of the funnel is covered with some solution. The collected crystals/precipitate can be washed with some chilled solvent. Do not discard the mother liquor (in the filter flask) as more compound can be recovered.

Figure 22a - A vaccum filtration setup

Caution: Running water can be sucked back into the filtration apparatus if the water pressure decreases. Be sure to break the vacuum by disconnecting the tubing at the aspirator before turning off the running water.

#7 - Techniques on Preparation of a Standard Solution

A ***primary standard*** is a substance that has a known high degree of purity, a relatively high molar mass, is non-hygroscopic and reacts in a predictable way. A ***standard solution*** is a solution having a very well known concentration of a solute. A measured volume of the standard solution then reacts with the substance being analyzed.

Potassium Hydrogen Phthlate ($KHC_8H_4O_4$) is a good primary standard because of the following properties:

1. High purity
2. Low toxicity
3. High molar mass (204.23 g/mole)
4. Soluble in water
5. Stable in air
6. A monoprotic acid and reacts rapidly with a base

When a standard solution of Potassium Hydrogen Phthlate is properly prepared, it can be used to determine the concentration of a sodium hydroxide (NaOH) solution. This process is referred to as the standardization of a solution.

Quantitative Transfer

Quantitative transfer is a technique, which ensures that in the transferring of the sample from one container to another, the ***entire*** sample is transferred. For example, a solid sample, which has been weighed in a weigh boat (container 1), is to be transferred to a beaker (container 2). In the transfer process:

1. Pour the sample from container 1 into the container 2. Depending on the type of container being used, sometimes tapping the container lightly, such as a weigh boat, will help knock the particles into the beaker.
2. Using a water bottle filled with distilled water, rinse container 1 thoroughly with distilled water.
3. Pour the rinse water into container 2.
4. Repeat steps 2 and 3 three times.

Steps to prepare a standard solution of Potassium Hydrogen Phthlate

1. Dry the Potassium Hydrogen Phthalate at 110^{o}C for several hours. Remove the $KHC_8H_4O_4$ and let cool in a dessicator.
2. Calculate the mass of $KHC_8H_4O_4$ that is required and determine the final volume of the solution, V_f, being prepared. Ensure that you have a volumetric flask of volume V_f available.
3. Obtain the glassware needed:
    a. A beaker which will hold approximately two-thirds the volume of the final solution.
    b. A volumetric flask which will hold the final volume, V_f, of the solution.
4. Using a weigh boat, weigh the $KHC_8H_4O_4$ to 4 decimal places on an analytical balance (See *Technique #4 for Techniques on Weighing*). In the steps to follow,

make sure that the level of accuracy is maintained when transferring reagents from one container to another.

5. Follow the steps on ***quantitative transfer***, and transfer the entire sample of $KHC_8H_4O_4$ from the weigh boat to the beaker.
6. Add distilled water to the beaker to dissolve the $KHC_8H_4O_4$ sample. The amount of distilled water should be about half to two-thirds of the final solution volume, V_f.
7. Using a stir rod, stir until the $KHC_8H_4O_4$ crystals are dissolved. Inspect the beaker closely.
8. Pour the solution from the beaker into the volumetric flask. (**Note:** A small funnel may be used if you are uncomfortable with pouring from the beaker.)
9. Follow the steps on ***quantitative transfer***, and transfer the solution from the beaker to the volumetric flask. (Note: If a funnel is used in this step, the funnel must be rinsed 3 times. All the rinse water must be poured into the volumetric flask.)
10. Follow *Technique #3 – Techniques on the Use of a Volumetric Flask* starting at Step 2 to finishing preparing the standard solution to a final volume of V_f.

#8 – Techniques on Titration

Titration is a method of analysis that allows you to determine the endpoint of a reaction. To perform a titration accurately, a precise quantity of the ***titrant*** needs to be dispensed into the reaction flask.

Figure 23 - An Erlenmeyer flask

In a titration, the reaction flask is usually an Erlenmeyer flask (Figure 23). It is used because it is conical in shape. This shape makes it easy to swirl the flask without spilling. For example, in an acid-base reaction, you may choose to pipette the acid into the Erlenmeyer flask. The base is introduced to the acid with the use of a burette. The solution in the burette, in this case, the base, is known as the titrant. A ***colour indicator*** such as ***phenolphthalein*** is introduced into the Erlenmeyer flask to detect the ***endpoint*** of the reaction. In acidic solution, phenolphthalein is clear in colour. In basic solution, phenolphthalein turns pink. The endpoint of the reaction is reached when the solution turns pink and the pink colour does not disappear after 30 seconds.

Steps in performing a titration

1. Follow the steps in *Technique #1 – Techniques on the Use of a Burette* and fill the burette with the titrant, the base.
2. Take an initial volume burette reading and enter it on the datasheet.
3. Follow the steps in *Technique #2 – Techniques on the Use of a Pipette* and pipette the acid into the Erlenmeyer flask (Figure 24).
4. Set up the burette and Erlenmeyer flask such that the tip of the burette is inside the neck of the Erlenmeyer flask (Figure 25). This ensures that all the base will be dispensed into the Erlenmeyer flask.

Figure 24 - Pipette the acid into the Erlenmeyer flask

Figure 25 - A titration setup

5. Add a few drops of colour indicator, such as phenolphthalein, to the acid solution. At the start of the titration, the acid solution in the Erlenmeyer flask should be clear. Add the titrant to the titration flask slowly and swirl the flask frequently. When the titrant touches the acid solution, the solution briefly turns pink in colour (Figure 25). Upon swirling, the pink colour will go away. Slow down the addition of the titrant when the trail of pink colour is taking longer to go away. Reduce the volume of the additions as the titration progresses. When you are near the ***endpoint***, the titrant should be added a drop at a time.
6. When it is judged that only a few more drops are needed, rinse down the walls of the Erlenmeyer flask. Quickly spin the closed stopcock 180^{o}. This allows a small shot of titrant to shoot out.
7. When the volume of titrant to be added is judged to be less than one drop, open the stopcock so that only part of a drop appears. Close the stopcock and touch the drop on the side of the Erlenmeyer. Use the wash bottle to rinse the partial drop into the Erlenmeyer flask with swirling.
8. The endpoint is reached when the colour change does not disappear after 30 seconds. The phenolphthalein colour change is from clear to pale pink (Figure 26).

1.

Figure 26 - The colour change observed at the endpoint of an acid-base titration. Flask#1 is the desirable colour change. Too much base has been added to flask #2.

9. Read the final burette volume. The difference between the initial and final readings on the burette is the volume of base used in the titration.
10. Repeat a titration at least twice. The burette volumes should be within +/- 0.10 mL or less.

Datasheet for a Titration

Trial 1:

1. Final burette reading _____ mL

2. Intitial burette reading _____ mL

3. Volume of titrant used in titration _____ mL

Trial 2:

1. Final burette reading _____ mL

2. Intitial burette reading _____ mL

3. Volume of titrant used in titration _____ mL

Trial 3:

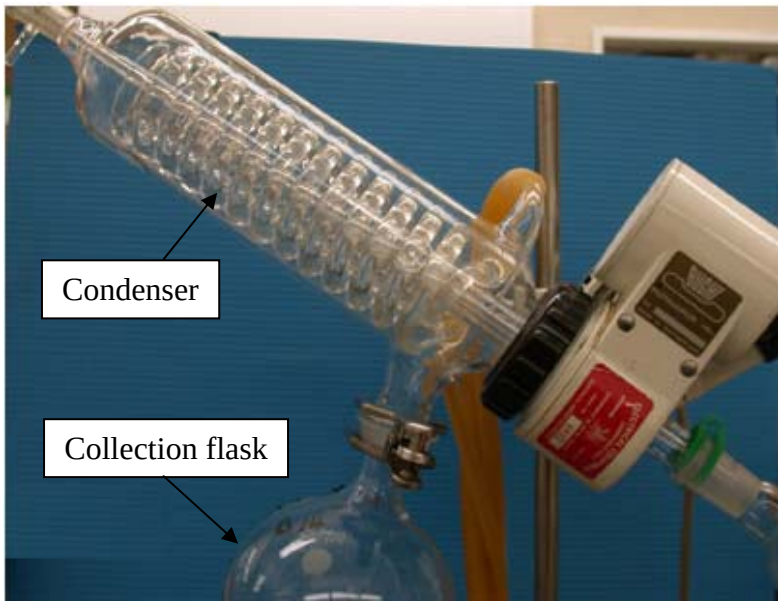
1. Final burette reading _____ mL

2. Intitial burette reading _____ mL

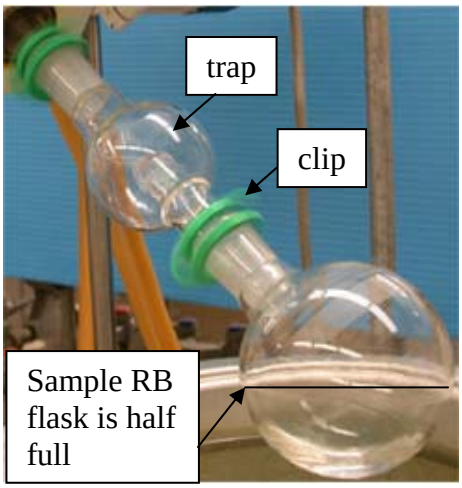
3. Volume of titrant used in titration _____ mL

#9 – Rotary Evaporator

The rotary evaporator is a piece of equipment that is designed to allow you to distill a liquid under conditions of reduced pressure. Since the pressure within the system is reduced, it means that the liquid can be distilled at a lower temperature than it would at atmospheric pressure.

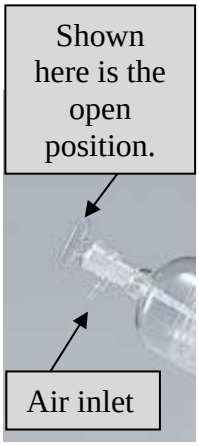


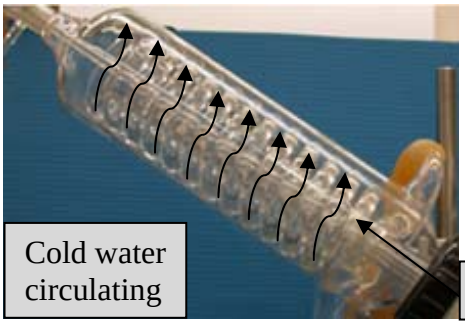
This is a very safe and fast method of distilling flammable solvents like dichloromethane. Dichloromethane normally boils at 40^{o}C at atmospheric pressure.



The sample to be "rotovapped" is added into the round bottom (RB) flask. The sample RB flask should never be more than half-full with liquid when it is attached to the rotary evaporator. The sample RB flask is connected to the trap and is secured using a circular clip. The clip has two sides. It is always oriented with the smaller circle on top.

The stopcock that is fitted at the top of the condenser needs to be in the closed position. To close, turn the stopcock such that it is cross-wise (90 degrees) to the air inlet. Turn on the water aspirator to evacuate the system.

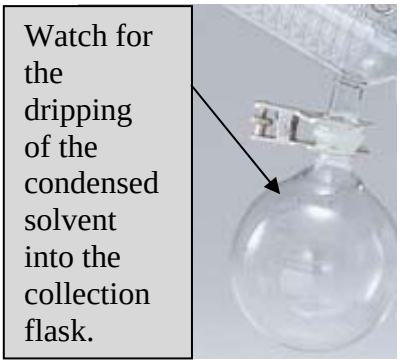




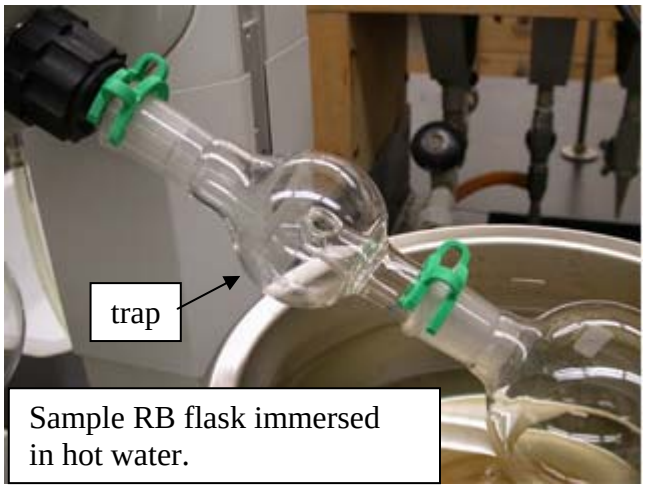
Ensure that the condenser has cold water running through the glass coil.

Vapour enters the distillation tube.

When the vapour enters the distillation tube inside the condenser, it will be in a cooled environment for the vapour to condense and drip into the large round collection flask to which it is attached.



Watch for the dripping of the condensed solvent into the collection flask.



Sample RB flask immersed in hot water.

There is a splash trap at the end of the distillation tube to prevent unwanted sample from splashing up into the condenser.

To speed up the rate of evaporation, the liquid sample can be warmed with hot water. Lower the flask by sliding the "Push" knob below the water bath. The temperature can be set using the dial on the side of the water bath.

To ensure even heating of the sample RB flask, turn the rotary dial to spin the sample RB flask that is immersed in the hot water bath.

Observe the condensation of vapour near the condenser. As the solvent condenses, you will see dripping of the condensed vapour collecting in the collection flask. Once the dripping stops, wait another minute and stop the rotary evaporator.

To remove the sample RB flask, disassemble by reversing the steps. Raise the sample RB flask from the hot water bath. Turn the rotary dial to stop the sample RB flask from spinning. Turn the stopcock that is fitted at the top of the condenser so that it is in-line with the air inlet. This will let air back into the system, and you should hear the sound of air rushing into the system. Turn off the water to the aspirator and the condenser. Remove the clip that is used to secure the sample RB flask. Gently give the neck of the sample RB flask a twist and remove the flask from the rotary evaporator.

Steps in operating the rotary evaporator

1. Turn power switch on. Turn water bath on. Adjust water bath to desired temperature.
2. Turn on water to condenser (slow to moderate flow).
3. Turn on aspirator full counterclockwise.
4. Attach the splash trap to the vapour tube and secure with clip.
5. Attach the sample RB flask to the splash trap and secure with clip.
6. Adjust rotational speed of the flask.
7. Turn the air inlet valve to the closed position to obtain a vacuum.
8. Slide the "Push" knob down to unlock the lift mechanism to lower the sample RB flask into the bath.
9. Watch for the dripping of the condensed solvent into the collection flask at the condenser. After the dripping stops, wait another minute to ensure that the evaporation has finished.
10. To disassemble, slide the "Push" knob to raise the flask.
11. Turn the rotational speed knob to the off position.
12. Turn the air inlet valve to the open position to re-establish atmospheric pressure.
13. Remove the clip and the sample RB flask.
14. Remove the clip and the splash trap.
15. Turn off aspirator, water to the condenser, water bath and the power switch.

Lab Exam #1: Assay of *m*-Toluic Acid by Titrimetry

Standardization of NaOH | Assay on m-Toluic Acid | Procedure | Part A,B Datasheet | Part C Datasheet

Introduction:

The amount of solute in solution is often determined by titrimetric method of volumetric analysis. The procedure involves the addition of a solution having a known concentration of solute (a standard solution) to a solution containing an unknown amount of solute until the reaction between the two solutes is complete. The point in the titration when the reaction is complete is called the equivalence point.

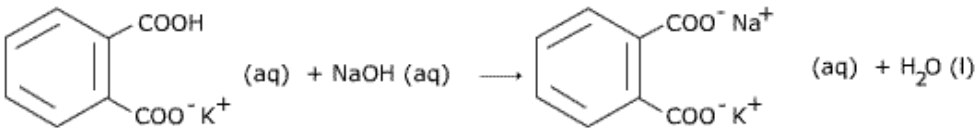
In this experiment, a sodium hydroxide solution of about 0.1 M is prepared and its exact concentration is determined in an acid-base titration. The equivalence point is detected using phenolpthalein as the indicator. Phenolpthalein is colourless in an acidic solution but pink (or red) in a basic solution (colour change occurs at pH 8.0 to 9.6). The point at which the phenolpthalein indicator changes colour is called the endpoint of the titration. Indicators are selected so that the pH at which the colour change occurs corresponds to the pH of the resulting solution at the equivalence point. The closer the point of colour change is to the pH at the equivalent point, the more accurate will be the titration.

Alkaline solutions (eg 0.1 M NaOH) should be protected from the atmosphere because it absorbs carbon dioxide and reacts with it:

$$OH^-\ (aq) + CO_2\ (g) \longrightarrow HCO_3^-\ (aq)$$

CO_2 absorption changes the concentration of a strong base over a period of time. It also decreases the extent of reaction near the endpoint in the titration of weak acids. If the alkaline solutions are stored in tightly capped polyethylene bottles, they may be used for about a week with little change in concentration.

Reagent grade sodium hydroxide may also contain sodium carbonate (from reaction with atmospheric CO_2 and water) and adsorbed water. Solutions of sodium hydroxide must be standardized against a primary standard. **Potassium hydrogen phthalate** (**KHP**) is among the most convenient primary standards for this purpose. It is a white, crystalline, nonhygroscopic, acidic solid with a high degree of purity. To determine the molar concentration of the NaOH solution, a measured mass of potassium hydrogen phthalate is dissolved in distilled water and the NaOH solution is added from a buret until the endpoint is reached. Sodium hydroxide reacts with potassium hydrogen phthalate according to the equation:



The amount (in moles) of potassium hydrogen phthalate titrated in the analysis can be calculated from the initial measured mass and the molecular weight (molar mass) of KHP.

$$\text{mass of KHP (g)} \times \frac{1 \text{ mole KHP}}{204.23 \text{ g KHP}} = \text{moles of KHP}$$

At the equivalence point, equal moles of KHP and NaOH have reacted.

$$\text{moles of NaOH} = \text{moles of KHP}$$

The molar concentration of the prepared NaOH solution is calculated from the moles of NaOH needed to react with the KHP and the volume of NaOH added from the buret.

$$\text{molarity of NaOH, M} = \frac{\text{moles of NaOH}}{\text{Volume of NaOH (liter)}}$$

Lab Exam #1: Assay of m-Toluic Acid by Titrimetry

Standardization of NaOH | Assay on m-Toluic Acid | Procedure | Part A,B Datasheet | Part C Datasheet

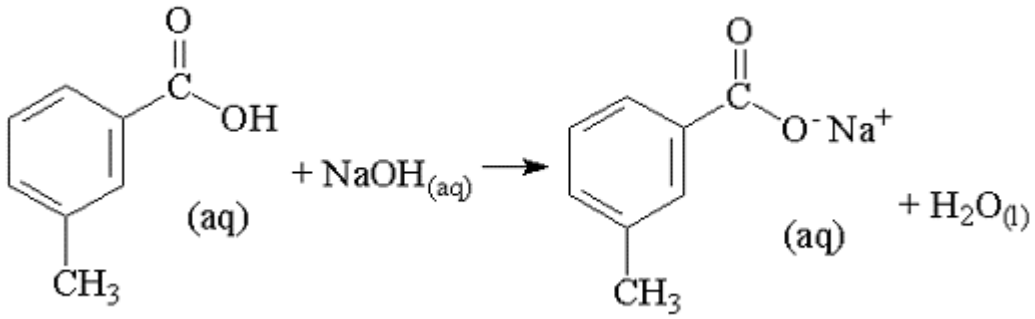
Introduction:

Assay on m-Toluic Acid Sample

In this experiment, the purity of m-toluic acid (MTA) from a commercial source is determined by titration with the standardized NaOH solution. This purity is known as an assay. Assaying of carboxylic acids by acid-base titrimetry is fast and convenient.

In the industrial production of m-toluic acid, m-xylene is oxidized with air in the presence of a cobalt catalyst. In a batch reaction, the reactor mass is analyzed by this method to determine the progress of the reaction and to determine when the reaction should be terminated and the next phase in the production process started. The final product is also assayed and the result of this assay is sent with the samples which are destined for shipment. The company that is purchasing the m-toluic acid sets the minimum specifications that the product must meet and it is the manufacturer's responsibility to ensure these specifications are met. The specifications are of concern to the purchaser because a large amount of impurities may affect the yield of a subsequent product or it may interfere with the effectiveness of the new product.

Assaying of m-toluic acid by titrimetry is carried out by adding standardized NaOH solution to a measured mass of m-toluic flakes dissolved in methanol until the endpoint is reached. The titration reaction involves the formation of water-soluble sodium m-toluate:



A blank titration is normally carried out to determine the volume of NaOH solution needed to neutralize any acidic impurities in the solvent. This blank volume is subtracted from the assay volume to give a corrected assay volume of NaOH solution.

From the corrected assay volume of NaOH solution and its molar concentration, the number of moles of NaOH added in the titration can be determined. From the balanced equation, one mole of MTA reacts with one mole of NaOH and thus the moles of MTA present in the titration is equal to the moles of NaOH added. The actual mass of pure MTA is calculated from the moles of MTA and its molecular weight.

moles of NaOH = (molarity of NaOH (M)) x (corrected volume of NaOH (L))

moles of MTA = moles of NaOH

$$\text{mass of MTA} = \text{moles of MTA} \times \frac{136.15 \text{ g MTA}}{1 \text{ mole MTA}}$$

The percentage of m-toluic acid in the sample is determined from the actual mass of pure MTA and the measured mass of the original sample.

$$\text{Assay (by titration) of m - toluic acid} = \frac{\text{mass of MTA (g)}}{\text{mass of sample (g)}} \times 100$$

Procedure:

Caution: The concentrated NaOH solution prepared in Part A - Step 1 can cause severe skin burns. Handle this solution with care. Wipe up any spills promptly and if you get any on your skin, rinse the affected areas immediately with cold water.

Part A - Preparation of 0.1 M NaOH Solution

1. Dissolve 3.0 g ± 0.1 g of sodium hydroxide pellets in 15 mL of distilled water in a 250 mL rubber-stoppered Erlenmeyer flask. Thoroughly mix and allow the solution to stand briefly for the precipitation of any sodium carbonate. Na_2CO_3 has a low solubility in a concentrated NaOH solution.
2. With a graduated cylinder, transfer 10 mL of the concentrated NaOH solution into a 500.0 mL volumetric flask. Do not transfer any of the Na_2CO_3 precipitate. Add distilled water to the mark and thoroughly mix the dilute NaOH solution.

Part B - Standardization of NaOH Solution

3. Clean a buret for titration. Acclimatize the buret by rinsing with three 5 mL portions of the prepared NaOH solution, making sure that the solution wets its entire inner surface including the tip. Fill the buret with the NaOH solution.
4. Measure 3 portions of 0.3 ± 0.05 g of dried potassium hydrogen phthalate (KHP) accurately to 4 decimal places in a clean, dry 125 mL Erlenmeyer flask. The KHP has been dried at 105^{o}C for several hours and allowed to cool in a desiccator.
5. Dissolve the KHP in 30 mL of distilled water and add 3 drops of phenolphthalein solution. The phenolphthalein solution has been prepared by dissolving 0.5 g of phenolphthalein in a 50 mL of ethanol and then adding 50 mL of water.
6. Titrate the KHP solution with the NaOH solution from the buret until the endpoint is reached. This occurs when a single half-drop causes the pink colour of the phenolphthalein indicator to persist for 30 seconds. Record the volume of NaOH solution added to an accuracy of ± 0.02 mL. Repeat two more times.
7. Repeat steps 5 and 6 at least two more times with accurately known amounts of potassium hydrogen pthlalate until the molar concentration of NaOH solution is within ± 0.001 M.

Part C - Assay of m-Toluic Acid Sample

8. Carry out a blank titration as follows. Add 15.0 mL (from dispenser) of methanol, 10 mL of distilled water, and 3 drops of phenolphthalein solution to a clean 125 mL Erlenmeyer flask. Titrate this mixture with the standardized NaOH solution (add half a drop at a time) until the endpoint is reached. Record the volume of NaOH solution added as the blank volume. This volume is to be subtracted from the assay volume obtained in step 11 to give the corrected assay volume. Repeat this step one more time.
9. Measure 0.3 g ± 0.04 g of the commercial m-toluic acid sample accurately to 4 decimal places in a clean, dry 125 mL Erlenmeyer flask.
10. Dissolve the MTA sample in 15.0 mL of methanol (reagent grade) and add 3 drops of phenolphthalein solution.
11. Titrate the MTA solution with the standardized NaOH solution until the endpoint is reached. Record the volume of NaOH solution added (assay volume) and determine the

corrected assay volume.

12. Repeat the titration (steps 9-11) at least once more with another 0.3 g of MTA. The reported assay is the average % m-toluic acid from at least two determinations.

Lab Exam #1: Assay of *m*-Toluic Acid by Titrimetry

Standardization of NaOH | Assay on m-Toluic Acid | Procedure | Part A,B Datasheet | Part C Datasheet

Data Sheet:

Part A - Preparation of 0.1 M NaOH Solution

Mass of sodium hydroxide pellets _____ g

Part B - Standardization of NaOH Solution

	Trial #1	Trial #2	Trial #3
Mass of KHP (g)			
Initial buret reading (mL)			
Final buret reading (mL)			
Volume of NaOH solution (mL)			
Concentration of NaOH solution (M)			

Average concentration of NaOH Solution = _____

Show sample calculation of the concentration of NaOH solution for Trial #1.

Lab Exam #1: Assay of *m*-Toluic Acid by Titrimetry

Standardization of NaOH | Assay on m-Toluic Acid | Procedure | Part A,B Datasheet | Part C Datasheet

Data Sheet:

Part C - Assay of m-Toluic Acid Sample

	Trial #1	Trial #2	Trial #3
Blank volume (mL)			
Mass of MTA sample (g)			
Initial buret reading (mL)			
Final buret reading (mL)			
Assay volume (final reading - initial reading) (mL)			
Corrected assay volume (mL)			
Mass of pure MTA* (g)			
Assay of MTA* (% m-toluic acid)			

Average assay of MTA = _____

* Show sample calculation of the actual mass of m-toluic acid contained in the sample and the assay of m-toluic acid for Trial #1.

Lab Exam #2: Determination of Copper in Water by Visible Spectrophotometry

Introduction | Procedure | Datasheet #1 | Datasheet #2 | Datasheet #3

Introduction:

A river water sample is analyzed for copper by visible spectrophotometry using a modern 'solid state' spectrophotometer. In order to do this analysis, the correct wavelength must be chosen, standard solutions must be accurately prepared and converted to the final coloured form.

The standard and sample solutions are firstly made acidic and then the copper (II) ion present is reduced to the cuprous (Cu^{+}) state. The solutions are then buffered to a pH of about 4.5 and the cuprous ions reacted with a complexing agent (bathocuproine disuphonate) which results in the formation of a yellow/orange-coloured complex. The intensity of radiation absorbed (absorbance) by each solution is measured using a visible spectrophotometer. By plotting a calibration curve of absorbance versus concentraction of copper (II), the concentration of copper (II) can be found in the unknown sample.

Notes to students:

In preparation for this experiment, the student should consider the following:

1. How to accurately prepare a series of standard solutions of known concentration from a stock solution (in this case a 50 ppm Cu(II) solution).
2. How to correctly use a graduated pipet.
3. How to correctly use the spectrophotometer for the determination of the optimum wavelength for analysis and for the recording of the absorbance of the standard and sample solutions.
4. How to correctly plot the required calibration graph of absorbance versus Cu (II) concentration in order to obtain the most accurate results.
5. How to calculate the concentration of Cu (II) in molarity given the concentration of Cu (II) in ppm.

Lab Exam #2: Determination of Copper in Water by Visible Spectrophotometry

Introduction | Procedure | Datasheet #1 | Datasheet #2 | Datasheet #3

Procedure:

1. Wash five 50-mL volumetric flasks several times with tap water and then rinse twice with distilled water. Ensure that they are marked 1 to 5.
2. Using the provided 2.00 mL graduated pipet, prepare a series of standards and a blank by adding the following quantity of stock 50.0 ppm copper (II) solution to the indicated flasks. After additions, bulk each flask to about 20 mL with distilled water.

Flask	1	2	3	4	5
Standard	1	2	3	4	5
Cu^{2+}	0.50 mL	1.00 mL	1.50 mL	2.00 mL	0.00 mL (blank)

3. Using automatic dispensers, add the indicated volumes of the following reagents to each of the five flask:

Hydrochloric acid (7%)	5 mL
Hydroxylamine hydrochloride (6%)	5 mL
Sodium citrate (20%)	8 mL
Bathocuproine (0.06%)	5 mL

   Make each flask up to the mark with distilled water and mix well.

   NOTE: The best results are generally obtained if you add the reagents in the order listed and mix well after each addition.
4. Use Standard #4, the blank solution (Standard #5) and the provided Bausch & Lomb Spectronic 21 spectrophotometer to record the visible absorption spectrum (Absorbance as a function of wavelength) of the yellow/orange copper bathocuproine complex.

   Choose a range of 400 nm to 550 nm. Initially change the wavelength by 25 nm each time and adjust the wavelength and then narrow down your search once you can see the general trend. You should aim to find the optimum wavelength to an accuracy of $\pm$ 5 nm. Remember to write down this wavelength and the absorbance on your data sheet.
5. Wash the 50-mL volumetric flask thoroughly with tap water and then rinse twice with distilled water. Using the microburets provided, measure 2.00 mL of the river sample into the volumetric flask. Bulk the flask to about 20 mL with distilled water. Add the appropriate reagents to the flask (as in step 3) and then make up to the mark with distilled water. Mix well.
6. Using the wavelength at which the absorbance is at a maximum value (λ_{max}) and the Bausch & Lomb Spectronic 21 spectrophotometer, measure the absorbance of the standards and sample solution from steps 3 to 5 using the blank solution (Standard #5) to set zero absorbance.

Lab Exam #2: Determination of Copper in Water by Visible Spectrophotometry

Data Sheet:

Solution spectrum - Copper bathocuproine complex

Determination of λ_{max}:

ROUGH SCAN			FINE SCAN	
Wavelength λ (nm)	Absorbance		Wavelength λ (nm)	Absorbance
400				
425				
450				
475				
500				

525			
550			

λ_{max} = _____

Lab Exam #2: Determination of Copper in Water by Visible Spectrophotometry

Introduction | Procedure | Datasheet #1 | Datasheet #2 | Datasheet #3

Data Sheet:

Absorbance values of standard and sample solutions

Solution	Cu concentration (ppm)	Absorbance
Standard #5		
Standard #1		
Standard #2		
Standard #3		
Standard #4		
Sample	----------	

Data Sheet:

Treatment of Data:

1. Plot a calibration graph (absorbance versus copper concentration) for the five copper standard solutions.
2. From the graph plotted, find the concentration of copper in the river water sample. Remember to take into account any dilution that you may have performed prior to the analysis.

Calculation of dilution factor:

Solution	Cu concentration obtained from graph	Dilution Factor	Original concentration (ppm)
Sample			

Calculate the molarity of the original river sample solution.

Atomic mass of copper is 63.546 amu